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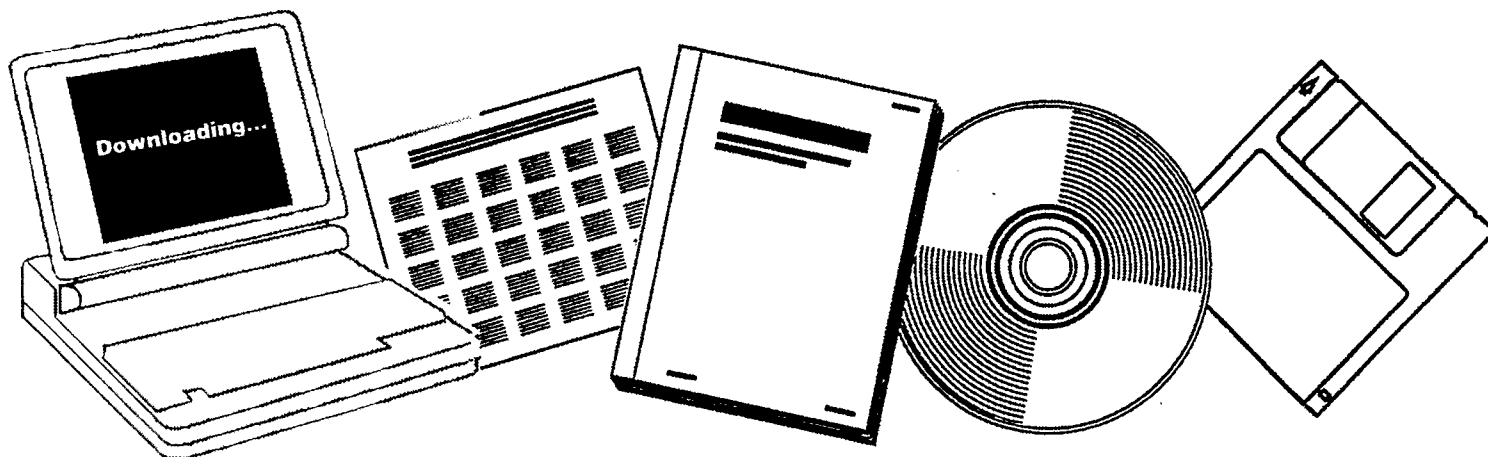
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**ALLOY CATALYSTS WITH MONOLITH SUPPORTS FOR
METHANATION OF COAL-DERIVED GASES.
QUARTERLY TECHNICAL PROGRESS REPORT,
SEPTEMBER 21-DECEMBER 20, 1979**

BRIGHAM YOUNG UNIV.
PROVO, UT

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ALLOY CATALYSTS WITH MONOLITH SUPPORTS FOR
METHANATION OF COAL-DERIVED GASES

Quarterly Technical Progress Report
For Period September 21, 1979 to December 20, 1979

Calvin H. Bartholomew
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Date Prepared -- January 5, 1980

PREPARED FOR THE UNITED STATES
DEPARTMENT OF ENERGY

Under Contract No. EF-77-S-01-2729

FOREWORD

This report summarizes technical progress during the ninth quarter (September 21, 1979 to December 20, 1979) of a two and one-half year study conducted for the Department of Energy (DOE) under Contract No. EF-77-S-01-2729. The principal investigator for this work was Dr. Calvin H. Bartholomew; Mr. Henry Pennline was the technical representative for DOE.

The following students contributed to the technical accomplishments and to this report: Erek Erekson, Ed Sughrue, Gordon Weatherbee, Don Mustard, Paul Moote, Clair James and Richard Bowman. Mr. Erekson and Dr. Bartholomew were the principal authors. Karen Barrett and Oscar Delgado provided typing and drafting services.

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ABSTRACT

Tests for catalyst deactivation by carbon deposition in a Berty reactor showed that CO partial pressure exerted a greater influence on deactivation than H₂ partial pressure. In kinetic studies in which H₂O vapor was added to the reactant gases, H₂O was found to inhibit the methanation reaction. H₂O inhibition was found to increase with temperature and loss of activity was observed at H₂O/CO ratios greater than one. The order of methanation with respect to H₂ and CO varies over the range of temperature from 498 to 598 K. Rate data indicate a change in mechanism or rate determining step at higher temperatures. Our experience with a quartz CFSTR has shown that this reactor is very delicate and needs near constant attention to maintain proper working order.

I. OBJECTIVES AND SCOPE

A. Background

Natural gas is a highly desirable fuel because of its high heating value and nonpolluting combustion products. In view of the expanding demand for and depletion of domestic supplies of clean fuels, economic production of synthetic natural gas (SNG) from coal ranks high on the list of national priorities.

Presently there are several gasification processes under development directed toward the production of SNG. Although catalytic methanation of coal synthesis gas is an important cost item in each process, basic technological and design principles for this step are not well advanced. Extensive research and development are needed before the process can realize economical, reliable operation. Specifically, there appear to be important economical advantages in the development of more efficient, stable catalysts.

From the literature (1,2), three major catalyst problems are apparent which relate to stability: (i) sulfur poisoning, (ii) carbon deposition with associated plugging, and (iii) sintering. Our understanding of these problems is at best sorely inadequate, and the need to develop new and better catalyst technology is obvious. Nevertheless, there has been very little research dealing with new catalyst concepts such as bimetallic (alloy) or monolithic-supported catalysts for methanation. This study deals specifically with sulfur poisoning, carbon deposition, and the effects of support (monolith and pellet) geometry on the performance of alloy methanation catalysts.

