CANADA

DEPARTMENT OF MINES

HON. W. A. GORDON, MINISTER; CHARLES CAMSELL, DEPUTY MINISTER

MINES BRANCH

JOHN MCLEISH, DIRECTOR

INVESTIGATIONS

OF

FUELS AND FUEL TESTING

(Testing and Research Laboratories)

1932

Sections I, II, III, VI, VII and VIII, published separately as advance sections of "Investigations of Fuels and Fuel Testing, 1932; and section IX, as Memorandum Series No. 60.

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OTTAWA
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ERRATUM

Please note the information on page 35A herewith, which is to be inserted so as to follow page 35 of "Investigations of Fuels and Fuel Testing, 1932."

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Annual Reports on Mines Branch investigations are now issued in four parts, as follows:—

Investigations of Mineral Resources and the Mining Industry.

Investigations in Ore Dressing and Metallurgy (Testing and Research Laboratories).

Investigations of Fuels and Fuel Testing (Testing and Research Laboratories).

Investigations in Ceramics and Road Materials (Testing and Research Laboratories).

Other reports on Special Investigations are issued as completed.

MINES BRANCH INVESTIGATIONS OF
FUELS AND FUEL TESTING, 1932

GENERAL REVIEW OF INVESTIGATIONS

B. F. Haanel, Chief of Division of Fuels and Fuel Testing
R. E. Gilmore, Superintendent of Fuel Research Laboratories

During 1932 investigations on Canadian fuels were conducted on both laboratory and plant scales, and were of much the same nature as in the three previous years. Large-scale coking and storage tests on specially prepared "Princess" and "Waterford" coals from the Sydney area in Nova Scotia were continued at the Lasalle plant of the Montreal Coke and Manufacturing Company, and the results to date have been reported.* Plant-scale coking tests on Waterford, N.S., coal and also on selected English coals were made at the gas works of the Ottawa Electric and Gas Company, and burning tests on the resultant domestic cokes were conducted at the Fuel Research Laboratories. Carbonization assays were continued on Canadian bituminous coals at both high and low temperatures, according to standardized laboratory-scale procedures, and special washing tests by a standard sink-and-float method were made on selected coals from the Maritime Provinces, and from Alberta and British Columbia. The results of these washing and carbonization assay tests have been supplied to the interested parties, and will be reviewed in the annual report of "Investigations" at a later date, when the respective surveys have progressed farther. The results of special batch-scale briquetting tests conducted on petroleum coke breeze, on hardwood charcoal breeze, and on fines from British Columbia coals, were also supplied to the producers of these (waste) materials, and plant-scale briquetting tests on Welsh anthracite screenings were in progress and are continued into the new year.

A more complete list of the investigations conducted and an outline of the activities of the technical staff of the Fuel Testing Division in the laboratory and in the field, is given in the Annual Report of the Department of Mines for the fiscal year ending March 31, 1933. Certain of the investigations that were sufficiently advanced to put into report form for publication have been released from time to time as preprints. These are assembled hereunder, and, with additions, comprise Investigations of Fuels and Fuel Testing, 1932.

Solid Fuels Investigations

The first five reports, it will be noticed, deal with the chemical and physical properties of coals and coke, and Reports VI and VII with the hydrogenation of coal, coal tar, and bitumen. The purpose, scope and results of the respective investigations are briefly reviewed hereunder.

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* Recent investigations on the nature, preparation, storage and coking of typical coals from the Sydney area, Nova Scotia, by R. E. Gilmore and R. A. Strong—Bull. Can. Inst. of Min. and Met., July, 1933.

Anthracite and Coke Analysis Survey. The purpose of this survey was primarily to study what improvement in quality was taking place in the anthracites and domestic cokes marketed as household fuels in central Canada. Most of the coals and cokes examined were purchased on the open market in Ottawa; representative samples of each size of two of the three different brands of by-product cokes were obtained direct from the producing plants. The size of the samples collected varied from one hundred to two hundred pounds, but since each was from tonnages in stock, they were considered as representative of the respective household fuels on the market at that time. The results of this analysis survey showed that the quality of the Welsh and Scotch anthracites, which had been of high quality during recent years, had slightly improved in respect to both ash content and sizing. Likewise, the domestic cokes showed a general improvement to the extent that the ash contents of the by-product oven sizes did not vary beyond the limits of 6·5 to 9·0 per cent, and the average ash of the stove size marketed was 7·5 per cent. Of special interest is the appreciable improvement noticed in the quality of the American anthracite. The average ash content of the samples used in 1925 in burning tests in an experimental domestic hot water boiler, as referred to in the appendix of Section I, was 14·5 per cent, and the corresponding calorific value was 12,090 B.T.U. per pound. In comparison the average values for the stove-size anthracite sampled in the 1932-33 heating season were 9·5 per cent ash and 12,900 B.T.U. per pound. This means that the average figure of 11·39 pounds used in 1925 as the quantity of fuel fired per therm (100,000 B.T.U.) delivered to the cooling water, may be revised to read 10·67 pounds. With this change, together with a revision of the corresponding values for the other fuels, the following table may be prepared to show the present interrelation of the different fuels as compared with eight years ago.

—	Equivalent tonnage to 10 tons of American anthracite	
	1925	1932-33
American anthracite	10·00	10·00
Welsh anthracite	8·40	8·85
Scotch anthracite	8·55	8·85
Gas coke (Ottawa)	9·85	9·65
By-product coke	9·10	9·85

The F.R.L. (Fuel Research Laboratories) Method for Rating the Grindability or Pulverizability of Coal, Correlated with the "Cross" and "Hardgrove" Methods. The title of this report by C. E. Baltzer and H. P. Hudson is self-explanatory, and since its purpose, scope, and the results are summarized in its preface by the writers of this general review, it need not be further discussed here.

A Laboratory Test on Coals for Predicting the Physical Properties of the Resultant By-product Coke, by Swartzman, Burrough, and Strong, is an important contribution, and should be of service to the by-product coke industry. Especially is this so when it is read in conjunction with the report "Classification of Coals using Specific Volatile Index," by the same authors, as published in Investigations of Fuels and Fuel Testing, 1930-31. The laboratory test referred to in the title concerns a method for determining "swelling index" of a bituminous coal, which may be defined as the percentage of swelling of the coke button per unit of volatile matter obtained at a temperature of 600° C. when one gramme of finely ground coal is heated in a platinum crucible at this temperature. Chart III is the feature of the report.

In this chart, where the "swelling index" is plotted as abscissa against percentage volatile matter at 600° C. as ordinate, coking coals are arranged in thirteen sections, the physical characteristics of the cokes from which are known as a result of tests in the experimental by-product oven at the Fuel Research Laboratories, and in commercial coke oven plants. From the position of a given coal on the chart, the physical properties of its resultant by-product coke may be predicted with a fair degree of accuracy. Hence, by means of ordinary proximate analysis to determine the "specific volatile index" supplemented by special, though simple, swelling index tests, it is possible to examine, quickly and with little expense, a large number of coals or coal blends as to their comparative suitability for use in a by-product oven without going to the expense of large-scale tests. On the coal or coals thus selected, full-size plant tests may then proceed to satisfy the coke producer that the predictions made are entirely satisfactory for the peculiar requirements of his plant and market.

Reports IV and V, by Nicolls and Swartzman, are in the form of laboratory notes. The first of these reports is a continuation of former work on the "forms of sulphur" in Canadian coals as published in previous "Investigations of Fuels and Fuel Testing" for the years 1926 and 1928. As to the effect of weathering on the forms of sulphur, the general conclusion drawn is that dampness or wet air, in contact with finely divided particles of coal, promote a comparatively rapid oxidation of pyritic sulphur to sulphate sulphur, and that the presence of water seems to promote a decrease in the total sulphur content, and more particularly the organic form.

As the title suggests, Report V is concerned with ash corrections for the purpose of arriving at a "pure coal" basis for coal classification. The application to the coals of the Maritime Provinces of "1·1 ash = mineral matter" originated by Stansfield for Alberta coals, and Parr's "unit coal" corrections, is discussed. For use in "Parr's modified unit coal formula," where it is necessary to know the proportion of pyritic sulphur to the total sulphur present, the following assumptions, resulting from actual determinations, are made:

Coals with less than 2 per cent sulphur..........	40 per cent is pyritic
Coals with 2 to 3 per cent of sulphur............	50 per cent "
Coals with 3 to 7 per cent of sulphur............	60 per cent "
Coals with over 7 per cent of sulphur...........	75 per cent "

It is concluded that the factor of 1·1 for converting ash to mineral matter, previous to calculating a coal analysis to the pure coal basis, is satisfactory for coals containing less than 2 per cent of sulphur; for coals with sulphur contents greater than this amount, the factor of 1·25 is recommended.

The application of two different ash-to-mineral matter corrections in the analyses of fusain and other banded constituents of Phalen seam coal from the Sydney area, Nova Scotia, and of Onakawana lignite from northern Ontario, is presented; a modification of "Turner's" method for preparing "pure coal" by removal of the inorganic mineral matter with hypochloric and hydrofluoric acids to certain high ash coals; and the comparison of the results of volatile matter determinations using the electric furnace and the gas burner methods are also included in this report.

Hydrogenation of Solid and Liquid Fuels

Reports VI and VII, by T. E. Warren and his collaborators, comprise a *"Report of Experimental Work on the Hydrogenation of Canadian Coal, Coal Tar, and Bitumen for the Production of Motor Fuel"*, which is the title of Mines Branch Preprint No. 737-3, containing these two reports. The hydrogenation experiments on low-temperature coal tar reported in the first report are a continuation of the small-scale laboratory tests on Alberta bitumen reported in Investigations of Fuels and Fuel Testing, 1930 and 1931. Comparing the results of the hydrogenation treatment of the coal tar with those obtained by "pressure cracking" in a Cross bomb, it was found that hydrogenation was successful in reducing coke formation from over 17 per cent to less than 1 per cent, and that the total oil recovery by hydrogenation was over 90 per cent, as compared with 78·5 per cent by pressure cracking. By hydrogenation the gasoline fraction was doubled, and the kerosene fraction was increased by nearly 60 per cent over the corresponding pressure-cracking yields. The best hydrogenation results were obtained at approximately 475° C. and 250 atmospheres pressure, using molybdic oxide as catalyst.

The feature of Report VII is the description of larger laboratory-scale apparatus for continuous rather than intermittent operation. This new apparatus is illustrated by means of flow-sheets and photographs and its details are amplified in the context. The results of three experimental runs, namely, No. 7 on low-temperature coal tar, No. 8 on bitumen, and No. 10 on a suspension of powdered coal in coal tar, are shown. Molybdic oxide was again employed as the catalyst, and pressures in the reaction chamber ranged from 183 to 187 atmospheres with the temperature varying from 428 to 452° C. The net yields of "dry volatile oil" from these three products were 90, 102, and 89 per cent by volume respectively of the dry charging-stock, which shows that over 100 per cent by volume is obtainable from the bitumen, and that the yield from the coal-coal tar paste was only one per cent less than that from coal tar alone. Carbon formation was avoided in all three runs and the volatile products from the three different materials charged had similar distillation ranges. Approximately 90 per cent distilled below 572° F. and 45 per cent below 410° F. as the crude kerosene and gasoline fractions respectively. It is proposed to replace the one gallon "Cook" bomb with a vertical tube reaction chamber, the upper portion of which will serve for vapour-phase hydrogenation, and future experimental runs are to proceed when these changes are made.

Natural Gas, Naphtha, and Gasoline

The report by Rosewarne, Campbell, and Offord, entitled *"A Study of the Natural Gas and Naphtha Products from Twenty-four Wells in Turner Valley, Alberta"* comprises the results of field work during the summer of 1932 in the Turner Valley field, and of subsequent laboratory tests at Ottawa. The purpose of the investigation was to ascertain the composition of the products from the producing horizons by examining the natural gas and condensate at the surface. By the term "condensate" is meant the total liquid caught in the separator, and the term "naphtha" is the product after certain of the more volatile constituents have been removed by what is known as "weathering". The naphtha, therefore, is weathered condensate ready for transportation either in pipe-lines or portable containers.

The results obtained by passing the natural gas through activated charcoal, and then distilling and condensing the absorbed hydrocarbons from the charcoal, with the pressure of the system maintained at 30 pounds and the temperature of the condenser at 32° F., were compared with the results obtained by fractionation of the gas in a Podbielniak column. It was found that the activated charcoal absorption method was effective in removing all the "pentane and higher hydrocarbons fraction" plus 30 per cent of the total normal and iso-butane fraction, as indicated by the "Podbielniak" fractionation. Of the total butane fraction, the proportions of normal butane, B.P. $-0\cdot6^\circ$ C. and iso-butane, B.P. $-12\cdot2^\circ$ C., averaged 65 and 35 per cent respectively. Other conclusions drawn were that the proportion of condensible hydrocarbons in the "stripped gas" after the separators was higher in the gas from low-pressure wells than in the gas from high-pressure wells, and that lowering the back pressure on the well increased the production of naphtha per day. This investigation was a continuation of previous work in this field, the results of which are to be found in Investigations of Fuels and Fuel Testing for the years 1929, 1930 and 1931.

The last report, No. IX, by H. McD. Chantler, though entitled *"Gasoline Survey for 1932"*, is comprised of the results of this analysis survey for two years, namely, 1931 and 1932. It contains the detailed results of all samples collected in these years, but in this report the section "Significance of Tests on Gasoline" is not included, as in former annual Gasoline Surveys. It should, therefore, be read in conjunction with Mines Branch reprint No. 725–4, which is a summary of the 1930 and 1931 surveys, and contains a description and interpretation of the tests just referred to. The main conclusions arrived at were: (*a*) the average gasoline sold during 1931 and 1932 continued to be of good quality, the variation in quality being less than in any preceding year, which in turn indicated a tendency towards a more uniform grade of gasoline marketed; (*b*) only about three per cent of the total samples collected in 1932 had poor Octane ratings, i.e. below 57, while the average Octane number of all the samples was 65, with that of 15 out of a total of 123 samples averaging over 75; and (*c*) fifty-two per cent of the 1932 samples were artificially coloured. The hearty co-operation of the officials and inspectors of the Department of Pensions and National Health, in the collection of the samples in fifteen cities outside of Ottawa, is again gratefully acknowledged.

I

ANTHRACITE AND COKE ANALYSIS SURVEY CONDUCTED AT THE FUEL RESEARCH LABORATORIES

This report comprises the results of analyses of eighty-six samples of different sizes of anthracite coals and domestic cokes as marketed in Ottawa during the winter of 1932-33. Sixty-four samples of anthracites were examined, fourteen of which were of Welsh, Scotch, and Westphalian origin, and the remainder were typical Pennsylvania anthracites. Of the latter, seven different kinds or brands were collected, the number of samples of each varying from four to ten. Of a total of twenty-two samples of coke, all but four represent coke shipped in from coke manufacturing plants at Montreal, Hamilton, and Detroit. Inasmuch as practically all the anthracites originated from mining and distributing centres in Great Britain and the United States, the analyses reported may be considered representative of both the cokes and anthracites marketed in central Canada, although the samples were collected mostly in Ottawa.

The selection of the samples comprising this survey, their collection and certain of the physical tests, were made by technical officers of the Mechanical Engineering Section, and the chemical analyses were conducted by Messrs. J. H. H. Nicolls, C. B. Mohr, and other members of the Solid Fuels Analysis Section of the Laboratories.

In Table I the proximate analyses showing the moisture, ash, volatile matter, and fixed carbon contents, together with the calorific values and ash fusion temperatures, are given, and Table II includes the bulk densities and screen analyses. In the last table, it will be noticed, the coals and cokes are arranged according to trade sizes, whereas in the first table the arrangement is according to kinds, as marketed. The different cokes and Pennsylvania anthracites are designated by brand numbers rather than by their usual trade names.

The analyses of anthracites and cokes collected during previous years are shown in the annual reports of Investigations of Fuels and Fuel Testing and in other Mines Branch publications.[1] A review of the quality of these household fuels, as judged by their chemical analyses, reveals that the cokes and British anthracites have been of consistently good quality from year to year. For the Pennsylvania anthracites it has been observed that

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[1] No. 671-1—Examination of typical cokes sold in Canada as household fuels, Invest. of Fuels and Fuel Test. 1925, pp. 8-32.

Memorandum Series No. 30 (1927): Analyses of cokes sold in Canada.

whereas prior to about 1920, when the tendency was towards higher ash, a gradual improvement in quality has been noticed during recent years, and that the different brands now marketed are fairly uniform and of comparatively good quality. Below is shown a comparison of the ash contents, calorific values and fusion points of ash (F.P.A.) for selected sizes of both cokes and anthracites on the basis of their average moisture contents as delivered.

—	Number analyses averaged	Ash %	B.T.U. per pound	F.P.A. ° F.
Welsh cobbles	3	5·0	14,250	2250
" buckwheat	3	5·0	14,350	2300
Scotch cobbles	2	5·0	14,150	2700+
" buckwheat (and nut) sizes	2	5·0	14,150	2650
Pennsylvania, stove size	11	9·5	12,900	2700+
" nut size	10	10·1	12,800	2700+
" buckwheat	9	11·4	12,600	2700+
By-product coke, stove size	5	7·5	13,000	2500
" " nut size	6	7·4	13,000	2450
Gas and oven coke, mixed	2	8·3	12,950	(2400)

TABLE I

Analyses of Anthracites and Cokes Marketed in Ottawa in 1933

Sample No.	Designation and Trade Size	Proximate Analysis and Calorific Value								Fuel† ratio	F.P.A. (fusion point of ash)
		Partially air-dried			Dry basis						
		Moisture	Ash	B.T.U. per lb.	Ash	Volatile matter	Fixed carbon	Sulphur	B.T.U. per lb.		
		%	%		%	%	%	%			° F.
	ANTHRACITES										
	Welsh										
1–43	Cobbles	0·8	4·6	14,670	4·6	8·0	87·4	1·2	14,800	10·90	2220
2–46	"	0·9	4·3	14,410	4·3	7·8	87·9	1·2	14,540	11·30	2280
3–66	"	2·8*	6·5	13,910	6·7	9·1	84·2	1·0	14,300	9·25	2270
4–44	Stove size	0·7	4·5	14,590	4·5	8·1	87·4	1·2	14,700	10·80	2250
5–67	"	1·9	5·1	14,410	5·2	8·0	86·8	1·2	14,700	10·85	2270
6–45	Nos. 1 and 2 Buckwheat	0·6	3·6	14,840	3·7	7·8	88·5	1·0	14,930	11·30	2300
7–47	Nos. 1 and 2 "	1·1	5·9	14,210	5·9	7·5	86·6	0·9	14,360	11·55	2400
8–56	Nos. 1 and 2 "	1·6	5·5	14,390	5·6	8·7	85·7	0·8	14,620	9·90	2230
	Scotch										
9–48	Cobbles	2·2	5·2	14,020	5·3	6·9	87·8	0·7	14,340	12·70	2700+
10–51	"	2·1	4·8	14,280	4·9	9·2	85·9	0·7	14,590	9·35	2700+
11–52	Nut size	1·7	3·8	14,390	3·9	9·8	86·3	0·8	14,650	8·80	2630
12–50	Nos. 1 and 2 Buckwheat	1·4	6·2	14,000	6·3	9·3	84·4	0·8	14,200	9·10	2710
	Westphalian										
13–53	No. 1 Buckwheat	0·5	5·4	14,690	5·4	9·1	85·5	0·8	14,770	9·35	2270
14–54	No. 2 "	0·8	6·2	14,400	6·3	9·5	84·2	1·1	14,520	8·80	2270
	Pennsylvania Brand No. 1										
15– 1	Egg size	2·2	11·4	12,890	11·7	5·1	83·2	0·7	13,170	16·35	
16– 6	"	3·5*	10·1	12,970	10·5	6·0	83·5	0·8	13,440	13·85	
17– 2	Stove size	2·3	10·0	13,230	10·2	5·6	84·2	0·9	13,530	15·10	2700+
18– 7	"	2·9	10·5	13,010	10·8	6·6	82·6	0·8	13,390	12·50	2700+
19– 3	Nut size	2·1	11·7	12,910	12·0	6·1	81·9	1·0	13,190	13·45	
20– 8	"	2·9	10·0	13,140	10·3	6·2	83·5	0·9	13,540	13·60	
21– 4	Pea size	2·1	9·5	13,340	9·7	6·8	83·5	0·8	13,630	12·30	
22– 9	"	3·6*	9·0	13,140	9·3	6·2	84·5	0·8	13,630	13·70	
23– 5	No. 1 Buckwheat	2·8	9·9	13,110	10·2	5·9	83·9	0·8	13,480	14·30	2700+
24–10	No. 1 "	3·2	8·7	13,360	9·0	6·7	84·3	0·8	13,810	12·60	2700+

	Pennsylvania Brand No. 2										
25–11	Egg size	2·5	9·1	13,320	9·3	5·3	85·4	0·8	13,670	16·05	
26–16	"	3·0	8·3	13,330	8·5	5·4	86·1	0·8	13,730	16·05	
27–12	Stove size	1·6	9·5	13,420	9·7	5·3	85·0	0·8	13,640	15·85	2700+
28–17	"	2·4	8·3	13,500	8·5	5·2	86·3	0·9	13,830	16·55	2700+
29–13	Nut size	2·8	10·2	12,970	10·5	5·2	84·3	0·8	13,340	16·40	
30–18	"	2·0	10·4	13,020	10·6	4·8	84·6	0·9	13,280	17·55	
31–14	Pea size	2·2	9·8	13,090	10·0	5·3	84·7	0·7	13,380	15·95	
32–19	"	1·8	7·7	13,610	7·9	5·4	86·7	0·9	13,860	16·05	
33–15	No. 1 Buckwheat	1·5	12·0	12,830	12·2	5·6	82·2	0·7	13,030	14·50	2700+
34–20	No. 1 "	1·4	14·8	12,390	15·1	5·5	79·4	0·7	12,570	14·30	2700+
	Pennsylvania Brand No. 3										
35–26	Egg size	2·5	10·3	12,990	10·6	5·7	83·7	1·0	13,320	14·65	2700+
36–27	Stove size	2·4	10·5	12,910	10·8	5·3	83·9	0·6	13,230	15·90	
37–64	"	4·0*	10·9	12,650	11·4	5·9	82·7	0·9	13,190	14·05	2700+
38–28	Nut size	2·0	12·2	12,850	12·5	5·9	81·6	1·0	13,100	13·80	2700+
39–29	No. 1 Buckwheat	0·8	11·0	13,240	11·1	6·1	82·8	1·0	13,360	13·60	2700+
40–30	No. 2 "	1·3	13·5	12,720	13·7	6·0	80·3	1·0	12,890	13·45	2700+
	Pennsylvania Brand No. 4										
41–21	Egg size	3·6*	8·4	12,970	8·7	4·1	87·2	0·6	13,460	20·95	
42–22	Stove size	3·1	8·7	13,020	9·0	3·6	87·4	0·7	13,440	24·00	2700
43–23	Nut size	2·6	10·3	12,710	10·6	3·7	85·7	0·7	13,050	23·20	
44–24	Pea size	2·3	7·6	13,280	7·8	4·0	88·2	0·6	13,600	21·80	
45–25	No. 1 Buckwheat	1·8	10·7	12,870	10·9	4·2	84·9	0·7	13,100	20·00	2700+
	Pennsylvania Brand No. 5										
46–31	Egg size	2·6	8·4	13,380	8·6	5·7	85·7	0·9	13,730	15·05	
47–32	Stove size	2·2	10·0	13,160	10·2	5·6	84·2	0·8	13,450	15·15	2700+
48–33	Nut size	1·7	9·6	13,340	9·7	5·9	84·4	0·8	13,570	14·40	
49–34	Pea size	1·9	11·7	12,830	11·9	5·6	82·5	0·8	13,070	14·80	
50–35	No. 1 Buckwheat	2·0	10·2	13,010	10·4	6·1	83·5	0·9	13,270	13·65	2700+

TABLE I—Continued

Analyses of Anthracites and Cokes Marketed in Ottawa in 1933

Sample No.	Designation and Trade Size	Proximate Analysis and Calorific Value								Fuel† ratio	F.P.A. (fusion point of ash)
		Partially air-dried			Dry basis						
		Moisture	Ash	B.T.U. per lb.	Ash	Volatile matter	Fixed carbon	Sul-phur	B.T.U. per lb.		
		%	%		%	%	%	%			° F.
				ANTHRACITES—*Concluded*							
	Pennsylvania Brand No. 6										
51-36	Egg size	2·5	10·7	12,980	11·0	5·9	83·1	0·7	13,300	14·15	2700+
52-57	"	3·5*	8·1	13,320	8·4	6·0	85·6	0·9	13,800	14·30	2700+
53-37	Stove size	2·3	8·8	13,290	9·0	5·5	85·5	0·9	13,620	15·45	2700+
54-58	"	3·4	9·1	13,260	9·4	6·0	84·6	0·9	13,730	14·05	2700+
55-59	Nut size	3·9*	8·8	13,130	9·2	5·8	85·0	0·9	13,670	14·55	2700+
56-62	"	4·1*	9·3	12,970	9·7	5·9	84·4	0·8	13,530	14·30	2700+
57-60	Pea size	3·0	8·6	13,320	8·9	6·6	84·5	0·8	13,720	12·70	2700+
58-65	"	3·4	18·8	11,370	19·5	4·9	75·6	0·7	11,770	15·30	2700
59-61	No. 1 Buckwheat	4·9*	14·7	11,890	15·4	5·4	79·2	0·7	12,490	14·75	2500
60-63	No. 1 "	2·5	12·4	12,860	12·7	7·6	79·7	0·7	13,200	10·40	2300
	Pennsylvania Brand No. 7										
61-39	Egg size	0·9	12·2	13,290	12·3	9·1	78·6	0·7	13,410	8·65	2700+
62-40	Stove size	1·1	14·4	12,950	14·6	8·8	76·6	0·8	13,100	8·65	
63-41	Nut size	0·7	15·0	12,890	15·1	8·6	76·3	0·7	12,990	8·80	
64-42	Pea size	0·8	15·0	12,860	15·1	8·6	76·3	0·7	12,970	8·85	

*These moisture contents are for samples having minimum exposure to room atmosphere before being analysed. The moisture contents of the different fuels as marketed and as assumed in the tabulated averages on page 7 above, are 4 per cent for the Pennsylvania anthracites, 2 per cent for the Welsh and Scotch anthracites, and 1 per cent for the cokes. Hence, the difference between these averages and the percentages here reported represents the amount of drying during the time the samples were stored indoors and during the screening test, prior to their preparation for chemical analysis.

†The Fuel Ratio is the ratio of fixed carbon to volatile matter and indicates comparative rank of coal.

TABLE I—Concluded

Analyses of Anthracites and Cokes Marketed in Ottawa in 1933

Sample No.	Designation and Trade Name	Proximate Analysis and Calorific Value								F.P.A. (fusion point of ash)	Apparent Sp. Gr.
		Partially air-dried			Dry basis						
		Moisture	Ash	B.T.U. per lb.	Ash	Volatile matter	Fixed carbon	Sulphur	B.T.U. per lb.		
		%	%		%	%	%	%		° F.	
	COKES										
	Gas and (By-product) Oven Coke—Mixed										
65–70	Crushed "New Process"	0·3	8·2	12,990	8·2	1·7	90·1	1·0	13,040	2370	0·88
66–71	" (Mixed)	1·0	8·3	12,950	8·4	1·6	90·0	1·1	13,080	2370	0·81
67–73	Nut size (Mixed)	....	9·9	....	10·6	2·5	86·9	1·2	12,790	2260	0·88
68–72	Blower size (Mixed)	8·9	10·7	11,470	11·7	2·3	86·0	1·2	12,590	2180	0·91
	By-product Oven Coke—Brand No. 1										
69–82	Egg size	0·1	7·2	13,210	7·2	0·7	92·1	0·8	13,220	2480	0·89
70–74	Stove size	0·1	7·1	13,090	7·1	1·0	91·9	0·8	13,100	2500	0·93
71–79	"	0·3	7·1	13,190	7·1	0·9	92·0	0·8	13,220	2490	0·91
72–83	"	0·1	7·1	13,200	7·0	0·7	92·3	0·8	13,210	2420	0·89
73–68	"	1·4	7·2	12,860	7·3	1·1	91·6	0·8	13,050	2470	0·92
74–75	Nut size	0·3	6·5	13,150	6·5	1·0	92·5	0·8	13,190	2380	0·89
75–80	"	0·1	7·4	13,010	7·4	0·7	91·9	0·8	13,030	2460	0·94
76–84	"	0·6	6·7	13,090	6·8	0·8	92·4	0·9	13,170	2360	0·89
77–69	"	1·4	7·5	12,820	7·6	1·0	91·4	0·8	13,010	2470	0·95
78–85	Pea size	0·1	7·1	13,150	7·1	0·7	92·2	0·8	13,170	2420	0·89
	By-product Oven Coke—Brand No. 2										
79–86	Stove size	0·8	9·0	12,850	9·0	1·3	89·7	1·0	12,950	2600	0·95
80–87	Nut size	1·7	8·9	12,690	9·1	1·2	89·7	0·9	12,900	2650	0·98
81–88	Range (Nut and Pea)	....	9·1	....	9·7	1·3	89·0	0·9	12,830	2665	0·99
82–81	Pea size	0·4	9·5	12,890	9·5	1·8	88·7	0·8	12,950	....	0·94
83–89	Buckwheat size	5·3	9·3	12,080	9·8	1·7	88·5	0·9	12,760	2700+	0·91
	By-product Oven Coke—Brand No. 3										
84–76	Egg size	0·3	7·4	13,210	7·4	1·0	91·6	0·6	13,240	2450	0·87
85–77	Nut size	0·4	7·4	13,170	7·5	0·9	91·6	0·6	13,220	2400	0·89
86–78	"Hickory" size	0·7	7·0	12,990	7·1	0·9	92·0	0·6	13,080	2410	0·87

TABLE II

Screen Analyses and Bulk Weights of Anthracites and Cokes Reported in Table I

Sample No.	Designation and Trade Size	Analysis as Tested				Screen Analysis—Using Square Mesh Screens								
		Ash	B.T.U. per lb.	Weight per cu. ft.	Cu. ft. per short ton	On 3″	On 2″ (−3″)	On 1½″ (−2″)	On 1″ (−1½″)	On ¾″ (−1″)	On ½″ (−¾″)	On ¼″ (−½″)	On ⅛″ (−¼″)	Through ⅛″
		%		lb.		%	%	%	%	%	%	%	%	%
	ANTHRACITES													
	Welsh													
1–43	Cobbles................	4·6	14,670	44·9	44·6	28·4	57·4	7·5	4·2	1·1	0·3	1·1*		
2–46	"	4·3	14,410	44·4	45·0	27·9	45·4	7·8	5·8	2·5	2·8	7·8		
3–66	"	6·5	13,910	44·1	45·3	54·2	43·8	1·2	0·4	0·0	0·2	0·2		
	Average...........	5·0	14,330	44·5	45·0	36·8	48·9	5·5	3·5	1·2	1·1	3·0		
4–44	Stove size..............	4·5	14,590	43·9	45·6	....	40·0	36·8	17·4	2·3	1·2	2·3		
5–67	"	5·1	14,410	48·3	41·5	....	15·1	49·5	30·0	3·1	1·0	1·3		
	Average...........	4·8	14,500	46·1	43·6	....	27·5	43·2	23·7	2·7	1·1	1·8		
6–45	Nos. 1 and 2 Buckwheat	3·6	14,840	43·5	46·0	....	....	....	....	....	....	40·4	59·0	0·6†
7–47	Nos. 1 and 2 "	5·9	14,210	47·0	42·5	....	....	....	....	....	....	39·3	49·6	11·1
8–56	Nos. 1 and 2 "	5·5	14,390	49·5	40·4	....	....	....	....	....	5·6	56·6	18·0	19·8
	Average...........	5·0	14,480	46·7	43·0	....	....	....	....	....	1·9	45·4	42·2	10·5
	Scotch													
9–48	Cobbles................	5·2	14,020	45·0	44·4	44·0	44·0	5·5	3·3	1·1	0·4	1·7		
10–51	"	4·8	14,280	45·6	43·8	24·8	40·3	18·3	13·7	1·4	0·5	1·0		
	Average...........	5·0	14,150	45·3	44·1	34·4	42·2	11·9	8·5	1·3	0·4	1·3		
11–52	Nut size................	3·8	14,390	46·0	43·5	....	....	10·9	36·5	29·0	15·8	7·8		
12–50	Nos. 1 and 2 Buckwheat	6·2	14,000	48·3	41·4	....	....	....	....	....	5·1	64·5	24·5	5·9
	Westphalian													
13–53	No. 1 Buckwheat.......	5·4	14,690	46·8	42·8	....	....	....	....	....	9·2	71·8	9·3	9·7
14–54	No. 2 "	6·2	14,400	45·8	43·8	....	....	....	....	....	....	18·4	62·5	19·1

*and †Percentages in these columns are totals through the ½-inch and ⅛-inch screens respectively.

TABLE II—Continued

Screen Analyses and Bulk Weights of Anthracites and Cokes Reported in Table I

Sample No.	Designation and Trade Size	Analysis as Tested				Screen Analysis—Using Square Mesh Screens					
		Ash	B.T.U. per lb.	Weight per cu. ft.	Cu. ft. per short ton	On 2″ (−3″)	On 1½″ (−2″)	On 1″ (−1½″)	On ¾″ (−1″)	On ½″ (−¾″)	Through ½″
		%		lb.		%	%	%	%	%	%
	AMERICAN (PENNSYLVANIA) ANTHRACITES										
15– 1	Egg size	11·4	12,890	50·5	39·6	86·1	8·0	3·0	0·7	0·7	1·5
16– 6	"	10·1	12,970	51·0	39·2	82·1	11·5	3·4	1·0	0·7	1·3
25–11	"	9·1	13,320	48·6	41·1	84·2	10·9	3·3	0·4	0·4	0·8
26–16	"	8·3	13,330	49·6	40·3	86·0	9·6	1·4	0·5	0·5	2·0
35–26	"	10·3	12,990	51·3	39·0	60·2	18·0	12·7	4·2	2·7	2·2
41–21	"	8·4	12,970	53·6	37·3	81·5	17·5	0·4	0·0	0·3	0·3
46–31	"	8·4	13,380	49·0	40·8	81·6	13·8	3·6	0·2	0·2	0·6
51–36	"	10·7	12,980	50·4	39·7	79·1	14·1	4·3	0·9	0·5	1·1
52–57	"	8·1	13,320	50·3	39·8	82·3	12·5	3·9	0·9	0·2	0·2
61–39	"	......	13,290	47·6	42·0	85·3	10·0	1·6	0·5	0·5	2·1
	Average	9·4	13,144	50·2	39·9	80·8	12·6	3·8	0·9	0·7	1·2
17– 2	Stove size	10·0	13,230	51·6	38·7	13·9	48·2	27·1	3·4	3·2	4·2
18– 7	"	10·5	13,010	51·4	38·9	11·4	67·4	18·0	1·7	0·5	1·0
27–12	"	9·5	13,420	49·8	40·2	6·9	47·8	36·8	3·3	3·3	1·9
28–17	"	8·3	13,500	49·9	40·1	12·8	43·7	32·2	5·3	3·0	3·0
36–27	"	10·5	12,910	53·0	37·7	14·8	58·4	24·0	1·5	0·4	0·9
37–64	"	10·9	12,650	51·0	39·1	22·0	57·2	18·7	1·1	0·4	0·6
42–22	"	8·7	13,020	56·8	35·2	0·0	48·0	34·6	8·1	5·7	3·6
47–32	"	10·0	13,160	50·0	40·0	9·8	69·7	17·5	1·5	1·0	0·5
53–37	"	8·8	13,290	50·5	39·6	6·8	65·2	24·9	1·2	0·7	1·2
54–58	"	9·1	13,260	50·0	40·0	6·7	66·9	25·0	0·7	0·2	0·5
62–40	"	......	12,950	49·4	40·5	24·3	55·2	16·7	1·5	0·5	1·8
	Average	9·6	13,127	51·2	39·1	11·8	57·1	25·0	2·7	1·7	1·7

TABLE II—Concluded

Screen Analyses and Bulk Weights of Anthracites and Cokes Reported in Table I

Sample No.	Designation and Trade Size	Analysis as Tested				Screen Analysis—Using Square Mesh Screens						
		Ash	B.T.U. per lb.	Weight per cu. ft.	Cu. ft. per short ton	On 1″ (−1½″)	On ¾″ (−1″)	On ½″ (−¾″)	On ¼″ (−½″)	On ⅛″ (−¼″)	On 10 mesh (−⅛″)	Through 10 mesh
		%		lb.		%	%	%	%	%	%	%
	AMERICAN (PENNSYLVANIA) ANTHRACITES—*Concluded*											
19– 3	Nut size	11·7	12,910	52·0	38·5	42·7	37·3	15·5	4·5			
20– 8	"	10·0	13,140	50·1	39·9	64·0	28·5	6·0	1·5			
29–13	"	10·2	12,970	51·9	38·5	25·3	35·0	32·2	7·5			
30–18	"	10·4	13,020	50·8	39·4	39·9	39·4	16·7	4·0			
38–28	"	12·2	12,850	51·4	38·9	(42·8	....	40·6	13·1	3·5)		
43–23	"	10·3	12,710	57·8	34·6	36·4	39·8	17·5	6·3			
48–33	"	9·6	13,340	49·5	40·4	65·7	24·4	8·5	1·4			
55–59	"	8·8	13,130	52·8	37·9	45·1	35·3	17·8	1·8			
56–62	"	9·3	12,970	53·4	37·5	38·0	32·4	22·0	7·6			
63–41	"	......	12,890	48·5	41·2	38·9	33·3	21·1	6·7			
	Average	10·3	12,993	51·8	38·7	44·0	33·9	17·5	4·6			
21– 4	Pea size	9·5	13,340	52·0	38·5	7·9	13·3	26·5	43·0	9·3		
22– 9	"	9·0	13,140	50·8	39·4	....	1·8	36·8	51·8	9·6		
31–14	"	9·8	13,090	50·8	39·4	....	2·4	60·2	28·7	4·6	4·1	
32–19	"	7·7	13,610	52·0	38·5	3·8	11·1	43·8	37·5	3·1	0·7	
44–24	"	7·6	13,280	54·0	37·0	3·2	2·9	56·6	32·6	2·2	2·5	
49–34	"	11·7	12,830	50·0	40·0	....	2·6	75·6	18·9	1·9	1·0	
57–60	"	8·6	13,320	49·5	40·5	1·2	6·3	23·8	43·6	14·0	11·1	
58–65	"	......	....	(56·8	35·2)	....	....	51·8	38·2	3·6	6·4	
64–42	"	......	12,860	49·3	40·5	....	....	54·2	39·4	3·4	3·0	
	Average	9·1	13,184	51·0	39·2	1·8	4·5	47·7	37·1	5·7	3·2	
23– 5	No. 1 Buckwheat	9·9	13,110	50·3	39·8	....	....	2·7	75·3	15·4	3·6	3·0
24–10	"	8·7	13,360	50·5	39·6	....	....	3·5	82·0	13·5	1·0	
33–15	"	12·0	12,830	50·8	39·4	....	....	....	88·8	10·4	0·5	0·3
34–20	"	14·8	12,390	51·0	39·2	....	....	....	87·7	11·5	0·5	0·3
39–29	"	11·0	13,240	47·8	41·8	....	....	4·2	86·9	8·1	0·4	0·4
45–25	"	10·7	12,870	52·8	37·9	....	....	....	86·0	9·1	0·6	4·3
50–35	"	10·2	13,010	49·0	40·8	....	....	....	87·3	12·0	0·4	0·3
59–61	"	14·7	11,890	52·5	38·0	....	....	1·9	80·4	11·0	3·8	2·9
60–63	"	12·4	12,860	49·5	40·0	....	....	2·2	84·7	8·7	2·2	2·2
	Average	11·6	12,840	50·5	39·6	....	....	1·6	84·4	11·1	1·4	1·5
40–30	No. 2 Buckwheat	13·5	12,720	48·3	41·5	....	....	....	12·0	76·0	11·7	0·3

Sample No.	Designation and Trade Size	Analysis as Tested				Screen Analysis—Using Square Mesh Screens						
		Ash	B.T.U. per lb.	Weight per cu. ft.	Cu. ft. per short ton	On 2" (−3")	On 1½" (−2")	On 1" (−1½")	On ¾" (−1")	On ½" (−¾")	Through ½"	Through ¼"
		%		lb.		%	%	%	%	%	%	%
	COKES											
69–82	Egg size	7·2	13,210	25·8	77·7	34·1	52·8	11·1	0·8	0·2	1·0	
84–76	"	7·4	13,210	26·5	75·5	49·8	40·2	9·4	0·3	0·0	0·3	
	Average	7·3	13,210	26·1	76·6	42·0	46·5	10·3	0·5	0·1	0·6	
70–74	Stove size	7·1	13,090	27·5	72·8	3·9	62·3	32·0	1·0	0·3	0·5	
71–79	"	7·1	13,190	27·8	72·0	7·3	75·3	14·5	1·7	0·5	0·7	
72–83	"	7·1	13,200	27·3	73·4	6·2	76·6	12·7	1·0	0·8	2·7	
73–68	"	7·2	12,860	29·0	69·0	5·0	56·0	34·1	4·1	0·4	0·4	
79–86	"	9·0	12,850	26·0	76·8	12·8	70·6	15·2	0·3	0·3	0·8	
	Average	7·5	13,038	27·5	72·8	..7·0	68·2	21·7	1·6	0·5	1·0	
65–70	"New Process"-crushed	8·2	12,990	25·9	77·3	10·8	45·3	34·7	6·0	2·2	1·0	
66–71	"Mixed-crushed"	8·3	12,950	22·6	88·4	8·0	49·2	36·7	3·2	0·8	2·1	
74–75	Nut size	6·5	13,150	27·9	71·7	....	19·3	66·6	12·5	0·8	0·8	
75–80	"	7·4	13,010	28·6	69·8	....	22·8	69·2	6·5	0·4	1·1	
76–84	"	6·7	13,090	27·5	72·7	....	9·3	59·9	25·0	3·7	2·1	
77–69	"	7·5	12,820	30·5	65·6	....	14·5	68·1	14·9	1·6	0·9	
80–87	"	8·9	12,690	28·3	70·6	....	3·4	90·8	4·8	0·3	0·7	
85–77	"	7·4	13,170	27·4	73·2	....	31·0	63·4	4·7	0·2	0·7	
	Average	7·4	12,988	28·3	70·6	....	16·7	69·7	11·4	1·2	1·0	
67–73	"Mixed Nut"	9·9	11,980	29·0	69·0	....	....	8·8	47·5	34·2	9·5	
86–78	"Hickory"	7·0	12,990	28·8	69·7	....	....	3·6	61·0	31·8	3·6	
81–88	"Range"	9·1	12,060	33·3	60·1	....	....	....	40·0	57·2	2·8	
78–85	Pea	7·1	13,150	27·5	72·7	....	....	....	29·2	67·2	2·2	1·4
82–81	"	9·5	12,890	27·9	71·7	....	....	11·6	72·6	13·4	2·4	
83–89	Buckwheat	9·3	12,080	31·3	63·8	....	....	....	37·7	58·7	3·6	
68–72	Blower	10·7	11,470	29·3	68·4	....	....	....	20·4	57·9	16·9	4·8

APPENDIX

RELATIVE VALUES OF ANTHRACITES AND COKES FOR USE IN DOMESTIC FURNACES

Messrs. E. S. Malloch and C. E. Baltzer, in their report entitled "Tests of Various Fuels made in a Domestic Hot Water Boiler at the Fuel Testing Station, in Co-operation with the Dominion Fuel Board", included a table[1] showing the relative value of various fuels tested, compared with American anthracite. This table may now be revised as in Table III below, since the results of recent analyses as reported in Tables I and II indicate appreciable changes in ash contents and calorific values, especially for the Pennsylvania anthracites, and such revision is feasible by the use of the average calorific values for the different fuels as shown on page 7. The same efficiencies as obtained in the actual burning tests are assumed and the average values of pounds of fuel fired per therm delivered to the cooling-water are adjusted in accordance with the 1933 analyses, thus eliminating the necessity of making a new series of burning tests.

TABLE III

The Relative Values of Various Fuels, Compared with American Anthracite

Fuel and size	Average value* for pounds of fuel fired per therm delivered to cooling-water	Equivalent tonnage to 10 tons of American anthracite
American anthracite (stove)	10·67	10·00
Welsh " (cobbles)	9·44	8·85
Scotch " (cobbles)	9·47	8·85
By-product coke (stove)	10·32	9·65
Gas (and oven) coke (mixed)	10·53	9·85

*Adjusted according to analyses made in 1932-33.

[1] Table V, page 63, Invest. of Fuels and Fuel Test. 1925.
Table X, page 92, Mines Branch Report No. 705 (1929).
Table on page 30, Second Progress Report of the Dominion Fuel Board, 1923-1928.

II

THE F.R.L. METHOD FOR RATING THE GRINDABILITY OR PULVERIZABILITY OF COAL

Correlated with THE "CROSS" AND "HARDGROVE" METHODS

C. E. Baltzer and H. P. Hudson

PREFACE AND REVIEW

The purpose of this investigation has been to select or devise a laboratory method for deducing an index which would show the comparative grindability or pulverizability of coals. An important requirement of such an index is that it must be capable of interpretation into terms of mill capacity. Hence, knowing the general relation existing between the index determined in the laboratory and mill capacity, the approximate amount of coal, say in tons per hour, that can be pulverized to a stated fineness in a given mill can be predicted. The power required to operate the mill at optimum capacity is another factor that requires correlation.

The coals available for this investigation were those from British Columbia, used in the series of pulverized fuel fired boiler trials recently conducted by E. S. Malloch and C. E. Baltzer. The pulverizer used was a high-speed, paddle-type mill, rated at 1,000 pounds per hour, and for each coal tested the mill performance in terms of power per ton, percentage of rated capacity, and screen analysis of the pulverized fuel delivered to the burner were recorded—see Table II, page 26. It is with these commercial-scale results that the laboratory grindability indices have been correlated. By this and further correlation with other pulverizers, including the ball-mill type, it is expected that in the future the determination of the grindability index of a coal can take the place, at the Fuel Research Laboratories, of elaborate and expensive large-scale burning tests in the pulverized fuel fired boiler installation.

Two methods for determining grindability indices already advanced have been examined, namely the Cross and the Hardgrove methods. A third method known as the F.R.L. method has been introduced, and it is the description and comparison of the results obtained by these three methods that is the subject of the paper herewith prefaced and reviewed. In the appendix is given a full description of these three methods, and in their paper Messrs. Baltzer and Hudson have described the development and advantages of the new F.R.L. method in relation to the two other methods. Below in tabular form the salient features of the three methods are given, and a comparison of the results obtained on twelve coals is shown in Table V.

Item	F.R.L. method	Cross method	Hardgrove method
Weight of sample used, grammes	500-1,500	200	50.
Fineness of sample before pulverizing	Just through 10-mesh screen.	Just through 10-mesh screen.	Through 16 on 30-mesh screen.
Type of mill	Laboratory jar mill.	Laboratory jar mill.	Special grindability machine.
Type of balls or pebbles	½ to 1-inch flint pebbles.	¾-inch steel balls.	1-inch steel balls.
Quantity of balls or pebbles	3,000 grammes; (⅓ jarful).	89 balls; (⅓ jarful).	8 balls.
Number of revolutions	3,000; 3 stages at 1,000 per stage.	400	60.

The above comparison indicates more grinding on a much larger sample by the F.R.L. method, and therefore it should give more representative results. By both the Hardgrove and Cross methods the grindability index is arrived at by calculation from the screen analysis results on the pulverized product from the test, whereas in the F.R.L. method the index is the difference between a constant figure and the amount retained on a screen of definite size. An interesting feature of the latter method is the addition to the residue remaining on the 100-mesh screen at the end of the first and second stages of grinding, of sufficient stock sample to bring the weight up to 500 grammes. This so-called "sweetening" of the charge is somewhat similar to commercial grinding where fresh coal is progressively charged as the pulverized product passes out of the mill.

The merits of a laboratory method for determining a grindability index may be judged by: simplicity of operation; availability of the apparatus used; ability to obtain check results; spread of indices for coals varying widely in grindability characteristics; ability to differentiate between coals having indices within a close range; and interpretation into terms of commercial mill capacities. The relative merits as to simplicity of operation and availability of the apparatus of the three methods examined are elaborated by the authors, and their respective merits according to the other points may be further discussed here.

Of the twelve coals tested, coal B was the hardest to grind, that is, most resistant to pulverization, and coal L was the softest. Accordingly, the range of indices obtained by the three methods for these two coals may be compared. The range of individual indices was 200 to 699 by the Cross method, 153 to 376 by the F.R.L. method, and 36·4 to 101·9 by the Hardgrove method. The differences between checks in duplicate determinations using the data on all twelve coals ranged from 0·0 to 7·4 by the Hardgrove, and 0 to 6 by the F.R.L. method. Similar differences in triplicate determinations by the Cross method were from 1 to 107. Hence, in respect to ability to obtain checks on individual coals, the order of the three methods was: first, the F.R.L.; second, the Hardgrove; and third, the Cross. For the two coals at the top and bottom, and for five of the intermediate coals for which the indices are close together, the average indices obtained by the three methods are shown below:—

Average Index Values by Duplicate Determinations

—	Hardgrove method	F.R.L. method	Cross method
Coal B	37·0	153	221
" K	69·2	245	430
" H	70·3	262	442
" G	74·7	271	479
" D	80·1	276	463
" I	79·5	282	496
" L	99·1	374	670

From the above it would appear that there is a fairly uniform relation between the indices obtained by the three methods, and as a result of comparison, the general conclusion may be drawn that any one of them may serve as a standard method for determining the grindability index of coal. The Cross and Hardgrove methods have been correlated with mill capacities, the relations being known by the respective manufacturers of pulverizer equipment represented by the authors of the methods. As for the F.R.L. method, it is planned eventually to correlate the indices obtainable with mills of different sizes and types, but in the meantime it is hoped that it will serve as a means of correlation between the two laboratory methods already examined and with any other meritorious method that may be advanced.

The investigation has been conducted in conjunction with the activities of Subcommittee VII on Pulverizing of Coal, American Society for Testing Materials, Committee D-5 on Coal and Coke. To Mr. A. C. Fieldner and Mr. John Van Brunt, Chairmen of Committee D-5 and Subcommittee VII respectively, acknowledgment is due for valuable suggestions and co-operation. The objective of the committee is to select and adopt as an A.S.T.M. standard, a method for determining a grindability index which will be of value to coal producers and consumers, as well as to the manufacturers of coal-pulverizing equipment. Hence the results obtained, while applicable first to Canadian coals, have an international significance.

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INTRODUCTION

The increased use of coal in the pulverized state has brought about a need for the development and general adoption of a simple laboratory method for rating coals in accordance with their grinding or pulverizing characteristics. Although this need has long been recognized by responsible builders of modern pulverizing machinery, it is only within recent years that the larger coal producers and consumers have realized the importance of evaluating coals in this respect. That the need is apparent, and acute, is evidenced by the number of research organizations in many parts of the world, which have lately included grindability investigations among their projects. Table I lists some of the agencies reported to be engaged on the problem.

TABLE I

Agencies Reported to be Engaged on the Coal Grindability Problem

(List not Claimed to be Complete)

Agency	Stated Problem
CANADA—	
1. Department of Mines, Mines Branch—Fuel Research Laboratories, Ottawa.	Correlation of various suggested methods.
ENGLAND—	
2. [1] Utilization of Coal Committee of the Institution of Mining Engineers, London.	Grindability of coals.
3. [1] Department of Fuel Technology, Sheffield University, Sheffield.	Grindability tests on British coals.
GERMANY—	
4. [2] Committee on Fuel of the Reichskohlenrat, Dresden.	Grindability of various classes of fuel.
UNITED STATES—	
5. [3] American Society for Testing Materials, Philadelphia, Pa.	Pulverizing characteristics of coal.
6. [3] Battelle Memorial Institute, Columbus, Ohio. (R. A. Sherman.)	Pulverizing characteristics of coal.
7. [4] Carnegie Institute of Technology, Pittsburg, Pa. (Prof. H. J. Sloman.)	Grindability of United States coals.
8. [4] Combustion Engineering Corporation, New York City. (John Van Brunt.)	Development of a method for the determination of the pulverizing characteristics of coal.
9. [4] Erie City Iron Works, Erie, Pa. (G. S. Brewer.)	Laboratory-scale pulverizing test correlated with commercial pulverizers.
10. [2] Fuel Engineering Company, New York City. (G. B. Gould.)	Grindability of coal for pulverizer service.

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[1] The Fuel Economist, December, 1932, page 156.

[2] A Survey of Research in Coal and Coal Utilization, November, 1931. Committee of Ten—Coal and Heating Industries, Chicago, Ill.

[3] Research Activities in the Mineral Industries of the United States. U.S. Bureau of Mines I.C. 6637.

[4] Private correspondence between members of Subcommittee VII, Committee D-5, A.S.T.M.

TABLE I—Concluded

Agencies Reported to be Engaged on the Coal Grindability Problem

(List not Claimed to be Complete)

Agency	Stated Problem
11. [2] Fuller Lehigh Company, Fullerton, Pa. (R. M. Hardgrove.)	Comparison of grindability of coal as indicated by two types of grindability machines.
12. [3] Rochester and Pittsburgh Coal Company, Indiana, Pa.	Pulverization characteristics of coal from various parts of the Freeport seam.
13. [4] University of Michigan, Ann Arbor, Mich. (Prof. A. H. White.)	Laboratory study on the grinding characteristics of various coals.
14. [4] U.S. Bureau of Mines, Engineering Station, Seattle, Wash. (Dr. H. F. Yancy.)	Study of methods for determining grindability of coal.
15. [4] Whiting Corporation, Harvey, Ill.	Grindability of coal.
16. [2] Yale University, New Haven, Conn.	Development of method for determining grindability of coal.

[2] A Survey of Research in Coal and Coal Utilization, November, 1931. Committee of Ten—Coal and Heating Industries, Chicago, Ill.

[3] Research Activities in the Mineral Industries of the United States. U.S. Bureau of Mines I.C. 6637.

[4] Private correspondence between members of Subcommittee VII, Committee D-5, A.S.T.M.

The lack of definite correlated information respecting the grinding characteristics of different ranks and grades of coal in varying types of commercial pulverizers, together with the difficulty of securing uniform agreement between all interested parties as to the best methods of attack, has delayed action by standards' associations, such as the American Society for Testing Materials, in selecting any one method for inclusion amongst their existing standards. Although it is true that the subject has received some attention in the technical press, the literature is by no means voluminous, and it is certain that much valuable information having a direct bearing on the subject is in the hands of industrial research organizations. The gist of it, however, has not been made public except in a very few cases, notably among which are the contributions by Hardgrove[1] and Gould.[2]

The methods these two writers have outlined are worthy of consideration as a means to the establishment of a recognized standard method, but their weakness probably lies in the fact that special apparatus, not common in the average laboratory, is called for. John Van Brunt and B. J. Cross[3] in a private report to Subcommittee VII on pulverizing characteristics of coal, Committee D-5, A.S.T.M., outline a simpler method than either of the above. This method follows along the general lines outlined by Hardgrove, but, whereas the Hardgrove method selects the coarser particles of the coal sample, after preliminary crushing, on which to make the test in a special grinding unit, the Cross method makes no attempt to segregate the sample after preliminary crushing and uses the ordinary laboratory jar and pebble mill in which to make the test. Its merit, from the writers' viewpoint, lies in the fact that the equipment used is such as is commonly found in the average laboratory. However, a serious drawback does exist in that the grindability index number determined there-

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[1] "Grindability of Coal," by R. M. Hardgrove, i/c Research and Design for Fuller Lehigh Company. A.S.M.E. paper No. FSP—54—5, presented at Birmingham meeting April, 1931.

[2] "Factors in Coal Selection—Pulverizing Quality and Fuel Value," by G. B. Gould, President, Fuel Engineering Company. Power, December 2, 1930, page 886.

[3] Chief and Assistant respectively, Research Department, International Combustion Engineering Company.

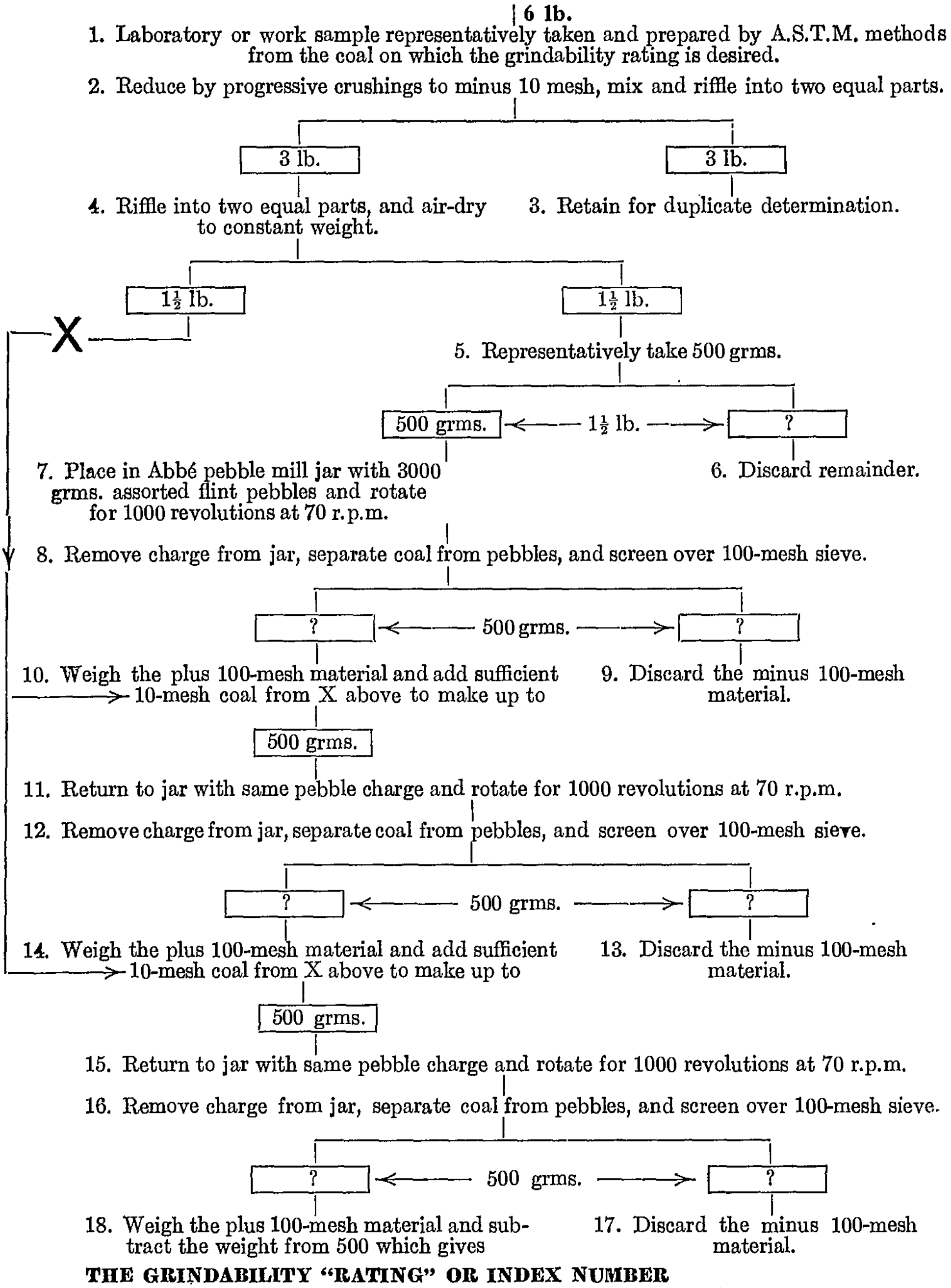
from, is likely to be on the low side because no means are provided for measuring the unit size of the super-fine material produced in the final milling operation. The Fuel Research Laboratory method, outlined herein, uses the same grinding equipment as the Cross method but differs from the latter in that extra precautions are taken in the preparation and preliminary crushing of the coal sample, the test sample is longer and more efficiently milled, means are provided for "sweetening" the mill charge so that there is assurance that the proper work has been done on the harder coal particles; and finally the index number is obtained by experimental means rather than by mathematical calculation. Notwithstanding all of the above, the method is very simple and almost as rapid as the Cross method, and slightly more so than the Hardgrove method.

