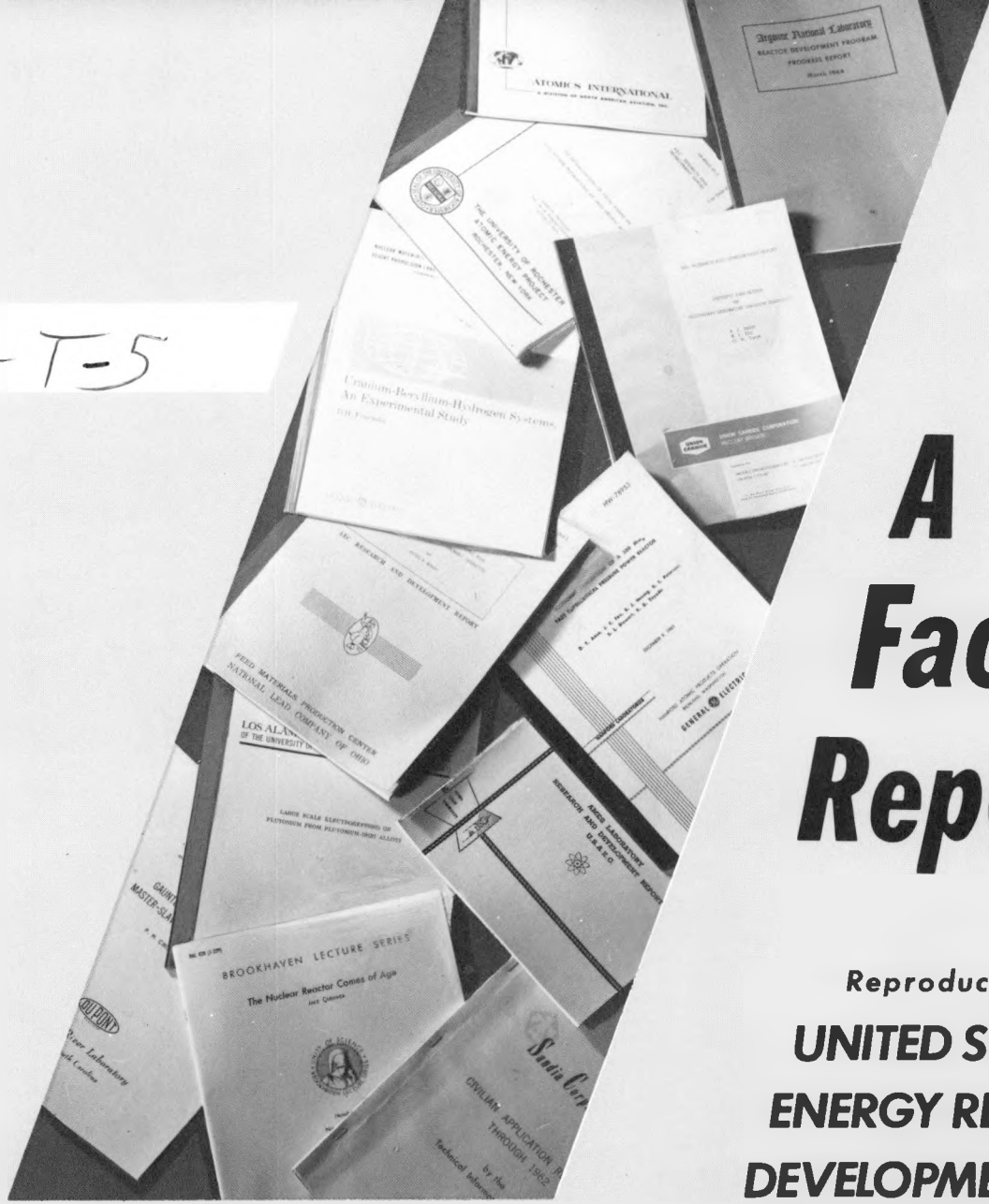


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FE--415-T5

**CONSOL LIGNITE GASIFICATION
PROCESS DEVELOPMENT**

Monthly Progress Reports for the
Period January - December 1968

Consolidation Coal Company
Library, Pennsylvania 15129

Prepared for
Office of Coal Research
U. S. Department of the Interior
OCR Contract No. 14-01-0001-415(2)

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FE--415-T5

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Monthly Progress Reports 44 through 55 covering each
month January 1 through December 31, 1968, respectively

CCDC-MPR--44

PROGRESS REPORT NO. 43
 OCR CONTRACT NO. 14-01-0001-415(2)
CONSOL. LIGNITE GASIFICATION PROCESS DEVELOPMENT

January 1 to 31, 1968

A. Summary

Phase I - Feasibility Study

Complete.

Phase II - Bench-Scale Experiments

Studies on operability limitations in the CO₂ acceptor process were extended to other sub-bituminous coals. Colstrip coal from Montana was run at the conditions with results summarized in the table below.

Run and Type	Gasifier		Regenerator	
	Temp.	Deposit Wt. % of Ash Fed	Temp.	Deposit Wt. % of Ash Fed
H-16				
Hydrodevolatilization of				
LTC Char				
D-4a	1525	None	Not operated	
D-4b	1600	0.4	1900	Very slight
D-4c	1500	None	1900	"
D-5	1500	None	1940	3%

It cannot be said that operation with Colstrip is completely operable. The ash deposition, however, was small, especially in view of the low fusion point of the ash in the feed.

Fundamental studies to discern the relationship between ash deposition and the ash composition of the feed coal are continuing.

Electron microprobe studies have shown that the Ca and Mg deposited from the coal ash on the outer shell of the acceptor are present largely as oxides.

Phase III - Pilot Plant Design

Engineering and review of final documents will be completed in February. Catalytic has requested an extension of their contract to March 1, 1968, and an increase in the contract budget to \$369,000.

Financial

Estimated expenditures in January were \$69,000. Cumulative expenses to date are \$1,510,485.

B. Technical Results and Program

1. Operation of the Continuous Gasification Unit (Project No. 532.00)

All operations during the month were concerned with Colstrip sub-bituminous coal.

a. Run 532-H-16, Hydrodevolatilization of LTC Colstrip Char

To insure a dense char, the Colstrip coal was precarbonized at 925°F in the same manner as described last month for the Rosebud coal. No deposits were found after 425 lb. had been fed at 1525°F hydrodevolatilizer conditions. Run conditions and results are shown in Table I. The product char density was high (80.0 lb/ft³).

b. Run 532-D-4, Operations with Colstrip Char at 1600°F Regenerator Temperature

During the first part of this run D-4(a), the gasifier was operated at 1600°F in the absence of acceptor to make a stock of regenerator fuel char. Run conditions and results are shown in Table II. After 96 lbs. of Run H-16 char had been fed, an iron-rich deposit amounting to 0.4% of the ash fed was found on the gasifier walls, in contrast with Run H-15 in which no deposit formed at the lower temperature of 1525°.

A peculiar behavior of the Colstrip char was revealed during Run D-4. During the early part of Run D-4(a), the Greenfield acceptor was circulated at 1500°F. On raising the gasifier bed temperature toward the desired 1600° level, the acceptor-char interface probe could no longer distinguish between char and acceptor at 1580°F. The acceptor was then drained from the vessel, and the run was continued to produce regenerator fuel char.

In Run D-4(b), the Tymochtee acceptor was used on the presumption that some impurity in the Greenfield acceptor had caused it to become electrically conducting at temperatures above 1580°. However, the same behavior was observed on heating the gasifier to 1600°F.

Since the purpose of the run was to determine the behavior of Colstrip char in the regenerator, no further effort was made to use this material in the gasifier. In Run D-4(c), the spent char from Run A-25 (Husky II) was used as gasifier feedstock. After ascertaining that the interface probe functioned normally at 1600°F with this material, the gasifier temperature was reduced to 1500°F for the remainder of the run.

Combustion of the Run D-4(a) char was carried out at 1900°F regenerator temperature for 31 cycles at which time the acceptor could no longer be withdrawn from the regenerator. After disassembly of the vessel, the bed was lightly agglomerated. A very slight deposit amounting to 0.0% of the ash fed was on the regenerator walls. The bed agglomeration apparently was caused by sintering of the outer shell on the acceptor. Run conditions and results are shown in Table III.

c. Run 532-K-2 Production of Regenerator Fuel Char

In this run, the gasifier was operated in the absence of acceptor to produce an additional stock of Colstrip regenerator fuel char. The run was similar to Run D-4(a) except that the inlet gas partial pressures were changed somewhat to obtain another gasification kinetics data point. Conditions and results are given in Table II. A total of 171 lbs. of Run H-16 char was fed.

d. Operation of Electrothermal Heating Plate

During Run K-2, the 1" x 8" heating plate was operated for 27 hours at a gasifier bed temperature of 1600°F. The heat flux was 40 watts/in², the same as the design condition for the Rapid City pilot plant. The plate, made from Type 310 SS, suffered no damage whatever.

e. Run 532-D-5, Operations with Colstrip Char at 1940°F Regenerator Temperature

Since the Colstrip char gave only a very slight deposit at 1900°F, this run was made at 1940° to determine the effect of temperature on ash deposition. The gasifier was operated at exactly the same conditions as in Run D-4(c).

Combustion of the Run K-2 char was carried out at 1940° regenerator temperature for 2 cycles, at which time the acceptor overflow weir plugged. The regenerator walls and internals were coated with a thin, uniform ash deposit amounting to 3.0% of the ash fed. The acceptor bed was not agglomerated, in contrast with Run D-4(c) at the lower temperature of 1900°. The weir was plugged by flakes of the deposit which had sloughed off the vessel walls. Run conditions and results are given in Table III.

The acceptor activities were the same as in Run D-4(c), showing that increasing the regenerator temperature to 1940° has no adverse effect. The activities were somewhat lower than at 1870°, however. The comparison is shown below.

Gasifier Conditions: Husky II Char at 1500°F
Acceptor: Tymochtee Dolomite

Run Regen. Temp., °F Cycle	D-4(c)	D-5	A-21
	1900	1940	1870
	Activity		
5	.80	.80	.90
10	.68	.68	.82
20	.55	.55	.65
30	.45	.45	.55
40	-	.39	.49

f. Program

All experimental work will be devoted to solving the regenerator ash deposit problem. The program is outlined in E-11678, Project No. 532.

2. Batch Kinetic Studies (Project No. 530.00)

a. Properties of Char Feedstocks and Products

Analytical data on all the raw coals and gasifier and regenerator feedstocks and products used to date are being compiled and will be presented next month.

b. Ash Studies

To aid in the understanding of the mechanism of ash deposition, two techniques have been applied to the raw, dried coal feedstocks. In the first, pristine ash from three samples of Glenharold lignite were obtained by low-temperature ashing (LTA) and various inspections were performed on the recovered ash. In the LTA process, the coal samples are oxidized by atomic oxygen at a temperature not exceeding 300°F. Literature references show that none of the common ash components are altered chemically or physically at LTA conditions, with the exception of partial or complete dehydration of gypsum (CaSO₄·2 H₂O). Results of the inspections made so far are shown in Table IV.

In the second technique, the dried, sized, raw Glenharold and Rosolud coals, as fed to the continuous unit, were separated into specific gravity fractions by float-sink. Results of the inspections are shown in Table V. Similar data for the Colstrip sub-bituminous coal are nearly complete and will be given next month.

Interpretation of the data in Tables IV and V will be deferred until they can be coordinated with results of X-ray diffraction studies, and with results of operation of the continuous unit regenerator in which the conditions will be varied in a systematic manner.

c. Carnegie-Mellon Studies

Electron beam microprobe studies of the Run A-26 regenerator deposit show that the major elements present (Ca, Mg, Si, Al, Fe, S) are distributed more diffusely than was expected from optical inspection. Only Fe showed any appreciable segregation. There was no evidence that any of the elements were concentrated as a grain-boundary material or cementing agent. Study of the X-ray micrographs and line scans is continuing.

Computerized processing of X-ray diffraction patterns of Husky I, Husky II and Glenharold char ashes, acceptor shells, and ash deposits has been completed at Penn State. A bewildering array of possible compounds in these materials confronts us. Interpretation will be time consuming.

After a rapid study of the results, the most significant finding is that the Ca and Mg (which chemically are the major components of the acceptor shells and regenerator ash deposits) are present mainly as oxides.* There is very little evidence that the Mg and Ca are combined with silica, alumina, or iron.

Phase III - Pilot Plant Engineering (Project No. 533.30)

The engineering work at Catalytic Construction was 95% complete as of January 31, 1968. The only remaining work included 33 piping drawings and 12 structural drawings. As of January 31, expenditures were \$359,000. Final cost is estimated to be \$369,000. An extension of contract time to March 1, 1968 is requested by Catalytic to permit review and approval of final drawings.

A meeting to carry out said review is scheduled for early February at the Catalytic offices.

Catalytic has provided a preliminary estimate of 8.8 MM \$ as the cost of completing the installation of the pilot plant.

C. Subject Inventions

None.

D. Financial Status

Expenses	Actual	Budget
Cumulative through December, 1967	\$ 1,441,495	\$ 1,415,000
Expended in Period	69,000	40,000
Cumulative through January, 1968	\$ 1,510,495	\$ 1,455,000

*In some cases, in conformity with the conditions in the continuous gasification unit, some of the calcium is present as CaS.

Att. - Monthly Progress Chart
2/14/68

TABLE I

Run Conditions and Results

Run No.	533-2-16
System Pressure, atm	20
Temp., °F	1525
Feedstock	LTC Colstrip Subbituminous Char
Feed Rate, lb/hr	7.69
Inlet Gas (Bottom of Bed)	
Steam, SCFH	80.5
Recycle, "	
Fluidizing Gas	197.4
Added N ₂ , SCFH	17.3
N ₂ , "	5.4
Composition, Partial Pressure, atm	
H ₂ O	5.36
H ₂	7.59
CH ₄	1.98
CO	2.10
CO ₂	0.97
N ₂	2.00
Outlet Gas (Top of Bed)	
Composition, Partial Pressure, atm	
H ₂ O	3.69
H ₂	8.20
CH ₄	2.52
CO	2.68
CO ₂	1.24
N ₂	1.76
Dry Exit Gas, SCFH	89.7
N ₂ Purges, "	5.8
Condensate, "	61.8
Recycle Gas Composition, mole %	
H ₂	48.99
CH ₄	15.08
CO	16.02
CO ₂	7.41
N ₂	12.50
Avg Fluidizing Velocity, ft/sec	.190
Avg Vapor Retention Time, sec	16.5
Yields	
Char, Wt % MF Feed	75.3
Carbon Bearing Gases, lb c/100 lb MF Feed	13.94
CH ₄	5.46
CO	5.80
CO ₂	2.68
Char Particle Density, lb/ft ³	80.0

TABLE II

Gasifier Run Conditions and Results

Run No.	532-D-4(a)	532-K-2
System Pressure, atm	20	20
Gasifier, Temp., °F	1600	1600
Char	Hydrodevolatilized LTC Colstrip	
Char Feed Rate, lb/hr	4.00	4.20
<u>Inlet Gas (Bottom of Bed)</u>		
Steam, SCFH	122.4	162.9
Recycle, "		
Fluidizing Gas	291.7	283.5
Char Carrier Gas (Middle of Bed)*	55.6	61.0
Added H ₂ , SCFH	14.7	21.0
CO ₂ , "	5.2	0
N ₂ , "	2.0	2.0
<u>Composition, Partial Pressure, atm</u>		
H ₂ O	5.61	6.94
H ₂	6.87	7.84
CH ₄	0.74	0.85
CO	2.83	2.55
CO ₂	1.52	1.07
N ₂	2.43	0.75
<u>Outlet Gas (Top of Bed)</u>		
<u>Composition, Partial Pressure, atm</u>		
H ₂ O	3.67	4.11
H ₂	7.81	9.20
CH ₄	0.93	1.12
CO	3.56	4.08
CO ₂	1.61	1.41
N ₂	2.41	0.78
Dry Exit Gas, SCFH	91.3	98.9
N ₂ Purges, "	14.0	3.5
Condensate, "	95.5	113.9
<u>Recycle Gas Composition, mole %</u>		
H ₂	46.30	57.47
CH ₄	5.51	7.00
CO	21.12	21.10
CO ₂	9.57	8.83
N ₂	17.5	5.6
Avg Fluidizing Velocity, ft/sec	.293	.313
Steam Conversion, %	22.0	30.1

*Contributes to outlet gas only.

TABLE III

Regenerator Run Conditions and Results

Run No.	532-D-4(c)	532-L-5
Duration, Cycles	31	42
Hour	21.3	25.9
Acceptor	Tymochtee-3 Dolomite	Tymochtee-5 Dolomite
Acceptor Circulation Rate, lb/hr (raw basis)	10.16	10.30
System Pressure, atm	20	20
<u>Regenerator</u>		
Temperature, °F	1900	1940
Fuel Char	Colstrip Subbituminous Char	Colstrip Subbituminous Char
	20% Burnoff (Run D-4)	30% Burnoff (Run K-2)
Fuel Char Fed, lb	18.1	23.5
Fuel Char Feed Rate, lb/hr	0.85	0.91
Atoms C burned/mole MgO·Ca	.915	.967
<u>Inlet Gas</u>		
Recycle, SCFH	250	250
Added N ₂ , "	35	35
CO ₂ , "	0	0
Air	94	90
<u>Recycle Gas Composition, Mole %</u>		
N ₂	Balance	Balance
CO ₂	20	20
O ₂	0	0
CO	0.5 - 0.7	0.5 - 0.7
Air in Inlet Gas, Mole %	24.2	24.0
CO ₂ Partial Pressure, atm	4.00	4.00
Equilibrium CO ₂ Partial Pressure, atm	5.90	7.60
ΔP Driving Force, atm	1.90	3.60
Fluidizing Velocity, ft/sec	1.11	1.15
Acceptor Activity	.66	.55

TABLE IV

Properties of Low-Temperature Ashed Samples

Source: Glenharold Lignite Cores

Core No. Ash Fusion, °F(1)	M607 Top			M606			189 Top		
	Start Contraction	First Liquid	Totally Melted	Start Contraction	First Liquid	Totally Melted	Start Contraction	First Liquid	Totally Melted
H ₂	1400	2070	2120(2)	1650	2260	-	1650	2040	2260(2)
N ₂	1480	2100	2140	1650	2250	2300	1960	2030	2210(2)
CO ₂	1540	2010	2080	2040	2160	2180(2)	1830	2010	2170
Components Present, by X-Ray Diffraction	Major	Minor		Major	Minor		Major	Minor	
	CaSO ₄	γ-CaSO ₄		CaSO ₄	γ-CaSO ₄		γ-CaSO ₄	CaSO ₄	
	α-SiO ₂	kaolinite		α-SiO ₂	kaolinite		α-SiO ₂	kaolinite	
	illite								
Ash Composition, Wt. %									
Al ₂ O ₃		13.7			10.4			9.3	
SiO ₂		43.6			17.8			64.4	
Fe ₂ O ₃		6.9			15.0			14.5	
CaO		18.5			34.2			13.5	
MgO		5.4			7.1			3.8	
Na ₂ O		7.2			13.2			3.6	
K ₂ O		1.8			0.75			0.74	
TiO ₂		2.1			1.5			0.69	
P ₂ O ₅		0.04			0.09			0.06	

(1) Measured in hot wire microscope, purged with gas as listed.

(2) Gas evolution.

<u>kaolinite</u>	Al ₂ Si ₂ O ₅ (OH) ₄ , [Al ₂ O ₃ .2 SiO ₂ .3 H ₂ O]
<u>illite</u>	NaAl ₂ (AlSi ₃ O ₁₀)(OH) ₂ , [Na ₂ O.3 Al ₂ O ₃ .6 SiO ₂ .3 H ₂ O]
<u>γ-CaSO₄</u>	Calcium sulfate derived from the dehydration of gypsum, CaSO ₄ .3 H ₂ O.

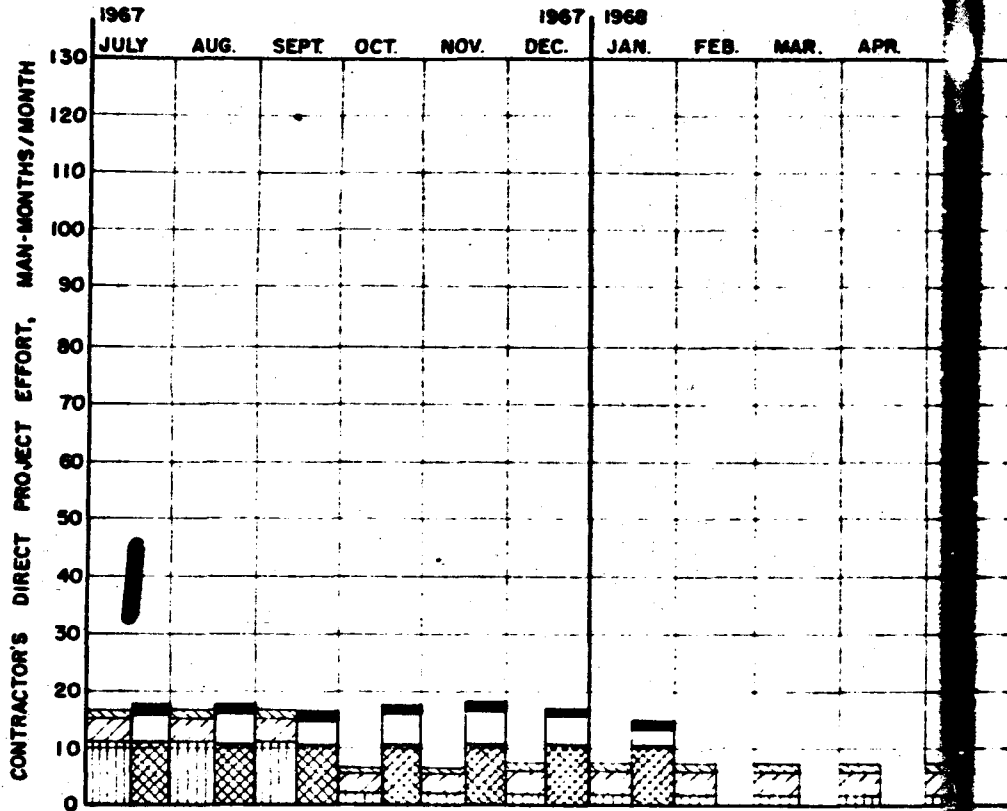
Progress Report No. 44
2/14/68

TABLE V

Inspections on Float-Sink Fractions

Specific Gravity	Glenharold(1)								Rosebud(2)						
	1.25 x 1.36	1.36 x 1.50			1.50 x 1.70			> 1.70	1.25 x 1.36	1.36 x 1.50		1.50 x 1.70			
Size, Tyler Mesh	-28	28x35	35x48	48x35	65x100	-100	-28	-28	28x35	35x48	-48	28x48	-48	-28	
% Ash, Dry Basis	6.79	5.71	5.99	6.03	6.17	6.67	11.49	56.75	2.55	2.57	2.48	4.36	4.77	34.00	
Al ₂ O ₃	10.4	11.0	10.4	9.6	10.0	6.7	12.0	9.9	24.3	24.0	24.3	26.8	26.4	20.4	
SiO ₂	25.1	15.8	15.1	15.2	16.4	17.5	31.5	37.7	16.3	14.7	13.8	36.2	36.6	45.3	
Fe ₂ O ₃	10.3	7.6	7.4	7.2	7.3	8.4	9.4	41.7	5.4	5.7	6.2	4.3	5.3	11.0	
CaO	31.5	41.3	43.0	42.7	40.7	39.8	28.6	3.9	34.6	35.1	33.9	19.5	20.2	4.0	
MgO	6.6	8.8	9.3	9.3	8.9	9.9	6.4	1.3	10.7	11.1	10.4	6.1	6.3	1.6	
Na ₂ O	7.2	12.1	11.8	12.3	11.9	10.6	7.1	0.78	0.72	0.61	0.27	1.2	1.3	0.40	
K ₂ O	1.3	1.7	1.5	1.5	1.2	1.0	1.3	1.3	0.29	0.17	0.23	0.63	0.61	0.15	
TiO ₂	0.06	0.49	0.45	0.46	0.53	0.46	0.47	0.16	2.2	2.3	2.7	2.1	2.2	1.1	
P ₂ O ₅	0.74	0.32	0.26	0.35	0.36	0.12	0.32	0.50	0.45	0.66	0.69	0.23	0.41	0.33	
Wt. % of Gravity Fraction	100	9	38	28	15	10	100	100	25	31	44	56	44	100	
		Weight % of Total Coal													
								(1)						(2)	
								1.25 x 1.36	3					72	
								1.36 x 1.50	88					23	
								1.50 x 1.70	5					5	
								> 1.70	4					0	

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MONTHLY EXPENDITURES*

	JULY	AUG.	SEPT.	OCT.	NOV.	DEC.	JAN.	FEB.	MAR.	APR.	MAY
PREDICTED	103.7 M	72.2 M	92.0 M	94.0 M	94.0 M	52.0 M	40.0 M	20.0 M	70.0 M	70.0 M	0 M
ACTUAL	75.5 M	78.0 M	106.6 M	84.5 M	97.1 M	93.4 M	69.0 M				

	JULY	AUG.	SEPT.	OCT.	NOV.	DEC.	JAN.	FEB.	MAR.	APR.	MAY
PREDICTED	1010.8 M	1083.0 M	1175.0 M	1269.0 M	1363.0 M	1415.0 M	1455.0 M	1475.0 M	1545.0 M	1615.0 M	1615.0 M
ACTUAL	981.3 M	1059.3 M	1166.1 M	1251.0 M	1348.1 M	1441.5 M	1510.5 M				

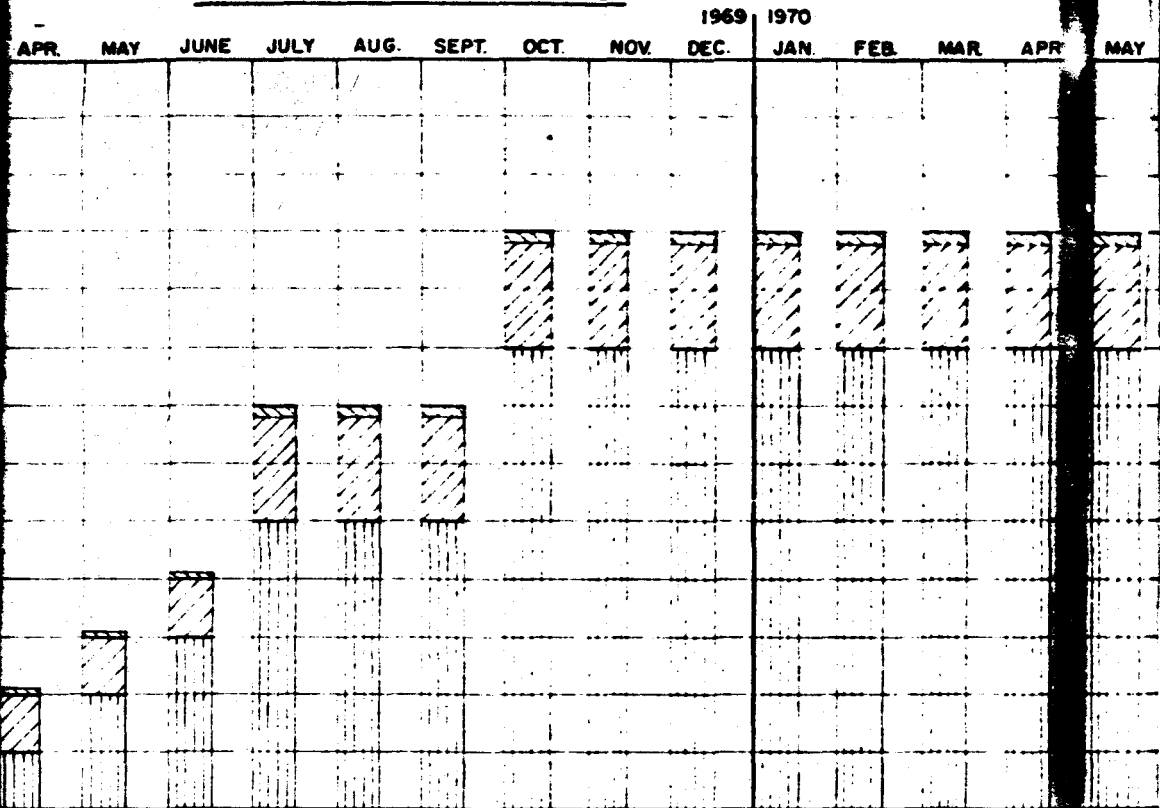
CUMULATIVE EXPENDITURES*

* Expenditures Include All Costs & Fees.

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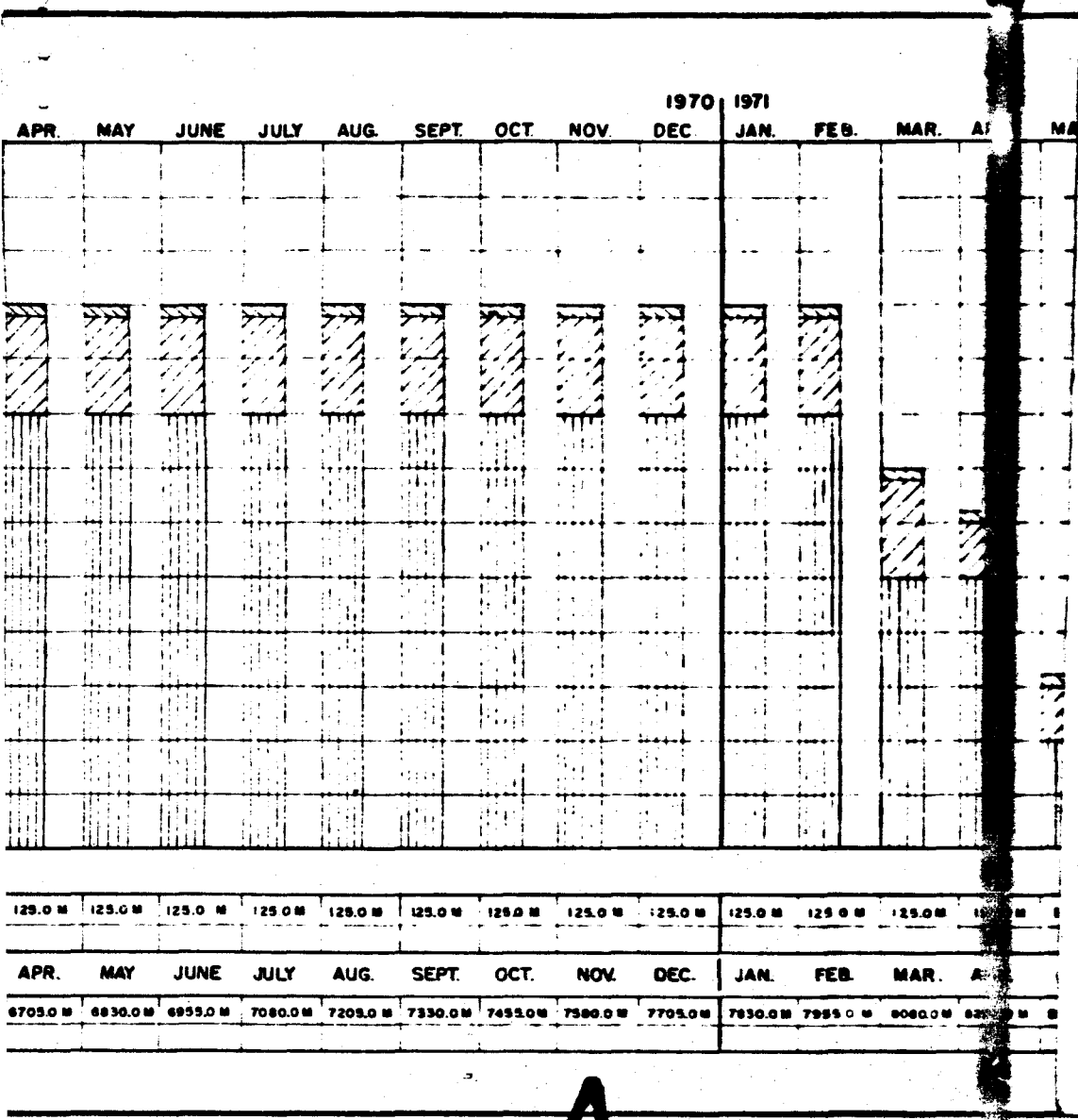
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APR. MAY JUNE JULY AUG. SEPT. OCT. NOV. DEC. JAN. FEB. MAR. APR. MAY

4815.0 M 4975.0 M 5455.0 M 5555.0 M 5655.0 M 5755.0 M 5905.0 M 6055.0 M 6205.0 M 6330.0 M 6455.0 M 6580.0 M 6705.0 M 6830.0 M

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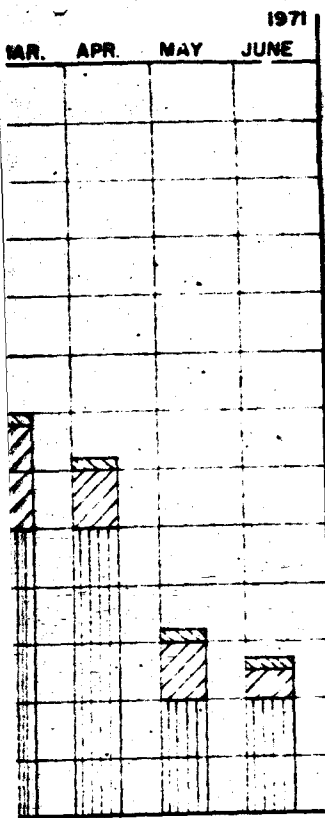


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CCDC-MPR-45

DATE: Feb 1971



PREDICTED **ACTUAL**

SUPERVISING [diagonal lines] [solid black]

PROFESSIONAL [diagonal lines] [white with circle]

TECHNICIAN [vertical lines] [diagonal lines with circle]

⊙ Includes Supervisory Exempt Technicians.
 ⊙⊙ Includes Clerical.

