

## CATALYTIC STEAM REFORMING OF LIGHT LIQUID HYDROCARBONS

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### Introduction

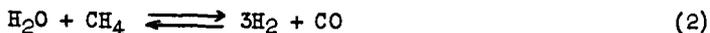
In the chemical process industry, catalytic steam-hydrocarbon reforming of natural gas or propane is widely employed to produce ammonia synthesis gas, high-purity hydrogen, and hydrogen-carbon monoxide mixtures for chemical synthesis. Currently, synthetic ammonia plants based on natural gas reforming account for 70-80 per cent of all ammonia produced in the United States, and a large percentage of domestic synthetic methanol production is derived from steam-hydrocarbon reforming.

The manufactured gas industry also employs steam-hydrocarbon reforming in the production of low-Btu carrier gas. Several continuous reforming units employing natural gas or propane feedstocks and steam-air oxidizing mixtures have been built in the United States. In a typical application, carrier gas and natural gas are blended to provide a mixture with the heating value, specific gravity, and hydrogen content required for satisfactory performance in customers' appliances. In Europe and Asia, carrier gas from steam-hydrocarbon reforming is commonly enriched with light hydrocarbons and diluted with inerts to about 0.5 specific gravity and 400-500 Btu higher heating value. The resulting mixture is used as a supplement or replacement for coke oven-carburetted water gas mixtures.

Steam-natural gas or steam-propane reforming techniques employed in producing ammonia synthesis gas, Fischer-Tropsch synthesis gas, hydrogen, and carrier gas have been described elsewhere /2,3,4,5,8/. In these and other applications of conventional fired-tube reforming furnaces, certain similarities are apparent. Feedstocks require careful desulfurization prior to conversion if temporary poisoning of nickel reforming catalyst and shutdowns for catalyst regeneration are to be avoided. Within the catalyst tubes, conversion of the original steam-hydrocarbon mixture occurs via highly endothermic chemical reactions yielding a product containing hydrogen, carbon monoxide, carbon dioxide, and small amounts of methane. When injection of air to the reformer is practiced, nitrogen becomes an additional component of the product. At the tube outlet, where gas temperatures of 1550°F may be obtained, product distribution is largely governed by equilibrium considerations. The water gas shift reaction



is found to be at equilibrium in reformer effluent, while the steam-methane reaction



attains a close approach to equilibrium.

Deposition of carbon on catalyst located near the tube inlet can be an operational problem in fired-tube reformers. Avoidance of carbon deposition is required to ensure long periods of continuous operation and prevent severe catalyst spalling. Freedom from carbon deposition is obtained by employing active catalysts

at high temperatures, and by using more than stoichiometric quantities of steam. Relative amounts of steam and hydrocarbon in the original mixture are conveniently specified by the steam-carbon ratio, moles steam per feedstock carbon atom. Selection of the optimum steam-carbon ratio is an important function of reformer design. Sufficient excess steam must be employed to prevent costly shutdowns and resulting lost production. However, a plant designed for an excessively high steam-carbon ratio also results in high manufacturing costs, since both investment and operating costs increase with increasing steam-carbon ratio.

Although continued growth of reforming capacity based on natural gas and propane feedstocks is predicted, an increased interest in utilizing heavier feedstocks has recently been indicated. In this paper, results of pilot plant and exploratory reforming tests on light liquid hydrocarbons are described and related to the known behavior of natural gas and propane. Particular consideration is given to the economically important steam-carbon ratio variable.

