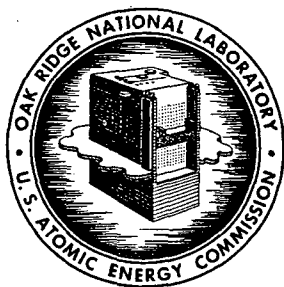


JUN 17 1964



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U.S. ATOMIC ENERGY COMMISSION



ORNL-TM-856

MASTER

DESIGN AND PRELIMINARY OPERATION OF THE EGCR
STEAM-GRAPHITE REACTION RATE EXPERIMENT

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R. E. MacPherson

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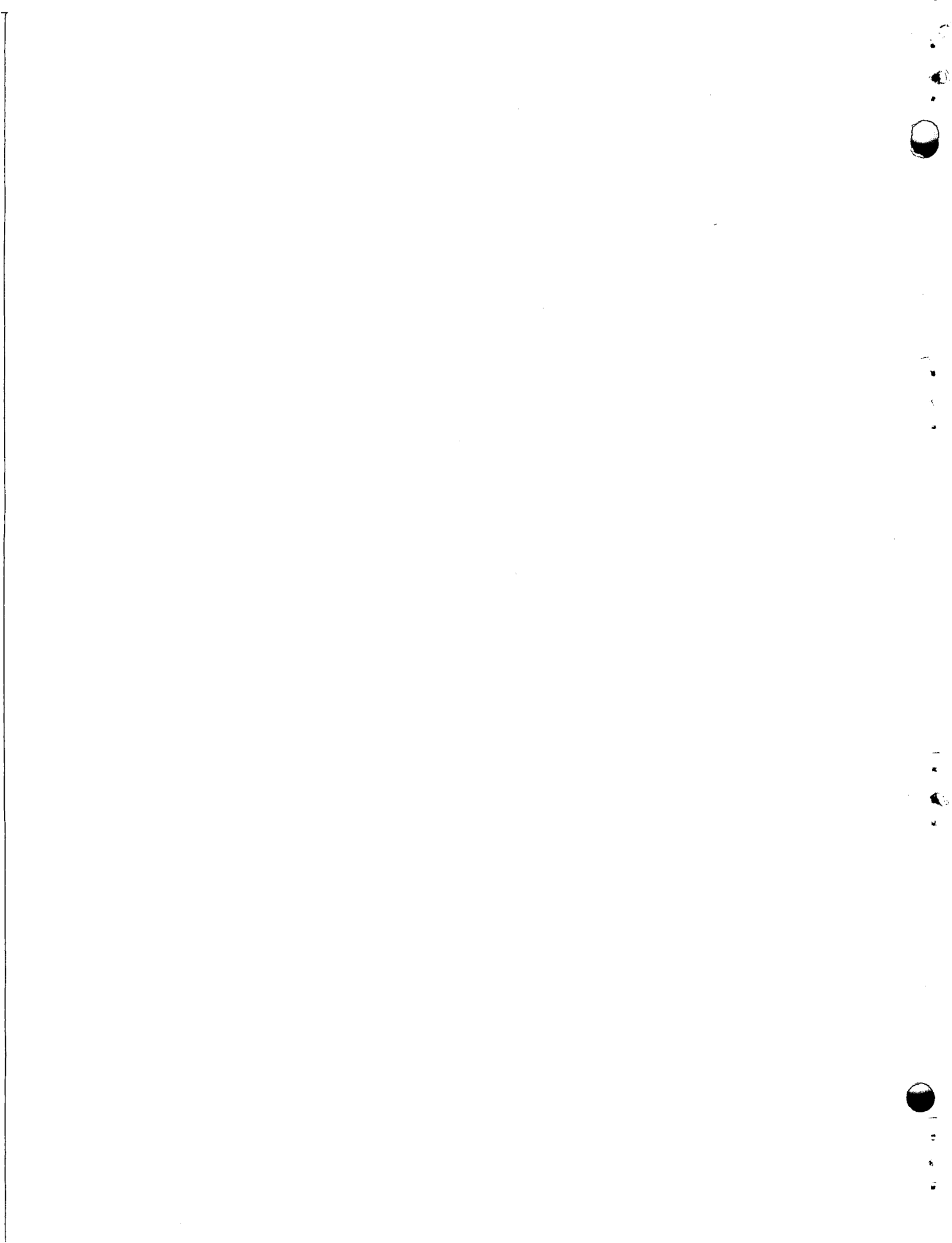
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R. E. Helms

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Abstract

A test facility has been designed, calibrated, and put into operation for the determination of reaction rates of graphite with high pressure, high temperature steam. The facility is designed to permit exposing graphite at temperatures of 1000-1800°F to steam in the pressure range of 30-450 psig at steam flow rates up to 120 lb/hr.

System calibration tests have been performed which have demonstrated the ability to measure reaction product gases quantitatively. These tests involved the introduction of carbon dioxide and hydrogen gas mixtures to the system and their subsequent recovery, measurement, and analysis for material balance purposes.

Initial studies will be concerned with the reaction of steam with full-scale Experimental Gas Cooled Reactor (EGCR) fuel element graphite sleeves and with identically shaped specimens of EGCR moderator graphite.

Two EGCR fuel element sleeves have been tested. During these tests, several operating problems were encountered and appropriate modifications were made to the system. As presently constituted, the facility can be employed for a wide range of investigations of steam-graphite reaction rates.

Introduction

The Experimental Gas Cooled Reactor (EGCR) is a graphite moderated, helium cooled reactor¹ designed to operate at a power level of 84.3 MW(t). The helium coolant system is operated at 315 psia with a fuel channel outlet gas temperature of 1075°F, producing superheated steam at 1250 psig and 900°F. The slightly enriched (2.46% U-235) uranium dioxide fuel is contained in stainless steel tubes which are assembled into seven-rod clusters and mounted in a graphite sleeve to form a fuel-graphite sleeve assembly. The fuel-graphite sleeve assembly is inserted in holes bored in the moderator graphite. The reactor core is composed of moderator graphite

containing 234 vertical fuel channels, each of which is charged with six of these fuel-graphite sleeve assemblies, axially aligned.

In the process of evaluating the hazards associated with operation of the EGCR, it has been necessary to examine the problems which might arise as a result of the rupture of the steam generator and the introduction of large quantities of steam into the helium coolant. One problem which must be considered is the hydrogen which is generated as a result of the reaction of steam with the moderator and the fuel-graphite sleeve assemblies. Two limiting conditions can be specified. The first of these is created by a rupture of the steam generator which results in steam entering the coolant stream and mixing with the helium inventory. For this condition, the steam-graphite reaction would proceed at a total pressure up to the level determined by system relief valve settings with some intermediate steam partial pressure. The second limiting condition would be created by a rupture of the helium coolant system which propagated a failure of the steam generator. In this latter case, the coolant system could be considered to contain essentially no helium, and the steam-graphite reaction would proceed at low steam partial pressures. Under both of these limiting conditions, the temperature of the graphite would be determined by after-heat considerations, heats of reaction, and control rod effects.

Because available literature^{2,3} on the steam-graphite reaction was not sufficiently definitive for the required hazards evaluation, a test program was proposed to provide reaction rate data for the specific graphites to be employed in the EGCR over the pressure and temperature ranges of interest.

An engineering-scale test facility has been designed and assembled which allows the measurement of steam-graphite reaction rates over the pressure range of 30-450 psig and the temperature range of 1000-1800°F. This facility is sized to permit the use of full scale EGCR fuel graphite sleeve assembly and similarly shaped samples of moderator graphite as test specimens. Steam flow rates are controllable up to a maximum corresponding to a steam Reynolds number of 10,000 at the reaction surface.

This facility has been operated thru a shakedown period and modified as a result of operating experience. As presently constituted, it is capable of precise measurement of steam-graphite reaction rates over the specified range of operating conditions.

System Description

Range of Variables

The EGCR steam-graphite reaction rate facility is designed to measure the rate of reaction between steam and graphite as a function of graphite temperature, steam pressure level, steam flow rate, and burn-off. The experimental program will be carried out on full scale specimens of EGCR fuel-graphite sleeve (Speer 901-RYL) and similarly shaped specimens of EGCR moderator graphite (National Carbon) at pressures up to 350 psig, temperatures to 1800°F and steam flow rates corresponding to steam Reynolds numbers at the reaction surface up to 10,000.

Mode of Operation

Three methods of testing were considered during early planning:

1. recirculating system using pure steam...,
2. recirculating system using steam and an inert gas...,
3. once-through system using steam and an inert gas....

The first method listed was the one chosen for the test program. Analysis of the problem of hydrogen generation due to the reaction of steam with graphite in the reactor core has led to the conclusion that the worst case is encountered when the coolant system is completely filled with steam.⁴ Design of the facility to accommodate recirculation of a steam-inert gas mixture, would allow investigation of the effect of inerts on the reaction rate, but the additional complexity introduced into the system does not seem warranted in view of the hazards-oriented nature of the present tests. The use of a once-through system is undesirable because of the large heat loads and inert gas usage which are inherent in testing full scale specimens at representative flow rates and because of the more difficult analytical problem in determining reaction product concentration in the once-through system discharge stream.

Test Facility

A high pressure natural (or forced) convection steam loop, built to determine the reaction rates of steam with graphite, is shown in Fig. 1

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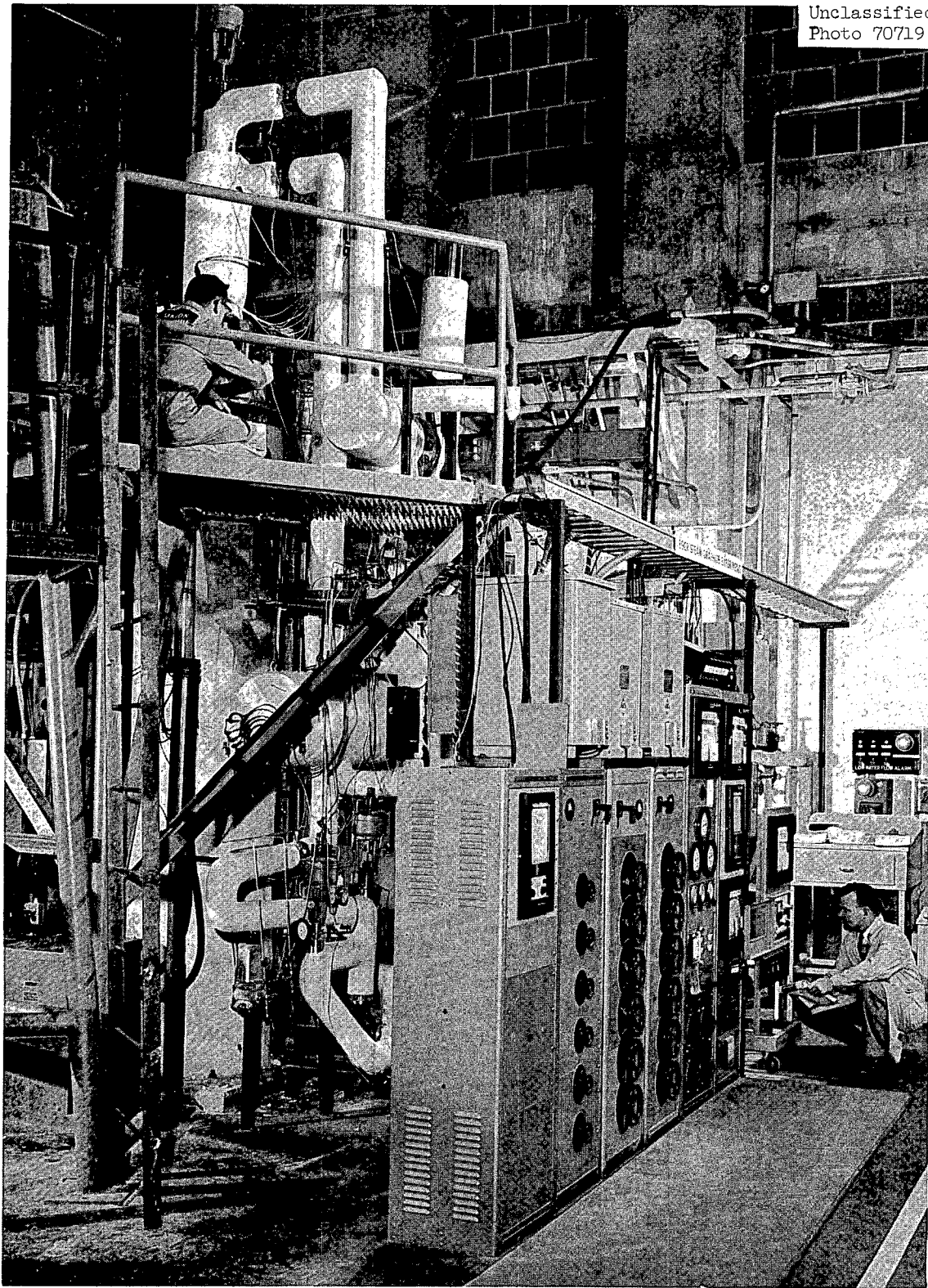


