

proportion of 10 Cb per unit of C present. Nominal dimensions of the completed reaction tube were: Length 20 ft. 6 inches; inside diameter 48.5 inches; wall thickness 0.5 inch.

The general appearance of the tube was excellent, with a smooth surface both inside and outside. Detailed measurements showed that the deviation in length of radii was within the specified limit of  $\pm 1/8$  inch. Figure 22 shows the tube being lowered into the retort for the first time. This picture clearly shows the uniform welds and the smooth surface of the tube. The dark portion near the lower end of the tube is the discolored 310-alloy flame guard, which had been used before (4).

To date the 310-alloy tube has been operated 1,862 hours, during which it was heated and cooled 3 times. After each run, numerous measurements and a careful inspection were made. The observations gathered indicated that corrosion at the present rate would not be a limiting factor for a tube life of 10,000 hours. The major portion of the inner surface exhibited little corrosion, and the outer surface was in excellent condition. Figures 23 and 23b show the same section of the inner surface before and after run 13. The area pictured is that of the most pronounced pitting and indicates little additional corrosion during run 13. Measurements of the depth of the pits showed a maximum penetration of only  $5/64$  inch after 1,862 hours of operation. In addition to this pitting, which occurred only between 6 to 10 feet from the flanged end of the tube, some tightly adhering magnetic scale was formed in a narrow band opposite the gas offtake. This narrow band of scale was not believed to be detrimental to tube performance.

When initially installed and after each run, radial measurements were made at 1 foot intervals over the entire tube length. In figure 24 the results of these measurements are plotted on a greatly exaggerated scale. Actually little deformation was visible and must be shown of this scale to be apparent. Even after run 12 the majority of the measurements were still within specifications for the original tube. However, the extent of the deformation of the tube increased during run 13. The maximum increase in the length of radius was  $24/32$  inch, and the maximum decrease was  $13/32$  inch. These changes in the dimensions of the tube were not sufficient to affect the smooth operation of the retort. Measurements of the tube length did not indicate any change.

From experience gained through a total of 5,772 hours of operation with various tubes and careful observations and measurements after each run with a 310-alloy, rolled-plate tube, it is believed that the problem of corrosion is no longer of major importance but that the problem of deformation or creep might be the limiting factor in the life of the tube.

#### INFLUENCE OF MASS VELOCITY OF GAS AND WATER VAPORS LEAVING GAS OFFTAKE ON BEHAVIOR OF GAS GENERATOR

Two factors may be considered as limiting the capacity of the gas generator. First, as shown during run 11, the capacity of the gas generator was limited when the feed rate was so high that gasification could not be maintained satisfactorily at normal operating temperatures in the combustion space. Second, at relatively high linear velocities of gas and steam vapors leaving the gas offtake, the retort did not operate smoothly, and increasing quantities of blowover dust were recovered from the dust collector. Although these effects depend upon the linear velocity of the gas and steam mixture, it is more consistent for the same installation to express the function as mass velocity.

These limiting factors of gas velocity were first investigated during period 11 MN. A feed rate of 395 pounds per hour was used and the rate of steam introduction progressively increased during the period. With an increasing steam rate, both gas and undecomposed steam volumes also increased. At an approximate mass velocity of 263 pounds of gas and water vapors per hour per square foot of gas offtake area, the unit was performing normally, but when a mass velocity of about 306 pounds per hour per square foot was reached, the fluctuations of static pressures and of the gas rate became more pronounced. The change in smoothness of operation is shown in figure 25, which is a reproduction of the product-gas recorder chart for the period 11-MN. From 4 to 5:45 a.m. at a mass velocity of 263 pounds per hour per square foot, an essentially uniform production is indicated. At 5:45 a.m., when the mass velocity had been increased to 306 pounds per hour per square foot, some erratic operation is shown by the jagged line, which might indicate a type of fluidization or "boiling" of partly gasified material at or near the gas offtake.

During runs 11, 12, and 13 the blowover dust was collected and weighed and the size consist determined from representative samples of each period. Figures 26 and 27 correlate the data. In each figure the abscissa of the curves is the mass velocity of gas and water vapors leaving the retort in pounds per hour per square foot of gas-offtake area. The gas-offtake area was in each instance 3.6 square feet. In figure 26, the ordinate is the total weight of blowover dust and dry sump mud, in pounds per hour, and in figure 27 it is the percentage of the size consist in the blowover dust above 30-mesh (0.0232-inch). In each figure, four curves, designated 1, 2, 3a, and 3b, are plotted. Curve 1 presents the data from run 11, curve 2 from run 12, and curves 3a and 3b from run 13. Curve 3a was obtained when the steam rate to the char zone was increased in 50-pound-per-hour increments from 50 to 300 pounds per hour without addition of steam to the upper reaction zone. Curve 3b was obtained when a constant steam load of 100 pounds per hour was maintained on the lower reaction zone and the rate to the lignite zone was increased in 50-pound increments from 50 to 300 pounds of steam per hour.

