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EXPERIMENTAL-RETORT TESTS OF ORIENT COAL

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The Bureau of Standards, in connection with two special coke-oven tests, found it desirable to investigate the influence of the temperature of coking upon the quality of the coke produced and upon the quantity and quality of gas made from the coal. Although several very similar coals were used in the oven tests, the experimental retort work was limited to coal obtained from the Orient mine of the Chicago, Wilmington & Franklin Coal Co., located at Orient in the south-central part of Franklin County, Ill. The data obtained from the coking tests with this coal at various temperatures are of such general interest that they are presented separately in this publication. In a separate technologic paper the Bureau is publishing an account of the operating test of the Koppers coke ovens when using straight Orient coal, which test was made at the plant of the Minnesota By-Product Coke Co., St. Paul, Minn.

This publication comprises a series of five experimental-retort tests made at various temperatures. The coking was done in a cylindrical retort of cast iron, set up as shown in Fig. 1. This apparatus was made available to the Bureau through the courtesy of the Bethlehem Steel Co., Sparrows Point, Md., who have used this equipment regularly for trying out coals or mixtures of coals intended for its by-product coke ovens. In this work the Bureau was fortunate in securing the advice of experienced technical men of this company who are regularly doing the experimental testing work. Special thanks for their counsel and assistance are

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due to Edwin Barnhart, engineer of tests, who developed the retort, and D. H. Duvall, assistant chemist, who has conducted the retort tests.

Data of a few similar retort tests on a variety of other coals which have been tested at this plant are presented for the sake of comparison.

Apparatus and Method Used.—The cylindrical retort, shown in Fig. 1, was inclosed in a refractory brick setting approximately

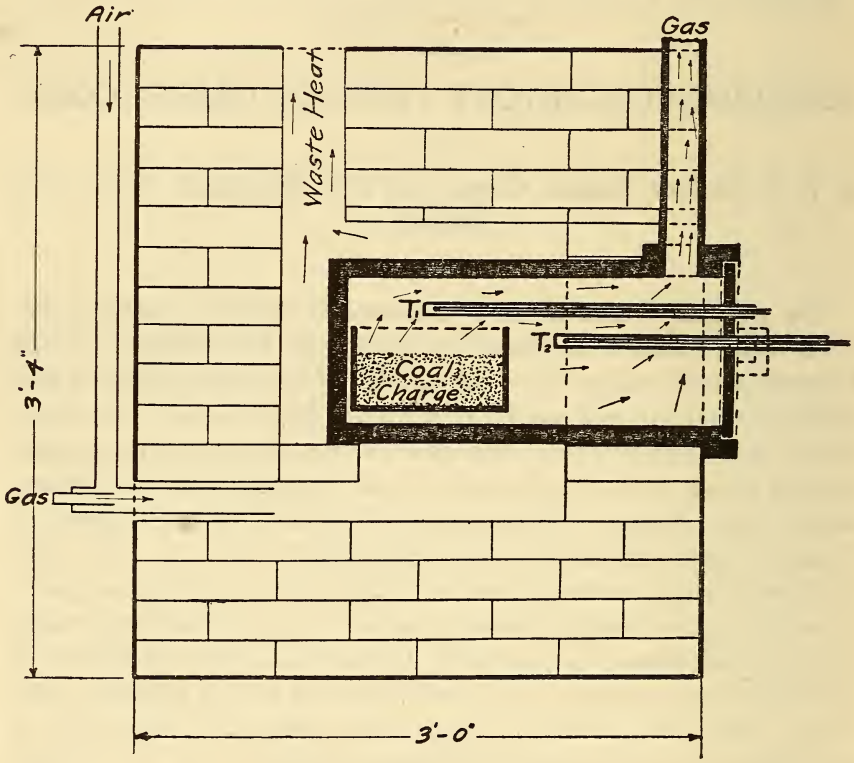

FIG. 1.—*Experimental retort*

Arranged for heating by bunsen burner, using coal gas. Inside dimensions of retort, 9½ inches diameter, 24 inches long; metal 1 inch thick. Inside dimensions of coal box, 9½ inches long, 4⅝ inches wide, 5 inches deep; metal ¾ inch thick. Space between outside of retort and masonry, 2¾ inches. Location of thermocouples, T_1, 5¾ inches from rear and 2 inches from top of retort; T_2 14 inches from rear and 4 inches from top of retort.