In presenting this new method the writers do so with some hesitancy as it is realized that the method may need some final refinement inasmuch as opportunity has been lacking for correlating it with the performance of a commercial pulverizer working at full-load ratings. The method, therefore, is advanced as a tentative one pending such correlation, and in this connexion it is hoped that others better equipped to do so will take the matter in hand. It is for this reason, as well as the promised co-operation of the staff of these laboratories in making a check-up of the Cross method, that the present progress report is submitted at this time to Subcommittee VII of Committee D-5, A.S.T.M.

DESCRIPTION OF THE METHOD

Briefly, the method consists of taking a representative portion of the coal on which a grindability rating is desired with the same care and attention to detail as is usual when sampling coal for chemical analysis. A 5- to 6-pound work or laboratory sample is taken from the portion so selected in accordance with the A.S.T.M. standard method. The laboratory sample is then carefully and progressively reduced in size, by means of a small roll crusher, so that it will all pass a 10-mesh sieve, due precautions being taken not to overcrush the coal. The sample is well mixed by rolling on an oilcloth sheet and then riffled into two parts. One part is set aside as a reserve for duplicate determination, if such is desired. The other part is again mixed and divided into two parts as before. Each of these parts is placed in a shallow pan and air-dried to constant weight.

Five hundred grammes of the air-dried coal are taken from the one pan (the coal remaining in this pan is discarded) and placed in a standard Abbé pebble mill jar of approximately one gallon capacity together with a 3,000-gramme charge of flint pebbles assorted in size between ½ and 1 inch. The jar with its coal and pebble load is then rotated for 1,000 revolutions at a speed of 70 r.p.m.; after which the load is removed and the coal is carefully separated from the pebble charge. The coal is screened over a 100-mesh sieve, the minus 100-mesh product being removed and discarded. The plus 100-mesh material is weighed and enough of the original coal from the second pan, previously mentioned, is added to make up or "sweeten" to 500 grammes. The "sweetened" sample is now returned to the jar together with the same pebble charge previously used and is milled a second time for 1,000 revolutions at 70 r.p.m. After this, the same procedure as outlined above is gone through again for a third



1. Laboratory or work sample representatively taken and prepared by A.S.T.M. methods from the coal on which the grindability rating is desired.

2. Reduce by progressive crushings to minus 10 mesh, mix and riffle into two equal parts.

3. Retain for duplicate determination.

4. Riffle into two equal parts, and air-dry to constant weight.

5. Representatively take 500 grms.

6. Discard remainder.

7. Place in Abbé pebble mill jar with 3000 grms. assorted flint pebbles and rotate for 1000 revolutions at 70 r.p.m.

8. Remove charge from jar, separate coal from pebbles, and screen over 100-mesh sieve.

9. Discard the minus 100-mesh material.

10. Weigh the plus 100-mesh material and add sufficient 10-mesh coal from X above to make up to

11. Return to jar with same pebble charge and rotate for 1000 revolutions at 70 r.p.m.

12. Remove charge from jar, separate coal from pebbles, and screen over 100-mesh sieve.

13. Discard the minus 100-mesh material.

14. Weigh the plus 100-mesh material and add sufficient 10-mesh coal from X above to make up to

15. Return to jar with same pebble charge and rotate for 1000 revolutions at 70 r.p.m.

16. Remove charge from jar, separate coal from pebbles, and screen over 100-mesh sieve.

17. Discard the minus 100-mesh material.

18. Weigh the plus 100-mesh material and subtract the weight from 500 which gives **THE GRINDABILITY "RATING" OR INDEX NUMBER**

Figure 1. Graphic description of the F.R.L. method for rating the grindability of coal.

milling period, which concludes the operation. The weight in grammes of the plus 100-mesh material obtained after the third milling subtracted from 500 gives the "Grindability Index Number" of the sample.

From the foregoing it will be noted: first, that the milling is done in three stages for a total of 3,000 revolutions at a constant rate of 70 r.p.m.; second, that the sample is "sweetened" with fresh make-up coal after removal of the fine material produced during the first and second milling stages; and third, that the index number is secured by purely experimental means requiring no technique or equipment outside the purview of the average laboratory. Plate I shows the equipment ready for test, while Figure 1 graphically shows the various stages of the process. Those readers who may wish to try the method for themselves will find the procedure written up in detail in the appendix, which for the sake of ready comparison also includes the detailed procedure used in both the Cross and Hardgrove methods.

DEVELOPMENT OF THE METHOD

The method outlined above was arrived at after some considerable experimentation and comparative check-up of both the Cross and Hardgrove methods, with each of which it was found to be in substantial agreement. It is advocated for the following reasons:—

1. The equipment required for the determination is simple and is such as obtains in the average small laboratory; the procedure is not unduly complex and is rapid enough to allow from 4 to 6 determinations per day by a single operative; and the technique involved is not beyond the average laboratory assistant or helper.

2. The laboratory sample on which the index number or grindability factor is obtained should be representative of the coal on which the rating is desired. This is provided for by using standard sampling methods such as those recommended by the A.S.T.M.

3. Preliminary crushing of the work sample is done in a progressive manner which not only guards against overcrushing of the sample but also ensures about the same amount of work being done on each sample before it is subjected to the grindability test. This, from the writers' viewpoint, is important as they believe that the "crushability" or friability of a coal has a bearing on its grindability. Wilson, Yancy, and Daniels[1] have also noted this point.

4. The coal undergoing test is milled for a reasonable length of time under as efficient conditions as are possible, with means provided for removing the finer particles and for adding fresh (make-up) coal to the sample as the milling proceeds. This conforms more nearly than either of the other methods to commercial milling practice in which the finer coal is constantly being bled from the pulverizer as fresh feed takes its place. Aside from the above, the writers' primary idea in "sweetening" the coal charge during the milling operation was to ensure that the proper amount of work was done on the harder coal particles which are cushioned or protected to some extent by the finer, softer particles, which are subjected to more than their proportionate share of the work, unless means are provided for exposing the harder particles to the grinding action.

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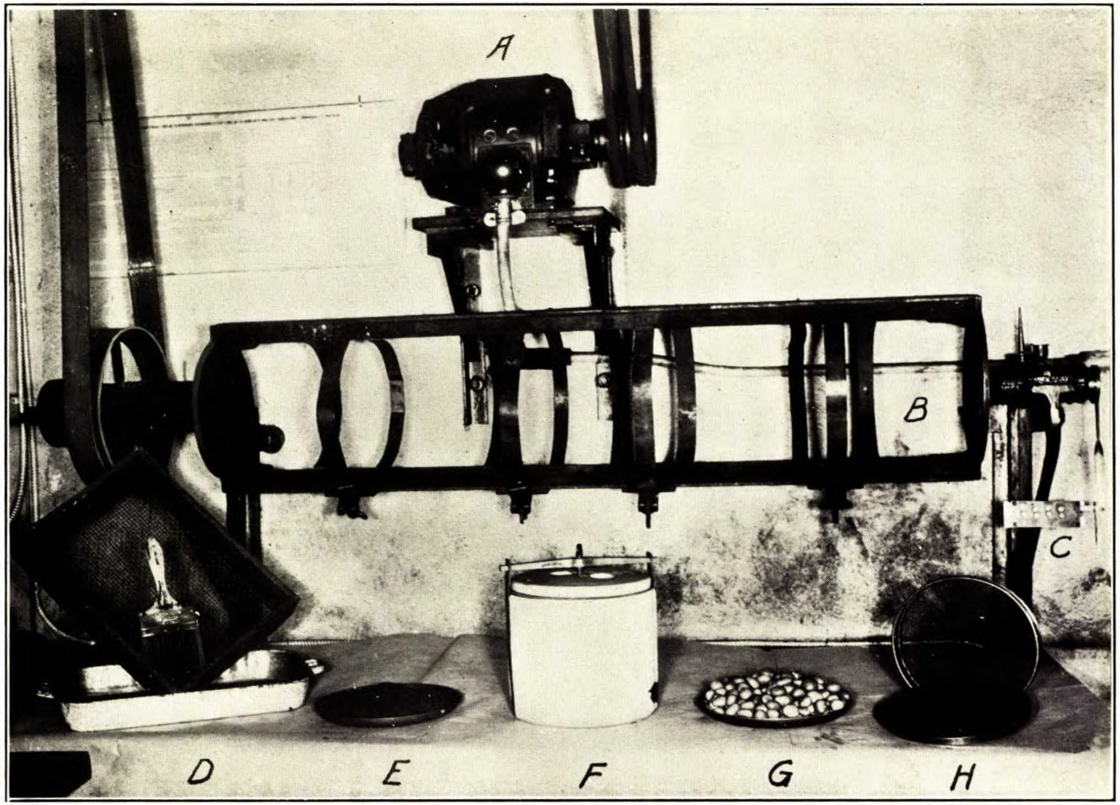
[1] "Preliminary Tests of Thirteen Washington Coals in a Powdered Coal Boiler Plant," University of Washington, Engineering Experiment Station, Bulletin No. 58.



A. Variable speed motor.
B. Abbé jar mill frame, speed of rotation 70 r.p.m.
C. Veeder revolution counter.
D. Box screen, ¼-inch mesh, brush, and pan.
E. 500-gramme sample of air-dried coal.
F. Abbé standard "Trojan" jar.
G. 300-gramme charge of assorted flint pebbles.
H. Tyler, standard, half-size, 100-mesh sieve with bottom pan and cover.

5. The index number or grindability factor is obtained purely by experimental means which do away with hypothetical, mathematical calculation.

During preliminary experimentation a Pittsburg seam coal known to give index numbers standing about midway of both the Cross and Hardgrove scales was selected for treatment in accordance with the foregoing procedure. A large sample of this coal was selected and split up into 5 separate work samples, each of which was crushed to pass a 10-mesh sieve. A screen analysis made on a representative portion of each of these samples gave quantities varying between 25 and 30 per cent through a 200-mesh sieve. A determination was then made on 3 of these samples and this gave index numbers of 261, 260, and 255 respectively. All the minus 200-mesh material was removed from the fourth work sample on which a determination then gave an index number of 259. The minus 200-mesh material removed from the fourth work sample was added to the fifth work sample on which a determination gave an index number of 269. From these results it was concluded that the method not only gave consistent results on the same coal sample but also permitted considerable variation in respect to the super-fine material in the original coal charge without unduly influencing the index number.

Determinations were then made on two coals varying widely in rank and known to have very poor and very good grinding characteristics respectively. These determinations gave index numbers of 153 and 376. From these results it was concluded that the index numbers derived by this method have a sufficiently wide range to permit of evaluating coals of intervening rank and grade. Determinations were next made on a number of coals, representative samples of which were readily available for the purpose. The results of these determinations in comparison with the results of similar determinations on the same coals by both the Cross and Hardgrove methods follow.

RESULTS OF TESTS

Twelve coals, for which the results of practical combustion tests were available for comparison, were selected for testing by the three different grindability methods mentioned. These coals ranged in rank from lignitic to high-grade bituminous. Without going into detail the principal results of both the combustion and grindability tests are presented in the following tables, which are self-explanatory.

In considering Table II which lists the average results of the combustion tests made with each fuel, it will be noted that the fuels are arranged in the order of their increasing calorific value (as fired). This, as would be expected, brings them roughly in line in respect to their rank as indicated by the specific volatile index. Moreover, as the average efficiencies and overall combustion conditions hold surprisingly uniform for all the fuels, the unit evaporation varies almost directly with these calorific values. No very definite relationship, however, is shown between calorific values or rank, and fineness of, or power requirements for pulverization, beyond a general trend towards increased fineness and decreased power as calorific values and rank increase. If, however, the fineness as given by the percentage through either the 100- or 200-mesh sieve for the individual tests on each fuel is plotted against the corresponding power requirements, a curve will be obtained for each fuel. From these curves a value may be

TABLE II

Average Data Taken from Combustion Tests[1] With Various Coals Burned in the Experimental Pulverized Fuel Fired Boiler Installation at the Fuel Research Laboratories, Ottawa, Canada.

Fuel[2] sample	Specific volatile index[3]		Screen analysis, fuel as fed to pulverizer					Proximate analysis fuel as fired				Calorific value fuel as fired gross B.T.U./lb.	Ash fusion temperatures		
	Number	Rank	$+\frac{3}{4}''$	$-\frac{3}{4}''$ $+\frac{1}{2}''$	$-\frac{1}{2}''$ $+\frac{1}{4}''$	$-\frac{1}{4}''$ $+\frac{1}{8}''$	$-\frac{1}{8}''$	Mois-ture	Ash	V.M.	F.C.		Initial	Soft	Fluid
			%	%	%	%	%						°F.	°F.	°F.
A	106·7	Black lignite (ortho lignitous).	0·0	5·6	38·8	24·5	31·1	23·0	13·6	26·9	36·5	8,162	1996	2093	2261
B	116·4	Black lignite (ortho lignitous).	0·0	6·4	40·7	24·0	28·9	19·9	9·2	30·6	40·3	9,405	2026	2129	2299
C	134·6	Sub-bituminous (meta lignitous).	0·0	3·1	32·3	24·3	40·3	9·8	11·2	32·5	46·5	11,118	2080	2237	2458
D	148·6	Sub-bituminous (meta lignitous).	0·0	0·0	0·4	24·6	75·0	5·9	17·0	34·7	42·4	11,305	2090	2179	2344
E	140·0	Sub-bituminous (meta lignitous).	0·0	3·8	29·0	22·3	44·9	9·1	11·7	34·9	44·3	11,310	2481	2604	2616+
F	161·1	Bituminous (para bituminous).	0·0	0·4	4·2	26·1	69·3	4·5	16·8	22·5	56·2	11,773	2606+	2700+	2700+
G	150·4	Sub-bituminous (meta lignitous).	0·0	0·0	0·7	34·1	65·2	4·6	14·1	36·3	45·0	11,985	2076	2237	2278
H	159·5	Sub-bituminous (meta lignitous).	0·0	0·0	19·0	31·2	49·8	3·8	14·6	30·1	51·5	12,268	2431	2462	2524
I	152·6	Sub-bituminous (meta lignitous).	0·0	4·1	30·6	24·6	40·7	3·5	12·0	36·3	48·2	12,528	2242	2312	2371
J	163·5	Bituminous (para bituminous).	1·6	7·5	22·4	23·9	44·6	3·5	12·9	30·6	53·0	12,690	2156	2183	2291
*K	166·2	Bituminous (para bituminous).	0·0	1·0	20·5	25·2	53·3	2·9	8·8	31·4	56·9	13,470	2465	2587	2622+
*L	176·5	Bituminous (ortho bituminous).	0·0	1·5	19·6	20·0	58·9	2·0	7·0	27·4	63·6	14,057	1968	2038	2358

TABLE II—Concluded

Average Data Taken from Combustion Tests with Various Coals Burned in the Experimental Fuel Fired Boiler Installation at the Fuel Research Laboratories, Ottawa, Canade.

Fuel[2] sample	Screen analysis, pulverized fuel as delivered to burner			Pulverizer[4]			Combustion data						
	On 50 mesh	Through 100 mesh	Through 200 mesh	Power per net ton fuel pulverized	Rate		Fuel fired per 1,000 lb. equivalent evaporated	Equivalent evaporated per lb. fuel fired	CO_2 in flue gas	CO in flue gas	Excess air	Rated boiler capacity developed	Thermal efficiency based on fuel as fired
					Coal fed per hour	Grind. % of rated capacity							
	%	%	%	kw. hr.	lb.		lb.	lb.	%	%	%	%	%
A	9·8	65·6	47·6	49·1	401	40·1	183·4	5·47	12·9	0·0	42	93	65·0
B	11·3	64·8	38·3	46·1	402	40·2	155·4	6·46	13·6	0·0	37	110	66·6
C	3·4	79·1	54·7	45·3	400	40·0	132·2	7·57	14·4	0·0	27	129	66·2
D	1·8	88·0	68·8	48·6	399	39·9	132·0	7·60	14·7	0·0	22	129	65·3
E	3·6	79·5	54·4	49·4	399	39·9	133·5	7·50	13·8	0·0	32	128	64·3
F	0·6	96·0	85·0	60·1	397	39·7	121·1	8·27	15·5	0·0	18	139	68·1
G	2·2	86·0	65·9	46·8	398	39·8	123·2	8·13	14·3	0·0	26	137	66·1
H	2·1	85·1	65·2	44·8	400	40·0	121·3	8·25	14·3	0·0	23	140	65·3
I	1·4	88·9	70·8	44·6	399	39·9	117·8	8·51	14·6	0·0	22	143	65·9
J	1·9	89·7	71·6	46·0	398	39·8	115·0	8·71	14·3	0·0	26	147	66·6
*K[1]	2·6	85·7	65·9	41·2	396	39·6	112·3	8·91	15·1	0·0	18	150	64·3
*L[1]	1·1	93·3	78·5	42·5	383	38·3	102·5	9·76	15·1	0·0	16	158	67·4

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[1] Twelve coals were included in the series; 4 tests were made on each of 10 of them; 3 on the remaining 2, which are so marked*. The rate of coal feed was approximately 200, 400, and 600 lb./hr. respectively on each of these coals, the fourth test being a repeat on 10 of them at the 400-lb. rate. The figures given in the above table are average values for all tests on each coal.

[2] The fuels were British Columbia coals with the exception of one, viz. sample K, which was a non-competing eastern coal, arbitrarily selected for standardization and general use purposes because of its constant quality and ready availability in the Ottawa market. It will be noted that the fuels are listed in the order of increasing calorific value.

[3] See "A Classification of Coals for Use in the By-product Coking Industry," by Burrough & Swartzman, Mines Branch Memorandum Series No. 55.

[4] Oversize pulverizer serving general purpose test installation in which peats and low-grade lignites were to be used as well as higher ranking fuels. Pulverizer rated at 1,000 lb./hr. on average grade bituminous coal. Combustion rates of over 600 lb./hr. with average grade bituminous coal are beyond the scope of practical operation for this installation.

secured in respect to fineness at one power rate, viz. the average power rate for all tests on all fuels, and such arrangement is shown by Table III to approximate closely to a similar arrangement of the fuels in respect to increasing grindability index numbers. This is the only correlation that can be made at the present time with the information at hand, between grindability ratings given by the laboratory method and similar results obtained in a commercial pulverizer.

Table IV gives the index numbers obtained by the F.R.L. method for independent determinations on duplicate samples of each coal from which an idea may be obtained of the effect the "sweetened" coal charge has on the index during the various stages of milling. An interesting feature shown by this table is that the average difference between the two determinations decreases with each stage of milling to a point after the third or final stage, which might well be considered to be within the limits of experimental error. The consistency of the final or "grindability" index numbers for the two determinations on each fuel is also shown, as well as the wide range that exists between the respective grindability index numbers for coals of low and high rank.

Table V correlates the grindability indices obtained on the same coal samples by the three methods discussed herein. From this table it will be seen that the F.R.L. method gives as consistent results as either of the other two in respect to the grindability ratings obtained for these particular coals. However, all three methods are virtually in substantial agreement in respect to grindability ratings, and any one of them might serve as a basis for standardization. The choice therefore will lie with the one that requires the simplest procedure and gives the greatest accuracy on a number of coals, and best agreement with results actually obtained with commercial grinding. The simplest procedure is, we believe, best provided by the F.R.L. method for reasons already given. Accuracy, however, can be settled only by correlation of the laboratory methods with commercial results on a wide variety of coals.

TABLE III

Fuels Arranged in Accordance With Various Factors to Show the Relative Order Holding Between Them

Fuels arranged in order of increasing									
Calorific value as fired		Rank as given by the Specific Volatile Index No.		Percentage pulverized fuel through				Grindability index number F.R.L. method	
				100-mesh sieve		200-mesh sieve			
				At an average (constant) power consumption (taken from data and curves not given)					
Cal. Val.	Fuel	S.V.I. No.	Fuel	Per cent	Fuel	Per cent	Fuel	Gr. Index No.	Fuel
8162	A	106·7	A	65·3	B	39·0	B	153	B
9405	B	116·4	B	65·3	A	48·2	A	172	A
11118	C	134·6	C	79·5	E	54·3	E	210	E
11305	D	140·0	E	79·5	C	56·0	C	228	C
11310	E	148·6	D	85·0	H	65·2	H	245	K
11773	F	150·4	G	86·2	K	66·0	K	262	H
11985	G	152·6	I	86·5	G	66·1	G	271	G
12268	H	159·5	H	87·3	D	69·2	D	276	D
12528	I	161·1	F	90·0	I	72·3	I	282	I
12690	J	163·5	J	90·3	J	73·0	J	283	J
13470	K	166·2	K	93·0	L	78·2	L	341	F
14057	L	176·5	L	95·8	F	83·3	F	374	L

TABLE IV

Comparative Study of the Effect the "Sweetened" Coal Charge has on the Index Number

Fuel[1] sample	First milling stage after 1,000 revolutions				Second milling stage after 2,000 revolutions				Third or final milling stage after 3,000 revolutions F.R.L. method			
	Preliminary Index No.				Intermediate Index No.				Grindability Index No.			
	Det. No. 1	Det. No. 2	Average	Range	Det. No. 1	Det. No. 2	Average	Range	Det. No. 1	Det. No. 2	Average[1]	Range
B	140	138	139	2	146	144	145	2	153	153	153	0
A	157	155	156	2	163	169	166	6	170	174	172	4
E	204	204	204	0	208	208	208	0	209	210	210	1
C	211	226	219	15	221	233	227	12	225	231	228	6
K	249	251	250	2	242	243	243	1	246	244	245	2
H	258	270	264	12	267	266	267	1	261	263	262	2
G	271	285	278	14	273	284	279	11	268	274	271	6
D	276	291	284	15	285	288	287	3	273	279	276	6
I	290	271	281	19	282	275	279	7	284	280	282	4
J	277	281	279	4	297	282	290	15	286	280	283	6
F	361	351	356	10	343	339	341	4	340	341	341	1
L	368	362	365	6	369	369	369	0	376	372	374	4
	Total range...........			101	Total range...........			62	Total range...........			42
	Average range........			8·4	Average range........			5·2	Average range.........			3·5

[1] Fuels arranged in the order of increasing grindability index number.

TABLE V

Correlation of Grindability Indices Obtained on the Same Coal Samples by the F. R. L., Cross, and Hardgrove Methods

Fuel[1] sample	Grindability Index Numbers obtained by												
	F.R.L. method				Cross method					Hardgrove method			
	Det. No. 1	Det. No. 2	Average[1]	Range	Det. No. 1	Det. No. 2	Det. No. 3	Average	Range	Det. No. 1	Det. No. 2	Average	Range
B	153	153	153	0	200	250	214	221	50	37·5	36·4	37·0	1·1
A	170	174	172	4	267	266	....	267	1	44·8	38·4	41·6	6·4
E	209	210	210	1	300	285	343	309	58	53·7	51·1	52·4	2·6
C	225	231	228	6	350	365	373	363	23	61·4	57·7	59·6	3·7
K	246	244	245	2	445	476	369	430	107	71·3	67·1	69·2	4·2
H	261	263	262	2	437	442	448	442	11	74·0	66·6	70·3	7·4
G	268	274	271	6	475	510	451	479	59	74·5	74·8	74·7	0·3
D	273	279	276	6	475	455	458	463	20	78·4	81·7	80·1	3·3
I	284	280	282	4	526	494	468	496	58	80·6	78·3	79·5	2·3
J	286	280	283	6	521	516	484	507	37	71·7	66·4	69·1	5·3
F	340	341	341	1	532	524	513	523	19	91·7	91·7	91·7	0·0
L	376	372	374	4	686	699	626	670	73	96·3	101·9	99·1	5·6

[1] Fuels arranged in the order of increasing (F.R.L. method) grindability index number.

APPENDIX

RECOMMENDED PROCEDURE FOR RATING THE GRINDABILITY OF COAL

A.—The Fuel Research Laboratories' (F.R.L.) Method.
B.—The Cross Method.
C.—The Hardgrove Method.

A. THE FUEL RESEARCH LABORATORIES' (F.R.L.) METHOD

PREPARATION OF SAMPLE

A representative laboratory or work (net) sample of approximately 6 pounds of the coal on which the grindability determination is to be made, is required. This amount should be obtained from the bulk or gross sample by the recommended A.S.T.M. method of progressive reduction which involves the standardized procedure of crushing, coning, and quartering. The total quantity of the net sample so obtained should then be passed over an 8-inch diameter, 9-mesh Tyler standard screen, U.S. Series equivalent No. 10. The minus 10-mesh product that is removed should be retained in a large pan, and the plus 10-mesh material is to be passed through a small roll crusher having its rolls spaced (by measurement) $\frac{1}{2}$ inch apart. The resultant product is again screened over the same 10-mesh sieve so as to remove the minus 10-mesh product, which is to be added to that previously obtained. This process of alternately crushing and screening is to be repeated a number of times, progressively reducing the clearance between the rolls of the crusher to $\frac{1}{4}$, $\frac{1}{8}$, and $\frac{1}{16}$ inch in succession and screening between each crushing so as to remove the minus 10-mesh product which is to be added to that previously obtained. After the sample has been passed through the crusher with rolls set to $\frac{1}{16}$-inch clearance and the minus 10-mesh product has been screened out, a considerable quantity of plus 10-mesh material will still remain. This is to be reduced in size by setting the crusher with the rolls just barely touching. Pass the remaining plus 10-mesh material through the rolls, screen out the minus 10-mesh product as before and again pass the plus 10-mesh material through the rolls *without altering the setting*. The above procedure should reduce the entire net sample to practically all minus 10-mesh and the plus 10-mesh material remaining after the 6th crushing should not exceed 1 per cent of the original sample. It will be noted that the sample has been reduced by a succession of six crushings with the roll clearance set to $\frac{1}{2}$, $\frac{1}{4}$, $\frac{1}{8}$, $\frac{1}{16}$ inch, just barely touching first pass and just barely touching second pass. These settings may be obtained accurately enough by means of a set of four wooden thickness gauges which may easily be constructed. If more than one net sample is to be treated great care should be taken that the same settings obtain for the different samples so that the crushing effect on each sample will be the same.

The entire net (6-pound) sample of minus 10-mesh material should now be well mixed by rolling on a square of oilcloth and then riffled into two equal parts. One part should be set aside in a sealed container as a reserve for a check determination if such is later required. The other part should be again mixed as before and again riffled into two equal parts, each of which should be thinly and evenly spread out in a shallow pan and be air-dried in the laboratory to constant weight and further treated as follows:—

METHOD OF TEST

After the air-dried coal has reached constant weight 500 grammes (approximately 1 pan full) is weighed out and placed in a standard Abbé pebble mill jar of approximately 1-gallon capacity, together with a charge of 3,000 grammes of flint pebbles assorted in size between ½ and 1 inch. The jar with its coal and pebble load should then be rotated for 1,000 revolutions at a speed of 70 r.p.m., after which the load should be poured into a ¼-inch mesh, box screen placed over a pan and the pebbles separated from the coal by brushing them back and forth with a soft bristle brush over the surface of the screen. The coal collected in the pan below should then be carefully screened over a 100-mesh Tyler standard screen, U.S. Series equivalent No. 100, and all the minus 100-mesh product removed and discarded. If a Tyler 8-inch diameter screen is used for this operation the most accurate results are obtained by screening the 500 grammes of coal in lots of approximately 100 grammes in order to prevent over-loading of the screen. The plus 100-mesh material remaining after the screening is retained. A note should be made of the weight and enough of the air-dried coal from the second pan should be added to make up to 500 grammes. This 500-gramme sample is again returned to the jar together with the same pebble charge previously used and again rotated for 1,000 revolutions at 70 r.p.m. The pebbles should then be separated from the coal as before and the minus 100-mesh product removed and discarded. After again adding enough of the air-dried coal from the second pan to make up to 500 grammes, the charge is again returned to the mill and rotated for another 1,000 revolutions at 70 r.p.m. The pebbles are again separated from the coal as before and the coal again screened over the 100-mesh sieve. The weight of plus 100-mesh material obtained is subtracted from 500 which gives the amount of the sample passing the 100-mesh sieve and this is the result to be recorded as a relative measure of the grindability of the coal.

PRECAUTIONS

Extreme care should be taken in screening the milled sample on the 100-mesh screen as the tendency is to overload the screen, with the result that an incorrect result may be obtained. If each ±100-gramme portion of the sample is screened a sufficient length of time so that only $\frac{1}{10}$ gramme passes through the screen in one minute a correct result has been obtained. By screening in a "Ro-Tap" mechanical sieve shaker for 3 minutes, then cleaning out the bottom pan and brushing the lower surface of the screen and again screening for 1 minute, the desired result will usually be obtained, but if a greater amount than $\frac{1}{10}$ gramme passes the screen in this time, the screening should be continued for 1-minute intervals until this condition is reached.

B. THE CROSS METHOD[1]

"The coal to be tested should be air-dried to constant weight in the laboratory. It should then be carefully crushed so that practically all goes through a 10-mesh screen, care being taken that it is not overcrushed. As a precaution against overcrushing the material through 10-mesh should be about 98 per cent. A sample of the crushed coal should then be screen-sized using A.S.T.M. screens Nos. 10, 20, 40, 60, 100 and 200 mesh. A sample of the crushed coal—200 grammes in weight—should then be ground in a ball mill for 400 revolutions. The ball charge should be 89 three-quarter-inch steel balls. The speed of the mill should be between 70 and 80 r.p.m. The ground coal should be again screened using the same screens used in the sizing of the initial product.

The surface factor for the initial and final products should then be computed using the following surface factors and the grinding index obtained by subtracting the surface factor of the initial product from that of the final product.

Material	Factor	Before Grinding		After Grinding	
		Per cent	Product	Per cent	Product
− 10 + 20	1				
− 20 + 40	2·25				
− 40 + 60	4·24				
− 60 + 100	7·10				
−100 + 200	12·7				
−200	25·4				
Totals		A		B	

Difference (B-A) Index No.

The grinding characteristics of the coal may then be taken from the curve Figure 14.[2] This curve has been constructed on data obtained in field tests on a 15-ton roller mill. This is the same curve as Figure 5 [2] except that the value of the abscissa has been changed. The value 15 tons per hour which corresponds to rated capacity of the mill has been called 100. This value corresponds to Pittsburg No. 8 coal which may be called an average bituminous coal. The abscissa values of the curve of Figure 14 may be interpreted as being the per cent of capacity to be expected if the mill is rated on Pittsburg coal."

C. THE HARDGROVE METHOD[3]

OPERATION OF GRINDABILITY MACHINES

"The capacity of any type of pulverizer depends on the resistance to crushing of the material being pulverized; this characteristic of the material has been given the name Grindability. Tests conducted over a period of years have shown conclusively that the capacity of a pulverizer

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[1] Quoted from "Progress Report on the Development of a Method for the Determination of the Pulverizing Characteristics of Coal," submitted by John Van Brunt to Subcommittee VII, Committee D-5, A.S.T.M.

[2] Not reproduced in this report.

[3] Quoted from "Operating Instructions Issued September 30, 1932," by the Babcock & Wilcox Company, builders of the Hardgrove grindability machine.

grinding a hard material such as cement clinker will be considerably less than the capacity of the same pulverizer when working on a softer substance such as limestone. Similarly, tests have shown that bituminous coals of low-volatile content crush more easily than those of higher volatile content. Hence, there came into being the need for some sort of a scale which would show the grindability of various materials as compared with a standard. The standard grindability which has been adopted is carefully explained in the paper entitled "Grindability of Coal," a copy of which is attached hereto.[1] It is suggested that this paper be read over carefully before running any tests on grindability as it covers the development work which led to the adoption of the Ball Machine as the Grindability Machine.

SAMPLING AND PREPARATION OF SAMPLE FOR TEST

As in all coal testing, emphasis must be placed on the fact that no laboratory results can be better or more representative than the sample submitted. It is, therefore, necessary to give the same attention to sampling and preparing a sample for grindability tests as required in sampling for any other analyses.

To ensure a true representative sample of material, the method employed in collecting a sample of coal for a calorific test should be followed. The gross sample should consist of at least two pounds of ¼-inch and finer material.

The gross sample (two pounds) after air-drying until the loss in weight is not more than 0·1 per cent per hour, is put on the No. 16 U.S. sieve nested with a No. 30 U.S. sieve and bottom pan. The nest of sieves can be hand tapped or put on a mechanical sieve-shaking machine for about two minutes, to remove all material finer than No. 16. The material remaining on the No. 16 sieve is put into a coffee mill with the set screw adjusted so that the coarsest particles are broken up. The sample, after passing through the coffee mill, is returned to the No. 16 sieve of the nest of sieves and again hand tapped or put on the mechanical shaker to remove material finer than No. 16. This operation is repeated several times, gradually tightening up the set screw until the entire sample has passed through the No. 16 sieve. The material remaining on the No. 30 sieve is then shaken to thoroughly remove any material finer than No. 30; the material passing the No. 30 being rejected. By using this method of preparing the sample for grindability determinations, a minimum of fines is produced, which is essential to obtaining representative samples for test.

OPERATION OF THE MACHINE

First, the predetermined counter must be set so that the machine will automatically stop just after 60 revolutions of the machine. To ensure stoppage of the machine within one-quarter of one revolution, two trippers are furnished. One tripper is fixed, and the other one may be set to correspond with the coasting of the machine after the switch is thrown. Therefore, the number of counts on the counter should be 120 or just twice the number of revolutions made by the machine. Second, the grinding elements should be thoroughly cleaned by brushing before the sample to be tested is added.

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[1] Not reproduced in this report.

A 50-gramme portion of the prepared sample (between No. 16 and No. 30 sieves) is weighed out. The eight 1-inch diameter balls are put in the bottom of the lower grinding element and the weighed sample is distributed evenly over the balls, any material falling on the elevated section of the lower grinding element being brushed toward the balls. The upper grinding element is then placed on the balls and the lower end of the shaft is fitted to the square opening in the disk, and the grinding elements are securely fastened in place by the two thumb screws on the sides of the lower grinding element. The utmost care should be taken to pull the bowl up evenly with the thumb screws as uneven pressure on one side will give erroneous results. The set screw on the collar, which supports the driving gears should always be kept tight, otherwise the weights will rest on the frame and there will be no pressure on the balls. The predetermining counter is set to zero, and the starting switch is closed. After the machine has been automatically stopped by the predetermined counter at 120 counts, or 60 revolutions, transfer the sample to the No. 16 sieve of a telescoped nest containing the Nos. 16, 30, 60, 100, 140, 200 and 230 U.S. sieves and No. 300 Tyler sieve, and bottom pan. The Nos. 16, 30, 60, 100 and 140 sieves can be half height or 1-inch screens, and the 200, 230, 300 and pan are best full height or 2-inch sieves; all sieves being 8 inches in diameter. The balls and upper and lower grinding elements are carefully brushed to remove all of the ground sample with a minimum loss of material.

SCREENING OF THE PULVERIZED SAMPLE

The physical properties of many samples of material, forwarded to laboratories for grindability test, are such that in the finely pulverized form they do not screen readily. Thus, to avoid any irregularities due to screening, the following procedure should be followed. The pulverized material is screened on the Nos. 16, 30, 60, 100, 140, 200, and 230 U.S. sieves, and No. 300 Tyler sieve. All samples should be screened for ten minutes on a mechanical shaker or be given an equivalent amount of hand screening. The screens are then separated and the under side of each screen is carefully brushed from underneath, while holding the sieve in an inclined position in such a way that any particles removed by brushing fall on the sieve beneath, thus keeping the loss of any portion of the sample at a minimum. The sample is then screened for five minutes more, after which the brushing of the under side of each screen is repeated. After screening for five minutes more, the material is ready for weighing. This gives a total of 20 minutes of screening with comparatively clean sieves.

WEIGHING THE SCREENED SAMPLE

Coals having a high residual moisture content, and this is especially true of lignites, lose some of their residual moisture in the pulverized condition, and for this reason rapid and fairly accurate weighing is essential.

A triple beam balance, such as the Cenco agate-bearing type having a sensitivity of 1 cg., with which weighings from 1 cg. to 111 grms. can be made, is well adapted to make the weighings, although a torsion or trip balance with suitable pan and of specified sensitivity can be used. Greater sensitivity than 1 cg. is not necessary for this work.

If the new surface units of the standard sample be 27,630, the grindability value equals 14,956 divided by 27,630 equals 54 per cent grindability.

The factors listed in the sample calculation are the reciprocals of the average diameter of the particles passing through the various screens. For instance, the opening of a No. 16 screen is 0·0469 in., the opening of a No. 30 screen is 0·0232 in., the average opening is 0·0350 in., making the reciprocal 29.

The material as prepared for grindability was originally between the No. 16 and No. 30 sieves. Multiplying through by the various factors, and adding the results, it is found that 8,983 surface units were produced upon the 50-gramme sample using the No. 1 set of sieves and 8,873 surface units upon another 50 grammes of the same material using No. 2 set of sieves, or a total of 17,856 surface units was produced upon 100 grammes of the sample. Since the original sample was between the No. 16 and No. 30 sieves, the reciprocal of the average opening, or factor of 29 surface units per gramme, or 2,900 surface units for 100 grammes, represents the sample before grinding and must be deducted from the final surface units of the sample after grinding, giving 14,956 new surface units produced in the grinding operation. If the new surface units of a standard sample treated in the same way were 27,630 the grindability value is taken as the ratio of the two, or 14,956 divided by 27,630 equals 54 per cent grindability.

In the example as shown, taken from an actual determination a discrepancy of 110 surface units exists between two 50-gramme samples and a recovery of 49·6 grammes was obtained in each case. The discrepancy of 110 surface units amounts to 0·4 per cent and is considered as a good check. If the discrepancy between two such determinations is more than 550 surface units, a third test should be made and the faulty one discarded. If proper attention is given to the details of operation and screening, there should be no trouble in obtaining satisfactory results with this machine.

CONCLUSION

Care should be taken in setting up the Grindability Machine, and after the machine is erected several runs should be made on the standard coal which is furnished with each machine. The machines have been calibrated in the laboratory before being sent out, so if the standard coal checks, the machine is working properly. Check samples on the standard coal should be made from time to time to make sure that the machine is in good order. It should be remembered that the human element enters into grindability determinations so care must be taken in all operations."

Accumulative weighing is used to weigh the screened portions to eliminate the necessity of cleaning the pan after weighing the contents of each screen. The material remaining on the No. 16 sieve is transferred to the balance pan and weighed to within 0·1 gramme and the weight recorded. The contents of the No. 30 sieve are added to the material on the balance pan and weighed and the weight recorded. The content of each screen is thus weighed accumulatively and the weight of material between any two screens is determined by subtraction. The amount of material through the No. 300 is taken as the difference between the total weight remaining on the 16, 30, 60, 100, 140, 200, 230, and 300 screens and the original weight of sample taken.

The material remaining on the pan can be weighed to determine how much of the original sample is recovered, but it does not enter into the calculation, the amount of material through the No. 300 being determined by difference as stated above. The loss in material after pulverizing, screening, and weighing should be less than two per cent of the sample. The material lost as fine dust would, without doubt, pass through the No. 300 sieve and is, therefore, calculated in this manner.

CALCULATION OF RESULTS

After weighing the screened portions of the pulverized sample, it is necessary to calculate the results to surface units produced, in order to obtain the grindability of the sample. A typical calculation of two 50-gramme portions of the same material, using two sets of sieves, is as follows:—

U.S. Series *sieves	Grammes between these two screens		Factor	Final surface	Units
	No. 1 Set of Sieves				
16- 30	14·3	×	29	415	
30- 60	17·3	×	61	1,055	
60-100	6·4	×	129	826	
100-140	3·2	×	202	646	
140-200	2·2	×	285	627	
200-300	1·4	×	377	528	
230-300	0·6	×	476	286	
Through 300	4·6	×	1,000	4,600	
					8,983
	No. 2 Set of Sieves				
16- 30	14·1	×	29	409	
30- 60	17·8	×	61	1,086	
60-100	6·7	×	129	864	
100-140	3·0	×	202	606	
140-200	2·0	×	285	570	
200-230	1·2	×	377	452	
230-300	0·6	×	476	286	
Through 300	4·6	×	1,000	4,600	
					8,873
Final Surface Units (100 grammes)					17,856
Less Original Surface Units					2,900
New Surface Units					14,956

*No. 300 sieve is made by Tyler.

III

LABORATORY TEST ON COALS FOR PREDICTING THE PHYSICAL PROPERTIES OF THE RESULTANT BY-PRODUCT COKES

E. Swartzman, E. J. Burrough, and R. A. Strong

INTRODUCTORY

The suitability of a coke for a specific purpose may be judged largely by its physical properties. The properties to be regarded as important are structure, size, impact hardness, resistance to abrasion, true and apparent specific gravities, porosity, and bulk density, and in most cases a definite numerical value can be assigned to them by means of standardized tests. Various laboratory test methods have been devised for predicting coke quality, and the consensus of opinion appears to be that none of these methods has sufficient merit to predict accurately the characteristics of the coke obtainable from a coal or blend of coals. However, the authors are of the opinion that by the aid of a single laboratory test, and the use of a chart and table as described below, coals can be satisfactorily classified in terms of quality of coke they will produce commercially.

The characteristics of coke in respect to structure and appearance comprise shape, cell structure, pebbliness, longitudinal and cross fractures, appearance of the natural surface, and colour. A coke may be blocky, fingery, or triangular in shape; it may have a uniform cellular structure or the cells may be of varying size with a decided lack of uniformity in the thickness of the cell walls; it may or may not be pebbly; it may have varying amounts of either straight or steppy cross fracture lines and varying amounts of longitudinal fissures; its surface may be smooth, granular, or irregular; and its colour may vary from steel grey to a dull greyish black. All these properties are assessed by visual observations to which no numerical value can be given, and yet they are mainly responsible for those physical properties which can be numerically evaluated. Three other properties which have a decided effect on the quality of coke and its consequent grading for a specific purpose may also be included here, namely hardness, toughness, and fragility.

The *size of coke* is determined by screening it as discharged from the ovens, on the following square mesh screens: 3-inch, 2-inch, 1-inch and ½-inch, the results being recorded as a percentage passing through and remaining on the respective screens. According to Mott and Wheeler[1], "the size specified in this way does not necessarily bear any relation to the length of the coke pieces, and thus to the oven width, but is governed by the maximum cross-sectional dimension". The size of coke, therefore, is to a degree controlled by the amount of longitudinal fracture.

The *impact hardness* of coke is evaluated by its resistance to shattering. This property is determined by means of a standard test, described by the A.S.T.M. in their publication under Serial Designation D141-23. In this test 50 pounds of coke is dropped four times from a height of 6 feet onto a steel plate, the shattered coke being screened on 3-inch, 2-inch, 1½-inch, 1-inch, and ½-inch square mesh screens. The cumulative percent-

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[1] "Coke for Blast Furnaces"—First Report of the Midland Coke Research Committee, 1930, pages 11 and 90.

age retained on the 2-inch screen is recorded as the "shatter index". It has been suggested by the Midland Coke Research Committee that the cumulative percentage on the 1½-inch screen should be used as a shatter index, especially when considering the use of coke for blast furnace operation. The experience of the authors, however, indicates that the 2-inch index enables a sharper division to be made of cokes into different classes. The impact hardness of coke, as determined by the shatter test outlined above, is directly related to the type and degree of both cross and longitudinal fractures in the coke.

The *resistance to abrasion* is evaluated in the Fuel Research Laboratories by the abrasion test developed by the Midland Coke Research Committee, which is called "The Sheffield Coke Abrasion Test"[1]. This test is outlined by that committee as follows: "In a drum 18 inches in diameter and 18 inches long, two cubic feet of three- to two-inch (dry) coke is revolved during a half hour at 23 r.p.m. A sieving analysis is then made, and the percentages of the charge remaining on a 1½-inch screen and passing through a $\frac{1}{16}$-inch screen are noted. The percentage remaining on the 1½-inch screen is taken to be the "abrasion index", and the percentage passing through the $\frac{1}{16}$-inch screen as the "dust due to abrasion."[2] The above test gives a numerical value for the friability of coke, and is especially important when the coke is to be used for blast furnace work where a high "abrasion index", over 90, and a low percentage of dust are imperative. The cell structure of a coke and its degree and type of fracturing are in the main responsible for the property of resistance to abrasion.

The *true and apparent specific gravities* and the *porosity* of cokes are determined by the A.S.T.M. methods described in their publication under Serial Designation D167-24. The true and apparent specific gravities are to a great extent related to the quantity of ash present in the coke, but the porosity depends on the size of the pores and thickness of the cell walls.

The *bulk-density* or weight per cubic foot of coke is dependent on the apparent specific gravity, shape, and size of the pieces of coke and the quantity of ash present. The bulk-density of coke is a measure of the quantity of voids in a charge and hence is a valuable criterion in comparing different cokes for use in any given process.

The above brief outline of the physical properties of coke and methods for their determination, indicates the difficulty encountered in any attempt to apply some simple test to coals, which will group together those coals used in the production of by-product cokes of similar physical characteristics. In a previous paper[3] on the classification of coal for by-product use, it has been shown that it is possible to classify a coal from its proximate analysis and calorific value, and to gain a general idea from the classification thus obtained of the physical properties of the resultant high-temperature coke. However, coals or blends of coals, although belonging to the same class, may yield cokes with varying physical characteristics, and hence the necessity for some specific laboratory test has arisen.

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[1] Op. cit.

[2] This terminology does not appear in the original quotation and has been inserted by the authors.

[3] A Classification of Coal and other Solid Fuels using Specific Volatile Index, by Burrough, Swartzman, and Strong—Invest. of Fuels & Fuel Test., 1930-31. Mines Branch, Dept. of Mines, Canada (1933).

The phenomenon of coking has been the subject of intensive study by many investigators, and a great deal of literature has been published on the subject. Considerable diversity of opinion exists as to the effect of the various constituents of a coal on its coking properties, but it is generally admitted that the physical changes occurring during coking due to differences in the chemical properties, play an important part in determining the type of coke which can be manufactured from any given coal. As a review of the work done on this subject and the variety of opinions held is essential in considering the problem of classifying coals according to the physical properties of their resultant cokes, the authors have included as an appendix to this paper a brief résumé of the opinions held by various outstanding authorities.

It is generally agreed that, when coal is charged into a hot oven, the temperature of that part of the charge in immediate contact with the heated walls rises very rapidly, and free moisture is driven off, much of it being temporarily condensed in the raw coal in the centre of the charge. The temperature of the charge rises fairly rapidly from 300° to 450° C. within which range the coal becomes plastic or fuses This fusion is preceded and accompanied by thermal decomposition, esspecially of the "beta" and "gamma" compounds[1], or the "oily" and "solid" bitumen[2], which are responsible in varying degrees for the coking and swelling powers of the coal. The degree of coking is not, however, solely due to these compounds but is to a great extent dependent on the physical condition of the non-fusible compounds (alpha fraction)[3]. Following the fusion period there is a rapid evolution of the volatile constituents, practically the whole of the tar being liberated below 600° C. leaving only a porous coke structure. The amount of the subsequent evolution of volatile matter above 600° C. is considered by many to be responsible for the quality of the coke produced, but as far as the authors can gather either from the literature or their own experiments, it does not appear that this residual volatile matter can be entirely responsible for the type of coke which results.

CRITICAL TEMPERATURE AND DEVELOPMENT OF METHOD

Previous work points to the fact that with practically all coking coals, the plastic stage has been passed at about 500° C. The plastic state of coal is accompanied by varying degrees of swelling and by the evolution of varying amounts of volatile matter. This swelling, which in the coke oven resolves itself into pressure, and the accompanying evolution of varying quantities of volatile matter, must play some part in determining the physical characteristics of the resultant by-product coke. However, it was found that at 500° C. there was no complete correlation between the quality of the coke and either the swelling, and the quantity of volatile matter evolved or the residual volatile matter at this temperature.

Between 500° C. and 600° C. the greatest thermal decomposition takes place, and shortly after 600° C. all the tar has been evolved. Hence

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[1] Wheeler, R. V.: The Chemistry of Coal, Fuel, vol. VI, No. 11, p. 1.

[2] Fischer, F., Broche, H., and Strauch, J.: The Coking Properties of Coal, Fuel in Science and Practice, V, No. 10, p. 466.

[3] Wheeler R.V.: Op. cit.

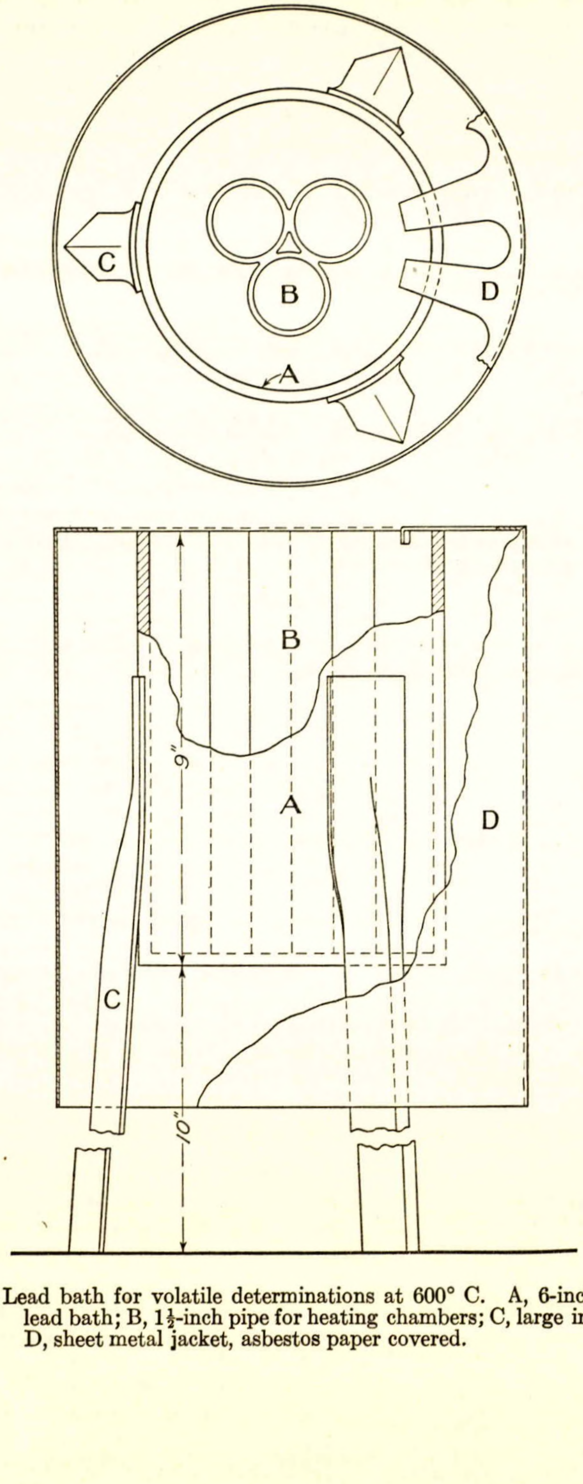


Figure 2. Lead bath for volatile determinations at 600° C. A, 6-inch pipe for lead bath; B, 1½-inch pipe for heating chambers; C, large iron tripod D, sheet metal jacket, asbestos paper covered.

it is considered that the temperature of 600° C. is a more critical point with respect to resultant coke quality than any other temperature and that the relation between swelling and volatile matter at this temperature may give the desired classification.

To check this conclusion a little more closely, the volatile matter evolved from the coals, and the volume of the resultant coke buttons, were obtained at 600° C. as well as at 500° C. and 950° C. The determination at 950° C. was according to the A.S.T.M. method, but for the lower temperatures of 500° C. and 600° C. a specially prepared lead bath of simple design as shown in Figure 2 has been found to be necessary for good temperature control. In all cases the 15 cubic centimetre platinum crucible is used, but at the two lower temperatures the usual seven-minute period of heating is increased to fifteen minutes to expel all the volatile matter given off at these temperatures. This increased period of heating did not change the volume of the "buttons" but gave the maximum amount of volatile matter.

The volatile matter determinations are calculated to the dry basis; and the volumes of the coke "buttons" determined in the following manner: the button is placed in a 200 c.c. graduated cylinder, while into another cylinder is measured 20 c.c. of fine sand, preferably screened so as to pass a 60-mesh screen and be held on a 100-mesh screen. This sand is carefully poured into the first cylinder containing the coke button until the sand reaches the 20 c.c. mark, the cylinder being tapped several times in order to obtain a uniform bulk density. The remaining sand is carefully measured in a smaller cylinder for sake of accuracy. The volume of the residual sand is equal to the volume of the coke button. In order to obtain uniformly shaped and sized buttons it is imperative to tamp the coal in the crucible so that the surface of the coal is level.

The marked variations in the volume of the buttons for the same coal or blend for the three temperatures 500°, 600°, and 950° C. are illustrated by the full-size photographs of a representative series in Plates II and III. It will be clearly seen that the buttons at 600° C. are entirely different from those at 950° C. and in most cases show the greatest amount of swelling.

A graph (Chart I) was prepared by plotting the volatile matter against the volume of the button obtained at the above three temperatures and for the sake of clarity only a few representative coals are shown on the chart. It will be noticed that the points for 600° C. seem to group coals which produce cokes of similar quality in such a way as to allow the formation of classes much better than when either of the other two temperatures is used.

SWELLING INDEX

The arrangement of coals tested at 600° C. does not entirely differentiate between the various types of coals and hence it is evident that at least one of the two factors used is not completely satisfactory for an acceptable classification. In analysing the two factors used, i.e. the volatile matter at 600° C. and the volume of the coke button obtained at this temperature, it was noted that two coals yielding different amounts of volatile matter may produce coke buttons of equal volume, hence it

PLATE II

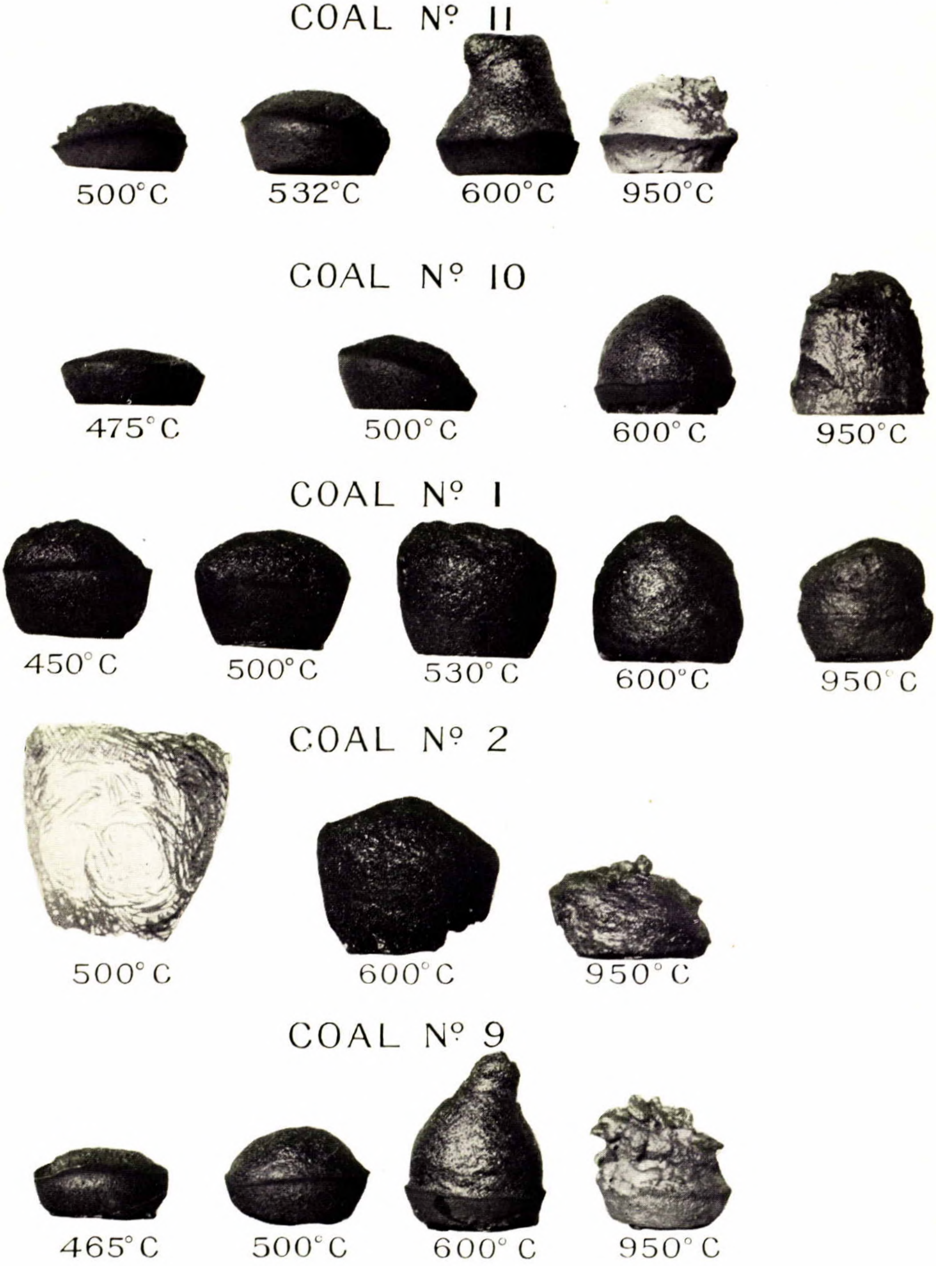

COAL Nº 11
500°C
532°C
600°C
950°C
COAL Nº 10
475°C
500°C
600°C
950°C
COAL Nº 1
450°C
500°C
530°C
600°C
950°C
COAL Nº 2
500°C
600°C
950°C
COAL Nº 9
465°C
500°C
600°C
950°C

PLATE III

COAL Nº 22

450°C 500°C 600°C 950°C

COAL Nº 33

500°C 600°C 950°C

COAL Nº 26

500°C 600°C 950°C

COAL Nº 28

500°C 600°C 950°C

COAL Nº 12

500° C 600°C 950° C

COAL Nº 16

500°C 600°C 950°C

is evident that the coal yielding the lesser quantity of volatile matter must have a greater swelling power in order to produce a coke button of equal volume to that produced from the coal yielding the greater quantity of volatile matter. This difference in the swelling of coals is not indicated by the volume of the button and must therefore be related to the volatile matter. Such being the case it may be expressed on the basis of unit volatile matter which is the percentage of swelling per unit of volatile matter evolved, and may be termed the "*Swelling Index*".

