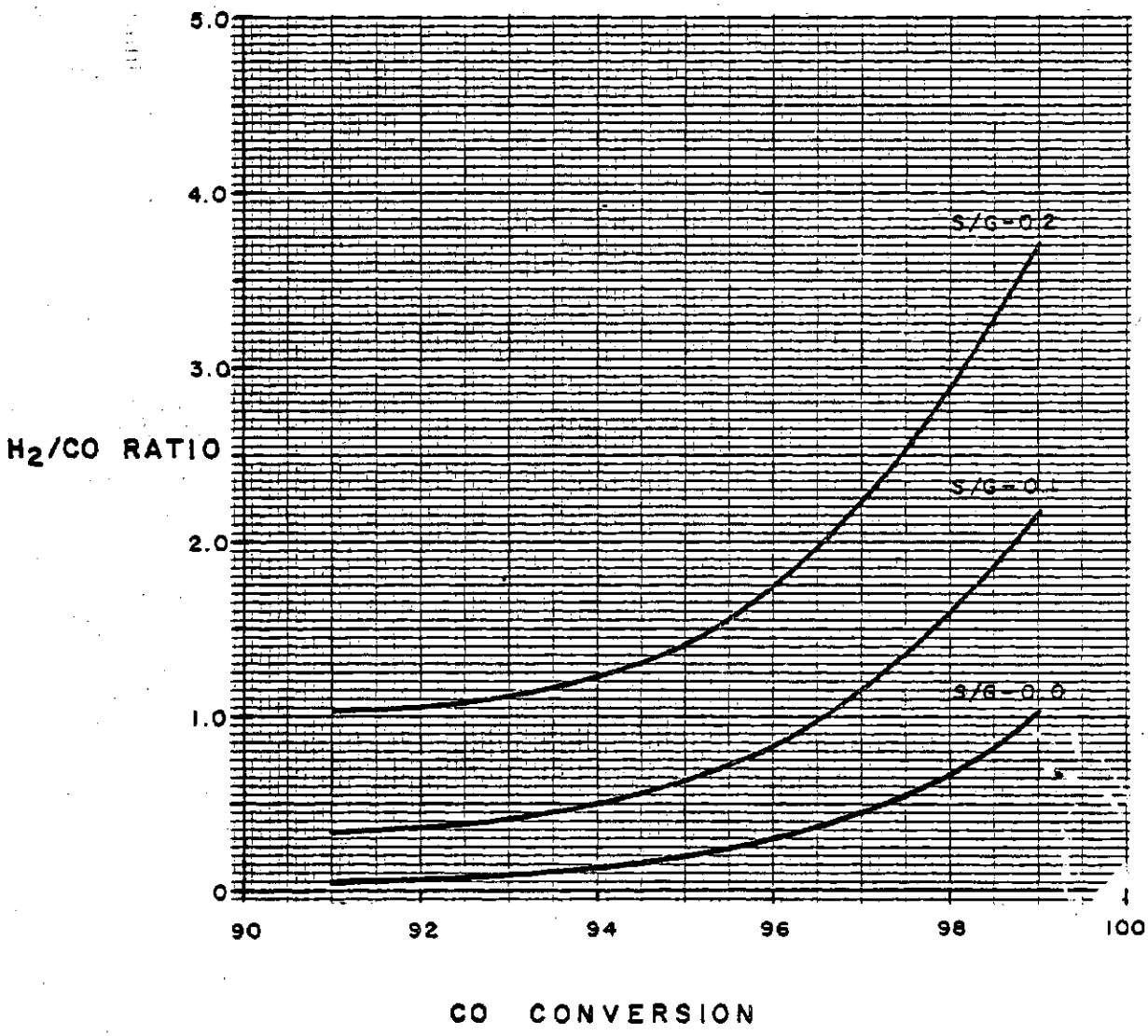
FIGURE IV-D-4b

EFFLUENT H_2/CO RATIO VS. CONVERSION

PROCESS DEVELOPMENT UNIT
CALSICAT Ni-230 REDUCED + STABILIZED
WITCO 40 MINERAL OIL
500 PSIG / 650°F
1.02 H_2/CO FEED GAS



completed. The recent PDU work corroborated the reaction model and system response as determined by the bench scale reactor program and established the basis for the future Pilot Plant studies. As required there will be continued effort to support the Pilot Plant program at the laboratory, concentrating on the following areas:

- Optimum H_2/CO levels for the LPM/S reactor.
- Optimum steam/dry gas ratios necessary to prevent carbon laydown in the polishing reactors.
- Evaluation of other promising catalyst candidates.

E. Pilot Plant

On July 15, 1975, a meeting was held at Industrial Contractors in Texas City, Texas. Present were representatives from Chem Systems, Institute of Gas Technology, Conoco, C.F. Braun, ERDA and Davy Powergas. Chem Systems reviewed the Liquid Phase Methanation process with respect to the pilot plant facility and how it would fit in with the existing gasification facilities at IGT and Conoco. Chem Systems recommended that IGT be the initial site for the pilot plant. The ERDA-AGA operating committee would review this. Comments by the participants were noted and Chem Systems reviewed the design to insure trouble-free operation at the proposed sites.

A subsequent meeting between Chem Systems and Davy Powergas was held in order to review the pilot plant status. Due to extended delivery dates for the seventeen (17) high temperature, high pressure, alloy valves and to a slacking off by Industrial Contractors' personnel, the completion date for the pilot plant had been extended to August 22, 1975.

On August 6th, Chem Systems personnel, accompanied by Davy Powergas personnel, visited Industrial Contractors to assess the status of their work. Even though a substantial amount of progress had been made, it was obvious that the plant would not be completed as promised.

The seventeen Walworth valves were due for shipment on August 11. Subsequently Walworth informed Davy Powergas that there would be a sixth delay in shipment since the first promised delivery date of April 7, 1975. A new delivery date of September 15th was promised after Chem Systems spoke to Walworth directly. This also required an extra charge of $1500 to cover special excess overtime costs to fabricate the valves. It must be noted that Davy Powergas could not exert any pressure on this vendor and this is indicative of their performance in failing to expedite other vendors as well.

At the August 6 meeting, modifications to the circulating oil pump seal system were finalized. These modifications were required due to a change in pump vendor from Union to Afton and Davy's failure to assess the implication of this change on the type of seals and instrumentation required.

By the end of August, Industrial Contractors had completed most of the piping work. Radiographic inspection of piping welds indicated poor welds in two trail lots. The weld defects were porosity, "cold spots" and penetration. Subsequently a third trail lot passed inspection. Chem Systems was concerned about this and started contacting outside testing companies to perform a completely independent inspection.

During September, Industrial Contractors completed the outstanding fabrication work on the LPM pilot plant. However, the plant has not been inspected or accepted by Chem Systems pending resolution of several outstanding items. The seventeen (17) Walworth valves did not arrive by the end of the month.

Davy Powergas' report on the radiographic inspection of the piping welds was received. After reviewing the report with ERDA/AGA, it was decided to hire an outside company to perform an independent inspection. This required removal of insulation from the piping.

During September 1975, Chem Systems met with C.F. Braun, at their request, to review the design basis and basic parameters to be used in Braun's evaluation of the LPM commercial concept. A design manual was prepared and given to Braun for their use in preparing the commercial concept design. The purpose of the manual was to organize the material necessary to carry out designs or evaluations for typical LPM plants and to present special design techniques, such as the LPM reactor design. Start-up and shut-down considerations were also provided in this manual.

In the fourth quarter, 1975, all seventeen (17) high temperature alloy valves were received from Walworth. The temporary spool pieces were removed, and the valves installed. Consolidated X-ray Service Corporation was retained to perform a 100 percent X-ray analysis of all the pilot plant high pressure butt welds. Arrangements were made with Davy Powergas and Industrial Contractors for the removal of piping insulation prior to the X-ray analysis.