Fig. 1. Steam-Graphite Experimental Facility.

and Fig. 2. At maximum operating conditions, saturated steam leaves the boiler at 435°F and 362 psia and enters the regenerator at approximately the same temperature and pressure. As shown by the schematic diagram, Fig. 3, the steam flows downward through the regenerator, through the annulus between the insulated graphite test section and an outer shroud which protects the primary pressure vessel from direct contact with the steam, then through a second annulus between the superheater container and the outer shroud. The steam turns 180° and enters the superheater at 1400°F, exiting at 1800°F to the inlet of the test section. Since the specimen is isothermal, the steam graphite reaction occurs nominally at the test specimen entrance temperature. Steam flow leaving the test section enters the regenerator and passes through to the condenser where the steam and the noncondensable gases are separated by condensation of the steam. The condensate is returned to the boiler either by natural convection or by forced convection using the boiler feed pump.

The noncondensable gases are bled from the high point of the condenser through a separate reflux condenser which improves the efficiency of purging the noncondensable gases from the system. From the reflux condenser, the gases are bled through a liquid trap. At the outlet of the liquid trap the noncondensables are divided into two streams. One stream is fed at a constant flow rate to a gas chromatograph for analysis. The discharge from the gas chromatograph is connected to a wet test meter. The second stream, the amount of gas in excess of that required for analysis, is bled directly to the wet test meter. Thus, the wet test meter records the total volume of noncondensable gases produced in the system.

The heater, the test specimen, and the regenerator are housed in an available autoclave that was designed for 800 psig and 1200°F service. This vessel can be operated at 1400°F with a system pressure of 578 psia and meet the requirements of Section VIII of the ASME Pressure Vessel Code. Using this available vessel as the primary pressure containment, the three components are mounted within a flow guide tube designed only to provide structural support and isolation of the flowing steam from direct contact with the pressure vessel.

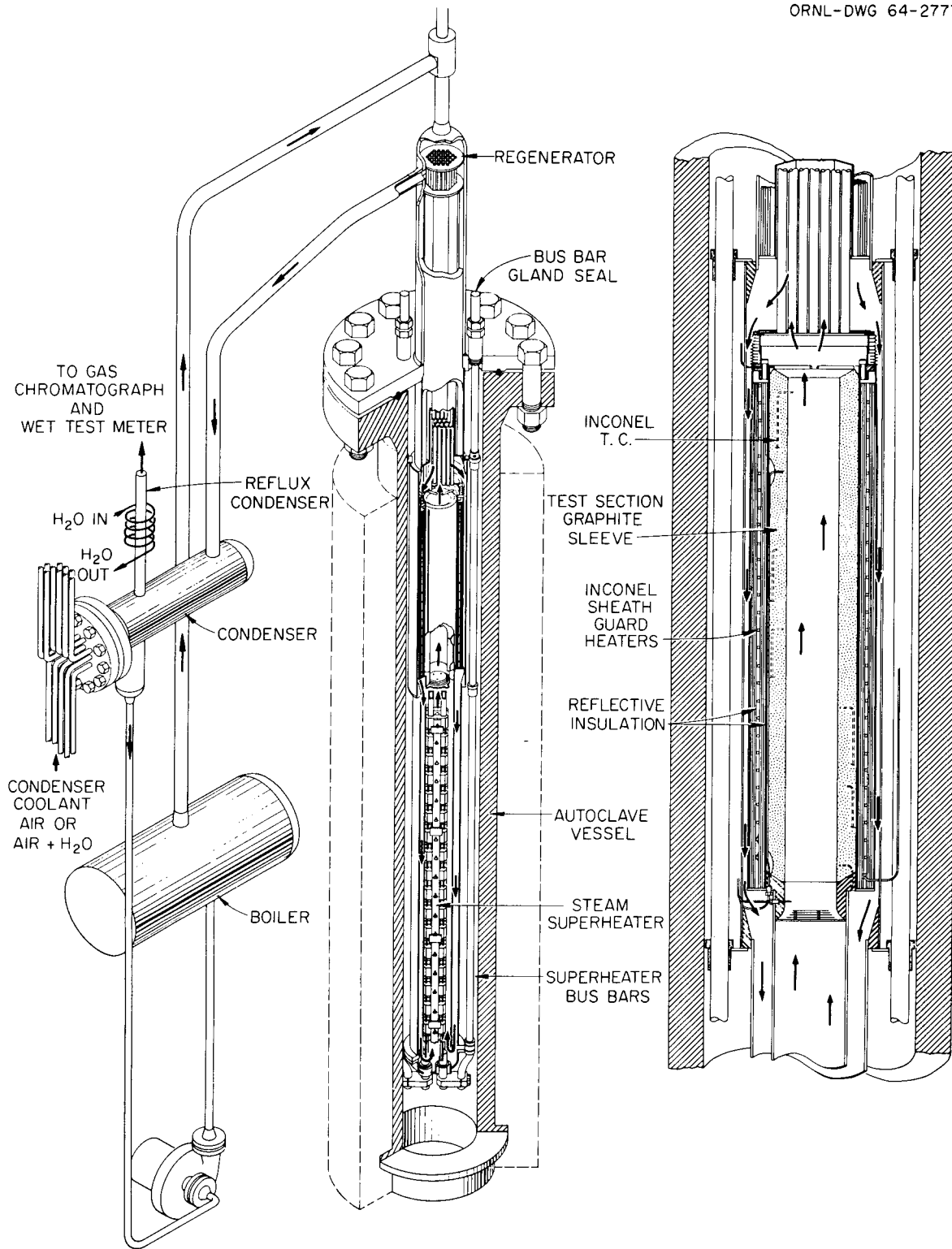


Fig. 2. Steam-Graphite Experimental Facility.

Test Specimen and Instrumentation

The test specimen is a 29 in. long graphite sleeve with a 3 in. inside diameter and a 5 in. outside diameter. The graphite test specimens of interest for the EGCR hazards investigation are the Speer 901-RYL fuel-sleeve graphite and a similarly sized moderator-graphite specimen machined from a piece of a National Carbon graphite core column.

During tests of the first two specimens of sleeve graphite (I & II) the test section did not have guard heaters to prevent heat loss from the test section. Radial heat flow from the test specimen thru layers of stainless steel reflective insulation was larger than anticipated, and as a result the test section could not be maintained isothermal. After these tests, the test section was altered to produce the geometry shown in Fig. 4. The test specimen was housed in a metal flow liner and reflective insulation was wrapped over the liner. Four Inconel sheath type heaters were spirally wrapped with a 1 in. spacing as shown in Fig. 5 and additional reflective insulation was wrapped over these heaters to complete the assembly.

Thermocouples are installed three at the inlet and three at the outlet of the 3 in. ID test specimen to measure the steam temperature. The signal from one of the inlet thermocouples is used to control the inlet steam temperature by adjustment of a Nichrome V strip heater that is located upstream of the test section. Seven thermocouples are inserted along the length of the graphite sleeve to measure any axial variation in the temperature of the specimen. These thermocouples are inserted from the outside to a location within 1/4 in. of the inside surface of the test specimen.

Boiler and Controls

The boiler (Fig. 6) is fabricated from 12 in. Schedule 80 316 stainless steel pipe and end caps. It is hung with the axis of the pipe in the horizontal plane to produce the maximum liquid surface area. The boiler is wrapped with 26 calrod heaters rated at 1500 watts each. This heat input to the boiler will produce 120 lb/hr of saturated steam at 435°F and 362 psia, the maximum operating conditions. Design conditions are 450 psig and 450°F.

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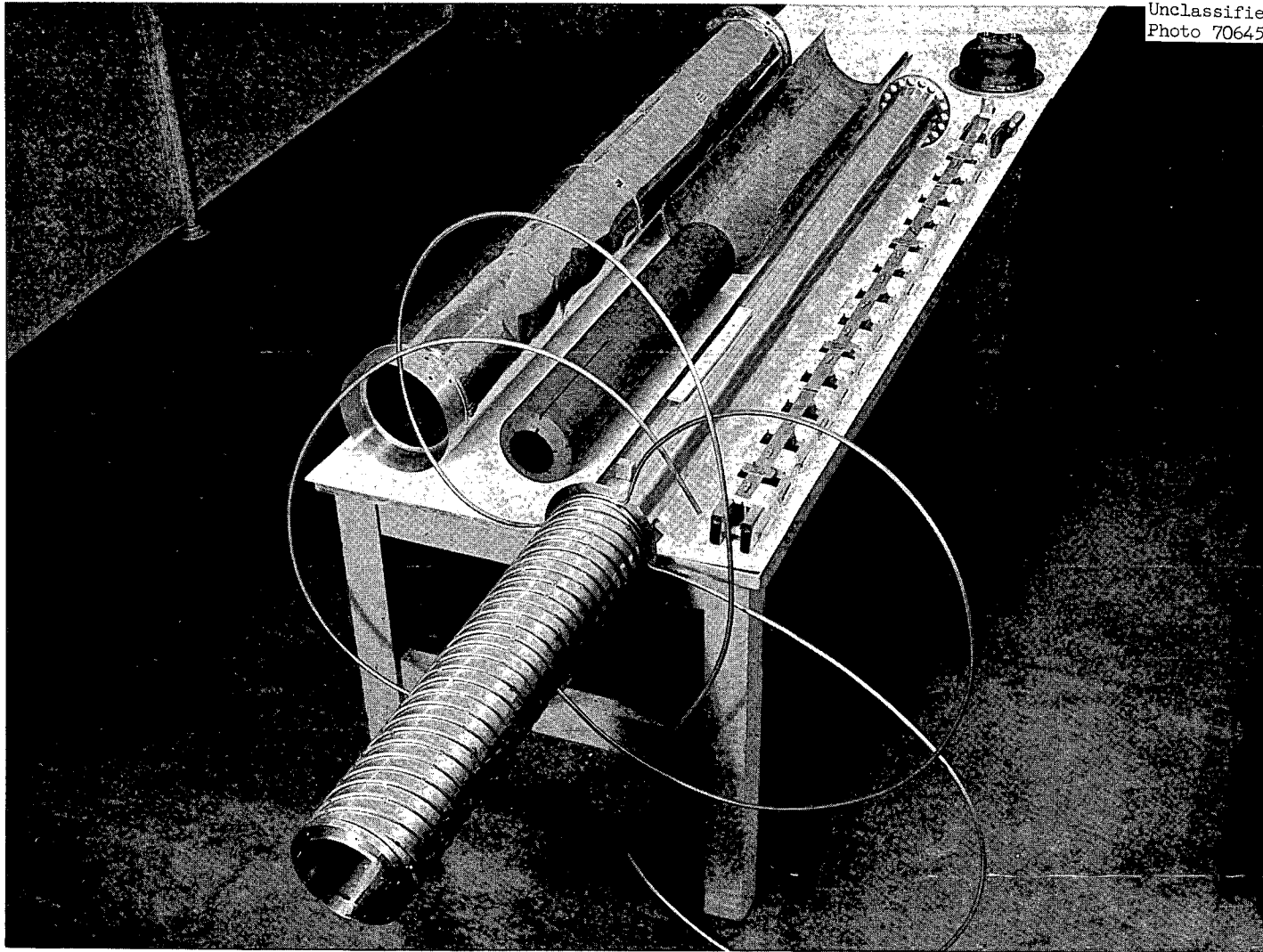