3 feet in each dimension. It was heated by a large bunsen-type burner, using coke-oven gas. The sample of coal to be tested was pulverized and dried thoroughly, generally by allowing it to remain overnight in an oven at approximately 100° C, before introduction into the small cast-iron box used to hold the charge. The retort was heated up to a temperature from 60° to 100° C

above the temperature desired for the test in order to allow for the unavoidable rapid cooling that takes place when the lid of the retort is opened for introduction of the charge. Immediately after each charge was placed in the retort an oil-soaked rag was thrown in. This rag quickly took fire and exhausted the oxygen in the retort space, thus eliminating the possibility of an explosion as the gases from the coal were generated.

The temperature of the retort was maintained at the desired point according to the indications of thermocouple T_1, the location of which is clearly shown in Fig. 1. Thermocouple T_2 furnished auxiliary temperature readings, but these were not used as the basis for temperature control. After introduction of the charge the lid was quickly closed and mudded up to prevent gas leakage. The gas driven off from the coal was discharged from the retort through the outlet pipe shown in the figure. As soon as the gas from the coal began to appear at a purge cock in this line, this purge cock was closed and the gas allowed to flow into a small storage holder in which the entire volume generated from the charge was collected. During the test period record was taken at intervals of five minutes, showing temperatures of T_1 and T_2, the volume of the gas collected in the holder, and the temperature of this gas. When the charge ceased gassing the coke was removed and quenched, and a record was made of the barometric pressure and of the temperature and volume of the gas in the holder.

TABLE 1.—Analysis of Coal as Used in Experimental-Retort Tests (Dry Basis)

		Sample from—	
		St. Paul, Minn.	Dover, Ohio
Volatile matter	per cent	37.8	38.0
Fixed carbon	do	52.3	53.4
Ash	do	9.9	8.6
Sulphur	do	.87	1.47
Phosphorus	do	.007	.002
Total carbon	do	73.0	76.3
Hydrogen	do	5.1	5.3
Oxygen	do	9.9	8.5
Ratio: hydrogen-oxygen	do	51.6	62.6
Btu per pound (dry)		12 800	13 000
Btu per pound (dry and ash-free)		14 200	14 200

After quenching, the coke was examined thoroughly, a description recorded, and an average sample prepared for analysis. The gas collected in the storage holder was analyzed, the candlepower

and specific gravity determined, and the heating value calculated from the analysis. From these data were calculated the Btu in the gas per pound of coal carbonized, the cubic feet of gas per pound of coal, and the candle-feet per pound.

Coal Used.—The coal used for four of these tests was that remaining from the sample taken at St. Paul, Minn., when the Bureau tested the coke plant of the Minnesota By-Product Coke Co., using Orient, Ill., coal. For the fifth test there was another smaller sample remaining from another shipment of Orient coal used by the Bureau in a separate coke-oven test at Dover, Ohio. Table 1 gives the analyses of these coal samples as used in the retort tests. These analyses agree very closely with those of the carefully prepared samples analyzed by the Bureau of Mines in connection with the plant tests mentioned above. The results of these tests, therefore, can be regarded as typical of the results for the larger shipment. Table 2 shows the size of the coal as crushed for the retort test.

TABLE 2.—Sizes of Crushed Coal as Used in Experimental-Retcrt Test

Mesh	Percentage through sieve
8	99.7
10	89.5
20	50.8
30	36.7
40	30.3
60	19.3
80	13.3
100	11.0

Results Obtained.—The results of the five retort tests are given in Table 3. In considering these data it must be borne in mind that only a very limited number of tests could be made because of the small samples and the short time available for the work. One should be very careful, therefore, not to try to draw too many conclusions from these results.

Test No. 1 was made as nearly as possible to duplicate the usual temperature conditions of testing followed by the Bethlehem Steel Co., so that a basis for comparison of this coal with others would be available. In this particular test the gas for heating the retort was cut off completely 40 minutes after the introduction of the charge. The data from this test are the only ones which can be compared with the results given in Table 4 for other coals.

The coke from this sample was fairly good, and the charge was thoroughly coked, showing considerable contraction in the sample box. The pieces were of medium size with no evidence of sponge and only a small amount of fine pebbly material on top of the coke. The coke had very small and regular cell structure, but was soft, shattering rather easily, having a longitudinal fracture and showing a tendency to finger. It was light in weight and of a dark silver color.