### Pilot Plant Experiments

#### A. Apparatus and Methods

A vertically mounted 1-inch reformer heated by a conventional electrical resistance furnace was used for screening of variables and determination of operability limits at 20-125 psig reforming pressure. The reactor consisted of a 57-3/4 inch length of 1-inch Inconel pipe. A catalyst support tray at the tube outlet and an axial thermowell extending the length of the reactor were provided. Attached to the upper or inlet end was a 2-inch welding-neck flange. The inner diameter of the flange provided surface for falling-film evaporation of water, and the annulus formed by the inner diameter of the flange and the outer diameter of the tube afforded volume for hydrocarbon vaporization and mixing of vaporized water and hydrocarbon. A preheater assembly, 28 inches of 316 stainless steel bar stock welded to a 2-inch blind flange, fitted snugly into the reactor, with 0.003 inches radial clearance. The flange and bar stock were suitably tapped for inlet connections and the axial thermowell, and the bar stock was threaded to provide heat transfer area. A stainless steel ring closure was employed. Catalyst bed depth was 30 inches.

In operation, water and hydrocarbon were pressured from calibrated feed tanks with nitrogen. Flow rates were controlled by small air-operated valves responding to pressure drops across hypodermic needle orifices. Water and hydrocarbon metered to the reactor inlet were vaporized in the falling-film evaporator section of the reactor, preheated by downward passage through the preheater spiral, and contacted with catalyst. Furnace windings opposite the vaporization, preheat, and reaction sections of the reactor were automatically controlled. Reaction products were cooled by indirect exchange, and unreacted water separated. Product gases flowed through a back-pressure regulator maintaining unit pressure to a wet-test meter, sampling manifold, and vent. Periodic samples were analyzed by mass spectrometer. Pressure drop across the catalyst bed was continuously measured by a differential pressure cell with a range of 0-100 inches of water.

In all experimental studies made in the 1-inch unit, 1/8 inch extrusions of commercial steam-hydrocarbon reforming catalyst were utilized to maintain a reasonable relationship between reactor diameter and catalyst size. The particular catalyst employed was a pre-shrunk preparation which contains 20-25 per cent by weight nickel (as NiO) uniformly distributed throughout each pellet. Fresh catalyst, containing 0.49 per cent by weight sulfur, was either pretreated extensively at high temperature with a steam-hydrogen mixture, or used in extended reforming runs at high steam-carbon ratios, until the catalyst sulfur content was reduced to 0.003-0.005 per cent by weight. Extensive pretreatment was mandatory, since unreliable results were obtained with catalysts containing as little as 0.01 per cent by weight sulfur.

Feedstocks employed were of 99+ mole per cent purity, containing a maximum of 5 parts per million sulfur by weight. Catalysts discharged after extended contact with feedstocks containing 5 parts per million sulfur uniformly analyzed 0.003-0.005 per cent by weight sulfur, indicating that the sulfur content of the catalyst was substantially constant during test periods.

After catalyst pretreatment, feedstocks were reformed in a series of 12 or 24 hour experiments conducted at progressively lower steam-carbon ratios. Testing was continued until an increase in reactor pressure drop was indicated by the differential pressure cell. A measurable increase in pressure drop was taken as evidence of carbon formation in the catalyst bed at the particular steam-carbon ratio employed.

### B. Experimental Results

For orientation, the reforming characteristics of 99.7 mol per cent cylinder propane were initially investigated, employing the technique described above. Typical experimental data obtained at 125 psig reforming pressure and substantially commercial temperatures and hydrocarbon space velocities are presented in Table 1. Tests made at steam-carbon ratios ranging from 3.77 to 1.51 were completed without evidence of carbon deposition. At 1.35 steam-carbon ratio, catalyst activity gradually declined during 24 hours of operation, and reactor pressure drop increased about 0.4 inches per hour. This behavior was interpreted as arising from deposition of carbon on the catalyst. Additional data from tests made without nitrogen diluent and a correlation relating rate of pressure drop increase to steam deficiency indicate that 1.50 moles of steam per carbon atom are required for operability at 125 psig when reforming propane.

Wet product gas analyses permit calculation of apparent gas temperatures at the tube outlet, assuming that the water gas shift and steam-methane reactions are at equilibrium in the product. Results of these calculations are presented in Table 1. The assumption of equilibrium for either reaction gives outlet temperatures which are generally in reasonable agreement with the measured outlet temperature. In larger pilot plants, the steam-methane reaction is usually further removed from equilibrium. The close approach of the steam-methane reaction to equilibrium at the outlet of the 1-inch unit reflects the high activity of 1/8-inch catalyst pellets relative to commercial-sized pellets when both are employed at the same hydrocarbon space velocity.