Fig. 5. Inconel Test Section, EGCR Graphite Sleeve III, Guard Heaters and Superheater.

The power to the boiler is supplied as shown by the power wiring diagram, Fig. 7. The safety alarm and auxiliary controls are shown in Fig. 8. A 1/8 in. thermocouple is inserted in the boiler liquid region through a Conax gland fitting to measure the liquid temperature. This thermocouple signal is fed to a Leeds and Northrop Series 60 "DAT" controller, No. 354165 which holds the boiler saturation temperature at the required setting.

A Meletron pressure switch Model 312 is connected to the boiler to prevent overpressurization of the system. The unit is set to cut-off the power to the boiler if the pressure exceeds the maximum operating conditions. A liquid level sight glass is also provided to permit observation of the boiler liquid level during operation.

Regenerator

A regenerator (Fig. 9) is employed in this loop design to limit the overall power requirements. This unit is a tube and shell heat exchanger made from 0.500 OD x 0.065 wall tubing arranged in a triangular pattern inside a hexagonal shell. The tubes are 4 ft 7 in. in length with the cold vapor outside of the tubes and the noncondensable gases and hotter vapor inside the tube.

The design inlet temperature to the regenerator from the boiler is 435°F with an exit temperature of 1400°F. The design inlet temperature from the test specimen is 1800°F with an exit temperature of 835°F. The design pressure and temperature of the containment vessel is 450 psig at 1000°F. Reflective insulation was installed between the containment vessel and the hexagonal shell side steam flow guide to reduce the vessel and flange design temperature.

Condenser and Liquid Level Control

The condenser (Fig. 10) is a U-tube and shell heat exchanger containing ten 0.75 in. OD stainless steel tubes. Steam is condensed on the outside of the tubes by air or an air-water mixture flowing through the tubes. The tubes are installed in a horizontal position inside a 6 in. pipe. The unit is designed for 450 psig pressure at 850°F.

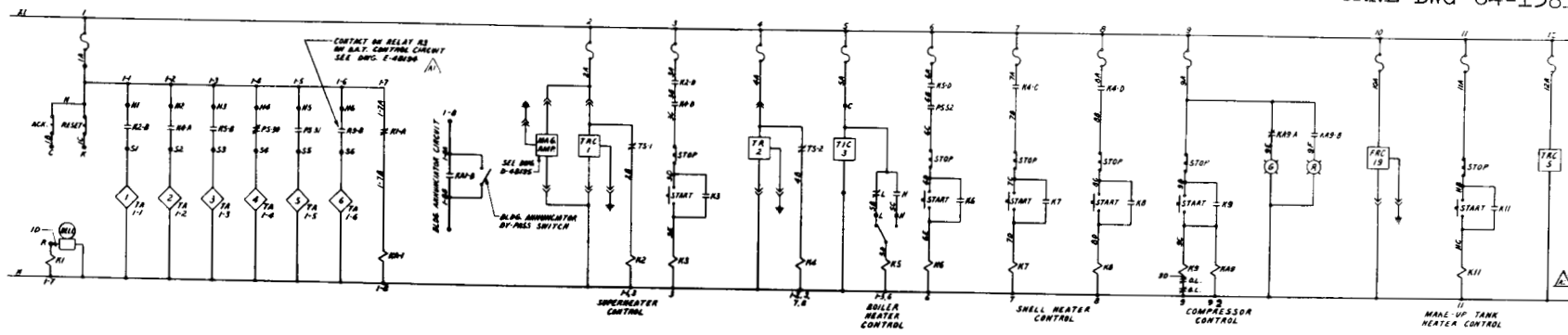
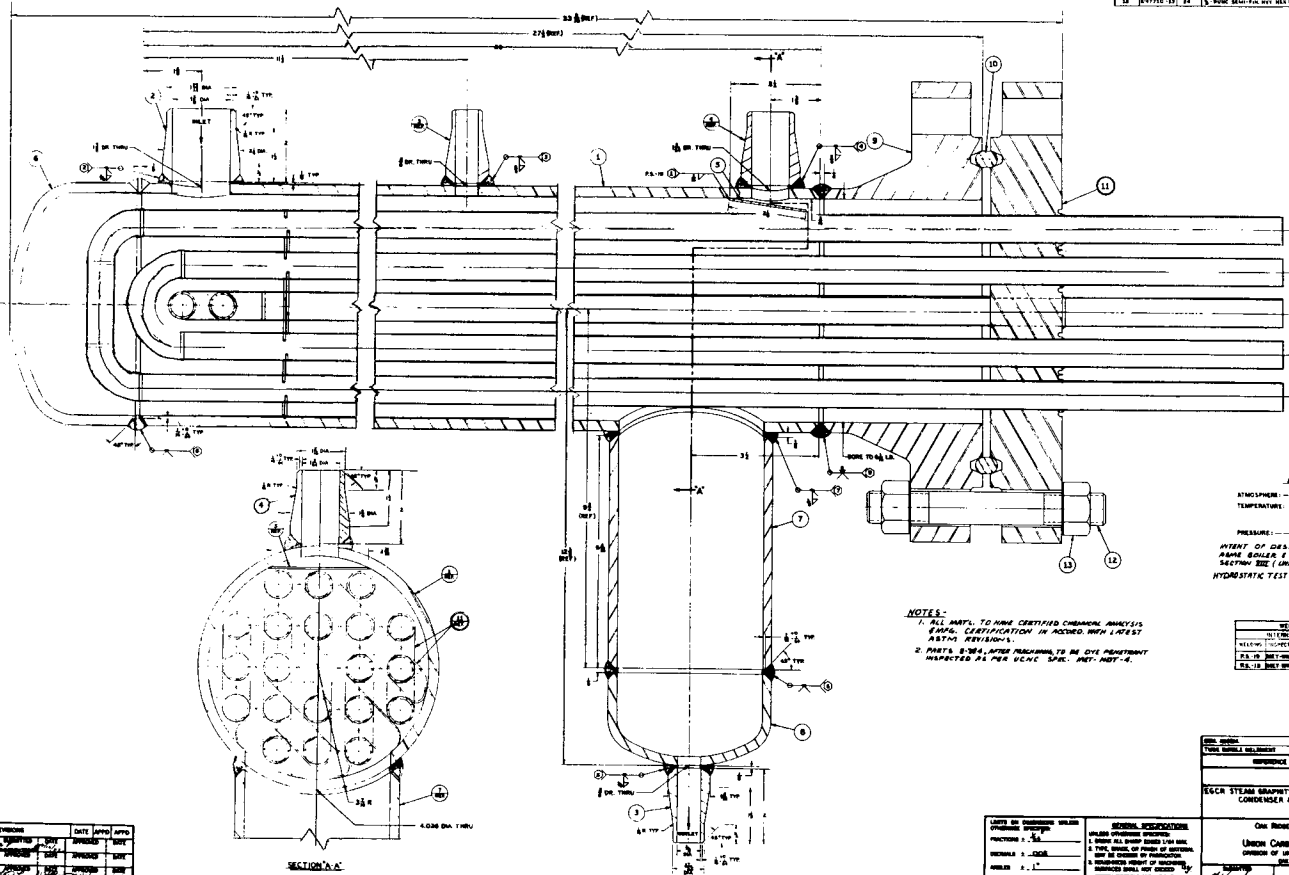


Fig. 8. Alarm Schematic and Auxiliary Controls.

Unclassified
ORNL-DWG 64-765

PARTS LIST				
ITEM NO.	QTY	DESCRIPTION (NAME, SIZE, ETC.)	MATERIAL	REMARKS
1	1	1" SCH 40 SEAMLESS PIPE 25' LB	SA-106	
2	1	1" SCH 40 SEAMLESS PIPE 25' LB	SA-106	
3	1	1" SCH 40 SEAMLESS PIPE 25' LB	SA-106	
4	1	1" SCH 40 SEAMLESS PIPE 25' LB	SA-106	
5	1	1" SCH 40 SEAMLESS PIPE 25' LB	SA-106	
6	1	1" SCH 40 SEAMLESS PIPE 25' LB	SA-106	
7	1	1" SCH 40 SEAMLESS PIPE 25' LB	SA-106	
8	1	1" SCH 40 SEAMLESS PIPE 25' LB	SA-106	
9	1	1" SCH 40 SEAMLESS PIPE 25' LB	SA-106	
10	1	1" SCH 40 SEAMLESS PIPE 25' LB	SA-106	
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15	1	1" SCH 40 SEAMLESS PIPE 25' LB	SA-106	
16	1	1" SCH 40 SEAMLESS PIPE 25' LB	SA-106	
17	1	1" SCH 40 SEAMLESS PIPE 25' LB	SA-106	
18	1	1" SCH 40 SEAMLESS PIPE 25' LB	SA-106	
19	1	1" SCH 40 SEAMLESS PIPE 25' LB	SA-106	
20	1	1" SCH 40 SEAMLESS PIPE 25' LB	SA-106	



DESIGN CONDITIONS:
 STEAM (WATER)
 TEMPERATURE: 633°F
 PRESSURE: 660 PSIA
 INTENT OF DESIGN TO BE IN ACCORD WITH
 ASME CODES & PRESSURE VESSEL CODE
 SECTION VIII (UNFURRED PRESSURE VESSELS)
 HYDROSTATIC TEST UNIT AT 766 PSIA @ 70°F(34)

NOTES:
 1. ALL WELDS TO HAVE CERTIFIED CHEMICAL ANALYSIS
 & WELD CERTIFICATION IN ACCORD WITH LATEST
 ASTM REVISIONS.
 2. WELDS TO BE INSPECTED TO BE ONE PERCENT
 INSPECTED AS PER UGHC SPK. INT. INT. 4.

