Correlation of data for the several test periods shows a definite relationship between percent carbon gasified and temperature measured at the inner wall of the gasification retort. In figure 14 carbon gasified is plotted as a function of inner tube temperature opposite point A (see retort diagram, fig. 2, for location of measurement positions). Relationship between carbon gasified and inner tube temperature opposite point B, also in the lower section of the gasification retort, was similar.

The demonstrated relationship between carbon gasified and inner tube temperature in the lower part of the gasification retort provides a clear indication that a heat transfer effect is controlling gasification rate. Low percent gasification corresponds to low inner tube temperatures; that is, to relatively poor heat transfer to the inner portion of the gasification retort. That the observed changes are necessarily related to heat transfer is evident from the type of relationship found. If the changes were primarily associated with differences in lignite reaction rate or reactivity for the several test periods and heat transfer characteristics were similar, then temperature at the inner wall would be lower for higher percent gasification - since the principal reactions involved are endothermic. The correspondence between high percent gasification and high temperature at the inner wall demonstrates that heat transfer to inner portions of the retort is the controlling factor.

Ash Deposits on Alloy Tube

The wide variation in carbon gasification during the several test periods of run 17 provides evidence of corresponding variation in heat transfer properties. From the available data, it appears that an important primary factor affecting heat transfer during the run was development of ash deposits on the alloy tube.

The total quantity of ash deposits on the alloy tube after each run of the present series was given in table 8. Quantity of ash attached to the alloy tube after run 17 was 109 pounds, or about twice that found in other runs of this series and in runs 11 through 13 (3).

Inner tube temperature in the lower part of the retort, opposite points A and B (see fig. 2), was measured for the first time in runs 15 through 17. Evidence on buildup of ash deposits during run 17 and a probable explanation of the primary trend in the gasification data is provided by the observed trend in inner tube temperature as the run progressed.

Inner tube temperature opposite point A during run 17 is plotted versus time of run in figure 15. In general, as would be expected, test periods at higher lignite feed rate show a lower inner tube temperature, at approximately equal time of run. However, the effect of time of run on inner tube temperature substantially exceeds that of lignite feed rate and within the apparent precision of the data all the points may be represented by a single curve.

The results for change of inner tube temperature with time of run show a definite trend of decrease in inner tube temperature with time in the first part of the run, up to about 20 days of operation. In the latter part of the run, there is a leveling off in inner tube temperature.

The observed change in inner tube temperature with time, as shown in figure 15, is believed to correspond to gradual building up of ash deposits on the inner wall of the alloy tube. Leveling off of inner tube temperature after about 20 days of operation corresponds, in the same basis, to maximum buildup of ash deposits, reaching approximately a steady-state condition.