B. Objectives

The general objectives of this research program are (i) to study the kinetics of methanation for a few selected catalysts tested during the first two years, (ii) to investigate these catalysts for resistance to deactivation due to sulfur poisoning and thermal degradation. The work is divided into five tasks.

Task 1. Characterize the surface, bulk and phase compositions, surface areas, and metal crystallite sizes for alumina-supported Ni, Ni-Co, Ni-MoO₃, Ni-Pt, Ni-Ru and Ru catalysts.

Task 2. Continue activity testing and support geometry studies of Ni and Ni-bimetallic catalysts initiated during the first two years. The tests include (i) conversion vs. temperature runs at low and high pressures, (ii) steady-state carbon deposition tests, (iii) in situ H₂S tolerance tests, and (iv) support geometry comparisons.

Task 3. Perform kinetic studies to find intrinsic rate data for alumina-supported Ni, Ni-Co, Ni-MoO₃, Ni-Pt, Ni-Ru, and Ru catalysts over a range of pressures and feed compositions. Detailed rate expressions for each catalyst will be determined at low and high pressure.

Effectiveness factors for monolithic and pellet-supported nickel on alumina will be obtained by comparing specific rates to those of finely powdered nickel on alumina.

Task 4. Determine H₂S poisoning rates, thermal deactivation rates, and operating temperature limits for Ni, Ni-Co, Ni-MoO₃, Ni-Pt, Ni-Ru, and Ru catalysts.

Task 5. Continue laboratory visits and technical communications. Interact closely with industrial and governmental representatives to promote large scale testing and development of the two or three best monolithic or pelleted alloy catalysts from this study.

C. Technical Approach

The technical approach which will be used to accomplish the tasks outlined above is presented in the statement of work dated May 20, 1977. The main features of that approach are reviewed here along with more specific details and modifications which have evolved as a result of progress. It is expected that various other aspects of this approach will be modified and improved as the project develops and as new data are made available. Nevertheless, the objectives, tasks and principle features of the approach will remain the substantially the same.

Task 1: Catalyst Characterization

A comprehensive examination of alumina-supported Ni, Ni-Co, Ni-MoO₃, Ni-Pt, Ni-Ru, and Ru catalysts will be carried out to determine surface, bulk, and phase compositions, surface areas, and metal crystallite sizes using the following techniques: chemisorption, x-ray diffraction, chemical analysis, ESCA and SIMS spectroscopy, Auger spectroscopy and transmission electron microscopy.

Hydrogen chemisorption uptakes will be measured using a conventional volumetric apparatus before each reactor test and before and after deactivation tests. X-ray diffraction measurements will be carried out to determine the active metallic phases and metal crystallite size where possible. Selected "aged" samples from Task 4 will be analyzed (by x-ray, chemical analysis, and perhaps ESCA) to determine carbon content and possible changes in phase composition or particle size. Also, transmission electron micrographs will be made to determine particle size distributions for catalyst samples. A few samples will be analyzed by EDAX to determine composition.

Task 2: Activity Testing and Support Geometry Design

Methanation activity and sulfur tolerance measurements initiated during the previous two years of study (3) will be completed. Pellet and monolithic alumina-supported Ni, Ni-Co, Ni-MoO₃, Ni-Pt, Ni-Ru, and Ru catalysts, (both high and low metal loadings) will be activity

tested over a range of temperatures, pressures, and H₂S concentrations. A comparison of steady state conversions for nickel on different pellet and monolith supports of varying geometry will be made. Low pressure activity and sulfur tolerance tests will also be made for pelleted Co/Al₂O₃ and unsupported Ni-Co and Ni-Mo alloys. A summary of the five test procedures and corresponding experimental conditions is listed in Table 1.

Task 3: Kinetic Studies

In order to make more extensive kinetic studies of the six catalyst metal combinations a new mixed flow reactor system will be constructed. This system will be capable of operation to 7500 kPa and 775 K and over a range of reactant compositions. The reactor for this system will be a "Berty" type constant volume mixed flow Autoclave reactor.

Intrinsic rate data will be obtained for alumina-supported Ni, Ni-Co, Ni-MoO₃, Ni-Pt, Ni-Ru, and Ru catalysts over a range of pressures and feed compositions in order to obtain detailed rate expressions at low and high pressures. To insure gradientless operation in the reaction-limited regime the rates will be measured at low conversions (0-5%) and low temperatures (525-600 K) for samples which have been crushed to obtain small particles.

Isothermal effectiveness factors for monolithic and pellet-supported nickel on alumina will be obtained by comparing their specific rates to those of finely powdered nickel on alumina using the same mixed flow reactor.

Task 4: Degradation Studies

H₂S poisoning rates and thermal deactivation rates at low pressure will be studied using a new quartz reactor system. Quartz was selected as the material for the reactor because it must operate at high temperatures (750-1000 K) and in a corrosive (H₂S) environment. This reactor is also a constant volume mixed flow type reactor according to the design of Katzer (4). The quartz reactor system will be constructed during the early part of the contract period. Thermal deactivation at high pressures will be studied using a tubular stainless steel reactor previously discussed (3).