In figure 26, curves 1 and 2 show an intermediate rapid increase in the amount of blow-over dust leaving the gas offtake at approximately 220 pounds of gas and water vapors per hour per square foot of gas offtake area. At equal mass velocities, curve 1 from the natural lignite run is consistently above that of curve 2 from the steam-dried lignite test. The difference in the weights of dust recovered at equal mass velocities might be due to the larger average size of the char originating from the dried lignite.

The influence of the position of the steam introduction is shown by comparing curves 3a and 3b. Approximately, curve 3a follows curve 1 in the region covered, whereas curve 3b shows that, at the lower mass velocities, up to about 196 pounds per hour per square foot of gas offtake area, more blowover dust is recovered when the steam is distributed between the upper and the lower reaction zones. At a mass velocity of about 196 pounds, curve 3b appears to level off but 3a increases rapidly, indicating that the fluidized condition was influenced more from the steam admissions to the char zone.

Figure 27 shows the percentage of plus-30-mesh particles in the blow-over dust as a function of the mass velocity of the gas and steam leaving the gas offtake. Curves 1 and 2 again show a marked increase at approximately 220 pounds per hour per square foot of gas offtake area. The blow-over dust collected from the gasification of dried lignite contained a higher percentage of plus-30-mesh particles than did the natural lignite, as shown by curve 2, being consistently higher than curve 1. Curve 3a shows a more rapid increase in the plus-30-mesh fraction when the steam was admitted only to the char zone. Also the plus-30-mesh portion was greater at equal mass

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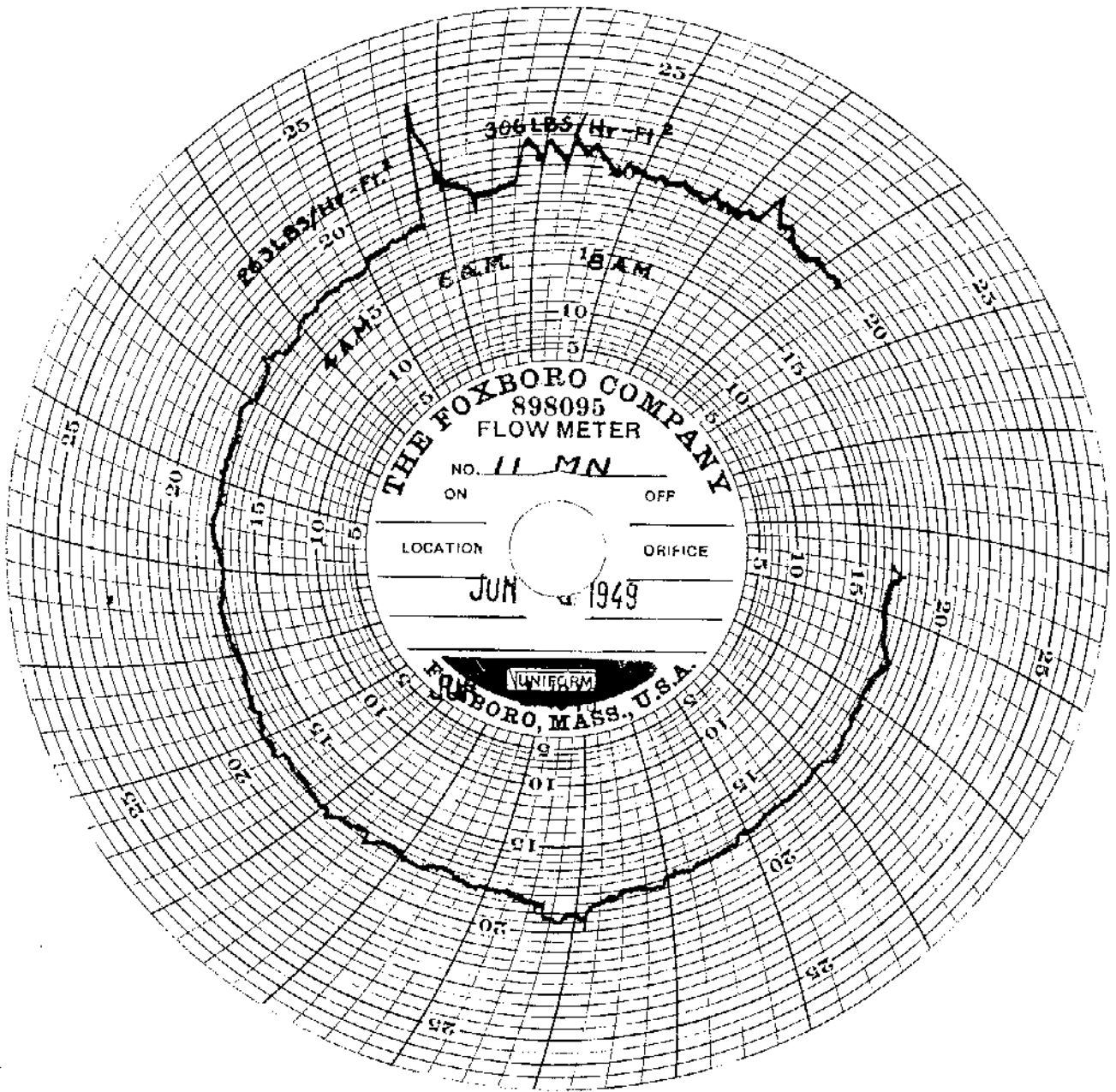