5

MONTHLY PROGRESS CHART

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 DEPARTMENT OF THE INTERIOR

CONSOLIDATION COAL CO.
 RESEARCH DIVISION LIBRARY, PA.

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125.0 M	125.0 M	170.0 M
MAR.	APR.	MAY JUNE
8205.0 M	8330.0 M	8500.0 M

PROGRESS REPORT NO. 45
 OCR CONTRACT NO. 14-01-0001-415(2)
CONSOL. LIGNITE GASIFICATION PROCESS DEVELOPMENT

February 1 to 29, 1968

A. Summary

Phase I - Feasibility Study

Complete.

Phase II - Bench-Scale Experiments

The Carnegie-Mellon subcontract on identification of the fundamentals of ash deposition has been completed. A final report is in preparation.

A number of runs were made in the continuous unit to seek regenerator conditions under which the ash deposition problem could be alleviated. Lower regenerator temperatures, lower carbon burnout, higher fluidizing velocity, and use of excess air were all helpful in reducing the amount of deposit formed. The low burn-out runs gave the most encouraging result, but even here the formation of a slight deposit was observed. Additional runs are planned to further define the factors influencing ash deposition in the bench scale of operation.

Phase III - Pilot Plant Design

The Catalytic design work is completed; copies of the design drawings and documents are in preparation for OCR.

Summary information on the Catalytic pilot plant estimate of \$8,800,000 is attached. Preliminary examination by Consol suggests this figure is at least 10% high.

Financial

Estimated expenditures in February were \$35,000. Cumulative expenses to date are \$1,546,513.

B. Technical Results and Program

1. Operation of the Continuous Gasification Unit (Project No. 532.00)

a. Regenerator Variable Studies

During the month, four runs were made to determine the effect of varying the regenerator conditions on the formation of ash deposits. The Husky II lignite char was used as regenerator fuel on the presumption that if any of the process changes prove to be successful with this troublesome material, then the ash deposit problem will have been solved for the more tractable lignite and sub-bituminous chars.

All the runs shared these common operating conditions:

System Pressure	11 atm
Gasifier Temperature	1500°F
Gasifier Feedstock	Husky II Char made in Run 532-C-9
Acceptor	16 x 28 mesh Tynochtee-6 dolomite

Regenerator Fluidizing Velocity 1.43 - 1.52 ft./sec.

All shutdowns were voluntary after the desired amount of fuel had been burned.

The acceptor activities all were essentially the same and were nearly identical with those of Run 532-A-21, reported in September, 1967. The results, with respect to formation of regenerator ash deposits are summarized below. Gasifier conditions and results are shown in Table I and similar data for the regenerator are shown in Table II.

Run	532-D-6	532-D-7	532-D-8	532-D-9
Purpose	Lower Temp.	Low Carbon Burnout	Excess Air	Duplicate of 532-A-24
Temperature, °F	1830	← 1870 →		
Fluidizing Velocity, ft/sec	1.43	1.50	1.52	1.50
<u>Offgas Composition, mol %</u>				
CO	0.3-0.6	3.5	0	0.3-0.6
O ₂	0	0	1.4	0
Carbon Burnout, %	~ 90	70	96	~ 90
Deposit, % of Ash Fed	2.7	0.6	2.0	6.0

All three process variants were beneficial in reducing the ash deposits. Sulfur rejection from the acceptor was complete in all runs except D-8 in which a small buildup of CaS-CaSO₄ (4.5% of the CaO) occurred after 52 cycles. Further discussion of the implications of these runs will be deferred until next month when results of further variations in regenerator conditions will be available.

b. Program

The next three runs will involve the following changes in regenerator conditions:

- D-10: Increase regenerator temperature to 1910°F
- D-11: Increase fluidizing velocity to 1.9 ft./sec.
- D-12: Add 2% (-200 mesh) acceptor fines to recirculating acceptor inventory

2. Batch Kinetic Studies (Project No. 530.00)

Tables of analytical data have been assembled for the materials listed below. Because of the large volume of this information, the tables will be issued in a separate memorandum.

1. regenerator fuel chars
2. gasifier product chars
3. gasifier feedstocks
4. devolatilizer feedstocks
5. acceptor feedstocks
6. acceptor outer shells
7. regenerator ash deposits
8. gasifier ash deposits
9. overhead ash from regenerator

Results of washability studies on the Colstrip sub-bituminous coal have been reported in E-11692, Project No. 530.00. The behavior was about the same as for the Rosbud coal reported last month in that, for a given specific gravity fraction, properties change only slightly with decreasing particle size and that wide differences in ash composition occur between the specific gravity fractions.

The Carnegie-Mellon subcontract was completed in February, and a final report will be written in March.

3. Supporting Patent and Legal Activities (Project No. 534.00)

The current laboratory reports and memoranda were reviewed for determination of Subject Inventions.

Phase III - Pilot Plant Engineering (Project No. 533.30)

1. Catalytic Design

The engineering work at Catalytic Construction Company was completed by March 1, as per the most recent schedule. Catalytic has submitted the original tracings and one reproducible copy of the general standards, process specifications, line tabulations, original calculations, vendor's data, estimates, as per contract. No further work or charges are anticipated. Copies of the Catalytic work are being prepared for OCR.

2. Pilot Plant Estimate

A detailed estimate of the pilot plant cost was provided by Catalytic and is summarized on Tables III A-H.

A preliminary analysis of this estimate indicates that the total of \$8,842,000 is probably 10-15% high. Referring to Table III-A, Consol projects the following reductions:

Item	Reduction	
	%	\$
Major Equipment and Freight	0	0
Foundations and Supports	25-30	40,000
Instrumentation	10	70,000
Buildings	15	140,000
Site Preparation	20	40,000
Field Charges, Temp. Const., & Misc.	30	300,000
Engineering	35	150,000
Overhead and Fee (at 6% of above)	-	45,000
Total Reduction, ex contingency		\$ 785,000
Contingency Reduction		79,000
Total		\$ 864,000

The use of 10% contingency seems high; however, until further checking can be done, Consol recommends the figure of \$8,000,000 for budgetary purposes. This is about twice the estimated cost included in the budget estimate for this project in 1966.

A comparison of the 1966 estimate and the Catalytic estimate shows the following:

	1966 Consol	1968 Catalytic
Section 100	477,000	720,000
Section 200	1,675,000	3,380,000
Section 300	278,000	880,000
Section 400	90,000	380,000
Section 500	530,000	1,110,000
Buildings and Site Prep.	570,000	1,530,000
	\$ 3,220,000	\$ 8,000,000

In the time interval between the two estimates, the basic process concept was not changed and no more than 10% escalation occurred. Accordingly, the disparity between the two estimates is primarily attributable to:

- 1) Recognition of the prevailing winter conditions at Rapid City and provision for basically indoor construction and more elaborate solids receiving and storage facilities (Section 100 and Buildings).

- 2) Provision of added operating flexibility by cooling and compressing gasifier offgases for feed to the devolatilizer and by cooling, scrubbing, and recycling regenerator offgases (Sections 200 and 300).

- 3) Failure in the earlier estimate to recognize the cost and complexity of providing mechanically adequate and safe equipment for the extreme conditions required for acceptor regeneration and recirculation (Section 200).

- 4) The decision to pilot two methanation systems (Section 400).

A more detailed examination of the Catalytic estimate and consideration of pilot plant revisions to reduce the investment cost will be carried out as time permits.

C. Subject Inventions

None.

D. Financial Status

Expenses	Actual	Budget
Cumulative through January, 1968	\$ 1,511,513	\$ 1,455,000
Expended in Period	35,000	20,000
Cumulative through February, 1968	\$ 1,546,513	\$ 1,475,000

Att. - Tables I, II,
III A-H
- Monthly Progress Chart
3/13/68

TABLE I

Gasifier Conditions and Results

Run No.	532-D-6	532-D-7	532-D-8	532-D-9
Cycle	36	20	7	34
Acceptor	Tymochtee-6 Dolomite			
Acceptor Circulation Rate (raw basis), lb/hr	9.02	9.17	10.49	10.56
System Pressure, atm	11			
Gasifier, Temperature, °F	1500			
Char	Husky 11 (Run C-9)			
Char Feed Rate, lb/hr	1.84	1.80	2.23	2.59
Inlet Gas (Bottom of Bed)				
Steam, SCFH	118.4	122.0	134.2	133.2
Recycle,				
Fluidizing Gas	152.2	157.2	160.5	159.0
Char Carrier Gas (Middle of Bed)*	34.0	35.1	35.8	35.5
Added				
H ₂ , SCFH	19.2	19.2	19.2	19.2
CO ₂ , "	15.0	15.0	15.0	15.0
H ₂ , "	1.8	1.8	1.8	1.8
Composition, Partial Pressure, atm				
H ₂ O	4.25	4.26	4.46	4.46
H ₂	3.51	3.72	3.67	3.68
CH ₄	.16	.17	.19	.17
CO	.98	.96	.76	.86
CO ₂	1.28	1.15	1.03	1.17
N ₂	.82	.82	.89	.86
Outlet Gas (Top of Bed)				
Composition, Partial Pressure, atm				
H ₂ O	2.81	2.78	3.06	3.10
H ₂	4.39	4.74	4.71	4.64
CH ₄	.24	.26	.29	.25
CO	1.52	1.37	1.19	1.32
CO ₂	1.16	.97	.83	1.01
N ₂	.88	.88	.92	.88
Dry Exit Gas, SCFH	84.7	87.9	94.3	95.6
N ₂ Purges,	10.1	10.4	12.8	9.0
H ₂ , "	-	-	-	-
Condensate,	89.6	91.4	107.2	110.6
Recycle Gas Composition, mol %				
H ₂	51.6	55.6	56.7	57.0
CH ₄	2.67	3.06	3.49	3.10
CO	17.83	16.03	14.30	16.21
CO ₂	13.65	11.41	10.01	12.43
N ₂	14.0	13.9	15.5	11.3
% Steam Conversion	24.3	25.1	20.2	17.0
Fluidizing Velocity, ft/sec				
Boot	.88	.90	.95	.91
Avg. Bed	.35	.36	.38	.38

* Contributes to outlet gas only.

TABLE II

Regenerator Conditions and Results

Run No.	532-D-5	532-D-7	532-D-8	532-D-9
Duration, Cycles	55	36	52	34
Hours	31.3	19.7	30.3	23.7
Acceptor	10 x 28 mesh Tymochtee-6 Dolomite			
Acceptor Circulation Rate, lb/hr (raw basis)	9.02	9.17	11.19	12.52
System Pressure, atm	11			
Regenerator	1870 Husky 11 Lignite Char			
Temperature, °F	1870 Husky 11 Lignite Char			
Fuel Char	48x65 (Run A-21, 22) -65 (Run A-21, 22) -48 (Run A-26)			
Fuel Feed Rate, lb/hr	30.0	1.31	30.0	0.98
lb Fuel Char Fed	30.0	25.6	30.0	23.3
Inlet Gas				
Recycle, SCFH	159.1	171.4	162.8	171.4
Added N ₂ , "	29.1	20.0	20.0	28.8
CO ₂ , "	0	8.6	0	0
Air	98.0	94.0	118.0	93.0
Recycle Gas Composition, mol %				
N ₂	Balance	Balance	Balance	Balance
CO ₂	22.3	22.3	22.3	22.3
O ₂	0	0	1.4	0
CO	0.3-0.6	3.5	0	0.3-0.6
Air in Inlet Gas, mol %	34.2	34.0	36.6	35.6
CO ₂ Partial Pressure, atm	2.45	2.45	2.45	2.45
Equilibrium CO ₂ Partial Pressure, atm	3.80	4.90	4.90	4.90
ΔP Driving Force, atm	1.35	2.45	2.45	2.45
Fluidizing Velocity, ft/sec	1.43	1.50	1.52	1.50
Acceptor Activity	0.56	0.70	0.81	0.64

TABLE III-A

Summary Estimate for Pilot Plant

	<u>Material</u>	<u>Labor</u>
Major Equipment	\$ 2,387,375	\$ 162,555
Freight	15,105	-
Foundations and Supports	60,335	81,320
Power Wiring	83,285	123,045
Process Piping	683,760	290,580
Instrumentation	387,550	169,915
Buildings	637,515	302,815
Site Work	148,400	49,350
Total	\$ 4,743,325	\$ 1,179,580
Supplies and Tools	58,450	-
Temporary Construction	47,750	47,750
Welder Qualif.	-	25,000
Misc. Directs	64,300	184,100
Winter Work	17,000	31,000
Total Direct	\$ 4,931,225	\$ 1,467,430
Direct M + L	\$ 6,398,655	
Insurance and Taxes	37,025	
Payroll Burden	183,430	
Field Supervision, Tools, etc.	540,000	
Engineering and Accounting	424,000	
Overhead and Fee	454,890	
Total Cost	\$ 8,038,000	
Contingency	804,000	
	\$ 8,842,000	

TABLE III-B

100 Area Summary

	<u>Material</u>	<u>Labor</u>
Major Equipment	\$ 308,520	\$ 28,480
Freight	4,020	-
Foundations & Supports	17,240	33,695
Power Wiring	5,780	14,140
Process Piping incl. Insul. & Steam Tr.	6,375	3,825
Instrumentation	81,150	24,750
	\$ 424,085	\$ 104,890

TABLE III-C

200 Area Summary

Major Equipment	\$ 1,363,740	\$ 59,935
Freight	5,575	-
Foundations & Supports	14,470	4,750
Power Wiring	25,965	28,390
Process Piping incl. Insul. & Steam Tr.	420,975	146,325
Instrumentation	338,630	102,480
	\$ 2,167,335	\$ 341,880

TABLE III-D

300 Area Summary

Major Equipment	\$ 424,045	\$ 26,935
Freight	390	-
Foundations & Supports	5,460	4,700
Power Wiring	3,340	11,415
Process Piping incl. Insul. & Steam Tr.	73,950	24,700
Instrumentation	61,575	18,740
	\$ 568,760	\$ 86,490

TABLE III-E
400 Area Summary

	<u>Material</u>	<u>Labor</u>
Major Equipment	\$ 147,915	\$ 11,610
Freight	2,250	-
Foundations & Supports	3,340	3,470
Power Wiring	4,605	10,470
Process Piping incl. Insul. & Steam Tr.	8,920	5,235
Instrumentation	60,345	18,370
	<u>\$ 229,375</u>	<u>\$ 51,155</u>

TABLE III-F
500 Area Summary

	<u>Material</u>	<u>Labor</u>
Major Equipment	\$ 342,355	\$ 35,595
Freight	2,870	-
Foundations & Supports	17,825	32,705
Power Wiring	43,595	58,630
Process Piping incl. Insul. & Steam Tr.	153,540	110,495
Instrumentation	17,850	5,375
	<u>\$ 578,035</u>	<u>\$ 243,000</u>

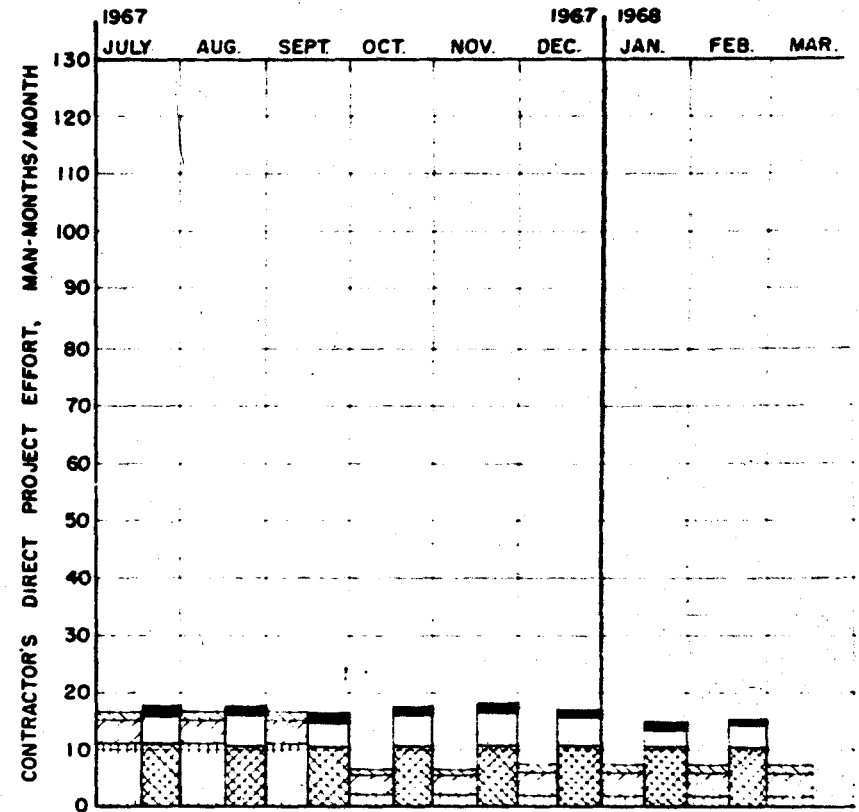
TABLE III-G
Buildings Summary

	<u>Material</u>	<u>Labor</u>
Administration		
Architectural	\$ 103,925	\$ 49,860
Heating & Ventilation	22,895	7,805
Lighting & Power Wiring	38,130	42,305
Plumbing & Service Pipe	10,795	7,690
Sprinklers	3,500	-
Total	<u>\$ 179,245</u>	<u>\$ 107,660</u>
Process Structure		
Architectural	381,715	182,225
Heating & Ventilation	14,285	4,410
Lighting & Wiring (incl. Admin. Bldg.)	-	-
Housing & Service Pipe	8,200	5,695
Sprinklers	2,400	-
Total	<u>\$ 386,600</u>	<u>\$ 172,330</u>
Unloading Bldg.		
Architectural	29,610	8,885
Total	<u>\$ 29,610</u>	<u>\$ 8,885</u>
Material Handling Bldg.		
Architectural	24,915	11,540
Total	<u>\$ 24,915</u>	<u>\$ 11,540</u>
Misc. Service Buildings	7,145	2,400
Total	<u>\$ 7,145</u>	<u>\$ 2,400</u>
Total Buildings	<u>\$ 627,515</u>	<u>\$ 302,815</u>

TABLE III-B

Site Summary

	<u>Material</u>	<u>Labor</u>
General Grading	\$ 25,750	\$ -
Paving & Sidewalks	29,505	4,300
Fencing	11,635	2,335
Landscaping	11,700	-
Underground Lines		
Steam	8,720	6,440
Sanitary	4,515	1,710
Fire Protection	13,170	7,185
Chemical Drain	18,320	4,265
Service Lines	9,810	18,770
	15,275	8,345
Railroad		
Total	\$ 148,400	\$ 48,350



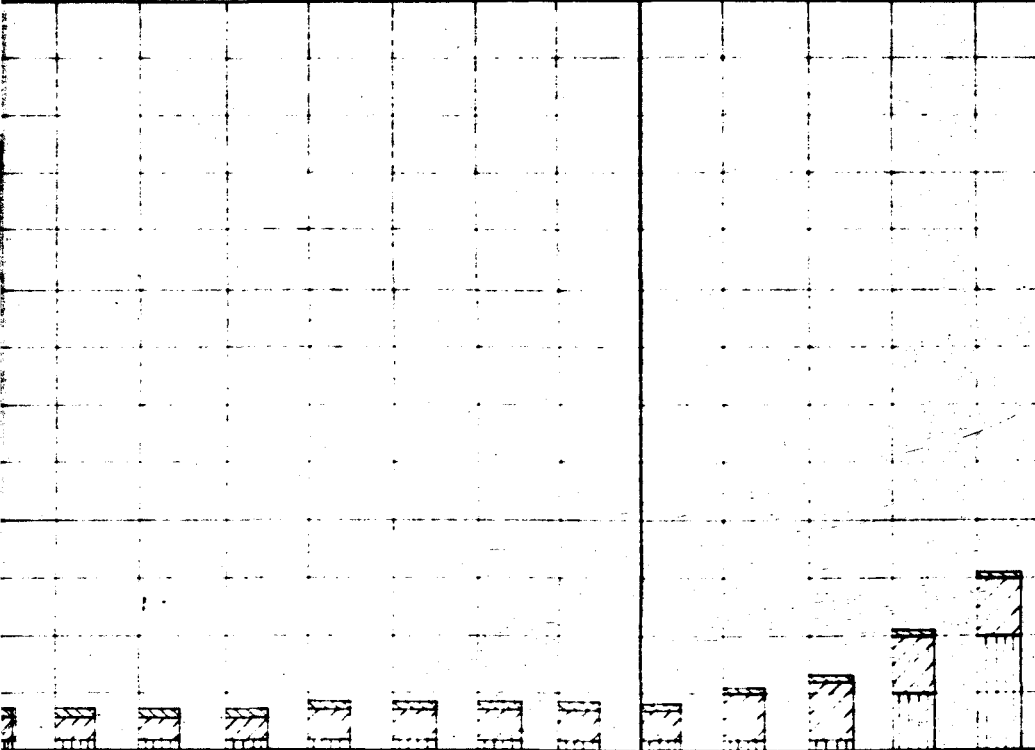
	MONTHLY EXPENDITURES*								
	1967	1967	1967	1967	1967	1967	1968	1968	1968
	JULY	AUG.	SEPT.	OCT.	NOV.	DEC.	JAN.	FEB.	MAR.
PREDICTED	103.7 M	72.2 M	92.0 M	94.0 M	94.0 M	52.0 M	47.0 M	20.0 M	70.0 M
ACTUAL	75.5 M	78.0 M	106.8 M	84.9 M	97.1 M	93.4 M	70.0 M	35.0 M	
PREDICTED	1010.6 M	1083.0 M	1175.0 M	1269.0 M	1363.0 M	1415.0 M	1455.0 M	1475.0 M	1545.0 M
ACTUAL	981.3 M	1059.3 M	1166.1 M	1251.0 M	1348.1 M	1441.5 M	1511.5 M	1546.5 M	

CUMULATIVE EXPENDITURES*

* Expenditures Include All Costs & Fees.

MOI

1968 1969
MAY JUNE JULY AUG. SEPT. OCT. NOV. DEC. JAN. FEB. MAR. APR. MAY



160.0 M 220.0 M 220.0 M 220.0 M 220.0 M 320.0 M 320.0 M 320.0 M 420.0 M 290.0 M 290.0 M 240.0 M 160.0 M

MAY JUNE JULY AUG. SEPT. OCT. NOV. DEC. JAN. FEB. MAR. APR. MAY

1955.0 M 2175.0 M 2395.0 M 2615.0 M 2935.0 M 3255.0 M 3575.0 M 3995.0 M 4285.0 M 4575.0 M 4815.0 M 4975.0 M

2

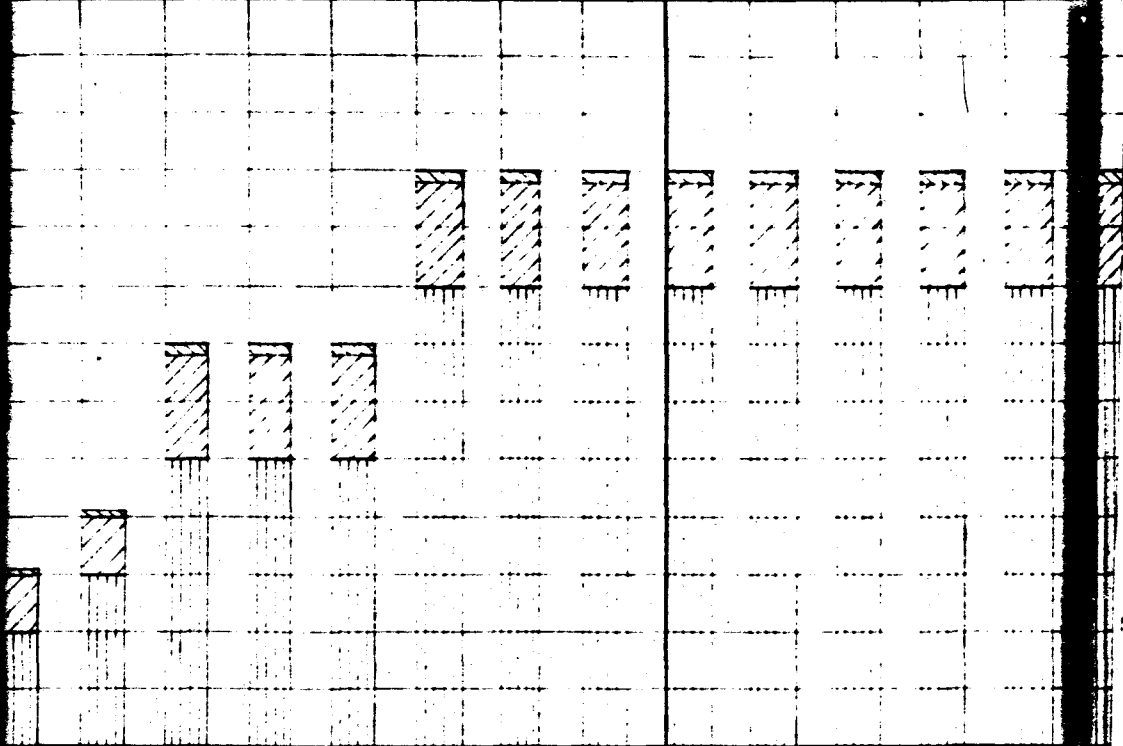
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MONTHLY PROGRESS CHART

CONTRACT No. 14-01-0001-415

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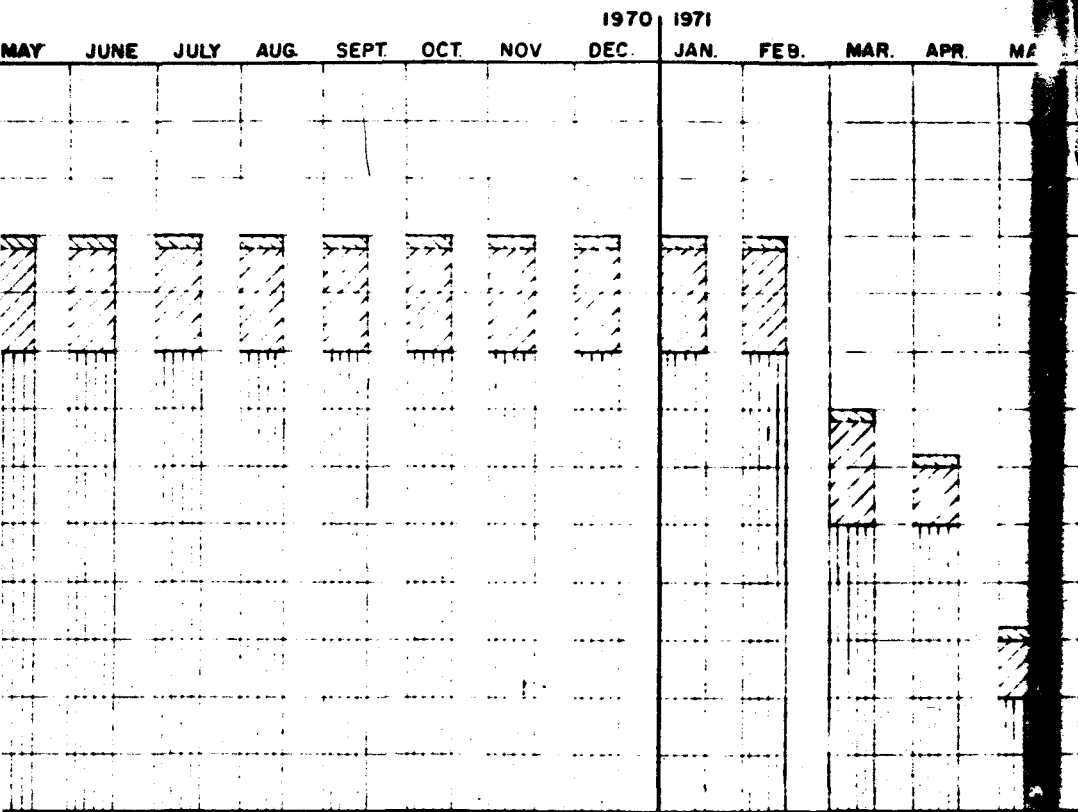
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MAY JUNE JULY AUG. SEPT. OCT. NOV. DEC. JAN. FEB. MAR. APR. MAY

4975.0 M 5455.0 M 5555.0 M 5655.0 M 5755.0 M 5905.0 M 6055.0 M 6205.0 M 6330.0 M 6455.0 M 6580.0 M 6705.0 M 6830.0 M

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MAY	JUNE	JULY	AUG.	SEPT.	OCT.	NOV.	DEC.	JAN.	FEB.	MAR.	APR.	MAY
830.0 M	8955.0 M	7080.0 M	7205.0 M	7330.0 M	7455.0 M	7580.0 M	7705.0 M	7830.0 M	7955.0 M	8080.0 M	8205.0 M	8330.0 M

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B. Technical Results and Program

Phase II - Bench-Scale Research

1. Operation of the Continuous Gasification Unit (Project No. 532)

Beginning with the runs reported this month, the gasifier feedstock used in the experimental program for study of regenerator ash deposits will be Disco char which was produced in Run C-10. Operations will be at 1500°F and top-of-gasifier conditions. Under these circumstances, very little carbon will be gasified and the product from one run will be used as feedstock to the succeeding run. CO₂ will be added to the inlet gas to allow recarbonation of the acceptor. Gasifier conditions and results are shown in Table I.

a. Run 532-D-10, Regenerator Operation at 1910°F

To continue the study on the effect of temperature on regenerator deposits from Husky II fuel char, this run was made at 1910°F. Previous runs in this series were:

D-6	1830°F
D-9 and A-26	1870
A-25	1890

A shutdown after four cycles was caused by a cracked fitting in the acceptor return line between the regenerator and gasifier. The regenerator was examined at this point and a deposit amounting to 5.0% of the ash fed was removed. The deposit was distributed uniformly over the vessel walls and internals. After repairs were made, the run was resumed and continued until the 24th cycle when the regenerator bed agglomerated. Inspection of the vessel showed that there was no deposit on the walls or internals. The acceptor was coated with a heavy shell which had caused the agglomeration of the acceptor inventory.