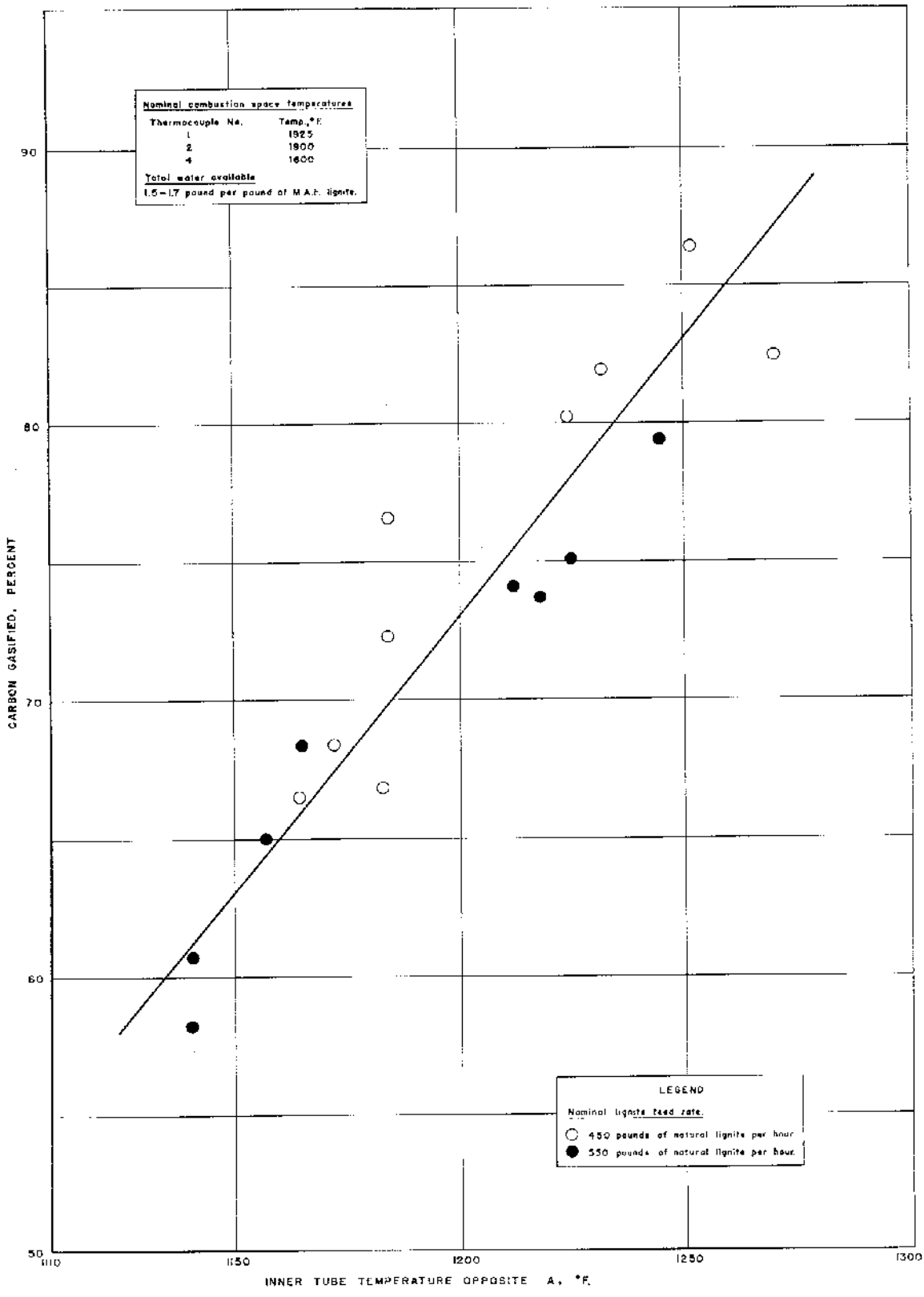

Figure 14. - Carbon gasification as a function of inner tube temperature.

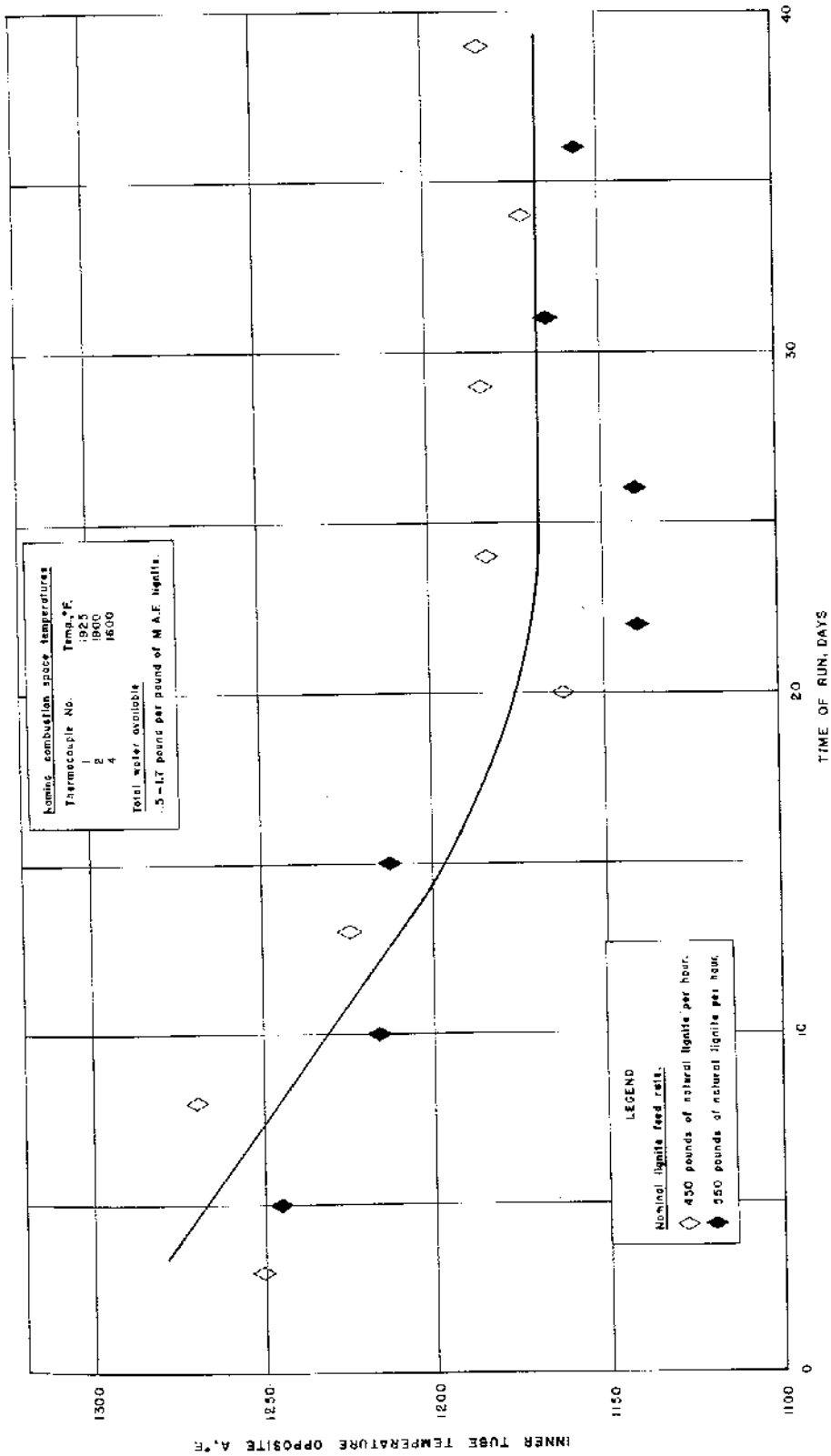

Figure 15. - Change of inner tube temperature with time, run 17.

The total amount of ash deposition (table 8) and the indicated reduction in gasification rate owing to ash deposit formation in run 17 are substantially larger than for previous runs of this series or earlier gasification tests (3, 4).

Combustion-space temperatures in run 17 were equal to the highest temperatures that had been used operating the alloy-tube gasification retort. In addition, combustion-space temperatures were maintained constant at this same distribution throughout the run. Both the high temperature and constancy of temperature could contribute to the formation of permanently attached ash deposits.

