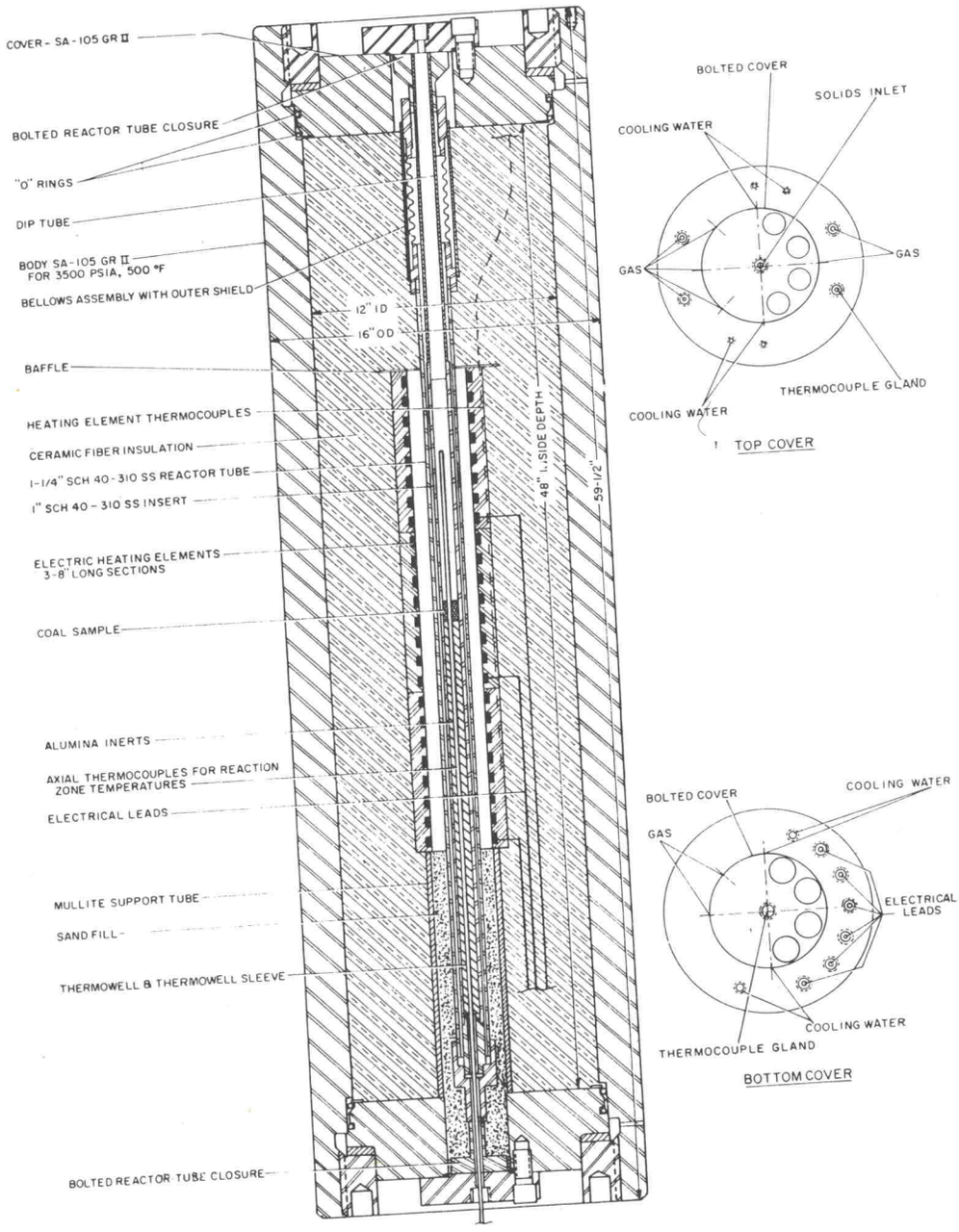

Figure 2. Balanced pressure reactor

Materials. In tests with hydrogen feed gas and steam-hydrogen feed gas mixtures, the hydrogen contained a small, accurately measured amount of helium or argon (usually about 1 mole %) as a tracer for exit gas flow rate measurement. The hydrogen–inert gas mixtures were mixed during compression and stored in a central gas storage system at pressures up to 3600 p.s.i.g. Commercially available grades of hydrogen (99.987% pure), helium (99.99% pure), and argon (99.998% pure) were used.