WELDING REQUIREMENTS			
WELDING PROCEDURE	WELDING PROCEDURE	WELDING PROCEDURE	WELDING PROCEDURE
WELDING PROCEDURE	WELDING PROCEDURE	WELDING PROCEDURE	WELDING PROCEDURE
WELDING PROCEDURE	WELDING PROCEDURE	WELDING PROCEDURE	WELDING PROCEDURE

DESIGNER	E-4777
TITLE SHEET	E-4777
DATE	NOV 1964
SCALE	AS SHOWN

EG&CR STEAM GRAPHITE REACTOR RATE LOOP CONDENSER ASSEMBLY		REACTOR BUILDING
DESIGNED BY: [Signature]		DATE: 11/1/64
CHECKED BY: [Signature]		DATE: 11/1/64
APPROVED BY: [Signature]		DATE: 11/1/64
PROJECT NO. E-4777		REV. NO. 1

REV.	DESCRIPTION	DATE	APP'D.
1	ISSUED FOR FABRICATION	11/1/64	[Signature]
2	REVISION	11/1/64	[Signature]
3	REVISION	11/1/64	[Signature]
4	REVISION	11/1/64	[Signature]
5	REVISION	11/1/64	[Signature]
6	REVISION	11/1/64	[Signature]
7	REVISION	11/1/64	[Signature]
8	REVISION	11/1/64	[Signature]
9	REVISION	11/1/64	[Signature]
10	REVISION	11/1/64	[Signature]

Fig. 10. Condenser Assembly.

Since the system steam flow rate is directly controlled by the steam condensation rate, the condenser is designed to permit operation over a wide load range. Individual valves are provided on each of the coolant tubes. Combined with the U-tube geometry to avoid stress problems, individual control of each tube permits cooling with any or all tubes to obtain the desired steam flow rate through the test section. Provisions are also made for injection of water into the cooling air stream for operation at higher loads.

The condensate liquid level in the hot well of the condenser is controlled automatically by a valve located at the inlet to the boiler. A differential pressure transmitter is connected to the hot well of the condenser and to the vapor region of the condenser. Variations in liquid level are detected by the transmitter that produces the instrument signal to a Foxboro recorder controller that regulates the air-actuated valve. Figure 11 illustrates this instrumentation among other items.

Superheater and Controls

The superheater (right side of Fig. 5) is rated for 17 kw at 40 volts. The heating elements are Nichrome V ribbons two inches wide and .055 inches thick. All other metal parts are fabricated from Inconel. The heating elements are five feet in length and will fit inside a 4.0 in. ID tube.

The heater is designed to produce 120 lb/hr of 1800°F superheated steam at 362 psia with an inlet temperature of 1400°F. The superheater is located in the system upstream of the test specimen to control the inlet temperature to the test specimen.

At the inlet (bottom) end of the superheater, two electrodes extend through an end cap and connect to two 1 inch diameter Inconel bus bars. These bus bars run vertically from the bottom of the containment vessel to the top, penetrating the vessel flange through specially designed gland seal.

A Leeds and Northrop Speedomax H unit Serial No. 56-50033-1-2 was used to control a 40 kva 460 volt saturable reactor. The saturable reactor feeds a 40 kva transformer that supplies the power to the superheater as shown in Fig. 7. Fig. 12 shows the Speedomax H unit wiring diagram among other items.

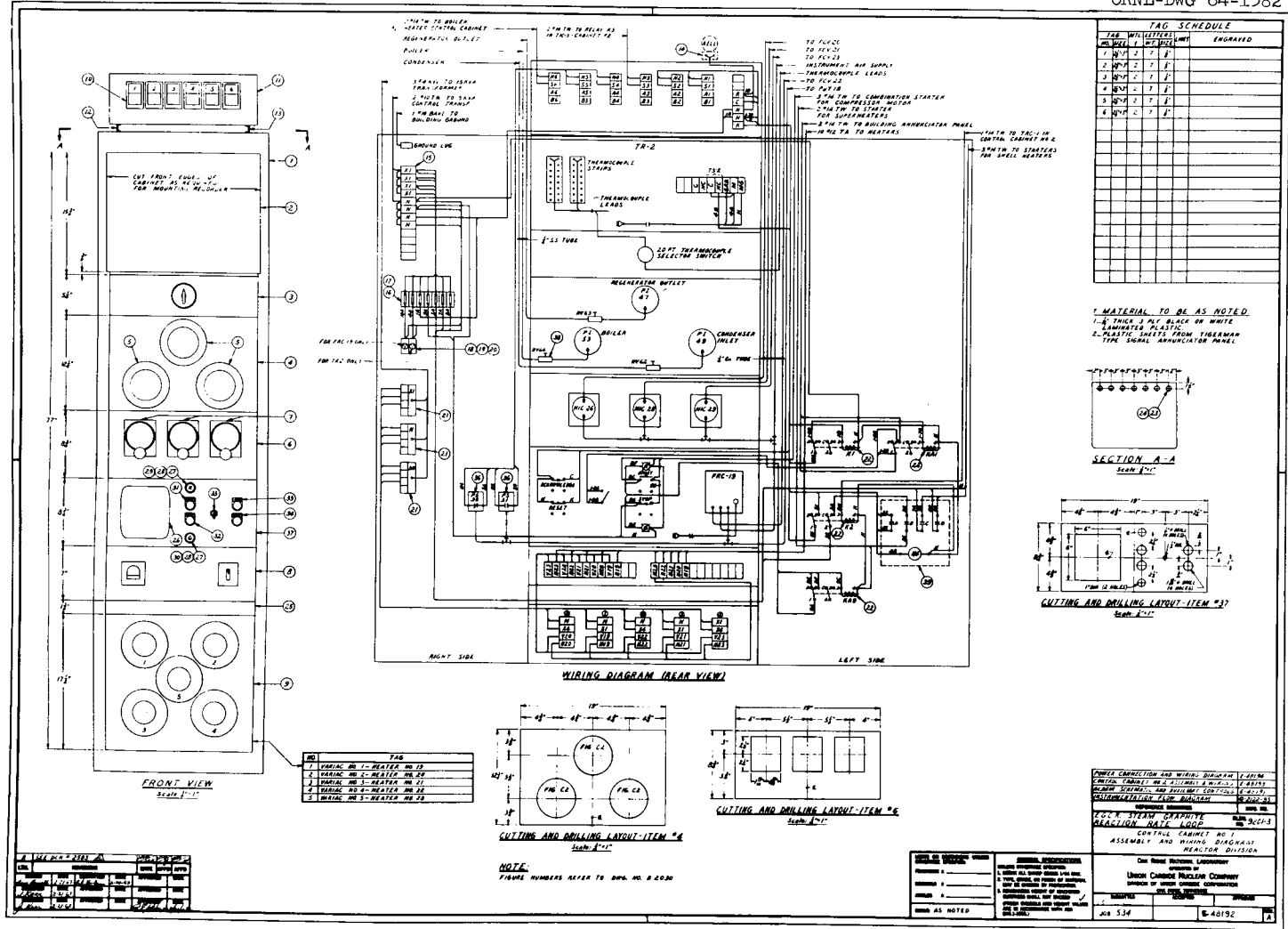


Fig. 11. Control Cabinet No. 1 Assembly and Wiring Diagram.

Condensate Pump

The condensate pump (Chempump Model GBT 7-3K-751-35) is a centrifugal pump purchased from the Fostoria Corp. The pump is a sealless unit with a canned rotor. The pressure containing parts of the pump are designed for maximum operating pressure of 425 psig at a temperature of 435°F. All parts exposed to demineralized water are fabricated from 316 stainless steel. The pump is designed to deliver 170 ft of head at a flow of one gallon per minute. A bypass line is installed around the pump to permit operation at a minimum flow of 1 gal/min independent of the system flow requirement. The pump was installed at the lowest point in the system.

Sample System and Gas Analysis

As described earlier, the noncondensable gases are bled from the main system through a reflux condenser that is connected to the highest vapor region of the condenser. The gases are bled to a liquid trap where small droplets of water are separated from the process stream. At the outlet of the liquid trap, the process gases are divided into two streams. One stream is metered to an on-line gas chromatograph for analysis, and the discharge from the gas chromatograph is connected to a wet test meter. The wet test meter is used to measure and record the total quantity of process gases produced from the test section.

The on-line gas chromatograph consists of three basic sections: the analyzer section, the control section and the recorder. The analyzer section employs chromatographic columns to separate the components of the gas stream and thermal conductivity sensors which have been calibrated against a gas of known concentration. The control section is designed to operate automatically, producing an analysis for H₂, O₂, N₂, CO₂, CH₄ and CO in this order on a twenty minute cycle. The recorder presents the analytical results in bar graph form.

Before connecting the gas chromatograph to the process system the instrument is calibrated by making an analysis of a standard gas (laboratory determined concentration) with a continuous recording of the thermal conductivity sensor output. With knowledge of the composition of the standard gas, the time at which individual components are evolved from the chromato-

graphic column can be determined. When a given gas component emerges, the appropriate cam on the programming timer is set so that an individual attenuating potentiometer is in the circuit at the time of the maximum sensor output for that component. These individual attenuating potentiometers are then set so that the peak height for each gas component will read percentage concentration to some convenient scale on the strip chart. After this calibration the instrument is shifted to automatic control and an unknown sample of gas from the process system is then bled into the instrument.

The analysis of the standard used to calibrate the gas chromatograph was determined by a mass spectrometer and by a separate gas chromatograph that had been calibrated using "pure gases" as standard. The results of these independent analyses were averaged and were used in setting the individual range attenuators. The maximum deviation of the average from the analytical results was within $\pm 1\%$. By careful calibration of the gas chromatograph, the analytical results for the process gas should be within $\pm 1\%$.

Operation

Start-Up of Test Rig

To operate the test rig, the vessel containing the regenerator, superheater and the test section is raised to 1000°F . This is accomplished using the Calrod heaters on the outside of the containment vessel.

The boiler is filled with demineralized water and the boiler Calrod heaters are turned on. The temperature set point on the boiler temperature control instrument is adjusted to correspond to the desired pressure level for the test. The condenser air coolant is turned on to start flow through the system. As the steam condensate accumulates in the condenser, the level in the condenser hot well is automatically controlled by either gravity flow or by pumping the condensate back to the boiler. An air-actuated valve regulates this flow as dictated by a differential pressure sensor located between the liquid level and the vapor region of the condenser. The system flow rate is established by adjusting the air coolant flow rate to the condenser.

When the condensate flow and the system pressure have reached a stable operating condition, the power to the superheater is turned on. The inlet temperature to the test specimen is increased to the desired temperature for a test. The set point on the superheater control corresponds to the inlet temperature to the test section and is maintained automatically by the superheater control. When the desired inlet temperature to the test section is established and stable system operation is obtained, the guard heaters around the test section are turned on. The power to the guard heaters is adjusted to produce an isothermal test section.

As the noncondensable gases from the steam-graphite reaction collect in the vapor region of the condenser, the micrometer valves to the gas chromatograph and wet test meter are adjusted until the bleed off rate from the system is equal to the production rate of the gas as indicated by some carry-over of condensate to the liquid trap downstream from the reflux condenser. The test run is continued at steady state conditions from four (4) to sixteen (16) hours to obtain one reaction rate data point. Different operating conditions are obtained by adjusting either the temperature set point on the boiler which changes the system pressure, or by changing temperature set point on the superheater and the guard heater to the test section to produce a different specimen temperature level.