Operating temperature limits (and specific reaction rates within this range), thermal deactivation rates near the upper use temperature (in the presence and absence of steam), and H₂S poisoning rates (at 525 K in the presence of 1 and 10 ppm H₂S in H₂) will be determined for Ni, Ni-Co, Ni-MoO₃, Ni-Pt, Ni-Ru, and Ru catalysts. The extent of carbon-carbide deposited in the thermal deactivation runs will be determined by chemical analysis and x-ray diffraction.

Table 1

Description of Reactor Tests for Task 2

<u>Test Procedures</u>	<u>Experimental Conditions</u>
1. <u>Temperature-Conversion Test</u> : Measure CO conversion and methane production as a function of temperature, with and without 1% (by vol.) of steam present in the reactant mixture.	475-675 K 140 kPa 30,000 hr ⁻¹ 1% CO, 4% H ₂ , 95% N ₂ (dry basis)
2. <u>Temperature-Conversion Test (high pressure)</u> : Measure CO conversion and methane production as a function of temperature at 2500 kPa.	475-675 K 2500 kPa 30,000 hr ⁻¹ 1% CO, 4% H ₂ , 95% N ₂
3. <u>Steady State (24 Hr.) Carbon Deposition Test</u> : Measure CO conversion and methane production at 500 and 525 K (250,000 hr ⁻¹) before and after an exposure of 24 hours at 675 K.	675 K (24 hrs.) 140 kPa 200,000-250,000 hr ⁻¹ 25% CO, 50% H ₂ , 25% N ₂
4. <u>In situ H₂S Tolerance Test</u> : Measure intermittently the production of methane and hydrocarbons (by FID) during 24 hours exposure to feed containing 1 or 10 ppm H ₂ S using a glass reactor.	525 K 140 kPa 30,000 hr ⁻¹ 1% CO, 4% H ₂ , 95% N ₂ 1 or 10 ppm H ₂ S
5. <u>Support Geometry Tests</u> : Measure CO conversion and methane production as a function of temperature for the same Ni/Al ₂ O ₃ catalyst supported on monoliths and pellets of varying geometries.	575-675 K 140 kPa 30,000 hr ⁻¹ 1% CO, 4% H ₂ , 95% N ₂

Task 5: Technical Interaction and Technology Transfer

The principal investigator will continue to communicate closely with other workers in methanation catalysis, continue distribution of quarterly reports to selected laboratories to stimulate interest and feedback, attend important coal and catalysis meetings, and visit other methanation laboratories.

He will also interact closely with Mr. A.L. Lee at the Institute of Gas Technology, with personnel at the Pittsburgh Energy Research Center and with other coal gasification representatives to promote large scale testing and development of the two or three best catalysts from this study.

II. SUMMARY OF PROGRESS

A project progress summary is presented in Figure 1 and accomplishments during the past quarter are summarized below. Figure 1 shows that task accomplishments are generally on schedule.

Accomplishments and results from the past quarter are best summarized according to task:

Task 1. Twenty-four low-loading monolithic catalysts were prepared this quarter for kinetic and degradation studies in Tasks 3 and 4. Hydrogen chemisorptive uptakes were made on several monolithic catalysts. Work continued on the stoichiometry of CO chemisorption on nickel catalysts.

Task 2. No specific progress was made in this task during the past quarter. This task is essentially completed.

Task 3. The Bertly reactor system was modified to deliver water vapor into the feed gas. Several tests were made to find the inhibiting effects of H₂O on methanation. Qualitative results were found. H₂O inhibits methanation increasingly with temperature and at high H₂O partial pressures the catalyst is deactivated.

Task 4. Four carbon deactivation tests were made on the Bertly reactor system. The CO partial pressure appears to have a stronger influence on deactivation than H₂ partial pressure. The quartz CFSTR system had air leaks and had to be revamped. Also, a graphite bearing was replaced in the impeller. These problems have caused us to fall behind schedule in our sulfur poisoning studies.

Task 5. The principal investigator presented a paper at the Alex Oblad Symposium at Snowbird, Utah, visited the Pittsburgh Energy Technology Center where he presented a seminar and prepared and submitted 6 journal articles.

