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DEVELOPMENT OF BEACON TECHNOLOGY

Quarterly Report for the Period July—September 1982

Work Performed Under Contract No. FC21-80MC14400

For
Morgantown Energy Technology Center
Morgantown, West Virginia

By
TRW Energy Development Group
Redondo Beach, California

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Prepared for
U.S. Department of Energy
Assistant Secretary for Nuclear Energy

Prepared by
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ABSTRACT

The BEACON process involves the catalytic deposition of a highly reactive form of carbon from a gas stream which contains carbon monoxide. The carbon-depleted gas is combusted with air to produce power, and the carbon is reacted with steam to produce methane or hydrogen.

Catalyst development at SOHIO showed that larger quantities (4 kg) of satisfactory C77-K2 catalyst could be prepared. Several preparation modifications were studied, and, although some showed satisfactory results, none were sufficiently attractive to be adopted.

Studies were conducted at SOHIO on promoters for the C77 type catalyst that would increase hydrogen production and suppress methane. Several were found, one of which operates at 550°C.

The C77-K1 catalyst was found not to be satisfactory for converting oil shale retort off-gas to hydrogen. The C77-K2 catalyst does perform this conversion under economically attractive conditions.

Physical characteristics for sieve fractions of a C77-K2 catalyst were determined. A new catalyst support was evaluated and found to be unsatisfactory.

Tandem Reactor development proceeded at TRW with completion of construction and the test readiness review and the initiation of shakedown testing.

Good fluidization with a C77-K2 catalyst was achieved with the installation of a stirrer in a 1.5-inch reactor at SOHIO. Good steady state carbon deposition and steaming rates were achieved over a 10 cycle test.

1. INTRODUCTION

The BEACON process converts low Btu gas into a substitute natural gas (SNG) and electric power. The original investigations of the chemistry and the necessary catalyst systems were performed by TRW. Since July 1978, the Standard Oil Company of Ohio (SOHIO) has joined with TRW to develop the process.

The joint TRW/SOHIO program has concentrated on the development of catalysts to perform the BEACON process chemistry, bench-scale testing to define suitable process conditions, reactor studies and engineering analysis pertaining to the development of conceptual commercial designs and studies of their efficiencies, costs and economics.

The current program operates under a cooperative agreement among DOE, SOHIO and TRW (Cooperative Agreement No. DE-FC-21-80MC14400). This report covers the period from July through September 1982.

2. SUMMARY

2.1 CATALYST DEVELOPMENT AT SOHIO

Duplicate 4 kilogram batches of C77-K2 catalyst were prepared in a 5-gallon reactor at the SOHIO pilot plant. Samples from both batches were subjected to carbon deposition at 450°C up to approximately 25% by weight of carbon. Satisfactory deposition rates were obtained with both batches. Similarly steaming to remove carbon down to approximately 10% by weight showed satisfactory rates with both batches, and the rates stayed constant over the four cycles tested. A scale-up procedure for producing large quantities of C77-K2 has been proven.

Two areas in the preparation of C77-K2 catalyst were studied to investigate their importance to catalyst performance. The first area, designated Modification I, involved changes in the treatment of the catalyst support. These experiments demonstrated that the standard treatment is better than Modification I.

The second area investigated had as its objective improving catalyst performance by modification of the catalyst preparation. These changes are given the general designation, Modification II. Two of the approaches were entirely procedural and are designated Modifications IIA and IIB. While Modification IIA resulted in a catalyst with increased reaction rates it also took a much longer time to prepare. Therefore this procedure will not be substituted for the standard preparation of C77-K2 catalyst material. Modification IIB was slightly poorer than the standard procedure. Modification IIC involved an entirely different approach to catalyst preparation. The results of this procedural modification were much poorer than a standard C77-K2 catalyst.

A series of experiments was performed using promoters for the purpose of increasing the hydrogen production through reducing methane formation during steaming. These promoters were designated Classes I, II and III. C77-K1 and C77-K2 catalysts were used with all three types of promoters.

Promotion of the catalysts with the Class I promoter did not work when the steaming temperature was 550°C. However at 650°C both catalysts showed consistent activity when promoted with the Class I material. The methane suppression was very good with the promoted C77-K1 catalyst and only marginal with C77-K2 catalyst. Class II materials are good promoters for increasing steaming rates but were found to be useless as methane suppressors.

The best results were obtained by promoting the C77-K2 catalyst with a Class III material. With this combination the rates were good and the methane suppression was excellent. An additional advantage was that the steaming could be performed at 550°C which is 100°C lower than the temperature needed for operating the C77-K1 catalyst promoted with a Class I ingredient.

In the two previous quarterly reports data were presented showing that the C77-K2 catalyst was attractive for converting an off-gas from an oil shale retort to hydrogen. Comparative evaluation of the C77-K1 catalyst for performing the same conversion was conducted this quarter. The C77-K1 catalyst was found not to be suitable for this conversion in the temperature range of 450°-550°C studied.

The physical characteristics of a fresh C77-K2 catalyst were determined by sieve cut and again after overloading with carbon and gasification. At all sieve fractions there was an increase in the smaller sized pores and a decrease in the volume associated with larger pores. A new catalyst support was investigated for the K1 and K2 catalysts, but it was found to be not as satisfactory as the C77 system.

2.2 BENCH-SCALE TESTING

Tandem Reactor development at TRW proceeded during the current quarter with the completion of construction, the conduction of a test readiness review and the initiation of shakedown. The Tandem Reactor system is a two reactor apparatus designed to permit simultaneous running of the carbon deposition and steaming operations of the BEACON process with the transfer of BEACON solids between reactors in the fluidized state. In the early shakedown process some difficulties were experienced with the aluminized coating on

the stainless steel reactor walls. With cleaning of the reactor walls by brushing the situation did improve. K1 based BEACON solids, selected on the basis of catalytic activity and fluidization behavior, will be used as the initial charge to both carbon deposition and steaming reactors. A particular batch of K1 based BEACON catalyst has been selected and qualified by laboratory testing.