A similar operability study was made on ASTM grade n-heptane, a representative light liquid hydrocarbon reforming feedstock. Results of these tests are also presented in Table 1. At 20 psig reforming pressure and commercial temperatures and hydrocarbon space velocities, reactor pressure drop did not increase during test periods at 3.03 steam-carbon ratio and higher. At 2.53 steam-carbon ratio, reactor pressure drop increased 0.4 inches per hour during 12 hours of steady operation. Additional tests confirm that, at 20 psig, about 3.0 moles of steam per carbon atom are required to prevent carbon deposition when reforming n-heptane.

Product gas ratios and calculated outlet temperatures obtained when reforming n-heptane closely resemble results obtained using propane feedstock. Except that higher steam-carbon ratios are required for operability, the reforming characteristics of n-heptane appeared to be fundamentally similar to those of propane.

### C. Effect of Hydrocarbon Molecular Weight on Operability

Operability limits determined for propane and n-heptane in this study can be compared with operability data from the literature /7/ to provide an estimate of the relationship between hydrocarbon molecular weight and minimum operable steam-carbon ratio at several reforming pressures. In Figure 1, published data obtained

in a 5-inch tube at essentially atmospheric pressure are compared with data from this study. A single relationship appears to represent both sets of data adequately. Required steam-carbon ratio is shown to increase logarithmically with increasing molecular weight at several pressures. The rate of increase is moderate, operability limits ranging from 1.1 steam-carbon ratio for natural gas or methane to 3.0 steam-carbon ratio for n-heptane.

While agreement between the two sets of data may be partly fortuitous, owing to differences in feedstock purity, catalysts, and operating conditions, additional studies in the 1-inch pilot plant tend to confirm the relationship shown in Figure 1. When reforming olefin-free and sulfur-free hydrocarbons, reforming pressures ranging from 20 to 125 psig are found to have no significant effect on minimum steam-carbon ratio.

### Study of Reforming Variables in Glassware

#### A. Apparatus and Methods

Since pilot plant tests indicated no major effect of pressure on minimum steam-carbon ratios when reforming olefin-free and sulfur-free hydrocarbons, the study of light liquid hydrocarbon reforming was continued in glassware at atmospheric pressure to further clarify the influence of reactor and feedstock variables on operability. Reactions of steam-hydrocarbon mixtures were studied over various commercial reforming catalysts at several temperatures and steam-carbon ratios in a conventional 1-inch diameter quartz reactor heated by an electrical resistance furnace. A 2-inch bed of catalyst was employed so that, in effect, the top portion of a full catalyst charge in the 1-inch pilot plant was being studied, this being the critical zone for carbon formation.

In operation, hydrocarbon feedstock was introduced from a pressured container and vaporized. Flow rate to the glass unit was measured by calibrated rotameters. Steam-carbon ratio was controlled by gas saturation. A metered stream of vaporized feedstock, saturated to the desired steam-carbon ratio, was preheated by rapid passage through an annulus formed by the inner tube wall and snugly fitted quartz plug, and contacted with catalyst. Reactor effluent was rapidly removed from the reaction zone through 3-millimeter tubing, cooled to condense unreacted steam, measured in a wet-test meter, and vented. Periodic product gas samples were withdrawn and analyzed by mass spectrometer. Feedstock conversion was varied by altering hydrocarbon space velocity at constant steam-carbon ratio and temperature. At the completion of each test, the catalyst charge was analyzed for carbon by combustion and absorption of carbon dioxide. Previously cited limitations on catalyst and feedstock sulfur contents were observed. While the apparatus and technique employed are not ideally suited for kinetic measurements, they permit very efficient screening of variables for large effects on operability and product distribution.