The shell was attrited from the acceptor and amounted to 11 wt.% of the total acceptor, which corresponds to 35% of the ash fed to the regenerator. By ash balance, based on the overhead ash from the regenerator, the deposit on the acceptor was 37% of the ash fed.

b. Run 532-D-11, Regenerator Operation at a Greater Fluidizing Velocity

In this run, the regenerator fluidizing gas velocity was increased from 1.5 to 1.94 ft./sec. which gave about twice the bed expansion normally used. The combustion air rate and exit gas composition were kept the same as in Run D-9. However, the more highly expanded acceptor bed was less retentive of the feed char particles, and the carbon burnout dropped to 69% compared with 90% in Run D-9. The run was terminated after 24 cycles

when the regenerator recycle gas compressor failed. Examination of the regenerator showed a uniform deposit on the vessel walls and internals which amounted to 3.8% of the ash fed. A thin, soft shell on the acceptor amounting to an additional 1.8% of the ash fed brought the total ash deposit to 5.6%, compared with 6.0% in Run D-9.

c. Run 532-D-12, Regenerator Operation with Acceptor Fines Added

This run was made at the same conditions as Run D-9 except that 2% of -200 mesh raw Tymochee dolomite was added to the circulating acceptor inventory. The fines addition increased the superficial surface area of the acceptor by a factor of about three. The purpose of the run was to determine whether the sticky ash deposit would adhere preferentially to the acceptor fines and thereby be swept from the vessel.

The run ended after only four cycles when the acceptor overflow weir in the regenerator plugged. Examination of the vessel showed a uniform, massive, soft deposit which amounted to 18% of the ash fed. The weir was plugged by chunks of the deposit which had sloughed off the walls.

The deposit was mostly acceptor fines which had been cemented together by the sticky ash. Table II shows the regenerator conditions and results for the three runs described above.

d. Conclusions from the Regenerator Variable Study

When the regenerator is operated with Husky II fuel char at "neutral" conditions, i.e., essentially stoichiometric combustion of the inlet air to CO₂ - the following conclusions are drawn:

1. Massive deposits form at all temperatures in the range 1830-1910°F. Increasing temperature increases the severity of the deposit.
2. At temperatures of 1870°F and below the deposit forms almost exclusively on the vessel walls and internals.
3. At temperatures above 1870°F, the deposit tends to remain on the acceptor as an outer shell, until, at 1910°F the deposit is exclusively on the acceptor.
4. Increasing the regenerator fluidizing velocity and bed expansion has almost no effect on deposit formation, in the range studied.
5. Low burnout of the fuel char carbon (70% vs. 90%) leaves a carbon skeleton which is ineffective in retaining the sticky ash which forms.
6. Acceptor fines contribute to the deposit by being cemented together by the sticky ash. Run D-10A confirms this conclusion because a vessel deposit formed in the early stage of the run (four cycles) when the fresh acceptor was attriting rapidly as the sharp edges of the particle were being rounded off.

7. The compositions of the recovered deposits are remarkably similar, regardless of the regenerator conditions and the origin of the fuel char and acceptor. The melting points of the as-recovered deposits are very high ($> 2800^{\circ}\text{F}$). The attrited shells from the acceptor and the vessel deposits have almost identical compositions.

At non-"neutral" conditions, i.e., when either O_2 or CO is present in the exit gas in appreciable amounts, two more conclusions may be drawn.

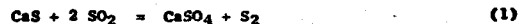
8. Excess O_2 diminishes the deposit (2.0% vs. 6.0%) but still gives a massive deposit.
9. The only drastic reduction in the amount of deposit occurred in Run D-7 in which a deficiency of air was used to give 3.3% CO content in the exit gas.

e. Proposed Mechanism of Ash Deposition

A mechanism which is consistent with all of the above conclusions is that a low-melting liquid forms during the combustion of the fuel char. This liquid diffuses rapidly from the char particles, then adheres to the acceptor, which in turn (at temperatures $\leq 1870^{\circ}\text{F}$) smears it uniformly over the vessel walls and internals. The liquid must exist only transiently and its decomposition product must be CaO or CaSO_4 to account for the observed compositions and melting points of the as-recovered deposits.

The source of the transient liquid very likely is CaS . Some of the line in the ash of both lignite and subbituminous chars is converted to CaS in the gasifier. In the regenerator this CaS is oxidized to CaSO_4 by either O_2 or CO_2 at "neutral" or excess oxygen conditions. Thus, the liquid forms when CaS and CaSO_4 are present simultaneously. The amount and nature of the resulting deposit is determined by the temperature and the lifetime of the transient liquid.

The CaS-CaSO_4 transient liquid has been confirmed experimentally. A sample of Tynochtee dolomite was converted to the MgO.CaS formed and heated in the hot-wire microscope in order that the following reaction would occur:



This reaction does not occur at process conditions because the SO_2 partial pressure is too low, but was used experimentally to insure that CaSO_4 was the principal end product. At an initial content of 100% CaS the sample melted, then froze within two seconds, at 2150°F . With a sample having both CaS and CaSO_4 initially present (ratio unknown), the same phenomena occurred at 1750°F .

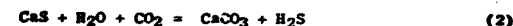
f. Run 532-C-11, Production of Desulfurized Husky II Char

To test the above mechanism of ash deposition, a run was scheduled in which no sulfur, in any form, would enter either the regenerator or gasifier. As a necessary preliminary, Run C-11 was made to thoroughly desulfurize the regenerator fuel char.

The gasifier was operated batchwise at 11 atm. pressure with the char product from Runs D-1C and D-5 as feedstock. This char, which previously was the char product from Run A-25, had a low organic sulfur content (0.23%) and a high sulfide sulfur (CaS) content of 1.27%. During the run, several combinations of operating conditions within the ranges listed below were used:

Temperature, $^{\circ}\text{F}$	1300-1400
Outlet Partial Pressures, atm.	
H_2O	4.7-8.0
H_2	2.9-6.2
CO_2	.07-.59
CO	.06-.29

Desulfurization proceeded rapidly at all conditions through the reaction,



g. Run 532-D-13(B), Operations in Absence of Sulfur

During the initial operations in Run D-13, a stock of Disco char for use as gasifier feedstock was desulfurized. The total sulfur content of the product was 0.12%. At the gasifier conditions used in Runs D-13(A) and D-13(B), no sulfur could have been transferred from this feedstock to the acceptor.

Run D-13(A) was terminated after three cycles, due to a leak in the acceptor discharge feeder. Examination of the regenerator showed no deposits of any nature. An unusual behavior of the acceptor was observed in this run in that the average particle size had decreased rapidly. Continued decrease at that rate over the first three cycles soon would have prevented the acceptor from showering through the gasifier bed.

In Run D-13(B), a fresh inventory of 16 x 20 mesh acceptor was used, rather than the normal 16 x 28 mesh size consist, in an effort to maintain the showering. The Tynochtee dolomite contains 0.2% total sulfur. In both Runs D-13(A) and D-13(B), the acceptor was circulated through the unit in the MgO.CaCO_3 form in order to desulfurize the inventory by reaction (2), above.

Run D-13(B) proceeded smoothly until the desired amount of fuel char had been burned in the regenerator. There were no deposits on the vessel wall. The internals had a slight deposit amounting to 0.06% of the

ash fed. This run showed a one hundredfold reduction in the amount of deposit over that in Run D-9 which was a comparable run except for the sulfur level involved. Regenerator conditions and results are shown in Table II.

The acceptor, sampled periodically during the run, was noticeably softer than usual and the attrition losses, caused mainly by the grinding action of the inlet and discharge rotary feeders, were twice the normal losses.

Elucidation of the ash deposit mechanism has two major process implications:

1. Process operability can be maintained by desulfurizing the regenerator fuel char. This route may be too costly. More design data for desulfurization via reaction (2) will be obtained so that a cost study can be made.
2. The formation of the transient liquid can be substantially suppressed by operating the regenerator with CO in the exit gas as in Run D-7.

h. Program

1. The entire unit will be operated at 1 atm. system pressure to determine the nature of the ash deposit at this pressure, which would be used in a proposed low-cost, interim pilot plant.
2. A demonstration run with Colstrip fuel char at 1940°F and CO in the exit gas will be made to prove that suppression of transient liquid formation can be accomplished at this higher temperature.
3. A run similar to Run D-13(B) will be made in which the acceptor will be allowed to go through the sulfur cycle in order to determine: (a) any interaction of acceptor-derived CaS with the desulfurized fuel char, and (b) to determine whether the acceptor strength will be maintained.
4. A series of runs will be made using 100% burnoff Husky II ash, while supplying the regenerator heat requirement by combustion of CO in order to determine what influence the vessel wall temperature and local (high) temperature of the burning char particles have on the amount and nature of the ash deposit.

2. Batch Kinetic Studies (Project No. 530)

The entire effort was devoted to routine processing of samples produced during operations of the continuous gasification unit.

The final report on the Carnegie-Mellon studies is being written and is expected during April.

3. Supporting Patent and Legal Activities (Project No. 534)

The current laboratory reports and memoranda were reviewed for determination of Subject Inventions.

4. Supporting Process and Economic Analysis (Project No. 533)

A draft of an interim report on the CO₂ Acceptor development was prepared, in which the status of the technology and the remaining problems were reviewed. A combined experimental design, and economic analysis program is recommended prior to decision on pilot plant construction on or about July 1, 1968. This report will be issued to OCR in April.

Phase III - Pilot Plant Engineering

5. Catalytic Design (Project No. 533.30)

All drawings and specifications for the gasification pilot plant were received from Catalytic Construction Company. An "Invitation to Bid" package for the plant was completed and will be sent to the Office of Coal Research for review and approval.

Some time was spent in review of Catalytic's process piping drawings.

The above "package" consolidates all of Catalytic's work to date. It is not planned to solicit bids from potential construction companies until an acceptable solution to the ash deposition problem has been found and its impact on process economics and pilot plant design has been established.

C. Subject Inventions

None.

D. Financial Status

<u>Expenses</u>	<u>Actual</u>	<u>Budget</u>
Cumulative through February, 1968	\$ 1,541,454	\$ 1,475,000
Expended in Period	90,000	70,000
	\$ 1,631,454	\$ 1,545,000

4/12/68

Att. - Monthly Progress Chart
Tables I and II

TABLE I

Gasifier Run Conditions and Results

Run No. 532- Cycle	D-10A 2	D-10B 9	D-11 22	D-13(B) 9
Acceptor	← 16 x 28 Mesh → 16 x 20 M ← Tymochee-6 Dolomite →			
Acceptor Circulation Rate (raw basis), lb/hr	8.93	12.05	9.59	9.28
System Pressure, atm	← 20 →			
Gasifier, Temp., °F	1500			
Char	← Disco Char →			
Char Feed Rate, lb/hr	Run C-10 2.86	2.86	Run D-10 3.20	Run D-13 2.32
Inlet Gas (Bottom of Bed)				
Steam, SCFH	88.9	82.6	104.3	114.0
Recycle, "				
Fluidizing Gas	280.0	258.6	118.2	131.8
Char Carrier Gas (Middle of Bed)*	57.1	52.8	33.1	36.9
Added H ₂ , SCFH	19.3	19.3	19.2	19.2
CO ₂ , "	12.4	12.4	15.0	15.0
N ₂ , "	1.5	1.5	1.8	1.8
Composition, Partial Pressure, atm				
H ₂ O	4.42	4.41	4.44	4.45
H ₂	7.02	5.86	3.20	3.65
CH ₄	1.91	1.51	0.20	0.13
CO	0.95	1.33	0.42	0.21
CO ₂	1.18	1.67	1.28	0.80
N ₂	4.52	5.22	1.46	1.78
Outlet Gas (Top of Bed)				
Composition, Partial Pressure, atm				
H ₂ O	4.20	4.33	4.27	4.02
H ₂	7.14	7.78	3.35	4.16
CH ₄	2.25	1.51	0.29	0.19
CO	1.12	1.33	0.59	0.30
CO ₂	0.66	1.67	0.90	0.31
N ₂	4.63	5.22	1.60	2.02
Dry Exit Gas, SCFH	49.3	52.3	39.4	40.8
H ₂ Purges, "	14.2	18.0	9.0	11.5
H ₂ , "	-	-	-	-
Condensate, "	98.8	95.6	115.1	114.2
Recycle Gas Composition, mol %				
H ₂	43.51	35.05	47.44	56.38
CH ₄	13.70	10.92	4.05	2.55
CO	6.85	9.60	8.31	4.11
CO ₂	4.04	7.23	12.67	4.26
N ₂	31.9	37.2	27.5	32.7
% Steam Conversion	**	**	**	**
Fluidizing Velocity, ft/sec				
Boot	.633	.880	.740	.794
Avg. Bed	.254	.238	.291	.310

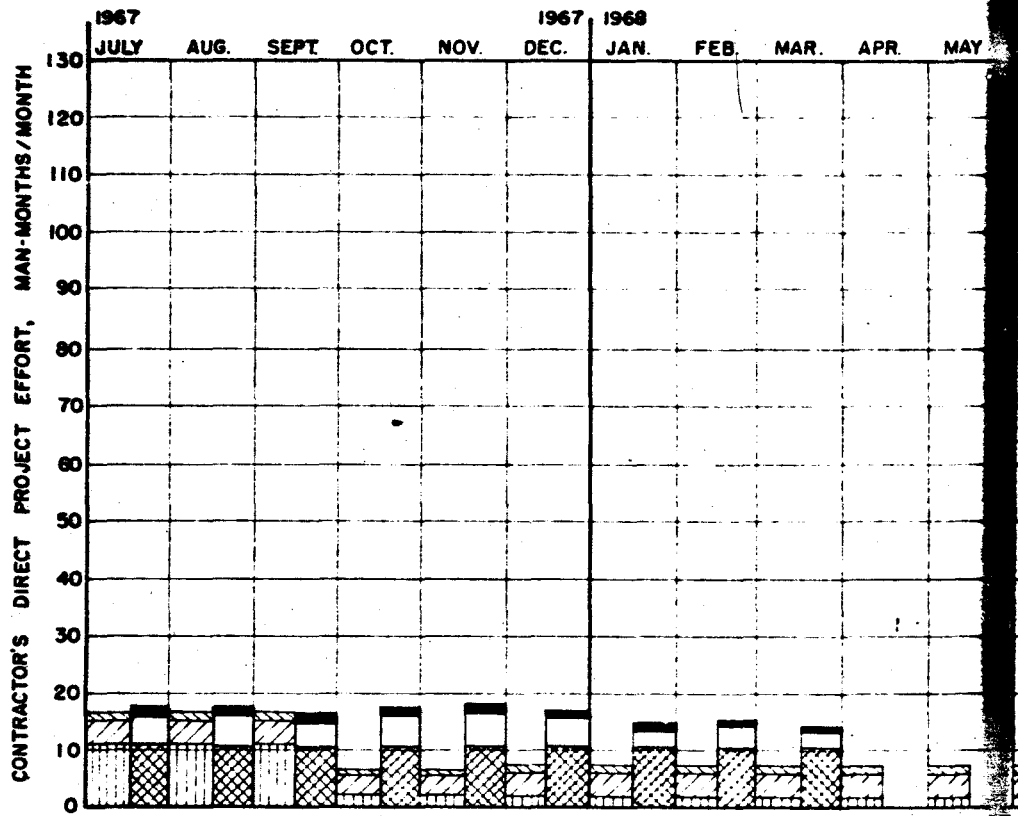
* Contributes to outlet gas only.

** Steam conversion negative due to low gasification rates and water gas shift reaction.

TABLE II

Regeneration Conditions and Results

Run No. 532-	D-10(B)	D-11	D-12	D-13(B)
Duration, Total Cycles	24	24	4	37
Hours	20.1	17.1	5.6	16.8
Acceptor	← 16 x 28 Mesh → 16 x 20 M ← Tymochee-6 Dolomite →			
Acceptor Circulation Rate (raw basis), lb/hr	11.28	9.59	8.62	9.28
% Acceptor Fines Addition	0	0	2.0	0
System Pressure, atm	20	11	11	11
Regenerator				
Temperature, °F	1910	1870	1870	1870
Fuel Char	← 48 M Husky II Lignite Char → -48 M Husky II, desulfurized			
Fuel Char Feed Rate, lb/hr	0.87	1.18	0.87	1.18
lb Fuel Char Fed	17.4	20.4	4.5	19.8
Inlet Gas				
Recycle	235	256.7	178.8	192.7
Added N ₂	35	20.0	20.0	20.0
CO ₂	8.0	5.4	0	3.0
Air	106	99	94	94
Recycle Gas Composition, mol %				
N ₂	← Balance →			
CO ₂	20.0	22.3	22.3	22.3
CO	0.3-0.6	0.3-0.6	0.3-0.6	0.3-0.6
O ₂	0	0	0	0
Air in Inlet Gas, mol %	27.5	26.0	32.1	30.4
CO ₂ Partial Pressure, atm	4.00	2.45	2.45	2.45
Equilibrium CO ₂ Partial Pressure, atm	6.30	4.90	4.90	4.90
ΔP Driving Force, atm	2.30	2.45	2.45	2.45
Fluidizing Velocity, ft/sec	1.10	1.94	1.49	1.58
Acceptor Activity	0.44	0.60	0.87	0.81
Corresponding Cycle No.	12	22	2	9



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MONTHLY EXPENDITURES*

	JULY	AUG.	SEPT.	OCT.	NOV.	DEC.	JAN.	FEB.	MAR.	APR.	MAY
PREDICTED	103.7 M	72.2 M	92.0 M	94.0 M	94.0 M	52.0 M	40.0 M	20.0 M	70.0 M	70.0 M	120.0 M
ACTUAL	75.5 M	78.0 M	106.6 M	84.9 M	97.1 M	93.4 M	70.0 M	29.9 M	90.0 M		

	JULY	AUG.	SEPT.	OCT.	NOV.	DEC.	JAN.	FEB.	MAR.	APR.	MAY
PREDICTED	1010.6 M	1083.0 M	1175.0 M	1269.0 M	1363.0 M	1415.0 M	1455.0 M	1475.0 M	1545.0 M	1615.0 M	1735.0 M
ACTUAL	981.3 M	1059.3 M	1166.1 M	1251.0 M	1348.1 M	1441.5 M	1511.5 M	1541.4 M	1631.4 M		

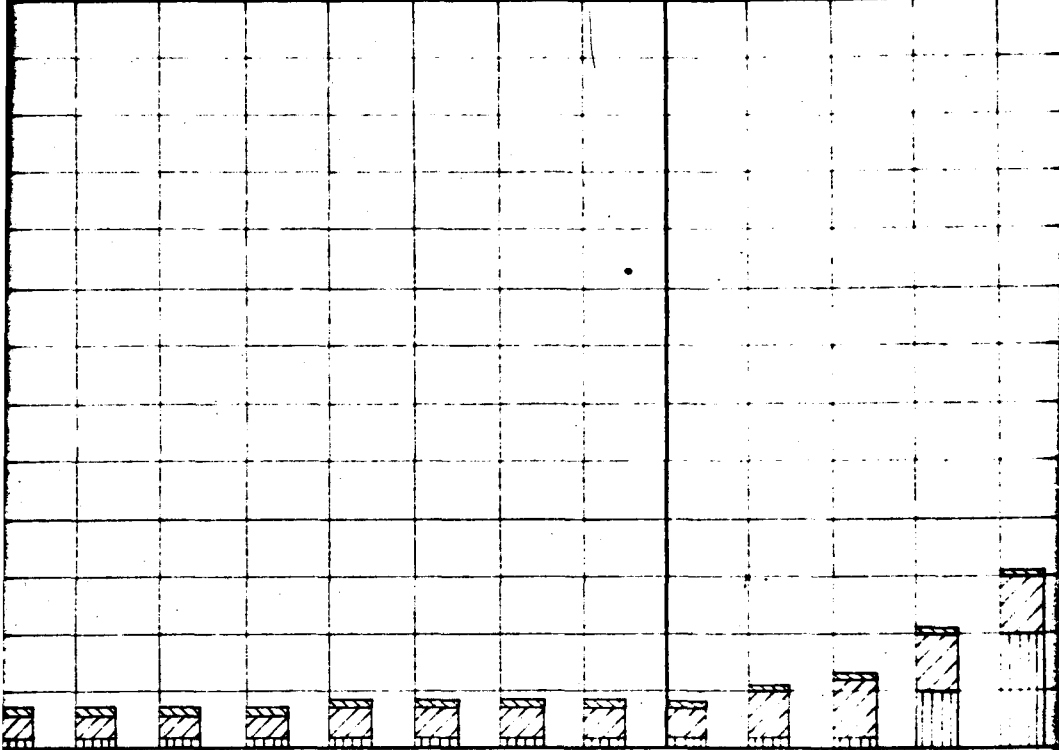
CUMULATIVE EXPENDITURES*

* Expenditures Include All Costs & Fees.

180.0 M
MAY
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1968 1969
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120.0 M 220.0 M 220.0 M 220.0 M 220.0 M 320.0 M 320.0 M 320.0 M 420.0 M 290.0 M 290.0 M 240.0 M 160.0 M

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1735.0 M 1955.0 M 2175.0 M 2395.0 M 2615.0 M 2935.0 M 3255.0 M 3575.0 M 3995.0 M 4285.0 M 4575.0 M 4815.0 M 4975.0 M

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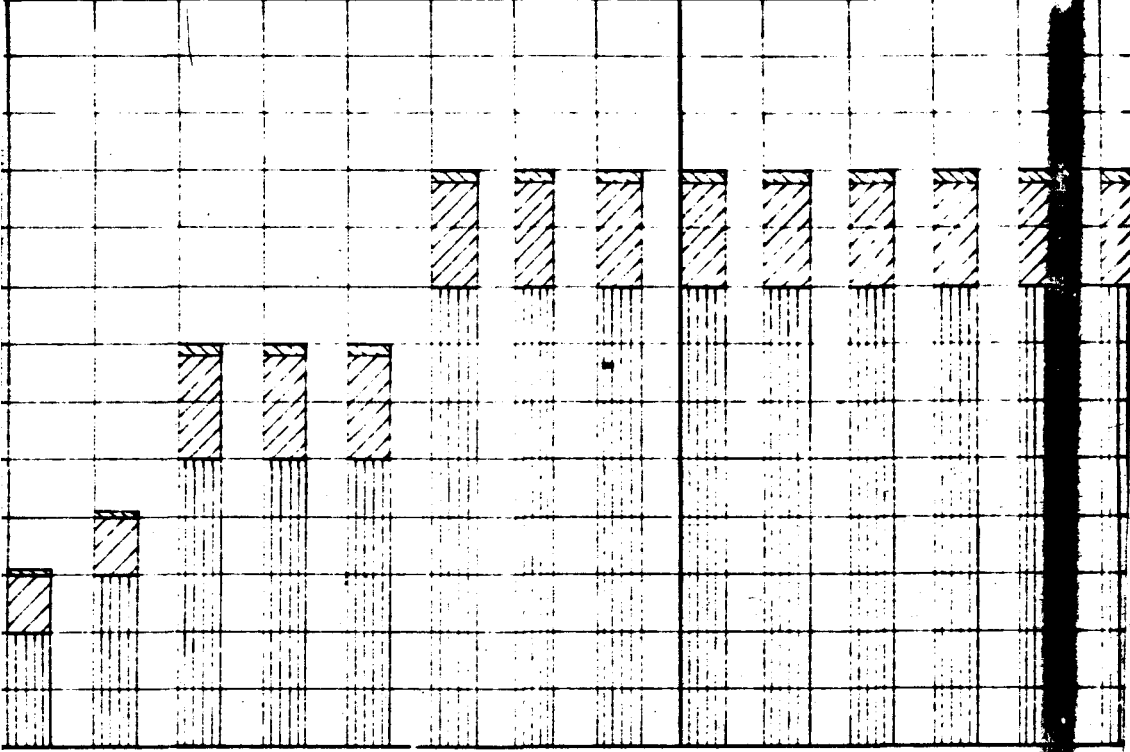
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MONTHLY PROGRESS CHART

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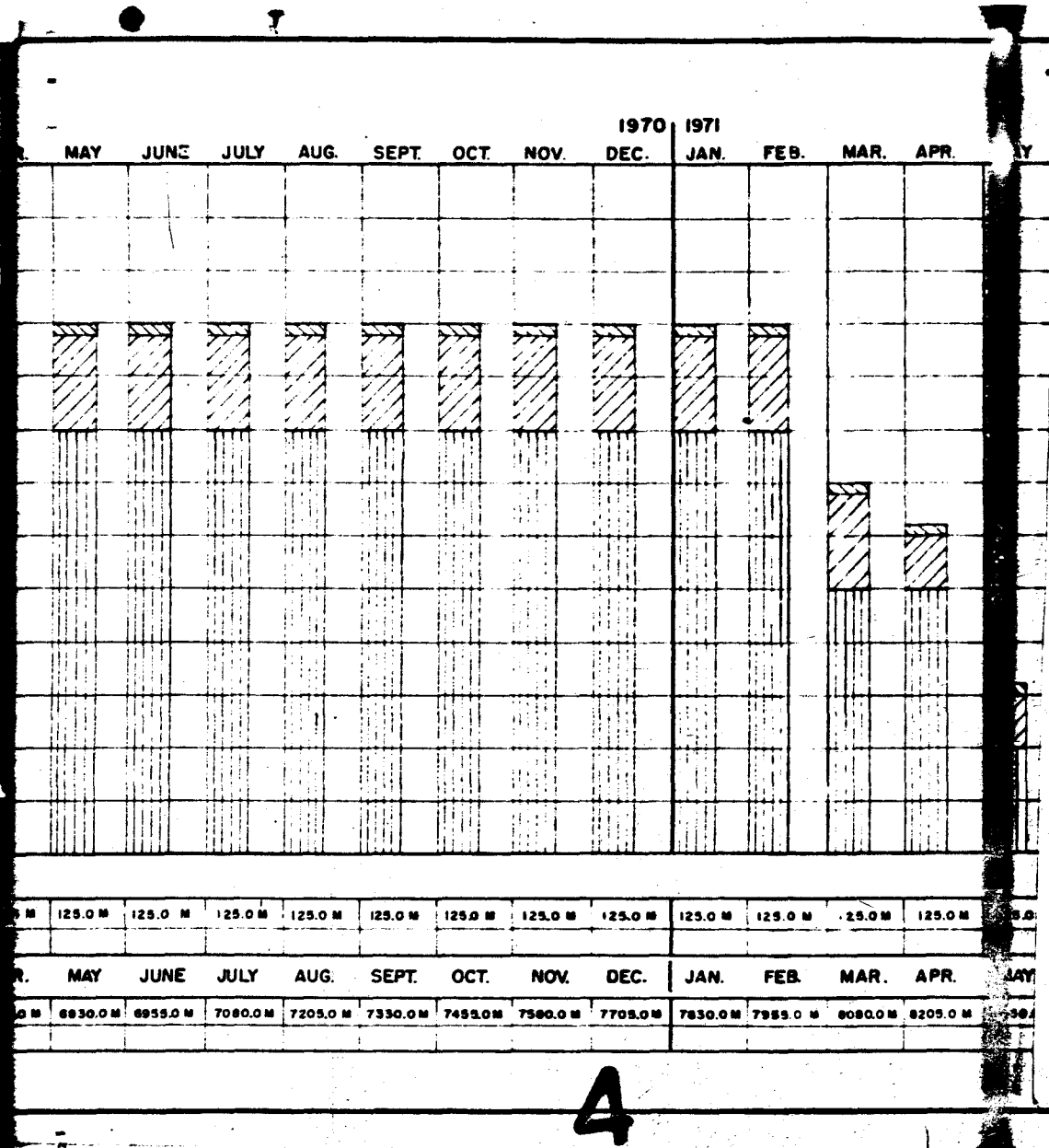
100.0 M	400.0 M	100.0 M	100.0 M	100.0 M	150.0 M	150.0 M	150.0 M	125.0 M	125.0 M	125.0 M	125.0 M	125.0 M	125.0 M	125.0 M	125.0 M
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4075.0 M	5455.0 M	5555.0 M	5655.0 M	5755.0 M	5905.0 M	6055.0 M	6205.0 M	6330.0 M	6455.0 M	6580.0 M	6705.0 M	6830.0 M	6955.0 M	7080.0 M	7205.0 M
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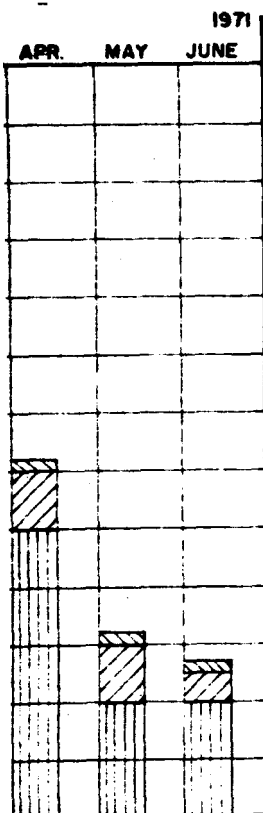
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DATE: March 1968



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PROFESSIONAL [Diagonal Lines Box] [White Box with *]

TECHNICIAN [Vertical Lines Box] [Hatched Box with *]

* Includes Supervisory Exempt Technicians.
 ** Includes Clerical.

MONTHLY PROGRESS CHART

OFFICE OF COAL RESEARCH
 DEPARTMENT OF THE INTERIOR

CONSOLIDATION COAL CO.
 RESEARCH DIVISION LIBRARY, PA.

CONTRACT No. 14-01-0001-415

5

PROGRAM REPORT NO. 47
 OUR CONTRACT NO. 14-01-0001-415(2)
CONSOL. LIGNITE GASIFICATION PROCESS DEVELOPMENT

April 1 to 30, 1968

A. Summary

Phase I - Feasibility Study

Complete.

Phase II - Bench-Scale Experiments

Work has continued on the program to define the mechanism of regenerator ash deposition and to evaluate alternate potential means of eliminating the problem.

The current work has shown that deposits of CaS-CaSO₄ eutectic occur (in the absence of reducing gas in the regenerator) regardless of whether the CaS is present in char ash or in acceptor. This suggests that desulfurization of fuel char and elimination of ash from the regenerator fuel may not be effective methods of controlling regenerator deposits.

Further studies are planned to define the CO content in regenerator gases necessary to prevent deposits as a function of char type, temperature, and acceptor sulfide content.

Phase III - Pilot Plant Engineering

No work was done on the Rapid City design in April.

Financial

Estimated expenditures in April were \$2,000. Cumulative expenses to date are \$1,665,403.

B. Technical Results and ProgramPhase II - Bench-Scale Research1. Operation of the Continuous Gasification Unit (Project No. 532)a. Run 532-D-14, Operations at 1 atm Pressure

The purpose of this run was to determine the ash deposition behavior of Husky II fuel char at nominally 1 atm system pressure. In the regenerator, O₂ was added to the N₂ fluidizing and carrier gas streams to give an inlet gas composition nearly the same as air. With this limitation on the O₂ content, the total gas flow rate required to give the same fluidized bed expansion as used in the runs at 11 and 20 atm system pressure reduced the heat release to about 60% of normal. The circulating acceptor was recarbonated in the gasifier boat at 1500°F while being fluidized with CO₂. No char was used in the gasifier.