Calibration Test

Of primary concern in the measurement of reaction rates by this apparatus is the efficiency of transfer of noncondensables from the test section to the condenser and of removal of noncondensables from the condenser volume to the analytical train. To establish the adequacy of the design, a calibration test was performed. With an empty test section, a graphite reaction was simulated by bleeding in a mixture of hydrogen and carbon dioxide through calibrated flowmeters into the boiler vapor region. These gases were recovered as they collected in the condenser by blowing down the condenser volume to a calibrated sample tank, shown in Fig. 3. The quantity of noncondensable gas in the sample tank was calculated and compared with the amount bled into the system. The test section inlet temperature was maintained at 1200°F with the condenser pressure at 140 psig during these test.

Determination of Gas Input to System. Bottles of CO_2 and H_2 were connected through pressure regulators and calibrated flowmeters to the vapor region of the boiler. The flowmeters were calibrated by means of a wet test meter, while maintaining an inlet pressure of 200 psig to the flowmeters. The flowmeters were subsequently operated at this inlet pressure level during the tests. The indicated flow rates on the flowmeters were monitored by a technician while the gas was fed into the system. The amount of gas fed into the system was calculated from the indicated flow rate (converted to standard conditions of 70°F and 14.7 psia) and the time.

Determination of Gas Output from System. The gas was purged from the system by cracking the valve between the condenser and the sample tank. At the completion of the test the valve was closed and the sample tank temperature was allowed to stabilize. During the blowdown operation some steam was bled into the sample tank with the noncondensable gases. When the sample tank had cooled to approximately room temperature, and the steam that was carried over with the gas had condensed, the sample tank pressure and the barometric pressure were read. The liquid was then drained from the sample tank into a thermos bottle, the temperature of the water was recorded, and the total liquid volume was measured. A sample of the gas was bled into a sample bomb for qualitative analysis by mass spectrometer.

The quantity of noncondensable gas in the sample tank was calculated from the following relationship:⁵

$$V_o = (V_T - V_L) \frac{T_{STD}}{T_2} \left(\frac{P_2 - P_{H_2O}}{P_{STD}} \right) \quad (1)$$

where

- V_o = Volume of noncondensable gas, cc (STP)
- V_T = Volume of sample tank, 14,000 cc
- V_L = Volume of liquid water drained from sample tank, cc
- T_{STD} = Standard temperature, 530°R
- T_2 = Temperature of liquid drained from tank, $^\circ\text{R}$
- P_2 = Stabilized pressure of sample tank, psia
- P_{H_2O} = Saturation pressure of water vapor corresponding to T_2 , psia
- P_{STD} = Standard pressure, 14.696 psia

Test Results. The gas volume recovered was compared with the input volume to determine the accuracy of this experimental procedure. The following table of results shows that the noncondensable gases injected can quantitatively be recovered from the system by the blow-down technique.

Table of Results

Test	Gas	Vol. Input cc (STP)	Vol. Output cc (STP)	% Error
CH-10-1,2	CO ₂ , H ₂	73,500	73,680	±0.25
CH-11-1	CO ₂ , H ₂	101,400	103,600	±1.93
CH-12-1	CO ₂ , H ₂	110,060	114,700	±4.20
CH-14-1	CO ₂ , H ₂	90,600	91,100	±0.55

Steam-Graphite Test

EGCR Graphite Sleeve Test I

After the calibration test, a reflux condenser was added at the high point of the condenser to reduce the amount of water vapor mixed with the noncondensable gases that are bled from the system. The outlet of the condenser was connected to a back pressure regulator which was set to relieve at 150 psig. From the back pressure regulator, the noncondensables were bled to the calibrated sample tank. An EGCR graphite sleeve (Fig. 13) was installed in the test section, and the results shown by Fig. 14 were obtained at approximately 1400°F test piece inlet temperature and 150 psig system pressure.

To obtain a reaction rate data point (Fig. 14) the noncondensable gases were diverted from the atmospheric vent line to the sample tank when steady state conditions had been achieved. The total time of blow-down to the sample tank was noted and the total quantity of gas collected in the sample tank was calculated using Eq. 1. A sample of the gas in the tank was bled into a sample bomb and the gas was analyzed by a mass spectrometer. Using the total calculated quantity of gas and the time of the data run, the gas production rate was calculated as follows:

$$R_G = \frac{V_G}{t} \quad (2)$$

where

Unclassified
Photo 70643

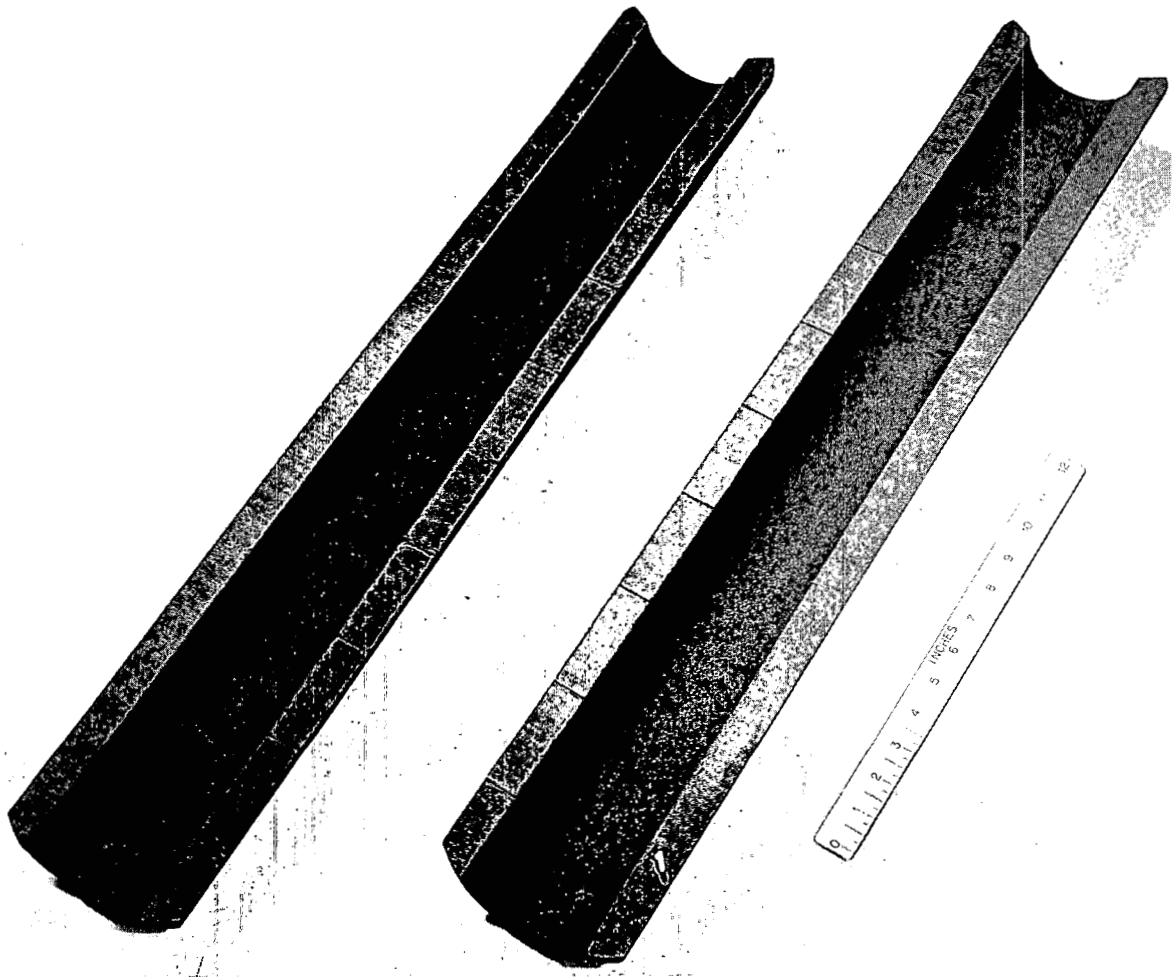


Fig. 13. EGCR Graphite Sleeve I -- After Exposure to Superheated Steam for Approximately 455 Hours at 1400°F Inlet Temperature.

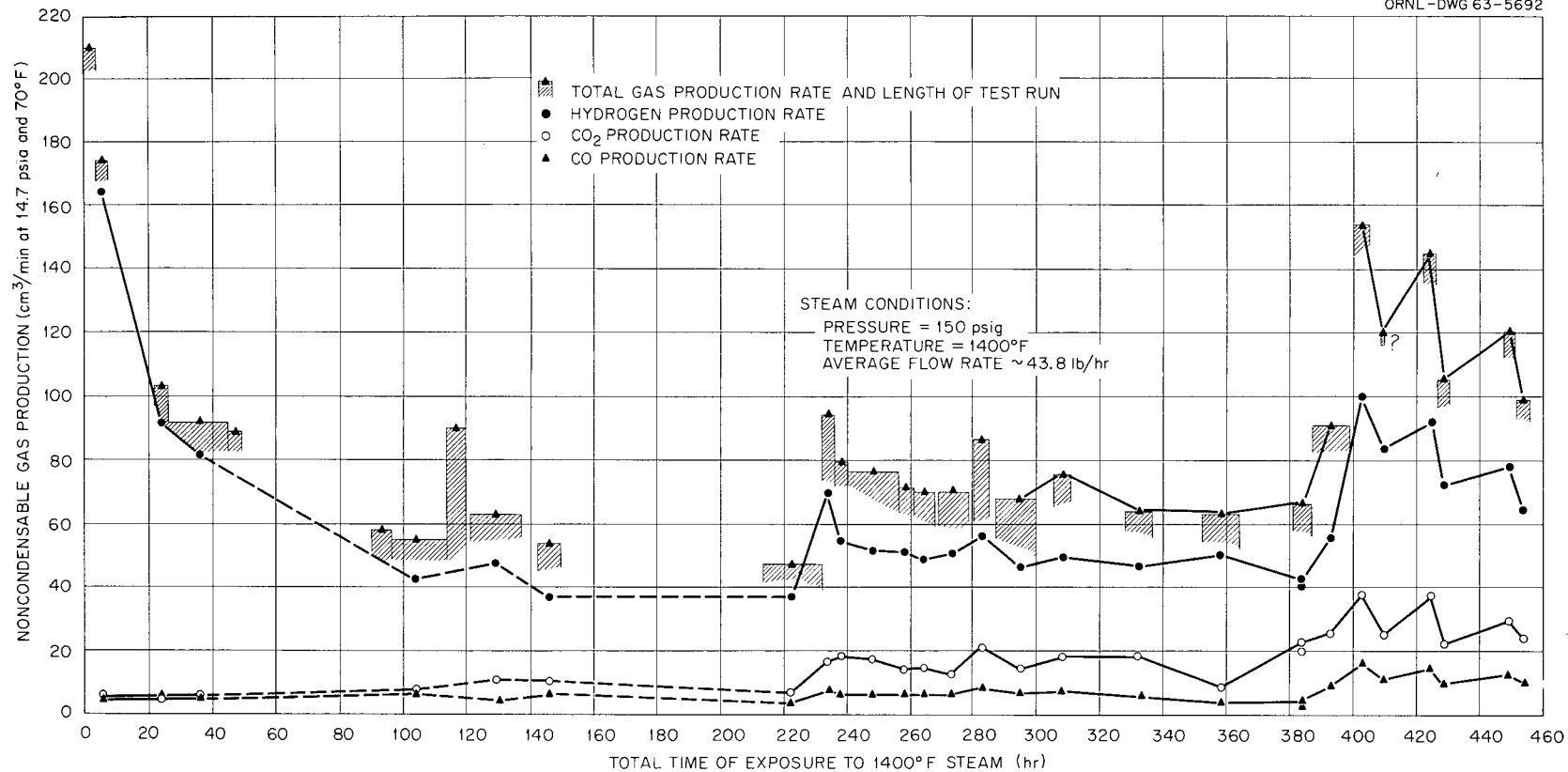


Fig. 14. Rates of Production of Noncondensable Gases Formed by Reactions of Steam and Graphite in a Forced-Convection Loop. EGCR Graphite Sleeve I.