Ten cycles of carbon deposition and steaming were achieved in a 1.5-inch reactor at SOHIO. A stirrer had been installed in the reactor and this apparently achieved the desired improved fluidization. The 10 cycles were performed using a C77-K2 catalyst. For carbon deposition the maximum rate achieved was 0.85 grams of carbon per hour per gram of active catalyst. In the steaming the best rate achieved under reliable conditions was 0.63 grams of carbon removed per gram of active catalyst per hour.

3. CATALYST DEVELOPMENT

The Catalysts appropriate for the BEACON chemistry are being developed at SOHIO and TRW. SOHIO is concentrating on supported catalysts and TRW works on developing unsupported carbonaceous catalysts. During the quarter reported here all of the catalyst development work was performed at SOHIO.

3.1 CATALYST DEVELOPMENT AT SOHIO

3.1.1 Preparation and Evaluation of C77-K2 Catalyst

3.1.1.1 Large Quantity Preparation and Evaluation

The C77-K2 catalyst developed at SOHIO can be considered as a candidate catalyst for performing the BEACON chemistry for converting low or medium Btu gas to a substitute natural gas. In previous reports the preparation and evaluation of this catalyst has been described. Up to the present time all the preparations have been on a laboratory scale.

To see if a satisfactory catalyst could be prepared on a larger scale, two 4 kilogram batches of C77-K2 were prepared in a 5-gallon reactor at the SOHIO pilot plant. The active catalyst material was introduced onto the support in equal quantity batches of this pilot plant prepared material. The same procedure was used in both batches.

The two batches then were subjected to 4 cycles of carbon deposition and steaming in laboratory scale equipment using a standard laboratory procedure which has been discussed in previous reports. The carbon deposition was conducted at 450°C with the attempt to deposit up to approximately 20% by weight of carbon. The subsequent steaming step was conducted at 550°C and attempted to remove the carbon down to about 10% by weight. The carbon deposition and removal rates were reasonably consistent for both batches over the 4 cycles studied. The first batch deposited carbon at rates varying from 1.32 to 1.47 grams of carbon deposited per hour per gram of active catalyst. The second batch showed similar deposition rates from 1.11 to 1.54. In the case of the first batch the amounts of carbon deposited

varied from 20.5% by weight to 27%. Similarly the second batch had carbon deposit varying from 17.7% to 24.6%.

Steaming of the first batch gave carbon removal rates varying from 0.95 to 1.01 grams of carbon per hour per gram of active catalyst. With the second batch the rates were from 0.88 to 1.05. In the first batch the carbon remaining after removal varied from 9.9% to 11.8% and with the second batch the remaining carbon varied from 9.2% to 12.4%.

The activity test of this large scale catalyst preparation showed that the scaling up was a success. The produced catalyst had excellent reactivity. Based on the similarity of the rates of the two batches it can be concluded that the incorporation of active catalyst into the support is very reproducible. A satisfactory scale up procedure for producing large quantities of C77-K2 catalyst has been provided.

3.1.1.2 Preparation Modifications

Two areas in the preparation of C77-K2 catalysts were studied to investigate their importance to catalyst performance. The first area studied was that of treatment of the catalyst support. These variations are described under the heading of Modification I. The second area investigated involved the procedures pertaining to incorporating the active catalyst on the support. These changes are designated under the heading Modification II.

Two sets of experiments were carried out as part of the Modification I investigation. The first set of experiments was designed to determine the influence of Modification I. Catalysts were prepared incorporating Modification I and compared with another set of catalysts where all steps in the procedure were standard. All of these catalyst batches were tested for carbon deposition and steaming reactivity in the usual manner.

The first set of experiments evaluating the importance of Modification I showed that the standard procedure was preferred over the Modification I. The standard catalyst support treatment results in good interaction between

the support and the catalyst. When Modification I was used the interaction between the support and the catalyst was poor and difficulties began to appear as soon as three cycles of carbon deposition and steaming.

In the second set of experiments involving Modification I a batch of C77-K2 catalyst was prepared using the standard procedure and divided into three batches. One batch was mixed with an equal volume of the catalyst support which had been treated according to Modification I. The second batch of the catalyst was sieved first. The -200 +270 mesh fraction was separated from the rest of the catalyst and was mixed with an equal volume of catalyst support which had been treated according to Modification I. The third batch was the control and the C77-K2 catalyst was mixed with an equal volume of catalyst support treated according to the standard procedure. Each of these test batches was subjected to carbon deposition and steaming to determine their reactivity in the usual manner.

Results of the second set of experiments are shown in Figures 3-1 and 3-2. Figure 3-1 shows a plot of the rates of carbon deposition or steaming vs cycle number when the catalyst is a 1:1 physical mixture of standard C77-K2 catalyst and its catalyst support treated according to Modification I. The solid line with circles represents the carbon deposition rates. There is a considerable drop in rate after the first cycle. The steaming, represented by the dash line and x marks, decreases much more slowly. A similar decrease in rates was observed when the -200 +270 mesh C77-K2 portion was diluted with equal amounts of support treated according to Modification I. Figure 3-2 shows a plot of rates vs cycle numbers for a 1:1 mixture of C77-K2 catalyst and catalyst support treated by the standard procedure. There is no decrease in reaction rates. These experiments demonstrated that the standard procedure is better than Modification I because the standard procedure incorporates an interaction between the catalyst and the support.

Experiments were initiated with the objective of improving catalyst performance by modifying the catalyst preparation. These changes are given the general designation Modification II. Three different approaches were tried and have been designated Modifications IIA, IIB and IIC.

Figure 3-1. Rates of Mix of Catalyst Support and C77-K2 Catalyst (Modification I Treatment of Catalyst Support)

Figure 3-2. Rates for Deposition and Steaming (Catalyst: 50% Catalyst Support With Standard Treatment and 50% C77-K2 Catalyst)

Modifications IIA and IIB were entirely procedural and Modification IIC involved a different approach. In all cases the level of the active catalyst was the same.