The coal char was separated by screening from the char used in pilot plant tests to ensure that the results obtained in these tests would be applicable to work to be done in the larger, pilot plant reactor system. This coal char was prepared by the Consolidation Coal Co. from Pittsburgh Seam bituminous coal from the Montour No. 10 mine by a low-temperature fluidized-bed pretreatment process. The analysis of the feed char is shown in Table I.

Procedure. In all the tests, a semiflow technique employing a flowing gas and a single, static coal char charge was used. It was essentially the same as that used in recent studies on the hydrogasification of solid fossil fuels (*5*, *6*). Test periods averaged about 1000 seconds.

Because heat losses by free convection from internally insulated reactors increase greatly when operating at high pressures, it was expedient to bring the reactor to operating temperature before pressurizing it to avoid long heatup times. When the reactor had reached the run temperature, the reactor pressure was brought to the desired level by increasing pressure inside the reactor tube. As pressure was increased, the pressure-balancing system maintained a balanced pressure on the reactor tube by admitting pressurized nitrogen to the insulated area surrounding the reactor tube. About 15 minutes was required to bring the pressure to 1000 p.s.i.g.

After the desired pressure was attained, the flow of reactant gas through the reactor tube was initiated. In tests with steam feeds, the reactor tube was first pressurized with nitrogen and then, when the steam generator pressure had been increased to slightly more than the reactor pressure, the steam was admitted to the reactor and the nitrogen was shut off.

This procedure was necessary to avoid introduction of large quantities of steam condensate into the instrument lines. In addition, since noncondensable exit gas flow rates were very small when pure steam feed gases were used, the exit gas system was purged at a controlled flow rate of 25 SCF per hour

Table II. Typical Results on Hydrogen–Coal Char Reaction

(Coal char sample weight, 2.5008 grams)

Time of sampling, sec.	0	20	30	40	60	100	180	360	480	600	1000	1500
Temp. at bottom of charge, ° F.	1800	1490	1510	1550	1662	1786	1800	1800	1800	1796	1800	1800
Unit pressure, p.s.i.g.	1010	1010	1010	1010	1010	1010	1010	1010	1010	1010	1011	1011
Feed hydrogen rate, SCF/hr.	39.3	39.4	39.3	39.1	39.3	39.8	39.8	39.8	40.8	40.8	40.8	40.8
Exit gas rate, SCF/hr.	39.3	40.0	39.5	39.0	38.1	38.6	39.0	39.7	41.0	41.0	41.0	41.4
Exit gas composition, mole %												
N_2	0.48	1.59	0.73	0.56	1.13	0.75	0.82	0.68	0.59	0.73	1.26	1.26
CO	...	...	...	0.40	0.50	0.20	...	...	...	...	...	...
H_2	99.47	98.36	99.21	97.44	92.19	94.19	97.09	98.62	98.96	98.93	98.54	98.59
CH_4	0.05	0.05	0.06	1.60	6.18	4.86	2.09	0.70	0.45	0.34	0.20	0.15
Total	100.00	100.00	100.00	100.00	100.00	100.00	100.00	100.00	100.00	100.00	100.00	100.00
Rate of formation of gaseous carbon, lb./lb. C fed-hr.												
As hydrocarbons	...	0.1	0.2	4.6	17.5	14.0	6.1	2.1	1.4	1.0	0.6	0.5
Total carbon	...	0.1	0.2	5.8	18.9	14.6	6.1	2.1	1.4	1.0	0.6	0.5
Conversion of carbon fed, %	...	...	0.0	0.3	10.4	29.4	51.6	70.0	75.5	79.5	88.2	95.6

with helium to avoid any distortion of the rate-time relationship because of backmixing or holdup in the exit gas system.

When feed gas flow rates, temperatures, and pressures had become completely stabilized, the feed gas was sampled. One minute later, the tests were initiated by opening the quick-opening ball valve between the hopper and the reactor. Single charges of either 2.5 or 5 grams of coal char were fed and reactant gas flow rates of 50 SCF per hour were employed in all tests. The coal charge was supported on a stainless steel screen on top of approximately 19 inches of high-purity alumina inerts in the form of cylinders, $^1/_8$ inch long by $^1/_8$ inch in diameter. Temperatures of the bottom of the coal char charge and of the centers of the top and bottom heating zones were recorded. Exit gas samples were taken in the early stages of a test at frequent intervals (as small as 10 seconds) and later as required to delineate the entire course of the reaction.