R_G = Production rate of noncondensable gas, scc/min

V_G = Volume of noncondensable gas at 14.7 psia and 70°F

t = Total time of the blow-down or total time to obtain a data point, min

Using the concentration of each component in the gas (CO , CO_2 , H_2 , N_2 , O_2 , CH_4) obtained from the mass spectrometer analysis, the production rate of each gas component was calculated as follows:

$$R_x = X R_G = X \frac{V_G}{t} \quad (3)$$

where

X = concentration of each gas component, % by volume

R_G = production rate of noncondensable gas, scc/min

R_x = production rate of each gas component (CO , CO_2 , CH_4 , H_2 , N_2 , O_2), scc/min

Since this experimental procedure was basically a batch process, steady state gas generation rates were, of necessity, assumed to exist when system operating conditions were reasonably stable. In subsequent tests, modifications were incorporated to allow measurement of gas generation rates over much shorter test periods. Thus the question of whether equilibrium had been achieved could be based directly on gas generation rate measurements.

EGCR Graphite Sleeve Test II

After the completion of EGCR graphite sleeve test I, the experimental facility was altered to permit continuous measurement and analysis of the gas produced during a data run. An on-line gas chromatograph was added to analyze the gas and a wet test meter was added to measure the total amount of gas being continuously bled from the system.

The gas chromatograph was calibrated to monitor and record the concentration by volume of H_2 , O_2 , N_2 , CO_2 , CH_4 and CO gases, producing one complete analysis every twenty (20) minutes. The concentration of O_2 and N_2 were monitored as a means of leak detection. The concentration of these latter gases was less than 0.01% during all the test operations reported herein. The gas chromatograph was calibrated with a known mixture of gases containing all components of interest at representative concentration levels.

When the system had reached steady state operating conditions, the reading on the wet test meter and a time reference point were recorded on an hourly basis. From the difference in reading on the wet test meter, the volume of noncondensable gases produced from the system, V_G , was determined. Using the time interval, t , and Eq. 2 the gas production rate, R_G , was calculated. Using the gas analysis X from the gas chromatograph, the gas production rate, R_G , and Eq. 3 the production rate of H_2 , CO_2 and CO were calculated. Each point on the graphs shown by Fig. 15 thru Fig. 19 represents a data point obtained in this manner.

When the bleed-off rate from the system is not as large as the gas production rate, gas will build-up in the condenser thus blanketing the condenser tubes. This reduction of the condenser effectiveness causes a decrease in the flow rate through the system as shown in the left hand portion of Fig. 17 and right hand section of Fig. 18. Uncorrected, this condition could result in complete loss of steam flow over a period of time.

Conversely, a bleed-off rate greater than the gas production rate will result in carry-over of steam condensate into the flow control valves and the analytical train. The former condition leads to erratic flow control while the latter condition could inactivate the adsorbent in the gas chromatograph. In order to permit smooth operation with bleed rates equal to (or slightly greater than) the gas production rate, a water separator was introduced into the bleed line just downstream of the reflux condenser. With this unit in place, bleed-off rates could be adjusted to avoid gas accumulation in the system without the attendant difficulties of steam condensate accumulating in the flow control valves or analytical train.

Using the CO_2 , CO , and CH_4 gas production rates, the steam-graphite reaction, in terms of the amount of carbon reacting per unit of time, was calculated using the following equation:

$$R_C = \left[R_{CO_2} + R_{CO} + R_{CH_4} \right] \left(\frac{12 \times 60}{22,400} \right) \quad (4)$$

R_C = grams of carbon reacting per unit of time, grams/hr

R_{CH_4} = production rate of CH_4 gas, scc/min

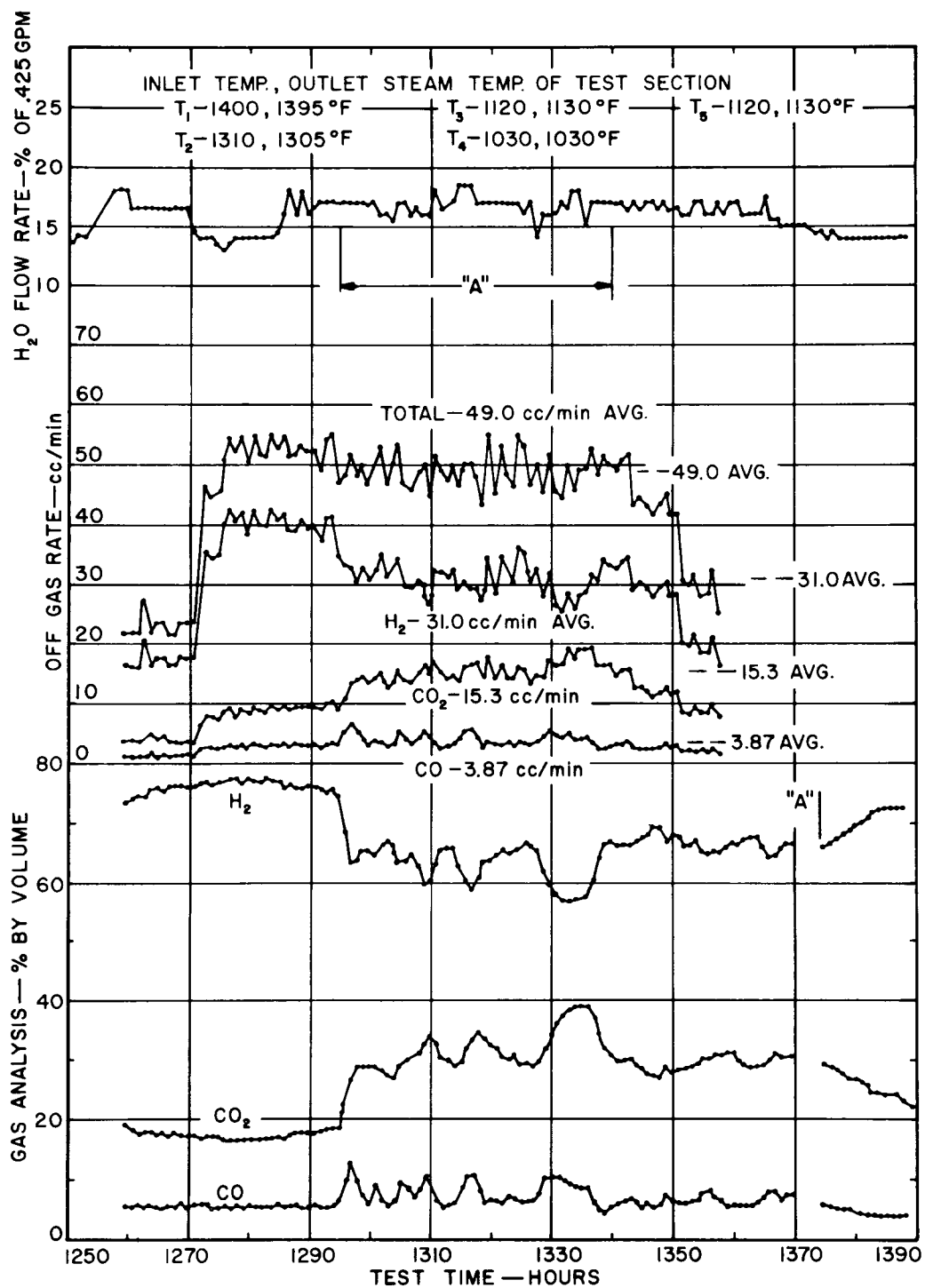


Fig. 15. Test at 140 psig and 1400°F Inlet Temperature EGCR Graphite Sleeve II.

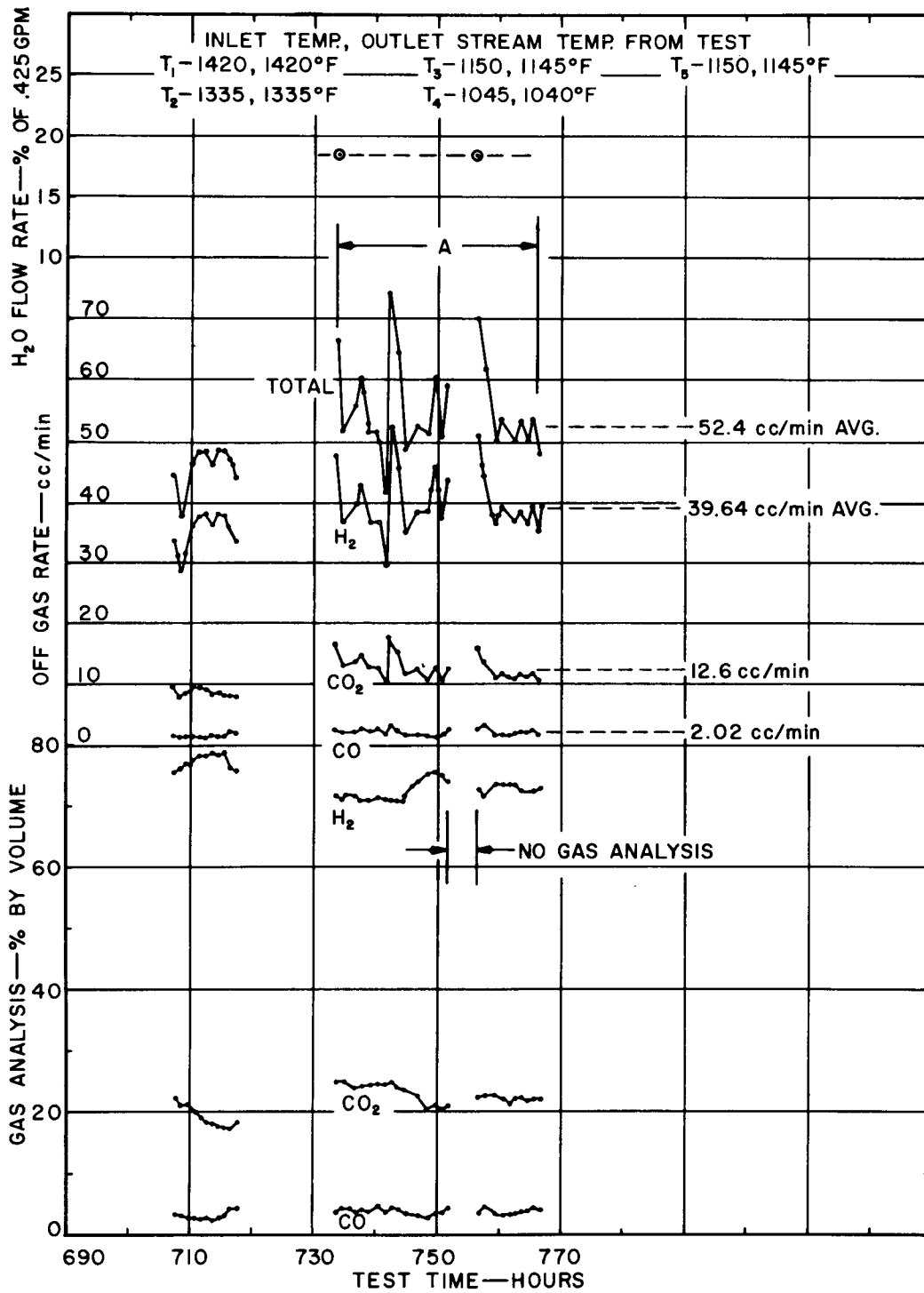


Fig. 16. Test at 200 psig and 1400°F Inlet Temperature EGCR Graphite Sleeve II.