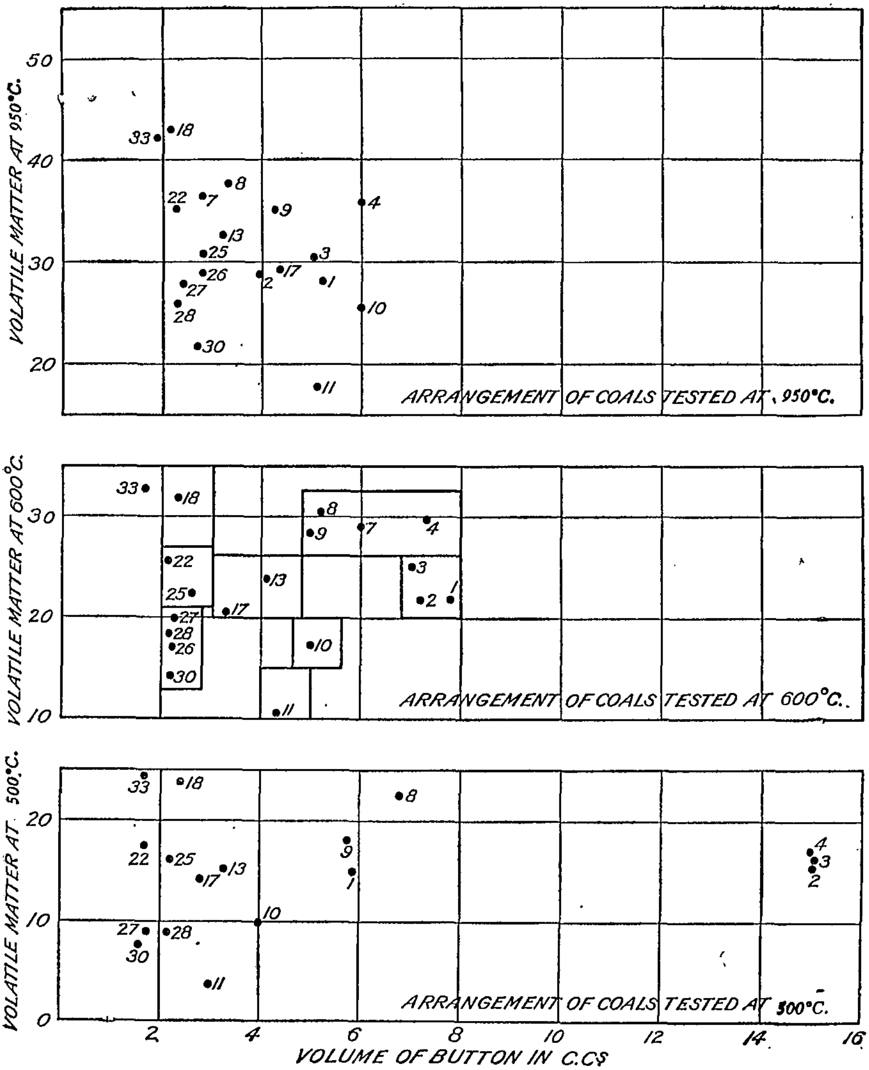


Chart I. Comparison of tests at 500° C, 600° C., and 950° C. with respect to the grouping of coals which produce cokes of similar characteristics

The calculation of this index is based on the assumption that the average volume of one gramme of finely ground coal is two cubic centimetres and thus the formula is as follows:

$$\text{Percentage Swelling} = \frac{(\text{Volume of button in c.c.} - 2)}{2\ \text{c.c.}} \times 100$$

$$\text{“Swelling Index”} = \frac{\text{Percentage swelling}}{\text{Volatile matter (dry basis)}} \times 100$$

The "swelling index" differs from the percentage swelling in that it takes into account the effect of the quantity of volatile matter on the swelling of the coal. Table I shows the swelling indices and the volatile matter contents (calculated to the dry basis) of a large number of coals and blends of coals upon which by-product coke data were available, and these are shown graphically in Chart II. Coals or blends of coals used for the production of cokes of similar quality were found to occur in groups so that it was possible to mark off these groups with arbitrary lines, forming a series of rectangles for which swelling index and volatile matter limits can be given. By reference to the chart it will be observed that, from Sections I to VII, the coals appear to follow the path of a curved band, but that at VII the curve is forked, one portion continuing up to VI and the other bending downwards to XII. The vertical line at zero swelling index is the division between coking and non-coking or agglomerating coals. The horizontal line at 20 per cent volatile matter separates those coals which produce cokes that are easily discharged from those more difficult to discharge, provided that the swelling index is over 400.

Chart III which is a duplicate of Chart II, without the coals plotted on it, was prepared to be used with the data obtained by means of the method described above, and it includes the general physical qualities of the coke corresponding to each section. It is an arrangement of coals on the basis of the physical properties of the cokes obtainable from them using the percentage of volatile matter and volume of the coke button obtained from the coal at a temperature of 600° C.

INTERPRETATION OF RESULTS

The portion of Section IV on Chart III between the swelling indices of 650 and 900 may be considered as the critical region for by-product cokes. From this region the physical properties of this type of coke diverge continuously in three directions through the sections in either decreasing or increasing magnitude. This is indicated on the chart, and the direction of the change in the properties is shown by the three curved arrows radiating from Section IV. This roughly groups coals into three major divisions:

1—Coals with a swelling index over 650.

2—Coals with a swelling index less than 650 and volatile matter above 27 per cent (on the dry basis).

3—Coals with a swelling index less than 650 and volatile matter below 27 per cent (on the dry basis).

TABLE I

Number	Coal Characteristics				Physical Characteristics of the Resultant Coke																	
	Volatile matter at 600° C. (dry basis)	Swelling	Swelling index*	Ash	Shatter Test		Abrasion Test		Size on Wharf			Apparent Sp. Gr.	Bulk density wt. c.f. 2- to 3-inch (dry)	Transverse shrinkage	Appearance of natural surface	Shape	Strength	Cross fracture	Longitudinal fracture	Cell structure	Sponge	Pebbly seam
					Index on 2-inch	Breeze through ½-inch	Index on 1½-inch	Dust through 1/16-inch	On 3-inch	Total on 2-inch	Breeze through ½-inch											
		%		%	%	%	%	%	%	%	%											
															Section I							
73	11·1	120	1,110	—	—	—	—	—	—	—	—	—	—	Nil	—	—	—	—	—	—	—	—
11	10·7	135	1,260	—	—	—	—	—	—	—	—	—	—	Nil	—	—	—	—	—	—	—	—
															Section II							
6	17·6	225	1,278	8·2	59·0	—	83·0	4·3	29·2	77·2	3·6	0·88	26·0	Fair, difficult to discharge	Steel grey, smooth	Blocky, square	Hard	Medium to large amount, fairly straight	Small to medium amount	Dense	Very little	None
10	16·6	150	961	10·6	56·0	—	84·7	3·4	38·3	78·6	5·4	0·95	26·0	“	“ “	“ “	“	“ “	“ “	“	“	“
															Section III							
1	21·3	290	1,360	6·6	60·0	—	89·5	3·5	33·2	86·0	2·2	0·93	25·7	Fairly good	Steel grey, smooth	Blocky, square	Hard	Medium amount, square	Medium amount	Dense	Very little	None
2	21·2	260	1,225	6·6	61·5	—	—	—	—	—	—	—	—	“	“ “	Square	“	“ “	“ “	“	“	“
3	24·1	250	1,037	7·5	62·6	—	—	—	46·8	83·7	1·0	0·92	—	Good	“ “	“	“	Small to medium, square	Small to medium amount	“	“	“
5	21·7	230	1,055	8·8	50·0	—	—	—	34·3	82·3	2·1	0·99	—	“	“ “	“	“	Medium amount, square	Medium amount	“	“	“
															Section IV							
4	27·9	270	970	6·6	56·2	2·5	—	—	—	—	—	1·0	—	Good	Steel grey, fairly smooth	Slightly triangular	Hard	Medium amount, steppy	Medium amount	Dense	Small to medium	None
35	25·5	220	860	7·0	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—
34	25·3	255	1,010	7·5	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—
7	28·0	200	715	8·7	50·0	—	85·0	4·9	46·1	85·9	2·3	1·06	28·2	Good	Steel grey, fairly smooth	Slightly triangular	Hard	Medium amount, steppy	Medium amount	Dense	Very little	None
															Section V							
68	29·6	118	400	2·6	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—
32	30·3	165	545	3·5	44·0	6·0	77·0	4·1	60·2	85·6	2·1	1·00	28·3	Good	Steel grey, and irregular	Triangular and fingery	Fragile	Medium to large amount, steppy	Medium to large amount	Medium	Medium amount, open	None
8	29·3	160	546	7·0	47·0	—	—	—	32·5	71·0	1·4	0·94	—	“	“ “	“ “	“	“ “	Large amount	Medium to large	Small to medium amount	“
															Section VI							
67	29·1	45	155	3·20	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—
43	37·5	70	209	3·35	36·5	6·0	76·7	3·9	50·0	80·0	2·0	0·83	23·0	Very good	Steel grey, irregular………	Triangular, fingery	Very fragile	Medium to large amount, steppy	Medium to large amount	Medium to large	Small to medium	None
65	30·0	55	183	3·70	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—
50	31·8	75	236	7·9	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—
18	28·9	30	103	13·3	26·0	—	57·5	7·0	40·0	70·0	5·0	0·85	25·3	Very good	Steel grey, irregular	Triangular, fingery	Very fragile	Medium to large amount, steppy	Large amount	Medium to large	Medium amount	None
															Section VII							
69	28·5	105	370	3·20	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—
46	26·5	155	585	3·50	61·0	3·0	94·0	1·5	41·7	85·6	1·9	0·85	25·0	Good	Steel grey, regular	Blocky, slightly triangular	Hard to fragile	Small to medium, slightly steppy	Medium amount	Small to medium	Small amount	None
44	27·3	165	604	3·69	72·0	3·0	84·2	4·3	45·8	84·5	3·0	0·83	23·8	“	“ “	“ “	“	Medium steppy	“ “	Medium	“	“
45	27·3	125	460	3·82	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—
49	27·5	135	490	3·87	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—
53	26·9	145	539	3·89	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—
47	26·5	145	550	4·08	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—
70	28·3	120	425	4·20	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—
54	26·0	120	460	4·24	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—
52	26·6	125	470	4·48	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—
56	26·7	125	468	4·55	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—
66	28·6	123	430	4·60	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—
51	25·7	110	428	4·63	72·0	3·0	93·6	2·7	46·4	77·6	4·0	0·83	23·5	Good	Steel grey, slightly irregular	Blocky, slightly triangular	Hard to fragile	Small to medium, slightly steppy	Medium to small amount	Medium to dense	Small amount	None
55	26·1	120	460	4·64	69·0	3·0	92·6	2·5	53·2	73·3	4·2	0·80	23·8	“	“ “	“ “	“	“ “	“ “	“ “	“	“
77	28·7	123	464	5·14	54·0	3·0	92·6	2·3	58·4	88·0	1·9	0·94	24·0	“	“ “	“ “	“	Medium amount, slightly steppy	Medium to large amount	“ “	“	“
61	25·3	150	590	5·26	71·0	3·5	96·1	2·0	53·0	81·4	4·5	0·85	25·5	“	“ “	“ “	“	Small amount, fairly square	Small to medium amount	Small to medium amount	“	“
75	25·3	115	455	5·60	70·0	3·5	95·2	2·5	69·2	89·7	1·7	0·91	26·0	“	Steel grey, regular	Blocky, fairly square	“	Small to medium, slightly steppy	“ “	“ “	“	“
42	27·0	110	408	6·02	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—
9	25·7	150	584	15·4	66·0	—	—	—	55·0	80·0	4·8	0·95	26·0	Good	Steel grey, regular	Blocky, slightly triangular	Hard to fragile	Small to medium, slightly steppy	Small to medium amount	Small to medium amount	Small amount	None
															Section VIII							
71	27·1	83	306	2·9	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—
72	27·8	80	290	3·4	48·9	5·2	80·4	3·0	16·7	42·0	2·9	0·85	24·3	Very good	Dull steel, grey, irregular	Triangular, fingery	Fragile	Large amount, steppy	Large amount	Medium to large	Medium amount, open	None
78	26·1	90	345	5·32	66·0	4·0	92·0	2·5	51·6	84·4	1·7	0·88	25·0	Good	Steel grey, irregular	Slightly triangular	Hard to fragile, friable	Small to medium, slightly steppy	Medium amount	Small to medium	Small amount	“
19	25·4	50	197	12·1	50·0	—	—	—	56·5	87·5	2·4	0·91	—	“	“ “	Slightly triangular, some fingery	Hard to fragile	Medium, steppy	“ “	Medium, irregular	“	“
21	26·3	25	95	12·5	50·0	—	68·4	5·1	56·0	85·6	2·2	0·91	24·5	“	“ “	“ “	“	“ “	“ “	“ “	“	“
36	27·9	90	322	12·6	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—
20	26·6	45	169	14·7	51·0	—	58·8	7·2	59·9	84·5	5·1	0·90	24·3	Good	Steel grey, irregular	Slightly triangular, some fingery	Hard to fragile	Small to medium, steppy	Medium amount	Medium, irregular	Small amount	None
															Section IX							
40	24·7	145	588	3·66	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—
64	23·6	110	465	3·81	69·0	5·0	93·6	2·3	49·5	78·5	4·6	0·81	23·5	Good	Steel grey, slightly irregular	Blocky, fairly square	Tough to hard	Medium, slightly steppy	Medium amount	Medium	Some	None
58	23·3	100	430	3·72	67·0	2·0	93·9	2·6	47·5	77·5	4·4	0·83	24·5	“	“ “	“ “	“	Small to medium, slightly steppy	“ “	“	“	“
59	23·6	110	465	4·08	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—
60	23·2	80	345	4·54	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—
63	24·7	105	425	4·63	72·0	3·0	93·6	2·7	46·4	77·6	4·0	0·83	23·5	Good	Steel grey, slightly irregular	Blocky, fairly square	Tough to hard	Small amount, slightly steppy	Small to medium	Medium to dense	Some	None
48	25·0	145	580	5·65	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—
76	24·9	153	624	5·76	71·5	2·5	94·3	1·7	57·1	90·7	1·6	0·92	26·5	Good	Steel grey, slightly irregular	Blocky, fairly square	Tough to hard	Small to medium	Small to medium	Medium to dense	Some	None
74	23·7	145	612	6·34	71·0	3·5	92·3	1·5	60·8	90·3	1·8	0·95	29·0	“	“ “	“ “	“	“ “	“ “	“ “	“	“
90	21·8	85	390	7·00	63·5	4·4	93·6	2·2	67·5	88·3	1·6	0·94	27·5	“	“ “	“ “	Tough	“ “	“ “	Dense	“	“
84	24·1	90	374	6·90	64·0	3·5	92·1	2·2	77·7	90·0	1·4	0·99	28·8	“	“ “	“ “	Tough to hard	Small, slightly steppy	“ “	“	“	“
82	24·7	50	202	7·40	64·5	4·5	83·3	3·4	66·7	86·3	1·7	0·93	24·0	“	“ “	Blocky, slightly irregular	Tough to hard, friable	Small to medium, slightly steppy	“ “	Medium to dense	“	“
87	23·8	90	378	7·40	59·5	5·0	91·4	2·2	73·5	90·2	1·4	0·94	29·0	“	Steel grey, fairly regular	“ “	Tough to hard	“ “	“ “	Dense	Small to medium	“
81	24·0	115	479	7·74	62·5	3·0	90·2	3·6	68·8	88·1	1·8	1·00	28·0	“	Steel grey, slightly irregular	Blocky, slightly triangular	“	“ “	“ “	“	“ “	“
83	24·9	103	414	8·20	66·5	4·0	84·9	2·9	76·9	91·1	1·5	0·99	29·8	“	Steel grey, fairly regular	“ “	“	“ “	Small amount	Medium to small	“ “	“
80	21·5	75	349	8·50	69·5	3·5	91·3	2·4	75·8	89·8	1·5	0·96	28·8	“	Steel grey, slightly irregular	“ “	“	“ “	Small to medium	“ “	Small	“
86	23·9	75	314	8·76	58·0	5·0	90·7	2·5	75·3	87·3	1·7	1·05	29·8	“	Steel grey, fairly regular	“ “	Tough to hard, slightly friable	“ “	“ “	Medium to irregular	Small to medium	“
89	21·9	65	297	8·44	68·5	5·0	89·6	2·6	75·7	89·9	1·5	1·00	28·8	“	“ “	“ “	Tough to hard	“ “	“ “	Medium to dense	Small	“
85	20·8	65	312	8·90	75·5	4·5	88·6	2·9	80·0	89·8	1·9	0·99	28·5	“	Steel grey, slightly irregular	Blocky, fairly square	“	Small amount, slightly steppy	Small amount	Dense	“	“
15	21·3	75	353	11·8	58·0	—	85·7	3·1	67·5	87·8	2·4	1·02	28·0	“	Steel grey, fairly regular	“ “	“	Medium, slightly steppy………	Small to medium amount	Medium to dense	“	“
16	22·2	70	315	12·4	68·0	—	—	—	66·4	91·0	2·9	0·94	—	“	Steel grey, slightly irregular	Blocky, slightly triangular	Tough to hard, slightly friable	Small to medium, slightly steppy	Small amount	“ “	“	“
12	20·2	115	570	12·8	70·0	—	90·0	2·3	60·9	89·1	2·5	0·98	27·5	Fair to good	Steel grey, fairly regular	“ “	Tough to hard	Medium amount, slightly steppy	Medium amount	“ “	Some	“
39	21·5	55	256	13·3	61·0	—	87·0	4·0	—	—	—	—	24·8	Good	Dull to steel grey, slightly irregular	Blocky, slightly irregular	Tough, slightly friable	“ “	Small to medium	“ “	“	“
14	21·3	90	423	13·5	65·0	—	88·9	3·4	67·9	89·8	2·0	1·08	29·3	“	Steel grey, slightly irregular	Blocky, fairly regular	Tough to hard	Small to medium, slightly steppy	“ “	Fairly dense	“	“
41	23·3	80	343	13·9	59·0	—	90·5	2·9	65·4	88·4	2·9	0·94	26·0	“	“ “	Blocky, slightly triangular	“	“ “	Small amount	Medium to dense	“	“
13	22·8	105	461	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—
															Section X							
23	22·6	25	110	11·1	71·0	—	—	—	71·9	91·3	4·0	0·89	—	Good	Dull to steel grey, slightly granular	Blocky, fairly square	Tough, friable	Small amount, irregular	Small amount	Dense, patchy	Slightly granular end	Very slight
24	21·9	35	114	12·6	64·0	—	86·0	4·6	—	—	—	—	—	“	Dull to steel grey, slightly irregular	Blocky, slightly triangular	“	Small to medium, slightly steppy	“ “	“ “	“ “	Slightly
25	20·9	30	144	17·95	71·0	—	—	—	—	—	—	1·00	—	Fair to good	“ “	“ “	Tough, slightly friable	“ “	“ “	Dense	Some	None
29	15·3	0	0	12·3	78·0	—	—	—	—	—	—	1·08	—	—	—	—	—	—	—	—	—	—
22	23·7	10	42	12·65	68·0	7·0	76·7	7·5	77·6	91·3	3·1	0·97	25·8	Fair to good	Irregular, granular	Blocky, irregular	Tough and friable	Small amount, steppy	Small amount	Coked portion medium	Granular end	Pebbly
27	17·3	10	58·0	13·25	68·0	10·0	76·3	9·4	79·4	90·5	4·0	1·12	28·5	Fair	“ “	“ “	“	“ “	“ “	Coked portion medium to dense	“ “	“
															Section XI							
28	15·7	20	127	11·22	70·0	5·0	89·1	2·8	70·0	90·0	2·5	1·05	30·8	Fair	Dull to steel grey, slightly irregular	Blocky, square	Tough	Small amount, very slightly steppy	Small amount	Irregular	Very little	None
26	18·9	20	106	12·09	69·5	4·5	85·7	3·8	76·6	91·0	1·4	1·07	29·8	“	“ “	“ “	“	“ “	“ “	“	“	“
17	19·8	65	329	13·0	72·5	4·0	90·4	2·4	76·2	89·4	1·7	1·10	31·0	“	Steel grey, slightly irregular	Blocky, slightly irregular	“	Small amount, slightly steppy	“ “	Medium to dense	Some	“
															Section XII							
30	14·2	10	71	11·5	88·1	—	—	—	—	—	—	1·04	—	Fair to poor	Dull grey, irregular	Blocky, irregular	Tough, friable	Very small amount, irregular	Very little	Irregular	Very little	None
															Section XIII							
37	29·2	−10	−34	13·0	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—
33	28·2	−30	−106	13·7	50·0	—	—	—	30·5	55·6	37·4	0·85	—	Good	—	—	Very fragile and friable	—	—	None	All granular	All pebbly
38	23·4	−15	− 64	12·7	69·0	—	87·8	3·3	61·0	84·6	3·5	0·91	26·5	“	Dull grey, granular	Blocky, irregular	“	Small to medium amount, steppy	Small to medium amount	Fairly dense, pebbly patches	Some granular ends	Pebbly
31	17·7	−20	−113	13·7	71·0	—	64·9	12·6	66·9	86·2	5·0	1·07	27·7	“	“ “	“ “	Tough and friable	Medium amount, irregular	“ “	Dense where coked	Granular ends	“

$$* \text{ Swelling index} = \frac{\%\text{ swelling} \times 100}{\text{Volatile Matter}}$$

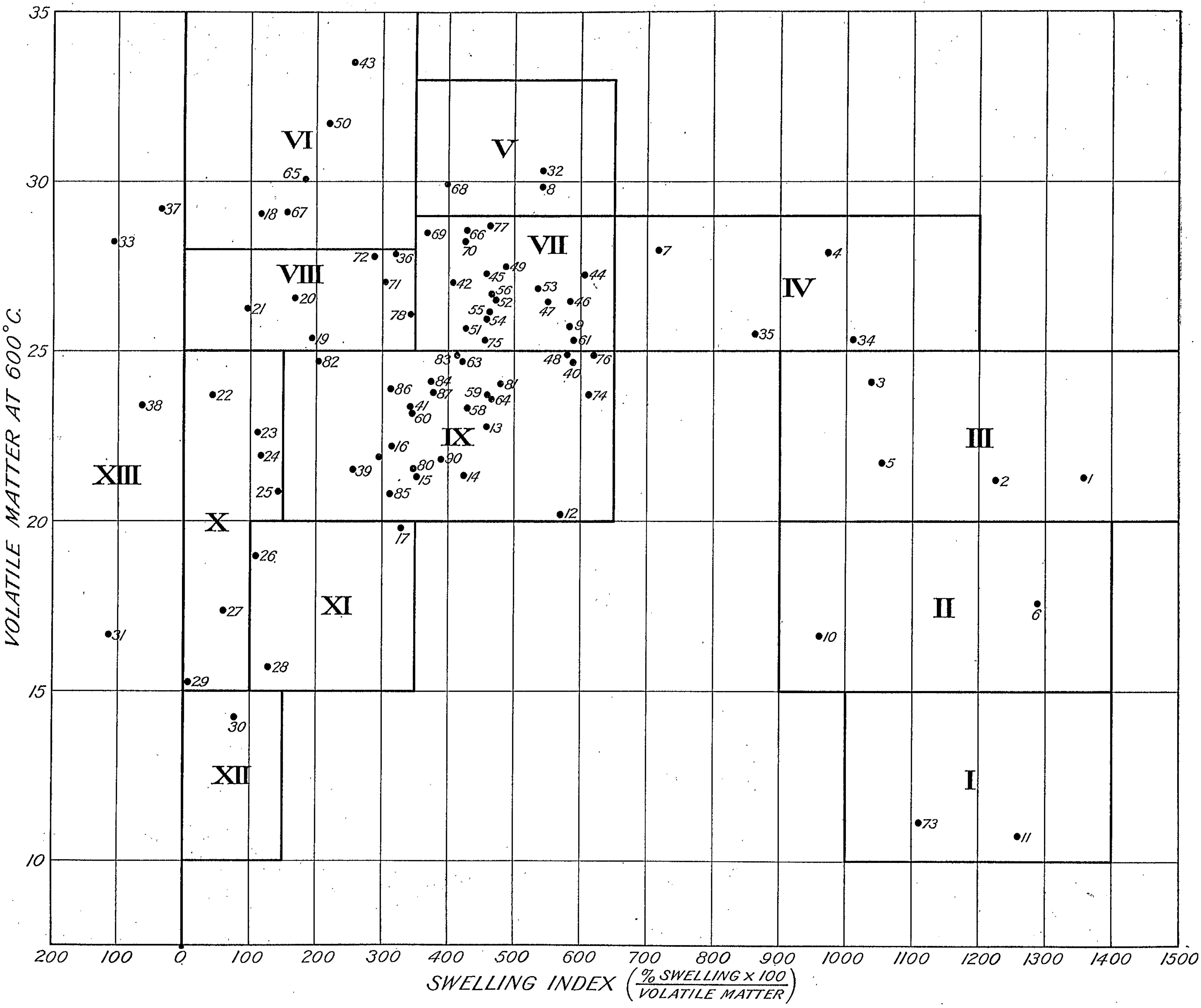
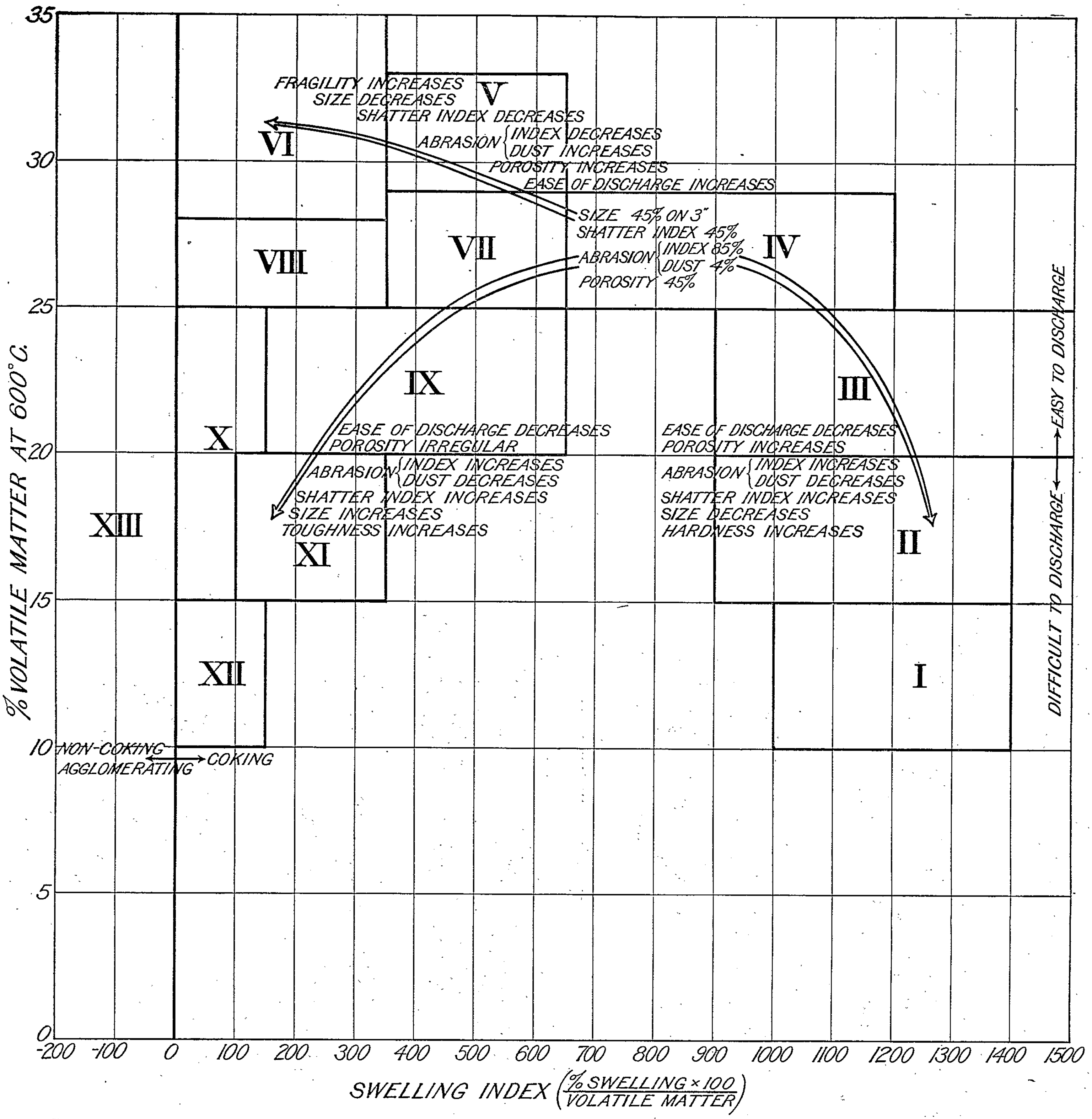


Chart II. Coals shown in Table I, plotted according to their volatile matter and swelling index at 600° C.



SECTION	ASH%	SIZE ON WHARF		SHATTER		ABRASION		DENSITY		TRANSVERSE	APPEARANCE OF NATURAL SURFACE	SHAPE	STRENGTH	CROSS FRACTURE	LONGITUDINAL FRACTURE	CELL STRUCTURE	SPONGE	PEBBLY SEAM
		% ON 3"	BREEZE	INDEX	BREEZE	INDEX	DUST	APP. S.G.	LB. C.F.	SHRINKAGE								
I		POCAHONTAS TYPE BLENDING COALS								NIL			HARD				VERY LITTLE	NONE
II		25-40	2·5-1·0	65-55	3·0-2·0	85-95	3·5 2·0	·85-·95	24-26	FAIR*	STEEL GREY AND SMOOTH	SQUARE	HARD	MEDIUM TO LARGE AMOUNT STRAIGHT	SMALL TO MEDIUM AMOUNT	DENSE	VERY LITTLE	NONE
III		30-45	2·5-1·0	50-65	3·0-2·0	85-95	3·5-2·0	·90-1·0	25-28	FAIR TO GOOD	STEEL GREY AND SMOOTH	SQUARE	HARD	MEDIUM AMOUNT, SQUARE	SMALL TO MEDIUM AMOUNT	DENSE	VERY LITTLE	NONE
IV		40-50	3·0-2·0	45-55	3·0-2·0	80-90	5·0-3·0	1·0-1·1	26-29	GOOD	STEEL GREY, FAIRLY SMOOTH	SLIGHTLY TRIANGULAR	HARD	MEDIUM AMOUNT, STEPPY	MEDIUM AMOUNT	DENSE	VERY LITTLE	NONE
V		30-50	4·0-2·0	40-50	6·0 4·0	75-85	5·0-3·0	1·0-0·9	26-29	GOOD	STEEL GREY, IRREGULAR	TRIANGULAR FINGERY	FRAGILE	MEDIUM TO LARGE AMOUNT STEPPY	MEDIUM TO LARGE AMOUNT	MEDIUM	SMALL TO MEDIUM AMOUNT	NONE
VI		30-50	2·0-5·0	25-40	6·0-4·0	55-80	7·0-3·0	·85-0·9	25	VERY GOOD	STEEL GREY, IRREGULAR	TRIANGULAR VERY FINGERY	VERY FRAGILE	MEDIUM TO LARGE AMOUNT STEPPY	LARGE AMOUNT	MEDIUM TO LARGE	MEDIUM AMOUNT	NONE
VII	0·5	40-50	4·0	55-70	3·0	80-95	2·5-5·0	·83	23·5	GOOD	STEEL GREY, IRREGULAR	BLOCKY; SLIGHTLY TRIANGULAR	HARD TO FRAGILE	SMALL TO MEDIUM, SLIGHTLY STEPPY	MEDIUM TO SMALL AMOUNT	MEDIUM TO SMALL	SMALL AMOUNT	NONE
	5-10	50-70	4·0	55-70	3·0	80-95	2·5-5·0	·83-·95	23·5-26	"	"	"	"	"	"	"	"	"
	10-15	70-50	5·0	55-70	3·0	80-95	2·5-5·0	·95	26	"	"	"	"	"	"	"	"	"
VIII		50-70	2·0-5·0	45-65	4·0-5·0	60-80	2·5-7·0	·85-·95	24-26	GOOD	STEEL GREY, IRREGULAR	SLIGHTLY TRIANGULAR AND FINGERY	HARD TO FRAGILE; FRIABLE	MEDIUM AMOUNT; SLIGHTLY STEPPY	MEDIUM AMOUNT	MEDIUM; IRREGULAR	SMALL AMOUNT	NONE
IX	0-5	45-55	4·0	70	3·0	93	2·5	·80-·86	22-25	GOOD	STEEL GREY, SLIGHTLY IRREGULAR	BLOCKY; SLIGHTLY TRIANGULAR	TOUGH TO HARD	SMALL TO MEDIUM; SLIGHTLY STEPPY	SMALL TO MEDIUM AMOUNT	SMALL TO MEDIUM	SMALL AMOUNT	NONE
	5-10	55-80	2·0	70-60	3·0-5·0	90	2·5	·86-1·1	25-31·5		"	"	"	"	"	"	"	"
	10-15	80-55	2·0-4·0	70-60	3·0-5·0	90	2·5	1·1-·92	31·5-25		"	"	"	"	"	"	"	"
X		70-80	3·0-5·0	65-80	10·0-5·0	70-80	10·0-7·0	·95-1·1	26-30	FAIR TO GOOD	DULL; GRANULAR	BLOCKY; IRREGULAR	TOUGH; FRIABLE	SMALL AMOUNT; SLIGHTLY STEPPY	SMALL AMOUNT	SMALL TO MEDIUM	GRANULAR ENDS	PEBBLY
XI		70-80	3·0	70-80	5·0	85-90	3·0-4·0	1·0-1·1	30-31	FAIR	DULL TO STEEL GREY; IRREGULAR	BLOCKY; SQUARE	TOUGH	SMALL AMOUNT; VERY SLIGHTLY STEPPY	SMALL AMOUNT	IRREGULAR	VERY LITTLE	NONE
XII		80	3·0	80	5·0	85	4·0	1·0-1·1	30-31	FAIR TO POOR	DULL GREY, IRREGULAR	BLOCKY; IRREGULAR	TOUGH; FRIABLE	VERY SMALL AMOUNT; IRREGULAR	VERY LITTLE	IRREGULAR	VERY LITTLE	NONE
XIII		25-70	4·0-3·0	30-70	20·0-5·0	50-80	15·0-5·0	·85-1·1	23·5-28	GOOD	DULL GREY, GRANULAR	BLOCKY; IRREGULAR	FRIABLE	SMALL TO MED. AMOUNT; IRREGULAR	SMALL TO MEDIUM AMOUNT	VERY LITTLE TO NONE	GRANULAR ENDS	PEBBLY TO NON-COKING

*DIFFICULT TO DISCHARGE

Chart III. Classification of by-product cokes according to their physical properties employing volatile matter and swelling index of the coal at 600° C.

These changes in the physical properties which have been shown diagrammatically on Chart III are tabulated below in Table II.

TABLE II

Properties	Division 1 — Swelling index over 650	Division 2 — Swelling index less than 650 and V.M. over 27 per cent	Division 3 — Swelling index less than 650 and V.M. less than 27 per cent
Ease of discharge	Decreases	Increases	Decreases
Porosity	Increases	Increases	Irregular
Abrasion index	Increases	Decreases	Increases
Dust due to abrasion	Decreases	Increases	Decreases
Shatter index	Increases	Decreases	Increases
Structural Strength			
Size	Decreases	Decreases	Increases
Hardness	Increases		
Fragility		Increases	
Toughness			Increases

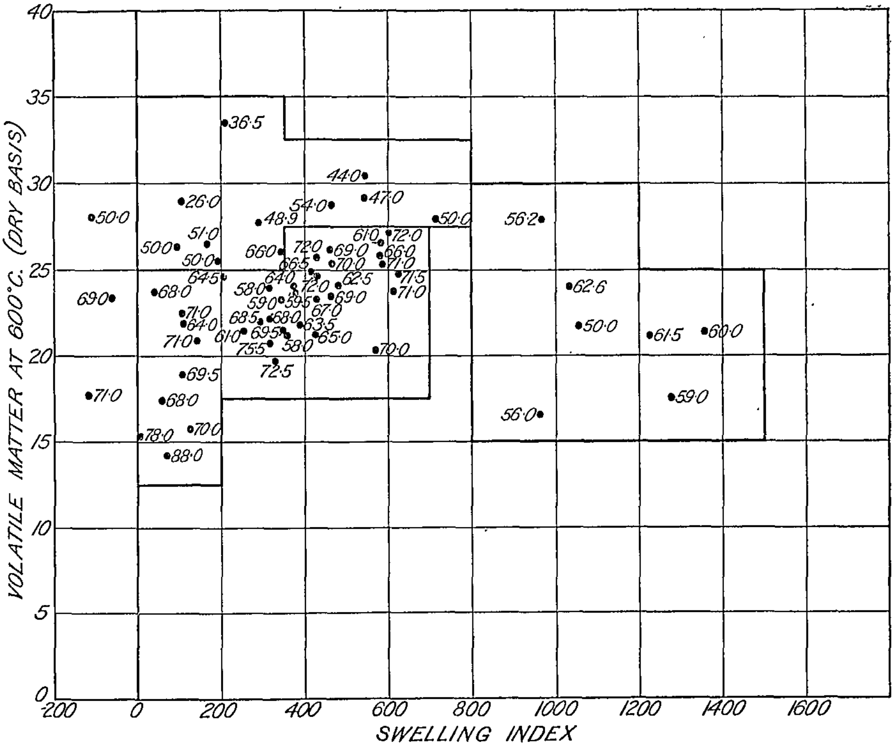


Chart IV. Shatter indices of the cokes showing the tri-radiating arrangement.

In Chart IV, the coals have been plotted as in Chart II, and the shatter index of each coke shown. The arrangement indicates the breaking up into the three major divisions, with the increasing and decreasing tendencies of this physical property through these divisions in the directions of the radiating curved arrows. Chart V is similar to Chart IV, with the exception that the percentage of the 3-inch size as discharged is indicated for each coke. Here, also, the generalization made above holds for this physical property of cokes, although in certain groups the ash also plays an important part in determining the size. This will be referred to later under the discussion of the individual groups or sections.

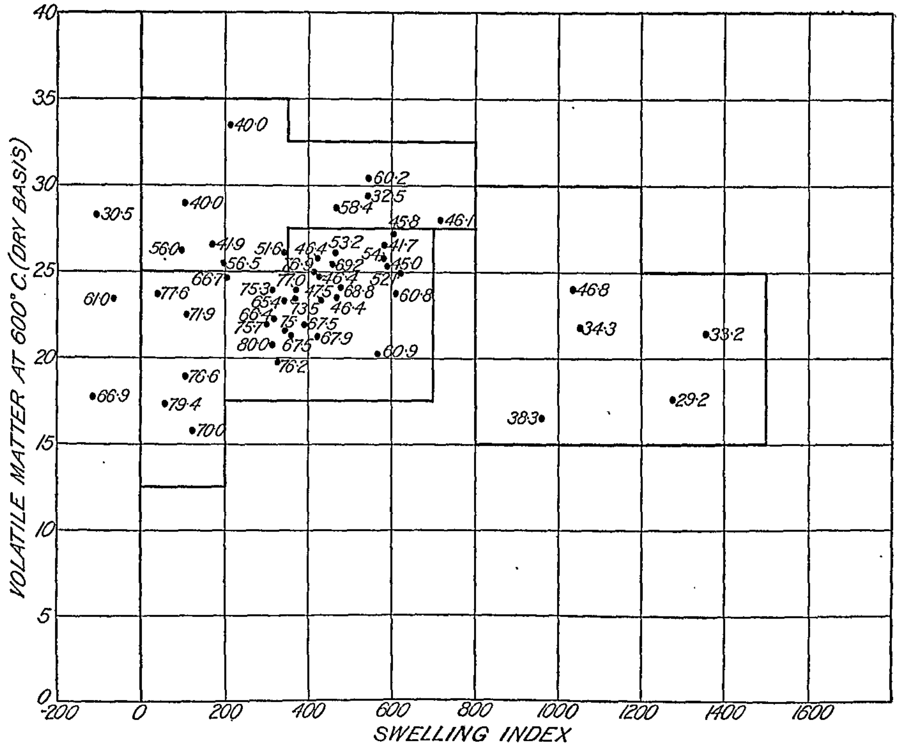


Chart V. Size on wharf (percentage on 3-inch) of the cokes showing the tri-radiating arrangement.

DISCUSSION OF GROUPS

By plotting coals on the basis of their swelling index and percentage of volatile matter at 600° C. as shown in Chart II, it will be noted that coals which produce cokes having similar physical properties tend to arrange themselves in groups. These groups or sections have been indicated on the chart by means of numerals and, although the lines are somewhat arbitrarily drawn, they do indicate definite changes in the characteristics of the various resultant cokes. The sections, together with the assigned swelling indices and volatile matter limits at 600° C., are shown in Table III and are described below.

TABLE III

Section	Swelling index limits	Volatile matter limits
I	1000—1400	10—15
II	900—1400	15—20
III	900—1500	20—25
IV	650—1200	25—29
V	650—350	29—33
VI	350—0	28—35
VII	650—350	25—29
VIII	350—0	28—25
IX	650—150	25—20
X	150—0	25—15
XI	350—100	20—15
XII	150—0	15—10
XIII	0—200	5—35

Section I.—This group contains all those high rank blending coals of the Pocahontas type, which are characterized by their marked swelling tendencies and low volatile matter contents. When blended with high volatile matter coking coals they improve the structure of the resultant coke, in some cases from an unsaleable to a saleable product. The coals in this group cannot be used alone in a by-product oven as they are non-shrinking and it would be impossible to discharge them.

Section II.—In this group are those high rank coals which may also be used for blending, but are not so satisfactory as those in Section I. These coals are higher in volatile matter content than those in Section I, and do not possess the same swelling power. They cannot be used alone for the production of coke owing to the difficulty of discharging them from the oven, but they can be and are used as substitutes for Pocahontas type coals for blending with the higher volatile gas coals to improve the structure of the coke.

Section III.—The coals falling into this group are those considered to be the "natural by-product coking coals". They are characterized by producing a dense and hard medium-sized square coke which is entirely satisfactory for either domestic or metallurgical use. Unfortunately there are very few coals produced which are of this particular type, and therefore it is necessary to blend several coals to obtain a mixture which will produce a coke similar to that made from coals in this group.

Section IV.—The coals occurring in this section, especially between the swelling indices of 650 and 900, are characterized by a high volatile matter content and strongly coking tendencies. They produce cokes of a characteristic structure, which are generally described as "true gas cokes". Coals of this type are used in some by-product plants for coke production, but the coke is not entirely satisfactory, especially when used for domestic purposes. It possesses a rather high density and low reactivity, making it difficult to control in the average domestic heating installation. From a production standpoint it also has certain disadvantages owing to its characteristic structure. The coke is usually triangular in shape with spongy ends, which tends to produce a large quantity of breeze and small sizes during preparation for the market. Coals in this section have a maximum swelling as well as a maximum contraction and this combination of properties places them in the centre of any scheme for arranging coals on the basis of the physical properties of their resultant cokes. The physical properties of cokes in other groups are a result of change in one or more of the properties of this group, hence this group may be considered as a hub from which the physical properties of other groups radiate in three directions in either decreasing or increasing magnitude.

Section V.—This group contains those high volatile matter gas coals which do not possess the strongly coking properties that characterize the coals in Section IV. Coke made from this type of coal is similar in shape to the "true gas cokes" but it is very fingery and highly fissured. As a result of these properties it does not handle well and produces a very large quantity of breeze and small sizes during preparation. Coals of this type are not suitable by themselves for the manufacture of by-product coke, but they can be utilized when properly blended with other types of coals.

Section VI.—Coals in this group are very similar to those in Section V, being low rank bituminous coals with high volatile matter contents. These coals are unsuited for use, by themselves, for the production of by-product coke, but may be utilized to a certain degree if blended with the proper type of coals. The coke from this type of coal is more fragile and more highly fissured than coke made from coals occurring in Section V, and likewise is wholly unsuited for either domestic or metallurgical use.

Section VII.—The coals in this group result in the production of medium to good quality cokes, but they are not entirely suitable because of their friability which makes for excessive degradation during handling. The ash plays a certain part in controlling the physical qualities of cokes in this group, as indicated in the table on Chart III.

Section VIII.—Coals in this group produce cokes which are superior to those in Section VI, but inferior to those in Section VII. They are midway between these two groups with respect to physical properties and these are such as to render them not entirely suitable for either domestic or metallurgical use. Coals occurring in this section can only be used in by-product ovens, when properly blended with suitable high rank coking coals, to increase their swelling tendencies, and reduce the volatile matter content.

Section IX.—In this section, coals of practically the same rank as those in Section III occur, but due to the difference in the swelling properties, cokes with entirely different qualities are produced. These cokes, generally speaking, are fairly good domestic and metallurgical fuels, being tough and strong, and having a porosity which gives them a reactivity quite suitable for most purposes. The distinctive difference between these cokes and those in Section III is their cell structure. In the case of the cokes from coals in Section III, the cell structure is uniform and the cells are of small size with thin walls, whereas those in this group have larger cells with thicker walls. This imparts to these cokes a property of toughness opposed to brittleness, which characterizes those in Section III. Some of the physical qualities of the cokes in Section IX are greatly influenced by the quantity of ash present. Chart VI, on which the percentage of the 3-inch size as discharged, the apparent specific gravity, and bulk density, for the cokes in this group are plotted against the percentage of ash in the coal, illustrates this point. The three properties mentioned have increasing values up to 10 per cent of ash in the coal, and from 10 per cent of ash up to probably 15 per cent, the values decrease. It is evident, then, that 10 per cent of ash in the coal or blend will give

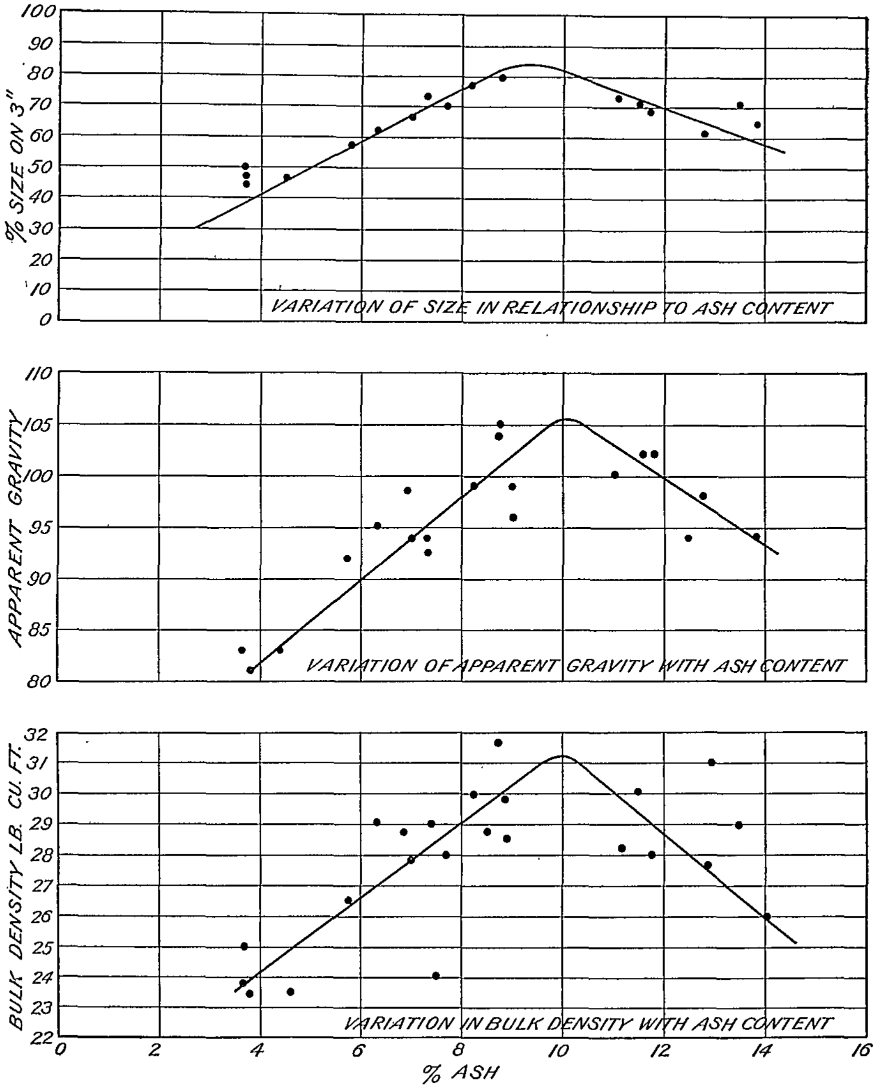


Chart VI. Curves showing varying physical qualities of cokes in Section IX with variation in the ash percentage.

the maximum value for the aforementioned properties. Other properties, such as resistance to shatter and abrasion, are more dependent on the chemical constitution of the coal alone, and, therefore, follow the general trend of the classification as indicated in the general discussion. It should be noted that those coals occurring closest to the swelling index of 650 in this group produce cokes most suitable for either domestic or metallurgical purposes, even more so than those found in Section III.

Section X.—This relatively small and irregularly shaped section contains coals which produce cokes that have a tendency to be pebbly, owing to the poor coking properties of the coals.

Section XI.—Coals in this group are usually of the same rank as those in Section II, but they differ materially in their swelling properties. As a result of the inferior swelling power of these coals, they are not suitable for blending with higher volatile matter coals to improve coke structure as are those in Section II. While not entirely suitable for the manufacture of by-product coke, it is possible to discharge coke made from these coals without much difficulty.

Section XII.—These coals are unsuitable for by-product use either alone or in blends where they are expected to improve appreciably the structure of the coke produced from high volatile matter coals. In other words, they cannot be used to replace Pocahontas type coals in by-product practice.

Section XIII.—The coals or blends in this rather wide region either produce very pebbly cokes or are non-coking. These coals may be neglected entirely when considering their use in the by-product industry, except when special treatment is resorted to.

CONCLUSIONS

It is believed that by the aid of Chart III and the laboratory method for obtaining the necessary data for use with the chart, it is possible to evaluate fairly accurately the physical properties of coke obtainable from any coal or blend in a standard by-product coke oven. The proposed method is serviceable when it is necessary to make a general survey of unfamiliar coals, or when a choice is to be made, for a particular purpose, from a large number of coals. Large-scale tests are expensive and sometimes impracticable, and by this procedure a coal or blend most suited for the particular purpose may be chosen, and a large-scale test used to confirm the results, if necessary. In this way expensive tests may be avoided, and only confirmatory evidence is required. The authors have had the opportunity of doing this, and predictions so made have invariably been corroborated by the results of later full-scale tests.

APPENDIX

The following brief résumé of the literature pertaining to the work on the coking properties of coals, although incomplete, serves to indicate the attempt on the part of various investigators to establish some laboratory test which will aid in evaluating coals for their use in the manufacture of coke. As the property of coking is dependent on the chemical nature of the coal, and as any variation in this is evidenced by physical phenomena during coking, a study of the coking properties of coals naturally falls into these two divisions.

CHEMICAL

Fischer's[1] researches on the exhaustive extraction of bituminous coal with benzene under pressure, and further resolution of the extract with petroleum ether, show that the "oily bitumen" (that part of the benzene extract soluble in petroleum ether) is responsible for the caking or agglutinating properties of a coal; whereas the ether insoluble brown powder, i.e. "solid bitumen", is essentially responsible for the swelling properties, and if its temperature of decomposition coincides with the temperature of fusion of the "oily bitumen" of the coal, it will yield a highly swollen coke.

It is obvious that the measurement of only one of the above-mentioned properties cannot give any reliable measure of the behaviour of coal during coking, and hence we have the failure of the caking index as a criterion of the coking properties of coals. This value only gives a reliable criterion when the second property, i.e. swelling, is zero or sufficiently small that it may be neglected.

Barash[2], in studying the extract due to the solvent action of pyridine, has come to the following conclusions:

(a) The fusible portion of bituminous coal represented by the "gamma fraction"[3], i.e. resins and hydrocarbons soluble in pyridine and in chloroform, is the cementing material to which is mainly attributable the coking and agglutinating properties of coal.

(b) The portion of coal corresponding to the "beta fraction"[3] (dispersed ulmins soluble in pyridine, but insoluble in chloroform) contributes to an appreciable extent towards these properties, being also fusible under carbonizing conditions.

(c) The manifestation of these properties by the "beta and gamma" fractions is influenced to a very marked degree by the type or condition of the infusible matter corresponding to the "alpha" fraction (undispersed ulmins, spore exines, cuticles and resistant woody structure insoluble in pyridine) which predominates in the coal mass with which the fusible compounds are associated.

(d) Treatment of coal, such as weathering or the action of a large variety of chemical reagents, which reduces or destroys the coking and agglutinating power, effects this through some modification produced in the infusible matter. Such treatment only affects the property of agglutination to a comparatively small degree, if at all.

(e) The difference between highly coking and non-coking (or poorly coking) coals depends essentially on the physical condition of the non-fusible compounds (alpha fraction) and is only dependent to a limited extent on the amount of agglutinant present.

Illingworth[4] contends that it is necessary for a coal to contain 5½ per cent of gamma compounds before it will yield a good quality coke, while Foxwell[5] takes exception to this and puts forward the argument that it is not so much the quantity of gamma compounds, but their distribution in the coal, their thermal stability and mode of extrusion from the individual pieces which determine the coking qualities of a coal. Foxwell's theory of coking is as follows:

"The first stage of coking is . . . the formation of these globules either by extrusion of molten matter from the interior of the piece of coal, or occasionally by fusion when very small particles are heated. A good deal of gas is evolved during this stage. The particles then cohere and form a net work. The surface tension effects become lessened and finally the process of cell formation which is nearly complete in the interior of the globule is carried on at the surfaces." This is practically the same as the theory advanced by Mott.

Damm[6] has shown that the results of benzene and pyridine extractions do not agree, and that neither gives complete extraction. In view of this, he suggested a different method of tackling the problem, i.e. by studying the coal as a whole. Others have also taken this view, and this brings us to the physical phenomena during coking.

PHYSICAL

I.—*Plasticity:* When coking coals are slowly and progressively heated in a suitable apparatus (e.g. by the Layng and Hawthorne plasticity apparatus) at a certain more or less defined temperature which is in the neighbourhood of 400° C., there is a softening of the coal particles and cohesion to form a plastic mass, which offers an increasing resistance to a constant stream of inert gas. This back pressure, as indicated by a water manometer, is plotted against the temperature and gives the so-called plasticity curve.

The coal, according to this theory, remains plastic up to about 500° C. and then becomes solidified into a semi-coke. The softening and hardening temperatures vary with different coals, and hence produce different plasticity curves, as shown in Chart VIII (curves for gas coal, by-product coal, and Pocahontas).

Foxwell[7] who was the originator of this method of investigation of the coking properties of coals, concludes that plasticity is necessarily antecedent to coking. The plastic stage is not a period during which the coal is particularly active chemically, although between the temperatures of 400° and 500° C. the almost complete destruction of beta and gamma compounds occur.

Preheating a coal reduces its plasticity, and the reduction is increased as time and temperature of preheating are increased. The general effect of weathering is to reduce the height of the plastic curve. The curves in Chart VII (artificially weathered coal) show this effect clearly.

Layng and his co-workers[8] have done a great deal of work on the plasticity of American coals, their results being much the same as Foxwell's; i.e. that, although they are able to study the coke formation with the aid of plasticity curves, they are not able to predict coke quality to any degree of accuracy. They have modified the curves and tried to obtain caking indices from plasticity data, but as stated before, caking index alone cannot serve as an index of coke quality. Chart VIII shows typical plasticity curves according to the method proposed by Layng.