Another factor that may be related to ash-deposit formation is ash fusibility of the lignite, as charged. Dakota Star lignite, used in most of the gasification experiments, has a relatively high ash fusibility. However, several of the lignites used in the early part of run 17 had lower ash fusibilities, with initial deformation temperatures ranging down to 1,940° F. Ash fusibility of the lignites as charged to the gasifier in the present series of runs is plotted in figure 16. A pronounced dip is evident for lignites tested in the latter part of run 16 and the first several test periods of run 17. A low ash-fusion temperature, probably in conjunction with some contamination from the retort tube, obviously could be a contributing factor in forming bonded ash deposits resistant to heat transfer at the tube surface. These ash deposits once formed are apparently quite permanent, at least when combustion-space temperatures are held constant, in view of the evidence in terms of leveling off of inner tube temperature in the latter part of run 17.

Effect of Lignite Size and Composition

During the period of ash buildup on the alloy tube, this phenomenon was predominant in determining gasification characteristics under operating conditions used in run 17. The gasification data of run 17 can be further analyzed with regard to factors affecting gasification characteristics during the latter part of the run after buildup of ash deposits to approximate steady state.

Experimental results during the latter part of run 17, after period 17-G, show a definite trend of gasification rate with size of lignite fed to the retort. As in previous runs, lignite fed to the gasifier was nominal 1-1/2- by 1/2-inch stoker size rescreened to 1-1/2- by 3/8-inch. In all runs there was some variation in size distribution within this range. In the runs with a single lignite, rescreened Dakota Star, average size of the lignite was in general from 0.8 to 1.0 inch. In run 17, during which lignite from 7 different mines was tested, average size of the lignite as charged to the gasifier showed a wider variation, ranging from 0.6 to 1.06 inches.

In figure 17, carbon gasified is plotted versus average size of lignite as charged for test periods 17-C through 17-O. The results show an evident upward trend of carbon gasified with increase in average size of the lignite. In figure 17 a single curve has been drawn to represent all data; however, there is also evidence, as would be expected, of a separation of results in terms of lignite feed rate. Results at each lignite feed rate, considered separately, show good correlation and the same general trend for carbon gasified versus average size of lignite.

The effect of size of lignite on gasification is believed to be related primarily to change in heat transfer properties with size. Rate of gasification at comparable temperature levels may also increase with increase in size, owing for example, to reduced channeling and improved gas-flow pattern. In the latter part of run 17 as elsewhere, however, the results for carbon gasified correlate reasonably