The resultant catalysts were tested for reactivity and compared to the activity of a catalyst made by the standard method. Table 3-1 shows the carbon deposition rates and steaming rates obtained with three different catalysts prepared according to the three modifications described above. For comparison similar rates are included for a catalyst prepared by the standard method. The catalyst prepared by Modification IIC had a low carbon deposition and a low steaming rate. It was concluded that Modification IIC is not an attractive revision of the preparation method. When the active catalyst was prepared according to Modification IIA significant improvement was obtained. With Modification IIB the rates were slightly lower than for the standard procedure. While Modification IIA resulted in a catalyst with increased reaction rates it also took a much longer time to prepare. Other possible ways to improve the catalyst may be explored before Modification IIA is accepted as part of the standard procedures.

Table 3-1. The Effect of Changes in Catalyst Preparation

Modification	Average Rates C Deposition	(qC/Hr/q Active Catalyst) Steaming
None	1.12	0.92
IIC	0.71	0.19
IIA	1.38	0.97
IIB	1.11	0.73

3.1.1.3 Promotion of C77 Catalyst for Hydrogen Production

A series of experiments was performed using promoters for the purpose of increasing the hydrogen production through reducing methane formation during steaming. C77-K1 and C77-K2 catalysts were used with three types of promoters. These promoters were designated Classes I, II and III. In all cases standard catalyst preparation procedures were followed with the promoters being added to the active catalyst ingredients. With the C77-K1 catalyst the Class I promoter was added to two levels designated Levels 1 and 2. With the C77-K2 catalyst the Class I promoter was added in the amounts designated Levels 2 and 3.

Two promoters of Class II designated IIA and IIB were tested with a C77-K2 catalyst. The Class IIA material was tested at a concentration Level 4 and the Class IIB material at a concentration Level 5.

The Class III material was tested with C77-K2 catalyst for its ability to suppress methane formation.

Promotion of C77-K1 and C77-K2 catalysts with Class I promoter at Levels 1 and 2 did not work when the steaming temperature was 550°C. When the promoter concentration was at Level 3 the activity of C77-K2 catalyst lasted a little bit longer at 550°C but without much improvement in methane suppression. However at 650°C neither the C77-K1 catalyst nor the C77-K2 catalyst promoted with Class I material at Level 2 showed any sign of decline in activity. The methane suppression was only marginal with the promoted C77-K2 catalyst, but it was very good with the promoted C77-K1 catalyst.

Class II materials are good promoters for increasing steaming rates but are useless as methane suppressors.

The best were obtained with C77-K2 catalyst promoted by Class III material. The rates were good and the methane suppression was excellent. As an additional advantage the steaming was carried out at 550°C which is

100°C lower than the temperature needed for operating a C77-K1 catalyst promoted with a Class I promoter. Good steaming rates were obtained in both the first and second cycles.

3.1.2 Evaluation of C77-K1 Catalyst for Converting Oil Shale Retort Off-Gas to Hydrogen

In the two previous quarterly reports the conditions used and the results obtained were described for converting an off-gas from an oil shale retort to hydrogen using a C77-K2 catalyst. It had been determined that for economic reasons the ratio in millions of Btu of the feedgas to the hydrogen produced should be less than three. Conditions for achieving this with C77-K2 catalyst had been identified, and they are reported in the last quarterly report.

The same single reactor system was used as described in the 1st quarterly report of 1982. The feed in these experiments was typical of that off-gas produced by an oil shale retort with the carbon dioxide removed. Twenty cc of C77-K1 catalyst was used with the carbon being deposited at 450°C, 500°C and 550°C with steaming at 550°C between depositions.

The results obtained with C77-K1 catalyst are shown in Table 3-2. The Btu values are disappointing, being higher than three, the target values. Table 3-3 contains the Btu ratios and percent conversion values obtained with C77-K2 and C77-K1 catalysts at 450°, 500° and 550°C. There are several differences between the two catalysts systems. It was surprising to find the C77-K1 catalyst to be insensitive to a change in temperature. Its Btu ratios are the same at all three temperatures, 4.61, 4.59 and 4.60. This is mainly the consequence of the invariance of the total carbon conversion at the three different temperatures (34.4%, 33.7% and 33.6%). In contrast the total carbon conversion increases with increasing temperature for the C77-K2 catalyst. This improved conversion is reflected in the improved Btu ratio for this catalyst. The Btu ratios are the same for both catalysts at 450°C but the ratio decreases for the C77-K2 catalyst at higher temperatures while the ratio remains the same for the C77-K1 catalyst.

Table 3-2. Summary of the Results Using Shale Retort Off-Gas With Catalyst C77-K1

Run No.	Temp (°C)	Average C Deposition Rate (gC/Hr/g Active Cat.)	Total C	Conversion* (%)			Btu Ratio Feedgas/H ₂	Overall Closure %
				CO	C ₂ +	CH ₄		
44	450	0.290	32.2	57.5	54.9	-11.2	4.87	98.4
45	450	0.326	36.6	69.5	57.0	-11.4	4.35	95.8
46	500	0.298	33.7	58.9	58.6	4.7	4.59	90.3
48	550	0.294	33.6	45.5	55.5	1.0	4.60	97.5

* Negative conversion (-) indicates net production of species.

Table 3-3. Comparison of C77-K2 and C77-K1 Catalysts Using Shale Retort Off-Gas

Temperature (°C)	Catalyst	Total C	Conversion* (%)			Btu Ratio Feedgas/H ₂
			CO	C ₂ +	CH ₄	
450	C77-K1**	34.4	63.5	56.0	-11.3	4.61
	C77-K2	34.6	60.7	73.3	-85.6	4.62
500	C77-K1	33.7	58.9	58.6	4.7	4.59
	C77-K2	42.8	49.8	83.9	-85.8	3.51
550	C77-K1	33.6	45.5	55.5	1.0	4.60
	C77-K2	52.4	28.3	95.2	-40.7	3.07

* Negative conversion (-) indicates net production of species.