Figure 25. - Product-gas recorder chart for period 11 MN, showing fluctuations in gas production.

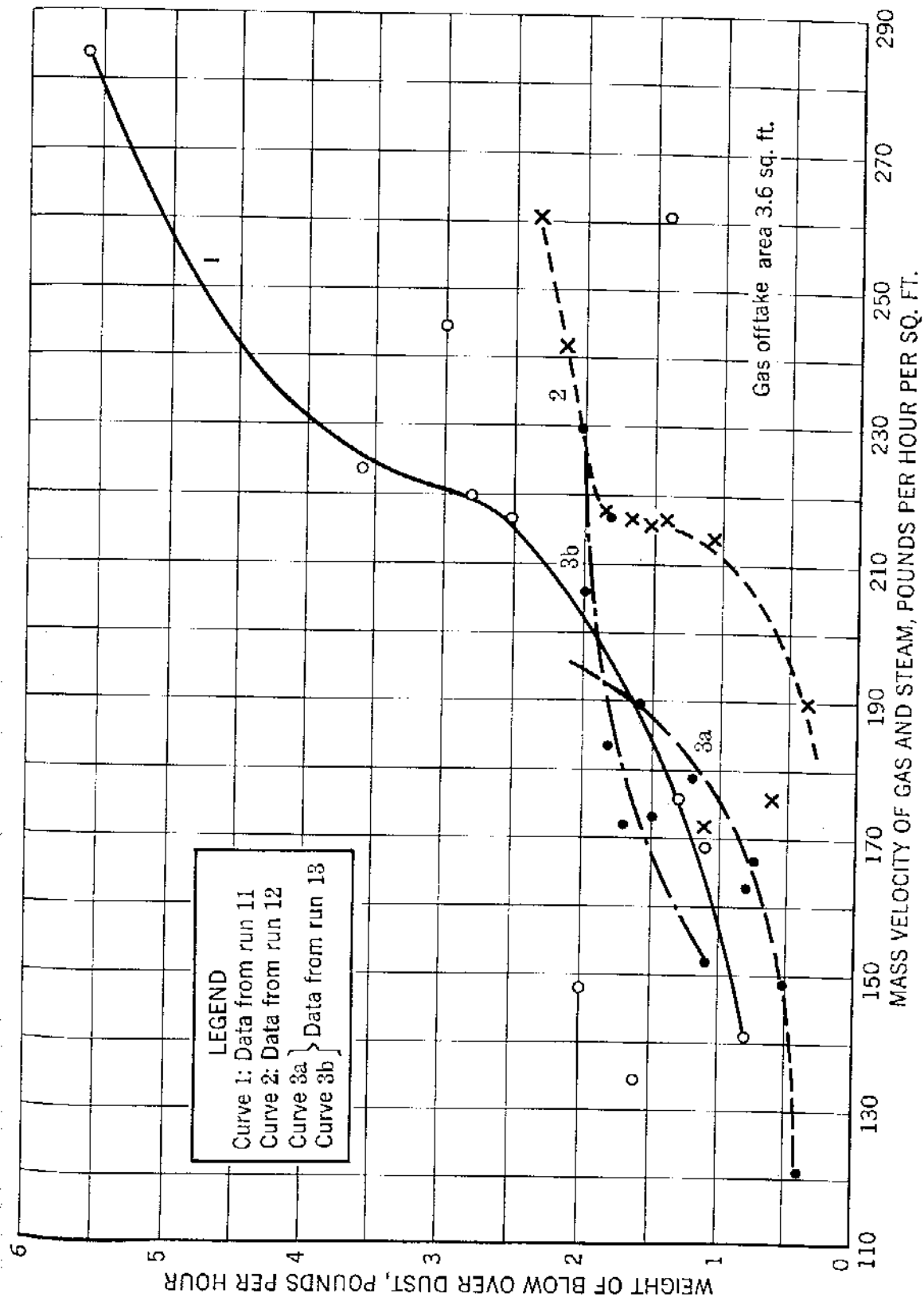


Figure 26. - Weight of blow-over dust as a function of mass velocity of gas and steam leaving gas offtake.

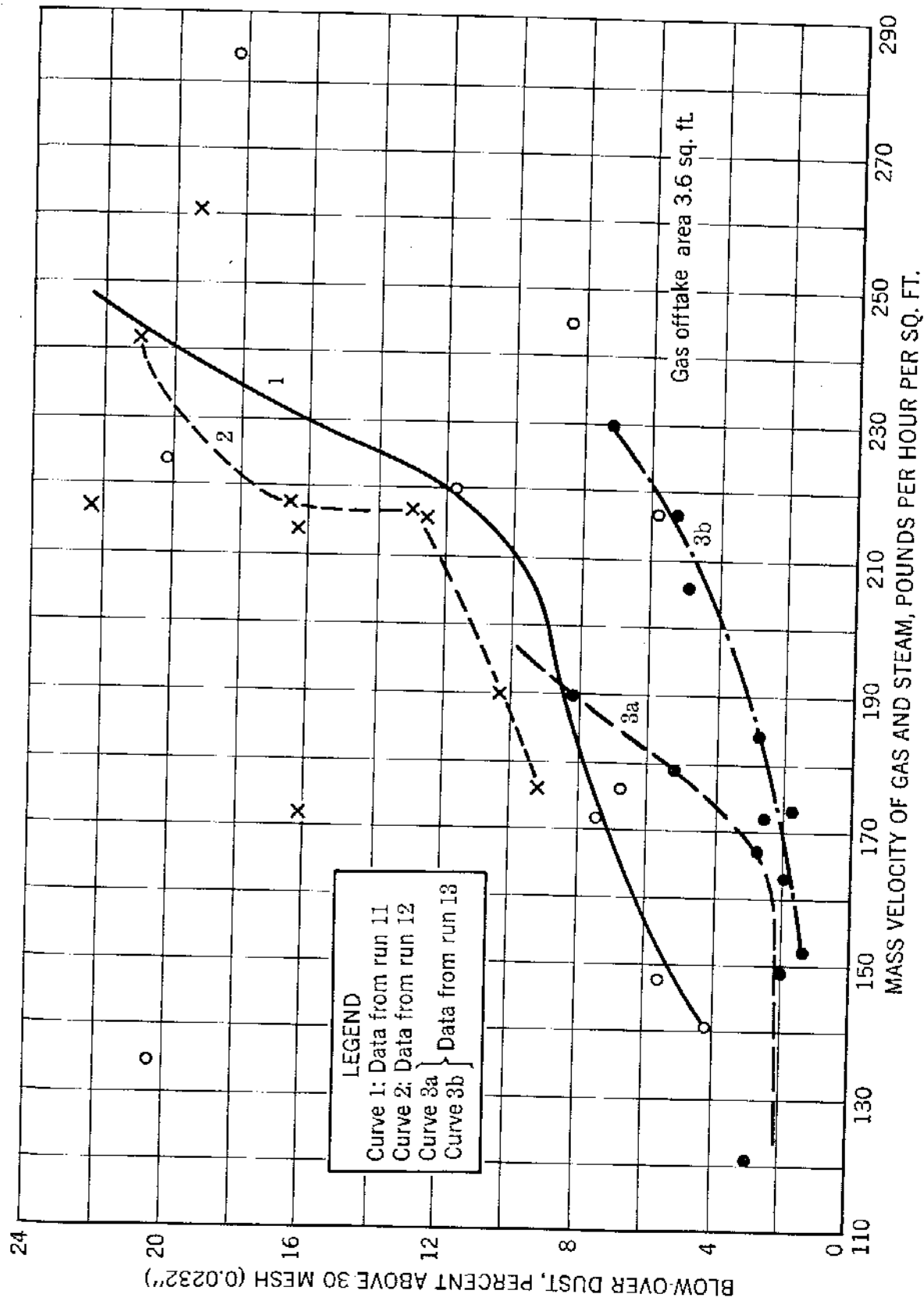


Figure 27. - Size consist of blow over dust as a function of mass velocity of gas and steam leaving gas offtake.

velocities when the steam was introduced into the lower reaction zone. The difference between the two curves, 3a and 3b, again indicates that the apparent fluidization of the particles at the gas offtake is more affected by steam admission to the char zone than to the lignite zone.