Chem Systems' personnel visited the pilot plant fabricators shop, Industrial Contractors, during the week of December 8, 1975. Arrangements were made to remove approximately one-half of the pipe insulation so that a radiographic inspection of the pilot plant pipe welds (performed by Consolidated X-ray Service Corp.) could commence. While the insulation was being stripped, Chem Systems initiated a check-out of the control room and pilot plant piping, comparing the latest set of P & I's with what actually existed. Because of the presence of the insulation crew on the skid, only P & I drawings C-607-Y3 and C-607-Y4 were completely examined. Some 25 deficiencies and/or discrepancies were noted and reviewed with Davy Powergas personnel at a subsequent meeting. In addition, it was also noted that several change orders that were approved during the months of August and September had not yet been completed.

As of December 31, 1975, 217 pipe welds had been analyzed by Consolidated X-ray Service Corp. Of these welds, 30 percent were found to be defective (porosity, incomplete penetration, non-fusion, etc.) according to the ANSI-31.3 code.

The X-ray analysis was completed at the end of January, 1976. Out of a total of 423 pipe welds examined, 215 did not meet the code requirements for 100 percent radiographic inspection. Of these, 92 would meet the code requirements for random radiographic inspection. The radiographic films of the defective pipe welds were shown to representatives of Davy Powergas

and Industrial Contractors at meetings held at the Davy Powergas offices on January 30 and February 3.

Chem Systems' personnel met with the ERDA/AGA Operating Committee to update them on the overall situation, with specific attention focussing on the radiographic inspection of the welds. Subsequently, another meeting was held with representatives of Davy Powergas in order to develop a course of action to repair the defective welds and settle other outstanding matters.

Chem Systems' personnel visited the pilot plant fabricator's shop to complete an inspection of the pilot plant skid and control room. Since the previous December inspection, no corrective work had been accomplished. At a subsequent meeting, Davy Powergas representatives were given a complete check list of the deficiencies uncovered during both inspections.

During the second quarter 1976, negotiations were finalized with Davy Powergas and agreement reached for completing the work on the LPM pilot plant. The remainder of work to be performed was described as follows:

1. All girth butt welds on pressure piping on the LPM unit not meeting 100 percent radiographic standards as specified in ANSI B 31.3-1973 would be repaired to meet such standards. Thus, the repaired welds would be subject to 100 percent radiography to ensure that the repaired welds meet such standards. Whether a weld currently meets 100 percent or random radiographic standards would be based on radiography already performed and evaluated by Consolidated X-Ray Services.

2. Socket and other fillet welds on pressure piping would be tested ultrasonically using random sampling and measured against random radiograph standards. It was understood that if the random test failed, 100 percent of such welds would be examined and measured against random radiograph standards.

3. Upon completion of all weld repair and reinspection, the LPM unit would be hydrostatically tested in the presence of representatives selected by CSI.

4. Upon CSI acceptance of the hydrostatic test, the LPM unit would be reinsulated.

5. DPG would supervise the aforesaid work, including the approval and inspection thereof, and the disassembly of the completed LPM unit into its component skids preparatory to loading for shipment, and arrange for crating and transport.

In addition to the above, certain agreements were made concerning outstanding invoices, costs for additional work by both Davy Powergas and Industrial Contractors, and costs for certain parts of the repair work.

The agreement was submitted to ERDA and AGA requesting their approval. In the meantime, Davy Powergas proceeded to begin the weld repair work and document all material purchases for settlement of outstanding invoices.

In June, verbal approval was obtained from the ERDA/AGA Operating Committee on the agreement reached with Davy Powergas for completing work on the LPM pilot plant. Official approval was still pending from the ERDA Contracts Department.

In the meantime, following agreement between Chem Systems and Davy Powergas on several outstanding invoices regarding materials purchased and services performed under the original contract, Davy Powergas started repair work on the pilot plant.