** Average of Run Nos. 44 and 45 of Table 3-2.

There is a big difference in the conversion of methane between the two catalysts. The C77-K2 actually produces large amounts of methane while the C77-K1 catalyst forms only small amounts of methane at 450°C. At 500°C and 550°C carbon is being deposited from methane with C77-K1 catalyst. The C77-K2 catalyst is very effective in converting the hydrocarbons to carbon and at 550°C practically all of the C_2+ are gone with this catalyst. At that temperature about 50% of the C_2+ component is still present with the C77-K1 catalyst. With both of the catalysts it was possible to remove the deposited carbon by steaming. Thus using a single reactor the C77-K1 catalyst is less effective than the C77-K2 catalyst in depositing carbon from a oil shale retort off-gas.

3.1.3 Physical Characteristics of Supported Catalysts for BEACON Chemistry

The data reported in this section are analytical results obtained on C77-K2 catalysts before and after carbon deposition and gasification. The physical characteristics of a fresh sample of C77-K2 catalyst are presented in Table 3-4.

The properties reported in Table 3-4 remain reasonably constant across the particle size ranges. The BET surface area averaged 41 m²/g.

A similar batch of C77-K2 catalyst was overloaded with carbon (approximately 35% by weight) and then the carbon removed by gasification with steam to approximately 22% carbon. Similar physical characteristics as reported in Table 3-4 for the fresh catalyst were determined on this sample and are reported in Table 3-5. The samples show an increase in surface area which can be attributed almost entirely to the carbon present. A plot of surface area vs carbon content for these sieve cuts would yield a straight line the intercept of which comes near the value of 41 m²/g obtained for the freshly reduced catalyst.

The pore volume distribution from this set of samples is shown in the lower part of Table 3-5. The differences between Tables 3-4 and 3-5 reasonably may be attributed to the residual carbon content of the catalyst. When an average particle size distribution is computed on the basis

Table 3-4. Physical Characteristics of Unused C77-K2 Catalyst by Sieve Cut

Sieve Cut (Mesh)	100x140	140x200	200x270	270x325	325xPan
BET Surface Area (m^2/g)	42.7	42.8	41.0	39.8	38.6
Weight % of Fraction	2.0	10.1	29.0	14.2	44.5
Bulk Density (g/ml)	.485	.540	.554	.564	.594
Pore Volume Distribution ($\mu\text{el/g}$) by Nitrogen Adsorption					
20 x 100 Å	19.2	19.4	14.0	16.6	15.5
100 x 200 Å	12.3	12.3	9.0	13.9	11.9
200 x 300 Å	20.8	20.3	19.8	28.3	23.5
300 x 400 Å	27.9	27.8	38.4	33.1	36.5
400 x 500 Å	36.9	31.2	74.1	47.4	40.7
500 x 600 Å	71.1	53.5	84.5	31.8	54.3
Total	188.2	164.5	239.8	171.1	182.4

Table 3-5. Physical Characteristics of a C77-K2 Catalyst By Sieve Cut After Overloading With Carbon and Gasification

Sieve Cut (Mesh)	100x140	140x200	200x270	270x325	325xPan
BET Surface Area (m^2/g)	72.5	69.3	67.4	64.0	54.1
Weight % Fraction	5.8	15.4	37.7	12.1	28.7
Weight % Carbon in Fraction	29.7	24.1	21.0	20.0	13.6
Bulk Density (g/ml)	.241	.230	.217	.245	.351
Pore Volume Distribution ($\mu\text{el/g}$) by Nitrogen Adsorption					
20 x 100 Å	38.8	41.2	31.1	28.1	24.7
100 x 200 Å	27.5	27.1	24.6	19.2	19.3
200 x 300 Å	29.5	29.8	32.1	37.5	29.1
300 x 400 Å	33.1	32.5	32.2	39.5	34.2
400 x 500 Å	25.7	28.6	33.7	38.5	35.7
500 x 600 Å	42.3	33.3	37.6	38.9	41.7
Total	196.9	192.5	191.3	191.7	184.7

of a log normal distribution for both catalysts the used catalyst exhibits a more uniform pore distribution. This difference reflects a large increase in the volume of the smaller pores relative to the larger pores in the overloaded catalyst. These analytical data suggest little change in the structure of the catalyst support after overloading with carbon deposition and partial gasification.

3.1.4 Chemical Characteristics of Some Supported Catalysts for BEACON Chemistry

The results described in Section 3.1.3 above and similar investigations described in the 1st and 2nd Quarter Reports for 1982 suggested the possible use of another catalyst support. The catalyst support selected had a BET surface area of $446 \text{ m}^2/\text{g}$ with most of the pore volume in the small pore size range. For example with a total pore volume of 797 microliters per gram 594 microliters per gram were in the $20 \times 100 \text{ \AA}$ range and 196 in the $100 \times 200 \text{ \AA}$ range. This is in contrast to the C77 support which had a much smaller pore volume in these pore sizes. The test program was performed in the 1-1/2" stainless steel laboratory reactor with a superficial velocity of about 0.13 fps under deposition conditions and about 0.03 fps under gasification conditions. These rates are slightly above minimum fluidization during gasification and well into fluidization during deposition with this catalyst material. However, the size of the reactor may be such that fluidization was not achieved.

Following each half cycle the reactor was unloaded and the catalyst visually examined. Samples were taken for carbon analysis and the remainder of each fraction was pushed through an 80 mesh screen, the settled bulk density determined and the reactor reloaded for the subsequent next half cycle.

The results with a K2 catalyst deposited on this new support are compiled in Table 3-6 over five cycles of carbon deposition and steam gasification. Materials balances calculated by different approaches achieved general agreement. The approaches were direct weighing, carbon analysis and GC mass balance. The results were disappointing in that the carbon monoxide conversions were low and the volume occupied by the catalyst increased steadily with the cycle number.