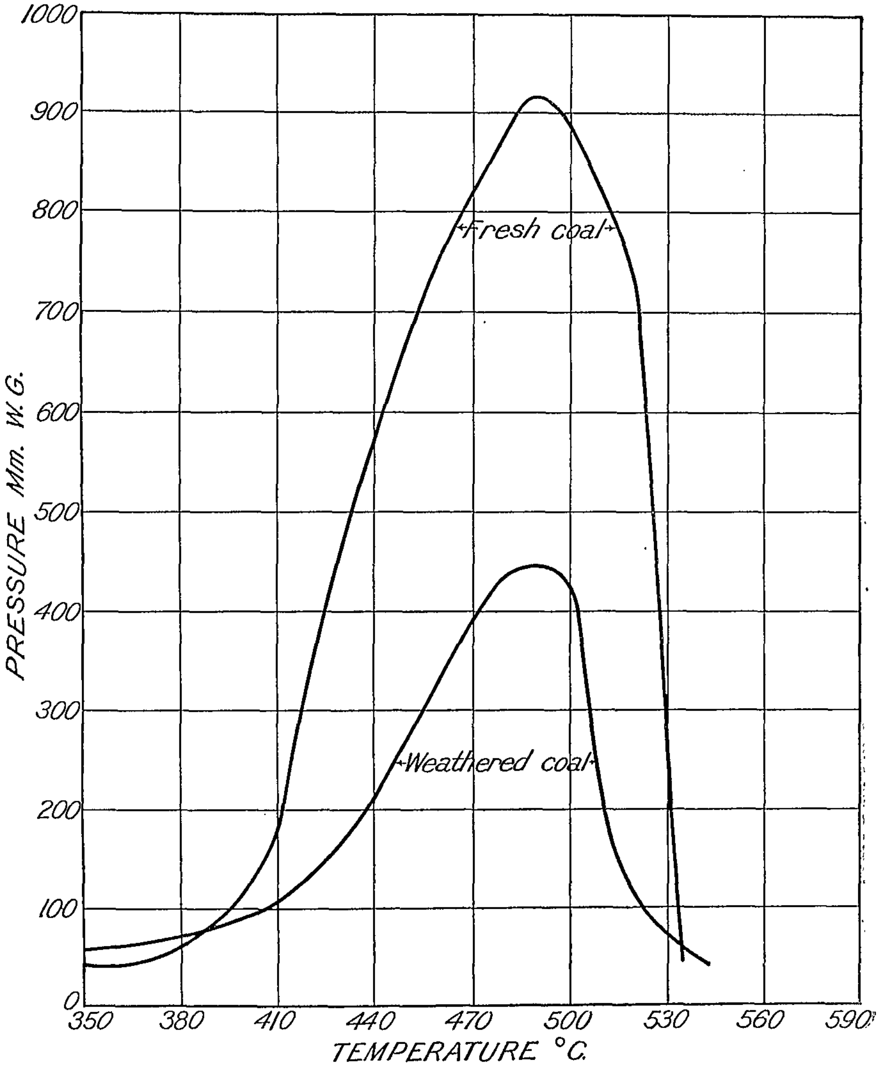


Chart VII. Plasticity curves showing effects of weathering on plasticity.

Audibert[9] draws the following conclusions from his work on the transient fusion of coals: Under the influence of heat coal begins to "melt". If, after passing the softening point, the temperature is progressively increased the viscosity of the pasty mass diminishes to a minimum and then increases until finally the pasty mass resumes the solid state. The chance that gas bubbles will be formed within a plastic mass of coal particles is an increasing function of the maximum fluidity. In a general way, the greater the fluidity obtained by the constituent matter during fusion, the more rapid the heating, the finer the original particles, and the more

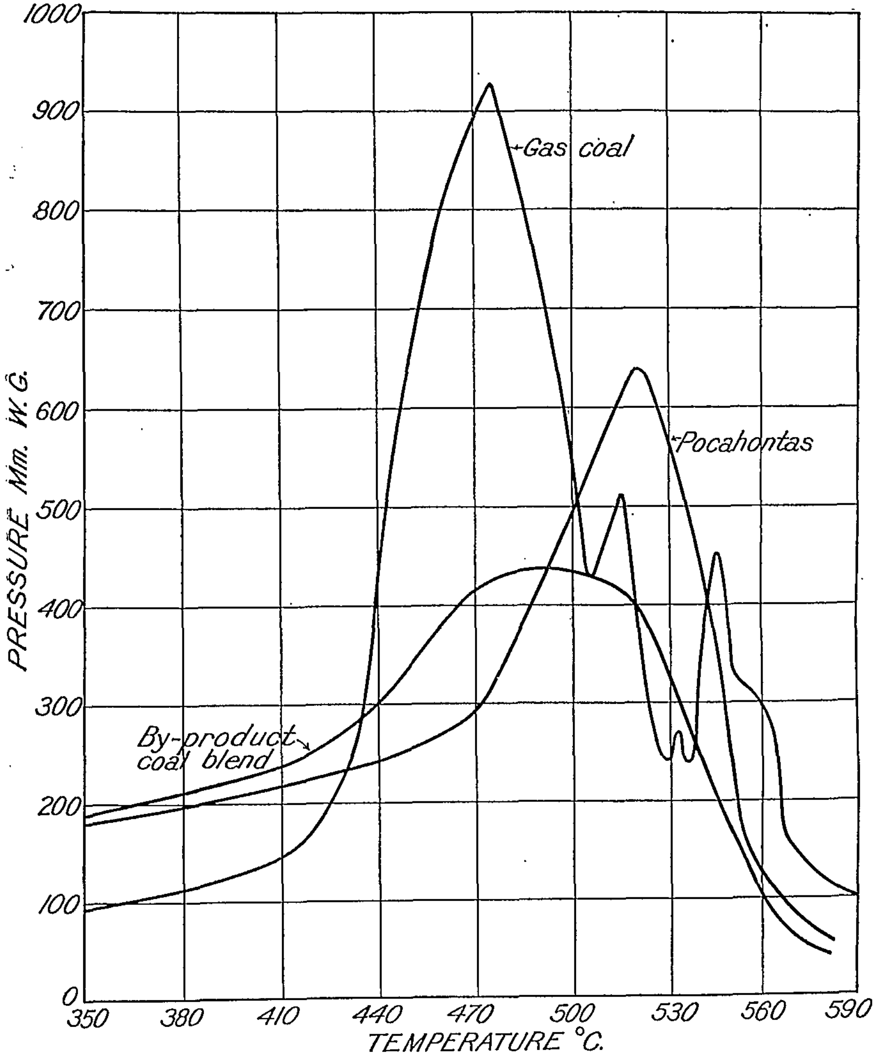


Chart VIII. Plasticity curves of typical coals.

nearly the density of the original mass approaches the density of the coal, the stronger will be the agglomerated mass of particles. Hence, again, we have a description of experiments to obtain the characteristics of coke in a general way, but nothing to classify coals or blends of coals.

A great deal of work has been done, and many interesting points brought to light but, as Foxwell[7] concludes, "there is . . . no numerical agreement between the area of the plastic curve and the quality of the coke, but . . . this is not to be expected, for the plastic curve gives a picture of the manner in which coking proceeds rather than a measure of the coking power".

II.—*Swelling:* The plastic state of coal is accompanied by varying degrees of swelling and by the evolution of varying amounts of volatile matter. This swelling resolves itself in the coke oven into pressure. Mott and Shimmura[10] conclude that swelling power is directly related to the coking power of a coal. Damm[6] comes to the conclusion that, to obtain a good coke, a coking coal must possess adequate caking properties as well as a certain swelling pressure. Coals with a low "coking index" give a coke which is firmer and harder the greater the swelling pressure. Slater[11] states that "though the swelling of the coke in the laboratory test" (Gray King apparatus with a maximum temperature of 600° C.) "cannot be accepted alone as an absolute criterion of the coking quality of the coal in practice, when coals from different coal fields are under examination, their general character can be used to differentiate them".

According to Mott and Shimmura[10] swelling has no relation to the amount of gas evolved over the plastic range, but is primarily dependent on the amount of tar produced, and the ease with which the rest of the coal is wetted by the tar. Barash[12] states that the percentage of swelling of the coke buttons "bears a distinct relationship to, and gives an indication of, the agglutinating value of the coal". It has been found, however, that the swelling power of coal is reduced by preheating, weathering, or treatment with chemical reagents.

III.—*Volatile Matter:* "It has been found (Rose, Fuel in Science and Practice, 1926, 5, 562) that there is some relation between the total volatile matter content and the nature of the coke obtained from a coking coal. High volatile coking coals give highly fissured cokes, while low volatile coking coals give blocky cokes with comparatively few fissures. It is during the period subsequent to the plastic range that the fissuring of the coke takes place, and the degree of fissuring should be related to the percentage of volatile matter evolved during this period"[13].

Damm[6] asserts that "whilst initial distillation and distillation during the fusion period determine the coking properties of a coal and the mechanism of carbonization, the extent of final distillation directly determines the quality of the coke produced . . . the greater the loss in the semi-coke produced by solidification of the plastic mass of coal, the greater will be the contraction of the coal, and hence the formation of fissures and cracks." The less the distillation after solidification "the less will be the contraction and fissuring of the coke. But at the same time, the risk increases of the coke contracting too little, adhering to the walls and becoming difficult to remove."

However, the authors do not believe that the above is generally true. The volatile matter in their experiments was determined at 500° C. (end of plasticity) for a series of coals, and the residual volatile calculated by subtracting the volatile at 500° C. from that of 950° C. The residual volatile does not agree very well with the shatter index of the cokes produced in the two-ton by-product oven of the Fuel Research Laboratories, as shown in Table IV.

TABLE IV

Coal No.	Residual volatile at 500° C. (unit coal basis)	Shatter index, per cent on 2-inch
5	13·2	41·0
6	13·9	59·0
1	14·0	57·0
7	14·7	45·0
12	14·8	70·0
10	15·4	54·0
14	15·5	65·0
16	16·6	68·0
28	16·8	77·0
8	16·9	53·0
9	17·3	64·0
20	17·6	59·0
18	18·5	26·0

From the above it will be readily observed that there does not seem to be any direct relation between the volatile matter evolved subsequently to the plastic stage, and the shatter index, which is a criterion of coke quality.

IV.—*Caking or Agglutinating Index:* It was first shown by Richters[14] that those coals which are recognized as falling within the best coke-producing class are capable of withstanding a higher mixture of inert material and still yield a carbonized residue of definite crushing strength, than are the more inferior coals. This phenomenon has since been thoroughly studied and methods developed, among which are those of Gray[15], Meurice[16], Barash[17], Burdekin[18], and Marshall-Bird[19].

Slater[20] has made a comparison of several of these methods and tried to correlate the results with the "shatter index". He concluded that the laboratory tests are capable, to only a very limited extent, of assessing the probable value of coals as coke producers. Within the limits of the range of carbon content of the coals he examined (i.e. 81·8 per cent to 91·4 per cent dry ash-free basis) the "shatter index" showed a fairly regular increase with increasing carbon content, whilst the "caking value" rose sharply to a maximum value with coals of approximately 86 per cent carbon, and then fell rapidly. Curves plotted from the above are dissimilar in form, and hence Slater concludes there can be no simple relationship between the two sets of values (i.e. "caking index" and "shatter index").

Audibert[9] claims that the "agglutination index" has no practical importance unless the rate of heating is of the same order as the rate at which the temperature rises in the plastic layer in the usual type of coke oven, that is, about 1° C. per minute. Foxwell[7], in a résumé of coking properties of coals, concludes that "there is little or no agreement between the coking index or the percentage of gamma compounds in the coal and its coking properties when carbonized in the coke oven".

As stated before, according to Damm[6] "caking index" alone is not sufficient and reliable in estimating the coking properties of a coal, and does not enable prediction of the nature of the obtainable coke. The authors have had the opportunity of confirming the above statement. Series of coals and blends, which had been carbonized in the two-ton by-product oven of the Fuel Research Laboratories, were subjected to two "caking index" tests, viz. the Gray and the Marshall-Bird.* There seems to be no definite relation between "shatter index", or "size on the wharf", and the "caking index" as determined by either test, nor does there seem to be complete correlation between the two methods of determining the "caking index". This is readily seen in Table V.

While these tests are of uncertain value for the purpose of assessing the value of a wide range of coals in their application to the production of by-product cokes, a knowledge of the "caking" or "agglutinating value" is of importance in the case of those coals which, prior to carbonization, may be mixed with inert carbonaceous material or non-coking coals. One can reasonably assume that, the higher the agglutinating value of a coal the greater will be the amount of inert material which may be added to it and still permit the production of a satisfactory coke.

V.—*Coke "Buttons" from Volatile Determination at 950° C:* These were and still are used to a limited extent to give an indication of the coking properties of a coal. However, it does not appear reasonable to attempt to assess the coking value of a coal by means of a test in which the coal is very rapidly heated when, in commercial use, the coal is so slowly heated that modification of the coking properties may occur during the earlier stages of the carbonization period.

Foxwell[7] in discussing this problem states that: "It happens frequently that conclusions based on this test are found reliable in practice, but this is merely because almost any coal which yields good coke in the oven will do the same in the crucible". The authors have tested many coals from which excellent "buttons" were obtained in the crucibles but the coals would not coke in the oven.

Kattwinkel[21] says that it has been definitely proven that "caking coals include both coking and non-coking coals" (i.e. coals unsuitable for large-scale coking). The authors' experience confirms what has been stated above with respect to the futility of judging coke quality from coke "buttons" obtained from the volatile matter test at 950° C.

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*Invest. Fuels & Fuel Testing, 1929, p. 36; Mines Branch, Dept. of Mines, Canada (1932 ,

TABLE V

Sample	Caking index		Shatter index, per cent on 2-inch	On 3-inch	Total on 2-inch (i.e. 3-inch + 2-inch)	Total through ½-inch
	Gray	Marshall-Bird (kilograms)				
B501........	33	15·62	57·0	33·0	86·0	2·2
B168........	27+	12·26	53·0	45·8	77·7	3·7
B248........	35·5	14·51	54·0	38·3	78·6	5·4
B500........	24+	12·40	45·0	44·0	85·0	2·3
B415........	27·5	8·56	77·0	69·9	88·6	2·5
B499........	22	12·95	64·0	55·0	86·0	4·8
B498........	20·5	7·06	20·0	40·0	70·0	5·0
			Blends			
B386........	39·5	15·19	76·0	29·2	77·2	3·6
B335........	36·5	9·92	68·0	66·4	91·0	2·9
B366........	31·5	11·58	58·0	67·5	87·8	2·4
B365........	31·0	7·64	64·0	....	....	..... (slightly pebbly).
B353........	24·0	5·98	70·0	58·1	82·5	5·2 (very pebbly).
B427........	20	4·61	71·0	66·9	86·2	5·0 (very pebbly).

List of References

[1] Fischer, F., Broche, H., and Strauch, J.: The Coking Properties of Coal, Fuel, vol. V, No. 10, p. 466.

[2] Barash, M.: The Coking of Coals, Journal of the Society of Ind. Chem., July 12th, 1929, p. 174 T.

[3] Wheeler, R. V.: The Chemistry of Coal, Fuel, vol. VI, No. 11-1.

[4] Illingworth, S. R.: Researches on Constitution of Coal, the Colliery Guardian Co., Ltd., London, 62 pp.

[5] Foxwell, G. E.: The Plastic State of Coal, Fuel, vol. III, No. 8, p. 277.

[6] Damm, P.: Properties of Coking Coals and their Behaviour on Coking, Fuel, vol. VIII, No. 4, p. 163.

[7] Foxwell, G. E.: Fuel, vol. III, No. 4, p. 122; No. 6, p. 206; No. 7, p. 227; No. 8, p. 276; No. 9, pp. 315, 318, 371, 373.

[8] Layng and co-workers: Industrial and Engineering Chemistry, vol. 17, p. 165 (1925); vol. 19, p. 924 (1927).

[9] Audibert, E.: A Contribution to the Study of the Mechanism of the Carbonization of Coal, Fuel, vol. VIII, No. 5, p. 225.

[10] Mott, R. A., and Shimmura, T.: A Further Study of Coke Formation, Fuel, vol. VII, No. 11, p. 472.

[11] Slater, L.: A Study of the Swelling Power of Coal at Different Rates of Carbonization, Fuel, vol. VI, No. 2.

[12] Barash, M.: Scientific Control of Carbonization with Particular Regard to the Agglutinating Power of Coal, Fuel, vol. VI, No. 12, p. 533.

[13] Davidson, W.: The Plastic Range of Coking Coals, Fuel, vol. IX, No. 10, p. 489.

[14] Richters, E.: Dinglers' Polyt. Jour. 1870, 195, 71.

[15] Gray, T.: Fuel in Science and Practice, 1923, No. 2, p. 42.

[16] Meurice, A.: Am. Mines Belg.—1914, No. 19, p. 625.

[17] Barash, M.: Proposed Standard Method for the Determination of Agglutinating Value of Coal, Jour. Soc. Chem. Ind. (1926) 45, 151 T, 176 T.

[18] Burdekin, J. T.: Physical and Chemical Survey of the National Coal Resources, No. 8, 1927.

[19] Marshall, S. J., and Bird, B. M.: Test for Measuring the Agglutinating Power of Coal, A.I.M.E., Coal Division, 1930, p. 340 (with selected bibliography).

[20] Slater, L.: A Comparison of Methods for Testing the Caking Properties of Coal, Fuel, vol. IX, No. 12, p. 586.

[21] Kattwinkel, R.: The Examination of Coking Coals and Estimation of their Value, Fuel, vol. V, No. 8, p. 347.

IV

CHANGES IN FORMS OF SULPHUR IN COAL UNDER VARIOUS CONDITIONS OF WEATHERING

J. H. H. Nicolls and E. Swartzman

At various periods during the past decade members of the staff of these laboratories have investigated the forms of sulphur in Canadian coals, particularly those containing comparatively large amounts of sulphur. It has been thought that finely disseminated pyrites might oxidize readily and promote heating of coal; also that changes from pyrites to the sulphates of iron might cause cracking or checking in coal and thus assist in its oxidation.

The authors indicated in a previous paper[1] that pyritic sulphur in coal is distinctly and steadily oxidized to sulphate in warm dilute hydrochloric acid, or water, and more particularly in water. At the time that the tests described in the paper were carried out, certain coals were being exposed to ordinary indoor conditions in order to observe changes in the forms of sulphur. It was subsequently decided to expose portions of these coals to atmospheres of different humidities, and to admixture with water, in addition to the experiments already under way. This paper describes the changes that took place while the coals were subjected to various kinds of exposure during a period of about 30 months.

There were five different methods of exposure of the coals, which had been ground in a pebble mill so as almost entirely to pass through a 100-mesh sieve. These methods were as follows:—

(1) Exposure in uncovered quart jars, each almost full of coal. These were protected from dust by a cloth thrown over a frame which was constructed so as to keep the cloth an inch or two above the jars and to allow circulation of air over them. For the first eight months the coals were stored in an upstairs room where the humidity was usually low; for the remainder of the exposure period they were stored in the basement, where the humidity was distinctly higher. Five of the coals, designated as exposed "in laboratory", were brought upstairs for the last seven months and stored on a covered shelf in order to parallel the conditions of exposure described as follows under (2). All the samples were stirred thoroughly fortnightly.

The samples submitted to conditions as described below were all taken from the coals in the quart jars after 16 months' exposure. They were comparatively small (less than 100 grammes), and were stirred weekly.

(2) Exposure in the laboratory in flat dishes. These were covered with watch glasses, and stored on shelves thoroughly protected from dust by overhanging paper.

(3) Exposure in air-drying apparatus. These were placed on the shelves of the apparatus used for air-drying coal, in which a condition of 60 per cent relative humidity is maintained.

(4) Exposure in a water-saturated atmosphere. The samples were enclosed in desiccators over water.

(5) Exposure in water. These were stirred to a paste with an excess of water, and stored in covered beakers, or dishes, with the other samples

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[1] Mines Branch publication No. 712; Investigations of Fuels and Fuel Testing, 1928, p. 28.

in the laboratory. Water was added from time to time so that the coal should not become completely dry.

Some of the coals exposed, especially those from the Sydney area high in ash, were distinctly hygroscopic. Therefore, the coals from the last three conditions of exposure were placed beside the samples on the shelves for a short period prior to each set of analyses, in order that the moisture contents might become constant previous to weighing. Nearly all the coals from the fifth condition of exposure, and some very hygroscopic coals from the water-saturated atmosphere, caked upon drying, so that they had to be removed with a spatula. It was considered advisable to mix all the coals that had been subjected to the 4th and 5th kinds of exposure in a mortar. Deposits, presumably mainly ferrous sulphate, separated from some of these coals and formed upon the sides of the beakers or dishes. Most of these were removed with a spatula and added to the corresponding coals before mixing in the mortar. Subsequently, the residual deposits were leached with water on to the mixed coal, which was again allowed to dry. Finally, this dried coal was again thoroughly mixed in the mortar, after which it was considered to be ready for analysis.

The results of the exposure tests are shown in Table I, and it is evident that the sulphate sulphur is the principal constituent affected. It has been assumed that the total sulphur remains unaltered in the dry coals, and repeat sulphur determinations were carried out only upon the coals from the three last conditions of exposure, and those only at the time of the final analyses.

A summary of the effects of the various conditions of exposure is as follows:—

(1) Exposure in quart jars.—A steady and, in many coals, a slow increase in sulphate sulphur accompanied by a decrease in the pyritic form.

(2) Exposure in laboratory in flat dishes.—Results very similar to the last, but, on the average, a little less sulphate sulphur formed.

(3) Exposure in air-drying apparatus.—In nearly all coals there is more sulphate sulphur than in either of the preceding kinds of exposure. The total sulphur and the organic form both seem to be less than in the original coal.

(4) Exposure in water-saturated atmosphere.—In this case there is, on the average, distinctly more sulphate sulphur formed than in any other kind of exposure. The final analyses show less total sulphur and less of the organic form than in any previous case.

(5) Exposure in water.—Although there is a very large amount of sulphate sulphur formed, it is not so great as that which results from exposure to the water-saturated atmosphere. The total amount of sulphur, and also of that in the organic form, seems to have decreased more than during any other condition of exposure.

The above conclusions may be further summarized by stating that dampness, particularly the action of wet air as distinct from a complete covering layer of water, promotes a comparatively rapid oxidation of pyritic sulphur to sulphate. The presence of water, and to a lesser extent of dampness, seems to promote a decrease of the total amount of sulphur present, more particulalry of that in the organic form.

TABLE I

Changes in Forms of Sulphur During Weathering of Coal

COALS FROM NOVA SCOTIA

All Coals in this Section of Table from Phalen Seam in Sydney Area

Date	Conditions and duration of weathering	Total sulphur, per cent of dry coal	Forms of sulphur—per cent of total sulphur			Total sulphur, per cent of dry coal	Forms of sulphur—per cent of total sulphur			Total sulphur, per cent of dry coal	Forms of sulphur—per cent of total sulphur		
			Sulphate	Pyritic	Organic		Sulphate	Pyritic	Organic		Sulphate	Pyritic	Organic
		No. 5166				No. 5173				No. 5177			
Dec. 11–13, 1928	Recently ground............	3·7	8·9	60·5	30·6	0·7	4·3	10·0	85·7	2·9	14·3	43·9	41·8
April 7–10, 1930	In quart jar..................	3·7	11·8	....	....	0·7	5·7	....	....	2·9	21·1	....	....
May 4– 7, 1931	“	3·7	14·3	51·0	34·7	0·7	5·5	8·2	86·3	2·9	27·1	31·0	41·9
		No. 5176				No. 5180				No. 5577			
Dec. 13–17, 1928	Recently ground............	1·3	13·0	18·7	68·3	6·1	8·6	71·0	20·4	2·4	2·9	62·8	34·3
April 16, 1929	In quart jar..................	1·3	14·6	....	....	6·1	8·9	....	....	....	....	....	....
April 1– 4, 1930	“	1·3	14·6	....	....	6·1	11·4	....	....	2·4	6·7	....	....
May 11–13, 1931	“	1·3	15·2	16·8	68·0	6·1	13·4	48·5	38·1	2·4	7·1	57·5	35·4
		No. 5170				No. 5580				No. 5582			
Dec. 11–19, 1928	Recently ground............	1·0	23·4	17·0	59·6	4·2	11·4	57·0	31·6	6·2	2·6	63·1	34·3
April 1–10, 1930	In quart jar................	1·0	25·5	....	....	4·2	15·6	....	....	6·2	7·7	....	....
Oct. 20, 1930	“	....	....	....	....	....	....	....	....	6·2	14·1	46·6	39·3
May 11, 1931	“	1·0	25·0	13·5	61·5	4·2	21·6	43·5	34·9	....	....	....	....
May 18, 1931	“ in laboratory...	....	....	....	....	....	....	....	....	6·2	15·1	49·9	35·0
Oct. 9–13, 1930	Thin layer, in laboratory since April 24, 1930........	1·0	24·2	13·1	62·7	4·2	18·9	45·7	35·4	6·2	12·0	49·0	39·0
May 21–28, 1931	Thin layer, in laboratory since April 24, 1930.......	1·0	27·1	15·6	57·3	4·2	19·8	47·7	32·5	6·2	13·1	50·0	36·9
Oct. 27–28, 1930	In air-drying apparatus since April 24, 1930.............	1·0	24·3	12·1	63·6	4·2	22·0	42·6	35·4	6·2	16·9	44·0	39·1
June 8–11, 1931	In air-drying apparatus since April 24, 1930.............	1·0	27·1	13·5	59·4	4·0	27·1	41·0	31·9	6·0	21·0	43·1	35·9

		No. 5168				No. 5181				No. 5581			
Dec. 11–19, 1928	Recently ground...........	0·8	11·4	8·9	79·7	12·7	4·4	67·8	27·8	5·4	3·2	57·1	39·7
April 16, 1929	In quart jar...............	....	....	....	....	12·7	5·0	....	....	....	....	....	....
April 1–10, 1930	"	0·8	12·6	....	....	12·7	7·5	....	....	5·4	7·6	....	....
Oct. 20, 1930	"	....	....	....	....	12·7	10·2	58·7	31·1..	....	....	....	....
May 4–5, 1931	"	0·8	13·7	6·3	80·0	....	....	....	....	5·4	12·5	45·6	41·9
May 18, 1931	" in laboratory...	....	....	....	....	12·7	11·3	59·5	29·2	....	....	....	....
Oct. 9–13, 1930	Thin layer, in laboratory since April 24, 1930.......	0·8	13·7	7·5	78·8	12·7	9·4	66·3	24·3	5·4	11·1	46·5	42·4
May 20–26, 1931	Thin layer, in laboratory since April 24, 1930......	0·8	16·8	10·8	72·4	12·7	9·6	67·1	23·3	5·4	12·1	47·8	40·1
Oct. 23–28, 1930	In air-drying apparatus since April 24, 1930............	0·8	13·2	4·8	82·0	12·7	12·4	55·5	32·1	5·4	15·3	41·6	43·1
June 4–10, 1931	In air-drying apparatus since April 24, 1930.............	0·8	14·3	9·1	76·6	12·1	15·5	60·7	23·8	5·2	19·1	41·0	39·9
Nov. 18–20, 1930	In saturated atmosphere since April 23, 1930.......	0·8	15·1	5·8	79·1	12·7	25·4	40·8	33·8	5·4	32·0	23·0	45·0
June 16–18, 1931	In saturated atmosphere since April 23, 1930.......	0·8	15·8	7·9	76·3	11·3	39·0	38·3	22·7	5·0	41·7	18·5	39·8
Nov. 27, 1930	In water since April 23, 1930.	0·8	13·3	8·4	78·3	12·7	18·1	49·1	32·8	5·4	27·7	27·9	44·4
June 24–25, 1931	In water since April 23, 1930.	0·8	16·9	11·7	71·4	11·3	23·6	51·0	25·4	5·0	32·7	30·7	36·6

		No. 5578				No. 5579				No. 5583			
Dec. 17–19, 1928	Recently ground...........	3·7	1·9	62·3	35·8	6·8	2·2	63·9	33·9	8·0	5·4	62·5	32·1
Mar. 31, 1930	In quart jar...............	3·7	6·1	....	....	6·8	7·3	....	....	8·0	9·4	....	....
Oct. 20, 1930	"	....	....	....	....	6·8	11·5	53·2	35·3	....	....	....	....
May 5–11, 1931	"	3·7	10·1	51·4	38·5	....	....	....	....	8·0	14·9	50·2	34·9
May 18, 1931	" in laboratory...	....	....	....	....	6·8	12·2	56·3	31·5	....	....	....	....
Oct. 9–15, 1930	Thin layer, in laboratory since April 24, 1930......	3·7	9·2	52·6	38·2	6·8	10·8	54·4	34·8	8·0	12·8	50·8	36·4
May 21–28, 1931	Thin layer, in laboratory since April 24, 1930.......	3·7	8·2	54·1	37·7	6·8	11·0	55·0	34·0	8·0	14·0	53·8	32·2
Oct. 23–29, 1930	In air-drying apparatus since April 24, 1930............	3·7	11·3	47·7	41·0	6·8	14·1	50·3	35·6	8·0	16·2	45·8	38·0
June 8–11, 1931	In air-drying apparatus since April 24, 1930............	3·6	13·8	49·7	36·5	6·6	17·2	51·0	31·8	7·6	20·5	48·6	30·9
Nov. 18–20, 1930	Jn saturated atmosphere since April 23, 1930.......	3·7	26·1	32·8	41·1	....	....	....	....	8·0	30·9	30·7	38·4
June 18–22, 1931	In saturate datmosphere since April 23, 1930.......	3·6	34·0	28·6	37·4	....	....	....	....	7·2	45·4	23·8	30·8
Nov. 26–27, 1930	In water since April 23, 1930.	3·7	30·9	29·5	39·6	....	....	....	....	8·0	26·1	36·0	37·9
June 25–30, 1931	In water since April 23, 1930.	3·6	34·8	28·4	36·8	....	....	....	....	7·4	32·4	41·5	26·1

TABLE I—Concluded

Changes in Forms of Sulphur During Weathering of Coal—*Concluded*
Coals from Nova Scotia—*Concluded*

Date	Conditions and duration of weathering	Total sulphur, per cent of dry coal	Forms of sulphur—per cent of total sulphur			Total sulphur, per cent of dry coal	Forms of sulphur—per cent of total sulphur			Total sulphur, per cent of dry coal	Forms of sulphur—per cent of total sulphur		
			Sulphate	Pyritic	Organic		Sulphate	Pyritic	Organic		Sulphate	Pyritic	Organic
		No. 5584, Phalen seam, Sydney area				No. D84, Inverness				No. 5589, Kimberley seam, River Hebert			
Dec. 19, 1928	Recently ground...........	5·8	5·3	58·8	35·9	......	......	......	......	......	......	......	......
Jan. 14, 1929	Recently ground...........	......	......	......	......	......	......	......	......	6·2	3·7	56·3	40·0
Mar. 22–31, 1930	In quart jar..............	5·8	8·9	......	......	......	......	......	......	6·2	14·2	......	......
Oct. 20, 1930	"	......	......	......	......	......	......	......	......	6·2	20·1	32·9	47·0
May 5, 1931	"	5·8	13·7	48·3	38·0	......	......	......	......	......	......	......	......
May 19, 1931	" in laboratory..	......	......	......	......	......	......	......	......	6·2	21·8	33·2	45·0
May 31, 1930	Recently prepared..........	......	......	......	......	7·5*	4·6	59·2	36·2	......	......	......	......
Oct. 15–20, 1930	Thin layer, in laboratory since April 24, 1930......	5·8	11·8	49·1	39·1	7·5*	9·4	48·0	42·6	6·2	19·9	33·1	47·0
May 28–June 4/31	(Except No. D84)......	5·8	12·9	51·0	36·1	7·5*	10·0	51·3	38·7	6·2	21·4	33·8	44·8
Nov. 3– 5, 1930	In air-drying apparatus since April 24, 1930............	5·8	15·2	46·5	38·3	7·5*	13·6	46·6	39·8	6·2	21·9	31·3	46·8
June 11–16, 1931	(Except No. D84)......	5·6	18·7	46·2	35·1	7·2	17·7	46·5	35·8	6·0	26·1	29·7	44·2
Nov. 20–24, 1930	In saturated atmosphere since April 23, 1930......	5·8	29·7	29·5	40·8	7·5*	27·4	28·0	44·6	6·2	31·8	22·3	45·9
June 22–23, 1931	(Except No. D84)......	5·4	41·0	22·5	36·5	6·8	39·4	22·6	38·0	6·1	37·4	24·5	38·1
Nov. 27–30, 1930	In water since April 23, 1930 (Except No. D84)......	5·8	26·3	32·2	41·5	7·5*	19·9	36·3	43·8	6·2	38·2	18·8	43·0
June 30, 1931	In water since April 23, 1930 (Except No. D84).....	5·3	31·8	35·3	32·9	6·8	26·1	38·4	35·5	5·6	45·4	16·4	38·2
		No. 5590, Queen seam, River Hebert				No. 5591, Joggins seam, River Hebert				No. 5592, Joggins seam, River Hebert			
Jan. 14, 1929	Recently ground............	6·9	1·8	70·5	27·7	5·5	1·5	70·8	27·7	6·6	3·1	59·6	37·3
April 16, 1929	In quart jar...............	......	......	......	......	5·5	1·3	......	......	6·6	2·5	......	......
Mar. 22, 1930	"	6·9	8·5	......	......	5·5	8·9	......	......	6·6	19·8	......	......
Oct. 22, 1930	"	......	......	......	......	......	......	......	......	6·6	26·0	25·7	48·3
May 5– 6, 1931	"	6·9	16·0	53·2	30·8	5·5	15·0	53·5	31·5	......	......	......	......
May 19, 1931	" in laboratory...	......	......	......	......	......	......	......	......	6·6	27·7	25·3	47·0
Oct. 16, 1930	Thin layer, in laboratory since April 24, 1930.......	6·9	13·5	53·5	33·0	5·5	13·2	52·1	34·7	6·6	25·7	26·7	47·6
June 1– 4, 1931	Thin layer, in laboratory since April 24, 1930.......	6·9	15·6	54·4	30·0	5·5	14·8	53·5	31·7	6·6	26·8	28·5	44·7
Nov. 3, 1930	In air-drying apparatus since April 24, 1930............	6·9	18·1	47·7	34·2	5·5	16·4	48·9	34·7	6·6	27·9	24·7	47·4
June 15–16, 1931	In air-drying apparatus since April 24, 1930............	6·8	22·0	47·6	30·4	5·4	19·7	50·1	30·2	6·4	32·1	24·0	43·9
Nov. 24, 1930	In saturated atmosphere since April 23, 1930.......	......	......	......	......	5·5	27·2	36·3	36·5	......	......	......	......
June 23, 1931	In saturated atmosphere since April 23, 1930.......	......	......	......	......	5·0	54·8	13·8	31·4	......	......	......	......
Dec. 1, 1930	In water since April 23, 1930.	......	......	......	......	5·5	38·3	25·6	36·1	......	......	......	......
June 30, 1931	In water since April 23, 1930.	......	......	......	......	5·0	45·5	28·1	26·4	......	......	......	......

* Moisture used in calculation approximated.

V

COAL FREE FROM INORGANIC MINERAL MATTER FOR THE PURPOSE OF CLASSIFICATION

J. H. H. Nicolls and E. Swartzman

During the past decade the classification of coal according to rank has been under frequent discussion on the continent of North America. For such a classification the composition of the pure coal substance free from inorganic mineral matter is necessary, or at least very desirable. It was originally assumed that the adjustment of a coal analysis to the (determined) ash-free basis would give the analysis of the pure coal. However, it was found that the analyses of pure coal calculated from two similar samples did not agree when the respective contents of ash-forming material were of different dimensions. This is due to the fact that the determined ash is not the same as the original mineral matter, because of changes in such substances as hydrated silicates, carbonates, and sulphides.

Probably the most thorough investigation of the nature of inorganic mineral matter in coal was carried out by the late Professor Parr and his associates of the University of Illinois and the Illinois Geological Survey. As a result of this investigation a method was devised for the calculation of the composition of "Unit Coal"[1]. This method assumed that, for purposes of calculations, most of the sulphur was present as pyrites, an assumption that subsequent investigation has somewhat altered. Recently, Fieldner and Selvig[2] suggested a modification in which the sulphur was assumed to be composed of equal proportions of the inorganic and organic forms.

In 1925 Stansfield[3], of the Alberta Research Council, after a thorough study of the coals of that province (which generally contain comparatively little sulphur, and that organic), found that 1·1 was a representative factor for converting ash, as determined, to inorganic mineral matter. It has been found by the writers that, as a general rule, pure coal analyses calculated by the Stansfield (in which mineral matter equals 1·1 × ash) and the Parr methods agree closely, except in the cases of certain high-sulphur coals from eastern Canada, in which the calorific values are somewhat greater by the Parr method. Because of its simplicity, and because it has been assumed that the differences between the results of the two methods of calculation would not be sufficiently great to disturb a general classification scheme, the Stansfield (1·1 × ash) calculation has been usually employed by the senior author in the study of Coal Classification.

Table I shows comparisons between the results of the ash-free calculation and of the Stansfield method (1·1 × ash) and the Parr method of calculation when applied to certain coals of high rank, selected because of their significant contents of ash-forming material. The upper portion of the table contains analyses of hand-picked samples; the lower portion those of samples prepared by separation in a heavy liquid. None of these samples

[1] "Unit Coal and the Composition of Coal Ash," Parr and Wheeler, Univ. of Illinois Bull. No. 37, vol. 6, No. 43, Aug. 9, 1909.

[2] "Present Status of Ash Corrections in Coal Analysis," Amer. Inst. Min. and Met. Eng., Trans. Coal Div., 1930, page 517.

[3] "A Chemical Survey of Alberta Coals", Bull. Can. Inst. Min. and Met., No. 156, page 406, April, 1925.

contains more than about 1 per cent of sulphur. The table compares percentages of volatile matter obtained with electric furnaces and gas burners, and shows that they are generally within permissible checking limits for any coal sample. Both these methods are employed in the Fuel Research Laboratories, though the furnace is preferred for coals belonging to the anthracitic class and for cokes.

It has been customary, in calculating to the "mineral matter"-free basis, to subtract from the volatile matter (and from the oxygen in the ultimate analysis) the amount added to the ash. This has the effect, particularly with the high-ash coals shown in Table I, of lowering the volatile matter and increasing the fuel ratio $\frac{\text{fixed carbon}}{\text{volatile matter}}$ and thus raising the rank of the coals. If, as seems admissible, some of the correction should be applied to the fixed carbon instead of to the volatile matter, the rank of the coals would be somewhat exaggerated by this customary calculation. It has been found that the "mineral matter"-free volatile matter and the "Unit"[1] volatile matter of Parr are almost identical, so that the latter is not shown in the table. In the case of the calorific values the differences are somewhat more pronounced, so that both are shown. However, these are comparatively close together, and much more uniform than those calculated to the ash-free basis.

The Stansfield (1·1 × ash) and the Parr methods of calculation agree satisfactorily for the coals of this country, except in the case of certain coals in the Maritime Provinces which contain notable amounts of pyrites. To illustrate this, Tables II and III have been prepared to deal respectively with representative coals from Glace Bay in the Sydney area in Nova Scotia, and from the Minto area in New Brunswick. The tables show the results of the methods of calculation so far discussed and, in addition, a modification of Parr's method deduced by the present authors after a study of the modification proposed by Fieldner and Selvig. They also show the results obtained by a simplified method of calculation giving values almost identical with those from the original Parr formula, deduced by G. Thiessen[2], of the Illinois State Geological Survey, and by a further modification of the same suggested by Fieldner. The last formula was taken from a private communication to the Fuel Research Laboratories.

All the coals analysed in each case were prepared from one large sample by separations in solutions of zinc chloride of various specific gravities. They cover a range of ash of from 2 to 26 per cent and of sulphur of from 1·2 to 10·8 per cent for the Sydney coal, and of from 6 to 44 per cent of ash and 3·6 to 16·5 per cent of sulphur for the Minto coal. The British thermal units resulting from each method of calculation were averaged, as shown in the bottom lines of the tables, and the divergences, or differences from the mean value, calculated for each individual coal sample. These divergences are shown in the last seven columns of the tables, and are, in turn, averaged in the bottom lines. It is assumed that, for each coal, the smallest mean divergence usually corresponds to the most satisfactory method of calculating the B.T.U. in the pure coal substance, though some allowance may be made for simplicity of calculation.

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[1] "The Classification of Coal", Parr, Univ. of Illinois Bull. No. 180, vol. 25, No. 428, July 31, 1928.

[2] Thiessen, G.: A Proposed Simplification of the Parr Unit Coal Formula; Fuel in Science and Practice, vol. XII, No. 12, p. 403.

It was stated in an earlier paragraph that Stansfield recommended a $\frac{\text{“mineral matter”}}{\text{ash}}$ factor of 1·1, because it was not far from the mean value for Alberta coals and because of its simplicity in calculations. Actually, he obtained values varying from 1·09 to 1·25. He prepared various coal fractions by means of separations in heavy solutions, as was done in connexion with the coals shown in the tables now under discussion, obtaining samples with widely varying amounts of ash. He plotted the analyses (on the dry basis) of the fractions on a diagram, the B.T.U. on the vertical axis and the corresponding ash percentages on the horizontal axis. A straight line drawn as nearly as possible through the points thus plotted when extrapolated cut the axis of zero calorific value at a point considerably short of 100 per cent of ash. He, therefore, came to the conclusion that 100 divided by the ash percentage corresponding to zero calorific value represented the ratio of “mineral matter” to ash. The figures shown at the head of the seventh and fourteenth columns of Tables II and III were obtained by the procedure just described, and therefore represent the correct Stansfield values for these particular Sydney and Minto coals.

The determination of the forms of sulphur (sulphate, pyritic, and organic[1]) in coals from the Maritime Provinces has led the writers to form the following general conclusion, for such coals as from their analyses may be thought to contain much pyrite.[2] If the sulphur be less than 2 per cent, 40 per cent may be assumed to be pyritic; if it be from 2 to 3 per cent, 50 per cent may be considered as pyritic; if it be from 3 to 7 per cent, 60 per cent may be taken to be pyritic; if it be over 7 per cent, 75 per cent may be considered as pyritic. The modified Parr's “Unit Coal” formula, as used in preparing Tables II and III, was evolved on the supposition that these conclusions are correct. The original “Unit B.T.U.” formula is:

$$\frac{\text{Indicated B.T.U.} - 5000\ \text{sulphur}}{1\cdot 00 - (1\cdot 08\ \text{ash} + \frac{22}{40}\ \text{sulphur})}$$

In the modified formula the figures by which the sulphur is multiplied change as follows:

Below 2 per cent..........	4530 in the numerator,		0·81 in the denominator		
2 per cent to 3 per cent....	4650	“ “	0·75	“	“
3 per cent to 7 per cent....	4770	“ “	0·715	“	“
Above 7 per cent..........	4950	“ “	0·64	“	“

Study of the last seven columns in the tables shows that the Stansfield factor resulting from the analyses tabulated (as distinct from 1·1) corresponds not only to the smallest divergences but also to the highest calorific values for the pure coal. (It may be stated that a factor based upon calculations supplied by J. L. Bowlby[3] gave almost as small divergences and almost as high calorific values, when applied to a series of sections taken from the Phalen seam at a locality designated “O.”) Next in order is the modified Parr formula, but the results from this are considerably less regular than the foregoing. Results fron Parr's original “Unit Coal” (or from Thiessen's substitute formula) are somewhat less regular than those from the modification of it, and the figures from the simplification suggested by Fieldner seem to be a little less satisfactory. Values calculated by the

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[1] Mines Branch publications: Investigations of Fuels and Fuel Testing, No. 618, (1923), p. 28; No. 689, (1926), p. 34.

[2] Usually coals containing only 1 per cent of sulphur contain little or no pyrites.

[3] Formerly of these Laboratories, and now with the Dominion Steel and Coal Corporation, Limited.

1·1 factor are less uniform than the preceding, particularly for the Minto coal, and those calculated directly to the ash-free basis are very irregular. The writers suggest that a $\frac{\text{“mineral matter”}}{\text{ash}}$ factor of 1·25 may be preferable to 1·1 for coals containing considerable amounts of pyrites.

Table IV contains the analyses of certain coal constituents calculated according to various conceptions of the "pure-fuel" basis. It has been included as a matter of general interest, principally because it contains analyses of "fusain" or "mother-of-coal". This constituent of coal is sometimes considered to vary so much from the other constituents in composition as to misplace a coal in the classification scheme when present in large amounts. It is clear that the "fusain" in the Sydney coal is different from the other two constituents, which correspond quite closely; it would, however, require a large quantity of such fusain to disturb seriously the classification of Sydney coal. The "fusain" in the Ontario lignite is not very different in composition from the other constituents. As the sulphur content of the lignite is small, except in the fusain, where it amounted to 3·6 per cent on the dry basis, the factor 1·3 (derived from the Phalen O series and therefore considered suitable for this table) was applied to that constituent only.

It is emphasized in Table I that the classification of coal with a large content of ash-forming material may be seriously disturbed. In order further to study the effect of inorganic mineral matter, coal samples were prepared from three sizes of a high-rank coal from Virginia. Table V shows analyses of "floats" and "sinks" obtained by separations of the coal in heavy solutions of zinc chloride, and, also, of the residues from extractions of the coal with hydrochloric and hydrofluoric acids to remove inorganic material. In addition to the figures shown in Table I, Table V shows analyses calculated to the correct "mineral matter"-free basis, as obtained from the "float" and "sink" samples by Stansfield's method.

The analyses of the "float" and "sink" samples, particularly those with high ash, show clearly that the rank assigned to a coal by its fuel ratio (or by its volatile matter) is very much higher on the "mineral matter" (1·1 × ash)-free basis than on the ash-free, and that the use of the factor 1·14 produces a still greater increase in rank. This is even more emphasized in the case of the calorific value; both the customary "mineral matter" calculation, and its modification for the Virginia coal, give distinctly higher values than does the Parr calculation. This is probably because all the sulphur in the Virginia coal (0·5 to 0·6 per cent) is organic.

The last seven analyses in the table show the effect of extraction of the pulverized pea-size coal by a method proposed by Turner, of Lehigh University, Bethlehem, Pa., or by modifications thereof. Turner's method which was submitted to the A.S.T.M. Technical Committee on the Scientific Classification of Coal for committee records, is briefly as follows:

Coal is digested with concentrated hydrochloric acid, diluted with an equal volume of water, for eight hours, and allowed to stand over night to settle. The acid is then decanted through a Buchner funnel. The residual coal is boiled with distilled water, filtered on the Buchner, and washed free of chlorides. After drying at a low temperature, the coal is brushed into a 3-inch platinum basin and covered well with hydrofluoric acid, which is then boiled until no more fumes are evolved. The residue is washed into a beaker containing 500 c.c. of 1:1 hydrochloric acid, boiled for one hour, filtered, washed free from chlorides and dried to constant weight at about 50°c.

Samples Nos. 6333, 6334, and 6335 show clearly that the effect of Turner's method is to increase fuel ratios (decrease the contents of volatile matter) but materially to lower calorific values; in other words, the aggregate effect of the two hot acids seems to be an attack upon the organic coal substance.

The next two samples show that extraction with boiling dilute hydrochloric for not more than four hours does not materially affect the organic coal substance; neither does it remove much inorganic material. Sample 6472 is of considerable interest, as it shows that prolonged extraction with cold hydrofluoric acid, although it reduces the ash from 18·8 to 6·0 per cent, does not materially affect the pure coal substance; in other words, the hot hydrofluoric probably attacked the coal during Turner's procedure. Finally, treatment of the residue from the cold extraction with hydrofluoric acid for a total of twelve hours with boiling dilute hydrochloric, although it removed almost all the inorganic material, had not a great effect on the organic coal substance. It is altogether likely that four hours' extraction of residue 6472 with the hydrochloric acid would have removed all the inorganic material, without altering the coal substance to any considerable extent.

These experiments, therefore, render available a method for removing the inorganic material from higher rank coal without seriously altering the coal substance proper.

SUMMARY

Various methods proposed for calculating the composition of "pure" or organic coal substance from routine laboratory analyses have been examined.

These methods of calculation have been applied to high-rank coals from Virginia containing considerable amounts of ash-forming material and but little sulphur, and to samples prepared from them in such a way as to show either very little or very much ash; also to coals from the Sydney area, Nova Scotia, and the Minto area, New Brunswick, containing notable amounts of pyrites, and to "float" and "sink" samples prepared from them.

It has been found that the factor suggested by Stansfield to convert ash to "mineral matter" previous to calculating a coal analysis to the "pure-fuel" basis, namely, 1·1, is satisfactory for all coals except, perhaps, those containing more than 2 per cent of sulphur, in which the pyritic form is found to predominate. In this case a general factor of 1·25 is suggested.

Analyses of the various constituents of Sydney, Nova Scotia, coal and of northern Ontario lignite are shown after calculation to the "pure-fuel" basis. Most of the constituents have been found to have similar analyses, though "fusain" differs somewhat from the others in composition.

A method is described for the removal of inorganic mineral matter, or ash-forming material, from higher-rank coal with hydrofluoric and hydrochloric acids, leaving the organic material or "pure coal" almost unaltered.

TABLE I

Comparative Analyses of Coals of High Rank, and of Fractions thereof, showing what Errors may be caused by the Use of Ash, as determined, as against Mineral Matter

("Mineral Matter" in this table represents Ash ×1·1.—All analyses reported on "Dry Basis")

Sample No.	Description	Ash	Volatile Matter		Fuel Ratio		B.T.U. per lb., Gross		
			Ash-free	M.M.-free	Ash-free	M.M.-free	Ash-free	M.M.-free	Parr's "Unit Coal"
		%							
2715	Virginia coal..	26·6	15·75	12·60	5·35	6·95	....	....	....
2717	"	15·1	12·20	10·60	7·20	8·40	....	....	....
2730	"	19·5	15·20	13·10	5·55	6·65	....	....	....
2743	"	19·2	14·10	12·05	6·10	7·30	15,100	15,470	15,440
2743A	Clean coal from No. 2743............................	7·9	12·65	11·90	6·90	7·40	....	....	....
2743B	Medium-run coal from No. 2743.....................	16·6	13·35	11·55	6·50	7·65	....	....	....
2743C	Dirty coal from No. 2743..........................	45·5	22·35	15·05	3·50	5·65	....	....	....
2776	Virginia coal..	23·5	14·10	11·10	6·25	8·05	15,020	15,500	15,430
2776A	Clean coal from No. 2776..........................	13·8	13·60	12·05	6·35	7·20	....	....	....
2776B	Dirty coal from No. 2776..........................	27·2	15·65	12·40	5·40	6·70	....	....	....
3058	Virginia coal..	18·8	14·30	12·30	6·00	7·15	....	....	....
3058A	"	17·2	13·95	12·15	6·15	7·25	....	....	....
3577A	"	17·1	18·70	17·00	4·35	4·90	....	....	....

3577B	"		21·4	26·85	24·75	2·70	3·05	....	....	....
3578A	"		16·8	20·55	18·95	3·85	4·25	....	....	....
3578B	"		26·5	12·70	9·40	6·90	9·60	....	....	....
6505	"		19·9	18·15	16·05	4·50	5·20	15,010	15,400	15,330
6086	" Stove	Chaddock*	16·0	16·85	15·20	4·95	5·55	15,290	15,590	15,550
		Fieldner. **	....	16·80	15·20	4·95	5·60	....	....	....
6087	" Chestnut	Chaddock.	18·5	16·55	14·60	5·00	5·85	15,260	15,610	15,570
		Fieldner...	....	16·65	14·70	5·00	5·80	....	....	....
6088	" Pea	Chaddock.	18·8	16·55	14·55	5·05	5·85	15,260	15,620	15,580
		Fieldner...	....	15·95	13·95	5·25	6·20	....	....	....
5789	Welsh anthracite	Chaddock.	5·8	8·40	7·85	10·90	11·75	....	....	....
		Fieldner...	....	7·85	7·25	11·75	12·75	....	....	....
5789A	Float from Welsh on 1·35 sp. gr. zinc chloride solution	Chaddock.	2·8	9·35	9·10	9·65	10·00	15,380	15,430	15,450
		Fieldner...	....	9·45	9·15	9·60	9·90	....	....	....
5789B	Sink corresponding to last	Chaddock.	15·1	10·45	8·80	8·55	10·55	15,030	15,300	15,280
		Fieldner...	....	10·20	8·60	8·80	10·65	....	....	....
6211	Scotch anthracite	Chaddock.	7·9	10·20	9·40	8·85	9·65	15,140	15,270	15,270
		Fieldner...	....	10·00	9·20	9·00	9·85	....	....	....
6211A	Float from separation of Scotch with 1·35 sp. gr. zinc chloride solution	Chaddock.	5·2	11·30	10·80	7·90	8·25	15,430	15,520	15,520
		Fieldner...	....	11·55	11·05	7·65	8·05	....	....	....
6211B	Sink corresponding to last	Chaddock.	14·9	11·10	9·30	8·00	9·50	14,920	15,190	15,170
		Fieldner...	....	11·30	9·70	7·85	9·30	....	....	....

* Volatile matter determined over Chaddock gas burners. ** Volatile matter determined in Fieldner electric furnaces.

TABLE

"Floats" and "Sinks" from Sample O, Phalen Seam, Glace Bay, Nova

—	Dry Basis			Ash-free	(1·1 × Ash)-free	(1·3 × Ash)-free*	Parr's "Unit Coal"	Modified Parr's "Unit Coal"
	Ash	Sulphur	B.T.U.	B.T.U.				
Float 1·30....	2·04	1·21	14,796	15,104	15,135	15,203	15,170	15,225
" 1·35....	2·54	1·28	14,569	14,948	14,987	15,070	15,025	15,083
" 1·35....	2·83	1·41	14,737	15,163	15,210	15,300	15,260	15,317
" 1·30....	2·93	1·34	14,717	15,160	15,207	15,293	15,250	15,310
" 1·40....	2·99	1·36	14,609	15,059	15,106	15,207	15,143	15,207
" 1·40....	3·24	1·46	14,533	15,020	15,070	15,175	15,110	15,180
" 1·25....	3·34	1·33	14,540	15,040	15,093	15,203	15,125	15,197
" 1·25....	3·64	1·45	14,483	15,030	15,086	15,207	15,130	15,193
" 1·20....	3·98	1·56	14,432	15,030	15,093	15,225	15,133	15,203
" 1·15....	5·40	2·09	14,233	15,047	15,133	15,307	15,190	15,270
Sink 1·25....	5·51	2·16	14,216	15,045	15,133	15,313	15,197	15,275
" 1·25....	6·27	2·47	14,013	14,947	15,053	15,260	15,125	15,213
" 1·20....	6·75	2·48	13,965	14,977	15,090	15,310	15,153	15,250
" 1·15....	14·69	1·49	12,273	14,387	14,637	15,147	14,640	14,707
" 1·30....	15·06	4·57	12,191	14,352	14,605	15,153	14,730	14,877
" 1·35....	15·39	4·49	12,249	14,473	14,743	15,313	14,857	15,010
" 1·30....	19·55	7·15	11,489	14,280	14,637	15,407	14,843	14,977
" 1·35....	26·50	10·83	10,048	13,668	14,183	15,350	14,530	14,760
" 1·40....	26·51	9·38	10,013	13,625	14,137	15,290	14,413	14,607
Mean values....	....	....	....	14,756	14,913	15,249	15,001	15,098

* Factor 1·30 derived from curve deduced from plotting anlyses of "float" and "sink" samples

TABLE

"Floats" and "Sinks" from Sample No. 4030 from Minto, New Bruns

—	Dry Basis			Ash-free	(1·10 × Ash)-free	(1·25* × Ash)-free	Parr's "Unit Coal"	Modified Parr's "Unit Coal"
	Ash	Sulphur	B.T.U.	B.T.U.				
Float 1·25....	6·26	3·58	14,299	15,260	15,350	15,513	15,470	15,580
1·40....	7·50	3·88	14,038	15,180	15,300	15,490	15,425	15,547
1·45....	8·36	4·21	13,853	15,117	15,260	15,475	15,397	15,530
1·50....	8·74	4·18	13,855	15,180	15,320	15,560	15,457	15,597
1·55....	9·96	4·33	13,624	15,125	15,300	15,560	15,433	15,570
1·60....	11·01	4·31	13,405	15,060	15,245	15,543	15,383	15,533
Sink 1·25....	17·61	6·57	12,173	14,770	15,090	15,610	15,313	15,540
1·30....	22·68	7·97	11,077	14,320	14,760	15,450	15,013	15,175
1·35....	25·18	8·76	10,681	14,270	14,765	15,587	15,080	15,257
1·40....	29·12	9·40	9,943	14,025	14,623	15,637	14,950	15,163
1·45....	32·01	10·00	9,270	13,630	14,280	15,457	14,640	14,867
1·50....	36·25	10·94	8,635	13,540	14,367	15,790	14,777	15,037
1·55....	40·36	13·62	7,786	13,060	14,003	15,713	14,523	14,847
1·60....	43·92	16·47	6,728	12,000	13,025	14,933	13,577	14,077
Mean values....	....	....	....	14,324	14,763	15,523	15,031	15,237

* Factor 1·25 is derived from curve deduced from plotting analyses of "float" and "sink"

II

Scotia—Comparison of Calorific Values Calculated to Various Bases

(1·08 ×Ash +0·2 ×S) -free	(1·1×Ash +0·1× S) -free	Ash-free	(1·1 × Ash)-free	(1·3 × Ash)-free*	Parr's "Unit Coal"	Modified Parr's "Unit Coal"	(1·08 × Ash +0·2 × S)-free (Thiessen)	(1·1× Ash +0·1×S) -free
B.T.U.		Divergence from Mean B.T.U.						
15,167	15,157	+ 348	+222	− 46	+169	+127	+170	+187
15,025	15,007	+ 192	+ 74	−179	+ 2	− 15	+ 28	+ 37
15,247	15,237	+ 407	+297	+ 51	+259	+219	+250	+267
15,243	15,230	+ 404	+294	+ 44	+249	+212	+246	+260
15,143	15,133	+ 303	+193	− 42	+142	+109	+146	+163
15,107	15,095	+ 264	+157	− 74	+109	+ 82	+110	+125
15,130	15,113	+ 284	+180	− 46	+124	+ 99	+133	+143
15,125	15,110	+ 274	+173	− 42	+129	+ 95	+128	+140
15,133	15,120	+ 274	+180	− 24	+132	+105	+136	+150
15,187	15,167	+ 291	+220	+ 58	+189	+172	+190	+197
15,190	15,170	+ 289	+220	+ 64	+196	+177	+193	+200
15,116	15,095	+ 191	+140	+ 11	+124	+115	+113	+125
15,150	15,130	+ 221	+177	+ 61	+152	+152	+153	+160
14,643	14,660	− 369	−276	−102	−361	−391	−354	−310
14,720	14,690	− 404	−308	− 96	−271	−221	−277	−280
14,850	14,823	− 283	−170	+ 64	−144	− 88	−147	−147
14,833	14,773	− 476	−276	+158	−158	−121	−164	−197
14,517	14,400	−1,088	−730	+101	−471	−338	−480	−570
14,417	14,323	−1,131	−776	+ 41	−588	−491	−580	−647
14,997	14,970	394	266	69	210	175	210	227

prepared from composite sample O.