The char combustion proceeded smoothly during the run. However, the char ash stripped from the regenerator bed caused restrictions in the exit gas piping with the result that the system pressure began to increase. After about one hour, the pressure has risen to 12 psig. Since the pressure balance system was not in operation, the run was suspended to prevent rupture of the thin-walled regenerator vessel. The outlet piping was cleaned out and the run was resumed. After the second hour, the pressure had increased again and the run was abandoned. One acceptor cycle had been completed. The restriction also occurs in the routine combustion runs made at 11 or 20 atm system pressure, but the resulting pressure drop across the outlet piping is of no consequence at the higher pressures. Regenerator conditions and results (before the pressure buildup) are shown in Table II.

Inspection of the regenerator revealed a uniform ash deposit on the vessel walls and internals which amounted to 5% of the ash fed. The amount and nature of this deposit show that the gas partial pressures have no major influence on the formation and decomposition of the transient liquid derived from CaS in the fuel char.

b. Run 532-C-12, Production of Desulfurized Fuel Char

In this run, another stock of thoroughly desulfurized Husky II fuel char was made. The feed to the run was a blend of previously gasified product char from Run D-4 and D-5 and the final char bed material drained from the gasifier in Runs D-6 and D-8. Since nearly all of the organic sulfur in this feedstock had been removed, the total sulfur content was almost exclusively in the form of CaS.

As in Run C-11, the char was desulfurized batchwise in the gasifier. Char samples were taken periodically from the gasifier bed by raising the fluidizing velocity temporarily and allowing char to spill over the discharge weir.

The sets of conditions were used in order to obtain some kinetics data for the reaction,



Run Conditions are shown below:

Run	C12A	C12B
Temperature, °F	1310	1300
System Pressure, atm	11	11
% S in Feedstock	1.67	1.67
<u>Exit Gas Composition</u>		
H ₂ O, atm	8.07	7.74
CO ₂	.362	.708
H ₂	2.51	2.43
CO	.06	.17
Total Exit Gas Rate, SCFH	286	290
% S in Final Product	0.09	0.09
% Carbon Burnoff	9	5
Duration of Run, min.	200	90

The rates of desulfurization are illustrated in Figure 1 where the sulfur contents of the char samples are plotted vs time. Both runs were prolonged beyond the time of the last samples shown in Figure 1, but the final sulfur content in both cases was 0.09%. Apparently, there is some refractory organic sulfur in this char which is not removed at the conditions used.

Figure 1 indicates that equilibrium for the reaction can be used for design purposes, in that the required solids retention time can be taken as about 50% greater than the theoretical retention time based on equilibrium in the system under consideration. Equilibrium constants for Reaction (1) are shown below:

Temperature, °F	$K_p = \frac{P_{\text{H}_2\text{O}} P_{\text{CO}_2}}{P_{\text{H}_2\text{S}}}$
1300	68
1400	145
1500	234
1600	490

Lower temperatures favor sulfur rejection and also prevent excessive carbon burnoff.

Gasifier Conditions

As explained last month, the gasifier feedstock for the ash deposition study runs is Disco char. Gasifier conditions and results for the runs reported below are shown in Table I. Since the Disco char has become desulfurized to a low level, the acceptor would not become sulfided while passing through the gasifier. To insure that the acceptor goes through the sulfur cycle, H₂S was added to the gasifier inlet gas beginning with the runs reported this month. The amount added was sufficient to convert about one mol % of the acceptor CaO to CaS on each cycle.

c. Run 532-D-15, Combustion of Colstrip Fuel Char with CO in the Regenerator

This run was a companion to Run D-5 in which Colstrip subbituminous char was burned in the regenerator at 1940°F. In Run D-15 the fuel char rate was increased while holding the same air rate as in Run D-5 in order to operate with about 3% CO in the regenerator product gas.

The run proceeded without incident for 42 cycles, when the desired amount of fuel char had been burned. The regenerator contained a deposit amounting to 0.3% of the ash fed, compared with a 3.0% deposit in Run D-5. This small deposit was localized at the bottom of the vessel walls and internals. Near the air inlet apparently some oxygen reaches the incoming CaS, even when the bulk gas contains 3% CO. The resulting oxidation of CaS to CaSO₄ led to the formation of a small amount of transient liquid.

Analysis of the gasifier product gas for H₂S showed that the acceptor had been sulfided by the H₂S added to the inlet gas to the extent of 0.9 mol % of the CaO during each cycle. After 42 cycles, the acceptor in the regenerator contained essentially no sulfur, showing that the acceptor CaS is rejected normally in the presence of 3% CO in the bulk gas. Regenerator data are in Table II.

d. Run 532-D-16, Operations with Desulfurized Fuel Char and Sulfided Acceptor

This run was a companion to Run D-15B in which desulfurized Husky II char was burned in the regenerator at neutral conditions and at 1870°F. Its purpose was to determine the effects of the acceptor sulfur cycle on ash deposition and on acceptor physical strength.

The run was ended voluntarily after the desired amount of the Run C-12 desulfurized fuel char had been burned. The regenerator contained a massive deposit, uniformly distributed over the vessel walls and internals, which amounted to 3.9% of the ash fed. The acceptor was coated with a thick, soft shell which amounted to 15% by weight of the total acceptor, which corresponds to 20.4% of the ash fed.

This totally unexpected result means that the SO₂ rejected from the acceptor reacts with CaO in the char ash to form CaS and CaSO₄, with consequent formation of the transient liquid. Apparently, inside the char particles, equilibria in the reactions,



are displaced sufficiently to allow formation of the sulfur compounds. In the bulk gas and inside the acceptor particles, CaO is the only stable solid component at the conditions used.

In the desulfurized fuel char most of the calcium in the ash enters the regenerator as CaCO₃, which calcines to form CaO. Analysis of the overhead ash showed that CaS indeed had formed.

The acceptor physical strength was normal in Run D-16, showing that the CaO formed from CaS probably acts as a "cement" for the crystallites. Regenerator data are in Table II.

e. Runs 532-D-17, D-18, and D-19, Operations in Absence of Fuel Char

In these runs no fuel char was used in the regenerator. The normal heat release of 9000 Btu/hr [250,000 Btu/hr (ft³ of bed)] was supplied by combustion of CO with air. The purpose of the runs was to determine, in the light of the Run D-16 results, what contribution the acceptor-derived CaS has on deposit formation in the complete absence of char ash. Regenerator conditions are shown in Table II. In the gasifier, the acceptor was sulfided to the extent shown below:

Run	Mol % Conversion of CaO to CaS per Cycle
D-17	.67
D-18	.99
D-19	1.15

* In the feasibility study, the conversion is 0.91%.

In Run D-17 at neutral conditions and at 1870°F, after two cycles the acceptor could no longer be withdrawn from the regenerator. A massive, uniform deposit had formed on the vessel walls and internals and the acceptor overflow weir was plugged with chunks of deposit which had sloughed off.

The deposit was removed and the run was continued. However, in the third cycle the char-acceptor interface probe in the gasifier failed. The regenerator was inspected during this shutdown and a uniform deposit again was found, but of lesser extent than previously. After removal of this deposit the run was continued through 21 cycles when shutdown was voluntary. The extent of the deposit formation is given below:

Cycle	Deposit, % of Acceptor Fed
0-2	1.2
2-3	0.51
3-21	0.004

These data imply that at neutral conditions the transient liquid formed from acceptor CaS is detrimental only during the first few cycles when the acceptor attrition rate is high, as the sharp edges of the particles are being rounded. At this time the transient liquid cements together the attrited fines.

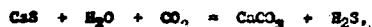
In Run D-18 the CO rate was increased to give 2.4% CO in the regenerator exit gas and the acceptor was sulfided to a greater extent than in Run D-17. Before the combustion period was begun, the regenerator was inspected after all of the char/acceptor had been fed through it and after the initial bed inventory had been calcined at 1800°F. No deposit was found, thereby eliminating the "pre-run" operations as a source of the initial deposit found in Run D-17.

After seven cycles and a voluntary shutdown, no deposits were found in the regenerator, showing that CO in the regenerator also is effective in suppressing the transient liquid during the high attrition rate period.

In Run D-19, the regenerator temperature was raised to 1940°F and CO combustion was carried out at neutral conditions. After seven cycles the regenerator contained a massive deposit but, in contrast with the uniform deposit formed in Run D-17, this material was highly localized in the region four to six inches above the bottom of the vessel. This local deposit was on all the internals as well as on the vessel wall. This result shows that the kinetics of formation and decomposition of the transient liquid is temperature dependent.

1. Discussion and Conclusions

The results of Runs D-16, D-17, and D-19 immediately raise the question of why no regenerator deposits were found in the early operations when the regenerator heat was supplied electrically and when Husky I fuel char was used. The implication is that in these runs the acceptor was not being sulfided in the gasifier. Recent insight gained on the reaction,



in Runs C-11 and C-12 confirms this implication. Further discussion will be deferred until next month when the sulfur contents of the various feed and product streams from all the runs will have become available.

The following conclusions are drawn:

1. Run D-16 proves the futility of desulfurizing the regenerator fuel char as a means of eliminating ash deposits.
2. Runs D-15 and D-18 show that operation of the regenerator with CO in the product gas is an effective way of suppressing the transient liquid, and therefore the ash deposit, regardless of the origin of CaS. Run D-15 was especially important in that it showed that the suppression is effective at temperatures up to 1940°F, and that with Colstrip char, at least, gross ash fusion does not occur at this temperature level.

2. Prognas

Run D-7 (combustion of Husky II char with CO in the regenerator) will be repeated under gasifier conditions where the acceptor assuredly is sulfided.

Subsequent effort will be devoted to finding the minimum CO content of the regenerator product gas as influenced by char type, temperature, and extent of acceptor sulfiding.

2. Batch Kinetic Studies (Project No. 530)

As in the recent past, no new experimental work was undertaken. All effort was devoted to routine processing of samples from the continuous gasification unit. The final report on the Carnegie-Mellon studies has been delayed and is now expected in June.

3. Supporting Patent and Legal Activities (Project No. 534)

The current laboratory reports and memoranda were reviewed for determination of Subject Inventions.

4. Supporting Process and Economic Analysis (Project No. 533)

In Interim Report No. 1, "Review of CO₂ Acceptor Process Development", April 5, 1963, a combined program of bench-scale experiments and economic appraisal was outlined. The combined program was designed to evaluate the most attractive of several commercial solutions to the ash deposit problem.

The detailed calculations required to evaluate the impact on commercial plant (250 MM SCFD) costs are now being made. Most emphasis will be placed on up-dating the basic Feasibility Study issued in 1958, and in evaluation of the incremental cost to operate the regenerator under reducing conditions (at a lower plant thermal efficiency), or to generate producer gas from char and operate the regenerator in the absence of char ash.

Phase III - Pilot Plant Engineering

5. Catalytic Design (Project No. 532.00)

No engineering work was performed under this project during April.

Five additional copies of the "Invitation to Bid" package have been prepared.

C. Subject Inventions

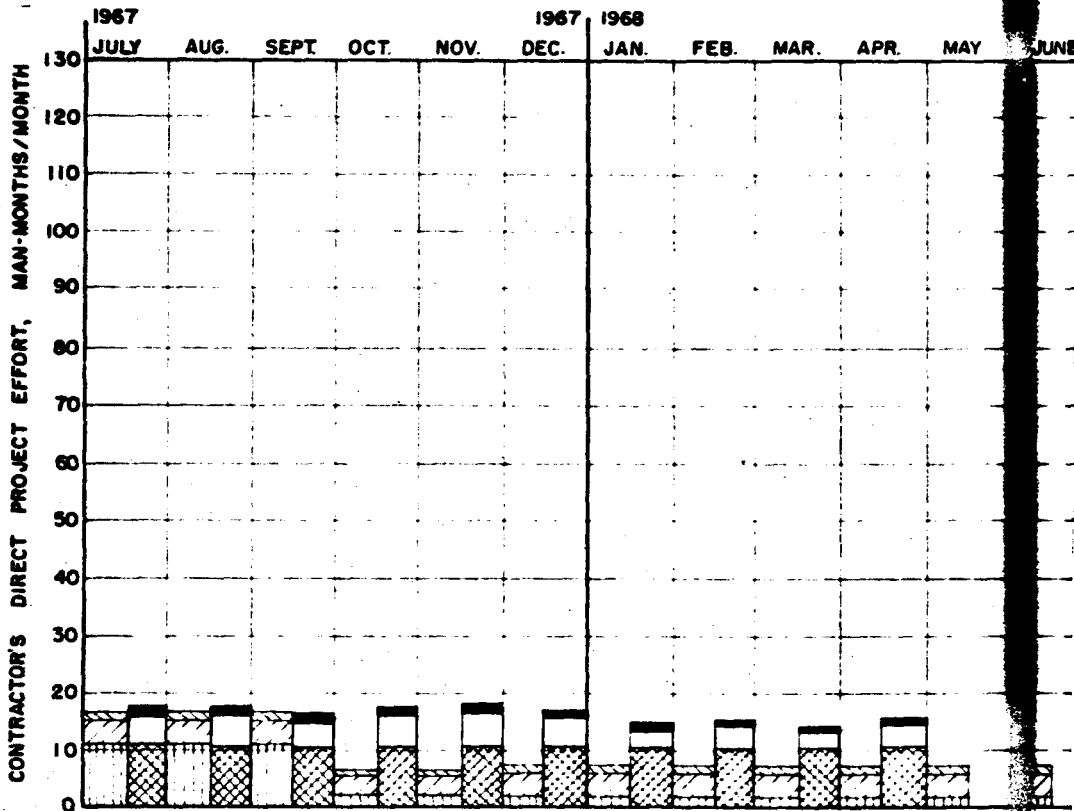
None.

D. Financial Status

<u>Expenses</u>	<u>Actual</u>	<u>Budget</u>
Cumulative through March, 1963	\$ 1,333,103	\$ 1,545,000
Expended in Period (Estimate)	32,350	70,000
Cumulative through April, 1963	\$ 1,365,453	\$ 1,615,000

5/13/63

Att. - Monthly Progress Chart
Tables I and II
Figure 1



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MONTHLY EXPENDITURES*

	JULY	AUG.	SEPT.	OCT.	NOV.	DEC.	JAN.	FEB.	MAR.	APR.	MAY	JUNE
PREDICTED	103.7 M	72.2 M	92.0 M	94.0 M	94.0 M	52.0 M	40.0 M	20.0 M	70.0 M	70.0 M	120.0 M	90.0 M
ACTUAL	78.5 M	78.0 M	106.8 M	84.9 M	97.1 M	93.4 M	70.0 M	29.9 M	92.9 M	32.0 M		

	JULY	AUG.	SEPT.	OCT.	NOV.	DEC.	JAN.	FEB.	MAR.	APR.	MAY	JUNE
PREDICTED	1010.8 M	1083.0 M	1175.0 M	1269.0 M	1363.0 M	1415.0 M	1455.0 M	1478.0 M	1545.0 M	1615.0 M	1735.0 M	1955.0 M
ACTUAL	981.3 M	1059.3 M	1166.1 M	1251.0 M	1348.1 M	1441.5 M	1511.5 M	1541.4 M	1633.4 M	1665.4 M		

CUMULATIVE EXPENDITURES*

* Expenditures Include All Costs & Fees.

MON

CONT

1968						1969						
JUNE	JULY	AUG.	SEPT.	OCT.	NOV.	DEC.	JAN.	FEB.	MAR.	APR.	MAY	JUNE
220.0 M	220.0 M	220.0 M	220.0 M	320.0 M	320.0 M	320.0 M	420.0 M	290.0 M	290.0 M	240.0 M	160.0 M	0.0
1995.0 M	2175.0 M	2395.0 M	2615.0 M	2935.0 M	3255.0 M	3575.0 M	3995.0 M	4285.0 M	4575.0 M	4815.0 M	4975.0 M	505.0 M

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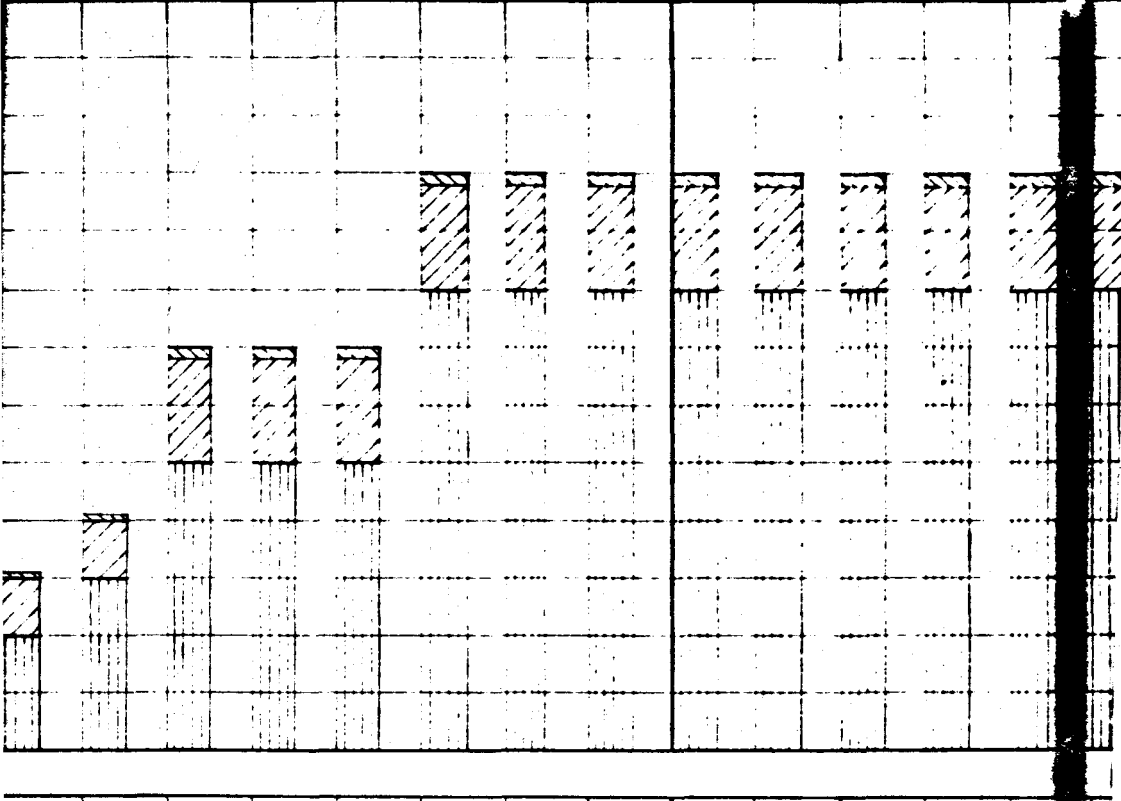
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MONTHLY PROGRESS CHART

CONTRACT No. 14-01-0001-415

1969 1970

MAY JUNE JULY AUG. SEPT. OCT. NOV. DEC. JAN. FEB. MAR. APR. MAY JU



160.0 M 480.0 M 100.0 M 100.0 M 100.0 M 150.0 M 150.0 M 150.0 M 125.0 M 125.0 M 125.0 M 125.0 M 125.0 M 125.0 M

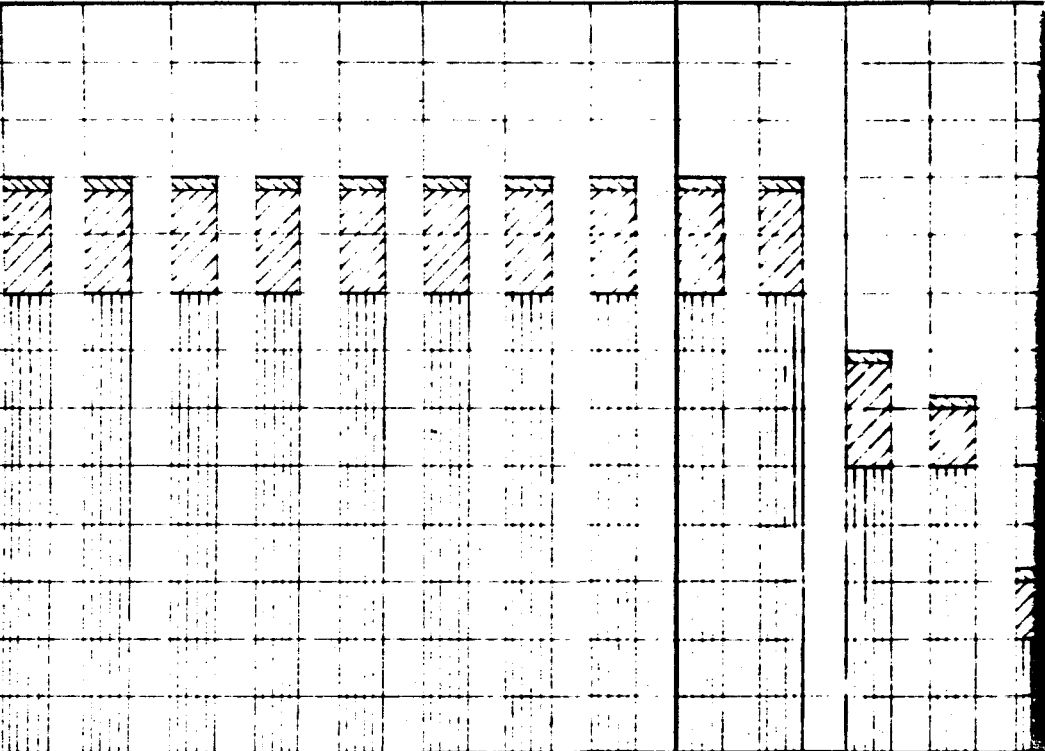
MAY JUNE JULY AUG. SEPT. OCT. NOV. DEC. JAN. FEB. MAR. APR. MAY

4975.0 M 5455.0 M 5555.0 M 5655.0 M 5755.0 M 5905.0 M 6055.0 M 6205.0 M 6330.0 M 6455.0 M 6580.0 M 6705.0 M 6830.0 M

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1970 1971
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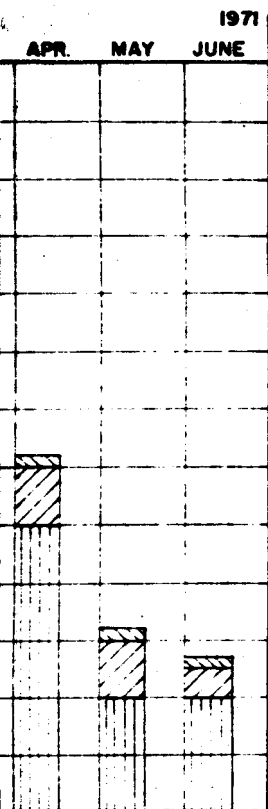
125.0 M 125.0 M 125.0 M 125.0 M 125.0 M 125.0 M 125.0 M 125.0 M 125.0 M 125.0 M 25.0 M 125.0 M

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6830.0 M 6955.0 M 7080.0 M 7205.0 M 7330.0 M 7455.0 M 7580.0 M 7705.0 M 7830.0 M 7955.0 M 8080.0 M 8205.0 M

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DATE: April, 1968



PREDICTED ACTUAL

SUPERVISING

PROFESSIONAL ⊕

TECHNICIAN ⊕⊕

⊕ Includes Supervisory Exempt Technicians.
 ⊕⊕ Includes Clerical.

MONTHLY PROGRESS CHART

OFFICE OF COAL RESEARCH
 DEPARTMENT OF THE INTERIOR

CONSOLIDATION COAL CO.
 RESEARCH DIVISION LIBRARY, PA.

CONTRACT No. 14-01-0001-415

Run No. 530-

Cycle

Acceptor

Acceptor Circulation Rate (raw basis), lb/hr
 System Pressure, atm.

Gasifier Temperature, °F
 Char

Char Feed Rate

Inlet Gas (Bottom of Bed)

Steam, SCFH

Recycle, "

Fluidising Gas

Char Carrier Gas (Middle of Bed)*

Added N₂, SCFH

CO₂, "

N₂, "

Composition, Partial Pressure, atm.

H₂O

H₂

CH₄

CO

CO₂

H₂

Outlet Gas (Top of Bed) Comp., Partial Pressure, atm.

H₂O

H₂

CH₄

CO

CO₂

H₂

Dry Exit Gas, SCFH

H₂ Purge, "

H₂, "

Condensate, "

Recycle Gas Composition, mol%

H₂

CH₄

CO

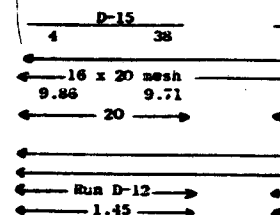
CO₂

H₂

Fluidising Velocity, ft/sec

Boot

Bed Avg.



4	P-15	38	-
9.86	16 x 20 mesh	9.71	-
20	Gasifier Temperature, °F		-
1.45	Char Feed Rate		-
87.9	Steam, SCFH	84.1	10
270.3	Recycle, "	261.5	12
57.3	Fluidising Gas	55.4	3
22.3**	Char Carrier Gas (Middle of Bed)*	22.3**	2
16.4	Added N ₂ , SCFH	12.4	1
1.5	CO ₂ , "	1.5	-
4.41	N ₂ , "	4.40	4
7.51	H ₂ O	7.20	3
0.38	H ₂	0.74	0
0.62	CH ₄	1.25	0
1.12	CO	1.42	1
5.76	CO ₂	4.99	1
3.95	H ₂ O	4.04	3
7.93	H ₂	7.31	3
0.71	CH ₄	0.89	0
0.76	CO	1.51	0
0.37	CO ₂	0.93	0
6.28	H ₂	5.32	1
44.4	Dry Exit Gas, SCFH	42.6	41
17.1	H ₂ Purge, "	13.8	11
-	H ₂ , "	-	-
87.5	Condensate, "	87.4	107
47.15	Recycle Gas Composition, mol%	44.02	51
4.25	H ₂	5.38	2
4.52	CH ₄	9.11	6
2.18	CO	3.59	6
41.9	CO ₂	35.90	32
.627	H ₂	.601	.7
.245	Boot	.238	.3

* Contributes to outlet gas only.
 ** Includes 0.20 SCFH H₂S.
 *** Includes 0.27 SCFH H₂S.

TABLE I

Gasifier Conditions and Results

D-16		D-17		D-18	D-19
2	23	6	13	5	3
Tymochtee-6 Dolomite					
16 x 28 mesh					
9.38	11.98	10.38	11.70	10.34	9.52
11		11		11	20
1500					
Disco Char					
Run D-15		Run D-16		Run D-17	Run D-18
1.06		1.03		1.10	2.51
28.6	108.4	108.5	103.7	104.9	86.4
24.9	123.9	117.9	109.8	109.8	283.1
35.0	34.7	36.7	34.1	34.2	56.3
21.2**	21.2**	21.2**	23.4**	21.9**	23.0**
16.4	13.0	16.4	20.5	21.5	18.2
1.8	1.8	1.8	1.8	1.8	1.8
4.38	4.45	4.44	4.40	4.45	4.40
3.47	3.40	3.65	3.32	3.25	7.48
0.14	0.14	0.11	0.12	0.12	0.45
0.31	0.20	0.22	0.29	0.32	1.00
1.00	0.78	0.87	1.26	1.30	1.51
1.69	2.03	1.71	1.61	1.56	5.16
3.97	4.14	3.89	4.07	4.02	3.93
3.88	3.64	4.26	3.70	3.75	7.88
0.21	0.20	0.18	0.19	0.20	0.56
0.46	0.29	0.33	0.47	0.52	1.25
0.51	0.35	0.29	0.62	0.63	0.72
1.97	2.38	2.05	1.95	1.88	5.68
41.5	35.2	42.1	41.9	45.7	43.1
11.6	11.6	12.2	12.0	12.8	14.5
87.5	109.6	101.1	102.3	102.4	85.1
51.92	49.78	56.18	49.92	50.06	47.07
2.83	2.79	2.31	2.50	2.68	3.34
6.20	4.01	4.38	6.30	6.96	7.47
6.85	4.82	3.83	8.38	8.41	4.32
38.20	38.60	33.30	32.99	31.9	37.8
.780	.768	.755	.742	.744	.618
.302	.297	.291	.284	.286	.241

2

Run No. 332-

Duration, Total Cycles
Hours

Acceptor

Size, Mesh
Circulation Rate
lb/hr (raw basis)

System Pressure, atm

Regenerator

Temperature, °F
Fuel Char

Feed Rate lb/hr
lbs fed

Inlet Gas

Recycle, SCFH
Added N₂, SCFH
CO₂, SCFH
CO, SCFH
Air, SCFH

Recycle Gas Composition, Mole %

N₂
CO₂
CO
O₂

Air in Inlet Gas, Mole %

CO₂ Partial Pressure, atm
Equilibrium CO₂ Partial Pressure, atm
ΔP Driving Force, atm

Fluidizing Velocity, ft/sec

Acceptor Activity
Corresponding Cycle No.

* O₂ flow
** O₂ in inlet gas, mole %

D-14

1
2.05

16 x 28

8.83

1.34

1670

Husky II
Lignite Char

0.73
1.30

None

42.5
None

None

11.7*

Balance

33.4
.3-1.6
0

19.7**

0.45
4.90
4.45

2.47

1.0
1

19.1
4.00
7.60
3.60
1.51
1.35
35

Ty

Busk

None

Balance

20.0
2.9
0

19.1

4.00
7.60
3.60

1.51

1.35
35

1

TABLE II

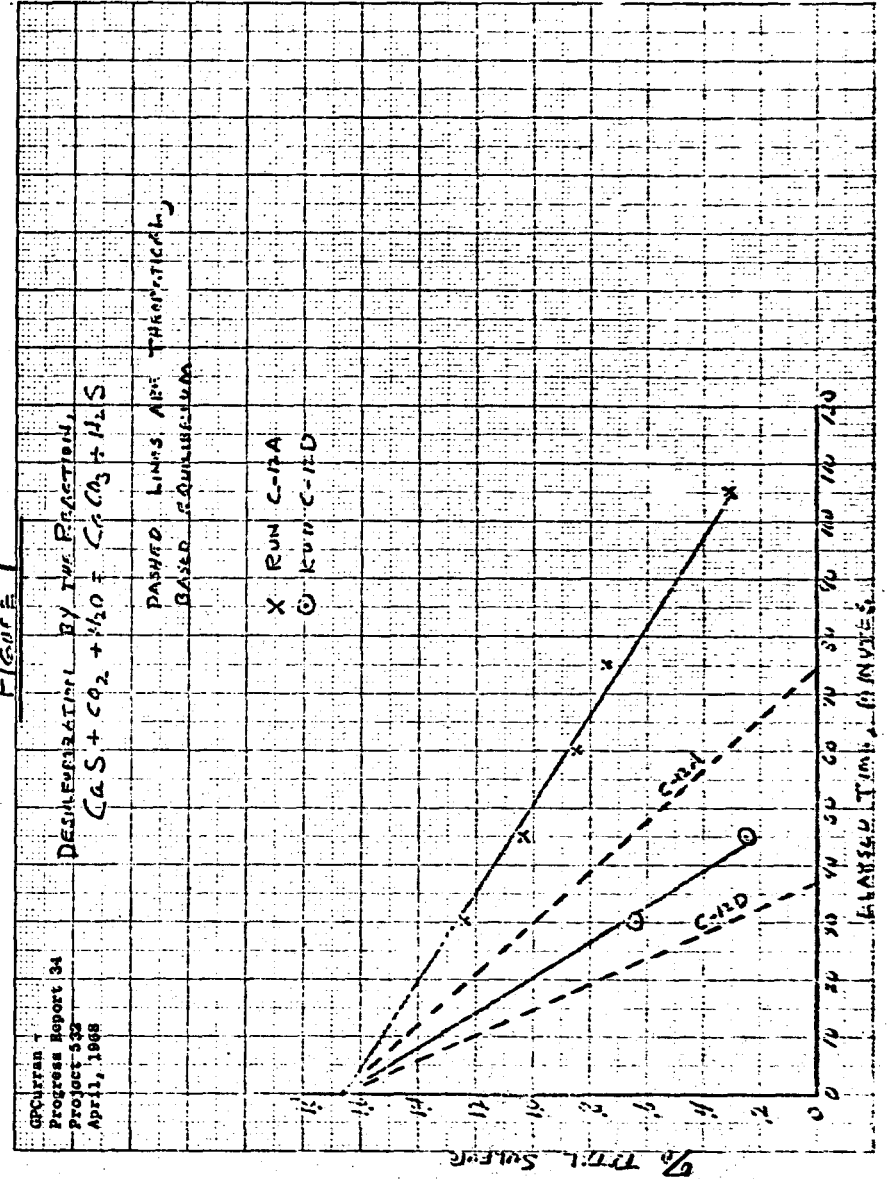
Regenerator Conditions and Results

D-16	D-17	D-18	D-19
31	21	7	7
19.8	15.0	6.8	6.3
Tymcoches-6			
16 x 20	Dolomite	16 x 28	16 x 28
12.00	10.58	10.06	10.52
11	11	11	20
1870	1870	1870	1940
Easy II Lignite Char		None	
(Run c-12)			
1.04	-	-	-
20.7	-	-	-
197.6	178.5	195.7	249
30	34.8	36.0	35
5.7	0	0	0
	28.0	31.6	25.0
95	66.0	66.0	67.0
Balance	Balance	Balance	Balance
22.6	29.2	27.9	28.7
.3-.6	0.3	2.4	0.7
0	0	0	0
29.8	21.5	20.1	17.6
2.49	3.21	3.07	5.22
4.90	4.90	4.90	7.60
2.41	1.68	1.83	2.38
1.51	1.46	1.56	1.10
.50	.79	.79	.76
25	9	4	7

2

COAST BROS. COMPANY, INC. BOSTON, MASSACHUSETTS.