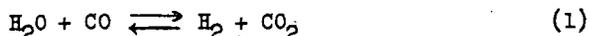
Before any reforming experiments were made, the catalyst space was filled with quartz chips, and steam-hydrocarbon mixtures passed through the unit at various combinations of temperature and contact time. In this way, temperature-contact time combinations sufficient to cause thermal cracking were determined for all feedstocks. Subsequent reforming tests were then carried out at conditions where precracking of feedstock could not occur, thus ensuring that catalytic reactions alone were being investigated.

In the tests to be described, two commercial catalysts were employed. Catalyst A is an impregnated type, containing about 5 per cent nickel (as NiO) on alumina. Catalyst B is the compounded preparation previously used for studies in the 1-inch pilot plant.

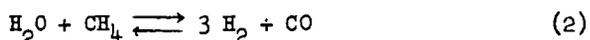
## B. Experimental Results

Typical data obtained when reforming 99.7 mole per cent n-butane to partial conversion at 1075°F or 1275°F and 3.0 steam-carbon ratio are presented in Table 2. At all conversion levels, dry product gases are observed to consist almost entirely of conventional reformed products and unreacted feedstock. Various olefinic and paraffinic butane decomposition products are present at low concentrations in all product gas samples. Methane, a final product in commercial reforming operations, is not detected in gases produced at low conversions of feedstock. These characteristic features of partially reformed gases are confirmed by other studies employing different temperatures, steam-carbon ratios, and catalysts.

Product ratios corresponding to equilibrium constants for the water gas shift reaction



and steam-methane reaction

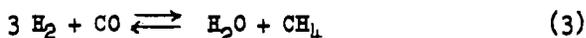


have been calculated and are reported in Table 2. At 1275°F, the water gas shift equilibrium constant is approximately 1.61. Shift constants calculated from experimental data obtained with compounded Catalyst B show a strong dependency on the extent of n-butane conversion. At conversions below 10 per cent, the shift reaction is far removed from equilibrium. However, in the range of 10-30 per cent n-butane conversion, equilibrium is rapidly approached, while at still higher conversions, equilibrium constants appear to scatter about the equilibrium value. At 1075°F, where the shift equilibrium constant is approximately 2.85, a parallel trend is evident from data obtained using Catalyst B. Data obtained with impregnated Catalyst A at 1275°F, indicate a much slower approach to equilibrium than with Catalyst B. However, the reproducibility of this apparent difference in catalyst activity for the shift reaction has not been determined. In general, all data in Table 2 indicate that the water gas shift reaction rapidly proceeds towards equilibrium in the presence of nickel reforming catalysts. Similar conclusions are made in a published study on natural gas reforming /6/.

The equilibrium constant for the steam-methane reaction at 1275°F is approximately 9.5. Calculated steam-methane equilibrium constants in Table 2 indicate that the approach to equilibrium over both Catalysts A and B, is very slow for this reaction, even at high n-butane conversions. This result is in agreement with commercial steam-hydrocarbon reforming results, where equilibrium is approached but not attained at much higher contact times than were employed in this study.

Methane production is further analyzed in Table 2 as a function of n-butane conversion at 1075°F or 1275°F and 3.0 steam-carbon ratio. In Figure 2, tabulated mole ratios of methane produced to n-butane converted are plotted against conversion and extrapolated towards zero conversion to determine if methane is a primary reaction product. As Figure 2 indicates, methane is produced only when the conversion of n-butane exceeds about 15 per cent. The relationship obtained appears to be essentially independent of catalyst type or temperature. Thus, methane is not a primary product of steam-butane reforming, and does not arise directly from catalytic decomposition of feedstock.

Consideration of other possible reactions yielding methane indicates that methane is probably formed by the methanation reaction



which is catalyzed by nickel at conditions similar to those employed in reforming. Production of methane is limited by the steam-methane back-reaction, since methane molecules can disappear by successfully competing with unreacted n-butane for active reforming sites on the catalyst.