III

wick—Comparison of Calorific Values Calculated to Various Bases

(1·08 ×Ash +0·2 × S) -free	(1·1× Ash +0·1 ×S) -free	Ash-free	(1·10 × Ash)-free	(1·25* × Ash)-free	Parr's "Unit Coal"	Modified Parr's "Unit Coal"	(1·08 × Ash+0·2 × S)-free (Thiessen)	(1·1× Ash +0·1×S) free-
B.T.U.		Divergence from Mean B.T.U.						
15,457	15,417	+ 936	+ 587	− 10	+ 439	+ 343	+ 444	+ 481
15,403	15,363	+ 856	+ 537	− 33	+ 394	+ 310	+ 390	+ 427
15,375	15,330	+ 793	+ 497	− 48	+ 366	+ 293	+ 362	+ 394
15,447	15,403	+ 856	+ 557	+ 37	+ 426	+ 360	+ 434	+ 467
15,417	15,380	+ 801	+ 537	+ 37	+ 402	+ 333	+ 404	+ 444
15,367	15,330	+ 736	+ 482	+ 20	+ 352	+ 296	+ 354	+ 394
15,280	15,220	+ 446	+ 327	+ 87	+ 282	+ 303	+ 267	+ 284
14,980	14,913	− 4	− 3	− 73	− 18	− 62	− 33	− 23
15,033	14,953	− 54	+ 2	+ 64	+ 49	+ 20	+ 20	+ 17
14,917	14,830	− 299	− 140	+ 114	− 81	− 74	− 96	− 106
14,617	14,533	− 694	− 483	− 66	− 391	− 370	− 396	− 403
14,727	14,627	− 784	− 396	+ 267	− 254	+ 200	− 286	− 309
14,500	14,357	−1,264	− 760	+ 190	− 508	− 390	− 513	− 579
13,663	13,443	−2,324	−1,738	− 590	−1,454	−1,160	−1,350	−1,493
15,013	14,936	775	503	117	387	322	382	416

samples prepared from sample No. 4030.

TABLE

Comparison of Analyses of Coal

Description	Volatile Matter			Carbon			Hydrogen	
	Ash-free	(1·1 × Ash)-free	(1·3 × Ash)-free	Ash-free	(1·1 × Ash)-free	(1·3 × Ash)-free	Ash-free	(1·1 × Ash)-free
Phalen Seam, Sydney Area, N.S.								
Bright coal	32·6	32·5	32·2	84·5	84·6	85·0	5·30	5·31
Bright coal	31·4	31·1	30·5	85·8	86·2	87·0	5·04	5·06
Dull coal	36·7	36·3	35·6	81·7	82·2	83·0	5·30	5·33
Dull coal	35·9	35·4	34·4	84·0	84·6	86·0	5·35	5·39
Fusain	30·2	28·4	24·4	85·1	87·3	92·1	3·78	3·88
Fusain	27·6	26·0	22·7	85·5	87·4	91·3	3·52	3·60
Onakawana Lignite, Abitibi River, Ontario								
Tree trunk, woody	48·8	48·5	....	70·0	70·5	....	5·01	5·04
Twigs, woody	50·4	50·1	....	69·5	69·9	....	4·86	4·88
Bark from last two	48·0	47·4	....	71·5	72·4	....	4·58	4·64
Hard, peaty	48·9	48·6	....	68·8	69·3	....	4·77	4·80
Soft, peaty	46·4	45·8	....	73·5	74·3	....	4·51	4·56
Fusain	40·0	39·2	37·6	74·0	74·9	76·8	3·95	4·01

IV

Constituents Calculated to Various Bases

Hydrogen	Oxygen			British Thermal Units				
(1·3 × Ash)-free	Ash-free	(1·1 × Ash)-free	(1·3 × Ash)-free	Ash-free	(1·1 × Ash)-free	(1·3 × Ash)-free	Parr	Modified Parr
Phalen Seam, Sydney Area, N.S.								
5·34	7·39	7·20	6·82	15,110	15,140	15,200	15,170	15,240
5·11	6·26	5·86	5·02	15,190	15,260	15,390	15,290	15,350
5·39	7·88	7·37	6·34	14,910	14,990	15,160	15,100	15,210
5·48	6·45	5·70	4·17	15,010	15,130	15,380	15,200	15,260
4·10	8·86	6·46	1·25	13,920	14,290	15,090	14,270	14,350
3·76	8·96	7·00	2·83	13,700	14,010	14,630	13,970	14,030
Onakawana Lignite, Abitibi River, Ontario								
....	23·7	23·3	....	11,650	11,720	....	11,720	....
....	24·6	24·2	....	11,700	11,770	....	11,760	....
....	21·2	20·2	....	11,790	11,930	....	11,940	....
....	25·1	24·5	....	11,500	11,580	....	11,570	....
....	20·1	19·2	....	11,780	11,910	....	11,910	....
4·10	17·4	16·4	14·2	12,030	12,180	12,500	12,220	12,320

TABLE V

Comparative Analyses of High-Rank Coal from Virginia, and of Fractional Parts thereof.

Sample No.	Description	Ash	Volatile Matter			Fuel Ratio			B.T.U. per lb., gross			
			Method	Ash-free	M.M.-free	Ash-free	M.M.-free	(1·14× Ash)-free	Ash-free	M.M.-free	(1·14× Ash)-free	Parr's "Unit Coal"
6086	Original coal—stove size..............	16·0	Chaddock..	16·85	15·20	4·95	5·55	5·85	15,290	15,590	15,710	15,550
			Fieldner....	16·80	15·20	4·95	5·60	5·90				
6087	Original coal—chestnut size..........	18·5	Chaddock..	16·55	14·60	5·00	5·85	6·25	15,260	15,610	15,770	15,570
			Fieldner....	16·65	14·70	5·00	5·80	6·20				
6088	Original coal—pea size...............	18·8	Chaddock..	16·55	14·55	5·05	5·85	6·25	15,260	15,620	15,780	15,580
			Fieldner....	15·95	13·95	5·25	6·20	6·60				
6099	Pea size through "coffee mill", Float from 1·37 sp. gr. solution.	12·4	Chaddock..	16·55	15·40	5·05	5·50	5·70	15,490	15,710	15,800	15,690
			Fieldner....	16·95	15·80	4·90	5·35	5·55				
6100	Sink corresponding to last..........	28·0	Chaddock..	19·00	15·80	4·25	5·35	5·95	14,850	15,450	15,720	15,370
			Fieldner....	19·05	15·80	4·25	5·35	5·95				
6234	Pea size through "coffee mill", Float from 1·34 sp. gr. solution.	14·7	Chaddock..	15·45	13·95	5·50	6·15	6·50	15,450	15,720	15,830	15,690
			Fieldner....	15·65	14·15	5·40	6·05	6·40				
6235	Sink corresponding to last...........	27·4	Chaddock..	17·95	14·75	4·60	5·80	6·50	14,960	15,550	15,750	15,470
			Fieldner....	17·95	14·75	4·60	5·80	6·50				
6236	Pea size through "coffee mill", Float from 1·31 sp. gr. solution.	12·8	Chaddock..	15·30	14·05	5·55	6·10	6·40	15,490	15,720	15,810	15,700
			Fieldner....	15·35	14·10	5·50	6·10	6·35				
6237	Sink corresponding to last............	23·4	Chaddock..	17·40	14·80	4·75	5·75	6·30	15,170	15,650	15,820	15,580
			Fieldner....	17·50	14·90	4·70	5·70	6·30				

6238	"Fines" (through 0·065 inch) from pea size through "coffee mill", Float from 1·34 sp. gr. solution.	14·0	Chaddock.. Fieldner....	15·60 15·30	14·20 13·90	5·45 5·55	6·05 6·20	6·35 6·50	15,530	15,790	15,890	15,770
6239	Sink corresponding to last...........	25·3	Chaddock.. Fieldner....	18·10 17·85	15·25 14·95	4·55 4·65	5·55 5·70	6·15 6·30	14,970	15,500	15,720	15,430
6240	"Coarse" (on 0·065 inch) from pea size through "coffee mill", Float from 1·34 sp. gr. solution.	9·2	Chaddock.. Fieldner....	14·25 14·45	13·40 13·60	6·00 5·90	6·45 6·35	6·65 6·55	15,540	15,700	15,780	15,690
6241	Sink corresponding to last...........	23·6	Chaddock.. Fieldner....	16·45 16·45	13·80 13·80	5·05 5·05	6·25 6·25	6·85 6·85	15,110	15,600	15,800	15,530
6333	Residue from extraction of pulverized No. 6086 by Turner's method.	0·7	Chaddock.. Fieldner....	12·70 12·00	12·65 11·95	6·85 7·30	6·90 7·35	6·95 7·40	15,370	15,380	15,380	15,400
6334	Residue from extraction of pulverized No. 6087 by Turner's method.	1·0	Chaddock.. Fieldner....	12·35 11·90	12·25 11·85	7·10 7·40	7·15 7·45	7·20 7·50	15,480	15,500	15,510	15,520
6335	Residue from extraction of pulverized No. 6088 by Turner's method.	0·8	Chaddock.. Fieldner....	12·25 12·90	12·15 12·10	7·15 7·20	7·20 7·25	7·25 7·30	15,460	15,480	15,480	15,490
6459	Residue from extraction of pulverized No. 6088 for 1 hour with boiling 1:1 hydrochloric.	16·2	Chaddock..	14·90	13·25	5·70	6·55	6·95	15,460	15,770	15,880	15,740
6460	Residue from extraction of pulverized No. 6088 for 4 hrs, with boiling 1:1 hydrochloric.	15·7	Chaddock..	14·10	12·45	6·10	7·00	7·50	15,540	15,830	15,960	15,800
6472	Residue from extraction of pulverized No. 6088 for 48 hours with cold 1:10 hydrofluoric.	6·0	Chaddock..	13·55	13·00	6·40	6·70	6·85	15,660	15,760	15,800	15,770
6473	Residue from extraction of No. 6472 for 12 hours with boiling 1:1 hydrochloric.	0·8	Chaddock..	12·45	12·35	7·05	7·10	7·10	15,660	15,670	15,670	15,690

VI

BATCH EXPERIMENTS ON THE HYDROGENATION AND CRACKING OF LOW-TEMPERATURE COAL TAR

T. E. Warren and A. R. Williams

This report comprises the results of batch hydrogenation experiments made in a one-litre autoclave and of comparative cracking tests on tar produced by the Illingworth low-temperature carbonization process from bituminous coal from the Sydney area, Nova Scotia. One reason for selecting this raw material was that tar produced by low-temperature carbonization has some of the chemical properties of coal. Accordingly, the conclusions obtained from the hydrogenation of low-temperature tar should be to some extent applicable to bituminous coal, although being a liquid it presents fewer experimental difficulties. A second reason was that a previous publication by this Department[1] has stated that it might be possible to apply a process of low-temperature carbonization to certain coals of the Sydney area with commercial success. If the operation were carried out on a scale of a thousand tons per day, as suggested, there would be produced about 500 barrels per day of tar, the profitable disposal of which would be essential to the success of the process. A market of almost unlimited extent would be made available by conversion of the tar to motor fuel. The process of hydrogenation has been used to convert coal tar and other carbonaceous materials to motor fuel on a commercial scale in Germany, although little has been made public concerning the conditions of operation. Therefore, the experiments reported in this paper have two objects in view, namely, to obtain general information on hydrogenation, and to begin the development of a method for the production of motor fuel from low-temperature tar from Sydney coal.

MATERIAL

An examination such as is ordinarily applied to crude oil, including a distillation by the Hempel method, gave the following data concerning the low-temperature coal tar used.

Characteristics

Specific gravity at 60°F............	1·068	Water, per cent.....................	7·5
Sulphur, per cent..................	1·1	Colour......	Black
Viscosity, seconds:		Calorific val	
Saybolt Univ. at 100°F.........	356	B.T.U. p pound..............	15,50
" " 130°F.........	127	" " Imp. gal............	165,500
" " 210°F.........	45		

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[1] Strong, R. A., and Burrough, E. J.: Invest. of Fuels and Fuel Test. 1929; Mines Branch, Dept. of Mines, Canada (1932).

Distillation, Hempel Method
(Tar dehydrated to 0·6% water)

Temperature °C.	Per cent cut	Cum. per cent	Sp. gr. cut	Viscosity Say. Univ. at		Cloud test °F.
				100°F.	212°F.	
Air distillation: Barometer, 765 mm. First drop, 77°C. (171°F.)						
Up to 100	0·5	0·5	0·846	....	....	....
100—125	0·2	0·7				
125—150	0·0	0·7	....	....	....	....
150—175	1·6	2·3	0·900	....	....	....
175—200	7·2	9·5	0·962	....	....	....
200—225	11·5	21·0	0·978	....	....	....
225—250	7·5	28·5	0·979	....	....	....
250—275	9·7	38·2	0·990	....	....	....
Vacuum distillation at 40 mm.						
Up to 200	3·8	3·8	1·006	51	33	20
200—225	6·0	9·8	1·029	83	35	45
225—250	8·3	18·1	1·050	225	39	65
250—275	7·5	25·6	1·083	1,520	53	90
275—300	9·9	35·5	1·107	16,370	115	100

Total distillate, 73·7%. Residuum, 25·0%. Distillation loss, 1·3%.
Carbon residue of residuum, 41·1%. Carbon residue of crude, 11·7%.

Since the A.S.T.M. Engler distillation was to be used in the examination of the products of both cracking and hydrogenation experiments, an Engler distillation of the dehydrated tar was made for the purpose of comparison. In it the yields up to 410°F., and between 410° and 572°F. were as follows:

—	Volume, per cent	Specific gravity
Fraction up to 410°F. (210°C.)	4	....
410° to 572°F. (300°C.)	35	0·991

It is to be noted that the fraction up to 410°F. is much smaller as determined by the Engler than by the Hempel distillation. This is probably due to the presence of the cresols which have boiling points about 200°C. (392°F.), and which would be included in the lower boiling fraction in the Hempel distillation where there is good rectification.

In the chemical examination of the tar, the proportion of acids in the whole tar was approximately determined, and the proportions of acids, bases, olefines, aromatics, and naphthenes plus paraffins determined more accurately in a fraction distilling up to 300°C. in the Hempel apparatus. The results of the first determination are as follows:

—	Weight, grammes	Per cent by weight
Undehydrated tar treated	348	100
10% NaOH used for extraction	659	....
Recovered neutral oil	205	58·9
Recovered tar acids	115	33·05
Water in tar	26·1	7·5
Total recovery	346·1	99·45
Loss	1·9	0·55
		100·00

In the more complete determination the fractions for analysis were obtained by distilling a large sample up to a temperature of 350° C., separating the water from the distillate and redistilling in the Hempel apparatus to obtain fractions boiling up to 410°F. and from 410° to 572°F. These were analysed separately according to the method of Kester and Pohle.[1] The results are tabulated as follows:

—	Fraction up to 410°F.	Fraction 410° to 572°F.	Combined fraction up to 572°F. (calculated)
Acids	48	31	36·9
Bases	2	4	3·3
Olefines	20	7	11·6
Aromatics	20	50	39·5
Paraffins and naphthenes	10	8	8·7
Total	100	100	100·0

The tar used in all of the experiments described in Part VI was dehydrated. Studies of the constitution and properties of low-temperature tars have been published by G. T. Morgan and collaborators[2] and by the British Fuel Research Board.[3] Both investigations have dealt more thoroughly with the chemical constitution of the tars than was considered necessary in the present work.

CRACKING EXPERIMENTS

It has been suggested that cracking as now applied in the petroleum industry might be used to produce motor fuel from low-temperature tar.[4, 5] Cracking is a simpler process than hydrogenation and in choosing between them it would be necessary to balance the smaller cost of the former against the greater yield of the latter. It was decided, therefore, to test the coal tar by the Cross experimental procedure in order to obtain results which could be compared with those of the hydrogenation experiments.

The apparatus used in the cracking tests[6, 7] consists of a strong, steel cylinder of 1·5 litres capacity. It is equipped with a pressure gauge, a gas outlet valve and a thermometer well, and is heated by gas burners. The procedure is to introduce 500 c.c. of the oil to be tested and heat it until the pressure has reached 810 pounds per square inch at 400°C. after which the cylinder is cooled to room temperature and the product removed for analysis. The time of heating should be between 55 and 70 minutes.

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[1] Kester and Pohle: Ind. Eng. Chem.; Anal. Ed., vol. 3, 1931, p. 294. *See also* Composition of the Fractions of Primary and High-temperature Tar, U.S. Bureau of Mines, Rept. of Invest. 3197.

[2] Morgan, G. T.: Jour. Soc. Chem. Ind., 51, 67T (1932).

[3] A Study of the Tars and Oils Produced from Coal, Fuel Research (British) Technical Paper 32, (1931).

[4] Egloff, G., and Morrell, J.: First International Conference on Bituminous Coal, p. 788 (1927).

[5] Dunstan, A. E.: Second International Conference on Bituminous Coal, p. 210 (1929).

[6] Warren, T. E.: Hydrogenation and Pressure-Cracking Experiments on Alberta Bitumen for the Production of Motor Fuel, Invest. of Fuels and Fuel Test., 1930-31, Mines Branch, Dept. of Mines, Canada (1932).

[7] Cross, Roy: Handbook of Petroleum, Asphalt and Natural Gas, 1928, Bull. No. 25, Kansas City Testing Laboratory, p. 664.

The results of two cracking experiments on dehydrated tar are given below:

—	Weight, per cent of tar charged	
	Experiment 3	Experiment 5
Crude gasoline boiling up to 410°F	7·0	8·9
Specific gravity of fraction up to 410°F	0·880	0·907
Crude kerosene boiling between 410° and 572°F	17·6	17·2
Specific gravity of fraction 410° to 572°F	0·993	0·991
Yield of water	1·3	1·3
Loss as gas	....	4·1
Loss as coke	....	23·1

Extraction of the materials soluble in petroleum ether from the coke of Experiment 5 reduced its weight 24·8 per cent. Assuming that the reduction represented oil retained in the coke, the yields of crude gasoline and kerosene in Experiment 5 would be raised to 9·3 and 18·5 per cent respectively. No chemical analysis of the product was made, but as the yield of water was relatively small it follows that the tar acids could not have been greatly reduced. The high specific gravity of the low-boiling fraction and the fact that the phenols in general are resistant to heat treatment[1] corroborate this conclusion.

HYDROGENATION EXPERIMENTS

The scope of the program of hydrogenation experiments of this report was purposely limited for the following reasons. Data of general application on some of the variables had already been obtained in experiments on another raw material[2] and repetition was unnecessary. The amount of product available in the small-scale, batch experiments was not sufficient for complete chemical analysis or engine tests. The properties of the product would be changed to some extent when the process was carried out continuously. Accordingly, the chemical characteristics of the liquid products were not determined and no study was made of the removal of non-hydrocarbon constituents from the charge. As formation of coke is a hindrance to continuous operation it would be necessary to eliminate it in a continuous process. For this reason the primary aim of the experiments was to find a catalyst and set of conditions in which good yields of light oil could be obtained without the attendant formation of coke.

Method and Apparatus

The apparatus used in these experiments has been described in a previous report[3] and will be only briefly outlined herein. It consists of a vertical autoclave of 920 c.c. capacity, heated electrically, and equipped with a stirring mechanism. Temperature is measured by a thermocouple

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[1] Hurd, C. D.: The Pyrolysis of Carbon Compounds (1929), p. 188.

[2] Warren, T. E.: Hydrogenation and Pressure-Cracking Experiments on Alberta Bitumen for the Production of Motor Fuel, Invest. Fuels and Fuel Test., 1930-31, Mines Branch, Dept. of Mines, Canada (1932).

[3] Warren, T. E.: Op. cit.

in a well, the end of which is about two inches from the bottom of the autoclave. Pressure is measured by a Bourdon gauge on the inlet line. The Bourdon gauge was calibrated against a piston gauge. A hydraulic system is used to compress the hydrogen from the shipping cylinders to the desired pressure.

The procedure which was nearly the same as that described in the previous report, was as follows: About 200 grammes of the charge was weighed into the autoclave, the catalyst added, and the autoclave and accessories assembled. Hydrogen was then passed in from the compression system until the required pressure was reached. After inspection for leaks the heat was turned on and the charge heated as rapidly as possible to the desired reaction temperature, maintained at it for the duration of the experiment, and cooled rapidly with a compressed-air blast. The duration was 30 minutes in all of the experiments except No. 81, in which it was 90 minutes. After the apparatus had cooled to room temperature the gas was released through a meter and analysed. The liquid product was weighed out of the autoclave and divided into "gasoline," "kerosene," and "heavy oil" fractions by distillation in an Engler apparatus. The solid remaining in the autoclave, consisting of the catalyst, coke and absorbed oil, was weighed and extracted with benzol in a Soxhlet apparatus to determine the amount of coke.

Results

To facilitate the discussion of results definitions of certain terms to be used are given below.

The *gasoline fraction* is the part of the product recovered as distillate up to 410°F. in an Engler distillation.

The *kerosene fraction* is the part of the product recovered as distillate between 410° and 572°F. in an Engler distillation.

The *coke* is the part of the solid product remaining after extraction with benzol and deduction of the weight of the catalyst.

The *temperature*, used in the sense of *temperature of experiment*, is the average temperature between the heating and cooling periods, i.e. over the duration. The point of measurement is near the centre of the charge.

The *initial pressure* is the pressure indicated before heating is begun by a Bourdon gauge connected to the autoclave. The Bourdon gauge was calibrated against a piston gauge.

The *reaction pressure* is the pressure indicated at the beginning of the reaction period of constant temperature.

The *pressure change* is the difference in pressure between the beginning and end of the reaction period. A pressure increase is a positive, a decrease is a negative, pressure change.

The *duration* is the length of time between the heating and cooling periods.

The *hydrogen used* is the hydrogen not recovered after an experiment, based on the volume and pressure of hydrogen before the experiment and the volume, pressure, and analysis of the gas after the experiment. It ncludes the hydrogen reacting with the charge to form either liquid or gas and also any loss of hydrogen occurring after the heating has begun.

The *total oil* is the amount of liquid recovered after an experiment including that extracted from the coke.

The *methane* and *ethane* are the amounts of these gases found in the total gas removed from the reaction chamber after cooling.

Catalysts

Most of the present work was devoted to an investigation of the effectiveness of various catalytic materials. The catalysts tested may be divided into three groups, namely: iron oxides, metallic copper, and molybdenum compounds.

Iron Catalysts: Although iron oxide catalysts had been recommended in the earlier literature on hydrogenation, it had been found in previous work[1] that ferric oxide (Fe_2O_3) had the effect of increasing carbon formation from bitumen under hydrogenating conditions. On the other hand ferric oxide which had been partly reduced with a high pressure of hydrogen was effective in eliminating carbon formation. In the present work it was thought advisable to try three types of iron oxide, namely: ferric oxide (Fe_2O_3), magnetic oxide (Fe_3O_4), and a mixture of the two containing about 46 per cent of the magnetic oxide. The mixture was produced by reducing ferric oxide at about 300°C. and 2,300 pounds per square inch pressure of hydrogen. The magnetic oxide was of natural occurrence. The ferric oxide and the product of reduction were about 200-mesh size, and the magnetic oxide about 100 mesh. They were not held on a carrier but added to the charge in powder form. The quantity used was 10 grammes, or nearly 5 per cent of the weight of the charge. The results of the work with the iron catalysts are tabulated below. In general, the effect of the iron catalysts was small. Only the natural magnetic oxide reduced the formation of coke and it did so to an extent which may be considered within the limit of error of the tests. The beneficial effect of partial reduction observed in the previous work with bitumen is entirely absent. The hydrogen used amounted to only one- to two-tenths of one per cent of the weight of the charge, and with the mixed catalyst was even slightly negative, i.e. some hydrogen was given off by the tar. This amount, however, is also within the limit of error.

Experiment No.	Catalyst	Temp. °C.	Pressure (atm.)			Per cent by weight of charge					
			Initial	Reaction	Change	Gasoline fraction	Kerosene fraction	Coke	Total oil	Methane + ethane	Hydrogen used
69	None................	450	76·8	163	21	12·3	19·6	13·0	71·3	4·4	0·127
70	Fe_2O_3................	449	76·8	157	29	13·4	17·4	19·1	68·5	4·5	0·204
73	$Fe_2O_3 + Fe_3O_4$......	453	70·8	159	30	13·2	17·4	14·5	73·9	6·7	−0·034
74	Fe_3O_4...............	453	76·8	163	25	13·4	19·6	12·3	75·0	No analysis of gas	

Copper Catalysts: Copper was first tried as a catalyst because it had been concluded from thermal data that sulphur was more easily removed from copper sulphide than from the sulphides of most other metals by the

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[1] Warren, T. E.: Op. cit.

action of hydrogen. It was thought, therefore, that copper might catalyse the removal of sulphur as hydrogen sulphide by the mechanism of first uniting with the sulphur of the tar and subsequently giving it up as hydrogen sulphide. Since copper oxide is easily reduced by hydrogen it was thought that oxygen might be removed from the tar by a similar mechanism. It was found experimentally that sulphur was removed from copper sulphide to an appreciable extent under the temperature and pressure conditions of hydrogenation, but that it was not removed from iron sulphide under the same conditions. It was further thought that since copper formed such easily reducible compounds it would be in the metallic state under hydrogenating conditions which might make it more effective than the oxide or sulphide catalysts.

Metallic copper was used as a catalyst in two forms. One of these was a wool of fine turnings about one millimetre in width, 0·1 millimetre in thickness, and several centimetres long. The other was fine (about 200 mesh) powder formed by the reduction of copper oxide at 316°C. with hydrogen at atmospheric pressure. Nine per cent of the former was used and only 5 per cent of the latter because it presented more surface per unit of weight.

The results of the runs with the two copper catalysts are tabulated below, together with the run in which no catalyst was used.

Experiment No.	Catalyst	Temp. °C.	Pressure (atm.)			Per cent by weight of charge					
			Initial	Reaction	Change	Gasoline fraction	Kerosene fraction	Coke	Total oil	Methane + ethane	Hydrogen used
69	None..............	450	76·8	163	21	12·3	19·6	13·0	71·3	4·4	0·127
75	Copper turnings......	453	80·2	140	15	14·2	21·3	6·3	82·9	No analysis	
77	Copper powder......	454	76·8	153	31	12·2	18·5	17·1	70·9	7·6	0·164

It is to be noticed that in the run with the catalyst of copper turnings there was a higher yield of gasoline and kerosene fractions and a lower yield of coke than in the run without catalysts or in any of the runs with iron oxide. The copper powder, on the other hand, gave very poor results. The difference in behaviour of the two forms of copper would be accounted for if the copper powder had sunk to the bottom of the charge, leaving it without a catalyst, and also causing local overheating in the bottom of the autoclave. Another explanation is that the effect of the copper turnings in reducing coke formation was due to the conduction of heat away from points of local overheating and to its auxiliary stirring action. It is not certain, therefore, that copper has any true catalytic activity and since molybdic oxide proved to be a superior catalyst the point has not been further studied.

Molybdenum Catalysts: In the recent literature oxides and sulphides of molybdenum have been frequently mentioned as good catalysts for hydrogenation of many different materials. Three forms were used in the present work, namely: molybdic oxide (MoO_3), molybdenite (MoS_2), and a mixture of equal parts of molybdic oxide and sulphur. The last-mentioned has been used by the British Fuel Research Board in some of their work on tar hydrogenation. All three were used without a carrier in the finely powdered form (about 200 mesh). The quantity of the oxide and sulphide was 10 per cent and that of the mixed catalyst 5 per cent of the weight of the charge.

A comparison of the effectiveness of molybdic oxide and copper turnings is shown by the following table. The same amount of copper turnings as of molybdic oxide was used (10 per cent).

Experiment No.	Catalyst	Temp. °C.	Pressure (atm.)			Per cent by weight of charge					
			Initial	Reaction	Change	Gasoline fraction	Kerosene fraction	Coke	Total oil	Methane + ethane	Hydrogen used
80	Copper turnings......	438	83·6	182	−13	15·3	32·0	4·2	94·7	2·7	0·226
82	MoO_3...............	437	76·8	146	− 3	14·0	28·0	0·8	92·7	3·0	0·605
84	Copper turnings......	465	143·0	255	− 9	17·1	27·3	1·9	88·4	6·6	1·43
86	MoO_3...............	476	141·7	252	−14	17·8	29·0	0·6	90·4	6·7	1·73

Comparing Experiment 80 with 82, and Experiment 84 with 86, it is to be noted in each case, that, although an advantage is given the copper (due to addition of hydrogen in Experiment 80 and the lower temperature in 84), the molybdic oxide did not allow the formation of as much coke. There was also a slightly greater absorption of hydrogen in runs 82 and 86 than in 80 and 84 respectively. Since the effect of the molybdic oxide cannot be attributed to improved heat transfer or stirring, it follows that it is the better catalyst.

The mixture of molybdic oxide and sulphur and the molybdenite were used in only one experiment each. The results are given below together with that of Experiment 75 which was run under similar conditions with a catalyst of copper turnings.

Experiment No.	Catalyst	Temp. °C.	Pressure (atm.)			Per cent by weight of charge					
			Initial	Reaction	Change	Gasoline fraction	Kerosene fraction	Coke	Total oil	Methane + ethane	Hydrogen used
75	Copper turnings......	453	80·2	140	+15	14·2	21·3	6·3	82·9	No analysis	
91	$MoO_3 + S$...........	448	73·9	146	+36	14·0	17·1	15·2	70·1	9·4	0·83
101	MoS_2...............	450	91·8	171	+10	15·9	24·5	3·8	85·6	5·0	0·98

The molybdenite catalyst appears to be fairly effective but the mixture of molybdic oxide and sulphur gave results comparable with those of the run in which no catalyst was used. However, as the addition of sulphur to molybdic oxide has been found to increase its activity[1] in hydrogenating coal tars it would be advisable to test it further before concluding that it is not applicable in the present case.

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[1] Morgan G. T., and Veryard, J. T.: Hydrogenation of Low-temperature Tar Products, Jour. Soc. Chem. Ind., vol. 51, No. 9, 80T (1932).

Hydrogenation of High-Boiling Product

After several experiments had been completed it was noticed that although there had been a large variation in experimental conditions and correspondingly in coke formation, the quantity of liquid product boiling above 572°F. varied within comparatively small limits. It was usually not less than 40 nor more than 50 per cent of the charge. Accordingly, some experiments were made in which it was attempted to convert this high-boiling oil to more volatile fractions. The special conditions of these runs were high temperature, long duration (Experiment 81), or the removal and separate treatment of the high-boiling fractions. The results of these runs are as follows:—

Experiment No.	Catalyst	Temp. °C.	Pressure (atm.)			Per cent by weight of charge						
			Initial	Reaction	Change	Gasoline fraction	Kerosene fraction	Coke	Total oil	Methane + ethane	Hydrogen used	Fraction over 572 °F.
81 (90 mins.)	Copper, 20 grms...	438	100·0	169	−24	20·2	28·6	2·3	90·6	4·7	1·29	41·8
85	Copper, 20 grms...	505	149·7	285	+13	16·7	17·8	21·3	60·4	8·5	2·01	25·9
89	MoO_3, 20 grms...	500	169·5	315	− 2	13·5	18·4	21·0	60·9	9·7	2·57	29·0
87	Copper, 20 grms...	467	143·5	268	− 6	....	....	......	......	10·6	1·68	....

In Experiment 81 the duration was 90 minutes, or three times that of the other runs. In it, although there were good yields of gasoline and kerosene and a low production of coke, the fraction above 572°F. was not greatly reduced. In Experiments 85 and 89, using copper and molybdic oxide respectively as catalysts, the temperature was very high and the fraction over 572°F. was appreciably reduced, but although a pressure of reaction of about 300 atmospheres was used it was not enough to suppress coke formation. In Experiment 87 the fractions boiling above 572°F. from previous experiments with the copper catalyst were united and hydrogenated again with the copper catalyst. The resulting product closely resembled the charge and could not easily be separated from the coke and catalyst because of its high viscosity. It was apparent, however, that very little light oil, if any, had been formed.

Hydrogenation of Neutral Oil

Two experiments were run on the neutral oil remaining after extraction of the tar with sodium hydroxide solution. It was thought that the neutral oil might be especially amenable to hydrogenation or that the value of the extracted phenols might justify the expense of their removal in commercial practice. The results of these experiments are given below.

Experiment No.	Catalyst	Temp. °C.	Pressure (atm.)			Per cent by weight of charge					
			Initial	Reaction	Change	Gasoline fraction	Kerosene fraction	Coke	Total oil	Methane + ethane	Hydrogen used
83	Copper, 20 grms.....	433	76·8	142	− 8	12·0	30·5	0·6	96·3	1·8	0·21
88	MoO_3, 20 grms.......	473	143·5	266	−14	11·7	26·0	0·6	88·0	8·6	1·65

It is remarkable that of these two runs the better result was obtained with the copper catalyst and at the lower temperature. In both cases the gasoline fraction was small, and in neither was there a tendency for carbon to form.

COMPARISON OF RESULTS BY HYDROGENATION AND CRACKING

Although the experiments reported herein on hydrogenation and cracking are all discontinuous and on a very small scale, some discussion of the comparative merits of the processes may be of value. The following table gives a comparison of the best results of the cracking and hydrogenation tests.

—	Weight per cent of charge	
	Cracking	Hydrogenation
Total oil recovery	78·5	90·4
Gasoline fraction	9·3	17·8
Kerosene fraction	18·5	29·0
Coke	17·4	0·6

A subject which has not been discussed in this paper is the conversion of phenols to neutral oil by hydrogenation. That this conversion is possible has been shown by work of the British Fuel Research Board.[1] Cracking does not offer this advantage and phenols, due to their gum-forming tendency, would lower the quality of a motor fuel, if present in large amounts.[2]

It seems probable, therefore, that hydrogenation has technical advantages over cracking as a process for the conversion of the tar to motor fuel which would justify its higher processing cost.

SUMMARY

Tar from bituminous coal from the Sydney area, Nova Scotia, produced by the Illingworth low-temperature carbonization process, has been subjected to laboratory-scale discontinuous hydrogenation experiments and cracking tests. In the cracking tests there was comparatively high coke formation and low recovery of oil and of volatile fractions. In the hydrogenation experiments the principal object was to find a catalyst which would suppress coke formation. Iron oxide, copper, molybdic oxide, molybdenum disulphide, and molybdic oxide mixed with sulphur were tested as catalysts. Copper and molybdic oxide were effective in reducing coke formation in the present experiments, but because of the possibility that the effect of copper was due to its physical form, molybdic oxide was considered to be the better catalyst. The best result was obtained using ten per cent of the weight of the charge of molybdic oxide at a temperature of 476°C., a reaction pressure of 252 atmospheres, and 30 minutes' duration. In it the recoveries in weight per cent of the charge were: total oil, 90·4; coke, 0·6; gas and unrecovered loss, 9·0; fraction boiling up to 410°F. (gasoline), 17·8; fraction boiling 410° to 572°F. (kerosene), 29·0. In most of the experiments over a wide range of conditions, a proportion of the total oil amounting to roughly 40 to 50 per cent of the charge had a boiling range above 572°F. The neutral oil left after the removal of the tar acids gave slightly lower yields of the gasoline fraction than the untreated tar under similar conditions of hydrogenation.

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[1] Cawley, C. M.: The Reactions of Phenol with Hydrogen at High Pressures, Fuel, 1932, 11, 217-221.
The Hydrogenation of Cresols and Dihydric Phenols, Fuel, 1933, 12, No. 1, 29-35.

[2] Morgan, G. T.: Jour. Soc. Chem. Ind. 51, 61T (1932).

VII

DESCRIPTION OF AN APPARATUS FOR CONTINUOUS HYDROGENATION AND EXPERIMENTS ON COAL TAR, BITUMEN, AND A SUSPENSION OF POWDERED COAL IN COAL TAR

T. E. Warren and K. W. Bowles

This report comprises a description of an apparatus for continuous hydrogenation and an account of runs in it of three types of charging-stock. The apparatus was built because it was advisable to employ a principle of operation which could not be applied in discontinuous runs and because larger amounts of product were required for more complete tests than had been made previously.

The three materials treated represent three of the general types which may be proposed as raw materials for motor fuel production in Canada, namely: coal tar, bitumen, and coal. The representatives of the first two types, low-temperature tar from Sydney, N.S., coal and Alberta bitumen, are of further interest because they have been the subjects of previous experimental work in the batch autoclave.

The principle of operation of the apparatus is very briefly as follows. The charging-stock is pumped into a heated reaction chamber through which is passed a comparatively rapid stream of hydrogen at high pressure. The hydrogen carries the volatile products of reaction out of the heated zone and they are condensed from the stream to form the major part of the product. The hydrogen is then purified and after making an addition to compensate for losses, recirculated through the charge. A small amount of the charge is removed as liquid carrying with it the constituents (e.g. mineral matter) which cannot be converted to volatile oil or gas. By this procedure the volatile oil, which is the desired product, is removed from the reaction chamber before it can be dissociated, while the less volatile oil is left for further treatment. This principle is at present used by the British Fuel Research Board and also, it is thought, by the large-scale developments of the Standard Oil Company, the I.G. Farbenindustrie, and Imperial Chemical Industries, Limited.

In deciding upon the scale of operations it was necessary to consider not only the amount of product required but also the availability of standard equipment, the cost of hydrogen, and the capacity of the reaction chamber. There was no way of foretelling more than the order of magnitude of the production capacity of a reaction chamber of given size. Further, it was thought that in future developments the reaction chamber would be the part of the equipment to be altered or replaced. Accordingly, the first reaction chamber selected has a total volume of about four litres and the auxiliary equipment a correspondingly larger capacity.

APPARATUS

The detailed description of the apparatus which follows is based on the drawing of the apparatus shown in Figure 3. In it the larger units are drawn to scale but the relative position of the parts is not the same as in the original, and much of the auxiliary equipment (e.g. motors, switches, etc.) has been left out. The apparatus is described under headings which group the various units according to their purpose.

Hydrogen Compressor. The hydrogen used in the present apparatus is purchased compressed at 2,000 pounds per square inch in cylinders, one of which is shown at the extreme left in Figure 3. Three of these may be attached to the inlet manifold through which the hydrogen is passed to a water-sealed, calibrated gas holder of 40 cubic feet capacity, from which it is drawn into a three-stage compressor. The compressor, which is operated by a 5 h.p. motor, may be used at pressures up to 5,000 pounds per square inch, and has a capacity of 4·1 cubic feet of hydrogen per minute at an outlet pressure of 3,000 to 3,500 pounds per square inch. From the compressor the gas is passed to two storage cylinders, the volume of each of which is 0·93 cubic foot. A manifold connects the cylinders with each other, with the compressor, and with the line leading to the reaction chamber. A low-pressure line is also arranged so that exit gas from the compressor may be passed back to the gas holder.

Liquid Feed Pump. The feed tank from which the charge is drawn to the liquid pump is shown in Figure 3 at the right of the high-pressure storage cylinders. The liquid to be charged to the reaction chamber is usually too viscous when cold to be easily pumped. Accordingly the feed tank is steam-jacketed and the lines leading from it to the pump are heated electrically. The feed tank has a capacity of seven litres and the volume above the point of withdrawal is indicated by a float and scale. The charging-stock in the tank is stirred by paddles driven by a belt from the liquid feed pump.

The high-pressure feed pump draws the charging-stock from the feed tank and injects it into a line leading from the high-pressure hydrogen storage cylinders to the reaction chamber. The liquid is prevented from flowing back into the hydrogen storage cylinders by the stream of hydrogen, or, if this stops, by a check valve. The pump is a single-acting one having an easily adjustable stroke by which the rate of output can be controlled from zero to full capacity. The valves are $\frac{3}{8}$-inch balls which lift and close by change of pressure. The pump is driven through a speed-reducing gear by a 2 h.p. motor and works at the rate of 60 strokes per minute, having a maximum capacity of five gallons per hour. The maximum working pressure allowable is 10,000 pounds per square inch. The body of the pump is heated electrically and in pumping very viscous liquids (e.g. Alberta bitumen) it is kept at a temperature somewhat above 100°C. as shown by a thermocouple between the valves.

Reaction Chamber. The reaction chamber shown in the centre of Figure 3 is a short cylinder with rounded bottom of about 4 litres capacity, made entirely of heat-treated chrome nickel (18-8) steel. The cover is attached by a tongue-and-groove joint held by eight studs of $1\frac{1}{4}$-inch diameter. The mixed hydrogen and charging-stock pass by a common inlet line through the cover into the liquid in the chamber. The hydrogen is withdrawn by an outlet line having its opening flush with the head of the chamber. The liquid level is kept constant by withdrawing excess liquid through an uptake standpipe. The volume of the chamber up to this level is 2,800 cubic centimetres. The temperature of the liquid charge is measured by a thermocouple in a well, the end of which is at about the centre of the reaction chamber. The temperature of the incoming charge and that of the outlet vapour are also measured, and a thermocouple screwed into the bottom of the reaction chamber measures the temperature of the outside of the wall at its hottest point. The charge is stirred by a large paddle rotating at about 60 r.p.m. The paddle is driven by a shaft passing through a packing on the cover of the chamber, and turned by a speed-reducing gear belt-driven from a ¼ h.p. motor.

The heater for the reaction chamber is an alundum core wound with chromel wire and surrounded by diatomaceous earth, which is held in a cylindrical, sheet steel box. It stands on a table which can be raised or lowered with a screw jack. The cover of the reaction chamber is supported from above and the heater, either alone or together with the receptacle of the reaction chamber, can be lowered from it. The heater has a maximum power of 7·7 kilowatts and is adjusted by a hand-operated rheostat in series with an ammeter. A compressed air line directed against the bottom of the reaction chamber can be used if quick cooling is desired.

Condenser and Receivers. The vaporized volatile product and hydrogen pass from the top of the reaction chamber to a T containing a frangible disk and thence to the condenser. The condenser is a coil made from a tube of chrome nickel steel, ½-inch outside diameter and 0·23 inch inside diameter and about 16 feet long. It is enclosed in a sheet metal cylinder with a water inlet at the bottom and outlet at the top. A thermocouple in the line between the condenser and the high-pressure receiver indicates the temperature of the liquid and gas leaving the condenser. The high-pressure receiver is a cylinder of about 1·1 litre capacity made from 2·5-inch seamless steel pipe of the double extra strong weight. The inlet line extends into the receiver about six inches from the top. The liquid product collected in the high-pressure receiver is run to the low-pressure receiver through a line made of ¼-inch extra heavy seamless pipe. The low-pressure receiver is a 2-litre glass flask equipped with a safety valve. The dissolved gas released at the low pressure is carried off through a ¾-inch pipe with a check valve to the exit gas manifold.

The heavy liquid product is tapped through the standpipe in the reaction chamber by a ½-inch outside diameter chrome nickel line to the low-pressure receiver which is identical with that for the volatile liquid product. Dissolved gas is led off through the same line as is used by the other low-pressure receiver.

Hydrogen Recirculating Equipment. Two systems of recirculating hydrogen were tried. The first of these, by means of a magnetic pump, was not used in the three runs to be described in detail. The second method, that of passing the hydrogen back through the compressor, was found to be more reliable than the other in its present stage of development.

In recirculating by means of the magnetic pump hydrogen is drawn from the top of the high-pressure receiver into the pump and by means of it, is passed through a line with a check valve to the inlet line of the reaction chamber. Valves on the inlet and outlet lines from the magnetic pump make it possible to shut it out of the system. The pump proper is shown at the right of the condenser in Figure 3. The barrel of the pump was made of bronze and the piston of iron. An air-cooled electro-magnet was

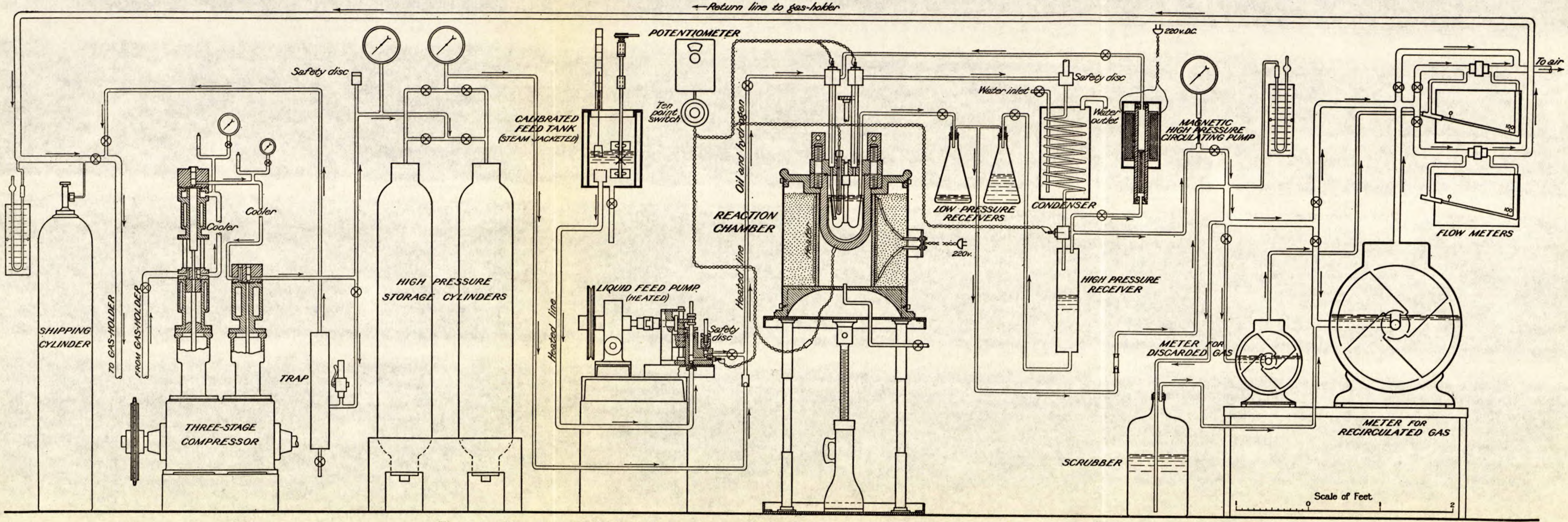
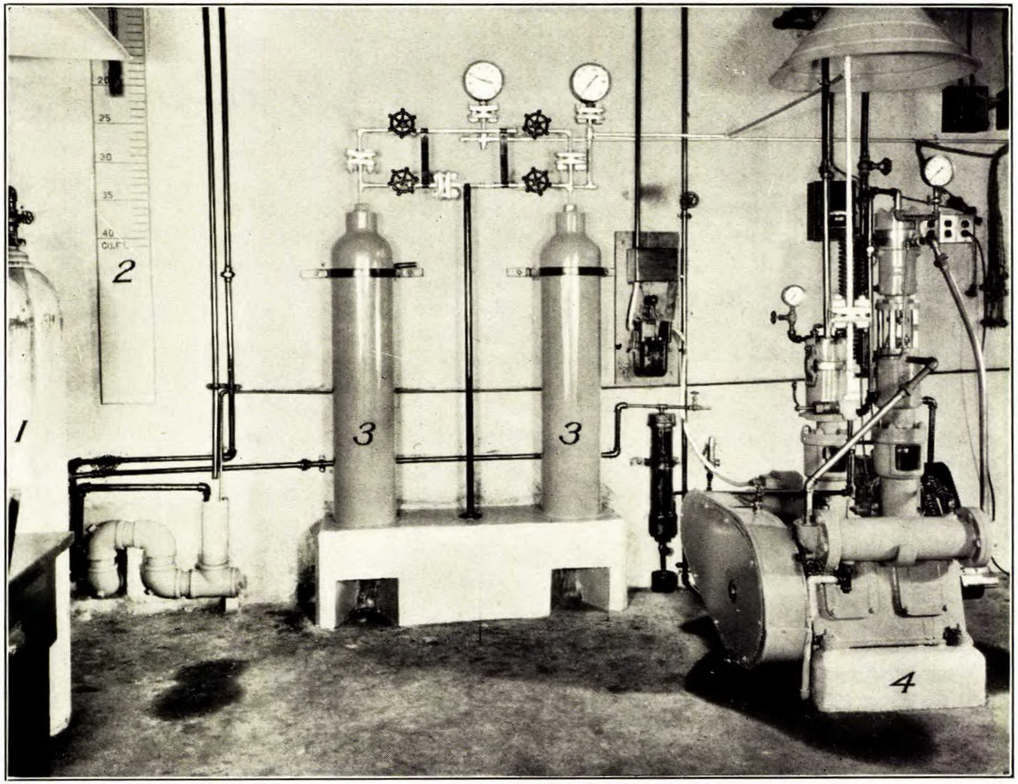
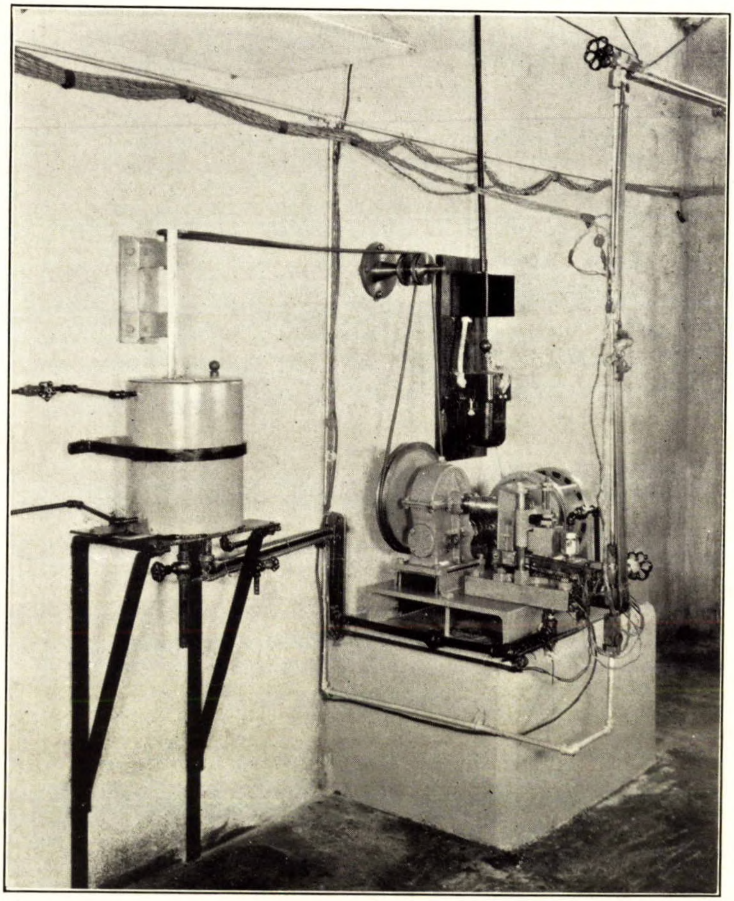


Figure 3. Diagrammatic sketch showing continuous hydrogenation apparatus in use at the Fuel Research Laboratories.

PLATE IV



Hydrogen compression system. 1. Hydrogen shipping cylinders. 2. Scale of gaz holder. 3. High-pressure storage cylinders. 4. Hydrogen compressor.



Liquid feed tank and pump.

mounted on the top of the barrel and by means of it the piston could be drawn to the top, from whence it fell to the bottom by its own weight when the current to the magnet was interrupted. The magnet was controlled by a switch which alternately turned the current on and off. The switch was operated by a variable speed motor so that the rate of the pump could be controlled at will. Operating at 20 strokes per minute at 200 atmospheres pressure it had about three times the maximum capacity of the compressor. There was also a considerable saving in power over the other method since the magnetic pump operated only against the hydrostatic head of the charge in the reaction chamber, while by the other method the gas had to be released to atmospheric pressure and completely recompressed. The magnetic pump, however, was too easily stopped by sticking of the piston or a slight obstruction of the line so that it was used only in the preliminary runs.

In recirculation through the compressor the hydrogen passes from the top of the high-pressure receiver through a valve to the low-pressure manifold, its pressure being released from about 200 atmospheres to one atmosphere. It then passes through a scrubbing system to a wet meter and flow meter and thence to the inlet manifold leading to the gas holder. In the gas holder it is mixed with fresh hydrogen, recompressed and passed back to the reaction chamber.

In either system of recirculation some of the gas coming from the reaction chamber is discarded in order to eliminate methane which accumulates and is not absorbed in the scrubber. The discarded gas is passed from the low-pressure manifold through a small wet meter and flow meter and then out of the building.

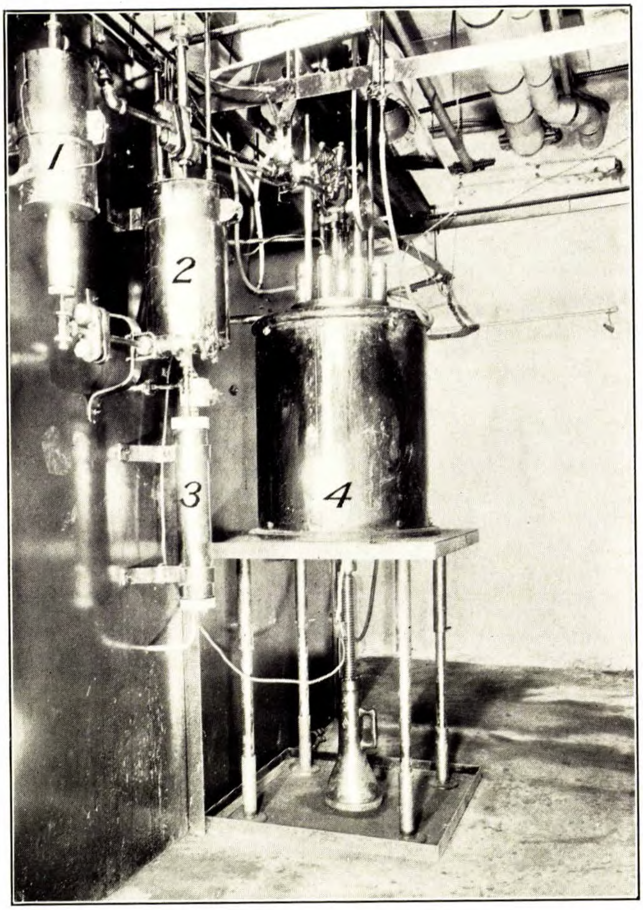
Temperature Measurement. Temperatures at six points on the apparatus are indicated by means of thermocouples, a multiple switch, and a potentiometer. The thermocouples are all iron-constantan, but are constructed differently. That in the feed pump is made by silver soldering a $\frac{3}{16}$-inch iron tube into a $\frac{1}{4}$-inch pipe plug, passing a constantan wire to the tip of the tube and silver soldering it there. The wire is insulated from the tube by a thin-walled capillary of glass. Iron-constantan duplex lead wire connects the iron tube and constantan wire to the potentiometer. The tip of this thermocouple is so placed in the pump that the liquid between the valves passes around it. It was used to find the best temperature at which to pump viscous materials. The thermocouples in the inlet and outlet lines of the reaction chamber are similarly made, excepting that each is held in place by a cone joint with a collar and gland nut. They are considerably longer, however, so that the tips are in the cover of the reaction chamber. The thermocouple in the inlet tube indicates the amount of preheating and that in the exit tube shows the temperature of the exit vapour which is a function both of the rate of volatile oil formation and of hydrogen recirculation. The thermocouple in the well in the reaction chamber is made by joining iron and constantan lead wire. Porcelain tubes are used as insulation for the hot section. This thermocouple measures the temperature of the liquid charge in the reaction chamber. A measurement of the temperature of the outer part of the wall of the chamber at its hottest point is obtained as follows: On the outside of the wall, near the bottom, two holes about $\frac{1}{4}$ inch deep were drilled. They were placed about $\frac{1}{4}$ inch apart and tapped to take 8-gauge wire. Sections of iron and constantan wires about 1 inch long were screwed into these holes and connected by lead wires to the potentiometer. This temperature measurement is necessary to prevent failure of the apparatus due to overheating at high pressures. The sixth thermocouple, placed below the condenser, is made in the same way as the two on the inlet and outlet lines of the reaction chamber. It is used to show if the condenser is operating efficiently.

Pressure Measurement. With the exception of the two gauges on the compressor, the pressure is measured at only three points on the high-pressure system. The first of these is on the line from the compressor to the high-pressure storage cylinder and indicates the pressure of the storage cylinder into which the gas is being passed. The second point is on the line between the storage cylinders and the reaction chamber. The third gauge is on the line leading from the high-pressure receiver to the low-pressure manifold. In operation these last two gauges should read the same since the pressure drop due to flow in the lines is usually negligible. This arrangement keeps the gauges out of contact with the liquid charge or product while permitting the pressure of reaction to be measured. The gauges were calibrated against a piston gauge.

Safety Precautions. Failure of the high-pressure apparatus would be due to incorrect design, flaws in material, too high a pressure, too high a temperature, or a violent reaction (e.g. combustion) taking place in a closed system. There is also the possibility of the failure of a closed piece of low pressure equipment which is connected with the high-pressure system. Together with failure of the apparatus there is the possibility of escape, and subsequent ignition, of hydrogen, vapour, and oil.