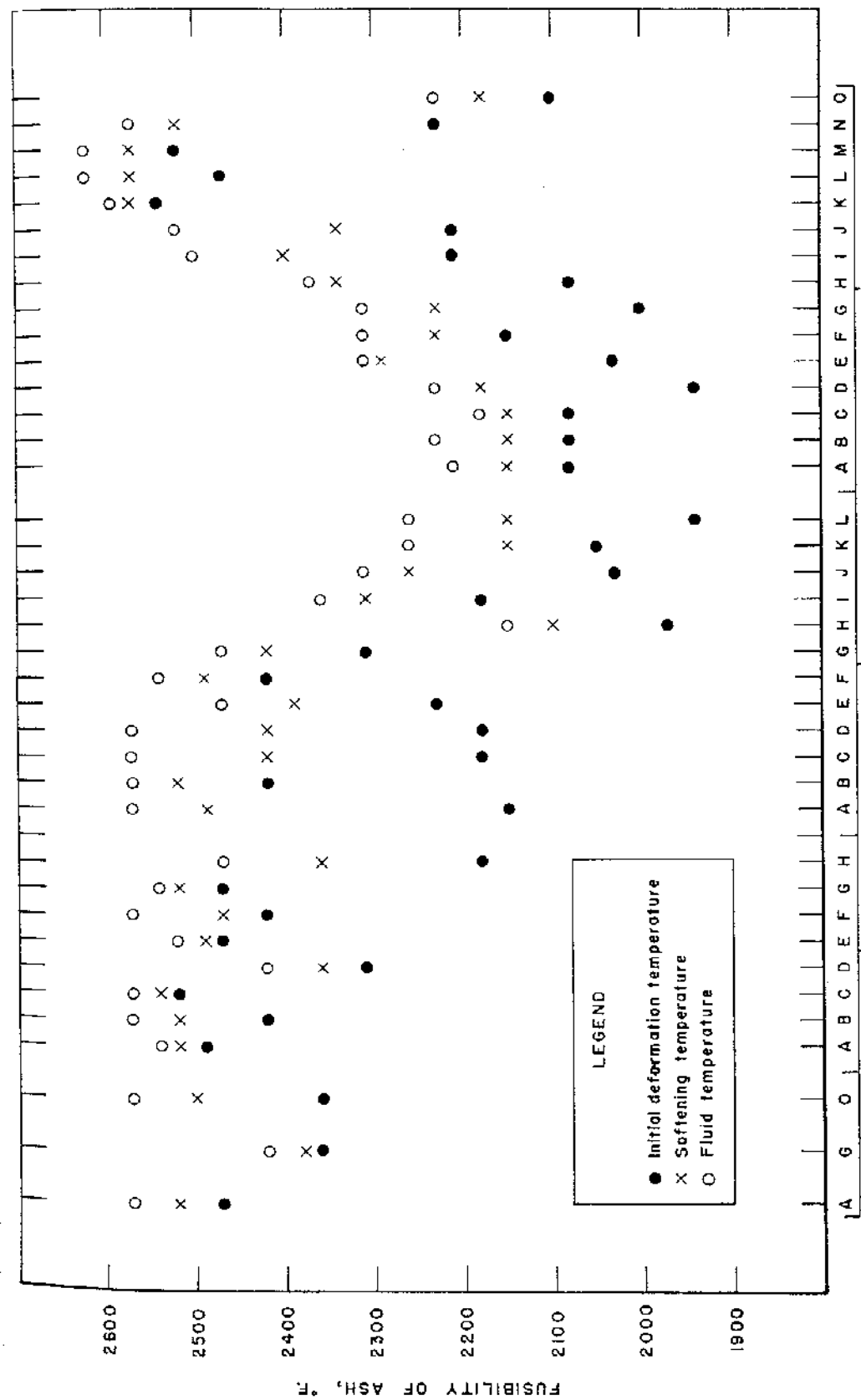

Figure 16. - Ash fusibility of lignite as charged, runs 14 through 17.

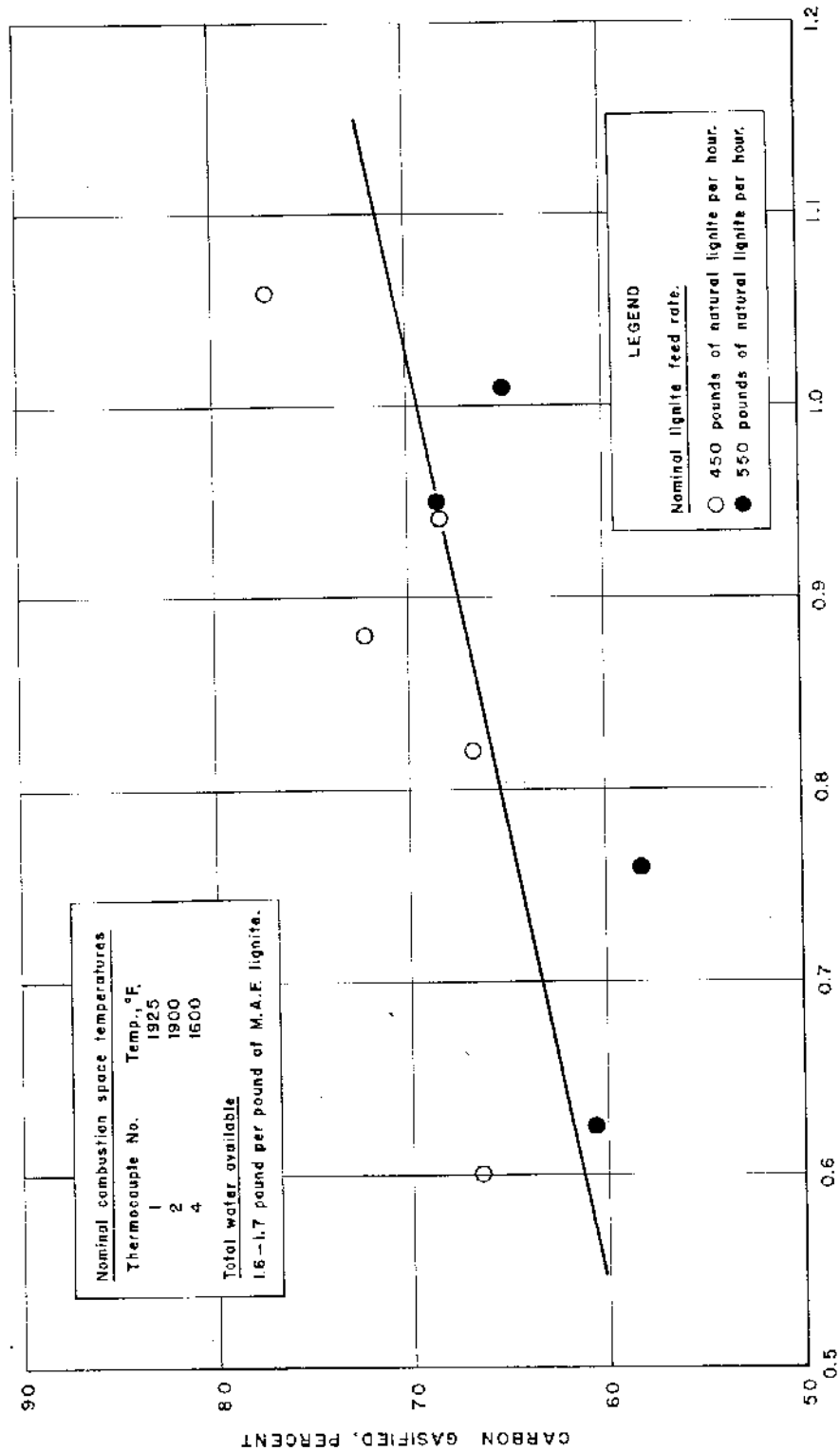

Figure 17. - Carbon gasification versus size of lignite as charged, periods 17-G through 17-O.

well with inner tube temperature (fig. 14, all data below 1,200° F.), and the trend of the data indicates primarily a heat transfer effect.

In considering gasification results of run 17, attention was also given to possible variation in gasification characteristics with change in composition of the lignites tested. Representative proximate and ultimate analyses of the seven lignites gasified during run 17 were given in table 2. The range of variation in proximate and ultimate analysis calculated on moisture- and ash-free basis is summarized in table 12. As is typical for lignites of the north central region, differences in ultimate composition on moisture- and ash-free basis were relatively small for the principal components, although the sulfur content of the lignites showed a substantial variation from 0.6 to 4.3 percent. Ash content and composition of the ash for lignites gasified during run 17 were listed in table 3.