Analyses of discharged catalysts indicate that, at steady state conditions, detectable amounts of carbon are always present on active catalysts. In general, carbon contents are higher at the lower reforming temperatures. Compounded Catalyst B contains less carbon than impregnated Catalyst A after comparable periods of exposure to steam hydrocarbon mixtures. The temperature dependency of catalyst carbon content is illustrated specifically in Table 3, where carbon levels are compared for runs made with Catalyst B at nominally constant conversion, constant steam-carbon ratio, and 900°F, 1100°F and 1300°F. Although absolute values for carbon content may be specific for the apparatus and conditions employed, these analyses indicate that carbon content decreases almost exponentially with increasing temperature.

All data from tests on n-butane have been evaluated to determine the apparent kinetics of n-butane disappearance. While the data are not sufficiently precise for a detailed kinetic analysis, the rate of butane disappearance appears to be approximately proportional to n-butane concentration for the temperatures, steam-carbon ratios, and catalysts studied. The apparent first order reaction rate constant increases exponentially with temperature. First order kinetics have also been shown to apply in natural gas reforming /1/. A comparison of rate constants for butane disappearance by steam-hydrocarbon reforming and by thermal cracking indicates that the reforming reaction is roughly 100 times faster at the same temperature, thus confirming that thermal cracking is not a major reaction in reforming.

#### Discussion

Information developed from partial conversion and pilot plant studies permits some reasonable speculation on the individual chemical reactions occurring in steam-hydrocarbon reforming. The overall conversion of paraffin hydrocarbons and steam to reformed products is indicated as occurring through a complex series of consecutive and competing reactions. Hydrocarbon molecules diffuse to the catalyst surface, are adsorbed, and undergo catalytic cracking-dehydrogenation reactions which yield strongly absorbed olefinic fragments. Further dehydrogenation-polymerization reactions occur, leading to formation of coke or carbon on the catalyst. The sum of these consecutive steps is an overall reaction in which feedstock molecules are converted to carbon on the catalyst at a rate proportional to feedstock concentration.

Carbon deposits are continuously removed from the catalyst by a competing reaction involving steam. Adsorbed steam molecules react with deposited carbon to form hydrogen and carbon monoxide or carbon dioxide. These products, hydrogen evolved in the cracking-dehydrogenation-polymerization step, and steam also participate in methanation and water gas shift side reactions. In addition, a portion of the methane produced by methanation is continuously reformed through a sequence similar to that followed by the original feedstock.

This qualitative mechanism is helpful in rationalizing the behavior of reforming units, since it predicts that net carbon formation and inoperability result when the rate of feedstock decomposition exceeds the rate of carbon removal by the steam-carbon ratio. Operable reforming units are thus characterized by equal rates of carbon formation and removal at all points in the catalyst bed. Any change in conditions which induces a relative increase in the rate of carbon removal from the catalyst will accordingly favor operability. Major operating variables capable of altering this relative rate are temperature and steam-carbon ratio. An

increase in catalyst bed temperature will, in general, favor the attainment of operability. Although the rates of reactions producing and removing carbon both appear to increase exponentially with temperature, the steam-carbon reaction rate apparently increases at a relatively greater rate, as demonstrated by catalyst carbon analyses. With all other reactor variables held constant, an increase in catalyst temperature will therefore increase the relative rate at which carbon is removed from catalyst at all points within the bed, or lower the equilibrium carbon content of catalyst.

An increase in steam-carbon ratio will also favor the attainment of operability by reducing the rate of hydrocarbon decomposition. When reforming n-butane, for example, an increase in steam-carbon ratio from 2.0 to 3.0 reduces butane concentration in the feedstock from 0.111 to 0.0796 mol fraction, a decrease of about 31 per cent. Thus the initial rate of butane decomposition to carbon near the tube inlet is decreased about 31 per cent. The corresponding slight increase in steam concentration, from 0.889 to 0.922 mol fraction, does not appear to greatly influence the rate of carbon-removing reactions. Small increases in steam concentration can therefore strongly influence the relative rates of carbon laydown and removal on catalyst located near the bed inlet.