Table 3-6. Results and Conditions From a K2 Catalyst Series

Cycle	1/2	1	1-1/2	2	2-1/2	3	3-1/2	4	4-1/2	5
Direction ¹	D	G	D	G	D	G	D	G	D	G
Temperature (°C)	455	597	453	593	450	595	469	594	461	596
Pressure (Psig)	10	6-9	10	6-9	10	6-8	10-11	6-8	10	6-8
Feed Composition (%)										
H ₂	1.2		1.1		2.2		0.0		1.0	
CO	9.3		10.8		10.2		10.5		9.9	
N ₂	Bal		Bal		Bal		Bal		Bal	
H ₂ O		100		100		100		100		100
Total Feed Rate (Mol/gC Free Catalyst/Hr)	.23	.046	.23	.048	.25	.050	.26	.053	.27	.053
Global Rate of Deposition ² (mg/min)	18.3	-76	18.1	-73	6.8	-62	21.1	-70	12.0	-64
Selectivities ³										
F1	.82	.12	.86	.10	.63	.10	1.00	.12	.82	.13
F2	.06	.61	.05	.64	.08	.68	-	.64	.04	.65
F3	.12	.27	.09	.26	.29	.22	-	.24	.14	.22
Cycle Efficiency (Mean, %)		85		85		91		83		82
(On Deposit Weight %)		89		92		106		89		88
CO Conversion (%)	48		38		22		42		29	
H ₂ Conversion (%)	55		50		49		-		60	
H ₂ /H ₂ O Ratio	4.4	.81	4.3	.81	8.6	.61	-	.70	5.3	.68
Peak H ₂ O Conversion (%)		53		52		44		49		47

¹ D = Deposition; G = Gasification

² Mean value shown for deposition; peak value shown for gasification.

³ Reaction bases for selectivities: Deposition: F1; $2\text{CO} = \text{CO}_2 + \text{C}$ Gasification: F1; $\text{C} + \text{H}_2\text{O} = \text{CO} + \text{H}_2$
 F2; $\text{CO} + \text{H}_2 = \text{H}_2\text{O} + \text{C}$ F2; $\text{C} + 2\text{H}_2\text{O} = \text{CO}_2 + 2\text{H}_2$
 F3; $\text{C} + 2\text{H}_2 = \text{CH}_4$ F3; $2\text{C} + 2\text{H}_2\text{O} = \text{CO}_2 + \text{CH}_4$

A similar series of tests were performed with K1 catalyst deposited on the same support and the results are presented Table 3-7. The volume changes with the K1 catalyst on this support were more severe than with the K2 catalyst. On a total product volume basis the change is about 0.7 ml/g/cycle with the K1 catalyst compared to 0.4 ml/g/cycle for the K2 catalyst. Neither value represents good stability. For the K1 catalyst the rate of deposition declined with the accumulation of deposit on the catalyst. Both catalysts showed the manufacture of significant amounts of methane which is undesirable in a catalyst designed for hydrogen production. For these reasons this new catalyst support is not considered attractive.

Table 3-7. Results and Conditions from a K1 Catalyst Series

Cycle	1/2	1	1-1/2	2	2-1/2	3	3-1/2	4
Direction ¹	D	G	C	G	D	G	D	G
Temperature (°C)	455	588	462	585	463	590	475	589
Pressure (Psig)	10	7-9	10	7-9	10	6-8	10	7-8
Feed Composition (%)								
H ₂	1.0	-	2.0	-	0.0	-	1.0	-
CO	10.0	-	10.0	-	9.7	-	9.9	-
N ₂	Bal	-	Bal	-	Bal	-	Bal	-
H ₂ O	-	100	-	100	-	100	-	100
Total Feed Rate (Mol/gC Free Catalyst)	.174	.036	.186	.037	.189	.038	.193	.039
Global Rate of ² Deposition (mg/min)	29.4	-79.6	34.7	-48.6	13.0	-54.7	30.1	-49.3
Selectivities ³								
F1	.91	.13	.84	.16	1.00	.17	.92	.16
F2	.05	.62	.07	.67	-	.62	.06	.70
F3	.04	.25	.09	.17	-	.21	.02	.14
CO Conversion (%)	65		52		28		63	
H ₂ Conversion (%)	39		35		-		33	
Peak H ₂ O Conversion (%)	-	56	-	42	-	44	-	43
H ₂ /H ₂ O Ratio	4.0	.93	6.3	.61	-	.64	3.7	.66
Cycle Efficiency (Mean, %) (On Weight, %)		77 81		78 82		106 126		62 71

¹ D = Deposition; G = Gasification

² Mean value shown for deposition; peak value shown for gasification.

³ Reaction bases for selectivities: Deposition: F1; $2\text{CO} = \text{CO}_2 + \text{C}$
 F2; $\text{CO} + \text{H}_2 + \text{H}_2\text{O} + \text{C}$
 F3; $\text{C} + 2\text{H}_2 = \text{CH}_4$
 Gasification: F1; $\text{C} + \text{H}_2\text{O} = \text{CO} + \text{H}_2$
 F2; $\text{C} + 2\text{H}_2\text{O} = \text{CO}_2 + 2\text{H}_2$
 F3; $2\text{C} + 2\text{H}_2\text{O} = \text{CO}_2 + \text{CH}_4$

4. BENCH-SCALE TESTING

4.1 TANDEM REACTOR DEVELOPMENT AT TRW

Tandem Reactor development proceeded very nearly on schedule during the current quarter with the completion of construction, the conduct of a test readiness review and the initiation of shakedown. Approximately one week of slip in schedule was almost entirely due to problems encountered with the Alon coating on the reactors (aluminization of the reactor stainless steel surfaces). Shakedown will be completed and testing will be initiated during the next quarter (4th Quarter CY 1982).