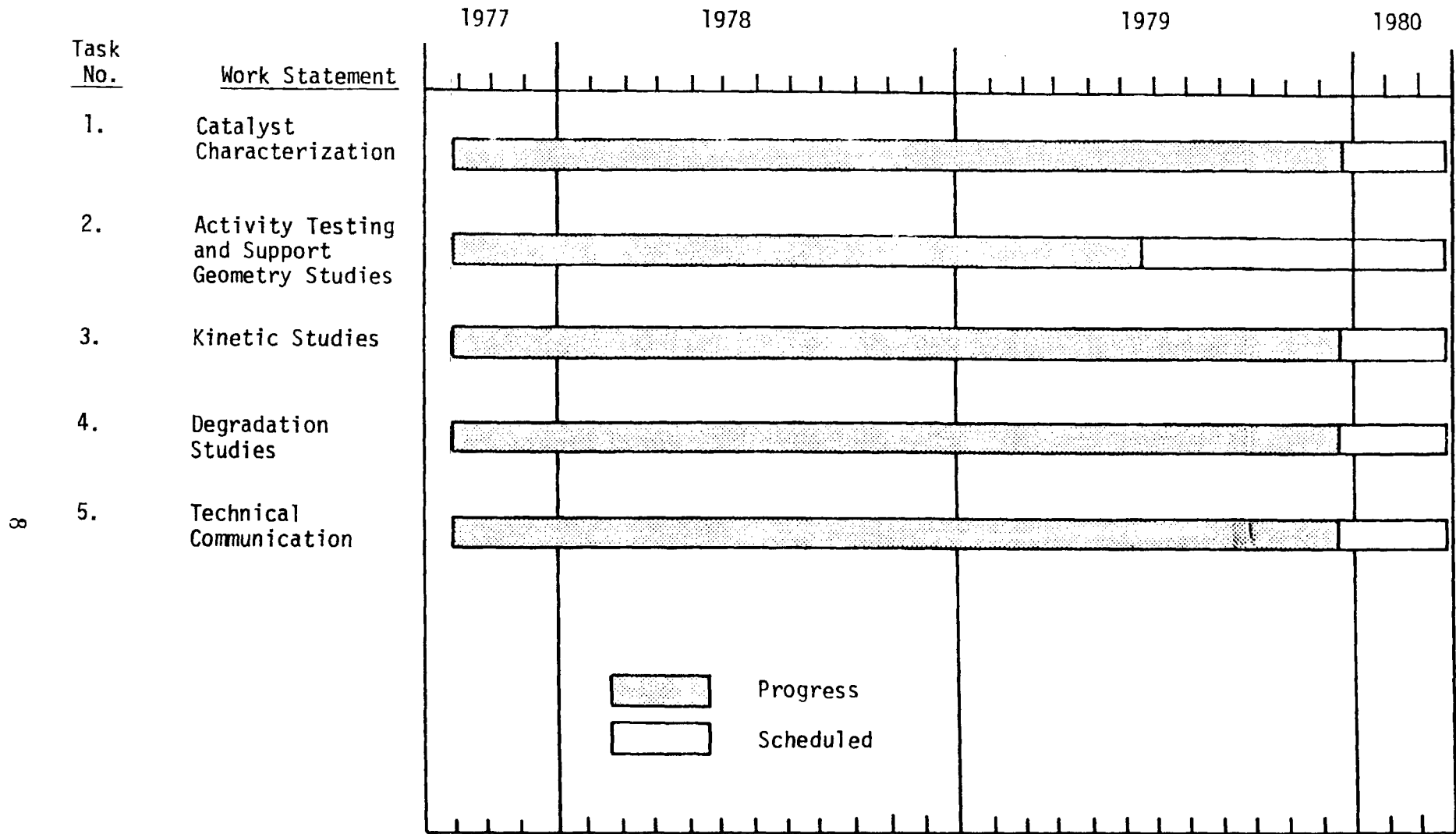


Figure 1. Project Progress Summary

III. DETAILED DESCRIPTION OF TECHNICAL PROGRESS

Task 1: Catalyst Characterization

1. Catalyst Preparation

Twenty-four low-loading nickel monolithic catalysts were prepared during this quarter for kinetic and deactivation studies in Tasks 3 and 4: Ni-M-500 to 504 and Ni-M-512 to 523 comprising 0.1% nickel and 2% alumina on Corning cordierite supports and Ni-M 531 to Ni-M 537 containing 0.5% nickel and 10% alumina on Corning cordierite. For all these monoliths the ratio of Ni to Al_2O_3 was maintained at 1/19 corresponding to a 5% Ni on Al_2O_3 loading. Strong support interactions occur at Ni loadings below 3% on alumina (5). The 0.1% nickel catalysts are used in tests above 573 K while the 0.5% nickel catalysts are for tests below 573 K.

2. Hydrogen Chemisorptions

Hydrogen chemisorptive uptakes obtained for several monolithic and pellet catalysts are listed in Table 2.

3. CO Chemisorption

Work on the stoichiometry of CO adsorption at various temperatures and before and after poisoning progressed slowly. H_2 and CO uptakes were repeated for 0.5 and 1.0% Ni/ Al_2O_3 catalysts (see Table 2), the resultant large values of CO/H suggesting suppression of H_2 adsorption and subcarbonyl CO adsorption as a result of strong metal support interaction. These results were incorporated in a paper submitted to the Journal of Catalysis.

The newly developed chromatographic sulfur detection method was found to give erratic results possibly because of contamination or aging of the reagents used in the test. Accordingly, new solutions were prepared and tests will be made during the coming quarter to determine the sulfur content in nickel samples from the CO stoichiometry tests.

Task 2: Activity Testing

No specific progress was made under this task during the past quarter. However, plans have been made to finish activity tests of monolithic nickel catalysts of different geometries during the next quarter.