NO. 316 25 DIVISIONS PER INCH BOTH WAYS. 140 BY 200 DIVISIONS.



McQuinn
 Progress Report 34
 Project 532
 April, 1968

CCDC-MPR-48

PROGRESS REPORT NO. 48
OCR CONTRACT NO. 14-01-0001-415(2)
CONSOL LIGNITE GASIFICATION PROCESS DEVELOPMENT

May 1 to 31, 1968

A. Summary

Phase I - Feasibility Study

Complete.

Phase II - Bench-Scale Experiments

Further studies were made on the efficacy of operating the regenerator with a reducing outlet condition (CO in offgas) in preventing ash deposits. It was demonstrated that this technique was successful in maintaining operability at 1870°F with a high sulfur fuel char (Husky II char) even when the acceptor feed was presulfided. The limiting temperature at which this technique could be successfully applied with Husky II char was determined to be about 1940°F.

Studies were made with a sub-bituminous char (Colstrip) on the possibility of operating the gasifier as a gas producer at 20 atm. and 1700°F. The interest here is in the possibility of the avoidance of ash fusion problems by means of a two-stage regeneration process. The tests were unsuccessful due to slugging of the ash at the point of air injection. Higher fluidizing velocities and the presence of an endothermic gasifying agent may be helpful in establishing operability. Such tests will not be carried out, however, because of lack of time.

Some results on hydro devolatilization and integral gasification kinetics of Colstrip char are reported. These were obtained incidental to feedstock preparation for the gas producer run.

An unsuccessful attempt was made to confirm earlier indications that the presence of sodium stabilizes the acceptor against activity loss.

Phase III - Pilot Plant Engineering

An evaluation of the impact on commercial plant economics of operating the regenerator under reducing conditions has been completed. An incremental cost of about 3¢/MM BTU pipeline gas was calculated.

Evaluation of the Catalytic Construction pilot plant design was continued. The investment associated with staged construction of various portions of the plant has been calculated.

Financial

Estimated expenditures in May were \$28,000. Cumulative expenses to date are \$1,699,164.

Lignite Gasification
Progress Report No. 48

-2-

May, 1968

B. Technical Results and Program

Phase II - Bench-Scale Research

1. Operation of the Continuous Gasification Unit (Project No. 532)

a. Acceptor Sulfiding During Early Operations

As mentioned last month, it had become apparent that in most of the runs made before Run D-15 (April, 1968), when H₂S was added purposefully to the gasifier, the acceptor was not being sulfided. Sulfur balances around the gasifier, coupled with the partial pressures of H₂O and CO₂, show that in general the equilibrium in the reaction,



was displaced toward the left and therefore little or no sulfur was picked up by the acceptor. In only three runs was the acceptor sulfided appreciably. These conclusions were confirmed by the sulfur analyses of the acceptor, sampled periodically from the gasifier during all the runs.

The main reason for this situation is, that in order to conserve the hydrodevolatilized feedstocks to the gasifier, low feed rates were used with the result that relatively little total sulfur was present in the gasifier. Fortunately, in the first two runs (Runs A-25 and A-26) made with Husky II fuel char the gasifier char feed rate was increased above the normal rate. This was especially so in Run A-25, made at 1600°F, when the feed rate was increased by a factor of about four in anticipation of the enhanced gasification kinetics. The third case of appreciable sulfiding occurred in Run D-10B when fresh Disco char was used as gasifier feed. With the low lime content of Disco ash most of the sulfur was rejected rather than being retained by the char ash, as was generally the case with the lime-rich Western coal feedstocks.

A summary of the sulfur loading to the regenerator and the extent of ash deposits for all the runs made at neutral or excess O₂ conditions in the regenerator is shown in Table I.

As in the past two months, the gasifier feedstock was Disco char used in previous runs. H₂S was added to the inlet gas to give 0.7 wt % conversion of the acceptor CaO to CaS on each cycle. Conditions and results for all the acceptor runs made this month are shown in Table II.

b. Run 532-D-20, Repeat of Run D-7, with Sulfided Acceptor

Run D-7 was the first in which a deficiency of air was used in the regenerator to provide CO in the product gas. However, little or no sulfiding of the acceptor occurred in this run. The purposes of Run D-20 were to determine what impact the additional acceptor CaS would have on the amount of CO required to suppress formation of the transient liquid and to determine the effect of the acceptor sulfur cycle on activity.

During the first six cycles, the average CO content in the regenerator product gas was 2.6%. On the sixth cycle, a shutdown was required to unplug the steam generator inlet line which had become choked with scale caused by use of H₂S in the inlet gas. Inspection of the regenerator showed a uniformly distributed deposit amounting to 4.3% of the ash fed. After removal of the deposit, the run was resumed with the CO content increased to 3.1%. On the eleventh cycle another shutdown occurred when the fuel char transfer line plugged because of a pressure upset. Inspection of the regenerator showed no deposits. Since too little char had been burned to prove conclusively that no long-term deposit might have formed, the run was continued with the CO content raised to 4.0%. The run was ended voluntarily after 52 cycles when the desired amount of fuel char had been burned. No deposits of any kind had formed.

Regenerator conditions and results are in Table III. This run was important because it showed that the acceptor activity is at least as good, with sulfiding of the acceptor, as in Runs D-7 and A-21 in which little or no sulfiding occurred. The data are plotted in Figure 1.

c. Run 532-D-21 - Effect of Sodium on Acceptor Activity

In Run A-17, made with high-sodium Glenharold char, the acceptor had picked up 1 mol % Na₂O after 27 cycles, and the activity was the highest observed up to that time.

Run D-21 was made to determine whether purposeful impregnation of the acceptor would provide enhanced activity. The Tycocote dolomite in the MgO.CaCO₃ formed was impregnated with an aqueous solution of Na₂CO₃ to give a 1.0% Na₂O content on the fully calcined (MgO.CaO) basis.

During the initial circulation of the impregnated acceptor in the MgO.CaCO₃ form, the attrition rate of the acceptor as measured by the amount of overhead fines removed by the regenerator cyclone, was unusually high. With the hope of stabilizing the acceptor, the entire inventory was circulated through the unit three more times, until the attrition rate had dropped to nearly zero.

After the first combustion cycle, the acceptor activity had dropped to 0.72, the lowest value ever observed at this stage for a dolomite acceptor. From about the seventh cycle the rate of decline in activity was about the same as for an unimpregnated acceptor. The reason for the abrupt loss of activity during the early circulation is not apparent yet. The activity data are plotted in Figure 1 and the regenerator conditions and results are shown in Table III.

d. Run 532-D-22 - Operations with Husky II Char at 1940°F

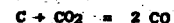
This run was made to determine the efficacy of the CO mode of regenerator operation for Husky II char at the higher temperature of 1940°F. It was therefore a comparison run to Run D-15, made with Colstrip char.

During Run D-22A, as in all the runs made with 3% or more CO in the regenerator product gas, a pronounced temperature gradient of about 50°F existed across the regenerator bed, with the highest temperature about 2-4 inches above the bottom where the air, char, and recycled CO enter the vessel. At neutral conditions the gradient is typically no more than 10°F. The gradient apparently is caused by rapid combustion of the incoming CO and O₂ near the inlet. Then the upper part of the bed tends to be chilled by the endothermic reactions



During the fifth cycle the temperature at the bottom of the bed suddenly increased to 2000°F, indicating a buildup of an agglomerate which had interfered with the normal flow pattern of the fluidized bed. To prevent acceptor deactivation by exposure to this high temperature, the run was discontinued. Inspection of the regenerator showed that a conglomerate of transient liquid deposit, acceptor and fused ash, had indeed formed in the lower six inches of the vessel.

For the D-22A conditions, if the assumption is made that all the CO entering with the recycle gas is burned immediately to CO₂, then carbon and oxygen balances show that nearly one-half of the carbon consumed in the regenerator is by the reaction,



In Run D-22B, after removal of the deposit, the amount of recycle gas was decreased and the amount of added CO₂ and N₂ was increased to the maximum allowed by the existing rotameters. The result of the changes was to decrease the amount of CO burned by a factor of two. The temperature profiles reflected this change, and the gradient was within 10°F. The run proceeded without incident while holding the CO content between 3.5 and 4.0% (average 3.77%) until the fifteenth cycle, when the supply of fuel char was exhausted. The regenerator contained no deposit derived from the transient liquid but a small aggregate of acceptor, cemented together with a brown, glassy material was found on the internals about six inches from the bottom. Thus, 1940°F is about the borderline of operability with respect to gross ash fusion for the Husky II char. In Run D-15, with Colstrip char at 1940°F, no gross ash fusion occurred.

e. Run 532-II-17 - Additional Stock of Hydrodevolatilized Colstrip Char

As in previous runs with sub-bituminous coal, the raw material was carbonized at 925°F in the five foot diameter rotary kiln prior to hydrodevolatilization. In this run, 350 lb. of the LTC char was fed. Conditions and results are shown in Table IV. This feedstock was a new shipment (Colstrip II). Analytical data on the new material are not yet complete and will be reported next month.

f. Run 532-K-3 - Gasification of Colstrip II Char

This run was made to provide a stock of pregasified char to be used in the succeeding run in which the gasifier was operated as a producer.

To obtain some gasification kinetics data, six different conditions were used which involved operation at 1500 and 1600°F and 11 and 20 atm. system pressure. The inlet H₂/H₂O ratio also was varied somewhat. Conditions and results are shown in Table V. Rate data will be reported next month, after the bed carbon inventories have been calculated.

g. Run 532-P-1 - Operation of the Gasifier as a Producer

The main purpose of this run was to determine the amount and nature of any ash deposit which may form under producer gas conditions. The gasifier was operated at 1700°F and 20 atm. pressure with recycle product gas and air as the inlet gas. Recycle gas was used to simulate the upper section of a tall fluidized bed. At this condition the air had to be injected through a separate inlet line and thus entered the bed at 20 atm. pressure. Dilution by mixing with the recycle gas was not possible since combustion of the CO would have melted the preheating coil.

In Run P-1A all the air was added at the bottom of the gasifier. After feeding 27 lb. of Run K-3 char in 6.3 hours, the temperature profile showed that an obstruction had occurred near the bottom of the vessel. Examination of the vessel showed that a dense, glassy blob of slagged ash had formed in the region surrounding the air inlet tube. The slagged ash amounted to 22% of the ash fed.

In Run P-1B, another air inlet tube was installed to allow about one-half of the total air to be injected halfway up the bed. After feeding 37 lb. of char in 9.9 hours, the same situation developed as in Run P-1A. Examination of the vessel showed two nearly identical deposits in the region around both diptubes. The total slagged ash amounted to 27% of the ash fed. Run conditions and results are in Table VI. Other than the slagged ash, no deposits of any kind were found on the vessel walls, showing that producer conditions do not introduce a new breed of deposit. Slagging at the point of air injection is the principal problem to be resolved. The presence of an endothermic gasification agent, i.e., CO₂, H₂O or CaCO₃ mixed with the air may be helpful in reducing this problem.

h. Program

Previous experience has shown that raw, dry sub-bituminous coal cannot be fed to the system at hydrodevolatilizer conditions without the occurrence of massive coking. The next operations will be devoted to determining what the minimum preoxidation conditions are which will prevent coking.

After establishing these conditions, a run will be made in which the preoxidized coal will be fed directly to the gasifier, to simulate single vessel operation.

All experimental work will cease on June 28 and report writing will begin immediately thereafter.

2. Batch Kinetic Studies (Project No. 530)

The batch differential rate data for the steam-carbon reaction at 1500°F were reviewed critically, revised, and recorrealted in preparation for a paper to be given at the September ACS meeting.

The final report on the Carnegie-Mellon studies has been delayed again by pressure of academic duties, but it is expected in July.

3. Supporting Patent and Legal Activities (Project No. 534.00)

The current laboratory reports and memoranda were reviewed for determination of Subject Inventions.

4. Supporting Process and Economic Analysis (Project No. 533)

In April, 1968, a combined program of experimental bench-scale work directed toward solution of the ash deposition problem and an evaluation of the commercial aspects of these studies was authorized. The most promising solution discovered in the bench scale work was to operate the acceptor regenerator under reducing conditions with 3-4 volume per cent CO in the regenerator offgas. Under these conditions, the formation of the transient liquid phase is inhibited and the ash deposition problem is eliminated. There is, of course, some additional carbon loss in the regenerator. For a fixed output of pipeline gas, more lignite is required, and the plant operates at a lower overall thermal efficiency.

The evaluation of the commercial-scale economics of this mode of operation was completed in May. Results will be issued in a formal report to OCK in early June.

This solution to the ash deposition problem increased the cost of synthetic pipeline gas (at 250 MM SCF/D) by about 3¢/MM BTU.

Phase III - Pilot Plant Engineering

5. Pilot Plant Design (Project No. 533.30)

The major work done during May was involved with evaluating the Catalytic Construction Company engineering package and investigating various alternates in plant design before making final recommendations to OCK.

Primarily Consol is considering building the Rapid City pilot plant in stages, in order to minimize investment risk until basic operability is established.

Basically, Consol is considering omitting devolatilization, gas cleanup, gas shift conversion and final methanization from the first version to be built.

A meeting was held with OCR to evaluate the primary gasification vessel design and hot-piping systems. An OCR alternate was also reviewed. The conclusion to be discussed in a future memo.

C. Subject Inventions

None.

D. Financial Status

<u>Expenses</u>	<u>Actual</u>	<u>Budget</u>
Cumulative through April, 1968	\$ 1,671,164	\$ 1,615,000
Expended in Period (estimate)	28,000	120,000
Cumulative through May, 1968	\$ 1,699,164	\$ 1,735,000

Att. - Monthly Progress Chart

Table I

Summary of Regenerator Sulfur Data

All runs made at neutral or excess O₂ conditions.

<u>Run</u>	<u>Special Features</u>	<u>Regen. Temp., °F</u>	<u>Grams/hr Sulfur Fed to Regenerator</u>			<u>Deposit, % of Ash Fed</u>
			<u>With Acceptor</u>	<u>With Fuel Char</u>	<u>Total</u>	
A-25	-	1890	6.4	6.4	12.8	26
D-10B	-	1910	10.0	5.9	15.9	35
D-16	desulf. char	1870	5.1	0.4	5.5	36
A-26	-	1870	1.0	5.7	6.7	8.0
D-8	low temperature	1830	~0	7.7	7.7	2.7
D-8	excess O ₂	1870	~0	7.4	7.4	2.0
D-9	-	1870	~0	6.7	6.7	6.0
D-11	increased velocity	1870	~0	8.0	8.0	5.8
D-12	added fines	1870	~0	8.2	8.2	18
D-13B	"no sulfur"	1870	~0	0.4	0.4	.08
<u>Husky I Char</u>						
A-12	-	1940	~0	2.6	2.6	0
A-20	-	1940	~0	2.8	2.8	0
A-21	-	1870	~0	2.7	2.7	0
A-22	-	1870	~0	3.1	3.1	0
<u>Glenharold Char</u>						
A-17	hi-sodium char	1940	~0	2.0	2.0	18
A-24	water extracted	1940	~0	2.8	2.8	18
<u>Colstrip Char</u>						
D-1C	-	1910	~0	1.9	1.9	.04
D-5	-	1940	~0	2.0	2.0	3.0
D-3	-	1910	~0	0.7	0.7	0

TABLE II
Gasifier Conditions and Results

Run No. 332 [*]	4	20	10
Cycle			
Acceptor	16 x 28 Mesh Tymnchtee 6 Dolomite		16 x 28 Mesh Tymnchtee 6
Acceptor Circulation Rate (raw basis) lb/hr	10.15	8.42	11.91
System Pressure, atm.	← 11 →		← 1500 →
Gasifier Temperature, °F	← Run D-19 →		← Disco Char →
Char	← 2.16 →		
Char Feed Rate			
<u>Inlet Gas (Bottom of Bed)</u>			
Steam, SCFH	103.9	100.1	91.9
Recycle, SCFH			
Fluidizing Gas	110.1	109.5	100.0
Char Carrier Gas (Middle of Bed)	34.2	34.1	35.9
Added H ₂ , SCFH	21.2**	21.2**	21.2**
CO ₂	20.5	15.7	19.4
H ₂	1.8	1.8	1.8
<u>Composition, Partial Pressure, atm.</u>			
H ₂ O	4.44	4.44	4.31
H ₂	3.28	3.30	3.69
CH ₄	0.11	0.13	0.10
CO	0.32	0.32	0.31
CO ₂	1.28	1.14	1.26
H ₂	1.57	1.77	1.42
<u>Outlet Gas (Top of Bed)</u>			
<u>Composition, Partial Pressure, atm.</u>			
H ₂ O	4.05	4.03	3.83
H ₂	3.74	3.65	4.22
CH ₄	0.17	0.20	0.17
CO	0.50	0.50	0.50
CO ₂	0.63	0.53	0.56
H ₂	1.91	2.79	1.72
Dry Exit Gas, SCFH	41.1	41.4	40.7
H ₂ Purges,	11.3	12.6	9.7
H ₂			
Condensate,	101.5	99.6	89.0
<u>Recycle Gas Composition, mole %</u>			
H ₂	50.55	44.76	55.52
CH ₄	2.33	2.66	2.20
CO	6.70	6.64	6.64
CO ₂	8.52	7.14	7.44
H ₂	31.90	34.60	28.20
<u>Fluidizing Velocity, ft/sec.</u>			
Boot	0.737	0.711	0.670
Bed, avg.	0.263	0.276	0.260

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*Contributes to outlet gas only
**Includes 0.2 SCFH H₂S

TABLE II
Conditions and Results

	D-21 14	43	D-22 10
1 Mesh Tynochtee 6 Dolomite Impregnated with Na_2CO_3			16 x 28 mesh Tynochtee Dolomite
11.91	12.87	14.39	17.38
←----- 11 ----->			
←----- 1500 ----->			
←----- Disco Char ----->			
←----- Run D-20 ----->			←----- Run D-21 ----->
	3.15		2.84
91.9	95.6	84.5	91.0
80.0	100.5	92.3	267.9
35.9	35.7	33.6	57.4
21.2**	21.2**	21.2**	22.3**
19.4	18.3	14.4	17.0
1.8	1.8	1.8	1.8
4.31	4.43	4.34	4.55
3.60	3.56	3.37	7.65
0.10	0.11	0.10	0.42
0.31	0.31	0.38	0.95
1.26	1.22	1.22	1.37
1.42	1.37	1.69	5.08
3.83	3.86	3.82	4.90
4.22	4.18	3.72	8.13
0.17	0.17	0.16	0.52
0.50	0.50	0.62	1.18
0.56	0.61	0.78	0.64
1.72	1.68	1.90	5.53
0.7	42.4	44.8	43.7
9.7	10.0	12.4	14.4
5.0	91.3	84.2	88.7
5.52	55.28	48.35	48.82
2.20	2.29	2.11	3.12
5.64	6.62	8.02	7.99
7.44	8.01	10.12	3.87
5.20	27.80	31.70	37.10
0.670	0.679	0.613	0.630
0.260	0.264	0.242	0.246

2

TABLE II

Regenerator Conditions

Run No. 532-	D-20		
	A	B	C
Duration: Cycles Hours	0 thru 6 5.53	7 thru 11 2.57	11 thru 11
Acceptor Size, mesh Circulation Rate, lb/hr (raw basis)	←----- Tynochtee-6 Dolomite ----->		
	←----- 16 x 28 ----->		
	10.15	10.35	3.4
System Pressure, atm	←----- 11 ----->		
Regenerator Temperature, °F	←----- 1870 ----->		
Fuel Char	←----- Hus ----->		
Feed Rate, lb/hr lbs fed	←----- +65 mesh ----->		
	1.29 7.15	1.37 3.94	1.4 24.3
Inlet Gas Recycle, SCFH Added N_2 , CO_2 , Air,	170.1 20.0 3.6 94.0	185.0 20.0 2.5 89.3	170.1 20.0 7.1 98.5
Recycle Composition, mol % N_2 CO_2 CO O_2	Balance 23.5 2.57 0	Balance 23.5 3.07 0	Balance 23.5 3.98 0
Air in Inlet Gas, mol %	21.68	30.22	30.00
CO_2 Partial Pressure, atm	2.59	2.59	2.5
Equilibrium CO_2 Partial Pressure, atm	4.90	4.90	4.9
ΔP Driving Force, atm	2.31	2.31	2.3
Fluidizing Velocity, ft/sec	1.51	1.51	1.5
Acceptor Activity	.59	.51	.7
Corresponding Cycle	4	9	20

1

and Results

	D-21	D-22	
		A	B
52	44	0 thru 5	5 thru 16
	31.15	6.17	10.10
	Sodium Impregnated		
→	Tynochtee-6 Dolomite	← Tynochtee-6 Dolomite →	
	16 x 28	← 16 x 24 →	
	11.91	9.76	11.16
→	11	← 20 →	
→	1870	← 1940 →	
→	II Lignite Char		
→	-45 M Runs D-6,7,8,9	48x65 M Runs D-6,7,8,9	
	1.37	1.38	1.26
	42.56	8.54	12.72
	179.3	314	176
	20.0	36	84
	5.7	12.4	31
	88.5	94.0	97.8
	Balance	Balance	Balance
	23.5	22.3	21.3
	2.70	3.41	3.77
	0	0	0
	30.10	20.99	25.15
	2.59	4.46	4.26
	4.90	7.30	7.30
	2.31	2.84	3.04
	1.50	1.32	1.12
	.64	.30	.63
	10	3	14

2

TABLE IV
RUN CONDITIONS AND RESULTS

Run No.	532-N-17
System Pressure, atm.	20
Temperature, °F	1525
Feedstock	LTC Colstrip Subbituminous Char
Feed Rate	7.87
<u>Inlet Gas (Bottom of Bed)</u>	
Steam, SCFH	90.3
Recycle, "	
Fluidizing Gas	189.1
Added H ₂ , SCFH	17.3
N ₂ , "	5.4
<u>Composition, Partial Pressure, atm.</u>	
H ₂ O	5.98
H ₂	6.75
CH ₄	2.09
CO	2.32
CO ₂	1.03
N ₂	1.83
<u>Outlet Gas (Top of Bed)</u>	
<u>Composition, Partial Pressure, atm</u>	
H ₂ O	4.08
H ₂	7.29
CH ₄	2.72
CO	3.01
CO ₂	1.34
N ₂	1.56
Dry Exit Gas, SCFH	99.08
N ₂ Purge, "	6.3
Condensate	72.27
<u>Recycle Gas Composition, mole %</u>	
H ₂	44.79
CH ₄	16.70
CO	18.51
CO ₂	8.24
N ₂	11.76
Avg. Fluidizing Velocity, ft/sec.	.194
Avg. Vapor retention time, sec.	16.1
<u>Yields</u>	
Char, Wt. % MF Feed	68.4
Carbon Bearing Gases,	
lb C/100 lb MF Feed	16.98
CH ₄	6.53
CO	7.23
CO ₂	3.22
Product particle density, lb/ft ³	77.5

TABLE V
Run Conditions and Results

Run No. 532-K-3	A	B	C	
System Pressure, atm.	69	30	11	
Gasifier, Temperature, °F	1600	1600	1900	
Char	← Hydrodevolatilized C			
Char Feed Rate, lb/hr	4.32	5.07	4.14	
<u>Inlet Gas (Bottom of Bed)</u>				
Steam, SCFH	124.2	141.6	118.2	
Recycle, "				
Fluidizing Gas	284.2	284.1	65.5	
Char Carrier Gas				
(Middle of Bed)	54.1	54.5	33.4	
Added H ₂ , SCFH	14.7	9.8	5.0	
CO ₂ , "	5.2	0	0	
N ₂ , "	2.0	2.0	2.0	
<u>Composition, Partial Pressure, atm.</u>				
H ₂ O	5.78	6.96	6.83	
H ₂	6.40	6.03	2.30	
CH ₄	0.78	0.78	0.13	
CO	3.08	2.82	0.65	
CO ₂	1.57	1.33	0.57	
N ₂	2.39	2.10	0.62	
<u>Outlet Gas (Top of Bed)</u>				
<u>Composition, Partial Pressure, atm.</u>				
H ₂ O	3.73	4.03	3.58	
H ₂	7.27	7.37	3.95	
CH ₄	1.00	1.01	0.28	
CO	3.92	3.74	1.14	
CO ₂	1.70	1.78	1.18	
N ₂	2.38	2.09	0.67	
Dry Exit Gas, SCFH	90.6	97.2	78.0	
N ₂ Purges, "	13.8	13.6	8.5	
Condensate, "	95.2	99.0	81.2	
<u>Recycle Gas Composition, mole %</u>				
H ₂	42.23	44.58	50.69	
CH ₄	5.94	6.12	3.56	
CO	23.34	22.63	17.17	
CO ₂	10.09	10.67	15.18	
N ₂	17.40	16.00	13.40	
Fluidizing Velocity, ft/sec.	.288	.275	.233	
Steam Conversion, %	23.4	30.1	31.4	

*Contributes to outlet gas only.