TABLE 12. - Variation in composition of lignites gasified during run 17, m.a.f. basis

Component	Range, percent	
	From	To
Volatile matter	44.6	49.8
Fixed carbon	50.2	55.4
Hydrogen	4.7	5.0
Carbon	70.0	72.7
Nitrogen	0.8	1.3
Oxygen	19.8	22.6
Sulfur	0.6	4.3
Heating value, B.t.u. per lb.	11,940	12,310

Analysis of the gasification data for run 17 has indicated that the main differences in gasification characteristics may be explained in terms of changes in heat-transfer properties resulting from accumulation of ash deposits and, to a lesser extent, from change in size of lignite. As previously discussed, one factor that may have contributed to ash-deposit formation is ash fusibility of the lignites as charged to the gasifier.

In general, and except for the possible effect of ash composition and fusibility, the results show no satisfactory correlation with composition of the lignites tested. Such differences as may exist between the lignites are apparently masked by the main trends in heat transfer behavior.

H_2-CO Ratio of Product Gas

Composition of the product gas expressed in terms of H_2-CO ratio showed a wide variation during run 17, from a minimum of 2.12 to a maximum of 3.92. Results indicate that H_2-CO ratio of the product gas was determined primarily by the extent of carbon gasification and the amount of unreacted steam available to shift the composition of the product gas. In figure 18, H_2-CO ratio of the product gas during run 17 is plotted as a function of amount of steam leaving the gasifier per mol of dry product gas. A typical correlation is indicated.

Summary of Factors Affecting Gasification Characteristics

In general, the observed differences in gasification behavior during run 17 are explainable on the basis of heat transfer as the controlling factor. There is no

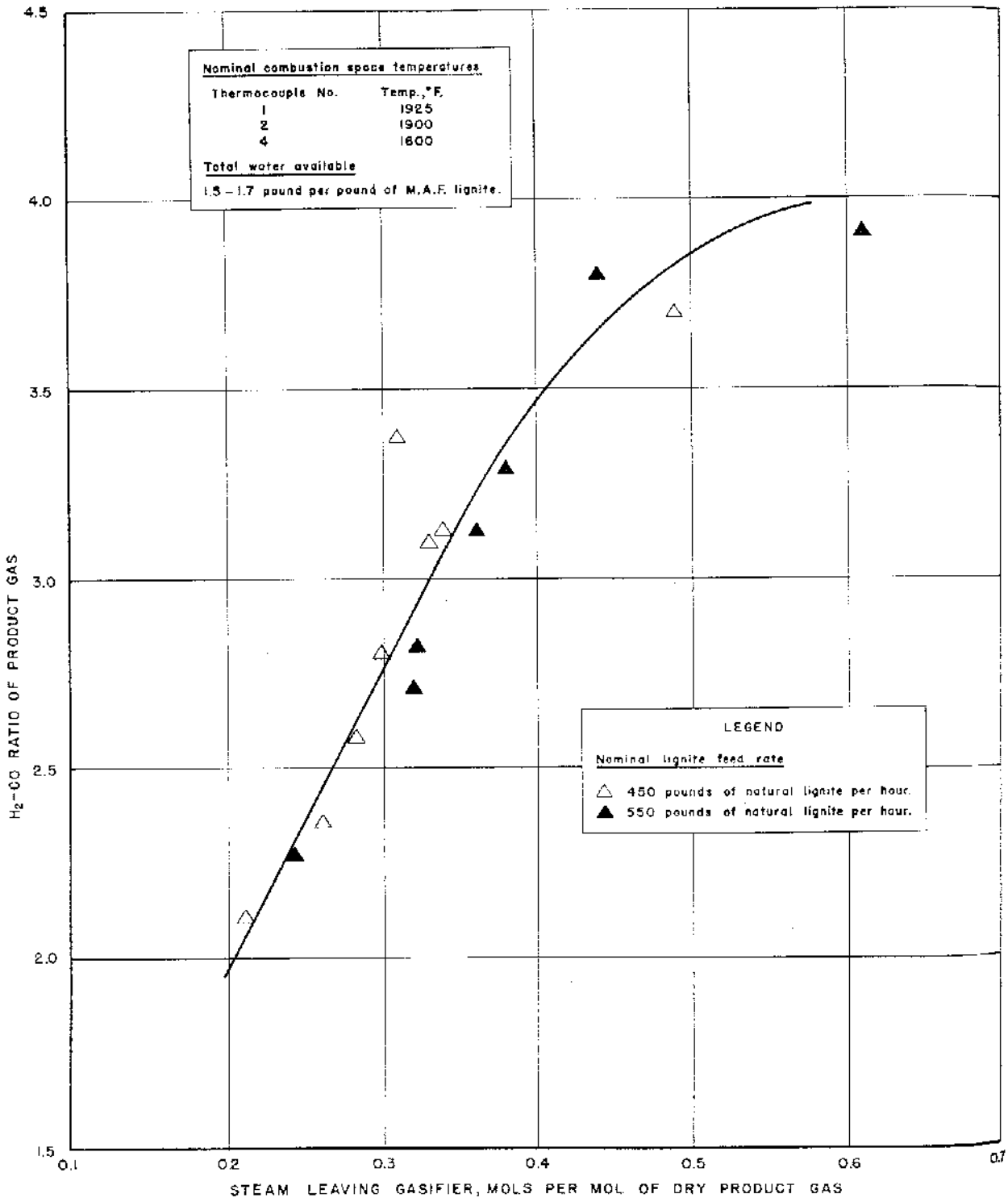

Figure 18. - H_2-CO ratio of product gas as a function of steam leaving gasifier, run 17.

definite evidence of differences resulting from differences in composition of the lignites tested.