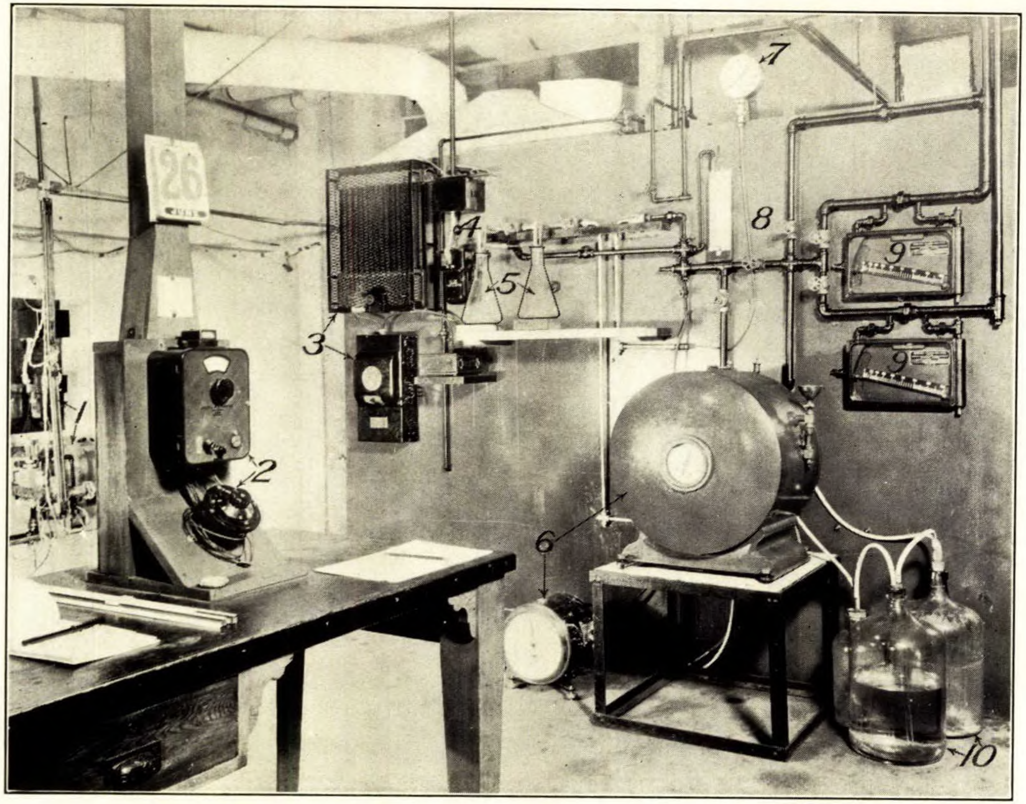
To avoid injury to the apparatus or operators due to any of the conditions enumerated above, the following special precautions have been taken. Each piece of high-pressure equipment was tested with water at a pressure twice as high as will exist under working conditions. The only exceptions to this rule were the compressor and the liquid pump, both of which are protected by safety valves and have only small volumes at high pressure. All parts which were to be subjected to both heat and pressure were made of chrome nickel steel. The reaction chamber, condenser, high-pressure receiver, and magnetic pump, together with most of the heated, high-pressure lines are situated behind a wall of $\frac{1}{4}$-inch sheet steel. Frangible disks which allow the escape of compressed gas at a pressure below the tested strength of the equipment, are situated on the inlet line to the high-pressure storage cylinders, the liquid feed pump, and the condenser. The temperature of the wall of the reaction chamber, as previously mentioned, is measured by a calibrated thermocouple to prevent overheating. Each cylinder of hydrogen before use is tested for purity with a Schilling apparatus to assure that the makers have not inadvertently filled it with oxygen or air. After the hydrogen is passed into the gas holder it is at all times at a pressure higher than atmospheric so that no air can be drawn into the system, except by a fault in the low-pressure line leading to the compressor. Closed low-pressure lines connected through valves to the high-pressure system are protected by safety valves.

An exhaust fan with air ducts over the shipping cylinders, compressor, liquid feed tank, and reaction chamber prevents the accumulation of



View behind the protecting wall showing:
1. Magnetic pump. 2. Condenser. 3. High pressure receiver.
4. Reaction chamber.

PLATE VII



General view outside protecting wall showing: 1. Feed pump and line. 2. Potentiometer. 3. Rheostat, ammeter and switch for reaction chamber heater. 4. Motor switch. 5. Low-pressure receivers. 6. Wet meters. 7. Exit pressure gauge. 8. Low-pressure manifold. 9. Flow meters. 10. Scrubbing bottles.

leaking hydrogen. The motors are of the induction type and are controlled by oil-immersed switches to prevent sparking. All electrical equipment may be turned off by switches located outside the room. As a fire extinguisher a cylinder of liquid carbon dioxide is located outside the room with valves and lines so arranged that either the reaction chamber or the room in general may be sprayed with liquid carbon dioxide by an operator either inside or outside of the room.

PROCEDURE

The general procedure has been outlined in the Introduction and many of the details were given in the description of the apparatus. However, it is necessary to give a more complete account before describing the results of the experiments. A flow-sheet of the process is given in Figure 4.

In preparation for a run the reaction chamber is charged with the catalyst and with about two litres of the material to be hydrogenated. The feed pump is tested, the line from the feed pump to the reaction chamber cleared, and the cover of the reaction chamber put on. The liquid is then completely drained from the liquid feed tank and a fresh quantity of about 6 litres weighed in. By this method the exact weight of liquid charged to the reaction chamber can be determined, since the initial conditions of the liquid feed system—i.e. a full line from the feed tank to feed pump, an empty line thence to the reaction chamber, and an empty feed tank—can be duplicated easily at the end of the run.

Hydrogen is then passed from the storage cylinders—after being tested and compressed to 3,000 pounds per square inch—to the reaction chamber and the heat turned on at the highest permissible amperage. During the heating period, which lasts about four hours, the hydrogen is circulated through the apparatus at the rate of about 5 to 10 cubic feet per hour, measured at atmospheric pressure. Near the end of the heating period the liquid feed pump is run intermittently to fill the reaction chamber to the level of the stand-pipe.

When the desired temperature, about 450°C., has been attained, the product produced during the heating period is removed, weighed, and retained as a separate sample. The rate of recirculation is increased to about 100 cubic feet per hour and the feed pump run thereafter either continuously or intermittently at intervals of about fifteen minutes. The pressure is allowed to fall from about 3,000 to 2,700 pounds per square inch, after which about 10 cubic feet of additional hydrogen is measured into the gas holder from the shipping cylinders and the compressor run until the pressure is again 3,000 pounds per square inch and the gas holder nearly empty. It is necessary to repeat this operation about twice every hour. At the end of each hour the high-pressure receiver is drained and the product removed, weighed, and separately bottled. The level of the liquid in the reaction chamber is tested at intervals of about fifteen minutes by opening the valve from the stand-pipe. As little liquid as possible is removed in this way. About 10 cubic feet per hour of hydrogen is measured, sampled, and passed outdoors. The duration of the three runs described herein is about 8 hours each plus the heating and cooling periods, or about 14 hours in all.

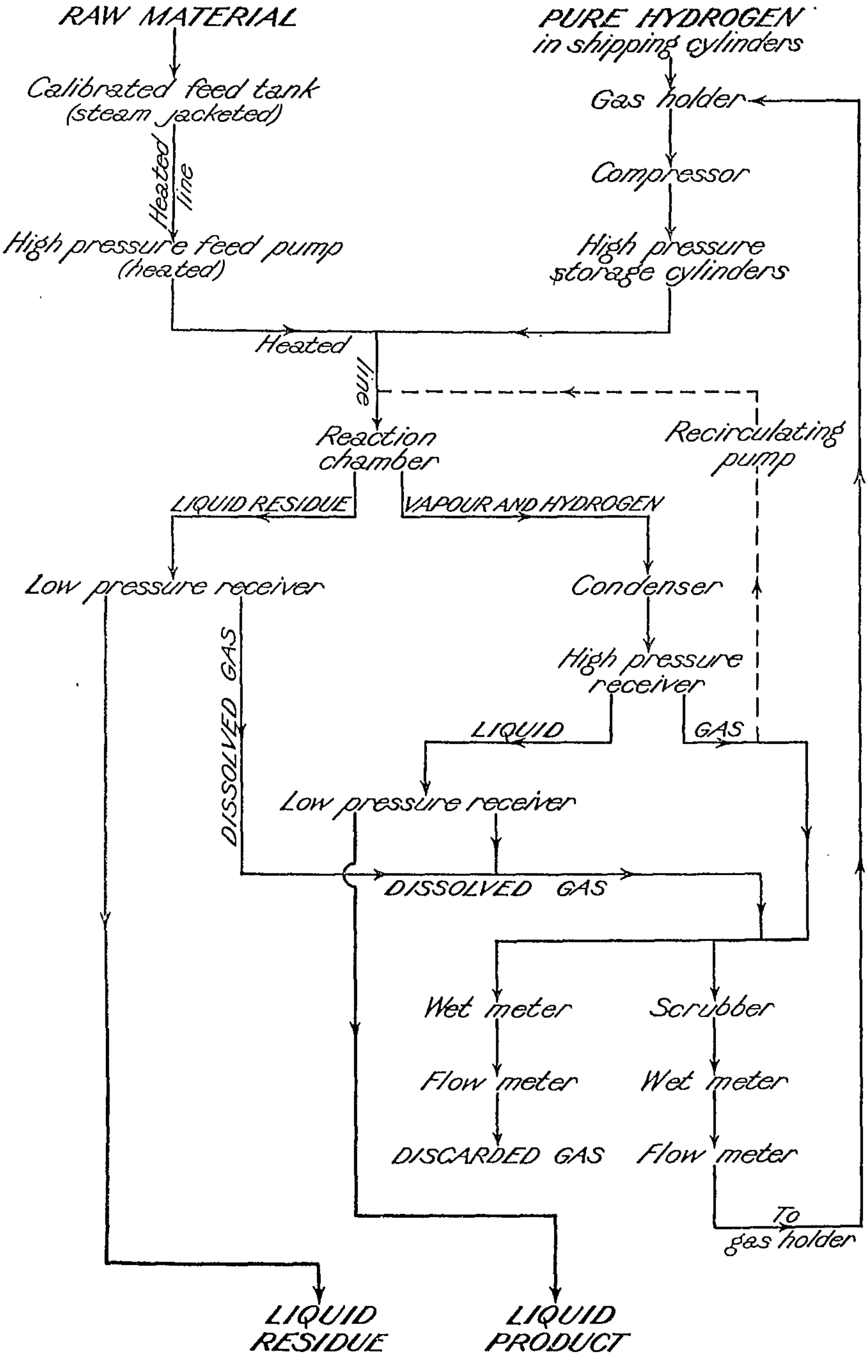


Figure 4. Flow-sheet of hydrogenation process at present in use at the Fuel Research Laboratories.

During the runs observations are recorded every fifteen minutes of the temperature of the charge, temperature of the wall of the bomb, the pressure at the storage cylinders, and at the condenser, the amperage of the heating current, the rate of recirculation, and the rate at which gas is discarded. After running the liquid feed pump the volume in the feed tank is recorded. Each hour the total amount of liquid tapped and the amount recovered in the previous hour is recorded. Each addition of hydrogen to the gas holder from the shipping cylinders is recorded at the time it is made.

When the apparatus has cooled the feed tank is drained, the reaction chamber opened and its contents quantitatively removed.

PREPARATION OF CATALYST

Although different catalysts were tried in the preliminary runs, only one was used in the three experiments to be described in detail. It was prepared in the following manner: One hundred grammes of ammonium molybdate ($(NH_4)_6 Mo_7 O_{24} \cdot 4H_2O$—Merck & Company) was dissolved in 500 cubic centimetres of hot water and 200 grammes of coke of 8- to 10-mesh size added. The solution was stirred and evaporated until solid and then heated several hours at red heat until ammonia could no longer be detected by smell and the colour had changed from white to light green. The amount of catalyst so obtained was about 260 to 280 grammes since there was a considerable loss in handling as well as the loss of ammonia and water of crystallization. An entire batch was used in each experiment.[1] It is about 10 per cent of the weight of the liquid in the reaction chamber during operation.

EXPERIMENTAL RESULTS

Preliminary Tests

Semi-continuous Procedure with Small Autoclave. Before the continuous apparatus was completed, five runs were made in the smaller autoclave using what might be called a semi-continuous procedure. That is, a charge of dehydrated low-temperature coal tar as used in the experiments of Part VI was placed in the autoclave and a current of hydrogen which carried out the volatile oil passed through it. The charge was not renewed so that it grew smaller and changed in composition as the run progressed. These runs were made to obtain data on the quantity of volatile oil carried by the hydrogen, which was to be used in the design of the continuous apparatus. The technique of these experiments was not well developed.

The yield of carbon was large, possibly due to inefficient stirring and high concentration of catalyst which would cause local overheating at the end of the run. However, carbon formation appeared to be greatly influenced by pressure. In the last two of the five runs the pressure was 3,500 pounds per square inch, and in them the solid residue was about 10 per cent of the charge as compared with nearly 45 per cent in the previous runs in which the pressure was about 2,000 pounds per square inch. However, since the catalysts in the four experiments are not strictly comparable, it is

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[1] This catalyst was first prepared on September 10, 1932, and first used on October 12, 1932.

not certain that the difference in results is due entirely to difference in pressure. In the high-pressure runs using a catalyst of 10 per cent of the charge of powdered molybdic oxide, the yields of volatile oil, including water, were 63·1 and 64 per cent by weight of the charge. Of the volatile oil, 84 and 80 per cent respectively boiled up to 410°F. The loss on refining of a composite gasoline fraction from the last two experiments with 80 per cent sulphuric acid was 10 per cent and on subsequent treatment with 10 per cent sodium hydroxide solution, was 12 per cent, making a total refining loss of 22 per cent. The yield of water amounted to about 6 per cent of the charge, which shows that a considerable proportion of the phenolic compounds had been hydrogenated.

Preliminary Tests on Continuous Apparatus. Before attempting the more carefully performed experiments, it was necessary to make a series of preliminary tests, the chief purpose of which was to indicate necessary changes in the apparatus or procedure. Six tests were made in which the apparatus was in operation for a total of about 50 hours. The charging-stock used was the low-temperature coal tar with a water content of 7·5 per cent.

Although analyses were made of the products of each run, the operating conditions of all but the sixth were so irregular that the results are not given herein. Alterations in the equipment as a result of these runs were of a minor nature although considerable difficulty was encountered in preventing leaks in the reaction chamber at the higher temperatures and in keeping the magnetic pump in operation during the required length of time. The principal alteration in procedure made as a result of the preliminary tests was the change of the form of catalyst and its method of introduction into the reaction chamber. Throughout the six preliminary tests the catalyst was finely powdered molybdic oxide which was added to the charging-stock in the liquid feed tank. This procedure was later abandoned because it was impossible to tell how much catalyst was in the reaction chamber during operation.

The second part of Run No. 6 of the preliminary tests is interesting because it is the only one using the magnetic circulating system and the powdered catalyst, in which the temperature and pressure control were sufficiently accurate to permit comparison of the results with those obtained later. This test, including heating and cooling periods, lasted 11 hours, of which 5 hours were at the desired conditions of reaction. The average temperature was 437°C., the average pressure 190 atmospheres, and the rate of recirculation calculated from the displacement of the magnetic pump was about 0·88 cubic foot per hour, which at atmospheric pressure would be 167 cubic feet per hour. The distillate product, including water, was collected during the 5-hour period at the average rate of 442 grammes or 494 cubic centimetres per hour. No coke was formed.

A complete material balance was not possible because a small amount of tar remained in the reaction chamber from a previous run. However, based on the materials removed and estimating the gas, vapour, and handling losses at 10 per cent of the charge, an approximate value may be assigned to the various quantities. Sixty-eight per cent by weight of the charge was recovered as distillate, 16 per cent was left in the reaction chamber at the end of the run, and 6 per cent removed from the levelling stand-pipe during the run.

The distillate product contained 9·9 per cent by volume of water. After separating the water the oil was distilled by both Engler and Hempel methods. The yields were as follows:—

—	Volume, %		
	Up to 410°F.	410° to 572°F.	Above 572°F.
Engler	46·0	48·0	4·0
Hempel	43·3	49·3	5·3

A determination of acids and bases in the dehydrated distillate product gave the following results:—

—	Acids	Bases	Neutral oil	Total
Per cent by volume	16	1	83	100

By comparison with the analysis of the raw tar given in Part VI, it can be seen that the acids have been reduced from about 33 per cent to 16 per cent.

The liquid left in the reaction chamber after the run had the green "bloom," characteristic of some lubricating oils and seemed to have lubricating properties. It was therefore examined by Hempel distillation and measurement of the viscosity and cloud point of the vacuum fractions, which method is commonly applied to crude oils. A summary of the analysis is given below.

Specific gravity at 60°F., 1·133. Water, per cent, none.

Distillation, Hempel Method

Temperature °C.	Per cent cut	Cum. per cent	Sp. Gr. cut	Viscosity Say. Univ. at		Cloud test °F.
				100°F.	212°F.	
Air distillation: Barometer, 765 mm. First drop, 150°C.						
Up to 150	....	....	....	....	....	....
150 to 175	0·2	0·2	0·953	....	....	....
175 to 200	0·7	0·9				
200 to 225	0·9	1·8	0·968	....	....	....
225 to 250	2·1	3·9	0·976	....	....	....
250 to 275	6·1	10·0	1·010	....	....	....
Vacuum distillation at 40 mm.						
Up to 200	1·0	1·0	1·036	52	34	10
200 to 225	15·2	16·2	1·052	63	34	30
225 to 250	20·2	36·4	1·071	111	36	40
250 to 275	16·6	53·0	1·091	447	41	60
275 to 300	11·7	64·7	1·120	3,167	59	75

The oil is considered to be a poor lubricant, chiefly because of the large variation of viscosity with change of temperature.

Experiment on Low-Temperature Coal Tar

Although the coal tar had been used in the preliminary tests and good results had been obtained in Experiment 6, it was decided to make another run because in No. 6 the input had not been measured and the catalyst concentration was not known.

The tar used in Experiment 7 was the same as that used in the preliminary runs. It was not dehydrated and contained 7·5 per cent of water. Its characteristics have been described in Part VI. The catalyst was molybdic oxide supported on coke, the preparation of which from ammonium molybdate has been previously described.

The general procedure which has been described was modified somewhat in Experiment 7. The quantity charged was determined by reading the calibrated scale of the liquid feed tank rather than by weighing as stated in the section on procedure. Hydrogen was recirculated through the compressor but no scrubber was used. Due to mechanical difficulties Experiment 7 was started four times, and since the reaction chamber and liquid feed line were not cleared after each start, it is necessary to include all four in the material balance, although the last is the only one which will be considered in detail. It should be mentioned, however, that during some repairs in the third run the pressure was allowed to fall for a short time to 700 pounds per square inch, while the temperature was at 400°C., which may have caused the formation of a small amount of carbon which was observed after Experiment 7.

The operating conditions of Experiment 7 are as follows: The heating period was of 3½ hours' duration after which the run proper was continued for 10 hours. The average temperature of the liquid charge over this 10-hour period was 427·5° C., the average pressure 183 atmospheres, and the rate of recirculation based on atmospheric pressure and room temperature 100·1 cubic feet per hour.

A summary of the material balance is as follows:—

—	Per cent by weight of charge
Distillate product, including water	62·8
Liquid removed from levelling stand-pipe	9·8
Liquid left in reaction chamber at end of experiment	14·8
Material not recovered, including coke, (2·29%), methane and ethane, vapour, ammonia, hydrogen sulphide and handling losses	12·6

The losses are grouped together because there is some uncertainty in separating the items.

The distillate product from the 10-hour run was light grey and somewhat turbid in appearance, with a small amount of black emulsion which collected as a film between the water and oil layers. It contained 10·9 per cent by weight of water. The distillate collected at the first of the run differed from that collected at the last as is shown by the following Hempel distillations of the dehydrated distillate product.

Fraction	Beginning of run		End of run	
	Volume, per cent	Sp. Gr. at 60°F.	Volume, per cent	Sp. Gr. at 60° F.
Up to 410°F	53·1	0·904	34·2	0·909
410° to 572°F	40·4	0·984	56·3	0·976
Above 572°F	....	....	6·5	....
Residue	3·0	....	2·5	....
Loss	3·5	....	0·5	....

Distillation of a sample composed of all the distillate product gave the following results:—

Fraction	Volume, per cent	Sp. Gr. at 60°F.
Up to 410°F	35·7	0·906
410° to 572°F	55·8	0·975
Above 572°F	5·0	....
Liquid residue	3·5	....

A chemical examination of the dehydrated distillate product using the method of Kester and Pohle referred to in Part VI gave the following results:—

—	Acids	Bases	Olefines and aromatics	Paraffins and naphthenes
Per cent by volume	27	3	53	17

It is to be noted that the acids have not been so much reduced as in preliminary Experiment 6. This point is verified by a balance of water input and recovery which shows 446 grammes of water introduced and 454 grammes removed in Run 4 of Experiment 7.

During the last six hours of the run the yield of distillate per hour decreased progressively from about 500 cubic centimetres to about 250. The temperature was about seven degrees Centigrade lower during the last half of the run than during the first half, but it is not known whether the decrease in yield should be attributed to temperature decrease, accumulation of refractory material in the reaction chamber, or deterioration of the catalyst.

The consumption of hydrogen during the fourth run of Experiment 7 was 6·4 per cent of the tar charged during that period.

Experiment on Alberta Bitumen

An estimate of the cost of producing motor fuel and road-paving material by topping and cracking Alberta bitumen has been published recently.[1] This estimate brings out the fact that in the proposed process the yield of road-paving tar would be greatly in excess of that of motor fuel, so that too much of the former would be produced for the comparatively limited market. Hydrogenation is the only process which could give a high ratio of motor fuel to residuum, and for this reason appears at present to be the most suitable means of converting Alberta bitumen to motor fuel. Accordingly, it was the second material to be tested in the continuous apparatus.

The bitumen used in Experiment 8 was a sample separated from the sand at the Clearwater plant near McMurray, Alberta, in 1930. It is part of sample No. 7295 which was the subject of previous work.[2] The Hempel distillation and analysis are given below.

Characteristics

Specific gravity at 60°F.	1·030	Water, per cent.	0·5
Sulphur, per cent.	5·84	Softening point, °F.	102
Viscosity, seconds:		Colour	Black
Furol at 210°F.	820		
" 250°F.	236		

Distillation, Hempel Method

Temperature °C.	Per cent cut	Cum. per cent	Sp. Gr. cut	Viscosity Say. Univ. at		Cloud test °F.
				100°F.	212°F.	
Air distillation: Barometer, 762 mm.						
Up to 125	....	....	....	....	....	....
125 to 150	0·9	0·9	0·809	....	....	....
150 to 175	0·8	1·7				
175 to 200	1·1	2·8	0·823	....	....	....
200 to 225	1·1	3·9	0·848	....	....	....
225 to 250	4·1	8·0	0·866	....	....	....
250 to 275	11·9	19·9	0·867	....	....	....
Vacuum distillation at 40 mm.						
Up to 200	1·6	1·6	0·878	36	32	below 0
200 to 225	3·2	4·8	0·929	66	36	" 0
225 to 250	6·1	10·9	0·947	118	39	" 0
250 to 275	6·4	17·3	0·958	178	42	" 0
275 to 300	10·6	27·9	0·972	508	52	" 0

Total distillate, 47·8%. Residuum, 49·5%. Distillation loss, 2·7%.
Carbon residue of residuum, 39·6%. Carbon residue of crude, 19·6%.

In Experiment 8 the heating period was three hours, after which the conditions varied somewhat for two hours, so that only the last six hours will be considered as the run proper. During this period the average temperature of the liquid charge was 439°C., the average pressure 184 atmospheres, and the average rate of recirculation 112 cubic feet per hour, measured at atmospheric pressure and room temperature.

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[1] Ells S. C.: Estimated Cost of Producing Solid and Liquid Hydrocarbons from Bituminous Sand; Invest. of Min. Res. and the Mining Ind., 1931. Mines Branch, Dept. of Mines, Canada (1932).

[2] Warren T. E.: Hydrogenation and Pressure-Cracking Experiments on Alberta Bitumen for the Production of Motor Fuel; Invest. of Fuels and Fuel Test., 1930-31. Mines Branch, Dept. of Mines, Canada (1932).

A summary of the material balance for the whole experiment is as follows:—

—	Weight per cent of charge
Distillate product, including water	60·5
Liquid removed from levelling stand-pipe	6·4
Liquid left in reaction chamber at end of experiment	21·6
Sulphur recovered in scrubber	2·9
Methane and ethane in gas	2·8
Vapour loss, handling loss and unrecovered impurities	5·8

No coke was formed and the consumption of hydrogen was 5·7 per cent of the weight of the total charge.

The liquid product collected throughout the run contained 2·2 per cent by volume of water, which amounted to 1·6 per cent by weight of the charge. The charging-stock contained only 0·5 per cent of water so that 1·1 per cent must have been formed from oxygen in the bitumen. The product was light grey in colour and was turbid, probably due to the presence of water. In order to note if there had been a change in composition of the distillate product during the six-hour period, the product was divided into two parts. The compositions of these parts from the first and second half of the run respectively are shown by the following results of distillation of the dehydrated distillate product in the Hempel apparatus.

Fraction	First half of run		Second half of run	
	Volume per cent	Sp. Gr. at 60°F.	Volume per cent	Sp. Gr. at 60°F.
Up to 410°F.	48·7	0·755	45·0	0·755
410° to 572°F.	39·7	0·881	41·4	0·885
Above 572°F.	8·3	0·930	11·3	0·938
Liquid residue	2·0	....	2·0	....
Loss	1·3	....	0·3	....

A composite sample of the distillate product from both halves of the run was distilled in a 3-litre flask and the following yield obtained.

Fraction	Volume, per cent
Up to 410°F.	47·3
410° to 572°F.	42·6
Liquid residue	9·5
Distillation loss	0·6

The fraction up to 410°F. from this distillation was submitted without further treatment to examination as a gasoline. The end point was found to be 469°F., probably due to entrainment in distillation. The gum formation was 105 milligrams per hundred cubic centimetres, as determined in a glass dish without an air jet. The colour was "yellow, transparent", and the odour "poor." The sulphur content was 0·6 per cent. The octane number of a redistilled sample with an end point of 434°F., determined in a Series 30B Ethyl Gasoline Corporation engine at 345°F. and 900 r.p.m., was 53. It is apparent, therefore, that refining would be necessary to produce a marketable gasoline from the distillate product.

The results of a chemical analysis of the dehydrated distillate product are given in the following table.

—	Volume, per cent			
	First half of run		Second half of run	
	Fraction up to 410°F.	Fraction 410°-572° F.	Fraction up to 410°F.	Fraction 410°-572°F.
Acids	0	0	0	0
Bases	1	1	1	1
Olefines	12	11	10	10
Aromatics	18	35	18	36
Paraffins and naphthenes	69	53	71	53

It is to be noticed that there are no acids in the product and that in spite of the hydrogenating conditions, 10 to 12 per cent of olefines are present.

As the experiment progressed the yield of distillate did not decrease to so great an extent as it had in Experiment 7. The rate of production of distillate product was increased by high temperature over the very limited range of variation in the experiment. The yield did not seem to be greatly influenced by change of pressure or rate of recirculation. The average rate of production throughout the six-hour period was 804 cubic centimetres per hour.

Experiment on a Paste of Powdered Coal and Low-Temperature Coal Tar

Interest in the direct hydrogenation of coal to motor fuel has been greatly increased since 1931 by statements of Imperial Chemical Industries, Limited. This company operated an experimental plant with a production capacity of about ten tons per day of motor fuel. The data obtained with it were used to estimate that in a commercial-scale plant, gasoline of good quality could be produced from bituminous coal at a total cost of 14 cents per gallon.[1] The published descriptions of the process are incomplete but it seems to be the same in principle as that described in this paper. The cost estimate has met with some criticism[2] and it must be remembered that it is the statement of a commercially interested organization. Results rather than operating conditions have been published and such important details as the composition and manner of use of the catalyst have not been given. Therefore, the experiment to be described had the double purpose of investigating the applicability of the present procedure to coal, and of making a preliminary test on a prospective Canadian raw material. The coal selected for the purpose was washed slack from the Princess colliery of the Dominion Steel and Coal Corporation in Cape Breton. The analysis and characteristics are as follows:

Proximate analysis (as received):	%	*Ultimate analysis:*	%
Moisture	7·0	Carbon	76·02
Ash	3·1	Hydrogen	5·76
Volatile matter	36·3	Ash	3·08
Fixed carbon	53·6	Sulphur	1·17
Calorific value:		Nitrogen	1·61
B.T.U. per pound	13,698	Oxygen	12·36

Coking properties—Good

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[1] Imperial Chemical Industries, Limited, Fuel, X, 481 (1931).

[2] Lander, C. H.: Proc. Roy. Inst., Great Britain, Nov. 20, 1931 (advance copy). Nature, 1932, 129, 640-641, 676-679.

It is necessary to mix powdered coal with oil in order to pump it into the apparatus. In continuous operation the oil might be a high-boiling fraction of the product previously produced. However, in beginning a run or in a short run, such as Experiment 10, some other oil must be used. In the present work the low-temperature tar from Sydney coal, which has been previously described, was selected for this purpose. This tar had the disadvantage of high viscosity so that if more than 40 per cent of coal were incorporated in it, there was difficulty in pumping the mixture. However, it was selected because its behaviour under hydrogenating conditions was better known than that of other available liquids. The coal was powdered in a ball mill until all of it passed a 100-mesh screen, although most of it was much finer. The coal tar was also filtered through a 100-mesh screen and the two mixed in proportions of 40 per cent of coal to 60 per cent of tar. The coal settled out only a little after standing without agitation for several weeks.

The procedure previously used was modified only slightly. A charcoal scrubber was placed in the gas recirculating system. The first trial on the paste (Experiment 9) failed because the mixture of coal and tar placed initially in the reaction chamber frothed badly and obstructed the condenser. For this reason Experiment 10 was begun with a charge of dehydrated tar in the reaction chamber. The tar-coal mixture was weighed into the feed tank.

The heating period in Experiment 10 was about six hours, and the run proper lasted seven hours. The average temperature during this period was 451·7°C., the average pressure 187 atmospheres, and the average rate of recirculation 87 cubic feet per hour at atmospheric temperature and pressure.

A summary of the material balance for the whole experiment is as follows:—

Input

—	Per cent
Dehydrated tar initially charged	23·5
Mixture coal	30·6
Mixture tar	45·9
Catalyst 4·0	

Output

—	Per cent
Distillate, including water	59·1
Material in absorbent charcoal	2·1
Liquid from levelling stand-pipe	0·7
Liquid left in reaction chamber	26·0
Methane and ethane	9·2
Unrecovered	2·9
Total	100·0

The distillate product was light brown and turbid with a black film between the aqueous and oil layers as in the run of coal tar alone. No adherent coke was formed although the oil left in the reaction chamber was much more viscous than in previous experiments. The hydrogen consumption based on the hydrogen added and the difference in uncombined hydrogen in the system before and after the experiment, was 7·2 per cent.

The liquid product contained 9·7 per cent by volume of aqueous liquor, which amount is in excess of the water introduced by about 58 cubic centimetres. The aqueous liquor contained 20·2 grammes of ammonia and 10 grammes of sulphur. The specific gravity of the product from the first two hours of the run was somewhat lower than that of the last five hours, probably because a fairly large proportion of the dehydrated tar initially introduced was still present in the reaction chamber. For this reason the distillation and chemical analyses were made on the product from the last five hours of the run. To note the change in product as the run progressed, separate distillations were made of the dehydrated distillate product from the first and last hours of the five-hour period. The results are given below.

—	First hour		Last hour	
	Volume, per cent	Sp. Gr. at 60°F.	Volume, per cent	Sp. Gr. at 60°F.
Up to 410°F.	43·0	0·901	39·3	0·888
410° to 572°F.	52·4	0·984	50·1	0·976
Above 572°F.	1·7	....	6·7	....
Residue	2·3	....	2·3	....
Loss	0·6	....	1·6	....

Determinations of the acids on fractions collected during the first and fourth hours of the five-hour run gave 21 per cent for the former and 20 per cent for the latter.

The results of the chemical analysis of the dehydrated distillate from the entire five-hour period are as follows.

—	Volume, per cent	
	Fraction up to 410°F.	Fraction 410° to 572°F.
Acids	21	21
Bases	3	5
Olefines	14	5
Aromatics	31	59
Paraffins and naphthenes	31	10

As in the previous run with bitumen, a considerable proportion of olefines was found. It is not possible to tell how much the phenols have been reduced because the coal adds an undetermined amount to the charge when it becomes liquid in the reaction chamber.

The liquid left in the reaction chamber after Experiment 10 was tested by distillation and extraction to find to what extent the coal charged had been liquefied in the reaction chamber. The total residue amounted to 2,189 grammes, of which 292 grammes was the catalyst. A sample of the residue, including catalyst, was extracted in a Soxhlet apparatus with benzol. The result is tabulated below.

—	Per cent	Weight
Extracted oil	55·9	1,224
Carbon and ash	30·8	673
Catalyst	13·3	292
Total	100·0	2,189

The total amount of coal charged to the reaction chamber was 2,230 grammes. Comparing this figure with the 673 grammes of carbon and ash, it is seen that up to the time of interruption of the experiment about 70 per cent of the coal had been liquefied. The remaining 30 per cent is probably made up chiefly of coal charged shortly before the run was stopped. The results of a distillation of the residue are given below.

Distillation, Hempel Method

Water—None

First drop—188°C.

Temperature °C.	Per cent cut	Cumulative per cent
Up to 250	2·0	2·0
250 to 275	3·5	5·5
Vacuum distillation at 40 mm.		
Up to 200	4·7	4·7
200 to 225	7·9	12·6
225 to 250	13·5	26·1
250 to 275	11·4	37·5
275 to 300	8·0	45·5

Residue, 91·0 c.c. (45·5 per cent by volume).
Distillation loss, 7·0 c.c. (3·5 per cent by volume).

The rate of production of distillate product decreased progressively during the five-hour period from 660 cubic centimetres in the first hour to 430 in the last hour. Changes in the conditions of temperature, pressure, and rate of recirculation were all insignificant during this period so that the decrease must be attributed to accumulation of refractory material in the reaction chamber or to deterioration of the catalyst. Since very little liquid was removed from the levelling stand-pipe, the former seems the more probable explanation.

Sulphur and ammonia were found only in very small amounts in the absorbent charcoal scrubber or in the bottles of sulphuric acid and sodium hydroxide which supplemented it. On the other hand, 20·2 grammes of ammonia and 10·1 grammes of sulphur (0·28 and 0·14 per cent of the weight of the charge respectively) were found in the liquor separated from the distillate product. The oil removed from the absorbent charcoal by heating with glycerine consisted chiefly of low-boiling unsaturated hydrocarbons.

DISCUSSION OF RESULTS

Although the results of the experiments have been in general satisfactory, it is probable that they can be improved by modification of the apparatus and procedure. It is to be borne in mind that the purpose of the process is to produce gasoline which is free from impurities and has a suitable distillation range, and it is apparent that liquid-phase hydrogenation as in the present experiments accomplishes this only in part. The process has succeeded, however, in preventing carbon formation and large losses as gas and vapour.

In discussing the yields of volatile oil, it is advisable not only to state those actually recovered but also to make an estimate of those obtainable had the runs been continued for a longer period of time. None of the experiments was continued so long that the quantity of material left in the reaction chamber could be considered a negligible fraction of the total charge, and therefore the recoveries of volatile oil were smaller than those which would have been obtained in longer runs. The yield which would be obtained in a run of long duration may be estimated if the material left in the reaction chamber at the end of the run is deducted from the total charge and the yield of volatile oil calculated as a percentage of the difference. Although this method is subject to some uncertainty, it has been adopted in calculating the two estimated values tabulated below. The values in the following table are percentages of dry volatile oil which is the "dehydrated distillate product" previously described. They are calculated to two different bases, namely, weight and volume per cent of the dry charging-stock.

—	Yields of dry volatile oil		
Experiment No	7	8	10
Material	Tar	Bitumen	Coal paste
Actual yield, per cent by weight of dry charging-stock	60·1	59·2	55·9
Actual yield, per cent by volume of dry charging-stock	67·5	73·3	65·5
Estimated ultimate yields, per cent by weight of dry charging-stock	80	82	76
Estimated ultimate yields, per cent by volume of dry charging-stock	90	102	89

Some experiments of longer duration are desirable, both for the purpose of verifying the estimated ultimate yields and of testing the durability of the catalyst. They were not performed in this investigation because sufficient staff was not available for shift operation.

The necessity of further treatment of the volatile oil is indicated by both the distillation range and chemical analysis. The distillation ranges of the volatile oils from the three experiments were very similar. About 90 per cent of the oil boils below 572°F. and of this about half boils below 410°F. From the results of the semi-continuous preliminary experiments it seems possible to increase the proportion of the gasoline fraction by circulating the hydrogen at a slower rate, but this change in procedure would be attended by a large gas loss and possibly the formation of carbon. The chemical analyses of the volatile oils show that appreciable quantities of olefines were present and that in the experiments with tar and coal, the tar acids were only partly eliminated.

The nature of the secondary treatment depends largely on the original raw material. The product from bitumen would require distillation and refining of the gasoline fraction, and further hydrogenation or cracking of the higher boiling fraction. The products from the tar and coal, because of their high phenol content, would require somewhat drastic refining unless the phenols were removed by further hydrogenation, and in this case also cracking or hydrogenation of the high-boiling fraction would be necessary. It seems advisable in future developments to subject all or part of the volatile oil to a second stage of hydrogenation. The second stage would preferably be in the vapour phase in which better contact with the catalyst is possible than in the liquid phase. Vapour-phase hydrogenation would take place both before and after separation of the gasoline fraction if the volatile product from the liquid phase contained phenols.

At the time of writing, a long vertical reaction chamber is being constructed in which the upper and lower halves are separately heated. It will be possible to have the liquid phase in the lower half and to treat the vapours carried from it in the upper half with a different catalyst and at a different temperature. When vapour-phase treatment is required both before and after separation of the gasoline fraction, a second passage through this reaction chamber will be necessary.

The present experiments have been essentially a test of the method rather than of the raw materials, and it is probable that better results could be obtained, even with the present apparatus and general procedure, if a prolonged program of experiments were carried out. The work is not sufficiently advanced to justify a definite statement concerning the commercial promise of any of the materials, although all of them give good yields of light oil with a fairly simple treatment.

SUMMARY

A continuous laboratory-scale, liquid-phase hydrogenation apparatus has been constructed at the Fuel Research Laboratories. In it, the volatile oil product is removed from the charge as vapour in a stream of hydrogen at high pressure. A constant volume of liquid is maintained in the apparatus by pumping in fresh charging-stock. The catalyst used in the later experiments described was molybdic oxide supported on coke of 8- to 10-mesh size. The concentration of catalyst was about 260 to 280 grammes in a liquid phase of 2,800 cubic centimetres. The temperatures of reaction were 428° to 452°C., the pressures 183 to 187 atmospheres, and the rates of hydrogen recirculation 87 to 112 cubic feet per hour, measured at atmospheric pressure and temperature.

The three materials hydrogenated were tar produced from Sydney coal by the Illingworth low-temperature carbonization process, Alberta bitumen, and a paste of 40 per cent of powdered Cape Breton bituminous coal mixed with the Sydney tar.

Somewhat similar results were obtained in the experiments with all three materials. Carbon formation was avoided and the total losses as gas and vapour were about 10 per cent of the material charged. The hydrogen combined and lost amounted to 5·7 to 7·2 per cent of the weight of the material charged. The experiments were of short duration so that the amount of partly converted material left in the apparatus at the end of the run prevented an exact determination of the yield of volatile oil which could be obtained in a long run. The yields, based on the total material charged, are 67·5, 73·3, and 65·5 per cent by volume of the dry tar, bitumen, and paste respectively. The yields based on the net material charged, that is, the total amount charged minus the partly converted oil left in the reaction chamber at the end of the run, are 90, 102, and 89 per cent by volume of the dry tar, bitumen, and paste respectively. It is thought that the higher figures are closer than the lower to the results which would be obtained in runs of long duration.

The volatile products from all three experiments had similar distillation ranges, approximately 90 per cent distilling below 572°F. and 45 per cent below 410°F. About 10 per cent of olefines was present in the volatile product. In the experiment on tar the phenols were not reduced appreciably, and in that on the paste only to a slight extent. It is probable, therefore, that both the distillation range and the purity of the volatile oil can be improved by further hydrogenation. It is proposed in future developments to add a vapour-phase stage to the present procedure.

VIII

A STUDY OF THE NATURAL GAS AND NAPHTHA PRODUCTS FROM TWENTY-FOUR WELLS IN TURNER VALLEY, ALBERTA

P. V. Rosewarne, W. P. Campbell, and R. J. Offord

INTRODUCTION

During the past four years the Division of Fuels and Fuel Testing of the Mines Branch has investigated various features relating to the natural gas and naphtha produced at Turner Valley in Alberta. These include the composition of the stripped gases, the naphthas, the condensates, the vapours lost on weathering, and the helium content.

In 1932, an attempt was made to determine the changes in composition of the stripped gas and condensate that may be expected with change of pressure. Part of the results obtained are presented in this paper. In order to achieve the desired end a great deal of supplementary data was, of necessity, collected. Much of this data has not yet been thoroughly collated.

OBJECT OF THE INVESTIGATION

The object of the investigation was to determine the composition of the material present in the gas-producing horizons as far as that might be possible from a study of the total production at the surface. The total production at the surface consists of gas and liquid, both of which are composed of hydrocarbons predominantly. The liquid is recovered in a trap, or separator, and the gas is burned or used for fuel. The liquid caught in the separator is then "weathered," or heated slightly to drive off the more volatile portions. In this paper the weathered liquid is called "naphtha," and the same material before it is weathered is called "condensate." These terms were chosen arbitrarily in order to avoid possible confusion in the discussion.

METHOD OF ANALYSIS

The best available method of analysis was admittedly a fractional distillation of the products. However, the time required for the examination of each sample was so long by this method that hope of using it was reluctantly relinquished. It was concluded from a study of the literature that a method using the absorptive power of activated charcoal, and a subsequent distillation of the absorbed products under a pressure of 30 pounds per square inch, could be substituted for the fractional distillation and would give sufficiently accurate results to enable general conclusions to be formed. The use of this method has been partly justified.

METHOD OF PROCEDURE

It was proposed at first that tests should be conducted on the products of a relatively few wells, and that these wells should be operated at different pressures, but the proration of production by the Turner Valley Natural Gas Committee necessitated the selection of a larger number of wells for examination, the pressures and production of which under proration covered the range that it was desired to study. On account of this enforced method of procedure some assumptions were made in compiling the results that may be questioned with a certain degree of propriety. One of these assumptions relates to the pressure. It was assumed that the "closed-in pressures," which were the only ones available, were related in a definite way with the production of naphtha. It is admitted that "rock pressures" would have been more reliable and desirable had they been available, since the closed-in pressure of a well differs from the rock pressure when there is liquid at the bottom of the hole. In some cases it was known that there was liquid at the bottom, and in others it was strongly suspected. It was assumed in addition that the composition of the hydrocarbons liquefied by the lowered temperature produced by the expansion of the gas was not influenced to an appreciable extent by passing through this accumulated liquid. It was also assumed at first that the size of the separator, its location, method of operation, etc., were not significant factors. However, the results obtained have indicated that the balance between pressure and volume of the gas, and radiation losses from the separator and gathering lines may be more delicate than was suspected. It is possible that were complete data available corrections might be applied to compensate for some of the consequent errors, but since such information is not available the data obtained are set forth in the following pages in the belief that they will assist towards a better understanding of this field and of others of a similar kind.

SELECTION OF THE WELLS TO BE TESTED

In selecting the wells to be tested several considerations were kept in mind. First, there must be included wells of high, medium, and low gas production; wells having high, medium, and low pressures; and wells which were peculiar for some reason. Second, the wells selected should cover the field from a geographical point of view. Third, the wells should represent as far as possible the various financial interests in the field.

The wells finally selected for testing were as follows:

1. Advance, No. 5A
2. Baltac, No. 1
3. Dalhousie, No. 7
4. East Crest, No. 3
5. Foothills, No. 1
6. Foothills, No. 2
7. Freehold, No. 2
8. Home, No. 4
9. Lowery, No. 1
10. Mayland, No. 6
11. Merland, No. 1
12. Mercury, No. 1
13. Mercury, No. 2
14. Miracle, No. 1
15. Model, No. 1
16. Okalta, No. 1
17. Richfield, No. 1
18. Royalite, No. 19
19. Royalite, No. 21
20. Royalite, No. 23
21. Sterling Pacific, No. 1
22. Sterling Pacific, No. 2
23. Structure, No. 1
24. Wellington, No. 1

Unfortunately, before the tests were completed six wells, namely, Baltac No. 1, Foothills No. 2, Freehold No. 2, Royalite No. 19, Royalite No. 21, and Sterling Pacific No. 1, were closed in and samples of their products could not be secured.

TABLE I

Classification of Wells According to Gas Production

	M. cu. ft. per day		M. cu. ft. per day
1. *High Gas Production—*		3. *Low Gas Production—*	
Mercury, No. 1	9,672	Baltac, No. 1	1,451
Merland, No. 1	8,039	Mercury, No. 2	1,315
Wellington, No. 1	7,133	Foothills, No. 2	1,245
Mayland, No. 6	6,226	Structure, No. 1	1,148
Sterling Pacific, No. 2	5,924	Royalite, No. 21	1,075
Lowery, No. 1	3,990	Sterling Pacific, No. 1	876
Dalhousie, No. 7	3,929		
Royalite, No. 19	3,929		
2. *Medium Gas Production—*			
Home, No. 4	3,500*		
East Crest, No. 3	3,355		
Okalta, No. 1	3,053		
Foothills, No. 1	3,022		
Royalite, No. 23	3,022		
Miracle, No. 1	2,781		
Model, No. 1	2,714†		
Freehold, No. 1	2,176		
Advance, No. 5A	1,934		
Richfield, No. 1	1,741		

*Estimated.
†At 800 lb. per sq. inch back pressure.

The selected wells are classified in Table I and Table II, according to gas production, and according to closed-in pressure, respectively. It will be observed that the whole range of pressures is very satisfactorily covered by the wells that were selected.

TABLE II

Classification According to Closed-in Pressures

	Lb. per sq. inch		Lb. per sq. inch
Sterling Pacific, No. 2	1,855	Structure, No. 1	1,190
Merland, No. 1	1,810	Richfield, No. 1	1,020
Stirling Pacific, No. 1	1,705	Royalite, No. 23	1,010
Model, No. 1	1,635	“ No. 19	960
Mercury, No. 2	1,580	Okalta, No. 1	930
Foothills, No. 1	1,550	Foothills, No. 2	925
Miracle, No. 1	1,540	Royalite, No. 21	900
Mercury, No. 1	1,465	Lowery, No. 1	850
East Crest, No. 3	1,360	Home, No. 4	850
Mayland, No. 6	1,350	Wellington, No. 1	815
Dalhousie, No. 7	1,255	Baltac, No. 1	790
Advance, No. 5A	1,190	Freehold, No. 1	780

As indicated above, some of the wells were selected on account of some peculiarity or unusual factor connected with them. For instance, Merland No. 1 was chosen because it was the most southerly; Foothills No. 1, because it was the most northerly; Royalite No. 19, because samples had been collected systematically from it for examination during the past three years, and Model No. 1, because it produced the largest amount of naphtha per thousand cubic feet of gas for any well in the field.

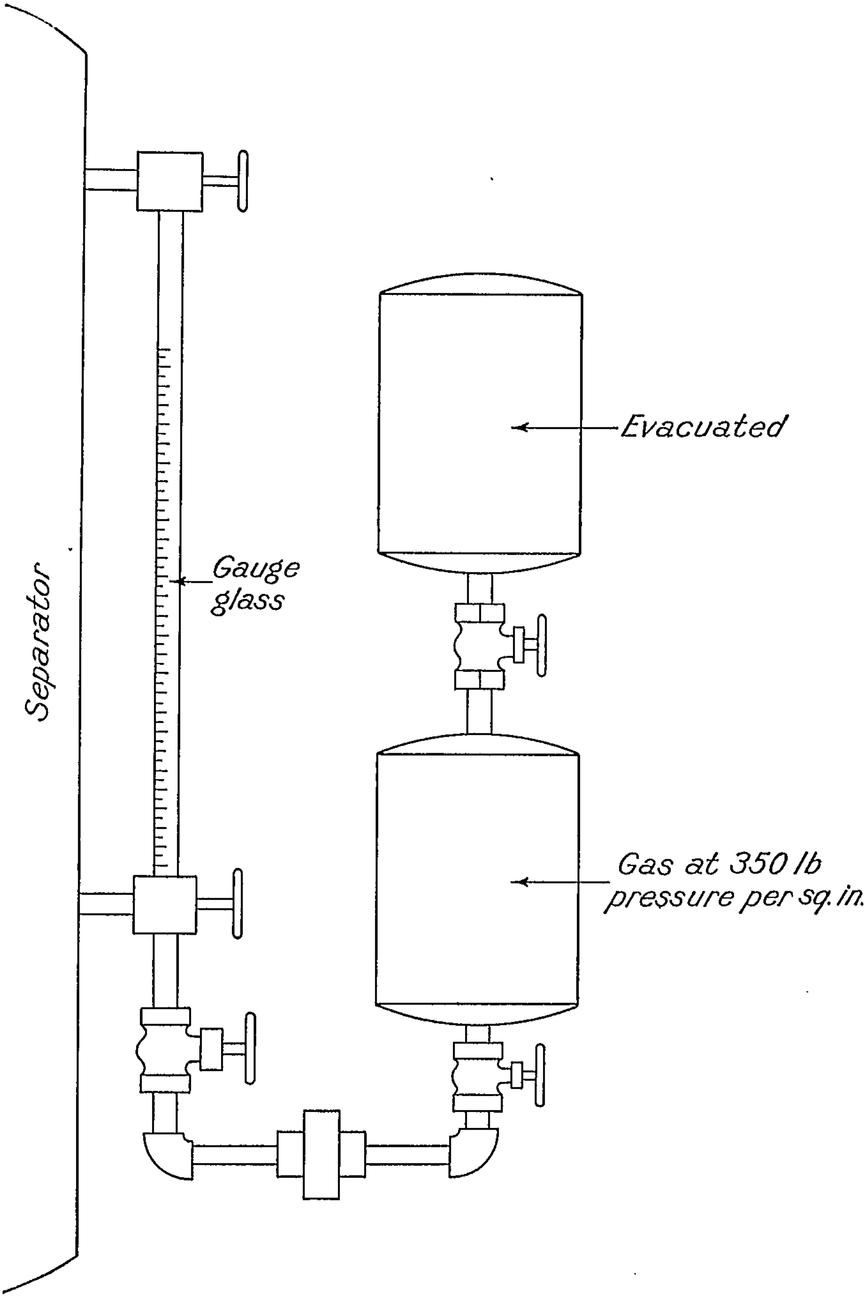

Figure 5. Sketch showing sampling tube and method of attaching to separator.

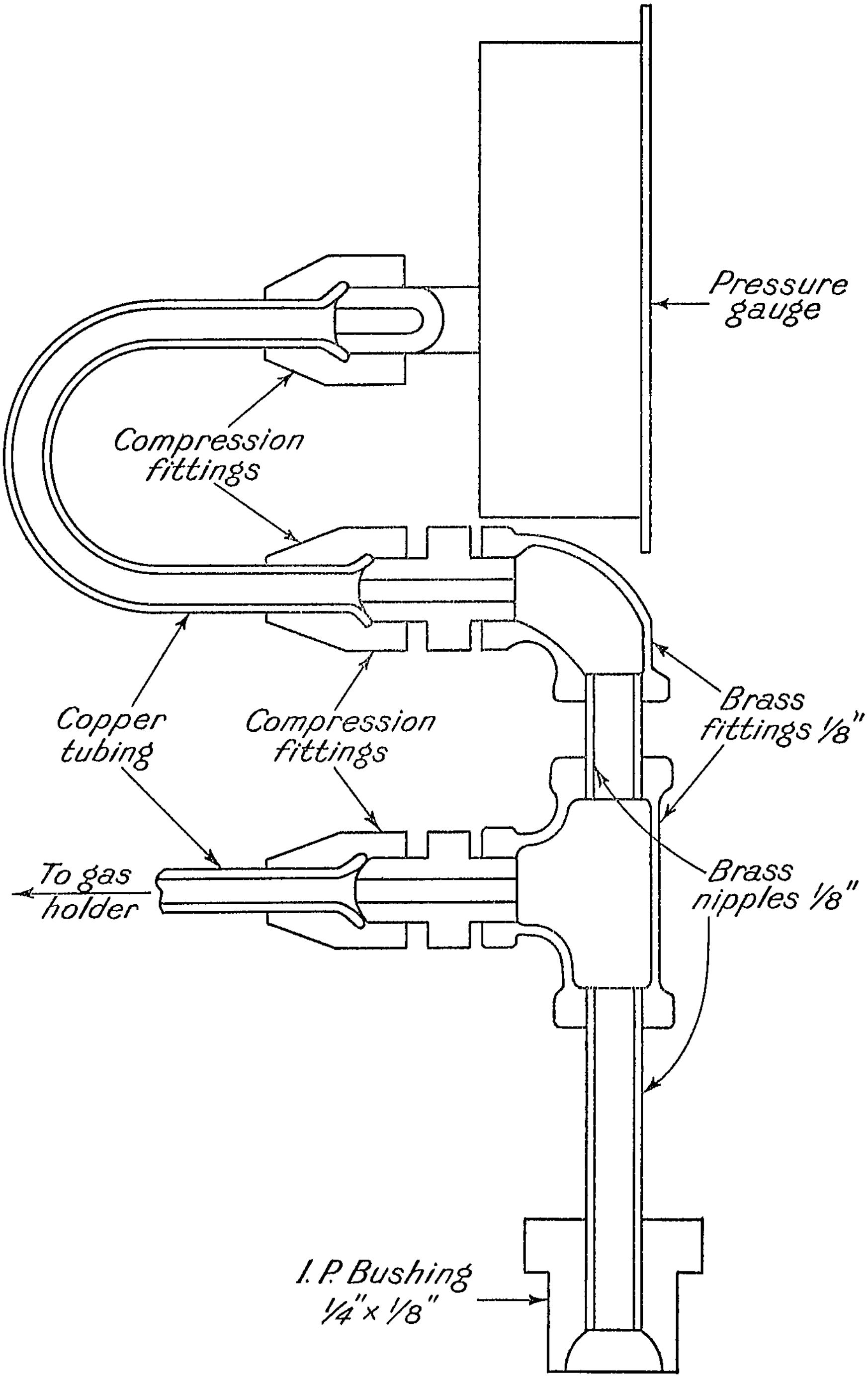

Figure 6. Sketch showing gauge and connexions for collecting condensate vapours.

The wells selected covered the field from a geographical point of view very thoroughly. For purposes of administration the Turner Valley Natural Gas Commission divided the field into 11 districts, which were numbered consecutively from the south to the north. Table III is arranged to show the number of wells in each of the districts adopted by the Commission, and also the number of wells in each district that was selected for testing.

TABLE III

Producing Wells by Districts

District No.	Number of wells in district	Number of wells selected for testing
1	1	1
2	12	5
3	8	2
4	16	4
5	1	1
6	1	0
7	6	3
8	16	4
9	18	2
10	1	1
11	1	1
Total	81	24

When the tests were begun there were 81 wells producing naphtha in the Turner Valley field. Of these 81 wells, 29 were owned by subsidiaries of the Imperial Oil Company, 14 were owned by independent companies who had contracted to deliver their production to the Imperial Oil Company, and 38 were owned by independent companies who disposed of their production independently. When the wells that were selected for testing were classified in the same manner, it was found that 7 wells belonging to Imperial Oil subsidiaries were chosen; that the production of 7 wells was under contract to the Imperial Oil Company; and that 10 wells were owned and operated independently. A summary of the above classification is shown in Table IV.

TABLE IV

Summary of Producing Wells in Turner Valley

Affiliations	Total wells in field	No. of wells selected for test	Naphtha production of wells selected, bbl.
Imperial subsidiaries	29	7	655
Under contract to Imperial	14	7	440
Independent	38	10	704
Total	81	24	1,799

EQUIPMENT USED IN THE INVESTIGATION

The equipment used for the investigation consisted of a number of specially designed sampling tubes for taking samples of condensate under pressure; an Oberfell charcoal absorption apparatus for testing samples of stripped gas; a water bath for heating the samples; a gasometer; and a distillation apparatus for distilling the absorbed material under a pressure of 30 pounds per square inch.

The sampling tubes consisted of two pieces of high-pressure steel tubing, 2½ inches in diameter, and about 5 inches long, with plates welded on both ends. One of the pieces had a short iron nipple welded in one end and the other piece had a nipple in each end. The two pieces were united with a needle-valve into which the nipples were screwed. A second needle-valve was screwed onto the remaining nipple. The assembled unit was then tested to at least 500 pounds pressure per square inch. A sketch of the assembled apparatus is shown in Figure 5.

The Oberfell charcoal absorption apparatus used was that part of the standard equipment which is used to bring a measured quantity of gas into contact with the activated charcoal. A modified method of distillation was adopted as is described below.

The water bath consisted of an ordinary galvanized iron pail partly filled with water, in which was immersed a perforated copper coil. This was connected to a steam line. The arrangement enabled the samples to be heated gradually to any desired temperature. A small pressure gauge, as shown in Figure 6, was attached to the line leading from the sample tube to the gasometer in order to indicate excessive pressures should they occur during heating.

The gasometer was made in the laboratory. It consisted of a steel drum supported in a framework of iron pipe and a large glass bottle which was carefully calibrated beforehand. Suitable equipment for counterpoising, for handling the gas and for measuring its volume were attached. The glass bottle was especially convenient because the amount of gas present was always apparent and could be measured with certainty. A sketch of the set-up is shown in Figure 7.

The distillation apparatus used for distilling the absorbed hydrocarbons from the charcoal was similar to that approved by the Southern California Natural Gasoline Association. Briefly, it consisted of a large receptacle filled with crushed ice and kept at a temperature of 32°F., in which was a metal coil having an available condensing area of 100 square inches, a steel distillation flask, a mercury pressure regulator, pressure gauges, a graduated receiver and receiver ice bath, and a vent for non-condensable gases. A sketch of the apparatus is shown in Figure 8.