These observations are consistent with operating experience in pilot plants and commercial units. Carbon deposition generally occurs near the tube inlet where catalyst temperatures are low; in this critical zone, the steam-carbon reaction proceeds slowly relative to decomposition. The effectiveness of increased steam-carbon ratio in suppressing carbon formation in the critical zone is well known.

The relationship between paraffin hydrocarbon molecular weight and minimum steam-carbon ratio presented in Figure 1 may also be a consequence of changes in the relative rates of competing reactions producing and removing carbon. Experience suggests that, at the same temperature and steam-carbon ratio, n-butane is catalytically decomposed to carbon more rapidly than methane. If the rate of reaction between steam and carbon is similar in both cases, net carbon formation or inoperability will be favored for the heavier feedstock. To attain an equal degree of operability, additional steam will be required to reduce the rate of n-butane decomposition to carbon. Hence n-butane will require a higher minimum steam-carbon ratio than methane to balance carbon production and removal. The relationship shown in Figure 1 may therefore arise from a regular increase in the rate of catalytic decomposition to carbon with increasing feedstock molecular weight.

### Conclusions

Pilot plant tests demonstrate that presently available steam-hydrocarbon reforming catalysts can be used successfully with light liquid hydrocarbon feedstocks. The similarity of behavior and results noted in tests with propane and n-heptane indicate that light liquid hydrocarbons and currently reformed feedstocks follow a similar reaction path. Partial conversion experiments suggest a mechanism involving simultaneous catalytic conversion of feedstock to carbon and catalytic removal of carbon by the steam-carbon reaction. Water gas shift, methanation, and steam-methane reforming side reactions are additionally indicated as occurring on the catalyst. The qualitative mechanism presented appears to rationalize some observed effects of reforming temperature and steam-carbon ratio on operability.