During the period of ash buildup on the alloy tube this phenomenon was predominant in determining gasification rate and carbon gasified. The apparent effect of ash buildup on gasification capacity was greater than in previous runs. Factors that may have contributed to ash-deposit formation in run 17 are: (1) High and constant combustion-space temperatures, and (2) low ash fusibility of lignites charged in the first part of the run.

Results for the latter part of run 17, after buildup of ash deposits to approximate steady state, indicate that lignite size was an important secondary factor affecting gasification characteristics, again apparently mainly through effect on heat transfer.

H_2-CO ratio of the product gas was determined by the extent of carbon gasification and the amount of unreacted steam available for water-gas shift.

Performance of Retort Tubes

From the conception of the annular-retort gasification process interest has been centered upon the performance of the externally heated metal reactor. Because this metal reactor is a vital part of the process equipment, successful development of the process depended to a great extent upon proving by experiment that an alloy of suitable heat and corrosion resistance under process operating conditions was obtainable economically.

Previous reports have discussed performance of and reasons for discarding a spray-coated (metcolized) tube, an alloy-clad tube (4), and a cast HK-alloy tube (3). Information concerning the performance of the present rolled-plate 310-alloy tube for 1,862 hours of operation has also been presented (3). Table 13 summarizes operational data for the various tubes, including the additional 2,879 hours of operation in runs 14 through 17 with the rolled-plate tube. The 310-alloy rolled-plate tube has accounted for 54 percent of the total time the plant has been operated and has been used in producing 60 percent of the total gas made during development of the process.

Deformation of 310-Alloy Tube

In the last progress report (3), measurements of the reaction tube after each run were presented in detail. On the basis of the information available at that time, some concern was expressed that progressive deformation or creep of the tube at the relatively high operating temperatures might limit useful life more than chemical corrosion. Again after each run in the present series, as a continuing check on deformation, eight radial measurements were made at 1-foot intervals over the length of the tube. Data from runs 14 through 17 are presented in figure 19 and compared with the original measurements. The horizontal scale on this plot is greatly exaggerated to show the deformation. The results show that additional deformation during runs 14 through 17 was relatively small. Maximum deviation from the original 24-1/4-inch radius was + 26/32 and - 22/32-inch, which, particularly for the positive deviation, was nearly the same as observed during runs 11, 12, and 13 (3). It should be noted that the variations in length of radii did not in general show a consistent trend with increase in time heated during runs 14 through 17, indicating that deformation was not necessarily progressive.