TABLE 3.—Experimental-Retort Tests of Orient Coal

	Test No. 1	Test No. 2	Test No. 4	Test No. 3	Test No. 5
Temperature average T_1 (° C)	840	775	700	600	605
Weight of dry coal (pounds)	4.48	4.00	4.00	4.00	3.20
Gas generated (cubic feet at 30 inches 60° F)	26.3	18.5	13.6	8.5	7.8
Gas per pound of dry coal (cubic feet)	5.87	4.62	3.40	2.12	2.44
Gas per net ton of dry coal (cubic feet)	11 750	9250	6800	4250	4850
Candlepower of gas	5.4	10.0	12.2	15.7	15.9
Candle-feet of gas per pound of dry coal	31.7	46.2	41.5	33.3	38.8
Specific gravity of gas	.465	.510	.625	.660	.655
Analysis of gas:					
CO_2	4.7	5.1	8.2	7.2	8.2
O_2	1.0	1.3	1.0	.8	1.6
Illuminants	2.2	3.1	3.8	5.0	5.2
CO	17.3	13.6	11.3	8.1	8.3
CH_4	25.1	30.6	36.6	37.1	39.5
H_2	46.3	44.7	33.6	30.3	26.0
N_2	3.4	1.6	5.5	11.5	11.2
Heating value, calculated from analysis (Btu)	510	570	600	610	625
Btu in gas per pound of dry coal	3000	2630	2040	1300	1520
Coke formed:					
Large (pounds), dry	2.99	2.66	2.79	2.72	2.19
Loose material (pounds), dry	.04	.06	.17	.21	.19
Total (pounds), dry	3.03	2.72	2.96	2.93	2.38
Coke yield:					
Large (per cent of dry coal)	67.0	66.5	70.0	68.0	68.5
Loose material (per cent of dry coal)	1.0	1.5	4.0	5.0	6.0
Total (per cent of dry coal)	68.0	68.0	74.0	73.0	74.5
Coke analysis (dry basis):					
Volatile	5.3	7.1	7.1	11.5	14.0
Fixed carbon	80.2	79.1	.....	.....	74.2
Ash	14.5	13.8	.....	.....	11.8
Sulphur	.78	.78	.....	.....	1.23
Phosphorus	.014	.014	.....	.....	.004

Test No. 2 was made at as nearly as possible constant vapor temperature, T_1, of 775° C. This was approximately the average temperature of the vapor above the coal as recorded in the test

of the coke plant of the Minnesota By-Product Coke Co., St. Paul, Minn., when the coking time was approximately 19 hours, but it is probable that for the same temperature of retort as of oven the vapor temperature in the retort would be higher than the corresponding vapor temperature in the oven. The coke results obtained in this case should probably, therefore, be more nearly comparable with the results of special oven tests made at St. Paul when the coking time was approximately 25 hours. The coke from this test was fair, contracted in the box, and broke up into large pieces with no sponge and only a small amount of fine material for about one-eighth inch on the upper surface of the mass. It had small and slightly irregular cell structure; but as a whole the coke was clean, soft, and somewhat tougher than in test No. 1, with longitudinal fracture but no cross fracture. It was slightly heavier than the material from test No. 1 and of dark color.

Test No. 3 was made with the idea of obtaining results at a somewhat lower vapor temperature above the coal than the average prevailing in the St. Paul test, but higher than noted in the other tests made by the Bureau at Dover, Ohio, where the vapor temperature above the coal averaged about 550° C. It was attempted to maintain an average throughout for T_1 of 600° C, and the results correspond very closely to this temperature. Judged by the results of this test, the coal would normally be characterized as only semicoking at this temperature, for the coke was very poor. There was some contraction in the box and no sponge formed, but there was a very large amount of loose material, that appeared almost uncoked, on top of the larger pieces of coke. The cell structure was small and very irregular. Because there was a very poor cementing action, even the large pieces were soft and easily broken in the hand. The material was fairly heavy with decided longitudinal fracture and no marked cross fracture. The color was very dark.

In view of the very poor results, judged by the character of the coke, obtained in test No. 3, it was decided to conduct test No. 4 at a somewhat higher temperature. The effort was to maintain T_1 constant at 700° C throughout this test. Even at this temperature the coal would be characterized as semicoking, although the coke was superior to that in test No. 3. It showed contraction in the sample box, large pieces, and no sponge; but there was a large amount of loose and apparently uncoked material on top of the coke. The small and somewhat irregular cell struc-

ture, with soft friable material resulting from a poor bond, corresponds to what would be expected from the results of previous tests. The material was fairly heavy with typical longitudinal fracture and no cross fracture, indicating that at this temperature there was a decided tendency to secure blocky coke rather than that of a fingery shape. As in the previous test, the material was very dark in color.

Test No. 5 was run with a small sample of coal remaining from the Dover test, in an effort to duplicate the conditions of test No.