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D	E	F	G
11	20	20	20
1600	1600	1600	1600
Used Colstrip Subbituminous Char			
Run M-17			
4.44	4.12	4.05	4.10
120.1	130.4	156.2	148.0
63.9	282.3	209.6	210.0
32.6	53.8	55.2	56.3
5.0	14.7	14.7	9.5
0	5.2	0	0
2.0	2.0	2.0	2.0
6.92	6.00	8.17	8.01
1.94	6.20	5.82	5.84
0.14	0.77	0.68	0.63
1.08	3.02	2.40	2.36
0.44	1.60	1.15	1.21
0.48	2.41	1.78	1.95
2.24	3.77	4.58	4.46
4.13	7.16	7.41	7.58
0.36	1.00	0.90	0.90
2.68	3.91	3.52	3.37
1.10	1.77	1.69	1.73
0.49	2.39	1.80	1.96
124.3	98.3	113.3	107.5
10.4	15.5	15.3	15.4
53.9	97.3	107.6	102.7
44.86	42.54	46.11	46.82
3.91	5.93	6.19	5.54
29.21	23.22	21.92	20.77
12.02	10.51	10.48	10.67
10.00	17.80	15.30	16.20
.254	.291	.261	.254
55.1	25.4	31.1	30.8

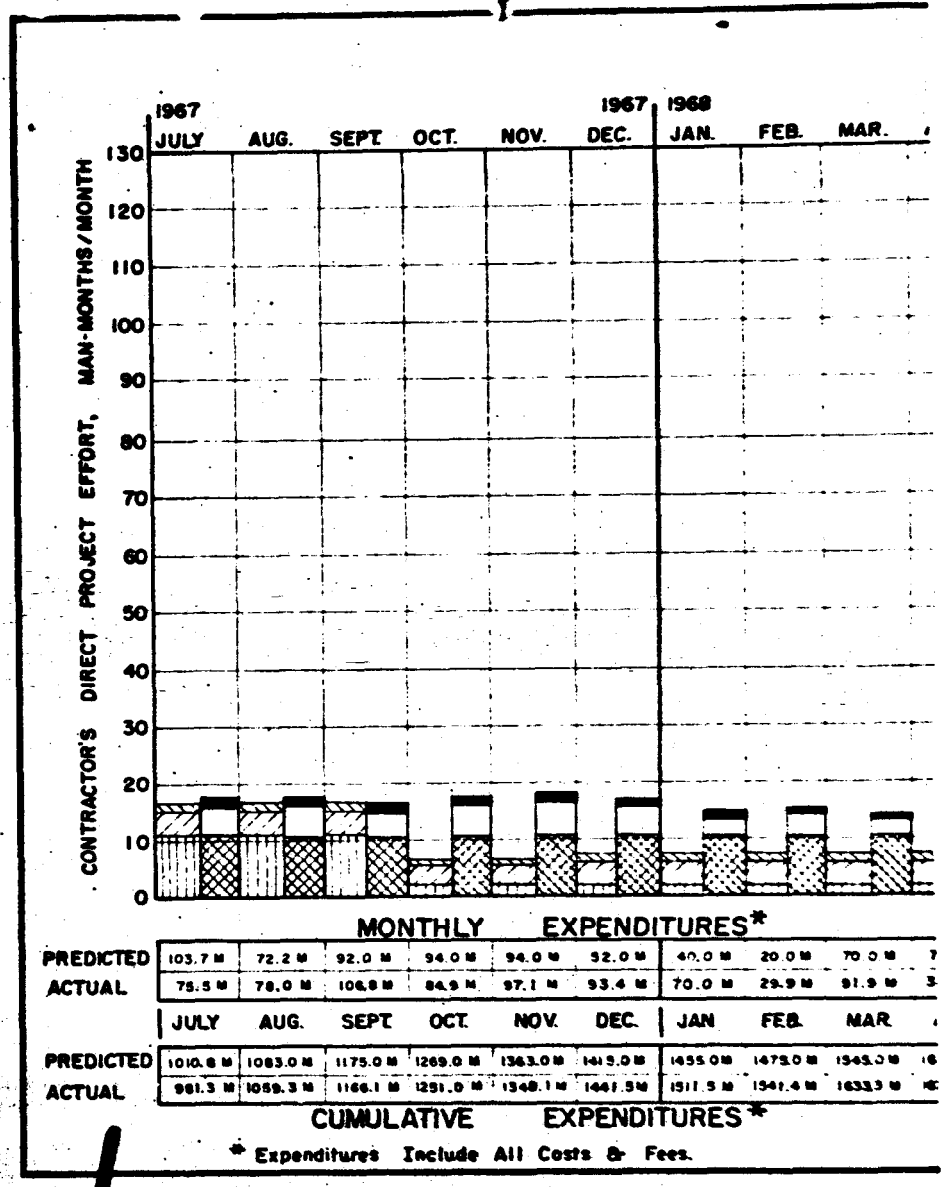
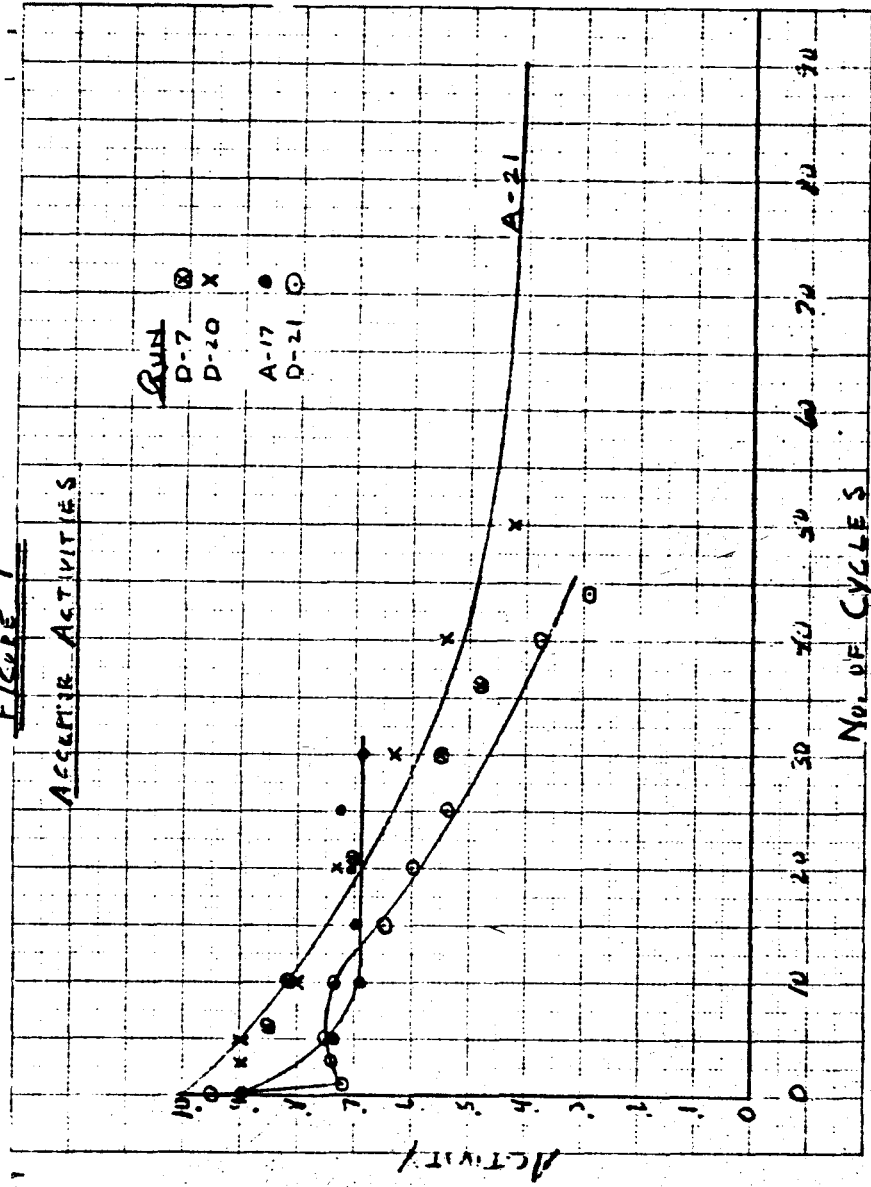
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TABLE VI
CONDITIONS AND RESULTS
Run 532-P-1

Run No. 532	P-1(A)	P-1(B)
Duration, Hours	6.03	9.87
System pressure, atm	20	
Bed Temperature, °F	1700	
Feedstock	Colstrip II Char	
Feed Rate, lb/hr	Run K-3, D, F, & G	
	4.4	3.8
<u>Inlet Gas (Bottom of Bed)</u>		
Recycle SCFH	278	291
Air, Bottom of Bed, SCFH	119.5	60.5
Air, Middle of Bed*	--	62.0
Added CO ₂	13.1	4.5
N ₂	2.0	2.0
<u>Composition, Partial Pressure, atm.</u>		
O ₂	1.22	0.71
CO	4.12	3.87
CO ₂	1.60	1.21
N ₂	13.06	14.21
<u>Outlet Gas (Top of Bed)</u>		
O ₂	--	--
CO	6.25	4.98
CO ₂	1.46	1.23
N ₂	12.29	13.79
<u>Exit Gas, SCFH</u>		
N ₂ Purges, "	170.0	169.5
	9.4	19.4
<u>Recycle Gas Composition, Mole %</u>		
CO	30.59	23.83
CO ₂	7.16	5.88
N ₂	62.25	70.29
<u>Fluidizing Velocity, ft/sec.</u>		
Bottom of Bed	.265	.230
Top of Bed	.282	.283

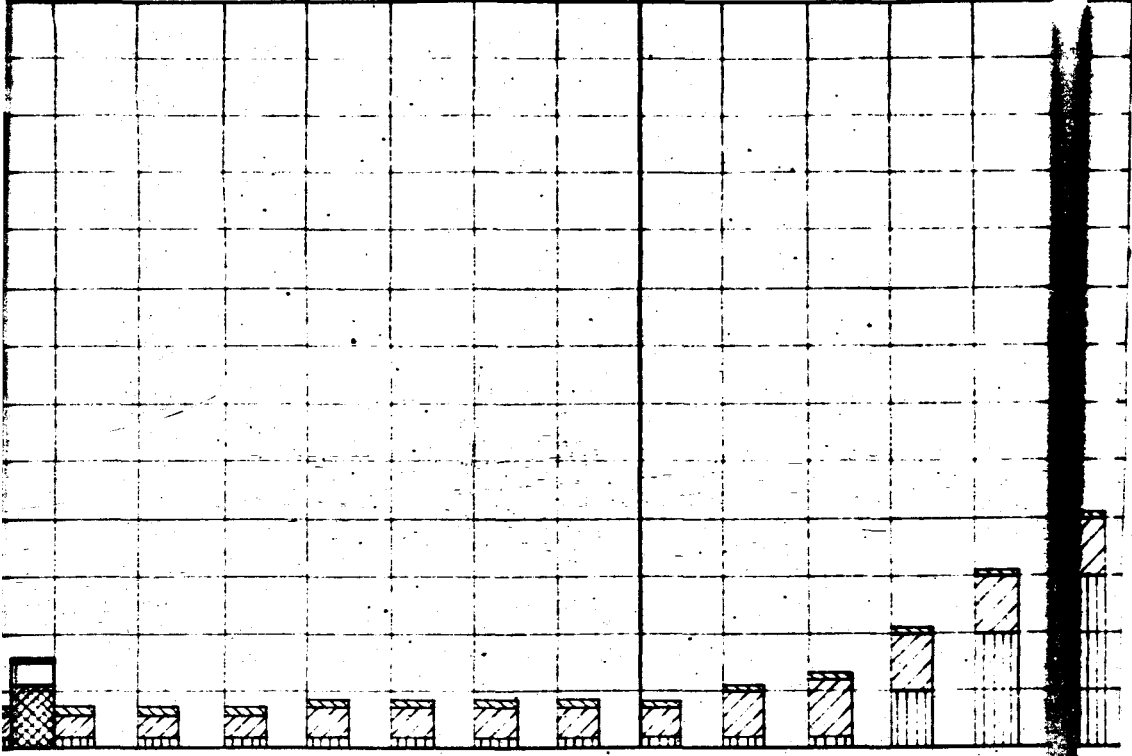
*Does not contribute to the inlet gas composition.

FIGURE 1



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1968 1969
JUNE JULY AUG. SEPT. OCT. NOV. DEC. JAN. FEB. MAR. APR. MAY JUNE



220.0 M 220.0 M 220.0 M 220.0 M 320.0 M 320.0 M 420.0 M 290.0 M 290.0 M 240.0 M 180.0 M 180.0 M

1968 JUNE JULY AUG. SEPT. OCT. NOV. DEC. 1969 JAN. FEB. MAR. APR. MAY JUNE

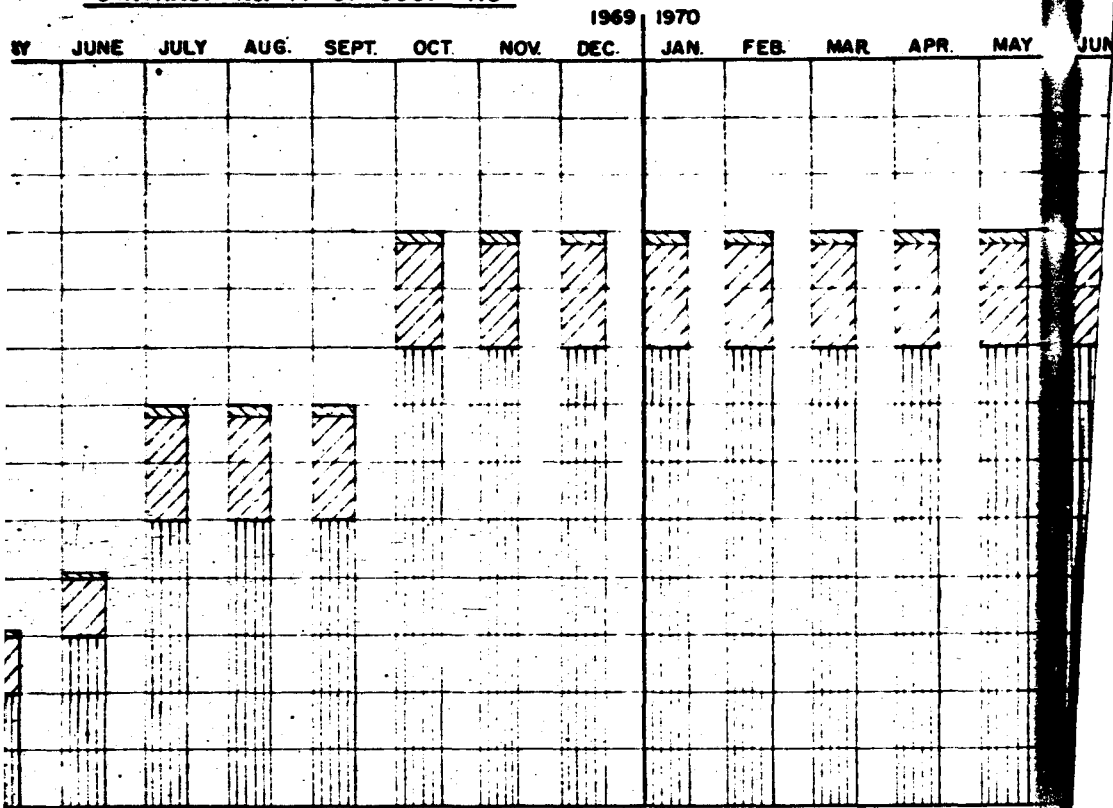
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MONTHLY PROGRESS CHART

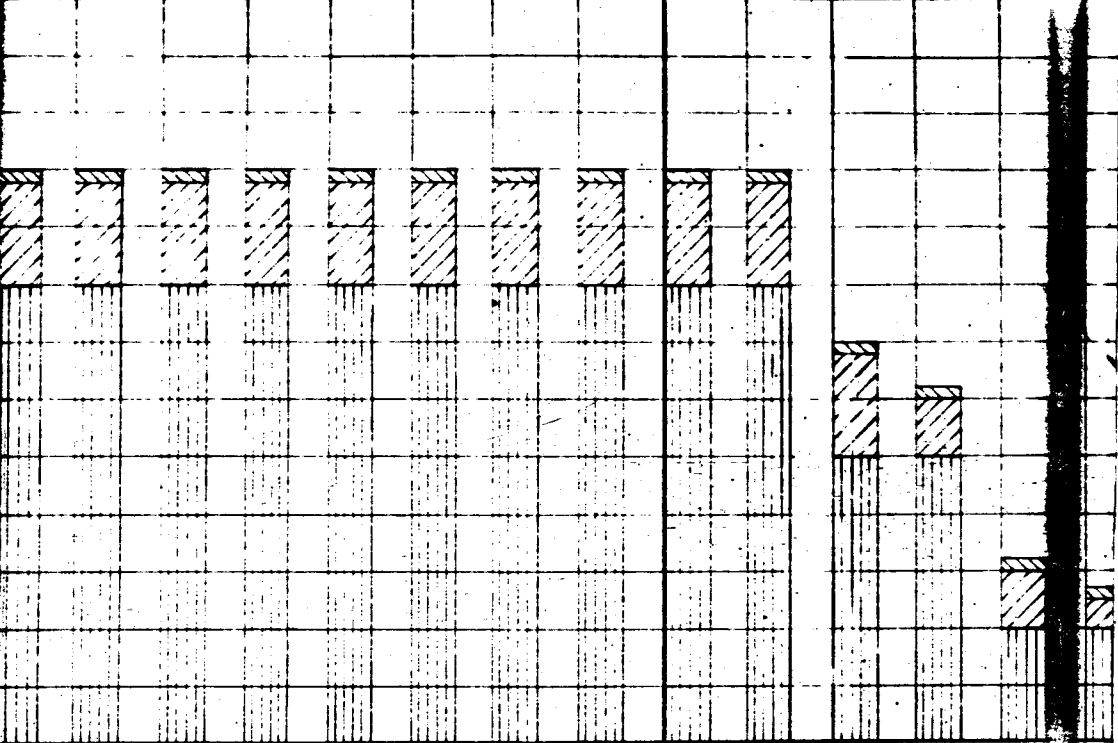
CONTRACT No. 14-01-0001-415



MAY	JUNE	JULY	AUG.	SEPT.	OCT.	NOV.	DEC.	JAN.	FEB.	MAR.	APR.	MAY
0.0 M	400.0 M	100.0 M	100.0 M	100.0 M	150.0 M	150.0 M	150.0 M	125.0 M	125.0 M	125.0 M	125.0 M	125.0 M
0.0 M	5465.0 M	5555.0 M	5655.0 M	5755.0 M	5905.0 M	6055.0 M	6205.0 M	6330.0 M	6455.0 M	6580.0 M	6705.0 M	6830.0 M

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1970 1971
MAY JUNE JULY AUG. SEPT. OCT. NOV. DEC. JAN. FEB. MAR. APR. MAY JUN



125.0 M 125.0 M 125.0 M 125.0 M 125.0 M 125.0 M 125.0 M 125.0 M 125.0 M 125.0 M 25.0 M 125.0 M 125.0 M

MAY JUNE JULY AUG. SEPT. OCT. NOV. DEC. JAN. FEB. MAR. APR. MA J

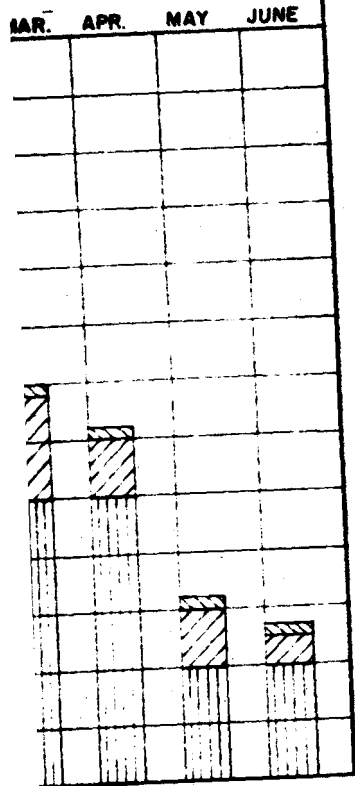
8830.0 M 8959.0 M 7080.0 M 7205.0 M 7330.0 M 7455.0 M 7580.0 M 7705.0 M 7830.0 M 7955.0 M 8080.0 M 8205.0 M 8330.0 M

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DATE: May, 1968

1971



PREDICTED ACTUAL

SUPERVISING [diagonal lines] [solid black]

PROFESSIONAL [horizontal lines] [white with border]

TECHNICIAN [vertical lines] [diagonal lines with circles]

⊙ Includes Supervisory Exempt Technicians.
 ⊗ Includes Clerical.

25.0 M	125.0 M	125.0 M	170.0 M
MAR.	APR.	MAY	JUNE
080.0 M	8205.0 M	8330.0 M	8500.0 M

5

MONTHLY PROGRESS CHART

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 DEPARTMENT OF THE INTERIOR

CONSOLIDATION COAL CO.
 RESEARCH DIVISION LIBRARY, PA.

CONTRACT No. 14-01-0001-415

PROGRESS REPORT NO. 49
 OCR CONTRACT NO. 14-01-0001-115(2)
CONSOL LIGNITE GASIFICATION PROCESS DEVELOPMENT

June 1 to 30, 1968

A. Summary

Phase I - Feasibility Study

Complete.

Phase II - Bench-Scale Experiments

Data from Run D-22 show that the carbon burnout in the regenerator increased from 73-86% at a constant CO level upon reducing the fluidizing velocity from 1.32 to 1.12 ft/sec. A high retention of char in the bed at the lower velocity was shown to be the cause of the improved combustion efficiency.

Integral rate data are presented from runs conducted earlier with Colstrip char in the continuous unit. The results indicate a lower reactivity relative to Husky lignite char.

A series of tests were carried out to determine the minimum amount of preoxidation required to render Colstrip coal operable in the hydrodevolatilization step. The results show that operability can be sustained with as little as 4% preoxidation.

A material balance run was conducted to simulate single-stage gasification of lightly preoxidized (4%) Colstrip coal at ca 1 hour residence time. This was done by circulating acceptor through the hydrodevolatilizer and eliminating extrinsic hydrogen addition. Material balance showed similar results (char yield was 52.8%) to those obtained with other feedstocks in the standard hydrodevolatilization runs without acceptor addition or preoxidation. The yield of CO was somewhat lower and that of H₂ was significantly higher, however, as was expected due to acceptor action. More extensive gasification could have been achieved by use of higher acceptor circulation rates and higher steam partial pressures. Experimental work has now been terminated and report writing is proceeding.

Phase III - Pilot Plant Engineering

A proposal for continuing the CO₂ acceptor development program through pilot plant construction and operation was submitted to OCR on June 28.

Solicitations for proposals for pilot plant construction and/or operation of the Rapid City facility are being prepared for review by OCR in July.

Financial

Estimated expenditures in June were \$31,000. Cumulative expenses to date are \$1,732,321.

B. Technical Results and ProgramPhase II - Bench-Scale Research1. Operation of the Continuous Gasification Unit (Project No. 532.00)a. Additional Data from Previous Runs(1) Run 532-D-21, Use of Sodium - Impregnated Acceptor

Results on activity loss of acceptor in this run were given in the May report. Data on sodium loss are reported here.

In this run the acceptor was circulated four times through the unit in the $MgO.CaCO_3$ form with the gasifier at 1500°F and the regenerator at 1600°F. During this period the activity declined sharply, even though no calcining had occurred, and most of the sodium was lost from the acceptor. The sodium loss continued on subsequent process cycles. The data are shown below:

<u>Na_2O Content, $MgO.CaO$ Basis</u>	
As Prepared	1.0 wt. %
After 1 $MgO.CaCO_3$ cycle	.85
After 4 $MgO.CaCO_3$ cycles	.28
After 1 process cycle	.19
After 20 process cycles	.11

The natural sodium content of the raw acceptor is 0.23%. The overhead attrited fines from the regenerator during the early $MgO.CaCO_3$ cycles will be analyzed for sodium. If this material shows no sodium enrichment, then the loss must have occurred in the gasifier.

(2) Run 532-D-22, Operations with Husky II Char at 1940°F

Analyses of the overhead ash from the regenerator during Run D-22A showed that the carbon burnout was 73%. In Run D-22B, after reducing the fluidizing velocity from 1.32 to 1.12 ft./sec., the increased retentivity caused the burnout to increase to 86% while a CO content of 3.8% was maintained in the exit gas.

At the end of Run D-22B, the fuel char feed was stopped and the combustion air and most of the recycle gas flows were removed in quick succession, thereby "freezing" the regenerator bed. The residual fuel char was recovered from the bed by screening at 48 mesh. Based on the weight and composition of the recovered material and compositions of the feed char and overhead ash as produced during the run, ash balances gave the fuel inventory range as shown below:

	<u>Regenerator Fuel Inventory,</u>	
	<u>Wt. % of Acceptor In Bed</u>	<u>Carbon</u>
Assuming char inventory bed composition of feed char	16.7	11.7
Assuming char inventory bed composition of overhead "ash"	6.3	1.7

Based on the ash contents of the recovered material and feed char and the feed char input rate, the average retention time of the fuel char was calculated as 14 minutes. This time is greatly in excess of that required for complete burnout at any appreciable O_2 partial pressure. The observed burnout at the long average retention time suggests that all of the incoming O_2 is consumed quickly at the bottom of the bed and that the remaining carbon burnout occurs in the upper part of the bed by the slow reaction, $C + CO_2 = 2 CO$.

(3) Run 532-K-3, Kinetics of Colstrip II Gasification

Compositions of the hydrodevolatilized Colstrip I and II sub-bituminous chars are shown in Table I. Char from the first shipment, Colstrip I, was used in Runs H-16, D-4, K-2, D-5 and D-15.

Integral gasification rate data are shown in Table II-A. Gas partial pressures were given in Table V of the May report. As with the lignite chars, the rates are enhanced, in general, by operation at 11 atm. total pressure rather than at 20 atm. However, the lower reactivity of the subbituminous char means that the gasifier would have to be operated at about 1600°F. At this higher temperature level the system pressure would have to be about 20 atm. in order to maintain heat balance via the acceptor reaction. Therefore, no advantage can be taken of the enhanced reaction rates at the lower system pressure.

Run K-3C was made at 1500°F and provided rate data which can be compared with those from the Husky II lignite char. The pertinent data are shown in Table II-B. A crude evaluation of the relative reactivities of the two chars may be made by comparing the ratios of the observed integral total gasification rates and the differential rates (obtained with Renner Cove lignite char) calculated at the exit gas partial pressures. On this basis, the Colstrip II char is 3.5 times less reactive at 1500°F.

b. Gasification of Preoxidized Colstrip II Coal

Run H-13, made with Rosebud coal, showed that raw, dry subbituminous coal cannot be fed directly to the hydrodevolatilizer without the occurrence of massive coking. The entire effort during June was devoted to determination of the minimum amount of preoxidation required to prevent coking, and to making a run simulating single-vessel gasification with the preoxidized Colstrip coal.

(1) Drying

To prepare a stock of dry coal for the preoxidation runs, the raw Colstrip Coal was dried in the gasifier at 400°F. Since our earlier work on preoxidation of Eastern bituminous coal showed that oxygen reacted preferentially with the -100 μ fine coal; the -25 μ coal, after being partially dried in air at less than 200°F, was screened at 100 mesh. The coal as received contained

31% moisture and the -28 M material was not free-flowing. The coal became free-flowing after being air dried to a moisture content of 18% or below.

In order to achieve complete drying and to avoid spurious preoxidation, the coal was dried in N_2 in the gasifier at 400°F. A system pressure of 11 atm. was used, not because of any process consideration, but because the system became insensitive to pressure upsets caused by temporary blockages in the exit piping (which are created by elutriated fines) at this pressure level. A total of 570 lb of dry coal was produced.

Analyses of the exit gas showed no detectable H_2 , CH_4 , or CO at 400°F. However, CO_2 was present to the extent of 0.09% of the dry coal.

(2) Preoxidation

Since the required level of preoxidation to prevent coking was unknown, three relatively small batches of about 80 lb each were prepared at 500°F and at the nominal levels of 4, 6, and 8% preoxidation. Conditions and results are shown in Table III. A system pressure of 29 atm. was used to achieve freedom from pressure upsets and because the process could be operated at this pressure level.

Each of the three batches was fed to the gasifier operated at hydrodevolatilizer conditions which were nominally the same as in Run H-11. After each run, the vessel was inspected for signs of coking. All three materials were completely operable with no coking whatsoever. The 6 and 8% char particles were not rounded or swollen, as was the case in Run H-14. A few of the 4% char particles showed some rounding. The mercury densities and size consists of the product chars are shown in Table IV. The mercury density of hydrodevolatilized LTC Colstrip II char was considerably greater (78 lb/ft³) than those of the chars derived from preoxidized coal. The difference is caused, not by swelling due to intumescence, but by cracks which developed along the bedding planes. The cracked particles are sufficiently rugged to resist attrition in the fluidized bed. Gross fracture of the particles did not occur.

In view of the incipient rounding and lower levels of particle shrinkage and mercury density, the 4% preoxidation level was taken as the minimum which would give freedom from coking. A large batch of coal (238 lb) for use as feedstock to the gasification run therefore was prepared at the 4% level. Another small batch of 8% material also was prepared with the intent of determining the effect of preoxidation level on char yield and gaseous product distribution. Conditions and results for these runs also are shown in Table III.

The yields of H_2O , CO_2 and CO are relatively independent of the preoxidation level over the range investigated. Analysis of the offgas showed the presence of 0.05% CH_4 at 500°F, which corresponds to 0.01% by weight of the dry coal.

(3) Gasification

The intent of this run was to simulate single-vessel operation of the gasification step wherein 4% preoxidized Colstrip II coal was fed to the gasifier at the highest practicable inlet steam partial pressure and with the use

of acceptor. The relatively low temperature level of 1525°F was chosen in the hope that, with simultaneous hydrodevolatilization and gasification, the reactivity of the freshly-created subbituminous char would be enhanced.

The original gasifier vessel, having the flow restrictor configuration at the bottom, was used because its longer length provided a greater retention time for decomposition of the tar vapors.

In previous runs (H-1 through H-17) in which LTC char or raw coal was fed to the gasifier vessel, no acceptor was used. The feedstocks were introduced through the normal acceptor discharge port at the bottom of the vessel in order to maximize the tar vapor retention time. The use of acceptor in the gasification run (532-G-1) introduced two problems: (1) The only available coal inlet line was the normal char inlet line which enters from the top and extends, as a dip tube, to the middle of the fluidized bed. This line was lengthened to just above the char-acceptor interface in order to increase the tar vapor retention time. The first effort at feeding preoxidized coal through this line, using the normal carrier gas velocity, resulted in a plug caused by heavy pitch which was formed as the coal passed through the hot dip tube. By increasing the carrier gas flow to decrease the coal retention time in the dip tube, pitch formation was suppressed and no further trouble was experienced. (2) Because of the low fluidizing velocity needed to assure adequate retention time for decomposition of tar vapor, it was uncertain whether sufficient velocity would be available to fluidize the acceptor layer below the char-acceptor interface. To aid this situation, the acceptor inventory size consist was reduced to 20 x 35 mesh, compared with the normal 16 x 28 mesh size range. Although it was desired to use the highest possible inlet steam partial pressure (~11 atm), the inlet partial pressure was reduced to 8.5 atm, to avoid agglomeration due to incipient melt formation in case fluidization could not be maintained in the acceptor layer.

As a result of the two problems, the effective inlet steam partial pressure at the bottom of the char bed was only 5.9 atm., a level much too low to prove out the single-vessel operation. It was found that a stable char-acceptor interface could be maintained and that a higher inlet steam partial pressure could have been used. Run conditions and results are in Table V, along with those from Runs H-14 and H-11 which are included for comparison.

The yields of char and total gaseous carbon are similar in all three runs. Compared with the other runs, the yield of CO_2 in Run G-1, which includes the CO_2 picked up by the acceptor, is increased at the expense of CO, as was anticipated. Another consequence of the use of acceptor was an increase in the amount of hydrogen made. In Run G-1 the partial pressures of both CO and CO_2 were relatively high when the normal acceptor circulation rate of about 10 lb/hr. was used. The next step was to operate with an increased circulation rate in order to reduce the CO_2 partial pressure, which in turn would reduce the severe CO inhibition of the gasification rate by virtue of the water-gas shift reaction.

Four efforts were made to continue the run at a higher acceptor circulation rate. No data were obtained because an unusual sequence of mechanical malfunctions terminated all attempts before steady-state was achieved.

However, freedom from coking at the 4% preoxidation level was confirmed, since no coke was found after feeding 170 lbs of the coal.

Throughout the operations involved in gasifying preoxidized coal, the regenerator heat requirement was supplied by combustion of CO. The regenerator bed temperature and CO content of the exit gas purposefully were allowed to vary between the limits of 1830-1940°F and 0.5-2.0%, respectively, in order to demonstrate that high acceptor sulfur loadings such as occurred at the conditions used in the gasifier can be accommodated at a relatively wide range of conditions without the formation of a deposit derived from the transient liquid. (The lower levels of CO content were used only after the initial high rate of acceptor attrition had ceased.) Heat releases as high as 300,000 BTU/(hr)(ft³) of bed were used. There were no deposits.

Were more time available, the original goal could be attained, since there is no inherent reason why a high acceptor circulation rate and continuity of operation with preoxidized coal cannot be achieved. However, the scheduled shutdown of the unit prevented any further effort.

c. Program

No further operations are planned. A final report on the bench-scale work will be prepared.

2. Batch Kinetic Studies (Project No. 530.00)

Samples from the continuous unit operations were processed routinely.

As with Project 532.00, all future effort will be devoted to report writing, covering the results obtained during the past four years.

3. Supporting Patent and Legal Activities (Project No. 534.00)

The current laboratory reports and memoranda were reviewed for determination of Subject Inventions.

4. Supporting Process and Economic Analysis (Project No. 533.00)

No work was done on this project.

Phase III - Pilot Plant Engineering

A proposal for the continuation of the CO₂ Acceptor Development through pilot plant construction and operation was submitted to OCR on June 28, 1968. After some discussion, Consol was directed to prepare solicitations for proposals from subcontractors for pilot plant construction and/or pilot plant operation. A list of companies to whom solicitations will be sent was approved.

Consol is now preparing drafts of these solicitations for review by OCR in July. The construction plan involves a two-phase program with the initial plant to be limited to the facilities required for raw water gas production.

C. Subject Inventions

None.