A kick-off meeting was held on June 4th at Industrial Contractor's shop in Texas City, Texas. Present were representatives from Davy Powergas, ICI Corp., Chem Systems, and the X-ray technician from Consolidated X-Ray Corporation who had performed the 100 percent radiographic inspection. Weld repair procedures, including radiography of repairs were reviewed. The weld radiography films of the original piping which were taken by Consolidated X-Ray Service Corp. were loaned to Davy Powergas for use by ICI Corp. during weld repairs. Also, a set of marked prints locating welds and films taken were also loaned to Davy Powergas for ICI use. It was arranged that weld repair work would start on June 7th. Davy Powergas would return the films and marked-up prints to Chem Systems upon completion of the repair work. ICI would grind-out defective welds (i.e., no chipping) either partially or totally, depending upon the extent of the defect. DPG took full responsibility for final acceptance of the repair job, and Chem Systems was not required to inspect the rework or to provide approval of the rework.

The meeting also reviewed Davy Powergas committments to perform the following:

1. Monitor and supervise weld repairs.

2. Advise 10 days in advance, and then, formally notify Chem Systems 5 days in advance when hydrostatic testing would commence. Advance notifications were required so that Chem Systems could arrange visits by ERDA and AGA personnel to witness the test.

3. Supervise hydrostatic testing. The hydrostatic test would be carried out on the same basis as the previous test. Three (3) sets of marked-up flowsheets and DPG's hydrostatic test procedure would be transmitted to Chem Systems for distribution to ERDA and AGA.

4. Expedite re-insulation of piping after completion of hydrostatic test.

5. Disassemble skid into four component sections, plus the control room trailer, prior to shipment to demonstration site.

Davy advised that they would attempt to complete work within a six-week schedule previously promised at the time the May agreement was reached.

Chem Systems would make a final check-out of the unit after the weld repair and during the hydrostatic testing.

A second meeting was held on June 17th at Industrial Contractors with Davy Powergas' inspection supervisor. The main purpose of the meeting was to review progress in weld repair on the pilot plant. Of the 213 welds requiring repair, 82 had been re-welded. Forty of the repaired welds had been X-rayed and 32 of these had passed 100 percent radiographic standards while 8 had failed and required further re-work. All the welds processed thus far were on 2" pipe. The pipe sections were removed from the skids and the repair work carried out in the shop. No piping had yet been re-installed.

Results of the ultrasonic testing of socket welds were presented to Chem Systems. Seventy-five welds were tested by an outside company hired by Davy Powergas. All welds passed the test including the revised piping in the seal flush system. However, a few socket welds were rejected based on visual observation and repaired. In addition, the LPM skid was partially checked-out to ascertain that previous check-list items and change orders had been completed. Some eight items were determined to be still not completed, and Davy Powergas was notified to finish the work as soon as possible. A complete check-

out had to wait for all the weld repairs to be completed so that all equipment and piping were found in the proper places.

As of June 29th, the ICI Corp. shop progress had achieved the following:

- 151 welds out of 213 defective welds had been repaired.
- 101 welds have been radiographed and 73 weld repairs had been determined as being acceptable.
- 28 welds required a second round of repairs.

On July 14, Chem Systems visited Industrial Contractors to review progress on the weld repair program and also interview prospective vendors who could supply the following services; preparation of the component skids for shipping, rigging of the skids onto trucks, and shipment to the designated coal gasification site.

The weld repair program proceeded slowly during the month due to the following factors:

1) A one week closure of ICI's shop for annual vacation.

2) "Lost" days to perform weld repairs because of inclement weather. There were 20 work-days between June 7 (when the program started) and July 2. An estimated four (4) work-days were lost because of strong winds and/or rainfall.

3) Excessive rust within the piping which caused numerous repaired welds to fail the 100 percent radiography inspection standards. This rust probably resulted from the long project delay and failure of ICI Corp. to dry out the piping after the hydrostatic

test of September, 1975. An adequate cleaning and welding procedure was devised to overcome this problem.