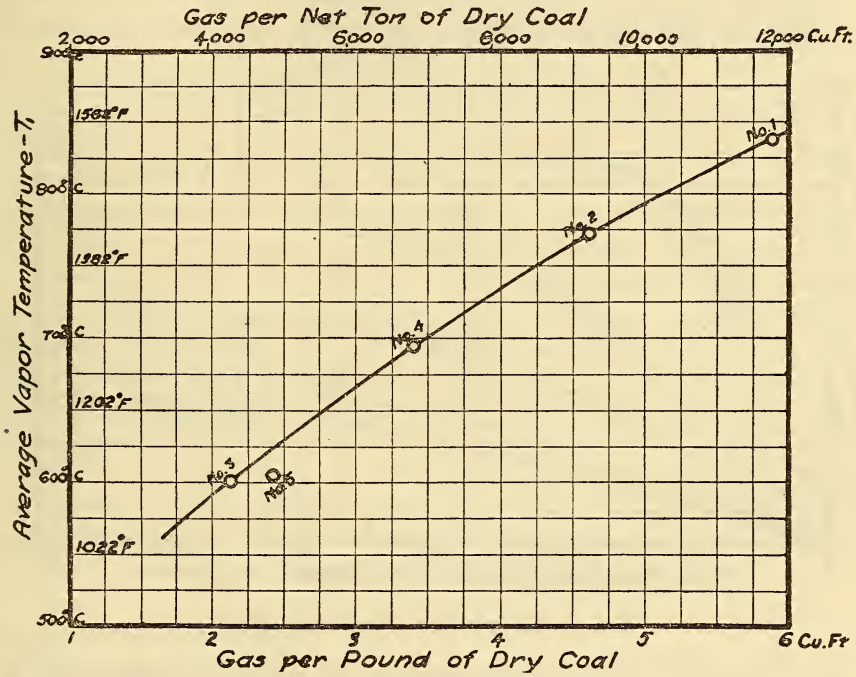

FIG. 2.—*Relation of gas yield to vapor temperature*

3—namely, an average for T_1 of 600° C. Although this coal came from an entirely different shipment from that used at St. Paul, the results were almost exactly the same as from the test No. 3 made at the same temperature.

Influence of Temperature Upon Gas Yield.—By comparison of the results for the five tests, the very large influence of temperature of coking upon the quantity and quality of gas is evident. In Fig. 2 is shown graphically the change in quantity of gas per pound of coal carbonized with changing temperature. In this connection it should be noted that the higher temperature results represent

not only a more complete elimination of the volatile material from the coal, but also a decomposition of the heavier volatile material into gaseous constituents. Naturally, as the vapors leaving the coal are subjected to this greater decomposition at higher temperature the average heating value of the gas is diminished. This diminution of heating value per cubic foot is, however, by no means enough to offset the effect of the larger volume, as is shown by the fact that the Btu in the gas per pound of coal carbonized at 840° is almost double that at 600°.

Fig. 3 shows the influence of temperature upon the rate at which the gas is generated and, of course, also the influence of

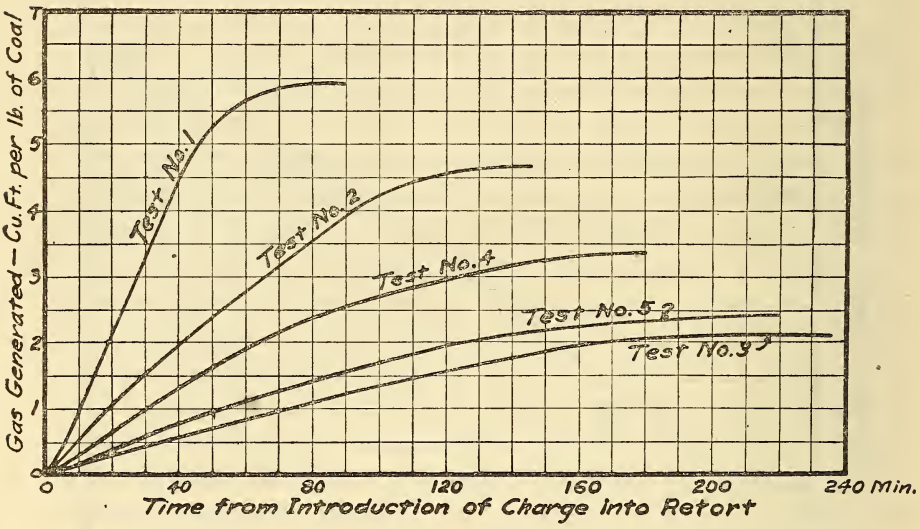

FIG. 3.—*Gas generated in experimental tests*

temperature upon the time required for complete gasification of the charge.