The following conclusions may be drawn from the correlation of the mass velocity of the gas and water vapors leaving the gas offtake and the amount and size consist of the blowover dust.

1. At a mass velocity of approximately 220 pounds per hour per square foot of gas offtake area, some type of fluidization or "boiling" of the partly gasified lignite occurred at or near the gas offtake and markedly increased the amount, and the plus- 30-mesh size, of the dust entrained in the gas and steam vapors leaving the gas offtake.
2. At a mass velocity of 300 pounds per hour per square foot of gas offtake area or over, some erratic operation of the retort may occur due to increased "boiling" or fluidization at or near the gas offtake.
3. At equal mass velocities the amount of blowover dust appears to be reduced, and the fraction of the particles above 30-mesh increased when dried lignite was gasified.
4. An increase in the steam rate to the char or lower reaction zone apparently increases the amount and size consist of the blowover dust more than an equal increase in the steam rate to the upper reaction zone.

#### DISTRIBUTION OF SULFUR DURING GASIFICATION OF LIGNITE

The concentration and distribution of sulfur in the products of the gasification process are important, as sulfur compounds are deleterious in most instances even though present in low concentrations. Lignite, as most fuels, contains sulfur in varying amounts. Although lignite generally has a relatively low sulfur content (averaging 0.6 percent), enough sulfur is transferred into the product gas to warrant investigation.

A considerable amount of information has been accumulated on the concentration, distribution, and types of sulfur in the lignite feed and in the gasification products. The following presentation summarizes this information.

The sulfur content of the Dakota Star lignite used in the gasification process varied between 0.6 to 1.6 percent, of which approximately 30 percent is present as organic sulfur, 60 percent as pyritic sulfur, and 10 percent as sulfate sulfur. As relatively large volumes of gas are produced per ton of lignite charged, the sulfur content of the product gas is unusually low, being in the range of 35 to 140 grains per 100 c.f., of which only 1.5 to 2.3 grains per 100 c.f. are organic sulfur. The actual concentration of the sulfur in the gas was found to be related to the operational conditions and the initial sulfur content in the feed. In table 18 typical data for runs 11, 12, and 13, showing the concentration of  $H_2S$  and organic sulfur in the gas, are presented. With such a low concentration of total and organic sulfur no major purification problem is presented.

TABLE 18. - Typical concentration of sulfur in product gas during runs 11 through 13

Run and period No.	Sulfur in H <sub>2</sub> S before scrubber gr./100 cu. ft.	Organic sulfur after scrubber gr./100 cu. ft.	Total sulfur in gas, gr./100 cu. ft.	Total sulfur in gas, percent of sulfur charged
11-D .....	35.8	2.1	37.9	16.3
11-F .....	135.5	1.8	137.3	40.8
11-J .....	71.5	1.7	73.2	39.4
12-D .....	49.6	1.7	51.3	23.3
12-E .....	71.7	2.1	73.8	21.2
13-C .....	82.8	2.1	84.9	33.9
13-M .....	77.7	1.6	79.3	46.7

The percentage of original sulfur transferred to the gas may be correlated with the fraction of undecomposed steam. The trend of an increasing percentage of original sulfur from the lignite in the product gas with an increasing fraction of undecomposed steam is indicated in figure 28, which presents the results of runs 11 through 13. The percentage of the original sulfur in the unscrubbed gas increased from about 16 to 57 percent when the fraction of undecomposed steam varied between 0.28 and 0.64. Scattering of the experimental points in this curve was caused by the influence of the location of the admission of steam on the percentage of sulfur extracted from the lignite. Part (a) of figure 29 shows the uniform increase of the percentage of the total entering sulfur extracted with increasing fraction of unreacted steam when the steam introduction was only to the char zone. Section (b) of the same figure shows a wider variation in the percentage extracted when the steam was added to both the lignite and char zones.