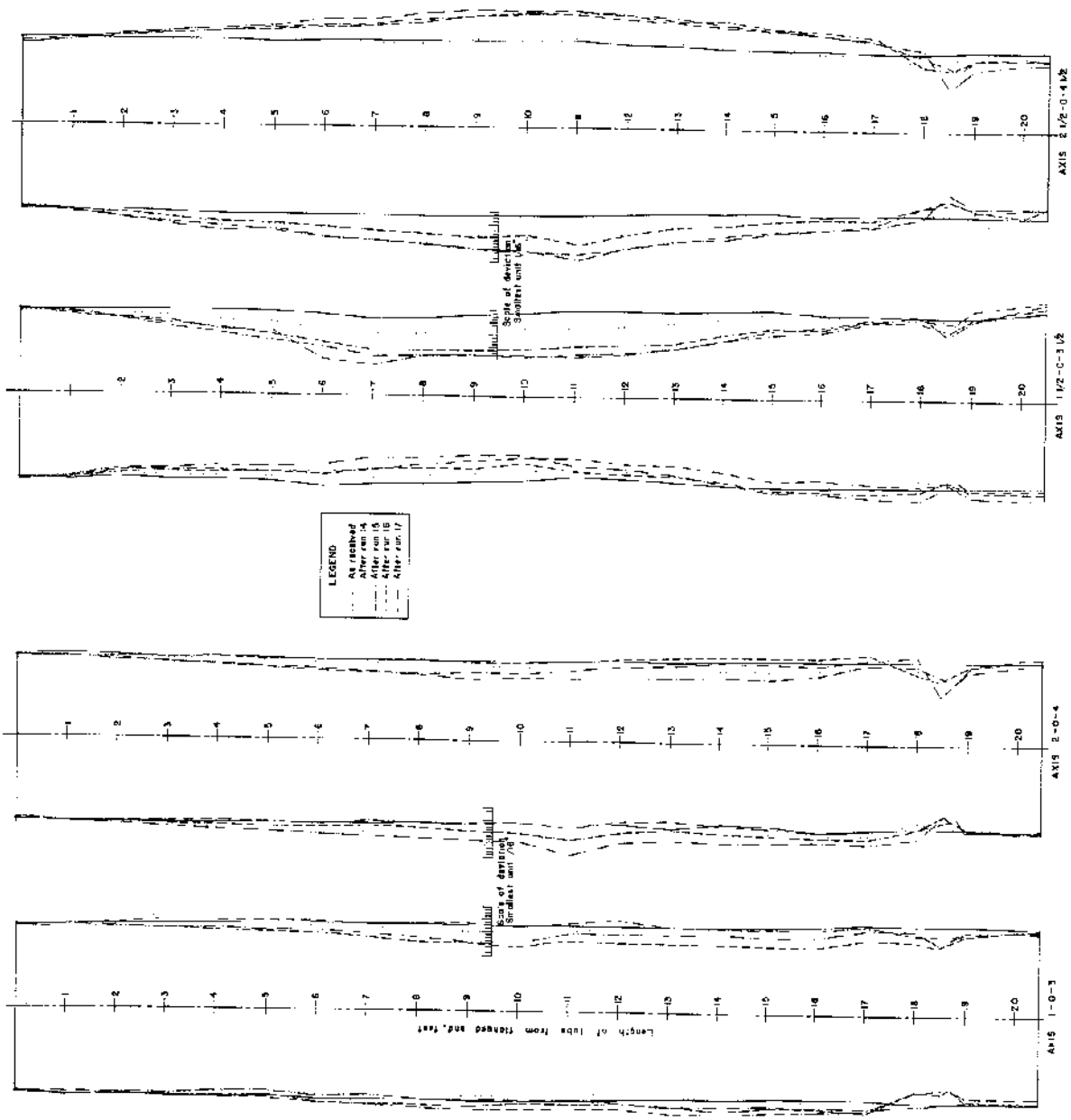

Figure 19. - Variation in radial dimensions of 310-alloy rolled-plate tube during runs 14, 15, 16, and 17.

TABLE 13. - Summary of operation using various reaction tubes

Run No.	Retort tube 1/	Date		Time operated, hours 2/	Lignite processed, tons	Gas produced, M cu.ft. 3/
		From	To			
Prelim. through 10..	1,2,3,4	2-27-45	1-21-49	3,910	4/761.9	33,228
11 through 13	5	5- 4-49	12-18-49	1,862	5/421.8	20,042
14	5	8- 1-50	9- 2-50	802	185.2	8,220
15	5	11- 8-50	11-27-50	466	129.0	4,874
16	5	4-20-51	5-16-51	637	153.1	6,965
17	5	8-11-51	9-20-51	974	219.2	10,261
Subtotal	5			4,741	1,108.3	50,362
Total		2-27-45	9-20-51	8,651	1,870.2	83,590

1/ 1 - Spray-coated (metcolized) tube; 2 - alloy-clad tube; 3 - reconditioned alloy-clad tube; 4 - cast HK alloy tube; 5 - 310 alloy rolled-plate tube.
2/ Time to nearest hour from lighting to closing of burners.
3/ Gas measured, 60° F., 30 inches Hg, saturated.
4/ Includes 7.6 tons of char.
5/ Includes 85.1 tons of steam-dried lignite.

The most noticeable deformation is the "nick" occurring at the 18.5-foot level measured from the flanged end of the tube. This nick first became apparent after run 13 during which the gasifier was operated at fairly high temperatures. This indentation is no greater than the deformation occurring at the 11-foot level, but it occurs rather abruptly and is easily visible. The 18.5-foot level is located opposite the first row of 6 burners where the combustion-space temperature is the highest. Within 2 feet toward the lower end of the tube the temperature has dropped approximately 800-1,000° F., thermally stressing the tube. The supporting brickwork for the bottom of the combustion space near the 19-foot level conforms closely to the tube and in conjunction with adjacent spacer bars on the inner tube may have imposed lateral stresses to start the observed deformation. Spacer bars on the inner tube at this level were removed after run 14 (see fig. 2). Also with the continuous annulus arrangement used after run 14, the gas offtake was lowered, maintaining higher temperatures below the level of the supporting brickwork.

From runs 14 to 17, the nick did not change greatly in character from run to run but did show a progressive tendency to increase in depth along certain axes (fig. 19). This further deformation appears to correspond to a compression effect. In each of these runs, support of the tube was arranged to give zero strain at one-third tube height as standard procedure. Adjustment to zero strain at one-third tube height left a substantial weight, about 1,300 pounds, for compression at the 18.5-foot level. It seems probable that the observed deformation at the 18.5-foot level could be arrested by lowering the point of neutral strain to that level.

Corrosion of 310-Alloy Tube

Previously, visual inspection and measurement of depth of corrosion pits on the alloy reaction tube after 1,862 hours of operation had indicated that chemical attack either on inside or outside of the wall was not serious (3). Careful observation of the corrosion deposits and pits after each run for a total additional operating time of 2,879 hours during runs 14 through 17 further strengthened this viewpoint as no appreciable increase could be noted.

The possibility of removing chromium and nickel with the gasification residue or char was considered. Neither nickel nor chromium could be detected in the char by conventional analytical methods. Low percentages of chromium and nickel were found, however, in the ash deposits adhering to the lower section of the tube wall after each run. Concentrations and total amounts of chromium and nickel in the ash deposits removed from the tube wall after runs 11 through 17 are listed in table 14. Total weight of chromium removed in the ash deposits for the 7 runs covering 4,741 hours of operation was 6.39 pounds. Total nickel removed was 1.64 pounds.

A rough estimate of the useful tube life with respect to nickel and chromium losses can be made based on the average rate of loss of these constituents during runs 11 through 17 and on the conservative assumption that a removal of 10 percent of either metal could be tolerated from the lower third of the reaction tube where the ash deposits were concentrated. On this basis, useful tube life with respect to chromium loss would be 33,000 hours and useful life with respect to loss of nickel would be 110,000 hours.