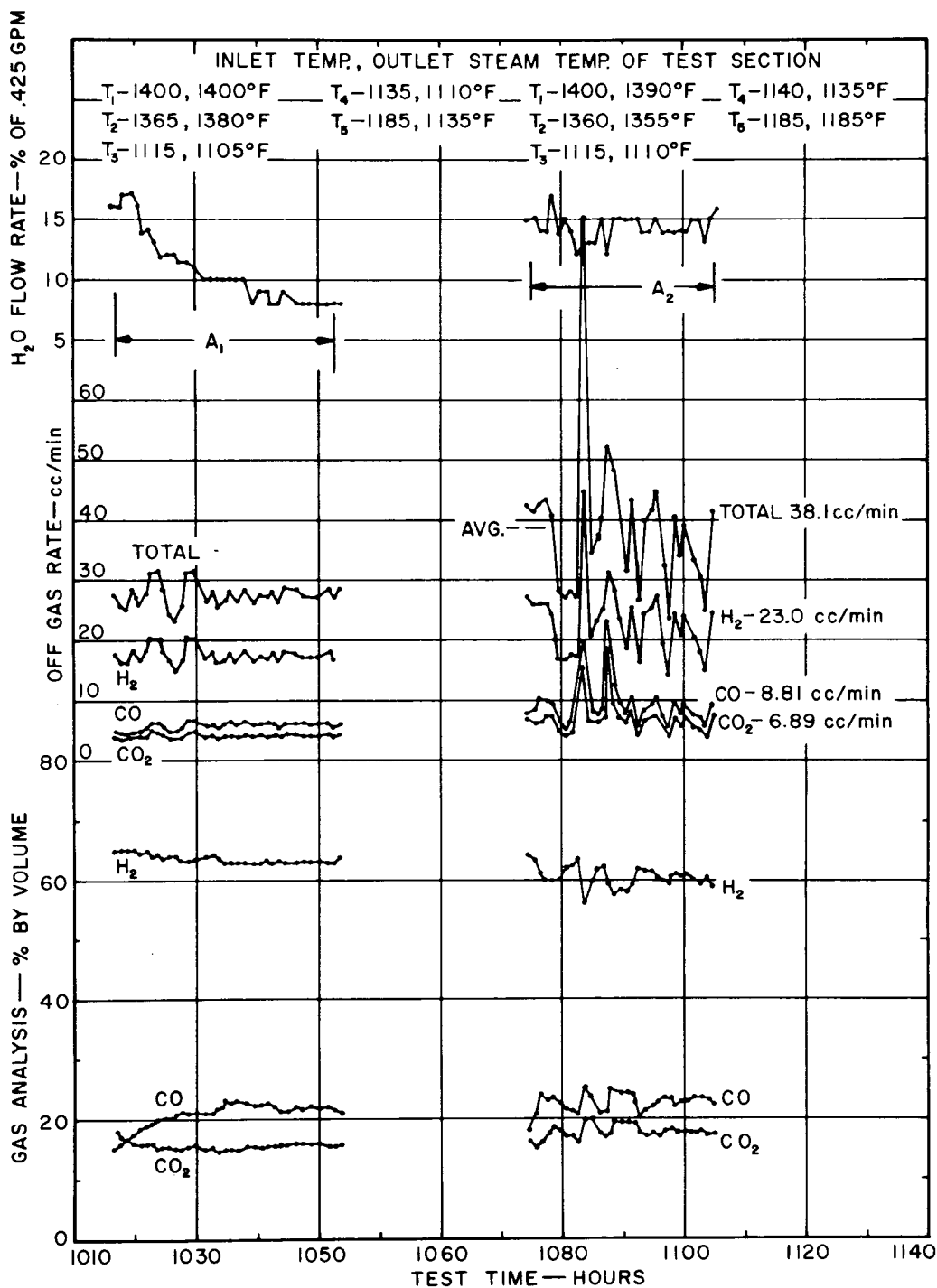


Fig. 17. Test at 30 psig and 1400°F Inlet Temperature EGCR Graphite Sleeve II.

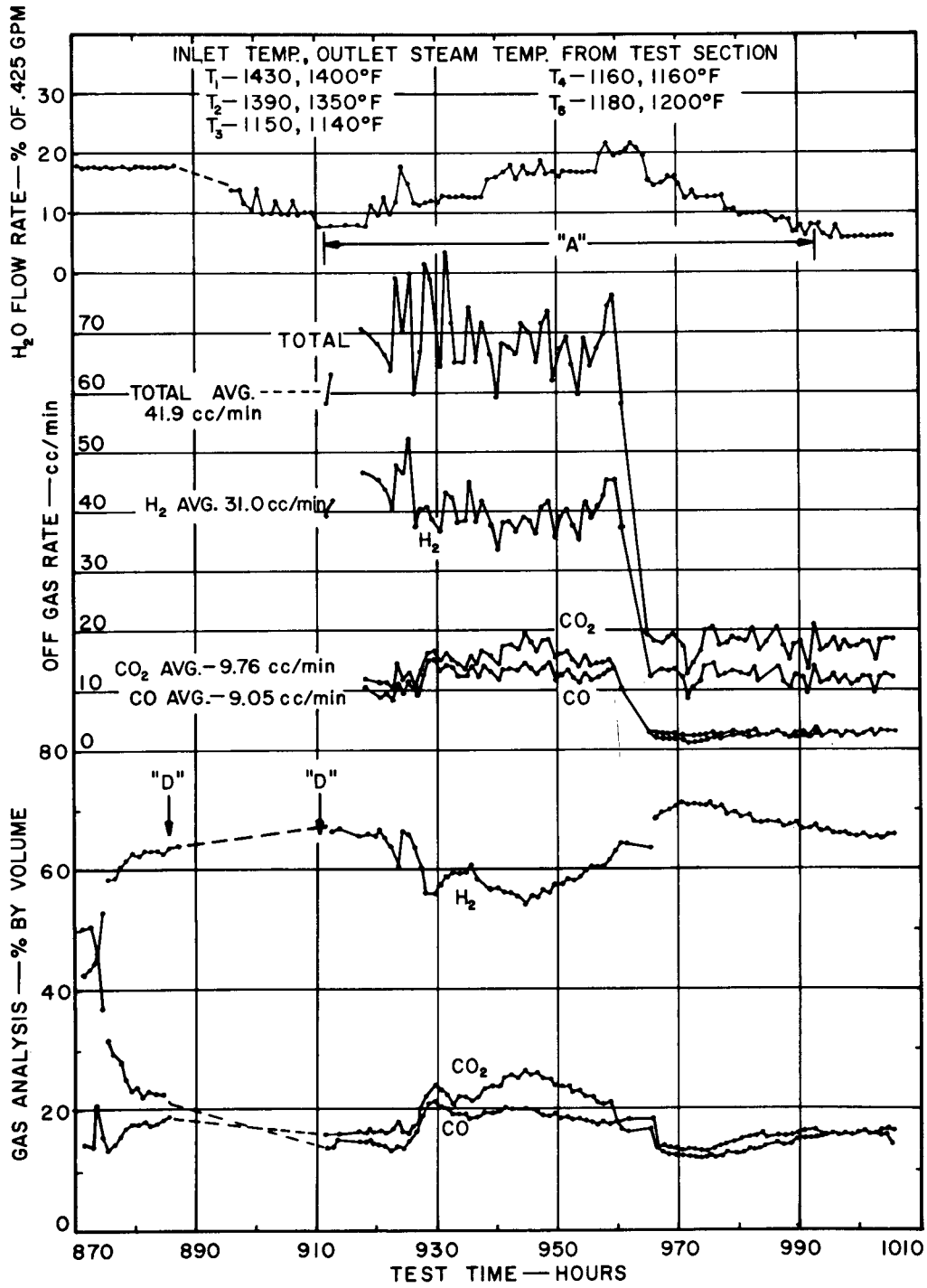


Fig. 18. Test at 50 psig and 1400°F Inlet Temperature EGCR Graphite Sleeve II.

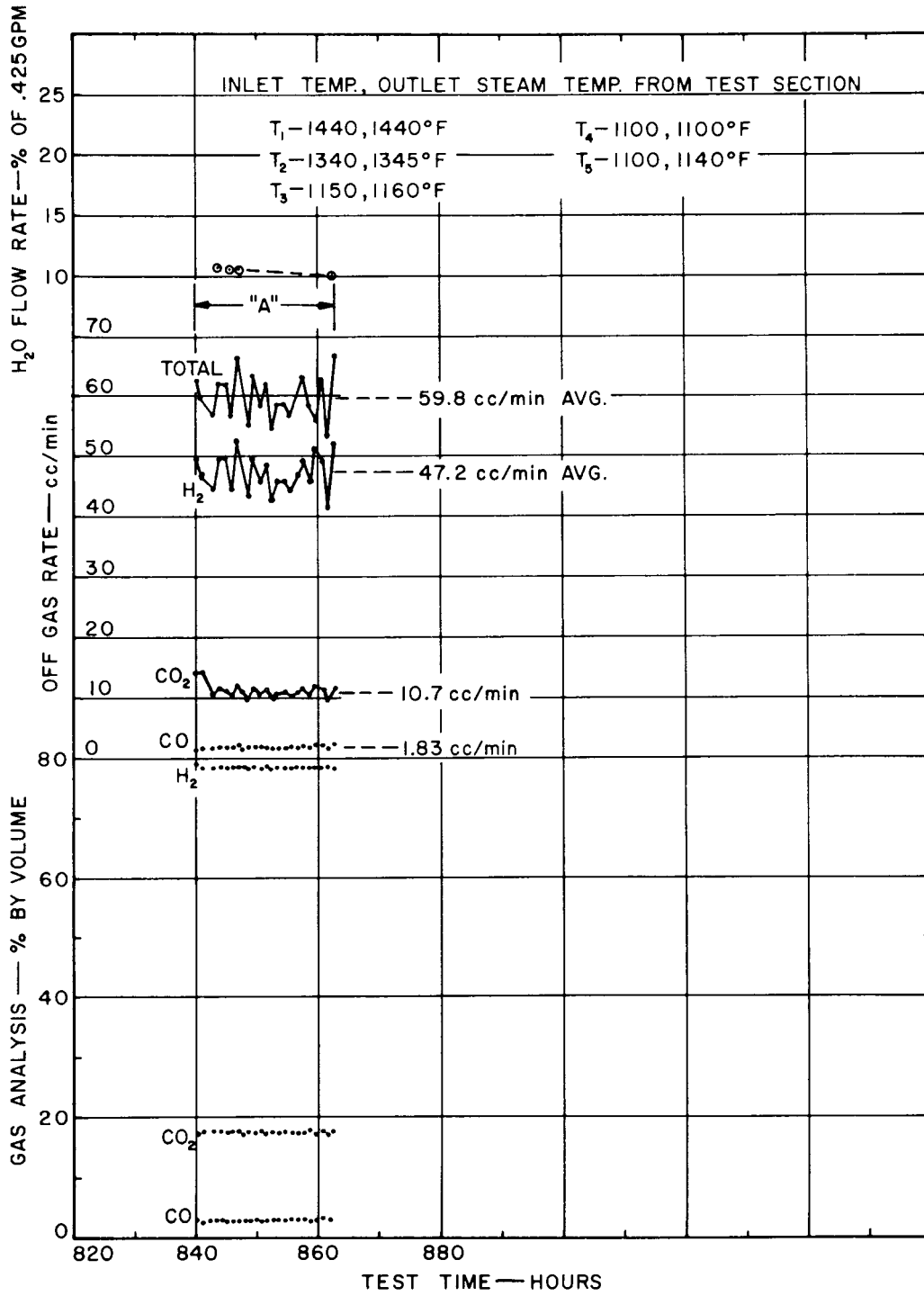


Fig. 19. Test at 300 psig and 1400°F Inlet Temperature EGCR Graphite Sleeve II.