4) The ICI shop welders' productivity was much lower than "anticipated" because of (a) difficulty of access to many sections of the skid piping, (b) inefficient positioning of welders and welding equipment because of lack of space and (c) inability to retain welder personnel because there is such an abundance of more desirable welding jobs in the Houston-Texas City area for union shop welders.

Nevertheless, by the end of July, all but five of the defective welds had been repaired and passed radiographic inspection. Sections of 2" pipe, which had been removed earlier for easier weld repair within the fabricator's shop were reinstalled on the pilot plant skid.

All weld repair and radiography to 100 percent x-ray standards was completed the first week in August. All railings and gratings were installed on the pilot plant skid and the unit prepared for hydrostatic testing. The hydrostatic test was performed on August 10 and witnessed by Chem Systems' representatives. The LPM skid assembly was partitioned into five sections, each of which was submitted to a hydrostatic test pressure of 150 percent greater than the normal operating pressure. Each section was held under test pressure for a period ranging from 1.5 to 2 hours. The five test sections performed well with only very minor leakage.

The small leakage and successful immediate testing was due to the fact that Davy Powergas required ICI Corp. to pre-test the LPM skid during the previous week and had then corrected the problems that arose.

At the end of the hydrostatic test, the ICI test team started drainage of the vessels and piping. Subsequently, on August 11 and 12, the team removed all piping blanks and reconnected the piping assembly.

Chem Systems inspected the LPM pilot plant on August 11. The "as-built" plant appeared to be fairly complete. There were a number of discrepancies and omissions which were noted and transmitted to Davy Powergas for immediate rectification. Davy Powergas agreed to up-date the P & I drawings to reflect the "as-built" conditions; correcting of omissions and additions of items due to revisions and change orders. The Engineering Data Books furnished by Davy Powergas were missing certain data and information. Davy Powergas agreed to transmit the missing pages as soon as possible.

Re-insulation of the piping was started on August 16 and completed by the end of the month. Paint retouching was also completed.

The construction phase of the Liquid Phase Methanation pilot plant was completed and a thorough inspection carried out by Chem Systems' personnel on September 9-10, 1976. The remaining electrical hook-ups were completed early in September. Except for minor details, all items checked-out satisfactorily. The completed skid assembly had an extremely good appearance. The insulation job appeared to be very good. The entire skid was carefully painted in standard gray color, and the control valves painted green.

The reactor circulating oil pump seal flush piping, upon inspection, were found to have screwed connections. Davy Powergas agreed to purchase new materials and ship them to the designated site for installation there. Davy Powergas also agreed to supply a new lift mechanism for the LPM reactor level detector instrument which could not function as installed. Since the mechanism material and assembly would not be ready before skid shipment, Davy Powergas agreed to ship the material to the demonstration site for field installation.

The control room building, pneumatic control systems, control valves, and electrical systems were not checked-out for operability because

(a) no electric power or air supply was available, (b) no inter-connections existed between the control room and the LPM skid and (c) the pump systems could not be checked without fluid in them.

Several vendors were interviewed during Chem Systems' July visit to Texas City. This was in respect to supplying services for preparing the pilot plant sections for shipment, rigging, truck shipment from Texas City to the designated demonstration site and chemical cleaning. The vendors were all recommended by Davy Powergas. Quotations for crating, rigging and truck shipment of the LPM pilot plant to one of the proposed demonstration sites were received by Chem Systems and subcontractors selected for this work.

Disassembly of the LPM Pilot Plant into its component modules was begun on September 15 in preparation for shipment to the first coal gasification demonstration site.

The disassembly, rigging, and start of crating were inspected by Chem Systems at the ICI Corporation, Texas City, Texas fabrication shop on September 21. The crater (First Class Export Crating, Inc.) was at the job-site on Friday, September 17 to make preparation for start of work on Monday, September 20. Severe area rainstorms on Monday prevented him from performing. Meaningful crating activities started on Tuesday, September 21.