Task 3: Kinetic Studies

Kinetic studies were continued during this quarter after some modifications were made in the Berty reactor system. A Milton-Roy minipump and a preheater were added to the reactor system in order

Table 2
Hydrogen Chemisorption Data

<u>Catalyst</u>	<u>Nominal Composition</u>		<u>H₂ Uptake (μmoles/g)</u>	<u>CO Uptake (μmoles/g)</u>	<u>CO/H</u>
	<u>% Al₂O₃</u>	<u>% Ni</u>			
<u>Monolithic Catalysts</u>					
Ni-M-193	2	0.1	2.0		
Ni-M-501	2	0.1	0.7		
Ni-M-503	2	0.1	1.2		
Ni-M-504	2	0.1	0.4		
Ni-M-506	10	0.5	8.8		
Ni-M-508	10	0.5	8.1		
Ni-M-513	2	0.1	3.1		
Ni-M-515	2	0.1	4.4		
Ni-M-516	2	0.1	3.2		
<u>Pellet Catalysts</u>					
Ni-A-124	99+	0.5	0.8 ^a	45	28
Ni-A-125	99	1.0	4.6 ^a	91	9.9

^aValue is average of several samples. Precision was +10%.

to introduce steam into the reactor feed gas. A blank test was made without catalyst to calibrate the pump under reaction conditions and to check for any significant gas-phase or reactor wall reactions. At 598 K no significant reactions occurred in the absence of H₂O. However, when steam was added in concentrations greater than twice the CO concentration, CO₂ began to appear and increased to approximately 0.2% at 20% H₂O. The dry feed gas was 96% N₂, 3% H₂ and 1% CO.

Two tests were subsequently performed with H₂O in the feed. However, in order to maintain 1% or less H₂O in the feed, it was necessary to operate the pump at approximately 0.01 ml/min H₂O. This extremely low flow rate was found to lie below the stable operating range of the minipump and the H₂O delivery system produced unsteady H₂O flows which resulted in the catalyst occasionally seeing high H₂O concentrations. Thus, the results collected in these tests are qualitative at best. Another water vapor delivery system has been designed and will be installed in February. These two tests did indicate that H₂O slows down or inhibits methanation and that the inhibiting effect is stronger at higher temperatures. At H₂O/CO ratios greater than one, the catalyst deactivated. Removal of H₂O from the feed did not completely restore initial catalyst activity.

Several tests were also made to check kinetic data obtained last quarter in tests at 498, 548, 573, and 598 K. These kinetic tests were performed at 690 kPa with a feed gas composed of approximately 96% N₂, 3% H₂, and 1% CO. Both H₂ and CO partial pressures were varied to determine their reaction order for the power law rate model, $r_{CH_4} = k H_2^x CO^y$. The results of these tests are compared with values determined by other investigators in Figures 2 and 3. Figure 2 is the order of reaction with respect to CO versus temperature while Figure 3 is the reaction order of H₂ versus temperature. Both figures provide indication of a mechanism shift taking place at higher temperatures. Figure 2 also shows very well the effects of competitive adsorption.

Task 4: Degradation Studies

Four tests were made at 598 K and 3450 kPa in the Berty reactor. Carbon deactivation kinetics were studied in these tests. Figures 4 and 5 report the results. The carbon monoxide partial pressure appears to be much more important than the hydrogen partial pressure in controlling the deactivation rate. The carbon deactivation will be continued next quarter.

During the last quarter H₂S mixing tests in the quartz stirred tank reactor showed that trace impurities from the reactant gases or the quartz reactor walls were reacting with the H₂S. We found that the teflon tubes, valves and fittings used to deliver the reactants to the reactor allowed oxygen from the air to diffuse into the reactant gas stream. The O₂ even at low levels (1 to 10 ppm) reacted with H₂S to form SO₂ in the reactor at temperatures above 500 K. The oxygen leaks were eventually eliminated by using glass and polyethylene tubing and leaving out the teflon valves for the reactant gas streams. The reactor was thoroughly cleaned twice and it is no longer suspected