Table 19 gives the sulfur balances for run 13. The amount of the total sulfur in the lignite recovered in the gas ranged from 22.1 to 46.8 percent and in the residue from 57.0 to 102.5 percent. The balances appear to be erratic, because in several instances the total sulfur recovered exceeded that charged. However, the positive and negative deviations from 100-percent recovery were approximately equal. The major reasons for the occasional unsatisfactory balances appear to be in obtaining a representative sample of char. Under the present experimental procedure, the operating conditions are changed from period to period during a run. The percentage of gasification and the resultant fraction of undecomposed steam are usually varied, causing the ash and sulfur content of the char and the gas composition to change. The gas composition is sensitive to operational changes and quickly responds, giving in a short time a nearly constant composition. However, the annular space at the end of an official period is filled with partly reacted material, the composition of which has been determined by the conditions of that period. As each official period is preceded by a transition period of 24 hours, calculations of throughput indicate that the char from the one official period should be replaced before the next official period begins. Thus, the char composition should correspond to the gas being made. In order to determine if a nearly constant composition of the char was reached, special char samples were collected during the later part of run 13 in addition to the regular composite sample. For the composite sample, a 3-percent, by weight, portion was taken with a pipe sampler from each can of char during the test period. The special samples, also 3 percent, by weight, were taken from the char cans removed at the beginning and the midpoint of each period, including transition periods as well as test periods. From table 20, which compares the results of the special samples with the representative sample obtained for each period, a periodical increase and decrease of the ash and sulfur content of the char may be noted. For example, the

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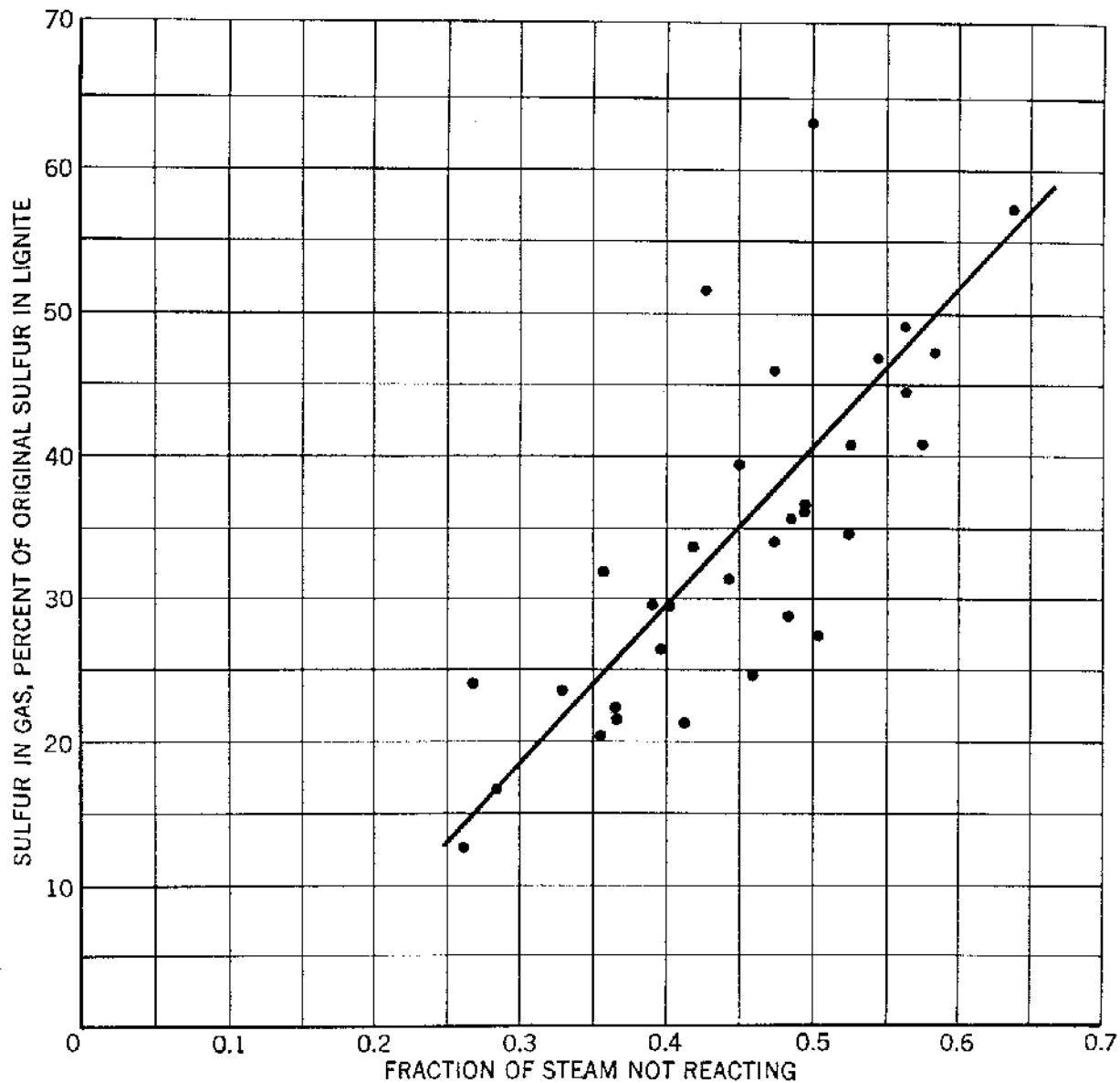