R_{CO} = production rate of CO gas, scc/min

R_{CO_2} = production rate of CO_2 gas, scc/min

Using the geometric surface area, the steam-graphite reaction rate, in terms of the amount of carbon reacting per unit of time per unit of surface area, can be calculated using the following equation:

$$R_{CA} = \frac{R_C}{A_s}$$

R_{CA} = grams of carbon reacting per unit of time per unit of surface area, g/hr.ft²

R_C = grams of carbon reacting per unit of time, g/hr

A_s = internal area of the test specimen, ft²

Using Eq. 4, the carbon reacting per unit of time, R_C , for the test runs shown in Fig. 15 thru 19 were calculated. These values are shown in the following table:

<u>System Pressure</u>	<u>Carbon Removal Rate</u>
Psig	g/hr
30	.505
50	.605
150	.616
200	.470
300	.403

Since the axial temperature gradient existed in the test specimen (Fig. 20) and due to the fact that the thermocouples measuring inlet steam temperature may have been influenced by radiant heat from the superheater upstream, it is not possible to specify a temperature level which can be associated with the reaction rates tabulated. At best, these measurements can be taken as an indication of the effect of system pressure level on the steam-graphite reaction. Based on operating experience gained in these shakedown tests, guard heaters have been added around the test section to permit better temperature control, radiation baffles have been added between the superheater and the test section inlet thermocouple wells, and additional thermocouples have been installed in the graphite test piece itself. Additional tests are now underway and the results will be treated in subsequent reports.

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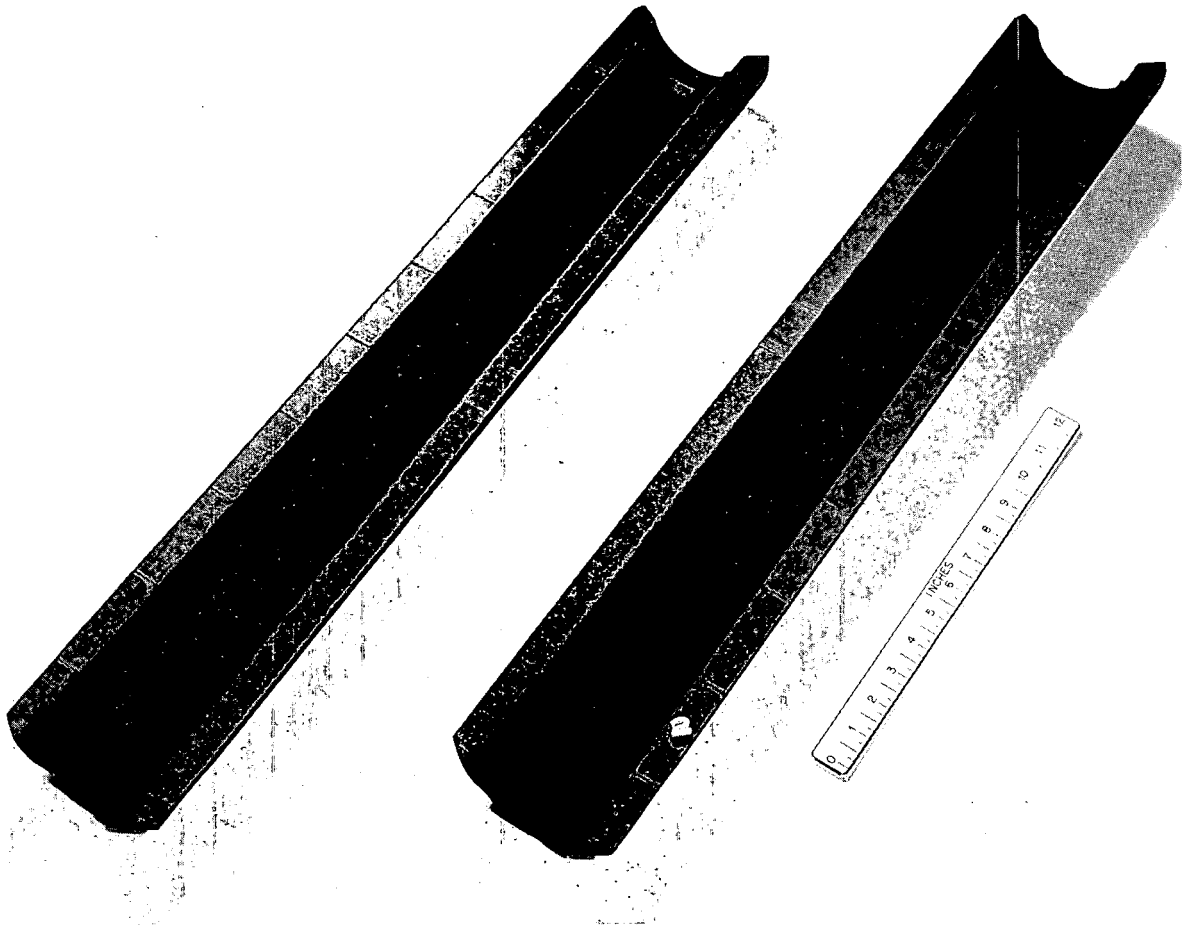


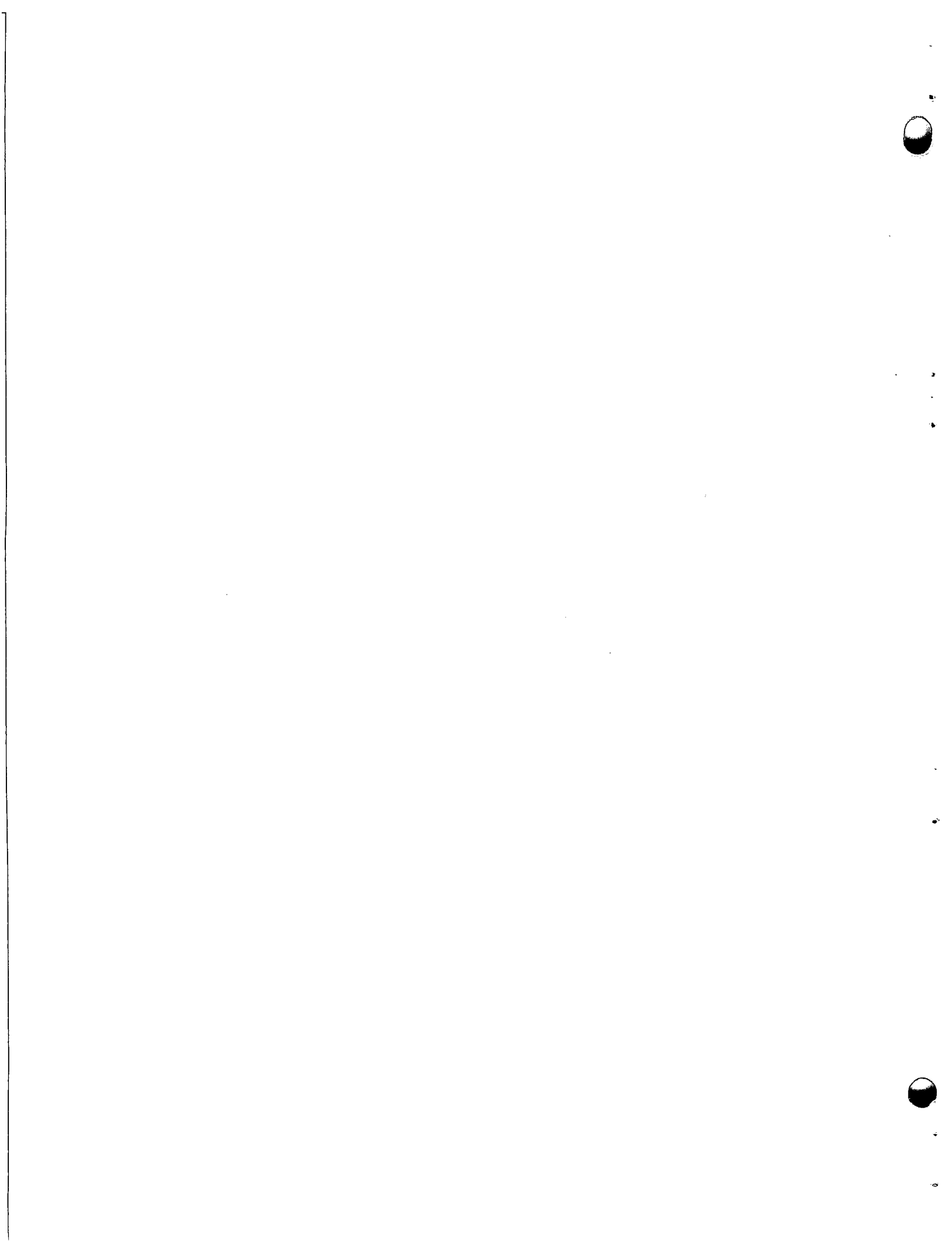
Fig. 20. EGCR Graphite Sleeve II - After Exposure to Superheated Steam for Approximately 455 Hours at 1400°F Inlet Temperature.

Nomenclature

- R_G = Noncondensable gas production rate, scc/min
- V_G = Volume of noncondensable gas at 14.7 psia and 70°F
- t = Total time of gas blow-down or total time to obtain a data point, min
- x = Concentration of each gas component, % by volume
- R_x = Production rate of each gas component (CO, CO₂, CH₄, H₂, O₂, N₂), scc/min
- R_C = Grams of carbon reacting per unit of time, grams/hr
- R_{CH_4} = Production rate of CH₄ gas, scc/min
- R_{CO} = Production rate of CO gas, scc/min
- R_{CO_2} = Production rate of CO₂ gas, scc/min
- R_{CA} = Grams of carbon reacting per unit of time per unit of surface area, grams/hr.ft²
- A_s = Internal area of the test specimen, ft²

Acknowledgments

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