The first reaction products appeared in the exit gas sampling system in about 15 to 35 seconds. This holdup time depended on the feed gas used, holdup times being shortest in tests with pure hydrogen feeds and longest in tests with pure steam feeds. In tests with steam-containing feed gases, a constant liquid level was maintained in the sight glass, so that gas holdup times in the exit system would not vary. This was necessary because a sizable variation in gas holdup times could distort the gasification rate–time relationship. When the reaction rate had reached a value too small to be measured accurately, the run was stopped. The feed gas flow was stopped, the reactor heaters were turned off, and the unit was depressurized to minimize further reaction of the coal charge after the run.

The feed gas and helium-purge gas orifices were calibrated before each run with the exit gas wet-test meter. Exit gas flow measurements were made also during each run by a wet-test meter to provide a check on the flow rates calculated with the helium or argon tracers. Gas analyses were performed by mass spectrometer at high sensitivity. Carbon monoxide was determined by infrared spectrophotometer, since it was not possible to measure the relative amounts of carbon monoxide and nitrogen with sufficient accuracy in the presence of methane with the mass spectrometer.

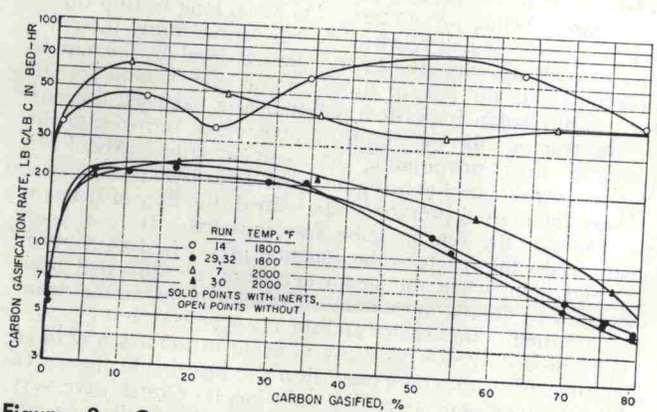

Figure 3. Comparison of results under conditions approaching adiabatic and approaching isothermal

Results

Hydrogen–Coal Char Reaction. Initial testing was done at 1000 p.s.i.g. and 1700° to 2100° F., with hydrogen feed gas, 5-gram coal char samples, and feed gas flow rates of 50 SCF per hour. At temperatures of 1800° F. and above, no consistent effect of temperature on rate of gasification could be observed. In addition, during the tests at 1800° F. and above, stainless steel screens which had been used to support the coal char charges were found to have melted, which indicated that very large temperature rises had occurred during these tests. Heat balance calculations made using finite difference techniques and assuming a heat of reaction equal to that for hydrogenation of beta-graphite to methane showed that, at the high rates of reaction obtained with this highly reactive feedstock, sample temperatures could easily rise to over 2500° F. from a starting temperature of 2000° F., even with the high gas flow rates and small sample sizes used. Further tests were conducted with 2.5-gram samples at 1700° to 2100° F. A 2.5-gram sample size was deemed the smallest that would give sufficient methane in the product gas for accurate measurement. However, even with such small samples, sample temperatures increased greatly. For example, when pieces of −16, +20 U.S.S. sieve size temperature-indicating pellets having fusion temperatures of 1700°, 1900°, 2100°, 2250°, and 2500° F. were mixed with the feed char in one check run made at 1700° F., both the 1700° and 1900° F. pellets melted.

Therefore, three more tests were conducted with 2.5 grams of coal char mixed with 30 grams of alumina inerts to act as a heat sink. The results of these tests at 1800° and 2000° F. (which approached isothermal conditions) are shown graphically in Figure 3 along with results of tests conducted without inerts at 1800° and 2000° F. (which approached adiabatic conditions). Typical tabulated data are presented in Table II. This dilution technique was used in all further tests with hydrogen feed gas, and all further results presented for hydrogen feed gas were obtained by this method. In tests where char and inerts were fed, the indicated bed temperature at first