Figure 28. - Percentage of total sulfur in charge transferred to product gas as a function of unreacted steam.



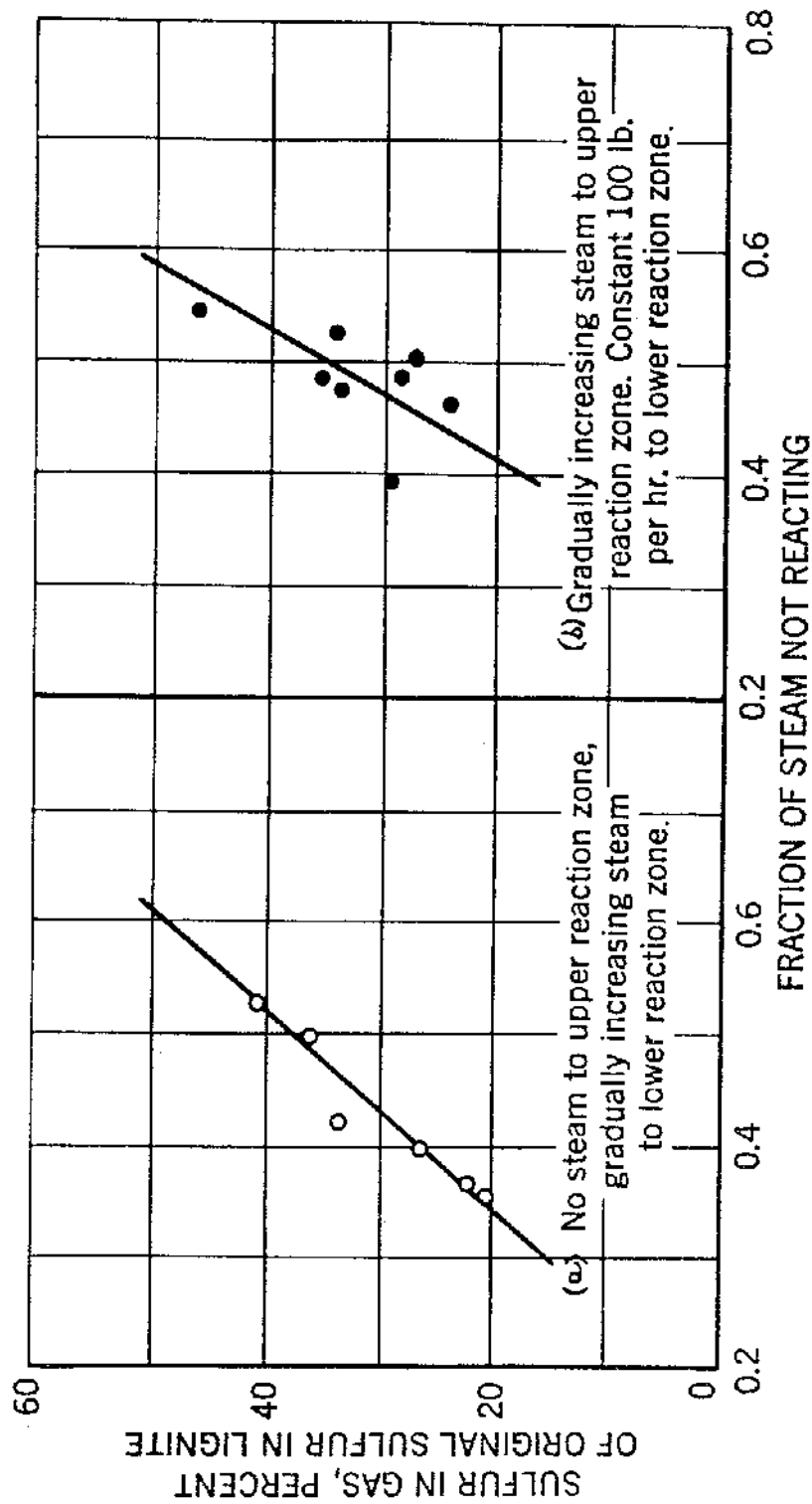


Figure 29. - Percentage of total sulfur in charge transferred to product gas as a function of unreacted steam and location of steam admission.