METHOD OF OPERATION

A flow-sheet is given in Figure 9 which shows the samples that were taken and the treatment each one received. The sample of stripped gas was taken through an orifice tip set facing the gas flow in the centre of the waste gas line between the separator and the flare. It was passed through the meter of the Oberfell apparatus and then through activated charcoal. The hydrocarbons absorbed by the charcoal were then distilled under a pressure of 30 pounds per square inch and the vapours condensed and measured at 32° F. The sample of naphtha was dipped from the storage tank at atmospheric pressure. A representative sample of condensate was more difficult to obtain, and the special sampling tubes were designed

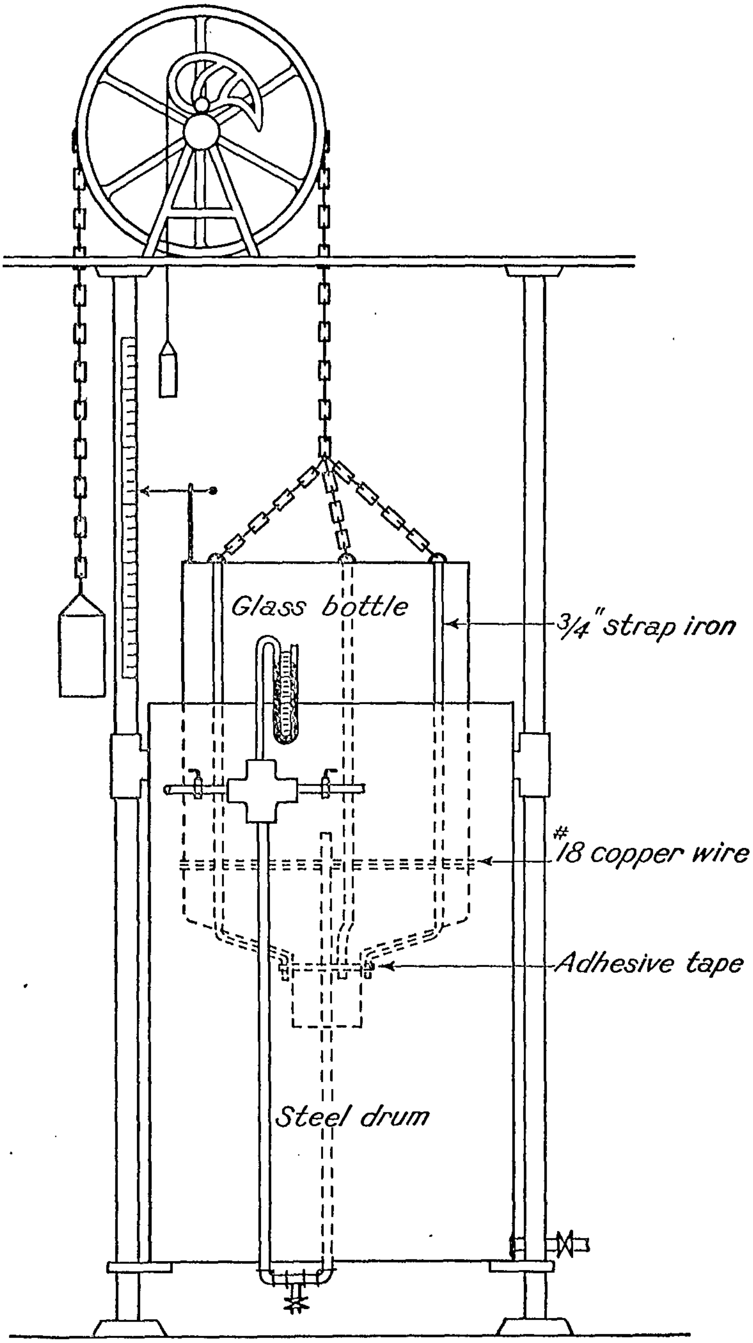

Figure 7. Diagrammatic sketch of gas holder.

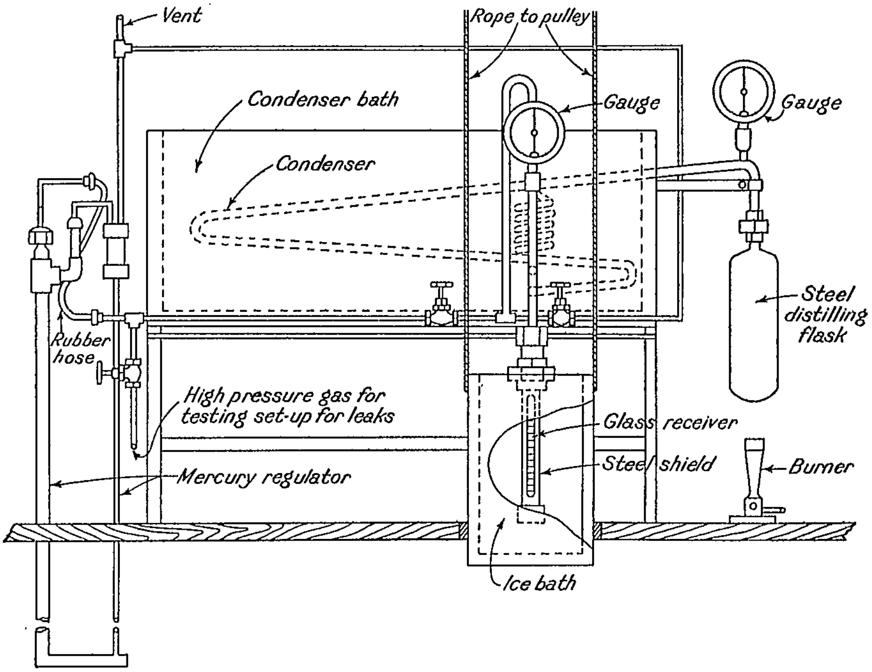

Figure 8. Pressure distillation apparatus for 30-pound – 32° – 32° test.

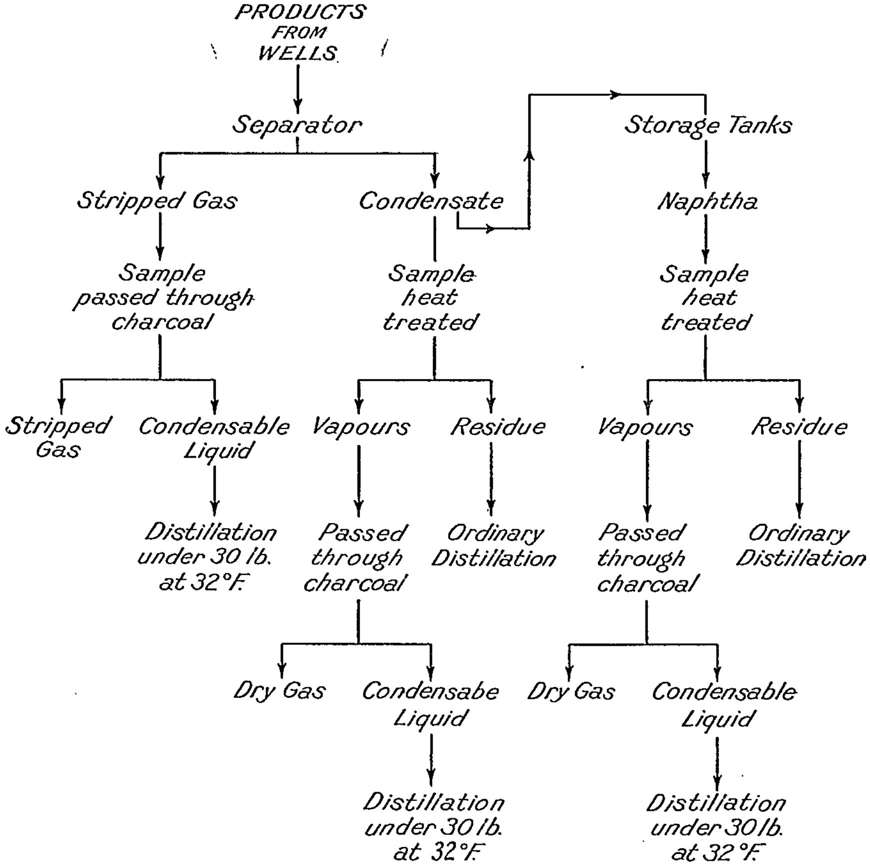

Figure 9. Flow-sheet showing treatment of samples.

on that account. Before taking a sample both parts of the tube were evacuated and both valves closed tightly. Then, natural gas was admitted to the lower portion only until a pressure was obtained slightly in excess of that on the separator from which a sample was to be taken. The whole unit was then connected to the separator, usually below the gauge glass, and the lower valve opened. This permitted some gas to be forced back into the separator due to the excess pressure in the sampling tube and was thought to ensure a more satisfactory sample by flushing any stagnant liquid out of the connexions. When the pressures in the lower part of the tube and in the separator were in equilibrium the middle valve was carefully opened to a very slight degree. The gas in the lower portion gradually seeped into the upper portion through this valve and was replaced by condensate from the separator, without the condensate being subjected to any appreciable reduction in pressure. The lower valve was then closed tightly; the unit was removed from the separator, it was inverted in order that the liquid and gas might be transposed and was taken to the laboratory for further examination.

The sampling tube and its contents were placed in the water bath described above and connected to the gasometer. The valve was opened gradually and the gas transferred to the gasometer and measured. When a quantity of gas had been recovered that was equivalent at atmospheric pressure to that which had been trapped in the tube at the separator pressure, the control valve was closed and the gas in the gasometer was discarded on the assumption that this gas was more or less comparable to that which had been used to compensate for the separator pressure. The valve was then opened again and the temperature of the water bath raised gradually to 85°F. The bath was maintained at that temperature for some time during which the contents of the sampling tube were well agitated by occasional shaking in order to remove all gases held in solution. In the meantime the temperature of the water in the gasometer was also raised to 85°F. to prevent partial condensation of the vapour. When consecutive readings on the gasometer showed that no appreciable quantity of gas or vapour was being given off, the sampling tube was disconnected, the volume and temperature of the evolved gas were noted, and a known quantity of it was passed through activated charcoal. Some samples were also taken from the gasometer for fractionation in a Podbielniak column at Ottawa.

The hydrocarbons absorbed by activated charcoal were distilled off under 30 pounds pressure per square inch and condensed at 32°F. in the apparatus described above. The condenser and the ice bath for the receiver were filled with crushed ice, and the charcoal with its absorbed material was emptied into the distilling flask. One hundred and fifty cubic centimetres of glycerine was added and the flask and graduated receiver fastened in place.

The assembled apparatus was then tested for leaks with natural gas at a pressure of 30 pounds per square inch. If no drop in pressure occurred within 10 minutes, heat was applied to the flask and the distillation started. The distillation was continued until all the hydrocarbons and 15 or 20 cubic centimetres of glycerine were recovered in the receiver. The volume of the supernatant layer of hydrocarbons was then determined and recorded.

The specific gravity and the distillation range of the residue left in the sampling tubes were determined by approved methods for gasoline and similar liquids. The greater part of these residues was shipped to Ottawa and examined there in order to save time in the field.

SUMMARY OF THE ANALYTICAL DATA

A summary of the data obtained in the investigation is given in tabular form in Tables V to IX inclusive. Table V shows the pressure on the well and on the separator at the time of the test, the amount and the specific gravity of the naphtha obtained, the amount of gas produced and the amount of liquid condensable from activated charcoal calculated in gallons per thousand cubic feet of gas and in barrels per day. Table VI shows the results of the heat treatment of the condensate, the amount of the residue after heating, the volume of the vapours recovered, and the calculated loss by the treatment. Table VII shows similar results for the heat treatment of the naphtha. Table VIII is a summary of the three previous tables. It shows the ratio of the condensate to a hundred barrels of naphtha, the condensable liquid in the vapours from weathering, and in the stripped gases, the total possible recovery from the well, and a theoretical efficiency of the separator calculated from the total possible recovery. Table IX shows the specific gravity and the distillation range of the residues from the heat treatment of the samples of condensate and naphtha given in Tables VI and VII.

TABLE V

Production of Naphtha and Natural Gas

Test No.	Name of well	Pressure		Naphtha			Natural Gas		
		On well in lb. per sq. in.	On separator in lb. per sq. in.	Amount recovered bbl.	Specific gravity at 60°F.	A.P.I. degrees at 60°F.	Gas flow M.c.f.	Condensable liquid from charcoal test	
								Gal/M.	bbl.
15	Advance, No. 5A....	500	265	92·0	....	....	4,850	0·3000	41·6
1	“ “	760	300	22·0	0·6950	72·1	1,960	0·190	10·6
27	“ “	1,150	300	13·4	0·6933	72·6	1,020	0·119	3·5
21	Dalhousie, No. 7.....	960	300	39·7	0·6872	74·4	3,960	0·167	19·0
22	East Crest, No. 3....	1,160	185	30·0	0·6764	77·7	2,800	0·203	16·2
20	Foothills, No. 1......	1,200	290	100·0	0·6973	71·4	6,300	0·154	27·7
3	Home, No. 4........	625	300	18·0	0·6935	72·5	3,500	0·269	......
24	Lowery, No. 1.......	675	300	....	0·7028	69·8	*6,200	0·221	......
8	Mayland, No. 6......	1,080	280	99·0	0·6790	76·9	6,620	0·206	38·9
10	Merland, No. 1......	1,700	250	146·0	0·6978	71·3	5,700	0·072	11·7
6	Mercury, No. 1......	1,200	285	169·0	....	....	9,500	0·160	43·0
9	“ “	1,200	265	169·0	0·6825	75·8	9,500	0·183	49·7
7	Mercury, No. 2......	1,180	250	27·5	0·6874	74·4	1,300	0·176	6·5
5	Miracle, No. 1.......	1,220	250	68·5	0·6860	74·8	2,750	0·145	11·4
12	Model, No. 1........	600	90	125·0	0·7672	52·9	3,109	0·186	16·5
13	“ “	700	200	133·0	0·7697	54·3	2,843	0·207	16·8
14	“ “	800	195	120·0	0·7584	55·1	2,714	0·114	8·8
16	“ “	900	300	102·0	0·7596	54·8	2,445	0·148	10·3
26	“ “	1,000	225	91·3	0·7552	55·9	2,250	0·163	10·4
17	“ “	1,000	300	86·2	0·7563	55·6	1,779	0·174	8·8
18	“ “	1,100	250	61·3	0·7583	55·1	1,465	0·121	......
4	Okalta, No. 1........	815	200	22·5	0·6951	72·1	2,223	0·168	10·7
23	Richfield, No. 1.....	870	230	8·1	0·6894	73·8	1,370	0·170	6·7
25	Royalite, No. 23.....	850	300	38·1	0·6869	74·5	7,550	0·123	26·5
11	Sterling Pacific, No. 2..............	1,520	270	102·0	0·6870	74·5	3,450	0·092	9·1
2	Structure, No. 1.....	1,010	220	13·6	....	....	3,000	0·241	20·7
19	Wellington, No. 1....	700	200	24·4	0·7013	70·3	8,050	0·172	39·5

*Estimated.

TABLE VI

Heat Treatment of Condensate

Test No.	Name and number of well	Residue after heating to 85°F. c.c.	Volume vapours recovered c.c.	Vapours calc. as liquid butane c.c.	Recovery from condensate c.c.	Calculated loss c.c.	Calculated volume of sample c.c.	Ratio sample to residue %
15	Advance, No. 5A	168·0	10,300	38·6	207	6·2	213	126·6
1	" "	124·0	16,975	63·6	188	6·1	194	156·5
27	" "	203·0	2,075	7·8	211	6·7	218	107·4
21	Dalhousie, No. 7	132·0	21,150	79·3	211	5·8	217	164·4
22	East Crest, No. 3	123·0	19,525	73·2	196	5·6	202	164·2
20	Foothills, No. 1	146·5	14,450	54·2	201	4·6	206	140·6
3	Home, No. 4	142·0	11,500	43·1	185	5·4	190	133·8
24	Lowery, No. 1	152·0	13,900	52·1	204	4·6	209	137·5
8	Mayland, No. 6	224·0	35,175	131·8	356	14·2	370	165·1
10	Merland, No. 1	68·5	16,550	62·0	131	5·9	137	200·0
6	Mercury, No. 1	98·0	20,550	77·0	175	5·2	180	183·5
9	" "	102·0	19,050	71·4	173	12·7	186	182·3
7	Mercury, No. 2	127·0	14,925	55·9	182	10·2	192	151·1
5	Miracle, No. 1	108·0	17,500	65·6	174	2·6	177	163·8
12	Model, No. 1	165·0	4,900	18·4	183	5·5	189	114·5
13	" "	146·0	9,550	35·8	182	5·5	188	128·8
14	" "	156·5	10,250	38·4	195	5·9	201	128·4
16	" "	177·0	14,525	54·4	231	6·9	238	134·4
26	" "	12·0	4,900	18·4	30	....	....	....
17	" "	129·0	12,250	45·9	175	5·2	180	139·4
18	" "	27·8	5,400	20·3	48	....	....	....
4	Okalta, No. 1	204·0	9,600	36·0	240	16·1	256	125·5
23	Richfield, No. 1	182·0	17,525	65·7	248	8·3	256	140·5
25	Royalite, No. 23	150·0	17,275	64·7	215	4·9	220	146·6
11	Sterling Pacific, No. 2	74·7	19,025	71·3	146	10·1	156	208·9
2	Structure, No. 1	184·0	4,900	18·4	202	5·4	207	112·5
19	Wellington, No. 1	137·5	9,150	34·3	172	15·7	188	136·8

TABLE VII

Heat Treatment of Naphtha

Test No.	Name and number of well	Volume of sample c.c.	Residue after heating to 85° F. c.c.	Volume vapours recovered c.c.	Vapours calc. as liquid butane c.c.	Recovery from naphtha c.c.	Loss c.c.	Loss %	Ratio sample to residue %
15	Advance, No. 5A	....	....	....	....	....	....	....	....
1	" "	499	472	3,025	11·34	483·3	15·7	3·26	105·7
27	" "	498	469	3,650	13·68	482·7	15·3	3·17	106·1
21	Dalhousie, No. 7	497	471	3,400	12·74	483·7	13·3	2·75	105·5
22	East Crest, No. 3	500	439	12,525	46·99	486·0	14·0	2·88	113·9
20	Foothills, No. 1	501	481	2,350	8·81	489·8	11·2	2·29	104·2
3	Home, No. 4	500	484	500	1·88	485·9	14·1	2·90	103·3
24	Lowery, No. 1	504	492	250	0·94	492·9	11·1	2·25	102·5
8	Mayland, No. 6	499	459	5,550	20·80	479·8	19·2	4·00	108·9
10	Merland, No. 1	498	476	100	0·38	476·4	21·6	4·53	104·6
6	Mercury, No. 1	....	....	....	....	....	....	....	....
9	" "	507	464	2,250	8·43	472·4	34·6	7·32	109·3
7	Mercury, No. 2	495	457	3,150	11·81	468·8	26·2	5·59	108·3
5	Miracle, No. 1	489	440	11,125	41·71	481·7	7·3	1·52	111·1
12	Model, No. 1	....	....	....	....	....	....	....	100·0
13	" "	....	....	....	....	....	....	....	100·0
14	" "	....	....	....	....	....	....	....	100·0
16	" "	....	....	....	....	....	....	....	100·0
26	" "	....	....	....	....	....	....	....	100·0
17	" "	....	....	....	....	....	....	....	100·0
18	" "	....	....	....	....	....	....	....	100·0
4	Okalta, No. 1	500	466	650	2·44	468·5	31·5	6·73	107·3
23	Richfield, No. 1	498	475	1,850	6·94	481·9	16·1	3·34	104·9
25	Royalite, No. 23	506	481	3,650	13·68	494·7	11·3	2·28	105·1
11	Sterling Pacific, No. 2	503	439	9,400	31·49	470·5	32·5	6·91	114·5
2	Structure, No. 1	500	485	500	1·88	486·9	12·9	2·67	103·1
19	Wellington, No. 1	497	455	100	0·38	455·4	41·6	9·14	109·2

TABLE VIII

Summary of Recovery

Test No.	Name of well	Ratio condensate per 100 bbl. naphtha	Amount naphtha recovered bbl.	Condensable liquid in		Total possible recovery from well bbl.	Theoretical efficiency of separator %
				Vapours from weathering bbl.	Stripped gas bbl.		
15	Advance, No. 5A	....	92·0	2·2	41·6	135·8	67·7
1	“ “	148·0	22·0	1·4	10·6	34·0	64·7
27	“ “	101·1	13·4	0·04	3·5	16·9	79·3
21	Dalhousie, No. 7	155·6	39·7	4·9	19·0	63·6	62·4
22	East Crest, No. 3	144·2	30·0	6·0	16·2	52·2	57·5
20	Foothills, No. 1	134·9	100·0	7·3	27·7	135·0	74·1
3	Home, No. 4	129·5	18·0	1·1	....	....	....
24	Lowery, No. 1	134·1	....	....	....	....	....
8	Mayland, No. 6	151·6	99·0	18·6	38·9	156·5	63·2
10	Merland, No. 1	191·2	146·0	27·5	11·7	185·2	78·8
6	Mercury, No. 1	....	169·0	....	43·0	....	....
9	“ “	166·7	169·0	30·8	49·7	249·5	67·7
7	“ “ 2	139·5	27·5	3·0	6·5	37·0	74·3
5	Miracle, No. 1	147·4	68·5	7·9	11·4	87·8	78·0
12	Model, No. 1	114·5	125·0	1·5	16·5	143·0	87·4
13	“ “	128·8	133·0	3·7	16·8	153·5	86·6
14	“ “	128·4	120·0	3·9	8·8	132·7	90·4
16	“ “	134·4	102·0	4·2	10·3	116·5	85·8
26	“ “	....	91·3	....	10·4	....	....
17	“ “	139·4	86·2	4·1	8·8	99·1	87·0
18	“ “	....	61·3	5·1	....	....	....
4	Okalta, No. 1	117·0	22·5	0·6	10·7	33·8	66·6
23	Richfield, No. 1	133·9	8·1	0·9	6·7	15·7	51·6
25	Royalite, No. 23	139·5	38·1	2·5	26·5	67·1	56·8
11	Sterling Pacific, No. 2	182·4	102·0	24·7	9·1	135·8	75·1
2	Structure, No. 1	109·1	13·6	0·1	6·9	20·6	66·2
19	Wellington, No. 1	125·1	24·4	0·6	39·5	64·5	37·8

TABLE IX
Residue from Turner Valley Condensate and Naphthas

Test No.	Residue	Degrees A.P.I.	Specific gravity, at 60°F.	Distillation Range, °F. First drop	5%	10%	20%	30%	40%	50%	60%	70%	80%	90%	95%	End point	Recovery	Residue	Distillation loss
1	Condensate*	....	....	80	99	110	133	148	166	184	204	230	276	......	......	466	89·5	3·5	7·0
1	Naphtha*	....	....	80	103	112	128	146	163	180	199	222	259	399	......	454	91·0	3·0	6·0
2	Condensate*	....	....	84	118	134	156	173	188	203	218	238	261	313	......	361+	94·0	1·0	5·0
2	Naphtha*	....	....	82	104	115	130	145	162	176	191	209	233	288	......	330	94·0	1·0	5·0
3	Condensate*	....	....	74	100	113	134	149	164	178	193	209	230	310	......	339	91·5	3·0	5·5
3	Naphtha*	....	....	78	104	116	136	151	166	178	192	208	224	268	......	308	95·0	0·5	4·5
4	Condensate	70·1	0·702	85	110	122	144	160	178	192	208	225	251	298	......	366	95·0	1·5	3·5
4	Naphtha	70·6	0·700	88	112	124	143	158	172	186	200	216	235	284	318	329	97·0	1·0	2·0
5	Condensate	69·6	0·707	92	110	123	136	152	169	189	208	229	261	327	......	342	92·5	4·5	3·0
5	Naphtha	73·3	0·691	81	102	110	126	141	157	175	194	214	240	298	348	361	96·0	1·2	2·8
6	Condensate	70·9	0·699	88	116	122	139	151	164	181	197	217	232	279	327	350	95·5	1·7	2·8
7	Condensate	70·6	0·700	82	105	116	134	150	165	181	200	221	246	293	344	370	97·0	1·4	1·6
7	Naphtha	73·0	0·692	81	104	114	130	146	163	180	198	218	244	295	360	363	95·5	1·1	3·4
8	Condensate	74·2	0·688	81	100	109	125	139	154	170	189	207	231	284	......	335	95·0	1·1	3·9
8	Naphtha	74·5	0·687	81	98	107	122	138	154	168	186	204	229	277	......	334	95·0	1·0	4·0
9	Condensate	72·4	0·694	83	104	113	127	141	156	171	189	206	228	268	308	334	96·5	1·1	2·4
9	Naphtha	69·2	0·705	90	122	138	153	162	172	192	212	234	265	320	394	396	96·0	1·3	2·7
10	Condensate	70·1	0·702	86	103	113	130	148	168	188	208	234	268	328	......	378	95·0	2·3	2·7
10	Naphtha	73·0	0·692	86	105	115	130	144	158	175	191	210	234	277	......	355	95·0	1·1	3·9
11	Condensate	70·1	0·702	86	101	112	126	146	166	187	209	236	280	331	......	385	94·5	2·0	3·5
11	Naphtha	70·9	0·699	84	101	113	130	147	167	189	209	234	267	338	......	401	95·0	1·5	3·5
12	Condensate	53·5	0·765	96	125	140	178	209	240	276	323	398	484	......	......	501	81·0	18·5	0·5
13	Condensate	54·0	0·763	97	120	142	177	210	244	284	338	423	......	......	......	470	80·0	18·0	2·0
13	Naphtha	53·5	0·765	94	125	146	182	213	245	282	334	418	......	......	......	494	78·0	20·2	1·8
14	Condensate	55·4	0·757	92	124	144	176	210	244	286	336	432	......	......	......	496	77·0	19·4	3·6
15	Condensate	63·7	0·725	88	121	139	164	181	198	212	230	254	296	401	......	468	93·0	5·0	2·0
16	Condensate	55·9	0·755	87	113	134	170	202	240	277	334	418	......	......	......	480	76·0	20·4	3·6
17	Condensate	56·9	0·751	86	114	131	162	194	225	259	304	382	......	......	......	458	77·5	20·0	2·5
17	Naphtha	54·9	0·759	88	123	142	175	206	237	278	326	406	......	......	......	492	78·0	18·0	4·0
18	Condensate	55·2	0·758	111	130	147	178	200	232	263	314	406	540	......	......	582	83·5	10·0	6·5
19	Condensate	70·9	0·699	89	112	124	142	156	170	184	196	210	226	256	310	316	96·0	1·0	3·0
19	Naphtha	69·5	0·704	90	117	129	144	161	176	189	202	216	233	265	308	334	97·0	0·9	2·1
20	Condensate	66·7	0·714	83	107	117	135	152	170	187	207	227	256	308	......	380	95·0	1·6	3·4
20	Naphtha	69·5	0·704	90	109	121	141	158	175	194	212	236	263	322	......	386	94·0	2·4	3·6
21	Condensate	72·1	0·695	86	106	116	130	144	158	174	190	206	230	270	312	322	96·0	1·3	2·7
21	Naphtha	73·0	0·692	82	104	115	130	144	159	174	190	206	228	264	313	333	97·0	1·0	2·0
22	Condensate	76·6	0·680	82	96	103	114	124	136	150	164	180	202	242	......	321	96·0	1·4	2·6
22	Naphtha	73·9	0·689	84	101	110	124	138	153	168	186	205	231	274	331	343	96·0	1·0	3·0
23	Condensate	70·4	0·701	86	110	123	142	159	174	189	204	221	243	290	......	328	94·0	1·0	5·0
23	Naphtha	71·8	0·696	81	107	118	135	148	163	180	194	210	228	262	......	326	96·0	0·8	3·2
24	Condensate I	70·1	0·702	84	109	120	142	158	174	190	205	218	240	286	......	336	95·0	1·4	3·6
24	Condensate II	70·4	0·701	86	110	122	141	158	174	188	201	218	238	286	......	330	95·0	1·3	3·7
24	Condensate III	70·6	0·700	86	114	126	144	160	177	192	206	220	240	279	......	327	95·0	1·0	4·0
24	Naphtha	68·9	0·706	86	118	131	151	167	180	195	208	223	242	274	......	336	96·0	1·0	3·0
25	Condensate	71·8	0·696	84	106	116	133	149	164	181	197	216	240	293	......	352	95·0	1·3	3·7
25	Naphtha	73·9	0·698	91	102	116	132	147	160	177	190	206	230	269	325	348	96·5	1·3	2·2
27	Condensate I	58·7	0·744	106	148	166	186	202	217	234	250	273	308	404	......	462	95·5	4·0	0·5
27	Condensate II	61·8	0·732	100	124	152	172	192	208	223	239	259	290	374	......	438	94·0	5·2	0·8
27	Naphtha	68·1	0·709	85	112	124	142	160	178	194	212	232	264	336	......	420	94·0	4·2	1·8

*These samples were distilled at Turner Valley and results have not been converted for low barometric pressure there.

CONDENSABLE LIQUID IN THE STRIPPED GAS

The quantity of liquid in the stripped gas that may be condensed by activated charcoal, used in the manner described, varied from 0·072 to 0·300 gallon per thousand cubic feet (gal./M.) of gas in those wells that were tested. It is obvious that the amount from any particular well will vary as operating conditions change, but the extent of the variation and the factor having the most influence on it is not so clear. Although actual temperatures were not taken, it appeared, from general observation, that the colder the gas the smaller the amount of condensable liquid obtained by charcoal, if the quantity and distribution of frost on the separator and pipe-lines may be taken to indicate the relative temperature of the gas at different wells.

The amount of condensable liquid obtained at the different wells, when the distillation was made under 30 pounds per square inch pressure, agreed very well with the results obtained by officers of the Turner Valley Natural Gas Conservation Board, who distilled the absorbed material from the charcoal under atmospheric pressure and this in spite of the fact that entirely different samples were used, the work was done at different times by different analysts, and in some cases, the samples were taken under slightly different conditions. The agreement is considered to be sufficiently close to indicate the reliability of the method within reasonable limits of experimental error. The distillation method using a pressure of 30 pounds had an advantage in that the distillation was made in less time, and required less attention by the analyst.

CHARCOAL ABSORPTION VERSUS FRACTIONATION

As was stated above some duplicate samples of gas were taken, one of which was examined in Turner Valley by the Charcoal Absorption method and the other was shipped to Ottawa for fractionation in a Podbielniak column. The results obtained are compiled in Tables X to XIII. The object was two-fold: first, to check the accuracy of the Charcoal method; and second, to obtain data by which results obtained by either method of analysis might be calculated to equivalent terms in the other.

The comparative results obtained are considered to indicate that the charcoal absorption method used gave results that were reasonably accurate if the quantity of liquid hydrocarbons recovered in the receiver was greater than about 5 cubic centimetres.

In seeking to obtain a conversion factor it was assumed that all the pentanes and higher hydrocarbons obtained by fractionation would be recovered from the charcoal, and that in addition a certain proportion of the butanes would also be recovered. If it be assumed that "Y" equals the calculated gallons per M. cubic feet obtained by charcoal absorption; that "A" equals the pentanes and higher hydrocarbons obtained by fractionation expressed in gallons per M. cubic feet; that "B" equals the butanes obtained and expressed in the same way; that "X" equals the percentage of butanes which when added to the pentanes yields a sum equivalent to the amount of liquid obtained by charcoal absorption, then the proportion of butanes required may be found from the equation:

$$Y = A + \frac{XB}{100} \quad \text{or} \quad \frac{XB}{100} = Y - A$$

$$\text{and } X = 100 \left\{ \frac{Y - A}{B} \right\}$$

The above equation was used to calculate the results shown in Table XIII. The third column was obtained by substituting for "B" the amount of the total butanes found. The fourth column was obtained by using the amount of the normal butane only. The reason for calculating a result in these two ways is that the separation of the iso-butane from the normal butane is not nearly so clear-cut as the separation of propane from iso-butane, particularly when the older type of fractionation column is used, or when the amount of iso-butane present is small. The averages found from these comparative tests show that the gallons per M. cubic feet (gal./M.) obtained by the charcoal method are equivalent to the sum of the pentanes and higher hydrocarbons and 29 per cent of the total butanes found; or, to the sum of the pentanes and higher hydrocarbons and 45 per cent of the normal butane found, all expressed in terms of gallons per M. cubic feet.

TABLE X

Results Obtained by Distillation of Vapours from Charcoal at 32°F. Under 30 Pounds Per Square Inch Pressure

Test No.	Name and number of well	Volume of gas taken c.c.	Volume of liquid recovered c.c.	Imperial gallons per M. cu. ft.
21	Dalhousie, No. 7	14,250	11·8	5·462
20	Foothills, No. 1	10,550	8·2	5·100
24	Lowery, No. 1	9,400	5·6	3·895
10	Merland, No. 1	12,500	10·2	5·477
9	Mercury, No. 1	19,050	20·4	7·100
25	Royalite, No. 25	12,500	7·7	3·980
19	Wellington, No. 1	5,650*	2·3	2·655

*Quantity too small for satisfactory determination.

TABLE XI

Results Obtained by Fractionation in a Podbielniak Column

Test No.	Name and number of well	Methane plus %	Ethane %	Propane %	Butanes Iso- %	Butanes Normal %	Pentanes plus %
21	Dalhousie, No. 7	32·52	13·55	21·68	6·78	12·74	12·74
20	Foothills, No. 1	32·27	13·55	21·61	6·79	13·86	11·94
24	Lowery, No. 1	37·85	17·80	20·05	5·65	9·04	9·60
10	Merland, No. 1	31·35	11·14	24·10	8·13	14·75	10·54
6	Mercury, No. 1	26·10	12·70	21·10	7·70	15·70	16·70
25	Royalite, No. 25	38·60	15·28	19·30	5·63	10·72	10·45
19	Wellington, No. 1	34·25	17·47	20·54	6·51	9·93	11·30

TABLE XII

Results of Charcoal Test Compared with Results of Fractionation in Terms of Imperial Gallons per M. Cubic Feet

Test No.	Name and number of well	Charcoal test gal./M.	Pentanes plus gal./M.	Total butanes only gal./M.	Normal butane only gal./M.
21	Dalhousie, No. 7	5·462	3·860	5·192	3·347
20	Foothills, No. 1	5·100	3·595	5·493	3·645
24	Lowery, No. 1	3·895	2·889	3·917	2·378
10	Merland, No. 1	5·477	3·170	6·094	3·879
9 6	Mercury, No. 1	7·100	5·030	6·229	4·132
25	Royalite, No. 25	3·980	3·145	4·354	2·820
19	Wellington, No. 1	2·655*	3·399	4·393	2·619

*See note under Table X.

TABLE XIII

Calculated Composition of the Liquid from Charcoal Test in Terms of Pentane and Butane

Test No.	Name and number of well	Total pentanes plus percentage total butanes	Total pentanes plus percentage normal butane only
21	Dalhousie, No. 7	30·85	47·70
20	Foothills, No. 1	27·40	41·28
24	Lowery, No. 1	25·70	42·35
10	Merland, No. 1	37·86	59·50
9 6	Mercury, No. 1	34·20	49·95
25	Royalite, No. 25	19·17	29·61
19	Wellington, No. 1*	....	....
	Average	29·19	45·07

*See note under Table X.

EFFECT OF BACK PRESSURE ON THE STRIPPED GAS

The results shown in Table XIV indicate a very definite tendency for the stripped gas to carry larger quantities of condensable liquid as the closed-in pressures drop, although some figures appear in the table that do not fall precisely in line with this general tendency. This is particularly true for tests on Mercury No. 2 and Structure No. 1, which are higher than would be expected, and for the test on Royalite No. 23, which is lower than was expected. No definite reason can be offered for these results except that conditions underground for these wells may be different, that operating conditions were different, or that the samples were not representative of the total gas production. It seems probable, therefore, that pressure is the largest factor in determining the liquid content of the gas, and that the size of the gas flow is next in order of importance. After these two it is probable that the condition of the separation, the length of pipe-line, atmospheric temperature, and other factors would have a place.

COMPOSITION OF THE CONDENSATE

The composition of the condensate can only be estimated from the data obtained. It was believed that changes in composition would be more apparent in the propane and butane fractions than in other fractions, and that these fractions of the vapours from the heat treatment would show a similar variation. Accordingly, the amount of condensate which would be equivalent to 100 barrels of naphtha was calculated, and the vapours from the heat treatment of seven samples were fractionated in a Podbielniak column. The results are shown in Table XIV, and it is apparent in regard to the calculated results that the more volatile condensates predominate among the high-pressure wells. The sum of the propane and butane fractions was obtained from the fractionations of the seven samples and these show a very definite gradation from the high- to the low-pressure wells. Only two samples out of the seven do not fall exactly into line. One of these was a sample from Foothills No. 1 and the other, from Wellington No. 1. It should be pointed out that one of these two wells was operating under conditions that were slightly different from the others. The other wells were operating continuously at approximately two-thirds of their respective closed-in pressures, and Foothills No. 1 was operated at a much smaller proportion of its closed-in pressure. It is considered highly probable, therefore, that under similar operating conditions, the higher the rock pressure is, the more volatile the condensate will be. It appears, further, that this greater volatility is due to larger proportions of propane and butane, and as a consequence, the greater will be the loss on weathering this condensate by the present practice.

TABLE XIV

Pressure Versus Volatility

Test No.	Name and number of well	Closed-in pressures in lb. per sq. in.	Gal./M. in stripped gas	Condensate per 100 bbl. naphtha (calc.)	Propane plus butanes %
11	Sterling Pacific, No. 2	1,855	0·092	182·4	....
10	Merland, No. 1	1,810	0·072	191·2	47·0
18	Model, No. 1	1,635	0·121	....	....
7	Mercury, No. 2	1,580	0·176	139·5	....
20	Foothills, No. 1	1,550	0·154	134·9	42·3
5	Miracle, No. 1	1,540	0·145	147·4	....
9	Mercury, No. 1	1,465	0·183	166·7	44·5
22	East Crest, No. 3	1,360	0·203	144·2	....
8	Mayland, No. 6	1,350	0·206	151·6	....
21	Dalhousie, No. 7	1,255	0·167	155·6	41·2
1	Advance, No. 5A	1,190	0·190	148·0	....
2	Structure, No. 1	1,190	0·241	109·1	....
25	Royalite, No. 23	1,020	0·123	139·5	35·7
23	Richfield, No. 1	990	0·170	133·9	....
4	Okalta, No. 1	930	0·168	117·0	....
3	Home, No. 4	850	0·269	129·5	....
24	Lowery, No. 1	850	0·221	134·1	34·7
19	Wellington, No. 1	785	0·172	125·1	37·0

RECOVERY OF NAPHTHA

The general practice in the field is to run the production from the well through a separator and collect there as much condensed liquid as possible. A considerable pressure is usually maintained on the separator, about 250 to 300 pounds per square inch, and a certain amount of fixed gas is held in solution on that account. The condensate from the separator is transferred as often as necessary to a convenient storage tank. This tank as a rule is open to the atmosphere, although some of them are equipped with valves to maintain a small pressure. As a result, as soon as the condensate is transferred to the storage tank the dissolved gases evaporate rapidly and carry away some of the higher boiling hydrocarbons. It follows then that the naphtha that accumulates in the storage tanks is considerably less than the quantity of condensate collected.

EFFECT OF BACK PRESSURE ON THE NAPHTHA

The data obtained that related to the effect of pressure on the naphtha produced were not exhaustive and covered only two wells, namely, Model No. 1 and Advance No. 5A. The results are shown in Table XV. It is apparent that an increase in back pressure on the well decreases the amount of naphtha recovered. It should be pointed out that Model No. 1 was using a "step-drip" and not a Smith separator as is usual in the field. It will be observed that the pressure carried on this step-drip varied considerably during the tests. In regard to the tests on Advance No. 5A, it should be noted that the test at 500 pounds was of comparatively short duration and the production might have been influenced thereby.

TABLE XV

Pressure Versus Naphtha Recovered

Test No.	Name and number of well	Pressure, lb./sq. in.		30-lb. charcoal test gal./M.	Gas flow in M. cu. ft.	Naphtha recovered in bbl. per day
		On well	On separator			
12	Model, No. 1	600	90	0·186	3,109	125
13	" "	700	200	0·207	2,843	133
14	" "	800	195	0·114	2,714	120
16	" "	900	300	0·148	2,445	102
26	" "	1,000	225	0·163	2,250	91
17	" "	1,000	300	0·174	1,779	86
18	" "	1,100	250	0·121	1,465	61
15	Advance, No. 5A	500	265	0·300	4,850	92
1	" "	760	300	0·190	1,960	22
27	" "	1,150	300	0·119	1,020	13

EFFICIENCY OF THE SEPARATORS

From the data obtained it is possible to reach several conclusions that were not anticipated at the beginning of the work. For instance, it is possible to calculate the naphtha actually recovered as a percentage of the total condensable product, which for want of a more convenient term has been called the efficiency of the separators. This figure is obtained by calculating the gal./M. in the stripped gas in terms of barrels of liquid per day, adding the figure so obtained to the amount of condensate necessary to produce the naphtha actually recovered and expressing the naphtha as a percentage of the above sum. It is of course apparent that the result is not an efficiency of the separator alone: it is rather an overall efficiency for the well, its equipment, and the method of operating it. It assumes that all the liquid hydrocarbon absorbed by charcoal and condensable at 32°F. under 30 pounds pressure per square inch could be recovered. As has been pointed out above these liquid hydrocarbons are equivalent to all the pentanes and higher hydrocarbons plus 45 per cent of the normal butane present. It will be noticed that the figures for an overall efficiency so obtained show that the highest percentages are obtained at high-pressure wells and the lowest percentages at low-pressure wells. The value of the calculation lies in the simple summing up of the total production and in the suggestion that by more complete understanding of the problem, operating conditions or equipment might be altered or improved to yield larger returns from many of the wells.

ACKNOWLEDGMENTS

The writers wish to take this opportunity of expressing their grateful appreciation of the assistance given them in the field by all of the operating companies whose wells were examined. They not only co-operated most readily but in many cases gave freely of their time to further the work. The writers are particularly indebted to the Royalite Oil Company and the Department of the Interior for the use of laboratory accommodation and equipment, and the best thanks of the writers are accorded to Mr. S. G. Coultis and Col. F. M. Steel who represented the above organizations. Mr. C. W. Dingman, Supervisory Engineer for the Turner Valley Gas Conservation Board, offered much helpful advice during the investigation and placed a great deal of valuable information at the disposal of the writers. To all of these and to others who in any way assisted in the work grateful acknowledgment is made.

SUMMARY AND CONCLUSIONS

1. An attempt has been made to determine the approximate composition of all the products from some wells in Turner Valley, Alberta, with a view to estimating the character of the material present in the limestone, on the assumption that the material is homogeneous and that preferential volatilization does not take place.

2. The equipment and methods used in the work have been described.

3. The results obtained by distilling absorbed hydrocarbons from activated charcoal under a pressure of 30 pounds per square inch and condensing the vapours at a temperature of 32°F. have been compared with

the results obtained by a fractional distillation in a Podbielniak column. A relationship was found indicating that the gallons per M cubic feet of gas obtained by pressure distillation from charcoal is equal approximately to the gallons per M cubic feet of gas calculated from the pentane and higher hydrocarbon fraction plus 30 per cent of the total butane fraction, or to the pentane and higher hydrocarbon fraction plus 45 per cent of the normal butane fraction as determined by fractional distillation.

The above results have also been compared with the results obtained by ordinary distillation of the absorbed liquids from the charcoal. The results show reasonable conformity, but the former method is preferred on account of the distillation requiring less time and attention. The results of the pressure method are probably somewhat more accurate also.

4. It has been shown that the stripped gas from high-pressure wells contains a smaller proportion of condensable hydrocarbons than the stripped gas from low-pressure wells. As the pressure on the well is reduced the stripped gas tends to carry a larger proportion of the higher hydrocarbons.

5. Lowering the back pressure on the well increases the production of naphtha per day.

6. From a study of the total production a theoretical overall efficiency has been calculated. The efficiency is highest for high-pressure wells and lowest for low-pressure wells.

IX

GASOLINE SURVEY FOR 1932

H. McD. Chantler

The Division of Fuels and Fuel Testing of the Mines Branch has made at the Fuel Research Laboratories a continuous study of the gasoline sold in Canada for the past eight years, and annual reports[1] have been prepared from the results obtained. This report contains the results in detail of the analyses of 125 samples of gasoline collected[2] from wholesalers or distributors in sixteen cities during July, 1932. It also includes the detailed analyses of 134 samples of gasoline collected in seventeen cities during July, 1931, which analyses were not shown in the summarized results[3] for the years 1930 and 1931. It was found that the average gasoline sold in Canada was of good quality, and that the variation in quality was practically the same for both years, when judged by the volatility. On the basis of their knock-rating, the samples collected in 1932 may be divided into four grades. These grades would have average Octane numbers of 75, 68, 61, and 53 respectively. Only four samples had knock ratings below an Octane number of 57. The average Reid vapour pressure of the samples collected in 1932 was 7·4 pounds per square inch. The average sulphur content of the samples collected in 1931 was 0·05, which is considerably less than the amount usually accepted as the limit for good gasoline. A marked tendency has been observed during the past six years to market gasoline that has been coloured artificially.

Results of Laboratory Examination.—A statement of the methods of analysis used, as well as a general discussion of the significance of the laboratory tests, together with the relationship between these tests and the actual operation of the fuel in an engine will be found in the report of Gasoline Surveys for 1930 and 1931.[3]

The results of the laboratory examination of the gasoline tested in 1931 and 1932 are shown by cities in Table I, and the average analyses are summarized in Tables II and III. The average results obtained by the examination of samples for the ten years from 1923 to 1932 are shown in Table IV, and Figure 10 shows graphically the ranges of average distillation temperatures for the same ten years. In order to determine the variation in quality of the gasolines, the average of the 10 per cent of samples having the highest index numbers and the average of the 10 per cent having the lowest index numbers were calculated for 1931 and 1932, and the results are shown in Tables V, VI, VII, and VIII. Table IX shows the difference between the average index numbers of the maximum and minimum 10 per cent of the samples collected in the ten years 1923 to 1932. Table X shows the knock ratings of the samples collected in the city of Ottawa in 1931, and, also, the percentage of the five series of hydrocarbons present in gasoline for the same samples. Table XI gives a classification according to average knock ratings of samples in grade in

1932. Table XII shows the average knock rating of the samples of 20 different brands of gasoline sold by eight oil companies in 1932, arranged according to arbitrary grades. Table XIII gives a classification of the samples collected in 1932 according to results of the Reid vapour pressure determination. Table XIV gives a classification of the samples collected in 1931, according to results of the sulphur determination. Table XV shows the percentage of artificially coloured gasoline in the past six years.

Volatility.—It is interesting to compare the results obtained with those obtained in previous years. In Table IV are given the average results of 88 samples collected in Canada, presumably in 1916 and reported[4] by the laboratories of the Department of Inland Revenue; the average results of the following samples collected[2] in Canada from 1923 to 1932, inclusive. The number of samples collected in the respective years was: 48, 59, 73, 76, 83, 77, 84, 124, 134, and 123. When judged by the distillation range, which has been the ordinarily accepted standard, it will be observed that the gasoline sold in Canada in 1931 shows an average of good quality, with a lower volatility than that sold during the three previous years. This decrease in volatility in 1931 is equivalent to approximately 3° F. rise in the average volatility of the 20, 50, 70, and 90 per cent points in the distillation range. The average gasoline sold in Canada in 1932 was of good quality, having a higher volatility than the average gasoline sold in 1931, and having practically the same volatility as that sold during 1930.

Table IX shows the difference between the average index numbers of the maximum 10 per cent and minimum 10 per cent of the samples collected in Canada in the ten years 1923 to 1932. The difference between the two averages has been used previously for the purpose of comparison, as a measure of the variation in quality. It will be observed that the variation in quality during 1931 was less than in any previous year and that the variation in quality during 1932 was practically the same as the variation in quality in 1931.

In 1931, the group having the higher volatility has an average volatility less than corresponding groups examined in previous years. The group having the lower volatility has an average volatility higher than corresponding groups examined in previous years, with the exception of the year 1929, when the volatility of these groups was practically the same.

In 1932, the group having the higher volatility has an average volatility higher than the corresponding group examined in 1931. The group having the lower volatility has an average volatility higher than corresponding groups examined in any previous year. This decrease in the difference of the average volatility in the higher and lower groups in 1931 and 1932 indicates a growing tendency towards a more uniform grade of gasoline.

Knock Ratings.—In 1931, knock ratings were determined only for the samples collected in Ottawa. The average found for the 29 samples was 66 Octane number, the highest being 75 and the lowest 57 Octane number. Three samples, or 10·3 per cent, had an Octane number of 73 or over; 18 samples, or 62·1 per cent, had Octane numbers ranging between 72 and 65; and 8 samples, or 27·6 per cent, had Octane numbers ranging between

64 and 57. The majority of the samples of gasoline collected in Ottawa in 1931 had a relatively good knock rating.

In 1932, the knock rating was determined for all the samples collected in Canada. The average knock rating of the 123 samples was 65 Octane number, the highest being 77, and the lowest 46 Octane number. Fifteen samples, or 12·2 per cent, had an Octane number of 73 or over, with an average Octane number of 75; 50 samples, or 40·7 per cent, had Octane numbers ranging between 72 and 65, with an average Octane number of 68; 54 samples, or 43·9 per cent, had Octane numbers ranging between 64 and 57, with an average of Octane number 61; and only 4 samples, or 3·2 per cent, were below 57, the average being an Octane number of 53.

It will be observed, therefore, that the samples of gasoline collected in Canada in 1932 may be divided, when classified according to knock rating only, into four grades, as follows:

Grade I—Gasolines of high knock rating with Octane numbers of 73 and over.

Grade II—Gasolines of medium knock rating with Octane numbers between 72 and 65.

Grade III—Gasolines of low knock rating with Octane numbers between 64 and 57.

Grade IV—Gasolines of very low or poor knock rating with Octane numbers of 56 and under.

In Table XII is given the average knock rating of 20 different brands of gasoline sold in Canada by eight oil companies in 1932. It will be observed that these brands of gasoline fall into the first three grades defined above, and that the average Octane number for Grade I is 75, for Grade II, 68, and for Grade III, 61.

Tetra-ethyl lead, which is blended with gasoline to increase the knock rating, was used only in "Ethyl" brands of gasoline in 1931; but in 1932 tetra-ethyl lead was used in other brands of gasoline, as well as in the "Ethyl" brands; 38·2 per cent of the samples collected in 1932 contained tetra-ethyl lead.

Vapour Pressure.—The vapour pressure of a gasoline is a measure of its tendency to vaporize and is usually expressed in units of pressure at a stated temperature and method of determination, e.g., Reid vapour pressure at 100° F. in pounds per square inch. The method of determination generally used is that adopted by the American Society for Testing Materials as tentative method No. D323–31T.

The Reid vapour pressure of a gasoline should be high enough to enable the engine to start easily but not so high that vapour lock occurs in the fuel system. The Reid vapour pressure is used to predict the temperatures at which vapour lock will occur. Two rules[5] govern the vapour lock problem, namely, "keep heat out of fuel system", and "keep propane out of the gasoline". Propane is a hydrocarbon, one per cent of which increases the vapour pressure of the gasoline in which it occurs by more than two pounds per square inch at 100° F. Vapour lock does not occur in all engines under similar conditions with fuels of the same vapour pressure, and on that account the Reid vapour pressure should not exceed 10 pounds per square inch, in the writer's opinion.

A classification of the samples collected in 1932 according to the results of the Reid vapour pressure determination is shown in Table XIII. This table shows that the average Reid vapour pressure was 7·4 pounds per square inch, and that 17·8 per cent of the samples had Reid vapour pressures of 6 pounds or less per square inch; 55·4 per cent had between 8 and 6·1 pounds per square inch, 22·7 per cent between 10 and 8·1 pounds per square inch, and only 4·1 per cent of the 123 samples had Reid vapour pressures over 10 pounds per square inch.

Sulphur Content.—The average sulphur content of the gasoline samples collected in Canada during 1930 was 0·07 per cent. This result was not reported in Memorandum Series No. 45 containing the results for that year. In 1931 the average sulphur content of the gasoline samples collected in Canada was 0·05 per cent and as indicated in Table XIV, only 6·7 per cent of the 134 samples tested had a sulphur content exceeding 0·1 per cent. In 1931 the average sulphur content of samples from the western provinces was 0·08 per cent, and the average for the eastern provinces was 0·04 per cent.

Colour.—Since 1927 there has been a gradually increasing tendency to colour artificially the gasolines being put on the market. According to the samples examined in the annual survey, the percentage of artificially coloured gasolines sold in Canada during the past six years was as follows: 10 per cent in 1927, 13 per cent in 1928, 18 per cent in 1929, 26 per cent in 1930, 34 per cent in 1931, and 52 per cent in 1932.

Summary and Conclusions

The gasoline surveys for 1931 and 1932 comprised the collection and analyses of 257 samples. The samples for each survey were collected in July, 134 coming from seventeen different cities in 1931, and 123 from sixteen cities in 1932. As these centres are widely separated and are distribution centres throughout the country, the samples taken may be accepted as representative of the gasoline sold in Canada at that time.

The analysis of the samples has shown that the average gasoline sold during 1931 and 1932 was of good quality. The average gasoline in 1931 was slightly less volatile than that in the three previous years, and the average 1932 gasoline was more volatile than in 1931 and had practically the same volability as that of 1930.

The variations in quality of the average gasoline during 1931 and 1932 was practically the same for both of these years, and this variation was less than in any preceding year. This indicates a tendency towards a more uniform grade of gasoline.

In 1931 only samples from Ottawa were examined for knock rating, but in 1932 all the samples collected in Canada were tested for knock rating. Thirty-eight per cent of the samples of gasoline collected throughout Canada in 1932 contained tetra-ethyl lead added to increase their knock ratings.

According to knock rating only, the 1932 gasoline samples may be divided into four grades, namely, Grade I with an average Octane number of 75, Grade II with an average of 68, Grade III with an average of 61,

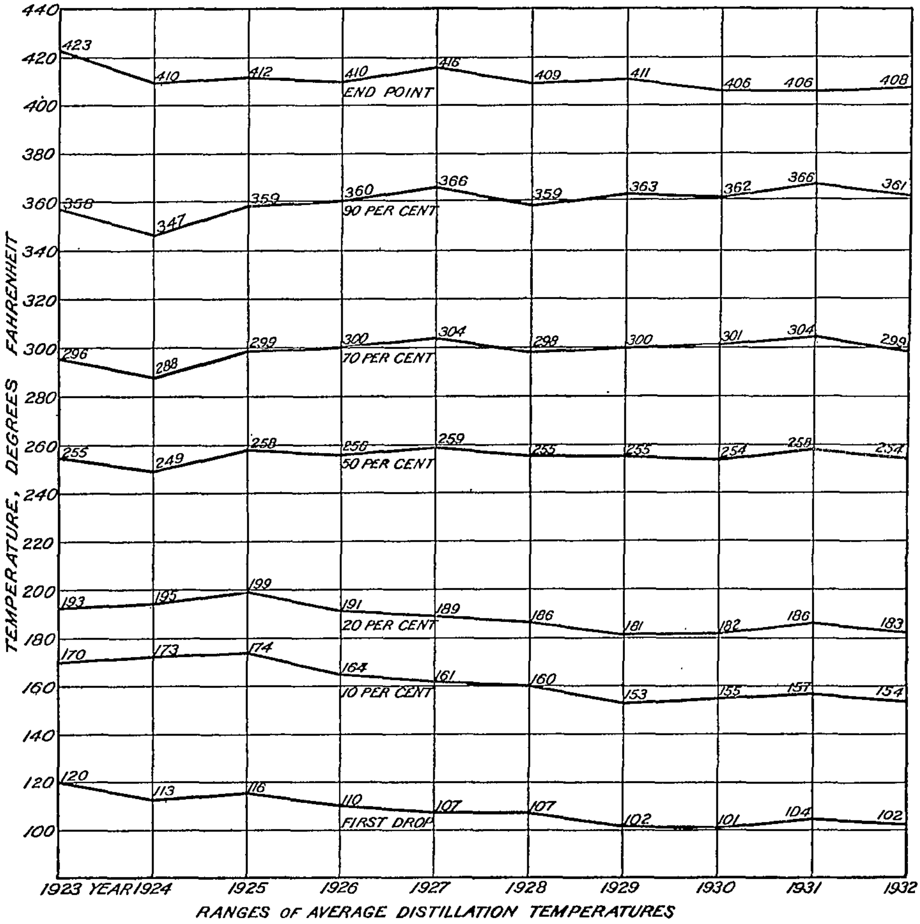

Figure 10

and Grade IV with an average Octane number of 53. Only four samples, or 3·2 per cent of the samples collected in 1932, had poor knock ratings, or were in Grade IV with knock ratings below 57 Octane number.

The average Reid vapour pressure of the 1932 gasoline samples was 7·4 pounds per square inch. Only 4·1 per cent of the samples had Reid vapour pressures exceeding 10 pounds per square inch.

The average sulphur content of the gasoline samples collected in 1931 was 0·05 per cent. This amount is considerably less than the amount usually accepted as the limit for good gasoline, viz. 0·10 per cent. In 1931 the average sulphur content of gasoline obtained in the western provinces was somewhat higher than for samples from the eastern provinces.

A marked tendency is shown during the past six years towards marketing of an increased number of artificially coloured gasolines. Fifty-two per cent of the gasolines collected in 1932 were artificially coloured.

LIST OF REFERENCES

1. Reports of Investigations of Fuels and Fuel Testing, 1923, 1924, 1925, 1926, 1927, 1928, 1929, and combined 1930 and 1931.
2. The hearty support and co-operation of the Department of Pensions and National Health in taking samples is gratefully acknowledged.
3. Report of Investigations of Fuels and Fuel Testing, 1930 and 1931.
4. Department of Inland Revenue, Bulletin No. 362 ("Gasoline").
5. Bridgeman, O. C., White, H. S., and Gary, F. B.—Oil and Gas Journal, November 19th, 1931, pages 22 and 101.