C&H Transportation Company delivered five low-boy trailers, as previously arranged, to the job-site on Monday, September 20. The rigger (Westheimer Rigging & heavy Hauling Company - subcontractor to C&H transportation) arrived on-site about 10 a.m. with nine (9) riggers/crane men; one 50-ton hydraulic crane and one 40-ton hydraulic crane. Two men were assigned to each crane; four men were employed to handle rigging of skid sections; and a superintendent supervised the work.

The first section removed and loaded onto a trailer was the topmost superstructure skid section containing the LPM reactor and the reactor separator. Including the half-hour lunch period, it took nearly four hours from 10 a.m. to 1:45 p.m. to rig this section onto the trailer. Rigging work was completed at 11:30 a.m. (Texas City time) on Wednesday, September 22. By September 28, all rigging and crating work was completed.

ERDA and AGA designated Institute of Gas Technology's HYGAS Plant in Chicago, Illinois as the first demonstration site.

On September 29, C&H Transportation Company truckers removed the five trailers from the ICI Corporation yard and the pilot plant was enroute to Chicago.

This completed all work on the construction phase of the LPM pilot plant. Installation and operation is covered under another contract.

F. Process and Economic Evaluation Studies

1. Carbon Formation Studies

As part of the LPM/S commercial design concept, there is a natural concern whether the polishing reactor will be operated at conditions that could lead to excessive carbon formation. In order to put this question in its proper perspective, the LPM/S polishing reactor design conditions have been compared with other commercial and proposed final stage methanators (polishing reactors).

Table IV-F-1a summarizes this comparison showing feed compositions, temperature, pressures and gas hourly space velocities. The first column represents a typical final stage methanator in a naphtha-based SNG plant. The second column represents the proposed design for the Ralph M. Parsons' Process (RMP) final stage methanator.* The final two columns present the polishing reactor conditions that would result from two different feed gas compositions to the main LPM/S reactor. The gas compositions for these four cases are plotted on a C-H-O ternary diagram (Figure IV-F-1a). This figure also indicates the carbon formation isotherm for 500 psig and 600°F. Since the carbon formation equilibrium changes little with temperature and pressure in the range of interest, the isotherm can be assumed constant over the range 125-500 psia and 400-900°F. All four points lie outside the carbon forming region. The gas composition of the naphtha-SNG case is achieved by having a substantial quantity (7%) of steam contained in the synthesis gas. In contrast, the final methanator feed composition in the proposed RMP process is achieved by complete water removal and CO_2 removal to 4 percent by volume. This gas also lies outside the carbon forming region. The two gas feed compositions to the LPM/S polishing reactor represent the dried product from a $2H_2/1CO$ and a 1.4 $H_2/1CO/0.36$ H_2O

*168th Annual Meeting of the American Chemical Society, Division of Fuel Chemistry, Vol. 19, No. 3, pp. 57-69, 1974.