D. Financial Status

<u>Expenses</u>	<u>Actual</u>	<u>Budget</u>
Cumulative through May, 1968	\$ 1,701,321	\$ 1,735,000
Expended in Period (estimate)	<u>31,000</u>	<u>220,000</u>
Cumulative through June, 1968	\$ 1,732,321	\$ 1,955,000

Att. - Tables I - V
- Monthly Progress Chart
7/12/68

TABLE I

Compositions of Colstrip Subbituminous Char

Hydrodevolatilized Char, Made In Run Number	Colstrip I H-16	Colstrip II H-17
H, wt: %, dry basis	.65	.80
C	84.92	82.08
H	.49	.65
O (diff.)	.88	2.39
S	.26	.62
Ash	13.39	13.70
Sulfide Sulfur	.19	.60
<u>Ash Composition, Sulfur-Free Basis</u>		
Al ₂ O ₃ , wt. %	24.1	23.4
SiO ₂	33.4	38.8
Fe ₂ O ₃	4.1	3.4
CaO	26.7	31.6
MgO	7.0	5.3
Na ₂ O	.3	.2
K ₂ O	.2	.2
TiO ₂	1.0	1.2
P ₂ O ₅	.7	.4
SrO	.2	.1
	97.8	94.6

TABLE II-A

Integral Gasification Rate Data

R = lb C gasified/min./lb C in bed x 10⁴

Run 532-13-	°F	Atm.	% Burnoff	R _C	R _{CH₄}	R _{Total}
B	1600	20	29	27.4	6.2	39.8
C	1500	11	26	23.8	3.0	29.8
D	1600	11	47	54.8	5.7	66.2
E		20	30	22.5	6.2	34.9
F			41	33.7	7.8	49.3
G			36	31.8	6.8	45.2

TABLE II-B

Comparison of Reactivities at 1500°F at 11 Atm Pressure

Run	D-6	A-21	K-3C
<u>Exit Partial Pressures, Atm.</u>			
H ₂ O	2.81	2.72	3.58
H ₂	4.38	4.90	3.95
CO	1.52	1.48	1.34
CH ₄	.24	.33	.28
% Burnoff	49	46	26
<u>Observed Rates</u>			
R _C	29.7	28.4	23.8
R _{CH₄}	3.8	4.2	3.0
R _T	37.3	36.8	29.8
Calculated R _T	29	26	77

TABLE III

Conditions and Results of Preoxidation Runs

Run	500°F 20 atm Dry Colstrip II Subbituminous Coal				
	I	II	III	IV	V
Total lb Fed	83	80	82	238	58
Feed Rate, lb/hr, dry basis	9.78	9.71	9.94	9.92	9.93
<u>Inlet Gas</u>					
Recycle, SCFH	530				
N ₂	27.4				
Air	34.5	22.8	45.6	.2	49.7
Inlet O ₂ Partial Press., Atm.	.24	.17	.31	.16	.34
Exit Gas Rate, SCFH	70.0	59.6	83.5	59.4	84.3
Purges, N ₂	12.1	12.5	15.6	12.1	12.2
<u>Recycle Gas Composition, mol %</u>					
N ₂	95.34	96.33	94.63	96.16	93.73
CO	.81	.68	.98	.68	1.01
CO ₂	3.80	2.98	4.19	3.16	5.04
O ₂	.05	.01	.20	0	.22
Fluidizing Velocity, ft/sec	.171	.167	.174	.167	.175
<u>Distribution of O₂, %</u>					
to H ₂ O	58.8	58.7	57.4	55.4	53.4
to CO ₂	36.7	37.0	36.6	40.3	40.7
to CO	4.0	4.2	4.3	4.3	4.1
not reacted	.5	.1	1.7	0	1.8
% Preoxidation*	6.1	4.1	7.8	3.9	8.5

*lb O₂ reacted/lb dry coal x 100

TABLE IV

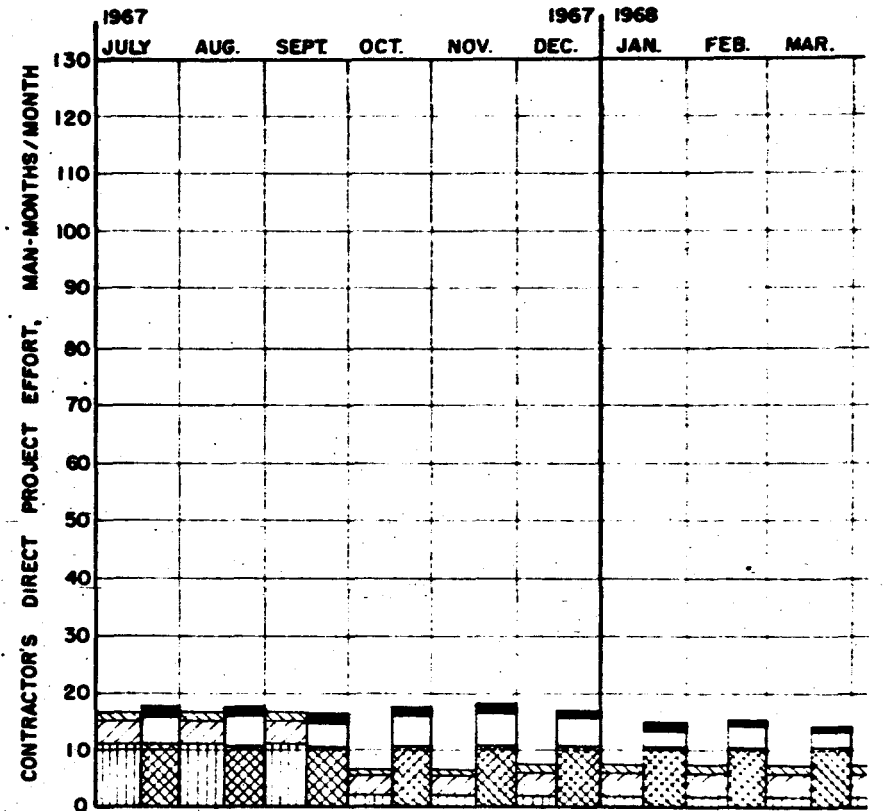
Char Properties after Hydrodevolatilization of Preoxidized Coal

	Dry Raw Coal	% Preoxidation		
		4	6	8
28 x 35 mesh, wt. %	14.0	7.0	5.9	6.0
35 x 48	26.8	28.3	27.0	25.6
48 x 65	24.6	27.3	27.8	27.1
65 x 100	17.0	17.8	18.6	18.5
100 x 150	12.1	12.7	13.7	14.6
-150	5.5	6.9	7.0	8.2
mean diameter, inches	.01081	.00994	.00970	.00949
% shrinkage	-	8.0	10.3	12.2
mercury density, lb/ft ³	~78	58.9	59.4	62.6

TABLE V

Run Conditions and Results

Run Number	G-1	H-14	H-11
Cycle			
Acceptor	Tymochtee-6, 20 x 35 M		None
Acceptor Circulation Rate (raw basis), lb/hr	9.3		
System Pressure, atm.		20	
Acceptor Activity	.67		
Gasifier, Temperature, °F	1525	1525	1510
Coal Feed	4% Preox. Colstrip	Rosebud	Glenharold
Feed Rate, lb/hr, dry basis	7.52	6.66	7.56
Inlet Gas (Bottom of Bed)			
Steam, SCFH	86.9	71.8	75.5
Recycle, "	200	178	172
Added H ₂ , "	0	0	15.0
N ₂ , "	6.8	11.3	5.4
Composition, Partial Pressure, atm.			
H ₂ O	5.92	5.56	5.64
H ₂	8.71	4.83	6.53
CH ₄	2.28	2.00	1.86
CO	2.40	2.67	3.01
CO ₂	.95	1.34	1.40
N ₂	2.76	3.60	1.56
Outlet Gas (Top of Bed)			
Composition, Partial Pressure, atm.			
H ₂ O	3.09	3.47	3.48
H ₂	7.40	6.10	7.11
CH ₄	2.92	2.53	2.44
CO	3.11	3.37	3.95
CO ₂	1.23	1.69	1.84
N ₂	2.25	2.84	1.17
Dry Exit Gas, SCFH	117.4	105.0	127.3
H ₂ Purges, "	13.0	9.8	6.0
Condensate, "	55.5	56.7	62.0
Recycle Gas Composition, mol %			
H ₂	41.9	35.6	42.2
CH ₄	16.57	14.78	14.47
CO	17.63	19.66	23.47
CO ₂	6.99	9.88	10.91
N ₂	16.9	20.1	9.0
Fluidizing Velocity, ft/sec. avg.	.193	.171	.183
Avg. Vapor Retention Time, sec.	17.7	16.5	18.2
Yields			
Char, Wt. % MF Feed	52.75	50.3	51.5
Carbon Bearing Gases, lb C/100 lb MF Fd	24.73	21.68	25.51
CH ₄	8.03	7.23	7.56
CO	8.54	9.62	12.25
CO ₂	8.19	4.83	5.70
Product Particle Density, lb/ft ³	59.1	32.7	60.0



MONTHLY EXPENDITURES*

	JULY	AUG.	SEPT	OCT.	NOV.	DEC.	JAN.	FEB.	MAR.
PREDICTED	103.7 M	72.2 M	92.0 M	94.0 M	94.0 M	92.0 M	40.0 M	20.0 M	70.0 M
ACTUAL	75.5 M	78.0 M	108.9 M	84.9 M	97.1 M	93.4 M	70.0 M	29.9 M	91.9 M

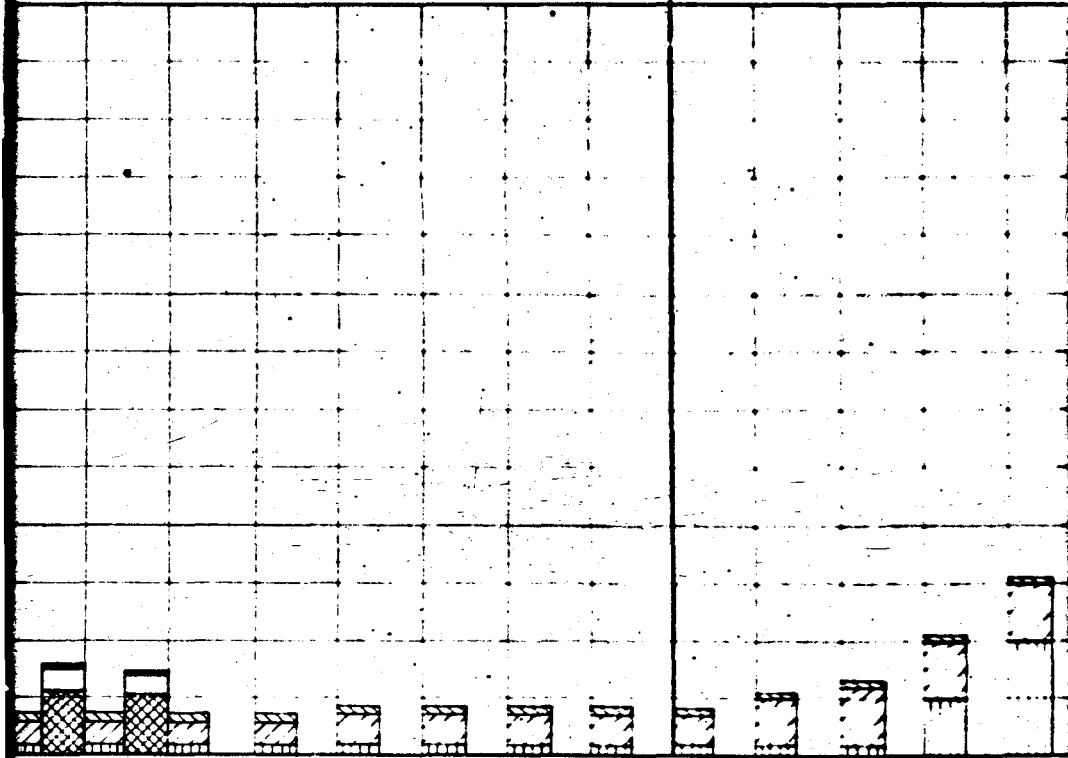
CUMULATIVE EXPENDITURES*

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PREDICTED	103.7 M	108.0 M	117.0 M	126.0 M	136.0 M	145.0 M	145.0 M	147.0 M	154.0 M
ACTUAL	98.3 M	105.3 M	116.1 M	125.0 M	134.1 M	144.5 M	151.5 M	154.4 M	163.3 M

* Expenditures Include All Costs & Fees.

MC

1968 1969
MAY JUNE JULY AUG. SEPT. OCT. NOV. DEC. JAN. FEB. MAR. APR. MAY



20.0 M	220.0 M	220.0 M	220.0 M	220.0 M	320.0 M	320.0 M	320.0 M	420.0 M	290.0 M	290.0 M	240.0 M	60.0 M
30.1 M	31.0 M											
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101.3 M	1732.3 M											

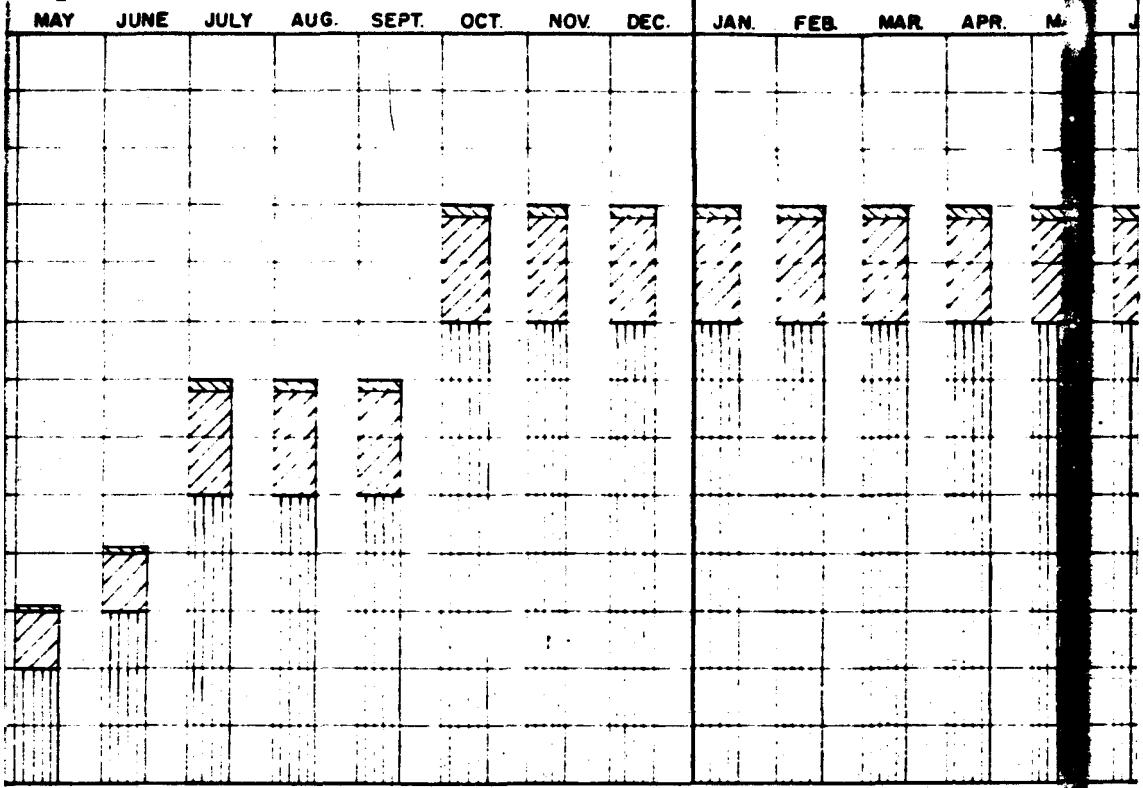
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MONTHLY PROGRESS CHART

CONTRACT No. 14-01-0001-415

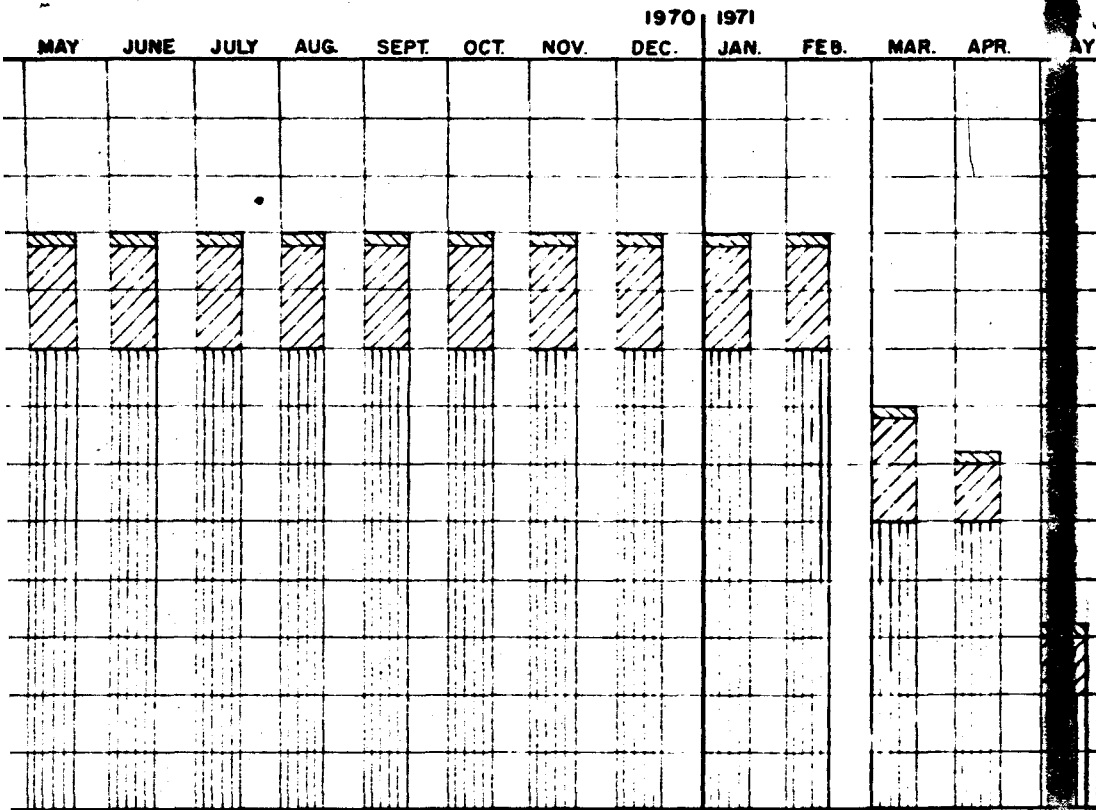
1969 1970



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OCT.	5905.0 M	
NOV.	6055.0 M	
DEC.	6205.0 M	
JAN.		6330.0 M
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MAR.		6580.0 M
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MAY		6830.0 M

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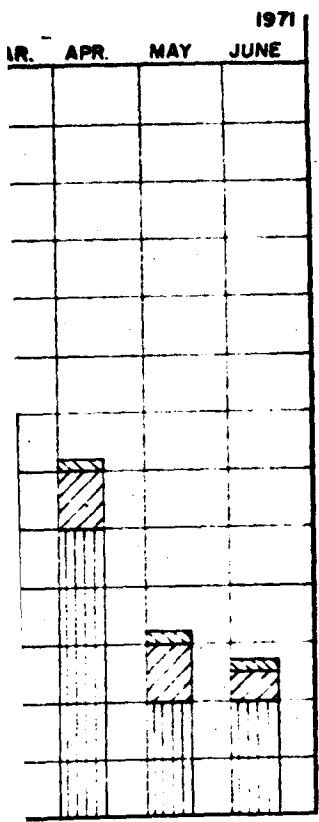


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4

DATE: June, 1968



PREDICTED ACTUAL

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PROFESSIONAL [diagonal lines] [white with border]

TECHNICIAN [vertical lines] [diagonal lines]

⊕ Includes Supervisory Exempt Technicians.
 ⊕⊕ Includes Clerical.

1 M	125.0 M	125.0 M	170.0 M
2.	APR.	MAY	JUNE
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MONTHLY PROGRESS CHART

OFFICE OF COAL RESEARCH
 DEPARTMENT OF THE INTERIOR

CONSOLIDATION COAL CO.
 RESEARCH DIVISION LIBRARY, PA.

CONTRACT No. 14-01-0001-415

5

PROGRESS REPORT NO. 50
 OCR CONTRACT NO. 14-01-0001-415(2)
CONSOL LIGNITE GASIFICATION PROCESS DEVELOPMENT

July 1 to 31, 1968

A. Summary

Phase I - Feasibility Study

Complete.

Phase II - Bench-Scale Experiments

Preparatory work has been carried out preliminary to writing the formal reports on Phase II of this development. This has included:

1. Preparation of outlines of the reports.
2. Completion of processing of experimental data carried out in prior months.
3. Completion of data correlation work.

A limited amount of experimental work on further development of the melt process for acceptor manufacture has also been in progress.

Phase III - Pilot Plant Engineering

Solicitations for proposals for construction of the Rapid City plant and for supplying services during operations were approved by OCR on July 29. Documents will be mailed to 13 interested contractors on August 5, 1968.

Financial

Estimated expenditures in July were \$27,000. Cumulative expenses to date are \$1,759,868.

B. Technical Results and Program

Phase II - Bench-Scale Research

1. Batch Kinetic Studies (Project No. 530.00)

In preparation for writing a final report on this project, the following work was done:

- a. The fluidized bed correlation was reviewed critically and modifications were made to include recent data taken at process conditions in the continuous unit.
- b. A correlation was developed for prediction of the incipient fluidizing velocity from existing literature data on pressure drop through the packed beds.
- c. The correlation for prediction of pressure drop in the pneumatic transfer lines was modified to allow for the effects of particle acceleration and of bends in the line.

Some laboratory work was done on modifying the properties of frozen melts in the $\text{CaCO}_3\text{-Ca(OH)}_2$ system. Since Run D-21 showed that sodium was ineffective in enhancing acceptor activity, the work so far has been devoted to addition of SiO_2 . Liquid phases have been found to exist at moderate temperatures and steam pressures in the systems, $\text{CaCO}_3\text{-Ca(OH)}_2\text{-SiO}_2$ and $\text{Ca(OH)}_2\text{-SiO}_2$. Dicalcium silicate also forms through the reaction



Two separate reports are under preparation on this subject entitled

- a. "Studies on Mechanics of Fluo-Solids Systems"
- b. "Laboratory Physico-Chemical Studies"

Outlines of the above reports have been prepared.

2. Operation of the Continuous Gasification Unit (Project No. 532.00)

The following important work was done in preparation for writing a final report on operations of the continuous gasification unit.

The number of calcining-recarbonation cycles achieved in any run has been reported routinely in terms of nominal cycles. One nominal cycle occurs each time the external acceptor inventory is fed through the unit. As long as the weight of the external inventory is equal to or greater than the internal inventory, a nominal cycle is equivalent to an actual cycle.

However, in all the runs, the internal inventory increases continuously because the particle density increases as the acceptor loses

activity. Since the total inventory remains approximately constant (no makeup was added), the external inventory decreases continuously. The decrease in external inventory is further augmented by losses due to sampling and attrition. The end result of these processes is that in nearly every run the number of actual cycles is less than the number of nominal cycles.

The method for calculating the actual number of cycles is rather complicated and is tedious to execute. The details will be given in full in the final report. The calculations have been completed for all 36 runs in which an acceptor was used. The largest number of actual cycles were achieved in Runs A-13 and A-21. The most extreme difference between actual and nominal cycles occurred in Run A-9, made with Pete Lien dolomite. In this case the physically weak stone caused very large attrition losses. A comparison of actual and nominal cycles is given below:

Run	Number of Cycles	
	Nominal	Actual
A-13	75	68
A-21	93	66
A-9	95	47

An outline of the final report has been prepared.

2. Supporting Patent and Legal Activities (Project No. 534.00)

The current laboratory reports and memoranda were reviewed for determination of Subject Inventions.

4. Supporting Process and Economic Analysis (Project No. 533.00)

No work was done on this project.

Phase III - Pilot Plant Engineering

Invitations for proposals on the construction of the Rapid City pilot plant and on supplying required services during the pilot plant operation were approved by OCR on July 29 for transmittal to qualified contractors.

Thirteen "packages" of documents, each weighing 50 lbs, were prepared in preparation for mailing on August 5. The list of contractors interested in proposing on this job is attached in Table I; one company (Lummus Company) expressed lack of interest. Proposals are to be submitted to Consol on or before October 7, 1968.

Catalytic is embellishing the model of the Rapid City pilot plant in preparation for a display in the Interior Building on September 9-13, 1968.

C. Subject Inventions

None.

D. Financial Status

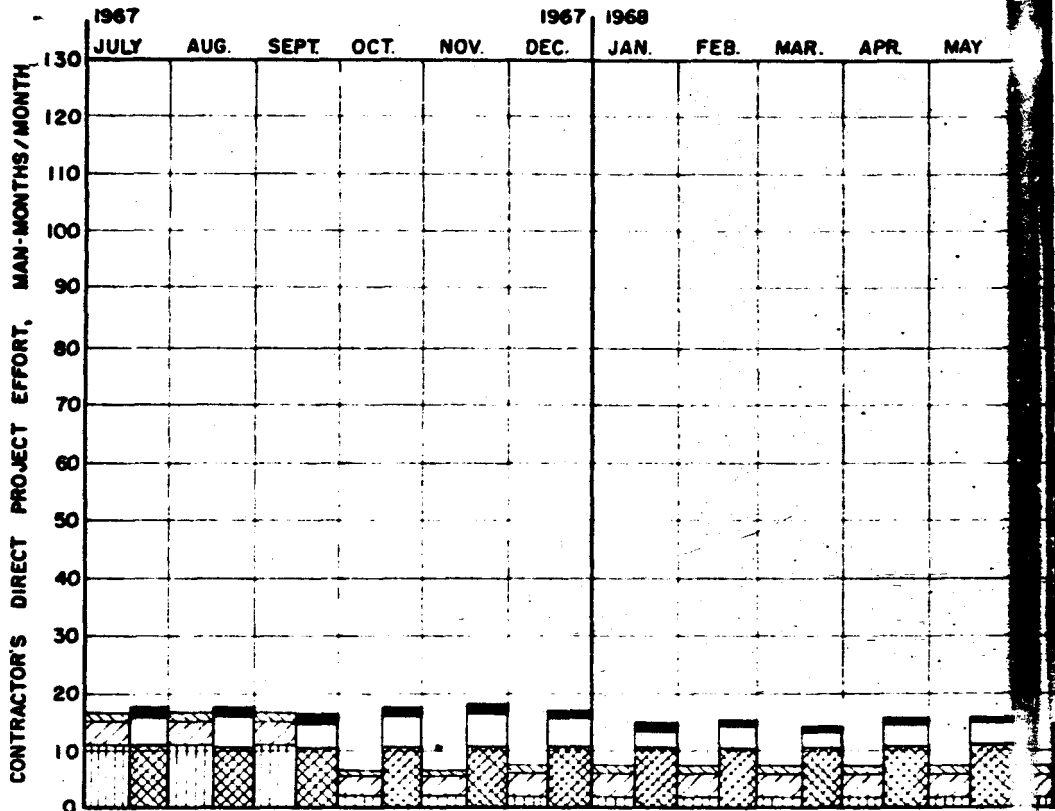
<u>Expense</u>	<u>Actual</u>	<u>Budget</u>
Cumulative through June	\$ 1,732,868	\$ 1,955,000
Expended in Period (estimate)	<u>27,000</u>	<u>220,000</u>
Cumulative through July	\$ 1,759,868	\$ 2,175,000

TABLE I

PROPOSED CONTRACTORS
FOR CSG GASIFICATION PILOT PLANT
CONSTRUCTION AND OPERATION

Bechtel Corporation
Bresina Construction Company
Catalytic Construction Company
Ford, Bacon & Davis, Inc.
H. W. Kellogg Company
Peter Lien & Sons, Inc.
Northwestern Engineering Company
Procon, Inc.
Stearns-Roger Corporation
Peter Kiewit & Sons, Inc.
Drave Corporation
Ralph M. Parsons Company
C. F. Braun & Company

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MONTHLY EXPENDITURES*

	JULY	AUG.	SEPT.	OCT.	NOV.	DEC.	JAN.	FEB.	MAR.	APR.	MAY	JUN.
PREDICTED	103.7 M	72.2 M	92.0 M	94.0 M	94.0 M	52.0 M	40.0 M	20.0 M	70.0 M	70.0 M	120.0 M	20.0 M
ACTUAL	75.5 M	78.0 M	106.8 M	84.9 M	97.1 M	93.4 M	70.0 M	29.9 M	91.9 M	37.9 M	30.1 M	32.8 M
PREDICTED	1010.6 M	1083.0 M	1175.0 M	1269.0 M	1363.0 M	1415.0 M	1455.0 M	1475.0 M	1543.0 M	1615.0 M	1735.0 M	1855.0 M
ACTUAL	981.5 M	1059.5 M	1166.1 M	1251.0 M	1348.1 M	1441.5 M	1511.5 M	1541.4 M	1633.3 M	1671.2 M	1701.3 M	1732.1 M

CUMULATIVE EXPENDITURES*

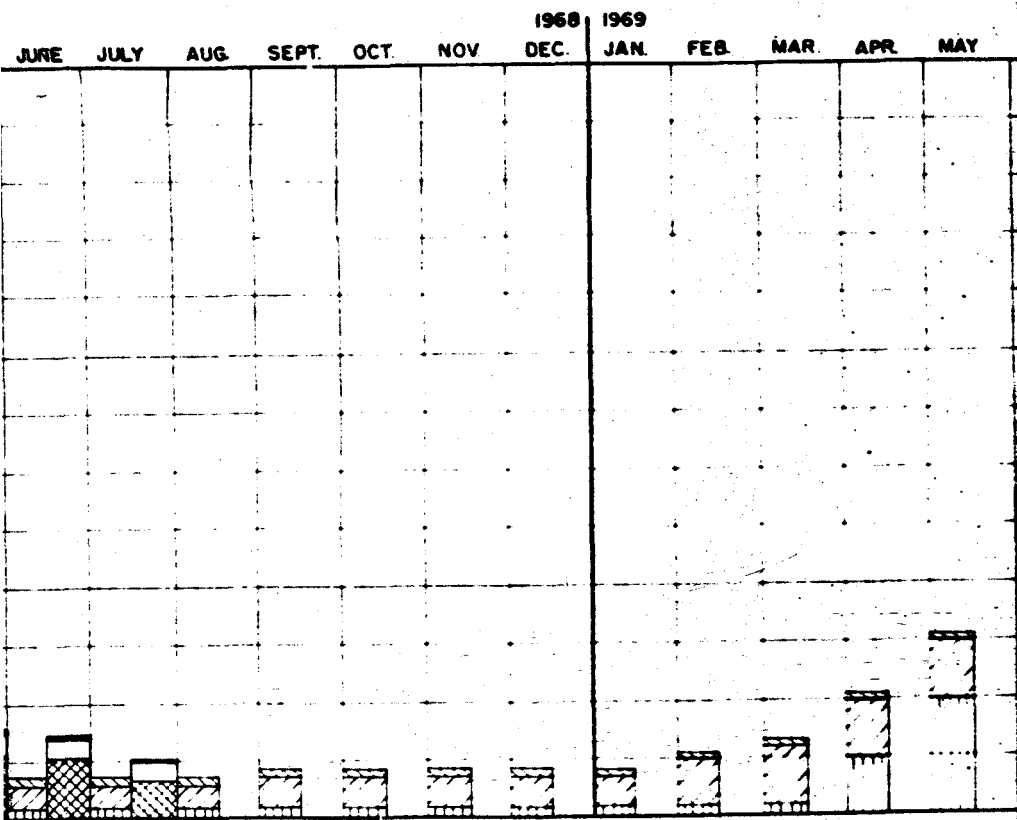
* Expenditures Include All Costs & Fees.