TABLE 14. - Chromium and nickel in ash deposits removed from alloy tube, runs 11 through 17

Run No.	Constituent[1] in deposit, percent		Weight of constituent removed with deposit, pounds	
	Chromium	Nickel	Chromium	Nickel
11	1.51	0.71	0.90	0.42
12	1.85	.24	.93	.12
13	4.31	.31	2.28	.16
14	1.16	.39	.60	.20
15	1.23	.47	.87	.33
16	.82	.16	.29	.06
17	.48	.32	.52	.35
Total..			6.39	1.64

1/ Percentage by weight of constituent in deposit removed from inner surface of alloy reaction tube after each run.

It can be assumed that as a result of the intermittent operation of the gasification unit and careful removal of the deposits after each run exposing a fresh surface to attack, losses of chromium and nickel indicated are greater than would occur during continuous operation over a similar period.

The outside of the 310-alloy tube showed no corrosion from products of combustion during the 4,741 hours that the tube was in use. The outer surface was discolored with a dark-greenish tinge, presumably from the formation of a thin tightly adhering film of chromium oxide that protected the underlying metal.

Figure 20 shows the lower flame guard of 310-alloy steel after more than 8,000 hours of direct exposure to temperatures as high as 1,950° F. No evidence of excessive corrosion was visible. From these observations it can be concluded that 310-alloy steel has enough resistance under normal combustion-side conditions to give indefinite tube life.

Corrosion of Mild Steel Inner Tube

The inner tube concentric with the alloy outer tube was fabricated from mild steel for reasons of low initial cost and ease of making alterations during development of the process. The outer surface of the inner tube was attacked somewhat by

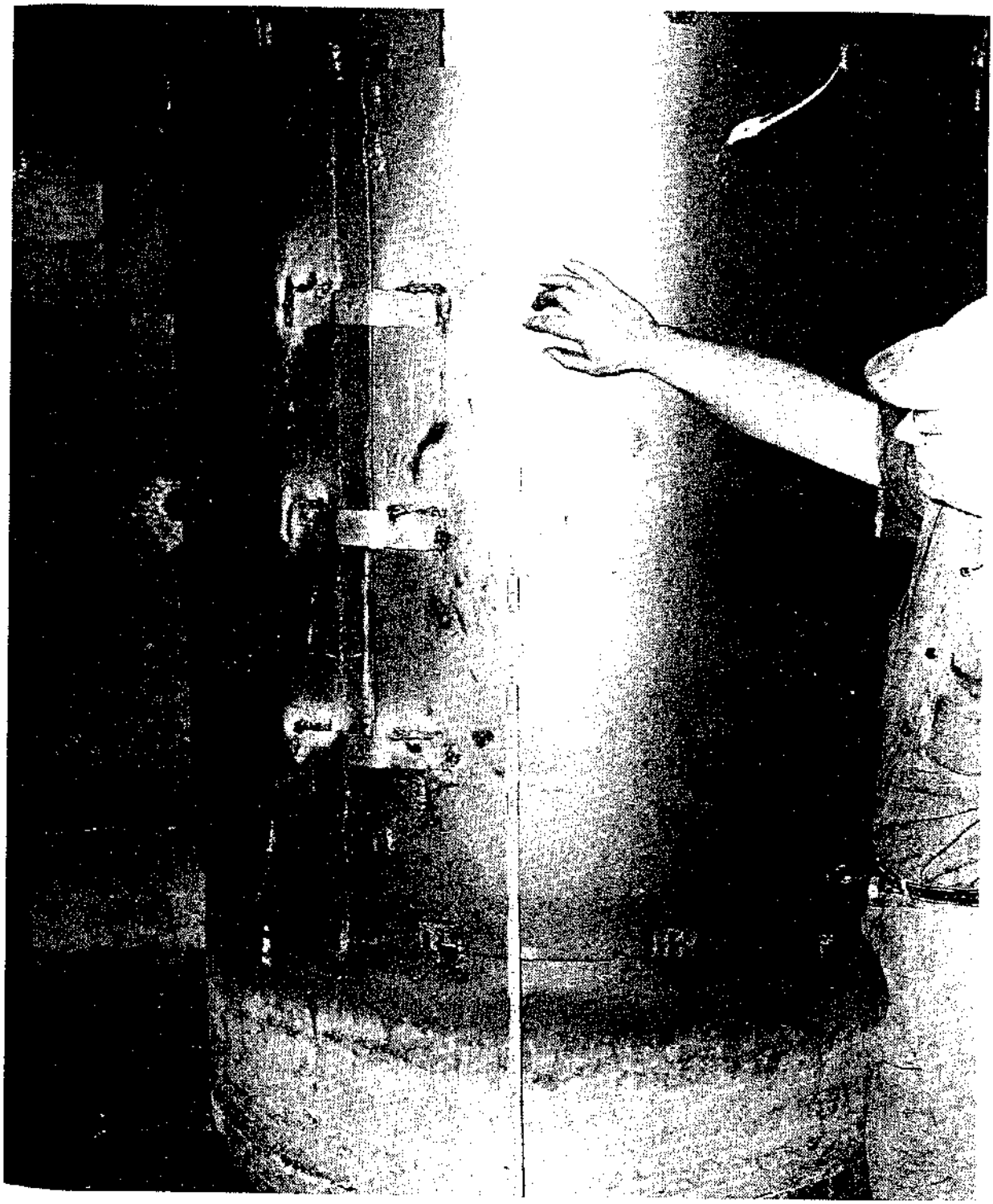

Figure 20. - Flame guard of 310-alloy steel after 7,000 hours of operation.