Table IV-F-1a

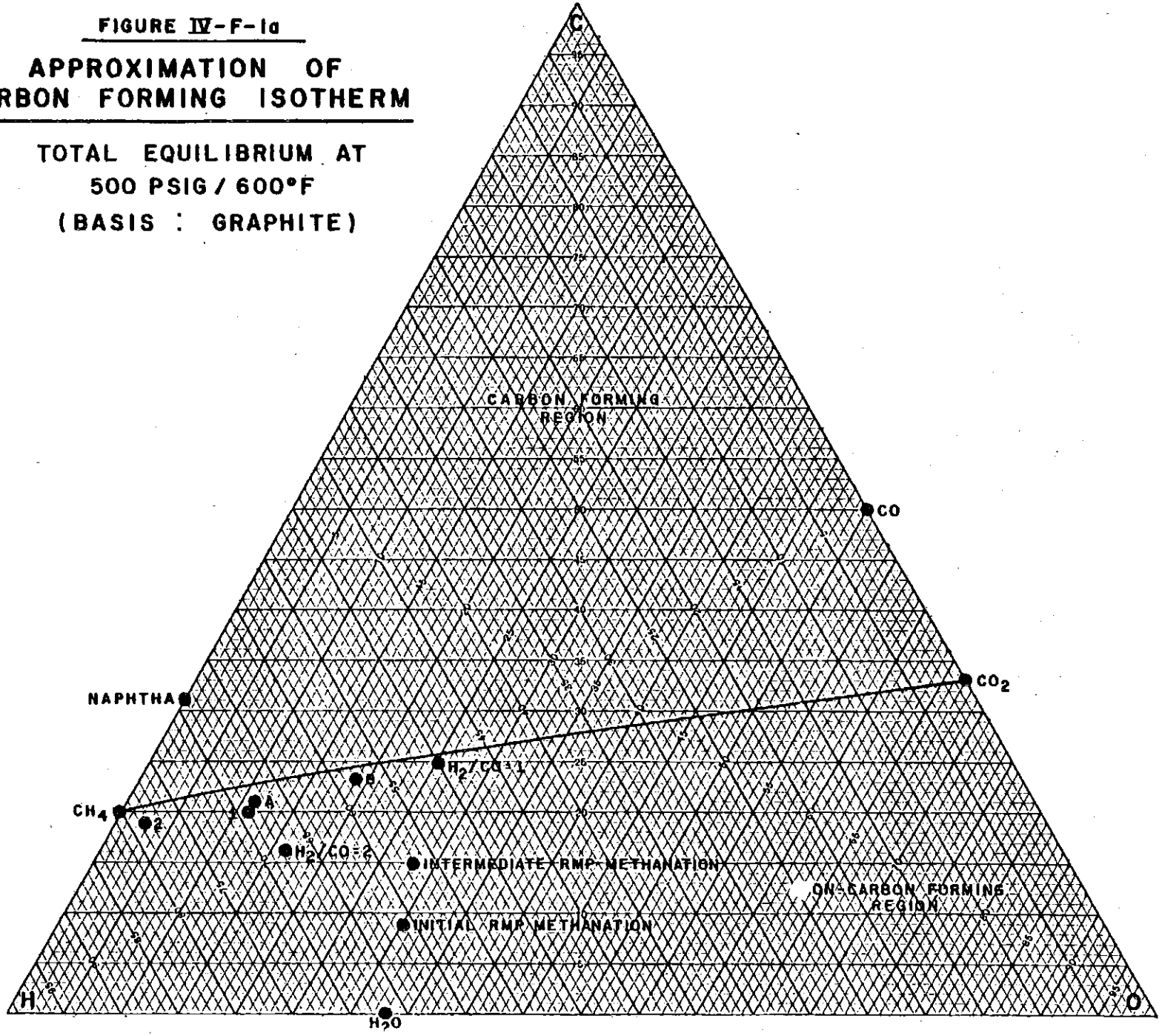
Comparison of Commercial and Proposed Final Methanators with the LPM/S Polishing Reactor

	Typical Final Methanation Step in a Naphtha-Based SNG Plant	Proposed RMP Process Clean-Up Methanation	LPM/S Initial Feed $2H_2/1CO$	LPM/S Initial Feed $1.4\ H_2/1CO/0.36\ H_2O$
Polishing Reactor Feed (Volume Percent)				
H_2	6.34	16.8	14.0	6.0
CH_4	66.79	77.6	63.0	55.0
CO_2	19.44	4.0	21.1	36.0
CO	0.49	1.6	1.9	3.0
H_2O	6.94	-	-	-
Temperatures (^oF)				
Inlet	575	550 (est.)	550	550
Outlet	677	850 (est.)	860	750
Average Pressure (psig)	507	300	500	500
Gas Space Velocity (hr^{-1})	9200	N.G.	11,300	9000
Point in Figure III-2	1	2	A	B