TABLE I

Gasoline Survey Analyses for 1931 and 1932, by Cities

Sample Number	Distillation Range							Re-covery	Residue	Distil-lation loss	Index No. °F.	Specific gravity	Degrees A.P.I.	Sulphur	Vapour pressure	Colour	Octane Number at 212° F. and 600 r.p.m.
	1st drop °F.	10% °F.	20% °F.	50% °F.	70% °F.	90% °F.	End point °F.										
HALIFAX, N.S.																	
1931— 1	106	158	189	275	324	384	419	97·0	1·2	1·8	1749	0·750	57·2	0·05	....	Blue......	—
2	105	157	187	251	287	340	394	97·0	1·2	1·8	1616	0·736	60·8	0·03	....	+28	—
3	110	175	204	262	300	353	395	97·0	1·3	1·7	1689	0·745	58·4	0·02	....	+27	—
4	108	172	200	257	295	348	395	97·0	1·1	1·9	1667	0·743	58·9	0·01	....	Red......	—
5	106	167	201	269	310	359	394	97·0	1·0	2·0	1700	0·745	58·4	0·02	....	+20	—
1931—Average	107	166	196	263	303	357	399	97·0	1·2	1·8	1684	0·744	58·7	0·03	....	....	—
1932— 1	108	168	190	242	280	336	387	98·5	1·1	0·4	1603	0·742	59·2	....	6·4	+27	64
2	102	161	193	258	296	349	390	98·0	1·0	1·0	1647	0·746	58·2	....	7·1	Green....	68
3	100	152	176	250	298	363	410	98·5	0·6	0·9	1649	0·747	57·9	....	6·3	Red......	74
4	94	140	168	242	292	358	407	98·0	1·1	0·9	1607	0·738	60·2	....	7·8	Blue......	69
5	108	161	194	274	322	385	419	98·0	1·3	0·7	1755	0·759	54·9	....	5·6	Green....	66
1932—Average	102	156	184	253	298	358	403	98·2	1·0	0·8	1652	0·746	58·2	....	6·6	....	68
SAINT JOHN, N.B.																	
1931— 6	114	174	200	264	297	353	396	97·0	1·0	2·0	1684	0·747	57·9	0·04	....	+15	—
7	102	155	185	254	294	350	396	96·5	1·2	2·3	1634	0·737	60·5	0·04	....	+18	—
8	106	165	200	263	302	353	396	97·0	1·2	1·8	1679	0·745	58·4	0·02	....	+19	—
9	98	168	193	270	320	380	418	97·0	1·3	1·7	1749	0·749	57·4	0·04	....	+ 6	—
10	100	150	183	259	302	355	396	97·0	1·2	1·8	1645	0·741	59·5	0·08	....	Yellow...	—
1931—Average	104	162	192	262	303	358	401	96·9	1·2	1·9	1678	0·744	58·7	0·04	....	....	—
1932— 6	107	163	191	255	292	345	388	98·5	1·2	0·3	1634	0·744	58·7	....	5·7	Red......	74
7	110	182	208	263	296	342	393	98·5	1·1	0·4	1684	0·751	56·9	....	5·0	+22	63
8	99	163	195	260	294	347	390	98·0	1·0	1·0	1649	0·745	58·4	....	7·7	+21	64
9	100	147	179	258	303	362	408	98·0	1·2	0·8	1657	0·747	57·9	....	7·4	Blue......	69
10	97	146	179	252	296	353	400	97·5	1·2	1·3	1626	0·741	59·5	....	7·3	Yellow...	64
1932—Average	103	160	190	258	296	350	396	98·1	1·1	0·8	1650	0·746	58·2	....	6·6	....	67

TABLE I—Continued

Gasoline Survey Analyses for 1931 and 1932, by Cities—Continued

Sample Number	Distillation Range							Re-covery	Residue	Distil-lation loss	Index No. °F.	Specific gravity	Degrees A.P.I.	Sulphur	Vapour pressure	Colour	Octane Number at 212° F. and 600 r.p.m.
	1st drop °F.	10% °F.	20% °F.	50% °F.	70% °F.	90% °F.	End point °F.										
QUEBEC, QUE.																	
1931—11	113	177	204	263	301	352	397	97·0	1·1	1·9	1694	0·745	58·4	0·03	....	+26	—
12	117	169	193	249	284	342	408	97·0	1·6	1·4	1645	0·729	62·6	0·02	....	+30	—
13	111	159	189	260	302	357	394	96·5	1·2	2·3	1661	0·743	58·9	0·06	....	Red	—
14	115	170	195	257	296	348	395	97·0	1·3	1·7	1661	0·745	58·4	0·02	....	Red	—
15	115	177	205	263	299	352	398	97·0	1·2	1·8	1694	0·745	58·4	0·02	....	+27	—
16	108	148	172	239	284	342	383	97·0	1·2	1·8	1568	0·738	60·2	0·06	....	Red	—
1931—Average	113	167	193	255	294	349	396	96·0	1·3	1·8	1654	0·741	59·5	0·04	....	....	—
1932—11	90	142	172	252	300	360	406	97·0	1·2	1·8	1632	0·741	59·3	....	8·3	Green	66
12	108	172	196	248	278	328	392	98·5	1·2	0·3	1614	0·744	58·7	....	5·8	+28	63
13	94	146	176	246	292	360	420	97·5	1·1	1·4	1640	0·735	61·0	....	9·0	+30	60
14	102	163	192	258	292	346	388	93·0	1·2	0·8	1639	0·748	57·7	....	6·9	Red	76
15	100	160	194	266	301	353	397	98·0	1·2	0·8	1671	0·749	57·4	....	7·3	Green	68
16	102	148	176	246	284	334	374	98·5	1·1	0·4	1562	0·739	60·0	....	7·3	Pink	67
1932—Average	99	155	184	253	291	347	396	97·9	1·2	0·9	1626	0·743	58·9	....	7·4	....	67
MONTREAL, QUE.																	
1931—17	101	142	168	251	310	388	420	95·5	1·2	3·3	1679	0·733	61·5	0·02	....	Red	—
18	97	135	160	250	307	373	416	96·5	1·2	2·3	1641	0·734	61·3	0·05	....	Green	—
19	109	166	195	272	315	375	414	97·0	1·2	1·8	1737	0·749	57·4	0·05	....	Red	—
20	107	162	192	274	319	379	412	96·0	1·3	2·7	1738	0·747	57·9	0·07	....	+14	—
21	104	153	182	264	315	382	419	96·5	1·4	2·1	1715	0·742	59·2	0·03	....	Blue	—
22	106	157	185	262	314	379	422	97·0	1·2	1·8	1719	0·755	55·9	0·03	....	Orange	—
23	112	158	186	249	290	344	382	97·0	1·1	1·9	1609	0·743	58·9	0·07	....	+29	—
24	108	158	179	239	286	345	382	97·0	1·1	1·9	1589	0·740	59·7	0·01	....	Red	—
25	110	157	191	276	325	385	417	96·0	1·5	2·5	1751	0·754	56·2	0·04	....	Blue	—
26	103	164	195	271	317	372	411	97·5	1·3	1·2	1730	0·753	56·4	0·06	....	+23	—
1931—Average	106	155	183	261	310	372	410	96·6	1·3	2·1	1691	0·745	58·4	0·04	....	....	—
1932—17	94	136	166	249	301	367	407	96·0	1·3	2·7	1626	0·736	60·8	....	10·1	Red	77
18	105	148	180	262	313	376	405	98·0	1·3	0·7	1684	0·743	58·9	....	8·5	Green	66
19	104	162	192	275	318	372	410	98·5	1·2	0·3	1729	0·755	55·9	....	6·0	Red	76
20	100	158	194	273	319	372	408	98·0	1·4	0·6	1724	0·752	56·7	....	6·7	Green	69
21	108	162	192	260	300	356	410	98·0	1·1	0·9	1680	0·746	58·2	....	5·7	+30	61

1932—22	99	150	178	247	294	360	421	97·0	1·4	1·6	1650	0·736	60·8	....	8·7	+30	60
23	104	162	194	272	316	374	414	98·0	1·4	0·6	1732	0·758	55·2	....	7·1	Blue......	68
24	107	154	184	271	318	372	407	98·0	1·2	0·8	1706	0·751	56·9	....	7·1	Red......	77
25	102	155	189	278	322	374	410	98·5	1·2	0·3	1728	0·754	56·2	....	7·1	Green....	70
26	105	162	188	258	292	355	410	98·5	1·0	0·5	1665	0·745	58·4	....	5·9	+30	63
1932—Average	103	155	186	264	309	368	410	97·9	1·2	0·9	1692	0·748	57·7	....	7·3	....	69

OTTAWA, ONT.

1931—27	113	178	214	281	324	380	416	97·5	1·2	1·3	1793	0·759	54·9	0·03	....	+14	60
28	102	133	153	244	304	372	417	96·5	1·4	2·1	1623	0·731	62·1	0·05	....	Green....	66
29	103	149	177	259	312	386	420	96·0	1·4	2·6	1703	0·739	60·0	0·04	....	Red......	69
30	100	138	160	235	284	362	408	96·0	1·0	3·0	1587	0·725	63·7	0·05	....	+26	61
31	103	153	182	268	317	376	415	96·5	1·2	2·3	1711	0·745	58·4	0·05	....	+19	66
32	108	150	178	261	310	375	419	97·0	1·3	1·7	1693	0·742	59·2	0·05	....	Red......	73
33	102	150	178	262	310	373	418	98·0	0·6	1·4	1691	0·741	59·5	0·06	....	+12	65
34	101	153	185	270	324	390	412	95·5	1·3	3·2	1734	0·737	60·5	0·01	....	+28	57
35	99	140	165	241	290	350	387	96·0	1·2	2·8	1573	0·734	61·3	0·02	....	Green....	72
36	102	164	194	272	319	375	412	97·0	1·2	1·8	1736	0·749	57·4	0·06	....	+20	65
37	95	132	154	235	287	351	397	96·0	1·2	2·8	1556	0·725	63·7	0·05	....	+21	69
38	100	126	146	235	291	359	393	96·0	0·9	3·1	1550	0·721	64·8	0·05	....	+21	69
39	112	161	196	272	314	370	415	97·0	1·4	1·6	1728	0·749	57·4	0·03	....	Red......	72
40	97	136	169	265	316	381	416	96·5	0·9	2·6	1683	0·740	59·7	0·05	....	Green....	67
41	110	158	192	273	320	378	418	97·0	1·0	2·0	1739	0·748	57·7	0·07	....	+14	66
42	117	164	187	257	302	365	412	98·0	1·4	0·6	1687	0·750	57·2	0·04	....	Red......	74
43	100	146	172	251	305	376	414	96·0	1·3	2·7	1664	0·734	61·3	0·01	....	+11	58
44	113	164	195	276	324	382	420	97·0	1·3	1·7	1761	0·759	54·9	0·05	....	Orange...	66
45	103	154	186	269	320	383	418	96·5	1·8	1·7	1730	0·748	57·7	0·04	....	Blue......	63
46	105	154	183	267	316	386	414	97·0	1·3	1·7	1720	0·741	59·5	0·04	....	Red......	58
47	96	145	172	249	300	370	420	96·5	1·0	2·5	1656	0·736	60·8	0·01	....	+21	58
48	105	147	170	238	283	341	382	97·0	1·0	2·0	1561	0·739	60·0	0·05	....	Red......	75
49	107	155	186	248	291	348	385	96·5	0·8	2·7	1613	0·741	59·5	0·06	....	+19	65
50	100	158	190	276	326	387	419	95·5	1·4	3·1	1756	0·753	56·4	0·06	....	Blue......	70
51	96	158	184	269	323	384	413	95·5	0·9	3·6	1731	0·749	57·4	0·05	....	+27	65
52	110	158	182	256	304	374	419	96·5	1·0	2·5	1693	0·741	59·5	0·02	....	+22	63
53	113	165	194	265	314	374	411	97·0	1·4	1·6	1723	0·748	57·7	0·05	....	+13	65
54	116	180	209	280	319	372	414	97·5	1·2	1·3	1774	0·755	55·9	0·03	....	Red......	71
55	97	132	156	244	294	360	397	96·0	0·7	3·3	1583	0·726	63·4	0·06	....	+13	70
1931—Average	104	152	180	259	308	372	410	96·6	1·2	2·2	1681	0·742	59·2	0·04	....	....	66
1932—27	95	150	182	260	308	374	403	97·0	1·0	2·0	1677	0·740	59·7	....	8·0	+26	61
28	91	148	177	245	290	353	403	96·5	1·3	2·2	1616	0·731	62·1	....	9·7	+30	61
29	92	136	162	244	294	357	389	97·0	1·1	1·9	1582	0·732	61·8	....	10·3	Red......	77
30	94	149	177	262	310	370	404	98·0	1·4	0·6	1672	0·746	58·2	....	7·9	Green....	67
31	102	152	185	259	310	372	404	98·0	1·3	0·7	1682	0·743	58·9	....	7·9	+30	59
32	106	159	188	267	312	369	412	98·5	1·4	0·1	1707	0·752	56·7	....	5·8	Red......	75
33	110	161	194	259	296	357	404	98·0	1·4	0·6	1671	0·748	57·7	....	5·4	Green....	62
34	99	150	184	267	311	367	406	98·5	1·5	0·0	1685	0·751	56·9	....	6·6	Green....	68
35	91	130	155	229	273	330	383	97·0	1·2	1·8	1500	0·750	57·2	....	9·9	Green....	71
36	95	140	168	238	284	362	414	97·5	1·2	1·3	1606	0·736	60·8	....	8·6	+27	64
37	112	163	192	270	315	369	412	98·5	1·4	0·1	1721	0·754	56·2	....	5·1	Red......	76

TABLE I—Continued

Gasoline Survey Analyses for 1931 and 1932, by Cities—Continued

Sample Number	Distillation Range							Recovery	Residue	Distillation loss	Index No. °F.	Specific gravity	Degrees A.P.I.	Sulphur	Vapour pressure	Colour	Octane Number at 212° F. and 600 r.p.m.
	1st drop °F.	10% °F.	20% °F.	50% °F.	70% °F.	90% °F.	End point °F.										
OTTAWA, ONT.—*Concluded*																	
1932—38	103	155	187	269	314	371	409	98·0	1·2	0·8	1705	0·750	57·2	....	7·0	Green....	69
39	110	170	198	267	308	365	412	98·0	1·0	1·0	1720	0·749	57·4	....	5·4	+30	61
40	97	142	172	246	296	367	417	97·0	1·4	1·6	1640	0·741	59·5	....	8·6	+22	64
41	101	148	176	253	303	363	409	98·0	1·2	0·8	1652	0·744	58·7	....	7·2	Red......	73
42	102	152	182	265	316	374	406	97·0	1·4	1·6	1695	0·745	58·4	....	7·8	Blue......	68
43	100	149	180	247	291	356	418	97·5	1·2	1·3	1641	0·736	60·8	....	8·3	+30	58
44	106	150	180	248	292	354	420	97·5	1·2	1·3	1644	0·738	60·2	....	7·9	Red......	59
45	106	149	172	246	296	362	417	97·0	1·4	1·6	1642	0·740	59·7	....	7·5	Green....	64
46	96	143	169	242	290	358	417	98·0	1·4	0·6	1619	0·737	60·5	....	9·1	+28	65
47	110	154	180	246	285	330	373	98·5	1·2	0·3	1568	0·741	59·5	....	6·3	Red......	74
48	104	150	176	246	284	330	374	98·5	1·0	0·5	1560	0·740	59·7	....	7·1	+30	66
49	107	151	178	240	286	355	416	97·5	1·2	1·3	1626	0·740	59·7	....	7·9	+30	61
50	101	153	176	241	286	353	416	98·0	1·3	0·7	1625	0·740	59·7	....	7·9	+24	60
51	102	158	194	272	318	371	414	98·0	1·2	0·8	1727	0·757	55·4	....	6·9	Blue......	67
52	111	169	198	269	312	374	439	98·0	1·4	0·6	1761	0·750	57·2	....	5·7	+20	60
53	95	154	188	272	318	372	408	98·0	1·3	0·7	1712	0·750	57·2	....	7·9	Red......	77
54	98	154	190	276	320	377	408	97·5	1·3	1·2	1725	0·751	56·9	....	7·2	Green....	70
55	106	162	191	260	298	354	405	98·5	1·2	0·3	1670	0·746	58·2	....	6·0	+23	63
1932—Average	101	152	181	255	301	361	407	97·7	1·3	1·0	1657	0·744	58·7	....	7·5	....	66
TORONTO, ONT.																	
1931—56	94	138	162	234	285	357	405	96·5	1·0	2·5	1581	0·727	63·1	0·01	....	+30	—
57	129	188	215	281	330	385	412	98·0	1·4	0·6	1811	0·755	55·9	0·03	....	+27	—
58	96	141	172	260	317	380	406	97·0	1·3	1·7	1676	0·745	58·4	0·04	....	+20	—
59	99	157	191	272	314	371	401	97·5	1·4	2·1	1706	0·742	59·2	0·07	....	Green....	—
60	103	158	184	253	294	355	396	96·0	1·1	1·9	1640	0·735	61·0	0·04	....	+12	—
61	97	160	192	275	321	377	407	96·5	1·0	2·5	1732	0·744	58·7	0·06	....	+ 7	—
62	104	162	189	254	300	371	413	97·0	1·3	1·7	1689	0·739	60·0	0·01	....	Blue......	—
63	115	193	239	323	349	374	405	98·0	1·1	0·9	1883	0·759	54·9	0·01	....	+20	—
64	107	168	202	274	317	369	398	97·0	1·2	1·8	1728	0·756	55·7	0·12	....	+28	—
65	92	139	164	236	285	354	407	97·0	1·3	1·7	1585	0·726	63·4	0·01	....	+30	—
1931—Average	104	161	191	266	311	369	405	97·1	1·2	1·7	1703	0·743	58·9	0·04	....	....	—

1932—56...............	92	144	177	258	307	367	396	97·5	1·2	1·3	1649	0·739	60·0	....	10·2	Green....	67
57...............	98	160	194	268	309	366	405	98·0	1·4	0·6	1702	0·743	58·9	....	7·3	+16	60
58...............	96	141	167	238	283	359	417	98·0	1·2	0·8	1605	0·733	61·5	....	8·9	+13	65
59...............	96	154	189	264	309	367	405	97·5	1·2	1·3	1688	0·739	60·0	....	9·2	Green....	68
60...............	109	163	189	261	308	367	411	98·5	1·2	0·3	1699	0·750	57·2	....	5·6	Blue......	68
61...............	96	149	179	260	305	361	397	98·0	1·1	0·9	1651	0·742	59·2	....	9·3	+23	66
62...............	106	161	197	274	319	376	417	98·0	1·2	0·8	1744	0·758	55·2	....	6·6	Blue......	68
63...............	98	154	188	264	307	364	406	98·0	1·2	0·8	1683	0·740	59·7	....	8·4	Green....	68
64...............	104	157	187	259	300	359	409	98·0	1·2	0·8	1671	0·738	60·2	....	7·2	Red......	57
1932—Average.........	100	154	185	261	305	365	407	97·9	1·2	0·9	1677	0·742	59·2	....	8·1	....	65

HAMILTON, ONT.

1931—66...............	107	165	190	257	305	371	412	97·0	1·2	1·8	1700	0·743	58·9	0·01	....	Red......	—
67...............	96	146	177	264	320	384	414	96·0	1·0	3·0	1705	0·738	60·2	0·03	....	Green....	—
68...............	105	147	172	241	291	360	407	96·5	1·2	2·3	1618	0·730	62·3	0·02	....	+27	—
69...............	98	139	166	240	292	353	390	96·5	1·1	2·4	1580	0·734	61·3	0·03	....	Green....	—
70...............	104	159	191	268	321	384	413	96·5	1·1	2·4	1736	0·740	59·7	0·03	....	+30	—
71...............	110	170	199	262	306	364	398	97·0	1·1	1·9	1699	0·731	62·1	0·05	....	+30	—
72...............	110	166	196	265	308	365	400	97·0	1·3	1·7	1700	0·740	59·7	0·05	....	Red......	—
73...............	100	159	193	274	319	374	404	97·0	1·4	1·6	1723	0·745	58·4	0·08	....	+14	—
74...............	112	164	192	274	325	387	425	96·5	1·5	2·0	1767	0·761	54·4	0·05	....	Orange...	—
75...............	101	160	185	254	304	384	418	96·5	1·3	2·2	1705	0·739	60·0	0·03	....	Blue......	—
1931—Average.........	104	157	186	260	309	373	408	96·7	1·2	2·1	1693	0·740	59·7	0·04	....	....	—
1932—65...............	97	142	177	258	305	368	402	97·5	1·2	1·3	1652	0·739	60·0	....	9·2	Green....	67
66...............	101	141	165	237	277	331	386	98·0	1·4	0·6	1537	0·734	61·3	....	7·9	Green....	69
67...............	103	142	171	242	287	360	413	97·5	1·2	1·3	1615	0·735	61·0	....	8·0	+30	64
68...............	99	155	189	263	307	365	405	98·0	1·2	0·8	1684	0·739	60·0	....	7·9	Green....	68
69...............	98	142	174	256	306	369	401	97·0	1·0	2·0	1648	0·735	61·0	....	10·7	Blue......	71
70...............	100	147	179	257	307	368	403	98·0	1·0	1·0	1661	0·739	60·0	....	8·5	+30	61
71...............	107	155	182	249	291	338	382	98·5	1·0	0·5	1597	0·741	59·5	....	6·3	+16	66
72...............	112	168	198	274	320	373	418	98·0	1·4	0·6	1751	0·757	55·4	....	6·8	Blue......	67
73...............	106	161	194	264	306	361	406	98·5	1·2	0·3	1692	0·743	58·9	....	6·6	Green....	68
74...............	106	168	198	268	307	362	406	98·0	1·0	1·0	1709	0·745	58·4	....	6·2	+27	62
1932—Average.........	103	152	183	257	301	360	402	97·9	1·2	0·9	1655	0·741	59·5	....	7·8	....	66

LONDON, ONT.

1931—76...............	96	144	174	264	319	383	412	96·0	1·2	2·8	1696	0·737	60·5	0·05	....	Green....	—
77...............	105	171	206	276	314	366	404	97·0	1·1	1·9	1737	0·742	59·2	0·03	....	+22	—
78...............	108	174	206	273	312	365	400	97·0	0·9	2·1	1730	0·741	59·5	0·03	....	+27	—
79...............	98	160	187	256	305	379	417	96·5	1·3	2·2	1704	0·741	59·5	0·02	....	Blue......	—
80...............	114	172	203	273	315	370	402	97·5	1·1	1·4	1735	0·748	57·7	0·09	....	Red......	—
81...............	101	156	191	276	327	388	420	96·5	1·2	2·3	1758	0·754	56·2	0·07	....	Blue......	—
82...............	102	166	201	270	308	362	400	97·0	1·2	1·8	1707	0·740	59·7	0·04	....	+29	—
83...............	100	174	207	272	310	361	400	97·0	1·0	2·0	1724	0·741	59·5	0·04	....	+30	—
1931—Average.........	103	164	197	270	314	372	407	96·8	1·1	2·1	1724	0·743	58·9	0·05	....	....	—

TABLE I—Continued

Gasoline Survey Analyses for 1931 and 1932, by Cities—Continued

Sample Number	Distillation Range							Recovery	Residue	Distillation loss	Index No. °F.	Specific gravity	Degrees A.P.I.	Sulphur	Vapour pressure	Colour	Octane Number at 212° F. and 600 r.p.m.
	1st drop °F.	10% °F.	20% °F.	50% °F.	70% °F.	90% °F.	End point °F.										
LONDON, ONT.—*Concluded*																	
1932—75	95	144	176	252	303	363	399	97·5	0·8	1·7	1637	0·737	60·5	....	9·2	Green	68
76	106	160	191	260	305	359	410	98·0	1·3	0·7	1685	0·740	59·7	....	6·6	+14	59
77	96	142	170	243	290	365	420	97·5	1·0	1·5	1630	0·734	61·3	....	8·6	+22	64
78	102	164	195	270	314	366	405	97·5	1·3	1·2	1714	0·744	58·7	....	7·8	Green	68
79	108	157	188	262	310	366	410	98·0	1·0	1·0	1693	0·748	57·7	....	6·3	Blue	66
80	99	152	180	258	306	362	396	97·5	1·1	1·4	1654	0·745	58·4	....	8·5	+21	66
81	126	183	211	282	325	377	423	98·0	1·1	0·9	1801	0·770	52·3	....	3·6	Blue	65
82	104	162	194	269	311	366	412	97·5	1·0	1·5	1714	0·744	58·7	....	7·6	Green	66
1932—Average	105	158	188	262	308	366	409	97·7	1·1	1·2	1691	0·745	58·4	....	7·3	....	65
FORT WILLIAM, ONT.																	
1931—84	106	167	201	268	308	360	401	97·0	1·2	1·8	1705	0·740	59·7	0·04	....	+27	—
85	132	196	218	274	312	359	402	98·0	1·2	0·8	1761	0·746	58·2	0·04	....	+28	—
86	107	160	187	250	288	343	386	97·5	1·3	1·2	1614	0·734	61·3	0·03	....	Red	—
87	103	168	204	271	311	363	406	97·5	1·3	1·2	1723	0·743	58·9	0·05	....	+26	—
88	95	140	167	233	276	340	387	97·0	1·2	1·8	1543	0·721	64·8	0·03	....	+26	—
1931—Average	109	166	196	259	299	353	396	97·4	1·2	1·4	1669	0·737	60·5	0·04	....	....	—
1932—83	100	150	186	259	300	360	402	97·5	1·1	1·9	1657	0·738	60·2	....	8·5	+21	59
84	107	158	188	254	300	354	397	98·0	1·4	0·6	1651	0·740	59·7	....	6·9	Red	72
85	98	151	181	255	299	364	402	97·5	1·2	1·3	1652	0·735	61·0	....	9·1	Green	66
86	104	152	187	259	301	362	402	97·5	1·3	1·2	1663	0·738	60·2	....	7·4	Blue	66
87	100	139	169	250	303	367	398	97·0	1·0	2·0	1626	0·734	61·3	....	9·9	+23	63
1932—Average	102	150	182	256	301	361	400	97·4	1·2	1·4	1650	0·737	60·5	....	8·4	....	65
WINNIPEG, MAN.																	
1931—89	102	164	200	269	309	364	400	97·0	1·1	1·9	1706	0·739	60·0	0·04	....	+29	—
90	104	163	198	271	311	364	398	96·5	1·3	2·2	1705	0·740	59·7	0·03	....	+28	—
91	108	168	198	270	311	365	400	97·0	1·1	1·9	1712	0·740	59·7	0·05	....	+27	—
92	108	150	172	238	288	363	418	97·0	1·5	1·5	1629	0·728	62·9	0·07	....	+30	—
93	98	142	168	250	298	360	398	96·0	1·0	3·0	1616	0·727	63·1	0·03	....	+28	—
94	107	146	164	227	280	368	436	97·0	1·4	1·6	1621	0·719	65·3	0·12	....	+27	—
1931—Average	105	156	183	254	300	364	408	96·8	1·2	2·0	1665	0·732	61·8	0·06	....	....	—

1932—88	104	156	188	260	305	359	403	97·5	1·2	1·3	1671	0·738	60·2	....	7·7	+23	60
89	103	154	185	260	303	359	404	97·5	1·0	1·5	1665	0·740	59·7	....	7·2	+20	60
90	97	120	136	202	262	363	409	97·0	1·1	1·9	1492	0·712	67·2	....	11·3	+30	64
91	100	154	186	258	301	362	404	97·5	1·2	1·3	1665	0·739	60·0	....	8·1	Green....	64
92	98	150	183	256	300	357	404	98·5	1·1	0·4	1650	0·739	60·0	....	7·7	Blue......	64
93	99	155	189	264	305	365	406	98·0	1·0	1·0	1684	0·742	59·2	....	7·4	+26	60
1932—Average	100	148	178	250	296	361	405	97·7	1·1	1·2	1638	0·735	61·0	....	8·2	....	62
BRANDON, MAN.																	
1931—95	91	143	170	234	275	338	387	97·0	1·1	1·9	1547	0·724	63·9	0·04	....	+30	—
96	103	161	197	270	312	364	403	97·0	1·2	1·8	1707	0·740	59·7	0·05	....	+28	—
97	102	160	191	266	309	365	405	97·0	1·1	1·9	1696	0·739	60·0	0·05	....	+17	—
98	95	145	171	237	280	345	399	97·0	1·2	1·8	1577	0·725	63·7	0·03	....	+27	—
99	109	168	201	271	313	363	402	97·5	1·3	1·2	1718	0·742	59·2	0·05	....	+27	—
1931—Average	100	155	186	256	298	355	399	97·1	1·2	1·7	1649	0·734	61·3	0·04	....	....	—
1932—94	100	152	188	260	304	362	412	98·0	1·3	0·7	1678	0·742	59·2	....	7·0	+20	59
95	101	158	188	260	302	360	403	97·5	1·0	1·5	1671	0·740	59·7	....	8·2	+29	58
96	98	154	186	259	301	358	403	97·5	1·1	1·4	1661	0·740	59·7	....	7·4	+27	59
97	95	154	186	260	304	362	404	97·5	1·1	1·4	1670	0·738	60·2	....	7·6	+19	61
98	102	159	193	267	312	371	420	98·0	1·2	0·8	1722	0·746	58·2	....	6·0	+29	58
1932—Average	99	155	188	261	305	363	408	97·7	1·1	1·2	1680	0·741	59·5	....	7·2	....	59
REGINA, SASK.																	
1931—100	98	149	178	260	310	373	421	97·0	1·3	1·7	1691	0·735	61·0	0·07	....	+18	—
101	104	148	170	237	293	377	425	97·5	1·4	1·1	1650	0·731	62·1	0·13	....	+28	—
102	94	144	177	265	316	378	417	97·0	1·2	1·8	1697	0·737	60·5	0·07	....	+14	—
103	100	148	177	267	318	381	419	97·0	1·3	1·7	1710	0·737	60·5	0·06	....	+ 5	—
104	113	166	197	276	322	375	405	97·5	1·3	1·2	1741	0·750	57·2	0·10	....	+28	—
1931—Average	102	151	180	261	312	377	417	97·2	1·3	1·5	1698	0·738	60·2	0·09	....	....	—
1932— 99	112	176	199	260	295	350	402	98·0	1·0	1·0	1682	0·743	58·9	....	4·2	+ 8	56
100	112	166	195	258	300	364	424	98·0	1·2	0·8	1707	0·741	59·5	....	4·9	+16	56
101	108	170	197	261	313	386	451	98·0	1·2	0·8	1778	0·744	58·7	....	5·3	+29	46
102	110	168	196	254	290	350	404	98·5	1·2	0·3	1662	0·740	59·7	....	5·3	+16	57
103	93	146	180	270	317	366	409	98·0	1·2	0·8	1688	0·739	60·0	....	7·7	Blue......	61
1932—Average	107	165	193	261	303	363	418	98·1	1·2	0·7	1703	0·741	59·5	....	5·5	....	55
SASKATOON, SASK.																	
1931—105	97	139	170	257	309	378	416	96·5	1·2	2·3	1669	0·732	61·8	0·07	....	+26	—
106	110	107	193	250	288	350	405	97·5	1·3	1·2	1656	0·740	59·7	0·03	....	Red......	—
107	100	152	179	256	307	372	414	97·0	1·2	1·8	1680	0·734	61·3	0·04	....	+13	—
108	98	146	176	261	310	378	416	97·0	1·4	1·6	1687	0·734	61·3	0·08	....	+18	—
109	100	142	163	224	284	380	424	97·0	1·2	1·8	1617	0·725	63·7	0·13	....	+27	—
110	100	157	187	270	323	376	401	97·5	1·2	1·3	1714	0·742	59·2	0·05	....	Red......	—
111	105	163	194	276	324	377	407	97·5	1·2	1·3	1741	0·749	57·4	0·09	....	+29	—
1931—Average	101	154	180	256	306	373	412	97·2	1·2	1·6	1681	0·737	60·5	0·07	....	....	..

TABLE I—Concluded

Gasoline Survey Analyses for 1931 and 1932, by Cities—Concluded

Sample Number	Distillation Range							Re-covery	Residue	Distil-lation loss	Index No. °F.	Specific gravity	Degrees A.P.I.	Sulphur	Vapour pressure	Colour	Octane Number at 212° F and 600 r.p.m.
	1st drop °F.	10% °F.	20% °F.	50% °F.	70% °F.	90% °F.	End point °F.										
CALGARY, ALTA.																	
1931—112..............	91	124	140	184	217	274	375	96·5	1·3	2·2	1314	0·699	70·9	0·16	....	+22	—
113..............	104	153	179	256	307	363	404	97·5	1·2	1·3	1662	0·744	58·7	0·09	....	+27	—
114..............	96	141	163	226	283	378	422	96·5	1·3	2·2	1613	0·724	63·9	0·12	....	+29	—
115..............	112	170	198	277	322	373	406	98·0	1·3	0·7	1746	0·750	57·2	0·10	....	+30	—
116..............	106	162	192	253	294	360	409	97·5	1·2	1·3	1670	0·749	57·4	0·08	....	+30	—
1931—Average.........	102	150	174	239	285	350	403	97·2	1·3	1·5	1601	0·733	61·5	0·11	....	....	—
1932—104..............	110	156	160	204	235	291	387	98·0	1·2	0·8	1441	0·719	65·3	....	5·9	+30	61
105..............	98	152	178	258	311	370	419	98·0	1·1	0·9	1688	0·744	58·7	....	7·3	+29	60
106..............	99	147	170	235	293	368	420	98·0	1·0	1·0	1633	0·734	61·3	....	7·6	+30	58
107..............	98	150	174	238	292	366	420	98·0	1·2	0·8	1640	0·736	60·8	....	6·9	+30	58
108..............	98	148	168	230	278	358	416	98·0	1·0	1·0	1598	0·726	63·4	....	7·5	+29	54
1932—Average.........	101	150	172	233	282	351	412	98·0	1·1	0·9	1600	0·732	61·8	....	7·0	....	58
EDMONTON, ALTA.																	
1931—117..............	102	150	173	229	272	347	402	97·0	1·2	1·8	1573	0·726	63·4	0·12	....	+30	—
118..............	103	157	183	260	309	365	405	97·5	1·2	1·3	1679	0·746	58·2	0·07	....	Red......	—
119..............	100	148	172	231	274	354	404	97·0	1·1	1·9	1583	0·728	62·9	0·11	....	+30	—
120..............	102	153	179	256	306	363	403	97·5	1·1	1·4	1660	0·744	58·7	0·10	....	+26	—
121..............	101	155	180	258	306	364	400	97·0	1·2	1·8	1663	0·744	58·7	0·07	....	Red......	—
122..............	94	138	162	225	282	380	425	97·0	1·5	1·5	1612	0·724	63·9	0·12	....	+26	—
123..............	110	163	193	275	323	374	407	97·5	1·2	1·3	1735	0·749	57·4	0·07	....	+28	—
124..............	108	165	192	254	294	357	405	97·0	1·0	2·0	1667	0·748	57·7	0·05	....	+27	—
1931—Average.........	103	154	179	249	296	363	406	97·2	1·2	1·6	1647	0·739	60·0	0·09	....	....	—
1932—109..............	107	152	166	200	228	281	375	98·0	1·1	0·9	1402	0·718	65·6	....	6·1	+30	60
110..............	99	148	176	252	290	346	412	98·0	1·1	0·9	1624	0·732	61·8	....	7·8	+30	59
111..............	93	133	151	225	361	493	531	97·5	1·2	1·3	1894	0·741	59·5	....	9·3	+26	57
112..............	102	148	172	237	288	371	415	97·5	1·3	1·2	1631	0·734	61·3	....	6·7	+30	59
113..............	107	151	168	215	254	334	412	98·6	1·3	0·2	1534	0·724	63·9	....	6·9	+30	57
1932—Average.........	102	146	167	226	284	365	429	97·9	1·2	0·9	1617	0·730	62·3	....	7·4	....	58

VANCOUVER, B.C.

1931—125	101	158	193	270	320	388	412	96·0	1·0	3·0	1741	0·755	55·9	0·06	....	Violet....	—
126	107	165	193	253	293	362	408	97·0	1·2	1·8	1674	0·748	57·7	0·07	....	+28	—
127	105	162	190	252	294	360	404	97·0	1·0	2·0	1662	0·749	57·4	0·10	....	+26	—
128	104	158	169	252	294	360	396	96·0	1·1	2·9	1649	0·743	58·9	0·05	....	+30	—
129	97	163	191	250	284	329	372	97·0	1·0	2·0	1589	0·745	58·4	0·05	....	Red......	—
130	99	163	192	258	297	356	397	97·0	1·0	2·0	1663	0·748	57·7	0·09	....	+29	—
1931—Average	102	162	191	256	297	359	398	96·7	1·0	2·3	1663	0·748	57·7	0·07	....	....	—
1932—114	106	156	186	252	296	368	414	98·5	1·2	0·3	1672	0·749	57·4	....	6·3	Violet....	67
115	106	158	185	244	284	356	421	98·5	1·3	0·2	1648	0·746	58·2	....	6·8	+30	65
116	105	160	186	244	280	356	420	98·5	1·2	0·3	1646	0·747	57·9	....	6·0	Red......	75
117	100	149	178	256	302	374	416	98·0	1·2	0·8	1675	0·746	58·2	....	8·1	Green....	76
118	98	147	178	246	288	356	407	98·0	1·2	0·8	1622	0·745	58·4	....	8·2	Yellow...	71
119	102	149	179	252	293	361	409	98·0	1·2	0·8	1643	0·747	57·9	....	6·9	Orange...	68
1932—Average	103	153	182	249	291	362	414	98·3	1·2	0·5	1651	0·747	57·9	....	7·1	....	70

VICTORIA, B.C.

1931—131	105	162	193	256	296	363	410	97·0	1·1	1·9	1680	0·749	57·4	0·06	....	+30	—
132	104	162	194	253	296	354	396	97·0	1·2	1·8	1655	0·747	57·9	0·09	....	+27	—
133	100	156	188	254	293	362	392	96·5	1·0	2·5	1645	0·745	58·4	0·04	....	+27	—
134	106	160	190	252	292	362	408	97·0	0·9	2·1	1664	0·747	57·9	0·10	....	+30	—
1931—Average	104	160	191	254	294	360	402	96·9	1·0	2·1	1661	0·747	57·9	0·07	....	....	—
1932—120	103	158	186	250	290	368	421	98·0	1·2	0·8	1673	0·749	57·4	....	6·4	+30	66
121	104	149	178	249	289	370	417	98·0	1·4	0·6	1652	0·746	58·2	....	7·8	Green....	75
122	98	145	176	245	289	358	416	97·5	1·3	1·2	1629	0·745	58·4	....	8·2	Yellow...	71
123	102	150	180	254	300	368	417	98·5	1·2	0·3	1669	0·748	57·7	....	6·9	Orange...	68
1932—Average	102	151	180	249	292	366	418	98·0	1·3	0·7	1656	0·747	57·9	....	7·3	....	70

TABLE II

Average of Gasoline Survey Analyses for Cities for 1931

City	Distillation Range							Re-covery	Residue	Distil-lation loss	Index No. °F.	Specific gravity	Degrees A.P.I.	Sulphur	Octane Number. at 212°F. and 600 rpm.
	1st drop °F.	10% °F.	20% °F.	50% °F.	70% °F.	90% °F.	End point °F.								
Halifax, N.S.	107	166	196	263	303	357	399	97·0	1·2	1·8	1684	0·744	58·7	0·03	—
Saint John, N.B.	104	162	192	262	303	358	401	96·9	1·2	1·9	1678	0·744	58·7	0·04	—
Quebec, Que.	113	167	193	255	294	349	396	96·9	1·3	1·8	1654	0·741	59·5	0·04	—
Montreal, Que.	106	155	183	261	310	372	410	96·6	1·3	2·1	1691	0·745	58·4	0·04	—
Ottawa, Ont.	104	152	180	259	308	372	410	96·6	1·2	2·2	1681	0·742	59·2	0·04	66
Toronto, Ont.	104	161	191	266	311	369	405	97·1	1·2	1·7	1703	0·743	58·9	0·04	—
Hamilton, Ont.	104	157	186	260	309	373	408	96·7	1·2	2·1	1693	0·740	59·7	0·04	—
London, Ont.	103	164	197	270	314	372	407	96·8	1·1	2·1	1724	0·743	58·9	0·05	—
Fort William, Ont.	109	166	196	259	299	353	396	97·4	1·2	1·4	1669	0·737	60·5	0·04	—
Winnipeg, Man.	105	156	183	254	300	364	408	96·8	1·2	2·0	1665	0·732	61·8	0·06	—
Brandon, Man.	100	155	186	256	298	355	399	97·1	1·2	1·7	1649	0·734	61·3	0·04	—
Regina, Sask.	102	151	180	261	312	377	417	97·2	1·3	1·5	1698	0·738	60·2	0·09	—
Saskatoon, Sask.	101	154	180	256	306	373	412	97·2	1·2	1·6	1681	0·737	60·5	0·07	—
Calgary, Alta.	102	150	174	239	285	350	403	97·2	1·3	1·5	1601	0·733	61·5	0·11	—
Edmonton, Alta.	103	154	179	249	296	363	406	97·2	1·2	1·6	1647	0·739	60·0	0·09	—
Vancouver, B.C.	102	162	191	256	297	359	398	96·7	1·0	2·3	1663	0·748	57·7	0·07	—
Victoria, B.C.	104	160	191	254	294	360	402	96·9	1·0	2·1	1661	0·747	57·9	0·07	—
Average* (134 samples)	104	157	186	258	304	366	406	96·9	1·3	1·8	1677	0·741	59·5	0·05	—

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

TABLE III

Average of Gasoline Survey Analyses for Cities for 1932

City	Distillation Range							Re-covery	Residue	Distil-lation loss	Index No. °F.	Specific gravity	Dgreees A.P.I.	Vapour [redacted] pressure	Octane Number at 212°F. and 600 rpm.
	1st drop °F.	10% °F.	20% °F.	50% °F.	70% °F.	90% °F.	End point °F.								
Halifax, N.S.	102	156	184	253	298	358	403	98·2	1·0	0·8	1652	0·746	58·2	6·6	68
Saint John, N.B.	103	160	190	258	296	350	396	98·1	1·1	0·8	1650	0·746	58·2	6·6	67
Quebec, Que.	99	155	184	253	291	347	396	97·9	1·2	0·9	1626	0·743	58·9	7·4	67
Montreal, Que.	103	155	186	264	309	368	410	97·9	1·2	0·9	1692	0·748	57·7	7·3	69
Ottawa, Ont.	101	152	181	255	301	361	407	97·7	1·3	1·0	1657	0·744	58·7	7·5	66
Toronto, Ont.	100	154	185	261	305	365	407	97·9	1·2	0·9	1677	0·742	59·2	8·1	65
Hamilton, Ont.	103	152	183	257	301	360	402	97·9	1·2	0·9	1655	0·741	59·5	7·8	66
London, Ont.	105	158	188	262	308	366	409	97·7	1·1	1·2	1691	0·745	58·4	7·3	65
Fort William, Ont.	102	150	182	256	301	361	400	97·4	1·2	1·4	1650	0·737	60·5	8·4	65
Winnipeg, Man.	100	148	178	250	296	361	405	97·7	1·1	1·2	1638	0·735	61·0	8·2	62
Brandon, Man.	99	155	188	261	305	363	408	97·7	1·1	1·2	1680	0·741	59·5	7·2	59
Regina, Sask.	107	165	193	261	303	363	418	98·1	1·2	0·7	1703	0·741	59·5	5·5	55
Calgary, Alta.	101	150	172	233	282	351	412	98·0	1·1	0·9	1600	0·732	61·8	7·0	58
Edmonton, Alta.	102	146	167	226	284	365	429	97·9	1·2	0·9	1617	0·730	62·3	7·4	58
Vancouver, B.C.	103	153	182	249	291	362	414	98·3	1·2	0·5	1651	0·747	57·9	7·1	70
Victoria, B.C.	102	151	180	249	292	366	418	98·0	1·3	0·7	1656	0·747	57·9	7·3	70
Average* (123 samples)	102	154	183	254	299	361	408	97·9	1·2	0·9	1659	0·742	59·2	7·4	65

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

TABLE IV

Annual Averages of Gasoline Survey Analyses for Canada

City	Distillation Range							Re-covery	Residue and distillation loss	Index No. °F.	Specific gravity	Degrees A.P.I.	Sulphur	Vapour pressure	Octane Number at 212°F. and 600 rpm.
	1st drop °F.	10% °F.	20% °F.	50% °F.	70% °F.	90% °F.	End point °F.								
1916	125	170	192	237	270	330	380	....	....	1579	0·732	61·8	....	....	—
1923	120	170	193	255	296	358	423	97·1	2·9	1695	0·737	60·5	....	....	—
1924	113	173	195	249	288	347	410	97·4	2·6	1662	0·736	60·8	....	....	—
1925	116	174	199	258	299	359	412	97·0	3·0	1701	0·739	60·0	....	....	—
1926	110	164	191	256	300	360	410	97·4	2·6	1681	0·739	60·0	....	....	—
1927	107	161	189	259	304	366	416	97·0	3·0	1693	0·741	59·5	....	....	—
1928	107	160	186	255	298	359	409	97·3	2·7	1667	0·737	60·5	....	....	—
1929	102	153	181	255	300	363	411	97·0	3·0	1663	0·736	60·8	....	....	—
1930	101	155	182	254	301	362	406	97·2	2·8	1660	0·741	59·5	0·07	....	—
1931	104	157	186	258	304	366	406	96·9	3·1	1677	0·741	59·5	0·05	....	—
1932	102	154	183	254	299	361	408	97·9	2·1	1659	0·742	59·2	....	7·4	65

TABLE V

Ten per cent of Samples Having Maximum Index Numbers* in 1931

Sample No. 1931	Index No. °F.	Distillation Range							Recovery	Residue	Distillation loss
		1st drop °F.	10% °F.	20% °F.	50% °F.	70% °F.	90% °F.	End point °F.	%	%	%
63...........	1883	115	193	239	323	349	374	405	98·0	1·1	0·9
57...........	1811	129	188	215	281	330	385	412	98·0	1·4	0·6
27...........	1793	113	178	214	281	324	380	416	97·5	1·2	1·3
54...........	1774	116	180	209	280	319	372	414	97·5	1·2	1·3
74...........	1767	112	164	192	274	325	387	425	96·5	1·5	2·0
85...........	1761	132	196	218	274	312	359	402	98·0	1·2	0·8
44...........	1761	113	164	195	276	324	382	420	97·0	1·3	1·7
81...........	1758	101	156	191	276	327	388	420	96·5	1·2	2·3
50...........	1756	100	158	190	276	326	387	419	95·5	1·4	3·1
25...........	1751	110	157	191	276	325	385	417	96·0	1·5	2·5
1...........	1749	106	158	189	275	324	384	419	97·0	1·2	1·8
9...........	1749	98	168	193	270	320	380	418	97·0	1·3	1·7
115...........	1746	112	170	198	277	322	373	406	98·0	1·3	0·7
Average......	1774	112	171	203	280	325	380	415	97·1	1·3	1·6

TABLE VI

Ten per cent of Samples Having Minimum Index Numbers* in 1931

Sample No. 1931	Index No. °F.	Distillation Range							Recovery	Residue	Distillation loss
		1st drop °F.	10% °F.	20% °F.	50% °F.	70% °F.	90% °F.	End point °F.	%	%	%
112...........	1314	91	124	140	184	217	274	375	96·5	1·3	2·2
88...........	1543	95	140	167	233	276	340	387	97·0	1·2	1·8
95...........	1547	91	143	170	234	275	338	387	97·0	1·1	1·9
38...........	1550	100	126	146	235	291	359	393	96·0	0·9	3·1
37...........	1556	95	132	154	235	287	351	397	96·0	1·2	2·8
48...........	1561	105	147	170	238	283	341	382	97·0	1·0	2·0
16...........	1568	108	148	172	239	284	342	383	97·0	1·2	1·8
35...........	1573	99	140	165	241	290	350	387	96·0	1·2	2·8
117...........	1573	102	150	173	229	272	347	402	97·0	1·2	1·8
98...........	1577	95	145	171	237	280	345	399	97·0	1·2	1·8
69...........	1580	98	139	166	240	292	353	390	96·5	1·1	2·4
56...........	1581	94	138	162	234	285	357	405	96·5	1·0	2·5
55...........	1583	97	132	156	244	294	360	397	96·0	0·7	3·3
Average......	1547	98	139	162	233	279	343	391	96·6	1·1	2·3

* The index number is the sum of the following points in the distillation range: 10%, 20%, 50% 70%, 90%, and the end point.

TABLE VII

Ten per cent of Samples Having Maximum Index Numbers* in 1932

Sample No. 1932	Index No. °F.	Distillation Range							Recovery	Residue	Distillation loss
		1st drop °F.	10% °F.	20% °F.	50% °F.	70% °F.	90% °F.	End point °F.	%	%	%
111	1894	93	133	151	225	361	493	531	97·5	1·2	1·3
81	1801	126	183	211	282	325	377	423	98·0	1·1	0·9
101	1778	108	170	197	261	313	386	451	98·0	1•2	0·8
52	1761	111	169	198	269	312	374	439	98·0	1·4	0·6
5	1755	108	161	194	274	322	385	419	98·0	1·3	0·7
72	1751	112	168	198	274	320	373	418	98·0	1·4	0·6
62	1744	106	161	197	274	319	376	417	98·0	1·2	0·8
23	1732	104	162	194	272	316	374	414	98·0	1·4	0·6
19	1729	104	162	192	275	318	372	410	98·5	1·2	0·3
25	1728	102	155	189	278	322	374	410	98·5	1·2	0·3
51	1727	102	158	194	272	318	371	414	98·0	1·2	0·8
54	1725	98	154	190	276	320	377	408	97·5	1·3	1·2
Average	1760	106	161	192	269	322	386	430	98·0	1·3	0·7

TABLE VIII

Ten per cent of Samples Having Minimum Index Numbers* in 1932

Sample No. 1932	Index No. °F.	Distillation Range							Recovery	Residue	Distillation loss
		1st drop °F.	10% °F.	20% °F.	50% °F.	70% °F.	90% °F.	End point °F.	%	%	%
109	1402	107	152	166	200	228	281	375	98·0	1·1	0·9
104	1441	110	156	168	204	235	291	387	98·0	1·2	0·8
90	1492	97	120	136	202	262	363	409	97·0	1·1	1·9
35	1500	91	130	155	229	273	330	383	97·0	1·2	1·8
113	1534	107	151	168	215	254	334	412	98·5	1·3	0·2
66	1537	101	141	165	237	277	331	386	98·0	1·4	0·6
48	1560	104	150	176	246	284	330	374	98·5	1·0	0·5
16	1562	102	148	176	246	284	334	374	98·5	1·1	0·4
47	1568	110	154	180	246	285	330	373	98·5	1·2	0·3
29	1582	92	136	162	244	294	357	389	97·0	1·1	1·9
71	1597	107	155	182	249	291	338	382	98·5	1·0	0·5
108	1598	98	148	168	230	278	358	416	98·0	1·0	1·0
Average	1531	102	145	167	229	270	332	388	98·0	1·1	0·9

TABLE IX

Difference between Maximum and Minimum Index Numbers*

Year	1923	1924	1925	1926	1927	1928	1929	1930	1931	1932
Maximum, 10%.....	1791	1806	1821	1815	1823	1791	1773	1787	1774	1760
Minimum, 10%......	1500	1428	1497	1524	1518	1488	1503	1471	1547	1531
Difference..........	291	378	324	291	305	303	270	316	227	229

* The index number is the sum of the following points in the distillation range: 10%, 20%, 50%, 70%, 90%, and the end point.

TABLE X

Knock Ratings and Chemical Analyses of Samples from Ottawa in 1931

Sample No. 1931	Octane No. at 212°F. and 600 r.p.m.	Hydrocarbons				Toluene value %	Index No. °F.
		Unsaturates %	Aromatics %	Naphthenes %	Paraffins %		
48...........	75	6	10	34	50	19·7*	1561
42...........	74	4	8	32	56	16·8*	1687
32...........	73	5	6	29	60	14·3*	1693
35...........	72	21	21	26	32	31·7	1573
39...........	72	4	8	26	62	15·3*	1728
54...........	71	4	10	29	57	18·1*	1774
55...........	70	9	10	24	57	17·8	1583
50...........	70	11	14	27	48	23·0	1756
29...........	69	8	11	26	55	19·1*	1703
38...........	69	8	8	24	60	15·6	1550
37...........	69	7	7	25	61	14·7	1556
40...........	67	10	14	26	50	22·5	1683
41...........	66	8	11	26	55	19·1	1739
28...........	66	10	7	25	58	15·3	1623
44...........	66	14	19	25	42	28·1	1761
31...........	66	7	10	27	56	18·2	1711
33...........	65	6	9	25	60	16·5	1691
49...........	65	6	8	33	53	17·5	1613
51...........	65	6	11	26	57	18·7	1731
36...........	65	5	11	27	57	18·8	1736
53...........	65	5	11	28	56	19·0	1723
52...........	63	9	9	26	56	17·3	1693
45...........	63	13	15	22	50	23·1	1730
30...........	61	3	4	30	63	12·1	1587
27...........	60	9	14	22	55	21·3	1793
46...........	58	8	9	24	59	16·6	1720
47...........	58	4	4	28	64	11·8	1656
43...........	58	4	5	29	62	13·1	1664
34...........	57	8	8	21	63	14·9	1734
Average......	66	8	10	27	55	18·2	1681

* These samples contain tetra-ethyl lead and therefore the toluene value does not indicate the anti-knock value of the fuel.

TABLE XI

Classification of the 1932 Samples according to Four Arbitrary Octane Number Grades

City	Grade I 73 and above Octane No.		Grade II 72 to 65 Octane No.		Grade III 64 to 57 Octane No.		Grade IV 56 and below Octane No.		Total number of samples
	Number of Samples	Average Octane Number	Number of Samples	Average Octane Number	Number of Samples	Average Octane Number	Number of Samples	Average Octane Number	
Halifax	1	74	3	68	1	64	....	....	5
Saint John	1	74	1	69	3	64	....	....	5
Quebec	1	76	3	67	2	62	....	....	6
Montreal	3	77	4	68	3	61	....	....	10
Ottawa	6	75	9	68	14	61	....	....	29
Toronto	....	....	7	67	2	59	....	....	9
Hamilton	....	....	7	68	3	62	....	....	10
London	....	....	6	67	2	62	....	....	8
Fort William	....	....	3	68	2	61	....	....	5
Winnipeg	....	....	....	....	6	62	....	....	6
Brandon	....	....	....	....	5	59	....	....	5
Regina	....	....	....	....	2	59	3	53	5
Calgary	....	....	....	....	4	59	1	54	5
Edmonton	....	....	....	....	5	58	....	....	5
Vancouver	2	76	4	68	....	....	....	....	6
Victoria	1	75	3	68	....	....	....	....	4
Number of samples in grade	15	....	50	....	54	....	4	....	123
Per cent of total samples	12·2	....	40·7	....	43·9	....	3·2	....	100
Average Octane No. for grade	....	75	....	68	....	61	....	53	....

Knock rating (Octane No.), average all samples	65
Knock rating (Octane No.), highest sample	77
Knock rating (Octane No.), lowest sample	46

TABLE XII

Average Knock Ratings of 20 Different Brands of Gasoline sold by 8 Companies in 1932, Arranged According to Arbitrary Grades

Company	Grade I Octane Number 73 and above		Grade II Octane Number 72 to 65		Grade III Octane Number 64 to 57	
	Number of Samples of Company's Brand	Average Octane Number	Number of Samples of Company's Brand	Average Octane Number	Number of Samples of Company's Brand	Average Octane Number
A	5	75	11	69	5	60
B	2	74	8	68	3	60
C	2	77	6	67	4	58
D	1	75	1	68	9	61
E	2	77	5	68	3	63
F	1	74	6	67	....	....
G	....	....	2	70	4	64
H	....	....	6	67	....	....
Total number of samples tested	13	....	45	....	28	
Average Octane Number	....	75	....	68	....	61

TABLE XIII

Classification of Samples according to Results of Reid Vapour Pressure Determination in 1932

City	Reid Vapour Pressure, pounds per square inch									
	12·0 to 11·1	11·0 to 10·1	10·0 to 9·1	9·0 to 8·1	8·0 to 7·1	7·0 to 6·1	6·0 to 5·1	5·0 to 4·1	4·0 to 3·1	Total
Halifax	......	......	......	......	2	2	1	......	......	5
Saint John	......	......	......	......	3	......	1	1	......	5
Quebec	......	......	......	2	2	1	1	......	......	6
Montreal	......	1	......	2	3	1	3	......	......	10
Ottawa	......	1	3	3	12	4	6	......	......	29
Toronto	......	1	2	2	2	1	1	......	......	9
Hamilton	......	1	1	1	3	4	......	......	......	10
London	......	......	1	2	2	2	......	......	1	8
Fort William	......	......	2	1	1	1	......	......	......	5
Winnipeg	1	......	......	1	4	......	......	......	......	6
Brandon	......	......	......	1	2	1	1	......	......	5
Regina	......	......	......	......	1	......	2	2	......	5
Calgary	......	......	......	......	3	1	1	......	......	5
Edmonton	......	......	1	......	1	3	......	......	......	5
Vancouver	......	......	......	2	......	3	1	......	......	6
Victoria	......	......	......	1	1	2	......	......	......	4
Total	1	4	10	18	42	26	18	3	1	123
Per cent of total	0·8	3·3	8·1	14·6	34·2	21·2	14·6	2·4	0·8	100

Reid Vapour pressure, average all samples	7·4
Reid Vapour pressure, highest sample	11·3
Reid Vapour pressure, lowest sample	3·6

TABLE XIV

Classification of Samples according to Results of Sulphur Determination in 1931

City	Sulphur, per cent														
	0·16	0·13	0·12	0·11	0·10	0·09	0·08	0·07	0·06	0·05	0·04	0·03	0·02	0·01	Total
Halifax	....	....	....	....	....	....	....	....	....	1	....	1	2	1	5
Saint John	....	....	....	....	....	....	1	....	....	....	3	....	1	....	5
Quebec	....	....	....	....	....	....	....	....	2	....	....	1	3	....	6
Montreal	....	....	....	....	....	....	....	2	1	2	1	2	1	1	10
Ottawa	....	....	....	....	....	....	....	1	5	11	4	3	2	3	29
Toronto	....	....	1	....	....	....	....	1	1	....	2	1	....	4	10
Hamilton	....	....	....	....	....	....	1	....	....	3	....	4	1	1	10
London	....	....	....	....	....	1	....	1	....	1	2	2	1	....	8
Fort William	....	....	....	....	....	....	....	....	....	1	2	2	....	....	5
Winnipeg	....	....	1	....	....	....	....	1	....	1	1	2	....	....	6
Brandon	....	....	....	....	....	....	....	....	....	3	1	1	....	....	5
Regina	....	1	....	....	1	....	....	2	1	....	....	....	....	....	5
Saskatoon	....	1	....	....	....	1	1	1	....	1	1	1	....	....	7
Calgary	1	....	1	....	1	1	1	....	....	....	....	....	....	....	5
Edmonton	....	....	2	1	1	....	....	3	....	1	....	....	....	....	8
Vancouver	....	....	....	....	1	1	....	1	1	2	....	....	....	....	6
Victoria	....	....	....	....	1	1	....	....	1	....	1	....	....	....	4
Total	1	2	5	1	5	5	4	13	12	27	18	20	11	10	134
Per cent of total	0·7	1·5	3·7	0·8	3·7	3·7	3·0	9·7	9·0	20·2	13·4	14·9	8·2	7·5	100

Sulphur, per cent, average all samples	0·05
Sulphur, per cent, highest sample	0·16
Sulphur, per cent, lowest sample	0·01

TABLE XV

Percentage of Artificially Coloured Gasoline in Different Years

Year	Artificially coloured gasolines	Year	Artificially coloured gasolines
	%		%
1927	10	1930	26
1928	13	1931	34
1929	18	1932	52

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