Influence of Temperature Upon Character of Coke Produced.—As would be expected, the amount of volatile matter left in the coke decreases as the temperature of coking increases; and correspondingly with more volatile matter in the coke the yield of coke is higher. However, at the lower temperature the percentage of fine material is much greater and the quality of coke very much inferior. In this connection, however, it should be noted that in none of the coking tests was it practicable to secure temperatures as high as are frequently used in oven practice with coals that are regarded as specially suited to the production of metallurgical coke. If such higher temperatures had been used another influ-

ence noted in connection with the St. Paul test would probably have appeared, namely, that at the high temperatures there is great tendency for the coke to be brittle, fingery, and small, instead of fairly tough and blocky.

TABLE 4.—Experimental-Retort Tests of Other Coals

[Data furnished to Bureau by the Bethlehem Steel Co. All tests with 4.48 pounds at approximate T_1 average of 850° C]

	40 per cent Washington 60 per cent Quemahoning	50 per cent Gauley 50 per cent Pocahontas coal	Washington coal	Quemahoning coal	Morrell, Fayette County, Pa., coal	Logan County, W.Va., coal	Mount Pleasant, Fayette County, Pa., coal	Tip Top, Fayette County, Pa., coal
Coal analysis:								
Volatile	25.42	28.85	33.02	17.23	31.20	26.08	31.55	30.63
Fixed carbon	66.58	63.98	58.51	75.45	57.80	45.95	59.86	59.07
Ash	8.00	7.17	8.47	7.32	11.00	27.97	8.59	10.30
Total carbon	79.63	79.69	77.78	83.15	72.26	59.77	72.45	70.71
Hydrogen	4.72	4.85	5.05	4.48	4.30	3.82	4.41	4.16
Oxygen	6.35	7.05	7.40	3.75	11.01	7.14	13.25	13.63
Ratio: Hydrogen-oxygen	74.33	68.79	68.24	119.46	39.05	53.50	33.27	30.52
Gas per pound of coal (cubic feet)	4.50	4.55	5.10	4.50	5.17	3.49	5.02	4.08
Gas per net ton of coal (cubic feet)	9000	9100	10 200	9000	10 340	6980	10 040	8160
Candlepower of gas	8.0	6.2	8.5	3.7	1.2	10.6	2.6	2.3
Candle-feet of gas per pound	36.0	28.2	43.3	16.7	6.2	37.0	13.1	9.4
Specific gravity of gas	.400	.476	.440	.424	.450	.466	.495	.551
Analysis of gas:								
CO_2	3.1	4.5	4.1	2.3	5.6	4.4	8.0	10.8
O_2	.9	.5	.7	.8	.6	.6	.4	.7
Illuminants	2.5	2.0	2.6	1.6	1.4	3.1	1.9	1.6
CO	10.4	12.6	11.7	9.9	15.6	8.6	15.5	14.6
CH_4	29.3	31.5	30.8	27.0	21.5	35.0	24.0	26.5
H_2	52.8	46.8	47.5	57.0	51.1	46.1	48.4	42.7
N_2	1.0	2.1	2.6	1.4	4.2	2.2	1.8	3.1
Heating value calculated from analysis (Btu)	557	556	561	526	465	600	492	490
Btu in gas per pound of coal	2510	2530	2860	2370	2410	2090	2470	2000
Coke obtained indicates	Extra good coal [a]	Good coal [a]	Fair coal [a]	Good coal	Semi-coking coal	Semi-coking coal	Non-coking coal	Non-coking coal
Shatter test of coke (per cent)	80	66	52	.....	.....	.....	.....	.....

[a] These results refer to coke from full-size oven tests.

As pointed out above, great care should be exercised in drawing conclusions from these few tests. However, these few conclusions are strong confirmation of the results of the special oven tests made at St. Paul and are therefore a very valuable bit of supplementary data, which it is hoped will be of interest to the by-product coke-oven operators in general, but most especially to the operators who use midcontinent coals.

Similar Results on Other Coals.—For sake of comparison there are included in Table 4 data for other coals similar to the data given in Table 3 for the Bureau's tests of Orient coal. A general comparison of these data is interesting, but no detailed conclusions should be drawn as comparison of such single tests is apt to be misleading.

WASHINGTON, April 2, 1919.