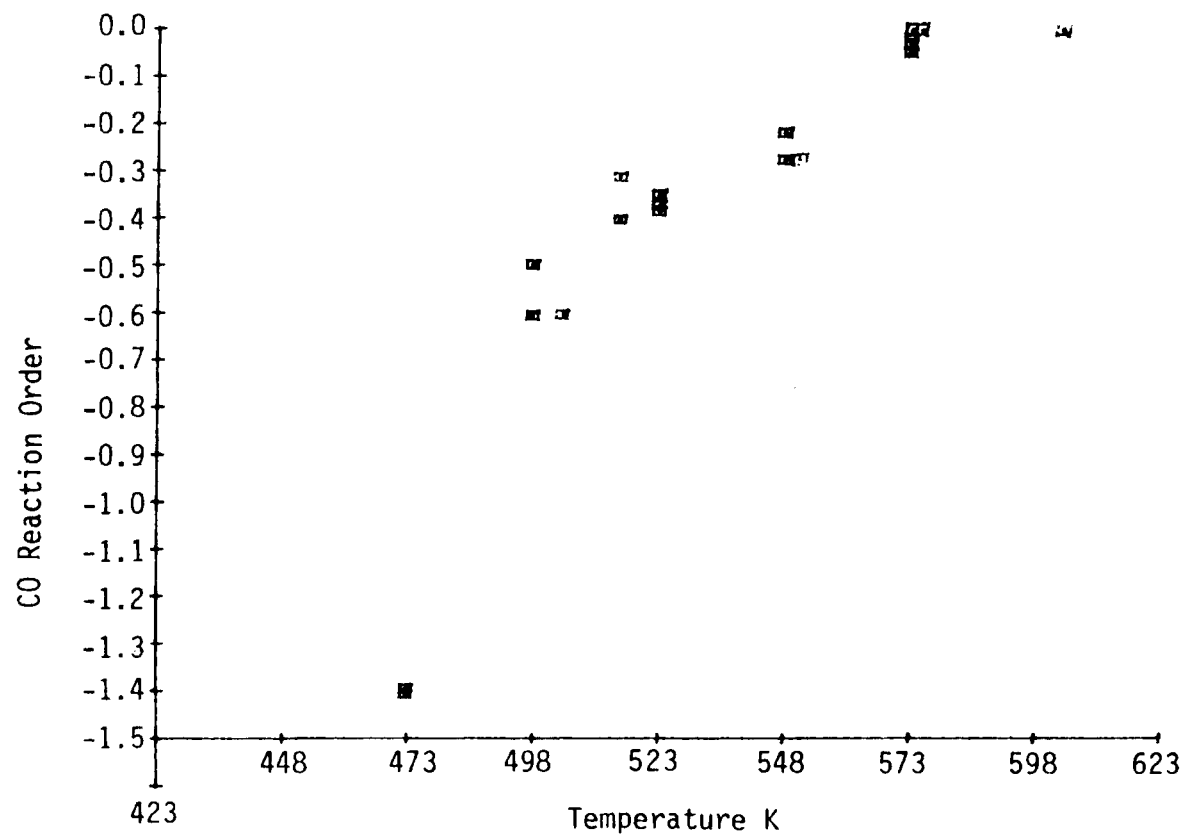


Figure 2. Methanation Reaction Order of CO vs. Temperature.

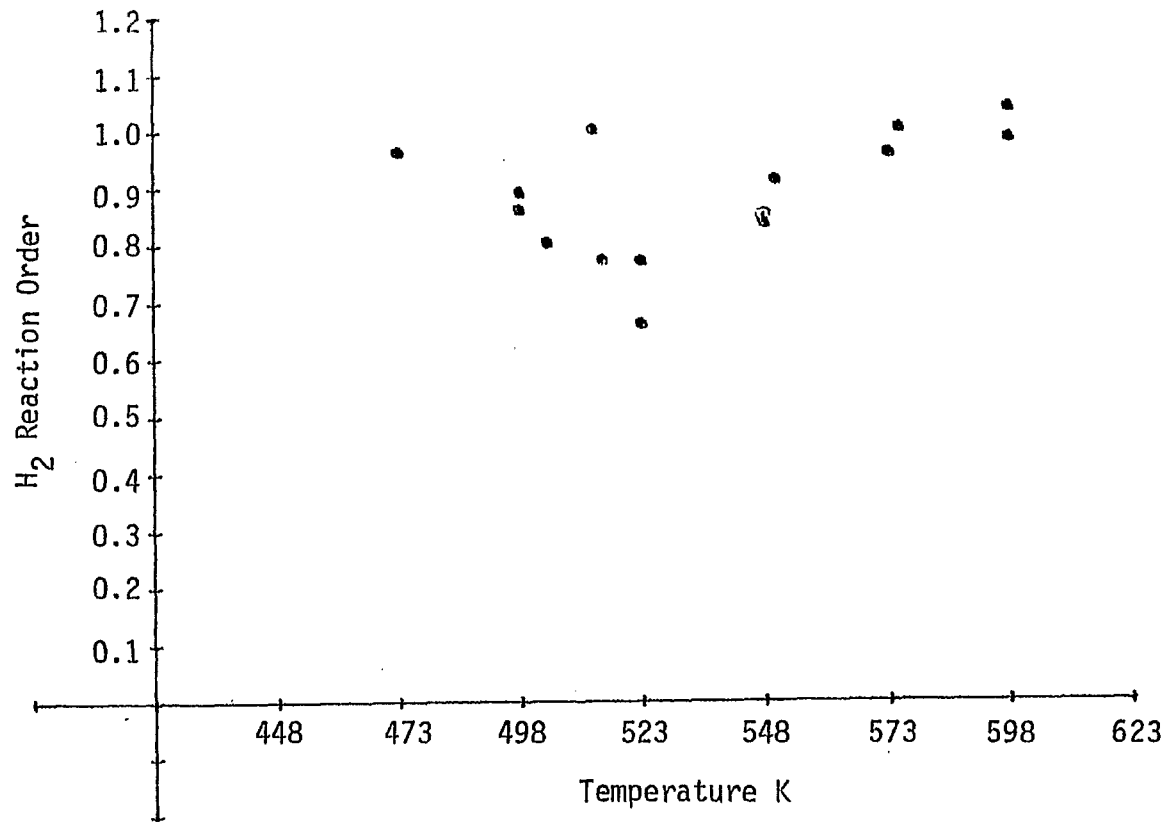


Figure 3. Methanation Reaction Order of H₂ vs. Temperature.