Table 3  
EFFECT OF REFORMING TEMPERATURE ON CARBON  
CONTENTS OF DISCHARGED CATALYSTS

Temperature, °F.	900	1100	1300
Carbon on Catalyst, Wt. %	1.34	0.46	0.12

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Table 1

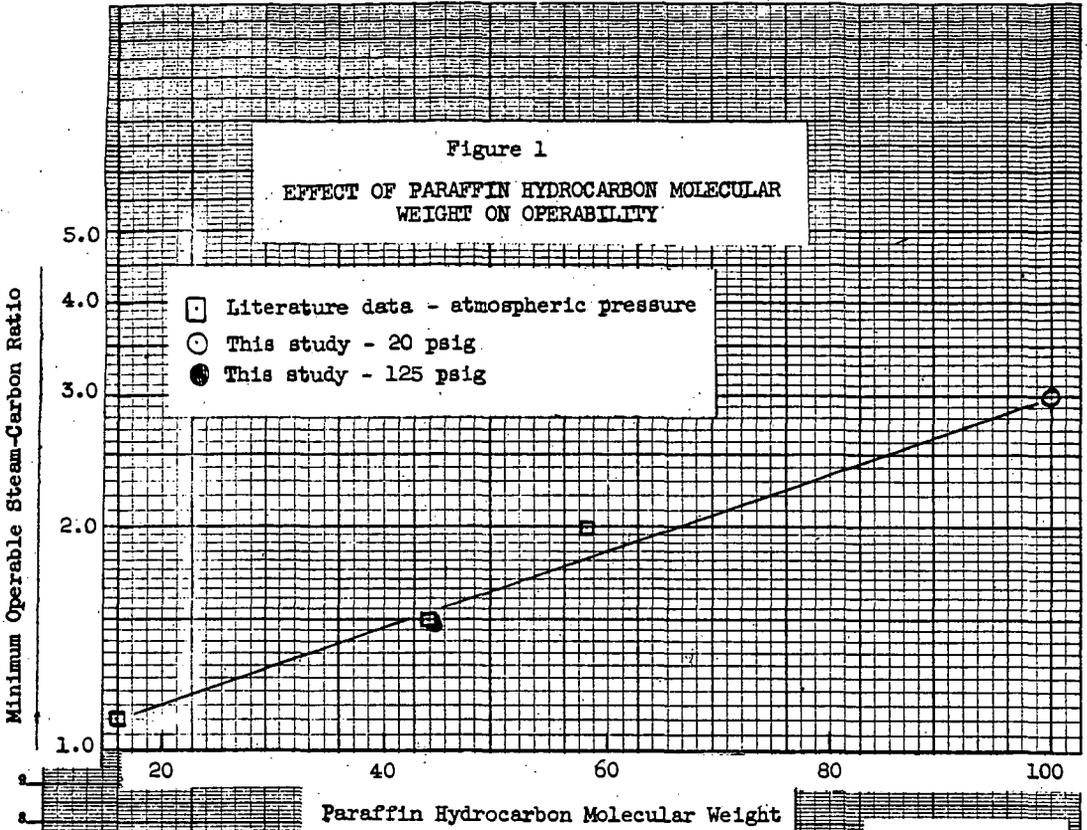
RESULTS OF PILOT PLANT REFORMING TESTS ON PROPANE AND HEPTANE

Feedstock	C <sub>3</sub> H <sub>8</sub>				n-C <sub>7</sub> H <sub>16</sub>							
	20	24	24	24	24	24	12	22-1/4	12	12		
Test Duration, hr	3.77	2.85	2.28	1.83	1.51	1.36	8.35	6.41	3.98	3.89	3.03	2.53
Steam-Carbon Ratio, moles steam per carbon atom	710	640	670	650	685	650	720	730	810	805	795	805
Reactor Inlet Temperature, °F	1450	1430	1440	1445	1440	1455	1455	1445	1450	1435	1400	1400
Reactor Outlet Temperature, °F	126	125	126	125	126	125	20	21	20	20	20	20
Rate of Reactor Δp Increase, in. water per hr	0.0	0.0	0.0	0.0	0.0	0.4	0.0	0.0	0.0	0.0	0.0	0.4
Dry Product Gas Analysis, mole %												
CO	12.2	14.1	15.0	19.1	19.3	19.8	7.3	9.2	14.4	14.6	15.9	18.9
CO <sub>2</sub>	10.7	9.6	7.7	5.6	5.1	4.3	18.2	17.0	12.7	12.4	10.7	9.4
H <sub>2</sub>	64.0	61.2	59.6	56.7	57.2	57.0	74.5	73.8	72.9	73.0	73.3	71.6
CH <sub>4</sub>	1.3	3.7	3.9	2.9	4.7	4.5/2/	-	-	-	-	0.1	0.1
N <sub>2</sub> /1/	11.8	11.4	13.8	15.7	13.7	14.4	-	-	-	-	0.1	0.1
Atomic Balances, %												
C	104.3	101.7	102.0	102.0	100.7	100.0	101.4	95.1	96.3	95.3	96.5	98.2
H	100.2	98.3	99.5	94.3	93.8	97.3	106.2	99.5	97.1	96.3	105.6	101.2
O	99.5	100.8	95.9	96.9	91.5	91.2	106.0	99.9	98.3	94.1	101.7	98.0
N	103.2	87.9	109.8	120.3	99.4	99.7	-	-	-	-	-	-
Outlet Temperature Calc. from Water Gas Shift Constant, °F												
	1490	1500	1495	1545	1515	-	1450	1440	1520	1515	1420	1455
Outlet Temperature Calc. from Steam-Methane Constant, °F												
	1470	1435	1460	1390	1510	-	-	-	-	-	-	-

FILE NO. E-3114  
JOY:LA 11/12/58

1/1 Nitrogen diluent from feedstock.  
2/ At start of test period. Severe catalyst deactivation was noted during test.





FILE NO. E-3116  
JCY:LA 11/12/59