4.1.1 Tandem Reactor Concept and Objectives

The Tandem Reactor System is a two-reactor apparatus designed to permit simultaneous running of the carbon deposition and steaming operations of the BEACON Process with the transfer of BEACON solids between reactors in the fluidized state. The concept is depicted in the schematic diagram of Figure 4-1.

The objective of Tandem Reactor Testing is to generate the missing data needed for BEACON Process scale up. Specifically, the Tandem Reactor unit will be used to evaluate the long-term performance of candidate BEACON catalysts, previously selected on the basis of batch testing, under quasi-continuous solids transfer between the deposition and steaming reactors. In the future (1983), the unit is expected to be used for the evaluation of hydrogen production promoters derived from BEACON solids technology. Successful operation of the Tandem Reactors will represent the first integrated demonstration of the two major unit operations of the BEACON Process.

Circulation patterns in each of the fluidized bed reactors tend to mix the solids thoroughly so that removal of the particles for transfer is a random process. Many particles stay in the reactor for less than the mean reactor holding time and some stay in appreciably longer. What effect this increased holding time will have on the chemical and physical

Figure 4-1. Tandem Reactor System Schematic

stability of the carbon and catalyst is not now known. The Tandem Reactor System will be used to investigate and quantify such effect, if any.

4.1.2 Construction Progress and Test Readiness Review

Assembly and instrumentation of the Tandem Reactor System was completed during this reporting period. Detailed description of the apparatus and its component parts, including photographs of completed sections, were presented in the previous quarterly report. Figure 4-2 shows the reactor section of the apparatus completely installed and instrumented.

Briefly, the Tandem System consists of the following sections: gas feed system, steam boiler, carbon deposition reactor, steam gasification reactor, product gas cleanup train, reactor pressure control and product gas metering system, solids transfer system, and data acquisition system. The deposition reactor has a six inch inside diameter and the gasifier a three inch inside diameter (the difference in reactor size was dictated by the difference in deposition and gasification rates at the desired operating ranges). Both reactors were constructed from 316 stainless steel parts and were surface aluminized by the Alon Process to prevent catalyst contamination from the reactor walls (especially through carburization of the deposition reactor walls).

The "Alonization Process" was supposed to produce a continuous protective coating of aluminum-steel alloy on the reactor surfaces, cyclone dip-legs, and solids transfer lines, but both visual observation and SEM photographs of sections cut from the dip-legs and transfer lines indicated defects in several places. Visual inspection revealed "rusty" spots on the surfaces; SEM photographs revealed hairline but deep cracks on the protective layer. The Alon Company representative inspected the reactor parts and concluded that the "rusted" spots were due to loose granules of steel and that after brushing them off, there is nothing to worry about. This contention appeared to be justified by short-term carburization (11 and 24 hours) performance tests on the empty carbon deposition reactor. These tests showed insignificant carbon deposition on the reactor walls, especially the test performed subsequent to brushing the reactor; the data

Figure 4-2. Tandem Reactors and Stand

are presented in the next section. However, tests performed on specimens cut from the dip-leg and the transfer line tubings showed appreciable carburization potential. It is conceivable that carburization may occur at areas of the reactor where the protective coating was defective or became damaged; but current evidence appears to indicate that such occurrence will not be extensive. Thus, the decision was made to continue with the current set of reactors but to closely monitor catalyst contamination from the reactor walls. If contamination appears to become a problem, the deposition reactor will be replaced by one constructed of Inconel 600 alloy.

On August 17, upon completion of construction and instrumentation of the apparatus, the "Test Readiness Review" of the facility was held. The review was based on up-to-date P&IDs, shakedown and operating plans, operating and safety procedure documentation, and physical inspection of the facility. The review committee, a group of managers and engineers not closely associated with the design and construction of the system but trained to spot potential safety and operational problems, was comprised of the Division's Health and Safety Engineer, the Chemistry and Chemical Engineering Operation's Chief Engineer, the BEACON Program Manager, and two levels of Line Management; the review meeting also was attended by the Project Manager and most of the Project's Operators. The review went very well with only nine "action items" generated concerning minor hardware and documentation modifications; the changes were made prior to start of shakedown for the most part and were completed before the end of the quarter.

4.1.3 Shakedown Plan and Progress - Operator Training

Apparatus shakedown and operator training started in the latter part of August and continued to the end of the quarter; it should be completed by the end of October or early November. Up to now the task has proceeded with very few problems; needed changes are made as soon as identified and defined, unless parts not immediately available are required. The shakedown plan is summarized in Table 4-1; items completed or underway are also indicated in the table.

Table 4-1. Shakedown Plan Summary

-
- Write test procedures and conduct test readiness review - completed
 - Perform "dry run" tests for operator training - completed
 - Verify mechanical integrity of system (pressure check, leak check, electronics check-out) - completed
 - Calibrate instrumentation for shakedown testing - completed
 - Perform empty reactor carburization test and verify results by thorough brushing of deposition reactor - completed
 - Perform hot gas test (nitrogen) to verify operability and adequacy of heaters and to test temperature sensor and recording system - completed
 - Load solids and perform the following tests (use cold or hot N₂ as required)
 - Verify pressure control system performance - completed
 - Verify DP system operability - underway
 - Determine solids entrainment vs gas velocity at operating temperatures and pressures - underway
 - Verify solids transfer system operability
 - Determine quality of fluidization (pressure drop fluctuation measurements, bed expansion, etc.)
 - Perform any hardware changes required - underway
 - Debug data acquisition system
 - Calibrate solids transfer system and feed & product gas instrumentation for planned operating parameter ranges
 - Verify shakedown findings with reaction gases under reaction conditions
-