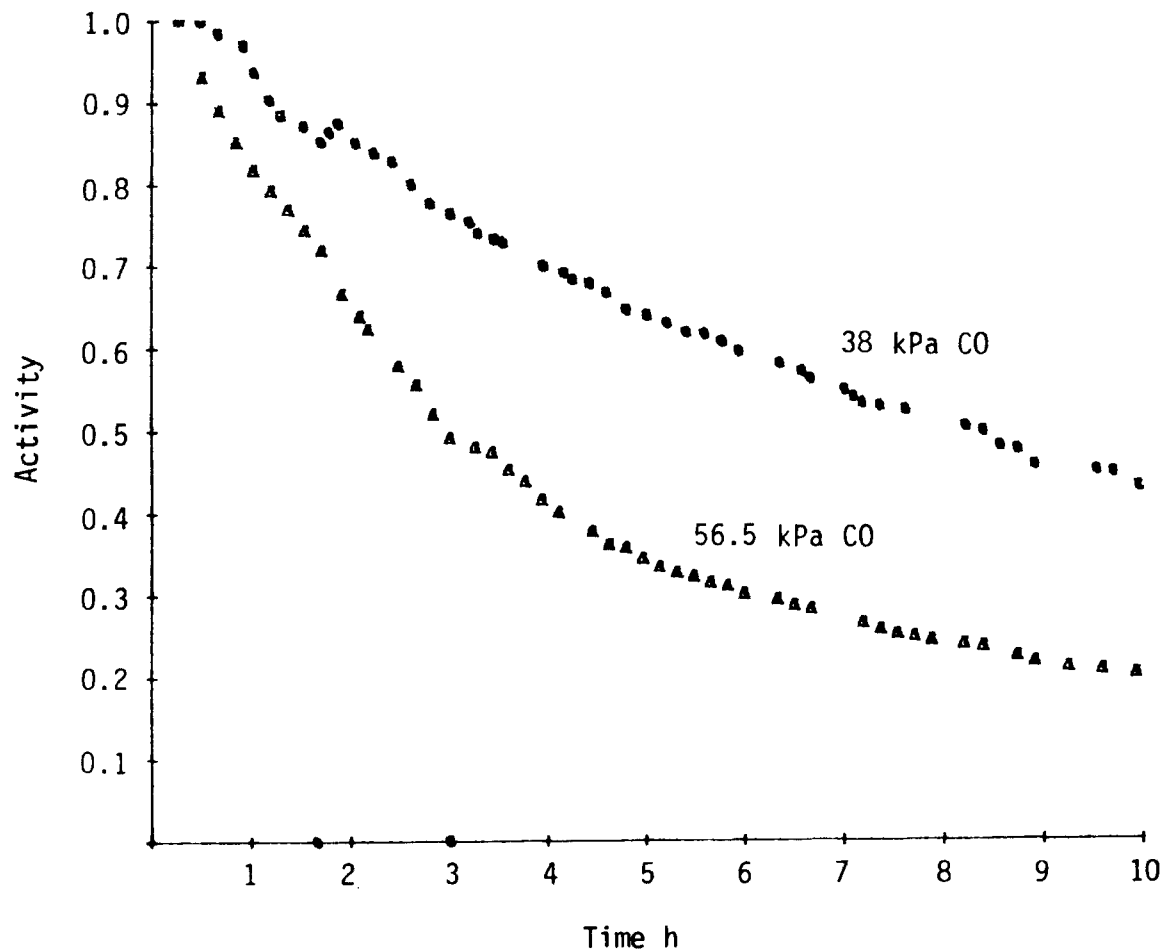


Figure 4. Dimensionless Activity versus Time at 598 K and 3450 kPa Total Pressure, comparing CO partial pressure. Dimensionless activity is defined as rate at any time divided by initial methanation rate per gram catalyst.