FIGURE IV-F-1a

APPROXIMATION OF CARBON FORMING ISOTHERM

TOTAL EQUILIBRIUM AT
500 PSIG / 600°F
(BASIS : GRAPHITE)



feed to the combined Liquid Phase Methanation/Shift Reactor. The two compositions are shown on the triangular diagram as points A and B. Both are outside the carbon forming region and only slightly closer to the carbon forming isotherm than the commercial methanator. One conclusion to draw from this analysis is that the LPM/S system would produce no more than the same amount of carbon found in the other systems. In fact, a 5 percent steam addition to the polishing reactor would place the composition at exactly the same distance from the carbon formation isotherm as is found in the commercial designs. This comparison should be valid since the reactors discussed above are all designed for similar operating conditions. In fact, the CRG-A catalyst which was tested is identical to that used in the final methanator of naphtha-based SNG plants. In practice, specific catalysts may respond differently to the Boudouard reaction driving force.* Thus, carbon formation must be determined by experiment.

2. Coal Gasification Product Survey

Existing and developing coal gasifiers produce synthesis gases with widely varying compositions. In order to evaluate the Liquid Phase Methanation/Shift process, it was necessary to first study typical synthesis gas compositions from various gasifiers and try to categorize them on a basis that would later facilitate evaluation of shift and methanation requirements. Representative gas analyses and other pertinent information for the Hygas process are listed in Table IV-F-2a. Similar information for other gasifiers utilizing Eastern and Western coals can be found in Tables IV-F-2b and c respectively. These lists are by no means exhaustive since many of the gasifiers produce synthesis gases which vary with coal utilized and with gasifier prototype. Also, there are many other gasifiers in various stages of development which are not listed on these tables. However, the gas compositions which are listed can be considered representative and typical of most medium or high-BTU gasifiers.

*F.W. Moeller Et Al, Hydrocarbon Processing, April, 1974, p. 69.

Table IV-F-2a

COAL GASIFIER PRODUCT SURVEY
HYGAS WITH EASTERN OR WESTERN COALS

	Electro-thermal	Steam-Oxygen	Steam-Iron
Clean Gas Analysis			
H_2 (Vol.%)	30.4	30.5	34.3
CO	26.5	24.1	11.4
CO_2	16.1	24.7	10.8
CH_4	24.6	18.9	39.9
C_2^+	1.3	0.7	1.5
Inerts	1.1	1.1	2.1
Coal-Basis	Any	Any	Any
Clean Gas Properties			
H_2/CO Ratio	1.15	1.26	3.01
H_2+CO (Vol.%)	56.9	54.6	45.7
HHV (BTU/SCF)	455	380	575
Pressure (psia)	1000	1000	1000
Separate Shift and Methanation			
Steam Required (Lbs/MM BTU SNG)	30.4	31.6	-
Combined Methanation/Shift			
Steam Required (Lbs/MM BTU SNG)	9.7	9.5	-
Acid Gas Removal			
CO_2 Removed (Lbs/MM BTU SNG)	80.3	120.7	23.1

Table IV-F-2b

COAL GASIFIER PRODUCT SURVEY
EASTERN COALS

	Lurgi	Synthane	Bi-Gas	Koppers-Totzek
Clean Gas Analysis				
H_2 (Vol.%)	38.8	27.8	32.4	35.6
CO	17.6	16.8	29.5	56.3
CO_2	32.4	29.1	21.7	7.4
CH_4	9.7	24.5	15.8	-
C_2^+	1.0	0.8	-	-
Inerts	0.5	1.0	0.6	0.7
Coal Basis	Ill.#6	Pittsburgh	Pittsburgh	Ill.#6
Clean Gas Properties				
H_2/CO Ratio	2.20	1.66	1.10	0.63
H_2+CO (Vol.%)	56.4	44.6	61.9	91.9
HHV (BTU/SCF)	300	405	360	295
Pressure (psia)	360	1000	1000	15
Separate Shift & Methanation				
Steam Required (Lbs/MM BTU SNG)	14.9	15.8	45.6	145.4
Combined Methanation/Shift				
Steam Required (Lbs/MM BTU SNG)	-	2.8	14.9	77.3
Acid Gas Removal				
CO_2 Removed (Lbs/MM BTU SNG)	162.9	108.9	132.6	205.7