As indicated in Table 4-1 the following items were either completed or were near completion at the end of September: Dry runs were performed for the training of the operators - especially the chief shift operator of each of the two shifts (a two-shift operation is planned for the latter stages of shakedown and for performance testing); operator training will continue until the end of shakedown. The mechanical integrity of the system was demonstrated through hydrostatic and pneumatic pressure testing and through gas leak checking. The system was rendered leak-proof to the degree required for the generation of reliable data; the criterion met was a leak rate lower than one percent of the minimum gas feed rate planned for use during testing. The instrumentation needed for shakedown was calibrated (orifices, gauges, temperature and pressure sensors, gas chromatographs, NDIRs, etc.). About 80% of the data acquisition software for converting the raw data into real time material balances has been written. The adequacy of the heating system was verified after a few supplemental heaters and insulation were added to the solids transfer lines and reactor expanders; also the adequacy and accuracy of temperature and pressure sensors were determined. Empty reactor tests were conducted in the deposition reactor to determine the catalytic activity of reactor surfaces (see data below). The reactor was loaded with the selected K-1 based BEACON solids (see Section 4.1.5) and testing is underway to verify DP system operability, extent of solids entrainment as a function of gas velocity, quality of fluidization, operability of the solids transfer system, and the efficiency of the solids return system (internal cyclones). Items to be initiated in October include debugging of the data acquisition system, calibration of solids transfer system and system checkout under reaction conditions with reacting gases (carburization gas and steam).

A 24 hour and a 12 hour carbon deposition tests were performed on the "empty" deposition reactor (no catalyst present) to determine the catalytic activity of the reactor surfaces. The tests were performed at 100 psig and 450°C with a feed gas containing 33% carbon monoxide, 17% hydrogen and 50% nitrogen by volume. In the 24 hour test, the average carbon deposition rate was approximately 2.5 grams carbon per hour (less than 500 ppm CO₂ in the reactor effluent gas). The reactor was brushed well and the 12 hour

test was performed. The carbon deposition rate was 11 grams in 12 hours or less than 1 gram per hour and the CO₂ concentration in the effluent was less than 150 ppm. These low deposition rates met the criterion of "minimum catalyst contamination" by metals leached from the reactor walls.

4.1.4 Test Plan Outline

Upon completion of shakedown, the Tandem Reactor apparatus will be used to investigate the validity of the Tandem concept under reaction conditions and to determine performance stability of K-1 based BEACON solids under simultaneous deposition/steaming operation. The following paragraphs outline the planned testing.

K-1 based BEACON solids, selected on the basis of catalytic activity and fluidization behavior, will be used as the initial charge to both carbon deposition and steaming reactors. The carbon content of these solids, which were prepared outside the Tandem Reactor System, will be adjusted by carbon deposition and/or steam gasification to meet the compositional requirements defined for each reactor bed (90% carbon solids for the deposition reactor bed and 70% carbon solids for the steaming reactor bed are probable selections). The final selection on the quantity and composition of solids in each bed will be determined during shakedown.

Testing will start, first two months approximately, with a brief parametric investigation to bracket system operability (solids transfer behavior, solids entrainment and return, etc.) and to test the validity of the Tandem Reactor concept. It will conclude, last three months, with a long term (multi-cycle) run at the selected optimum operating conditions. For the most part testing will be performed in two shifts; however, it is planned to perform at least one 80 hour continuous run in 3 shift operation.

The operating pressure will be virtually the same for both reactors but all other operating conditions may differ (temperature, gas velocity,

quantity of solids, etc); the carbon content of the solids will differ. The following table summarizes the ranges of operating parameter values available for use in the selection of the set of operating conditions for multi-cycle testing:

	<u>Carbon Deposition</u>	<u>Steam Gasification</u>
● Temperature	400-550°C	550-650°C
● Pressure	5-100 psig	5-100 psig
● Gas Velocity	0.3-1.0 fps	0.3-1.0 fps
● Feed Gas Composition	Any composition up to 5 components	
● Solids Composition	To be determined from cold-flow testing	

The data collected will include the following: feed and effluent flowrate and composition; reactor temperatures and pressures; reactor gas velocities; pressure drop at several reactor bed levels (bed density and quality of fluidization); solids transfer rate; extent of solids entrainment in the product gas; changes in solids composition and physical properties as a function of processing time.

4.1.5 Selection of Catalyst for Tandem Testing

Early lab scale testing narrowed the choice of BEACON catalysts for methane production to K-1 and K-2 based solids. More recent batch mode testing at bench scale led to the conclusion that the most promising candidate catalyst was the unsupported K-1 solids with a possible back-up the supported K-2 catalyst being developed at SOHIO. Thus, the decision was made to use the available K-1 solids, prepared about a year ago for batch testing, as the initial charge of solids to the Tandem Reactor System.

These solids, labeled "neat" in Table 4-2, were screened for activity at lab scale prior to loading into the Tandem apparatus. The results are summarized in the first two columns of Table 4-2 and are compared to the composition and steam utilization predicted by thermodynamic equilibrium for steaming of graphite carbon (last two columns of Table 4-2); the data are virtually identical which indicates that aging of K-1 based BEACON solids and exposure to air have no effect on their activity in steam

Table 4-2. Activity Screening Tests of BEACON Solids for Tandem Reactor Use*

Product Gas Composition, % V/V (Dry Basis)						
Species	K-1 Carbon "Neat"		K-1 Carbon Diluted 2 : 1 With Alumina		Composition Predicted By Thermo.	
	600°C	650°C	610°C	650°C	600°C	650°C
CH ₄	8	5	7.5	5.5	8.8	5.3
H ₂	48	50	50.2	51.3	49.3	51.0
CO	17	27	17.5	25.1	16.7	25.7
CO ₂	27	18	24.8	18.1	25.2	18.0
	<u>100</u>	<u>100</u>	<u>100.</u>	<u>100.</u>	<u>100.</u>	<u>100.</u>
Steam Utilization	70%	78%	72%	78%	70.7%	78%

* Screened at ambient pressure and at 600°C and 650°C. The steam feed rate was one gram per hour per gram of carbon initially charged to the reactor; at least 80% of the carbon was gasified. K-1 based BEACON solids were used both "neat" and diluted with inert solids (alumina).

gasification. A different sample from the same solids was mixed with twice its weight of alumina granules* and the mixture was steam gasified at 610°C and 650°C. There was no measurable effect on steam gasification performance as shown by the data presented in the middle two columns of Table 4-2 (the slightly lower methane concentration in column 3 versus column 1 is due to the 10° higher temperature and not to the presence of the diluent alumina).