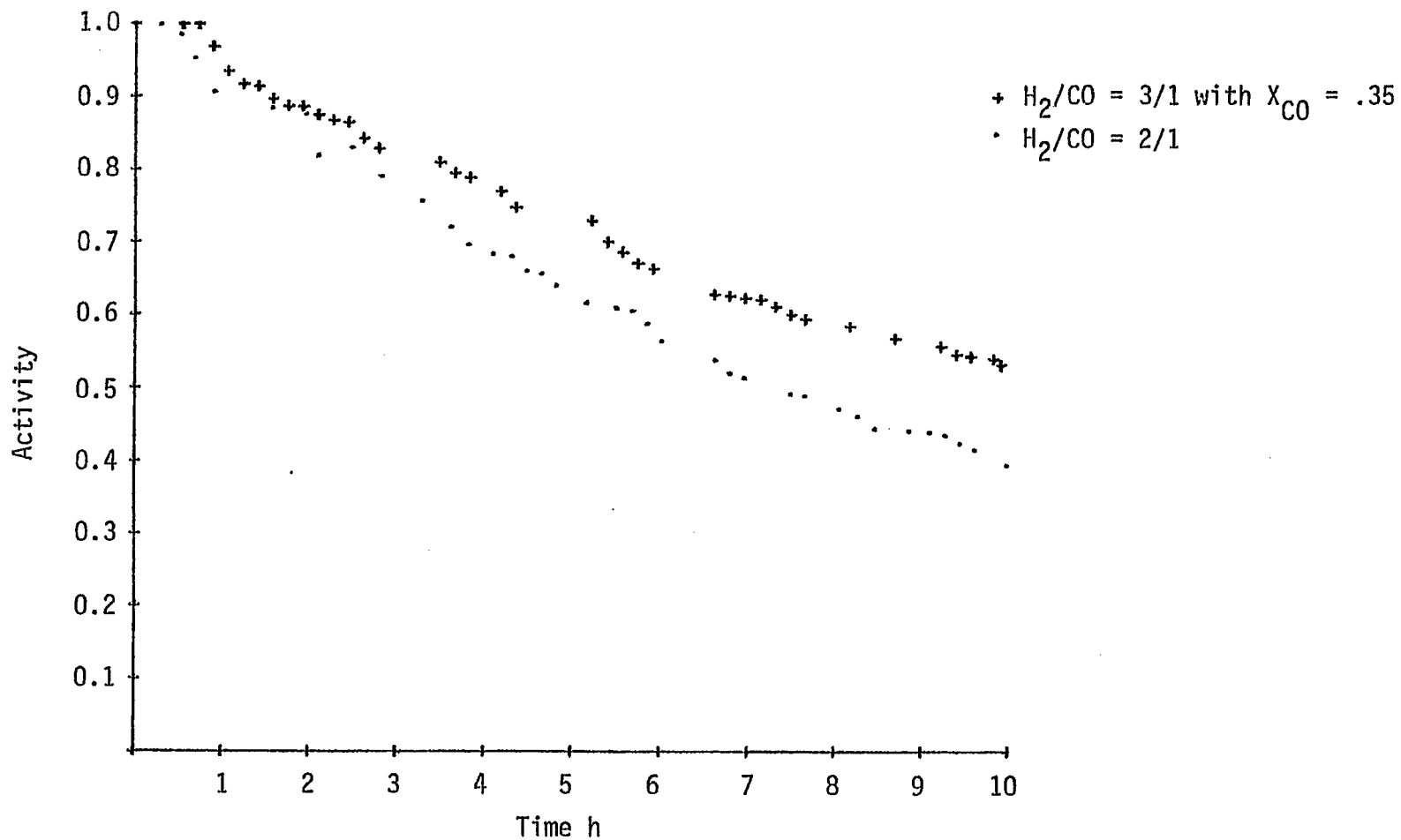


Figure 5. Dimensionless Activity versus Time at 598 K and 3450 kPa Total Pressure, comparing H₂ to CO ratio. Dimensionless activity is defined as rate at any time divided by initial methanation rate per gram catalyst.

to be causing interference with H₂S or any other gases used in the degradation experiments.

Toward the end of the ninth quarter, one of the graphite bearings in the quartz impeller began to break up. The impeller has two graphite bearings which hold the impeller shaft in place - one on the top and one on the bottom. The bearings are cylindrical with a small groove cut in the face to guide the impeller shaft as it rotates. The impeller was sent to a glass shop in order to replace the worn out bearing. During the last week of the quarter the impeller was returned and tests were made of the spinning stability. The tension on the bearing was adjusted several times to improve the spinning of the impeller. From our experience with this quartz CFSTR, it is clear that the parts are very delicate and need near constant attention to maintain proper working order.

Unfortunately because of the difficult experimental problems with H₂S analysis and with smooth operation of the quartz CFSTR reactor, we have fallen behind schedule in our sulfur poisoning studies. However, since we have solved most of the difficult problems, we expect to be able to obtain a great deal of data during the next quarter. Nevertheless, we will probably not finish the experimental work by the end of this quarter and have therefore requested a no-cost-extension through the summer for this purpose.

Task 5: Technical Interaction and Technology Transfer

The principal investigator and four graduate students, Erik Erikson, Edward Sughrue, Jeffery Rankin and Gordon Weatherbee attended Advances in Catalysis I, a symposium held in honor of Professor Alex Oblad, October 3-5 at Snowbird, Utah. Dr. Bartholomew presented a paper on "Surface Intermediates in Methanation of CO." The meeting was well organized and well attended by the most noted and productive researchers in catalysis. It was thought by most participants to be one of the finest dealing with catalysis in years.

On October 31, Dr. Bartholomew enjoyed a stimulating and productive visit with research personnel in the Process Sciences and Engineering Divisions at the Pittsburgh Energy Technology Center. He toured research facilities concerned mainly with testing of Fischer-Tropsch catalysts and presented a seminar on "Metal Boride Catalysts in CO Hydrogenation." He was also able to obtain ideas for micro reactor design and zeolite supported iron catalysts for testing in our DOE sponsored study of FT catalysts.

On the following day, Dr. Bartholomew participated on an NSF proposal review panel in Washington, D.C.

During the past quarter, Dr. Bartholomew was engaged in several writing projects including the completion and submission of 6 different journal publications of which 4 were based on work performed as part of this contract. Two of the most significant of these publications deal with the stoichiometry of H₂ and CO on supported nickel and metal-support interactions in CO hydrogenation on nickel.

Donald Mustard completed work on his M.S. thesis dealing with metal particle size characterization by TEM of supported nickel and began work with AMOCO Oil Co. in Houston in early December.

IV. CONCLUSIONS

1. Tests showed that qualitatively H_2O inhibits the methanation reaction. This inhibition increases with temperature. Moreover, at H_2O/CO ratios greater than one, catalysts deactivated.
2. In connection with last quarter's work on the shift in activation energy, several kinetic tests were made to show the changes in reaction order for both CO and H_2 between 498 and 598 K. These data also indicate a change in mechanism or rate determining step over this temperature range.
3. Carbon deactivation tests in the Berty reactor showed that CO partial pressure had a much stronger influence on deactivation rates than H_2 partial pressure.
4. Experience with the Quartz continuous flow stirred tank reactor shows that this reactor is very delicate and needs near constant attention to maintain proper working order.

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