four special samples taken for periods IJ (transitional period), and J (test period) shows the ash content to be 29.7, 37.3, 37.2, and 25.6 percent and the sulfur content 2.4, 3.0, 2.8, and 1.9 percent, respectively, despite the fact that the experimental conditions were constant. Fluctuations in composition during the transition period would be expected, but not during the test period. The composite sample for the official period gave 41.3 percent ash and 3.3 percent sulfur, percentages not reached in the special samples. These variations indicate the difficulty of obtaining matching char and gas samples.

TABLE 19. - Summary of sulfur balances for run 13

Period	Sulfur entering in lignite, lb./hr.	Sulfur accounted for					
		In residue		In gas		Total	
		Lb./hr.	Percent S charged	Lb./hr.	Percent S charged	Lb./hr.	Percent S charged
A	3.80	2.62	69.0	0.84	22.1	3.46	91.1
B	3.27	2.59	79.2	.86	26.3	3.45	105.5
C	3.32	2.11	63.6	1.12	33.9	3.23	97.3
D	4.36	2.22	51.0	1.57	36.0	3.79	87.0
E	2.70	2.19	81.1	1.10	40.7	3.29	121.8
F	3.47	2.63	75.9	.70	20.1	3.33	96.1
G	2.58	2.62	101.6	.76	29.5	3.38	131.1
H	1.77	1.13	63.8	.60	33.6	1.73	97.7
I	2.81	2.88	102.5	1.00	35.6	3.88	138.1
J	3.37	2.47	73.3	.82	24.4	3.29	97.6
K	3.42	2.13	62.3	.94	27.4	3.07	89.8
L	2.91	1.66	57.0	1.00	34.3	2.66	91.4
M	2.84	1.81	63.6	1.33	46.7	3.14	110.4
N	3.43	2.14	62.4	.99	28.8	3.13	91.3

TABLE 20. - Composition of special char samples collected during run 13

Period <sup>1/</sup>	Hours after beginning of period <sup>2/</sup>	Ultimate analysis of char					Composite sample	
		Ash	H	C	N	S	Ash	S <sup>3/</sup>
		Percent						
13-IJ	0-2.75	29.7	1.2	69.3	0.5	2.4		
13-IJ	9.83 - 11.83	37.3	1.1	63.6	.5	3.0		
13-J	0-1.35	37.2	1.1	62.7	.4	2.8		
13-J	9.35 - 11.35	25.6	1.2	72.3	.5	1.9	41.3	3.3
13-JK	0-1.47	43.5	.9	58.1	.4	3.5		
13-JK	9.47 - 11.47	41.8	1.0	59.0	.4	3.1		
13-K	0-2.0	51.2	.9	51.4	.4	3.8		
13-K	10.0 - 12.0	43.3	1.0	57.6	.4	3.2	41.7	3.3
13-KL	1.82 - 3.82	44.3	1.0	56.5	.4	3.3		
13-KL	9.82 - 11.82	54.5	.9	49.3	.3	4.0		
13-L	0-1.23	51.4	1.0	50.6	.3	3.6		
13-L	16.73 - 18.73	34.2	1.1	63.4	.4	2.2	51.5	3.0
13-IM	8.55 - 10.55	58.9	.8	44.4	.3	4.0		
13-M	2.0 - 4.0	52.1	.9	49.1	.3	3.4		
13-M	10.0 - 12.5	48.0	1.0	53.2	.4	3.2	49.6	3.4
13-MN	0.0 - 2	53.6	1.1	48.5	.3	3.9		
13-MN	9.50 - 11.50	52.5	.9	56.2	.4	4.2		
13-N	0-1.42	23.4	1.2	73.6	.4	1.6		
13-N	10.42 - 11.92	38.0	1.0	63.0	.4	3.2		
13-N	21.42 - 22.92	32.6	1.0	66.4	.4	2.2	34.1	2.5

<sup>1/</sup> Double letters represent transitional periods.

<sup>2/</sup> Hours after beginning of period refers to time during which the char, represented by the analysis, was collected.

<sup>3/</sup> Composite sample is regular official sample.