4.2 BENCH-SCALE TESTING AT SOHIO

The testing for this quarter was performed in the 1-1/2" BEACON reactor. A stirrer was installed to improve fluidization of the catalyst bed. A schematic drawing of the unit is presented in Figure 4-3.

The stirrer has five impellers of four blades each and operates at 65 rpm. Some nitrogen does leak from the shroud through the primary seal into the reactor and this distorted the nitrogen material balances from about 10% to 40%. Temperature is indicated only by the sand-bath thermocouple (TC6) which can be as much as 40°C off from the internal bed temperature.

Ten cycles of carbon deposition and steaming were satisfactorily performed in the 1.5" reactor. The catalyst used was a C77-K2 type. There were transition and induction periods but within each cycle a good period of steady state operation was achieved. These results are shown in Table 4-4 for the carbon deposition and Table 4-5 for the steaming. Deposition over Cycles 1-9 varied from 0.65 to 0.85 gmC/gm of active catalyst per hour. For a similar number of cycles of steaming the uptake rates varied from 0.39 to 0.63 grams of carbon removed per gram of active catalyst per hour. These figures are exclusive of Runs Q2S, Q3S and Q4S where problems in water feed occurred. These rates are also plotted in Figure 4-4.

* Diluent alumina may be used at later testing as a means of enhancing heat transfer and/or to keep carbon off reactor walls.

Figure 4-3. BEACON 1.5-Inch Reactor With Stirrer

Table 4-3. Run Q Depositions - Process Data, Deposition Rates

Cycle	Q10D	Q2D	Q3D	Q4D	Q5D	Q6D	Q7D	Q8D	Q9D	Q10D
Duration, Min	132	77	64	55	66	55	77	57	66	44
TC6, Deg-C	409	400	404	400	404	450	454	450	454	450
Psig	10	10	10	10	10	1	0	2	1.5	2.0
Gas, In, SCFH	2.73	3.00	3.06	2.95	2.94	2.83	2.79	2.88	2.73	2.89
Out	2.03	2.07	2.46	2.12	2.11	1.96	2.03	2.09	2.07	1.84
Gas In Comp., Pct										
H ₂	10.3	11.1	10.9	10.9	11.1	13.3	14.4	13.4	13.3	14.0
N ₂	30.3	30.3	29.7	28.6	29.1	27.8	28.3	28.7	28.7	28.1
CO	59.2	58.6	59.4	60.5	59.5	58.9	57.4	57.6	58.0	57.9
Gas Out Comp.										
H ₂	1.4	3.2	2.26	2.8	3.0	5.8	5.8	5.8	5.8	5.5
CO ₂	40.8	32.5	37.0	37.1	34.5	36.2	34.5	34.1	31.1	34.7
N ₂	45.2	44.0	45.4	45.6	44.4	42.8	44.1	45.0	46.2	45.9
CO	6.4	14.6	9.3	8.9	12.6	9.7	9.9	10.0	12.3	9.7
CH ₄	6.3	5.6	6.0	5.6	5.4	5.5	5.7	5.0	4.7	4.3
Deposition Rate										
GM-C/Hr	8.0	10.1	8.1	10.5	9.8	10.1	8.8	9.6	9.0	11.7
GM-C/Hr/Active Cat.	0.65	.81	.65	.85	.79	.81	.71	.77	.73	.94
Total GM-C Deposition	15.1	13.0	9.7	9.5	10.6	9.2	11.3	9.1	9.9	8.6

Table 4-4. Run C Steamings - Process Data, Uptake Rates

Cycle	Q1S	Q2S	Q3S	Q4S	Q5S	Q6S	Q7S	Q8S	Q9S	Q10SA	Q10SB
Duration	297	121	91	103	106	93	118	148	132	132	121
TC6, Degree C	576	570	570	570	572	550	550	550	550	550	550
Psig	10	10	10	10	10	0	2	1.3	1.5	1.5	2
Gas, In, SCFH	3.80	0.80	0.89	0.80	0.85	0.75	0.79	0.76	0.76	0.78	.76
Out	1.64	2.87	2.37	2.05	1.82	1.77	1.78	1.81	1.73	3.19	2.91
Water, In, G/Hr	33	43	39	50	34	34	35	34	35	34.0	35.5
Out											
Gas In Comp, %											
N ₂	100	100	100	100	100	100	100	100	100	100	100
Gas Out, %											
H ₂	29.3	36.8	30.4	34.7	23.9	32.4	29.8	32.4	32.1	26.4	29.8
CO ₂	16.6	19.3	16.3	18.0	13.0	16.1	14.8	15.4	15.7	12.6	14.2
N ₂	49.9	39.6	48.0	42.7	57.8	46.9	52.4	49.8	49.4	58.2	53.7
CO	1.4	2.0	2.5	2.1	2.2	2.0	2.0	1.7	2.0	1.9	1.9
CH ₄	2.8	2.3	2.8	2.4	2.4	1.4	1.0	.7	0.9	0.9	.5
Uptake Rate											
GM-C/Hr	5.2	10.3	7.8	7.0	5.1	5.3	4.8	4.9	4.9	7.8	7.4
GM-C/Hr/G Active Cat.	.42	.83	.63	.56	.41	.43	.39	.40	.40	.63	.60
Total GM-C Up	14.2	20.3	13.6	12.1	13.2	13.9	20.8	21.0	22.0	15.1	14.9

Figure 4-4. Reaction Rates, Run Q - SOHIO 1.5-Inch Reactor