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MAY

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31.6 M	27.0 M										
JUNE	JULY	AUG.	SEPT.	OCT.	NOV.	DEC.	JAN.	FEB.	MAR.	APR.	MAY
1955.0 M	2175.0 M	2395.0 M	2615.0 M	2935.0 M	3255.0 M	3575.0 M	3995.0 M	4285.0 M	4575.0 M	4815.0 M	4975.0 M
1732.9 M	1759.9 M										

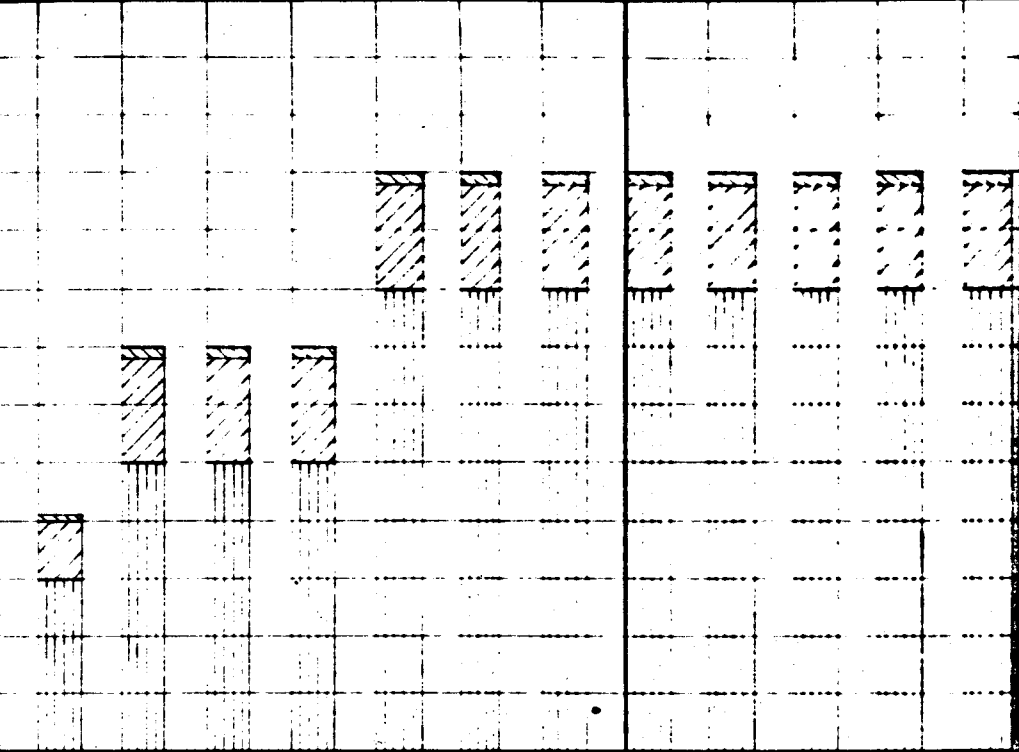
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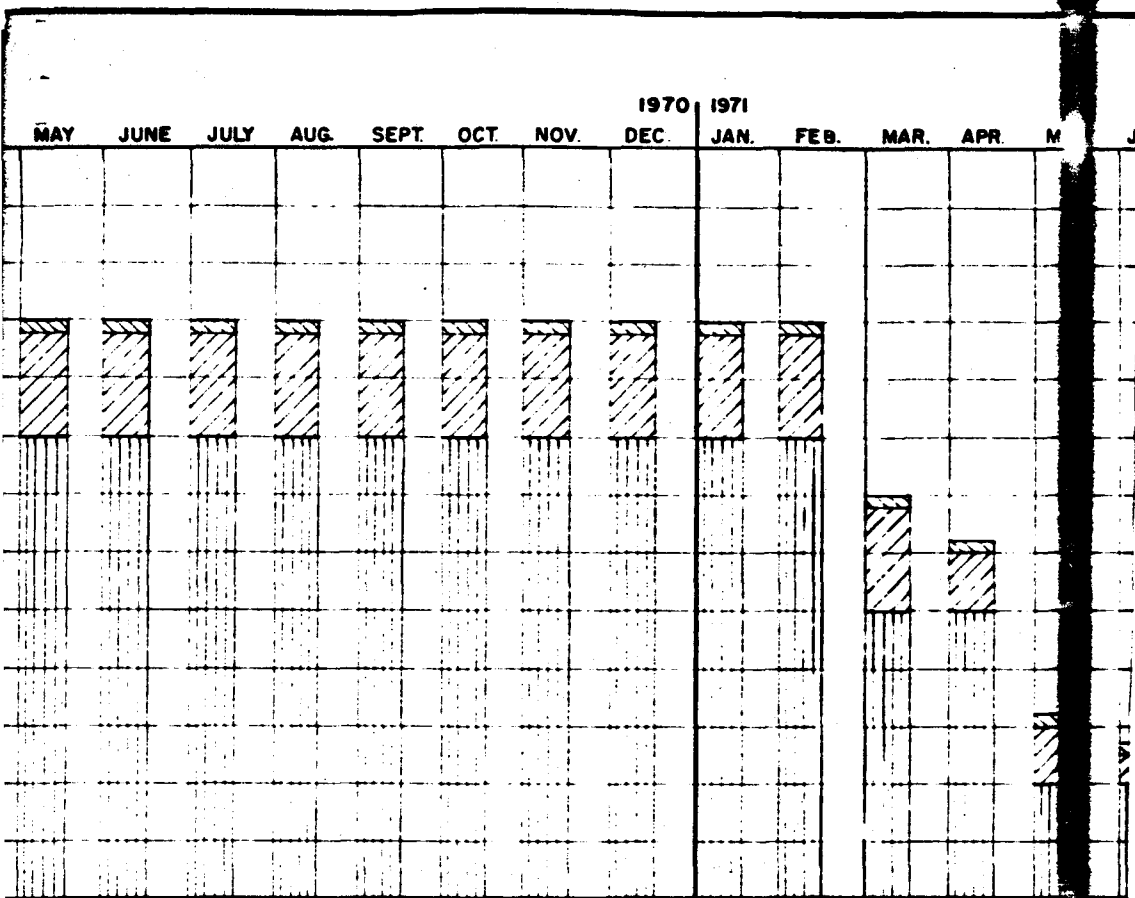
480.0 M 100.0 M 100.0 M 100.0 M 150.0 M 150.0 M 150.0 M 125.0 M 125.0 M 125.0 M 125.0 M 125.0 M

JUNE JULY AUG. SEPT. OCT. NOV. DEC. JAN. FEB. MAR. APR. MAY

5455.0 M 5555.0 M 5655.0 M 5755.0 M 5905.0 M 6055.0 M 6205.0 M 6330.0 M 6455.0 M 6580.0 M 6705.0 M 6830.0 M

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MAY	JUNE	JULY	AUG.	SEPT.	OCT.	NOV.	DEC.	JAN.	FEB.	MAR.	APR.	MAY	JUNE
6830.0 M	6955.0 M	7080.0 M	7205.0 M	7330.0 M	7455.0 M	7580.0 M	7705.0 M	7830.0 M	7955.0 M	8080.0 M	8205.0 M	8330.0 M	8455.0 M

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E. Technical Results and Program

Phase II - Bench-Scale Research

1. Experimental Projects (Projects 530, 532, etc.)

All experimental work has ceased and efforts are under way to prepare a final report on this phase of the CO₂ Acceptor development. The target completion date for the first draft of this report, which encompasses three years of experimental effort, is December 31, 1968.

2. Supporting Patent and Legal Activities (Project No. 534.00)

The current laboratory reports and memoranda were reviewed for determination of Subject Inventions.

3. Supporting Process and Economic Analysis (Project No. 533.00)

The OCR has requested that a portion of the final report on the bench-scale effort include a section devoted to an updated analysis of commercial economic feasibility. The same subject will be presented at the Cleveland meeting of the A.I.Ch.E. in May, 1969; draft of this paper is required in November, 1968.

Phase III - Pilot Plant Engineering

4. Catalytic Design (Project No. 533.30)

Nine contractors remain on the list of those supplying proposals for the construction and/or operation of the Rapid City pilot plant; Bechtel, Ford, Bacon & Davis, Parsons, and Peter Kiewit have declined. Minor questions from the various contractors are being handled by mail, with the provision that copies of all such questions and answers are mailed to all of the contractors.

Catalytic Construction has completed the embellishment of the Rapid City pilot plant model. Catalytic will assume the responsibility for installing the model in the Interior Building during the week of September 9.

A group of specialists from Ponca City visited Library for an in-depth review of the technical feasibility of the CO₂ Acceptor Process as well as detailed consideration of the process design, mechanical design, safety provisions, and start-up and shut-down provisions incorporated in the Rapid City pilot plant. While several areas were earmarked for further consideration, no major revisions of the present pilot plant design were recommended.

C. Subject Inventions

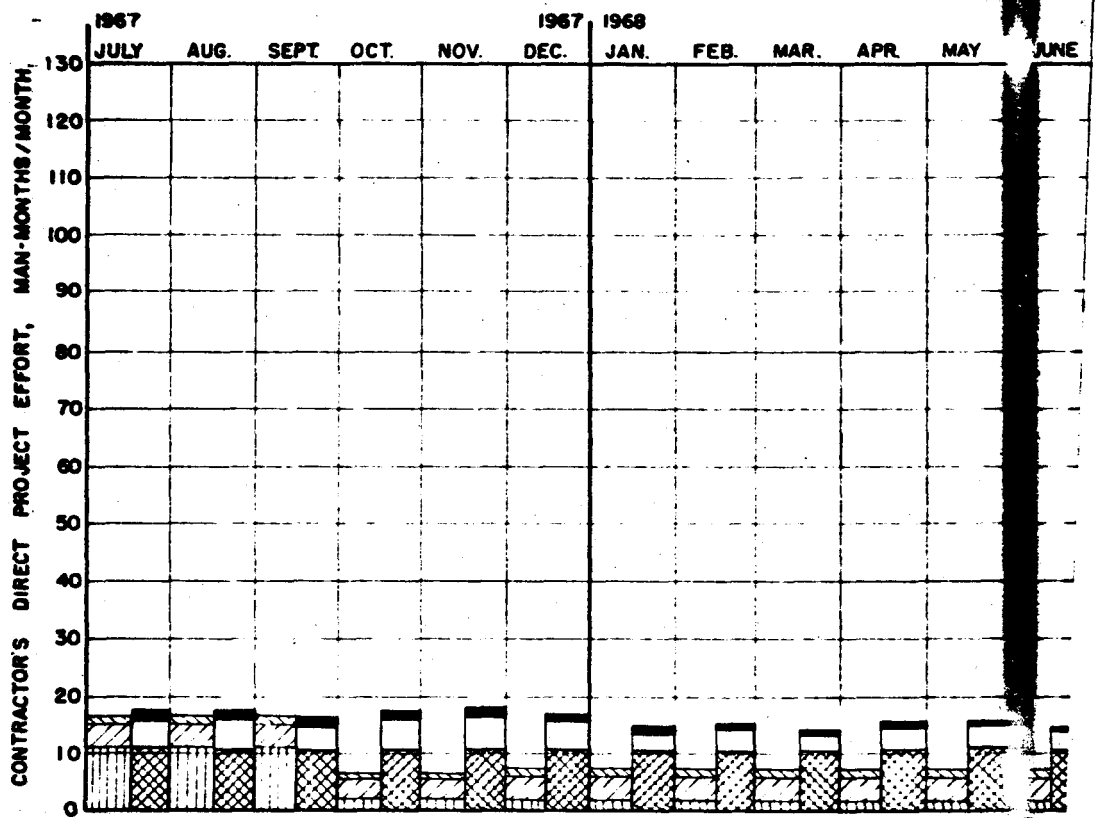
Subject Invention No. 2, entitled "Avoidance of Ash Deposition in the Regenerator of the CO₂ Acceptor Process," was transmitted to the OCR on August 30, 1968.

D. Financial Status

<u>Expense</u>	<u>Actual</u>	<u>Budget</u>
Cumulative through July	\$ 1,752,684	\$ 2,175,000
Expended in period (estimate)	17,000	220,000
Cumulative through August	\$ 1,769,684	\$ 2,395,000

Att. - Monthly Progress Chart

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MONTHLY EXPENDITURES*

EDICTED	103.7 M	72.2 M	92.0 M	94.0 M	94.0 M	52.0 M	40.0 M	20.0 M	70.0 M	70.0 M	120.0 M	20.0 M
TUAL	78.5 M	78.0 M	106.8 M	84.9 M	97.1 M	93.4 M	70.0 M	29.9 M	91.9 M	37.9 M	30.1 M	31.6 M

	JULY	AUG.	SEPT.	OCT.	NOV.	DEC.	JAN.	FEB.	MAR.	APR.	MAY	JUN.
EDICTED	1010.8 M	1083.0 M	1175.0 M	1269.0 M	1363.0 M	1415.0 M	1495.0 M	1479.0 M	1949.0 M	1615.0 M	1735.0 M	995.0 M
TUAL	981.3 M	1089.3 M	1166.1 M	1251.0 M	1348.1 M	1441.5 M	1511.5 M	1541.4 M	1633.3 M	1671.2 M	1701.3 M	732.0 M

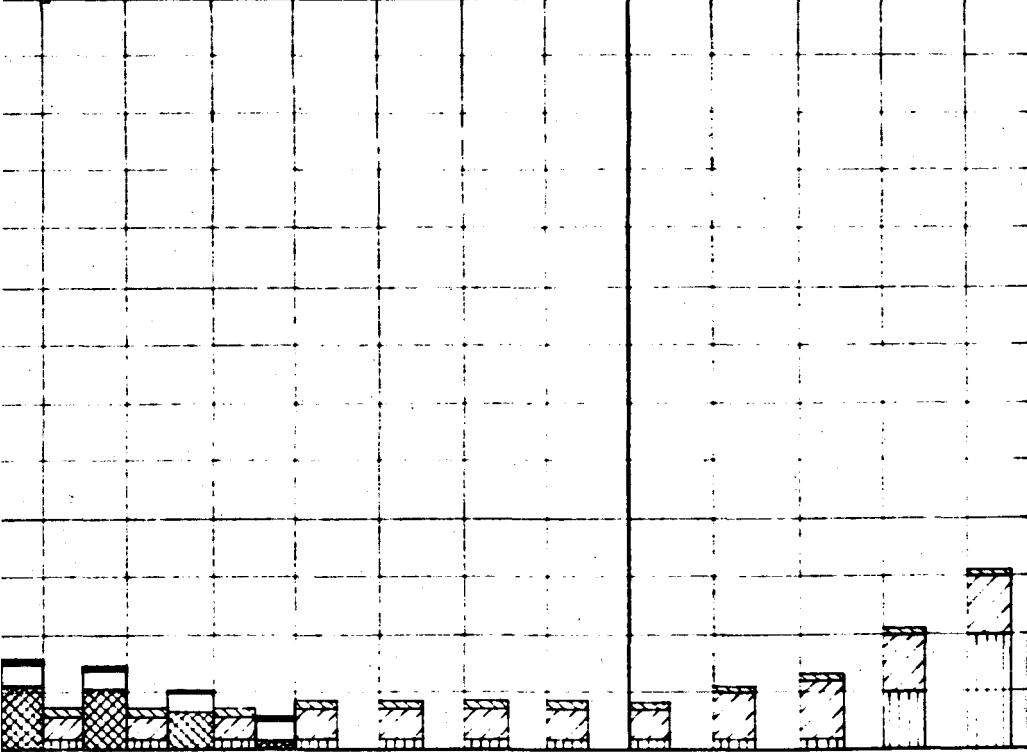
CUMULATIVE EXPENDITURES*

* Expenditures Include All Costs & Fees.

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	1968					1969						
Y	JUNE	JULY	AUG.	SEPT.	OCT.	NOV.	DEC.	JAN.	FEB.	MAR.	APR.	MAY



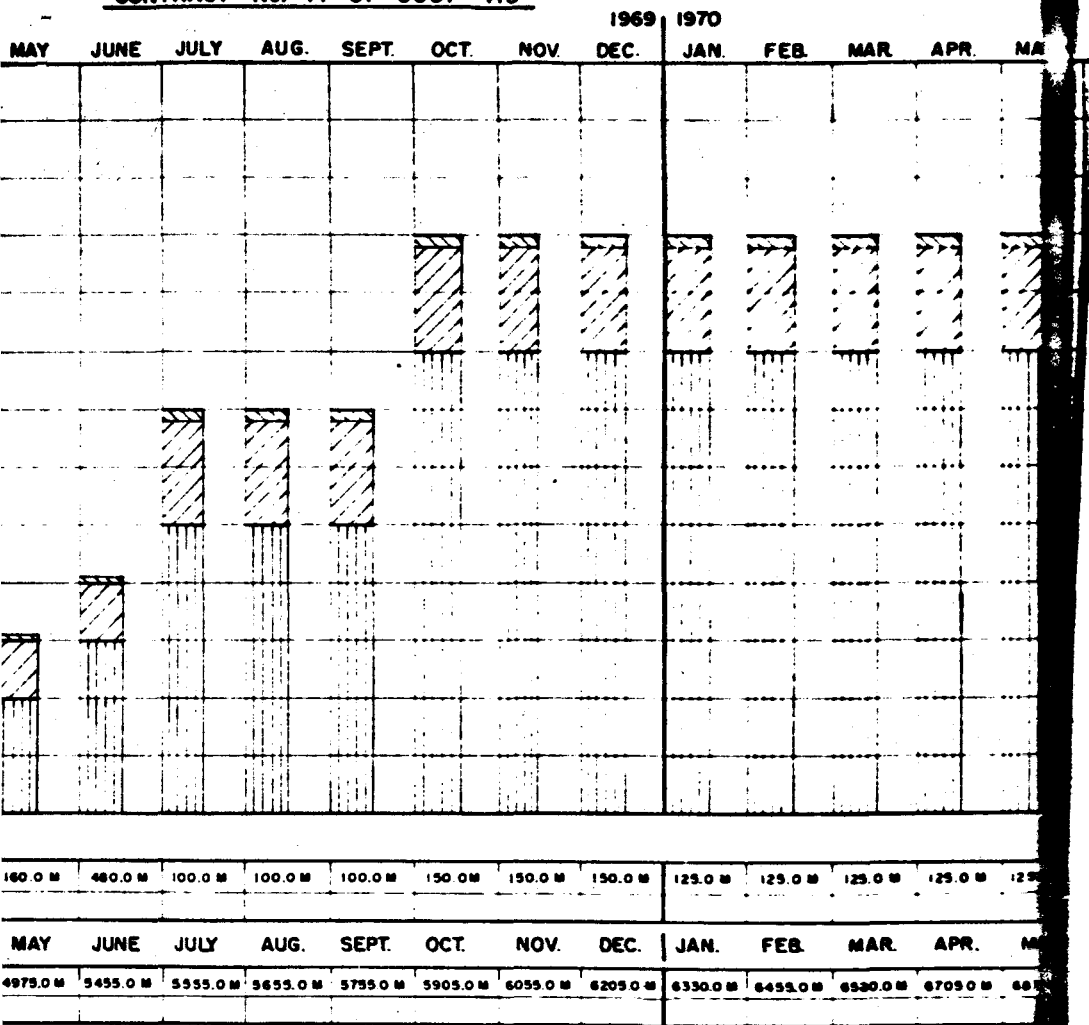
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M	31.6 M	19.8 M	17.0 M									
Y	JUNE	JULY	AUG.	SEPT.	OCT.	NOV.	DEC.	JAN.	FEB.	MAR.	APR.	MAY
OM	1955.0 M	2175.0 M	2395.0 M	2615.0 M	2935.0 M	3255.0 M	3575.0 M	3995.0 M	4285.0 M	4575.0 M	4815.0 M	4975.0 M
S M	1732.9 M	1732.7 M	1769.7 M									

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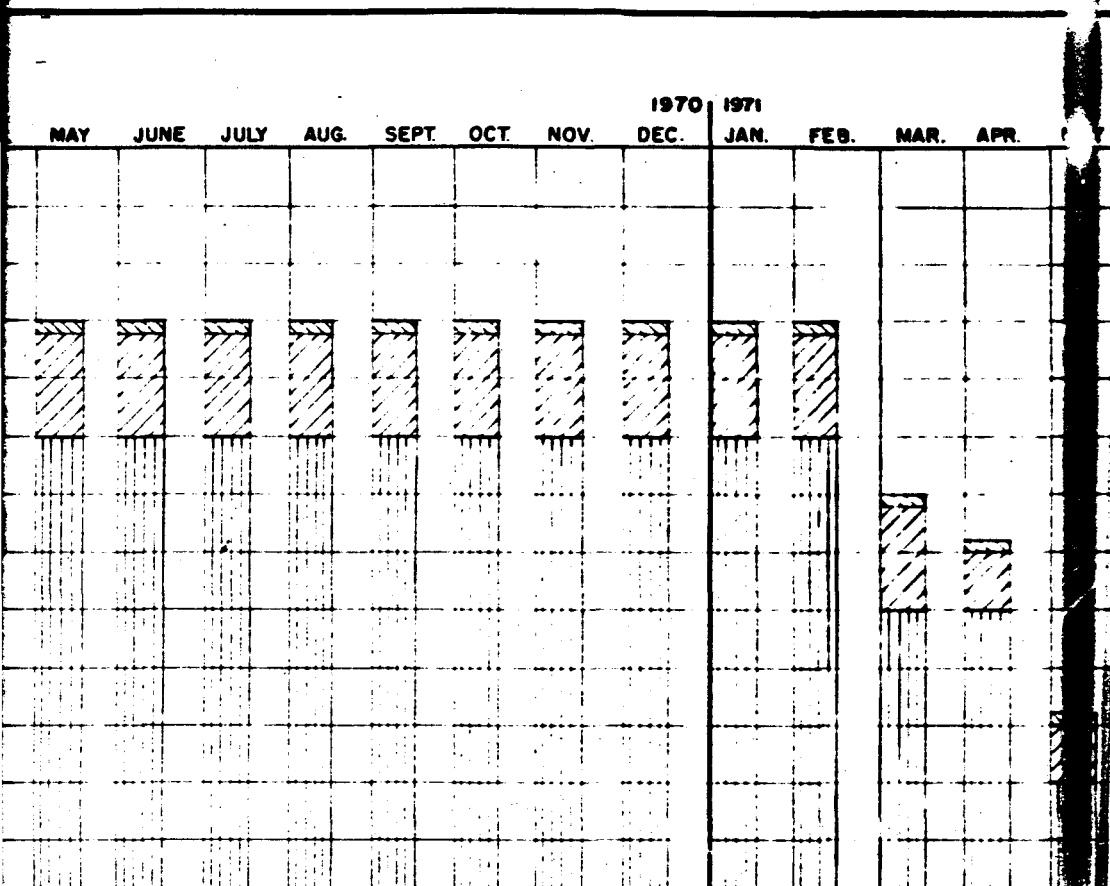
MONTHLY PROGRESS CHART

CONTRACT No. 14-01-0001-415



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


M	125.0 M	125.0 M	125.0 M	125.0 M	125.0 M	125.0 M	125.0 M	125.0 M	125.0 M	125.0 M	25.0 M	125.0 M
	MAY	JUNE	JULY	AUG.	SEPT.	OCT.	NOV.	DEC.	JAN.	FEB.	MAR.	APR.
M	6830.0 M	6955.0 M	7080.0 M	7205.0 M	7330.0 M	7455.0 M	7580.0 M	7705.0 M	7830.0 M	7955.0 M	8080.0 M	8205.0 M

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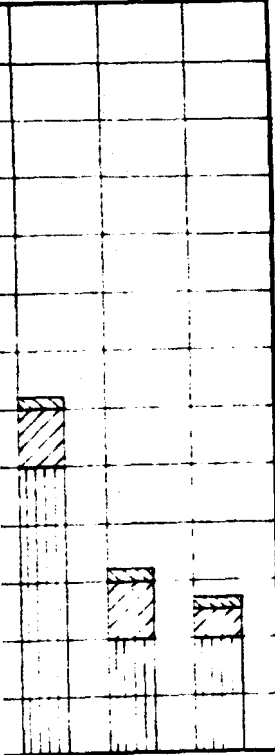
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DATE: August, 1968

1971
APR. MAY JUNE

	PREDICTED	ACTUAL
SUPERVISING		
PROFESSIONAL		 ⊙
TECHNICIAN		 ⊙⊙

⊙ Includes Supervisory Exempt Technicians.
 ⊙⊙ Includes Clerical.



125.0 M	125.0 M	170.0 M
APR.	MAY	JUNE
8205.0 M	8330.0 M	8500.0 M

MONTHLY PROGRESS CHART

OFFICE OF COAL RESEARCH
 DEPARTMENT OF THE INTERIOR

CONSOLIDATION COAL CO.
 RESEARCH DIVISION LIBRARY, PA.

CONTRACT No. 14-01-0001-415

5

PROGRESS REPORT NO. 52
 OCR CONTRACT NO. 14-01-0001-415(2)
 CONSOL LIGNITE GASIFICATION PROCESS DEVELOPMENT

September 1 to 30, 1968

A. Summary

Phase I - Feasibility Study

Complete.

Phase II - Bench Scale Experiments

All effort in this area is devoted to the preparation of a final report on Phase II activities.

Phase III - Pilot Plant Engineering

Contact was maintained with the various contractors preparing proposals on pilot plant construction and operation. Proposals are expected in early October from four major contractors.

Financial

Estimated expenditures in September were \$11,000. Cumulative expenses to date are \$1,777,353.

B. Technical Results and Program

Phase II - Bench-Scale Research

1. Experimental Projects (Projects 530, 532, etc.)

All experimental work has ceased and efforts are under way to prepare a final report on this phase of the CO₂ Acceptor development.

2. Supporting Patent and Legal Activities (Project No. 534.00)

The current laboratory reports and memoranda were reviewed for determination of Subject Inventions.

3. Supporting Process and Economic Analysis (Project No. 533.00)

At OCR's request, a paper entitled, "An Interim Study of the Economics of Pipeline Gas from Lignite" was prepared for presentation at the A.I.Ch.E. Meeting in Cleveland, Ohio in May, 1969.

The paper has been approved by the Consol Publication Committee and will be submitted to OCR on October 4, 1968.

Phase III - Pilot Plant Engineering

4. Catalytic Design (Project No. 533.30)

Kellogg and Braun have declined to submit proposals on the Rapid City pilot plant. Thus proposals from major contractors are now limited to Catalytic Construction, Dravo, Procon, and Stearns-Roger. Minor questions from these contractors were handled during the month.

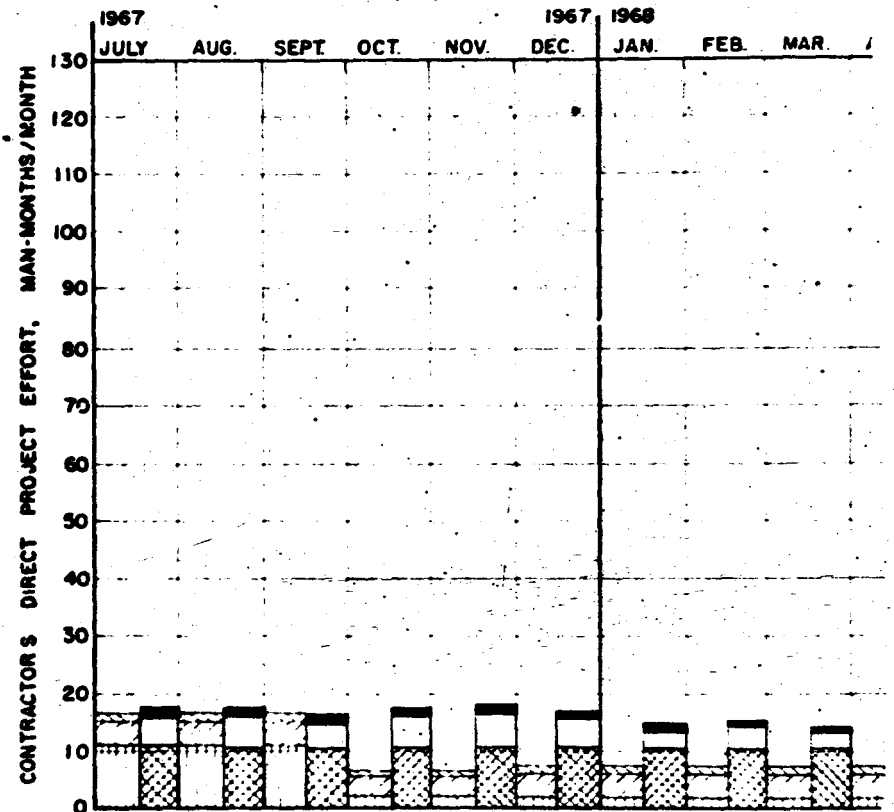
C. Subject Inventions

None.

D. Financial Status

Expense	Actual	Budget
Cumulative through August	\$ 1,766,353	\$ 2,395,000
Expended in period (estimate)	11,000	220,000
Cumulative through September	\$ 1,777,353	\$ 2,615,000

Att. - Monthly Progress Chart



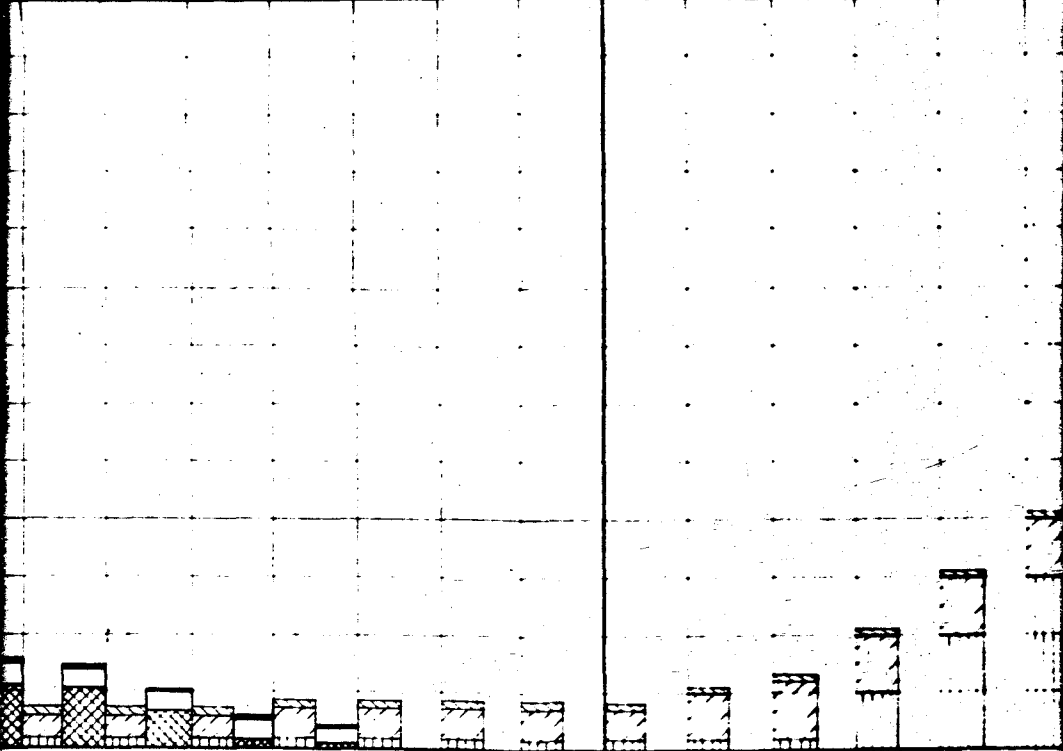
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MONTHLY EXPENDITURES*										
PREDICTED	103.7 M	72.2 M	92.0 M	94.0 M	94.0 M	52.0 M	40.6 M	20.0 M	70.0 M	70
ACTUAL	75.5 M	76.0 M	106.9 M	84.9 M	97.1 M	93.4 M	70.0 M	23.9 M	91.9 M	37
PREDICTED	1010.8 M	1083.0 M	1177.0 M	1269.0 M	1363.0 M	1415.0 M	1455.0 M	1475.0 M	1543.0 M	1615
ACTUAL	981.3 M	1059.3 M	1166.1 M	1251.0 M	1348.1 M	1441.5 M	1511.5 M	1541.4 M	1633.3 M	1671

CUMULATIVE EXPENDITURES*

* Expenditures Include All Costs & Fees.

MONTH

1968 1969
JUNE JULY AUG. SEPT. OCT. NOV. DEC. JAN. FEB. MAR. APR. MAY JUN.



220.0 M	220.0 M	220.0 M	220.0 M	320.0 M	320.0 M	320.0 M	420.0 M	290.0 M	290.0 M	240.0 M	160.0 M	400.0 M
31.6 M	19.8 M	15.7 M	11.0 M									
JUNE	JULY	AUG.	SEPT.	OCT.	NOV.	DEC.	JAN.	FEB.	MAR.	APR.	MAY	JUN.
1955.0 M	2175.0 M	2395.0 M	2615.0 M	2935.0 M	3255.0 M	3575.0 M	3995.0 M	4285.0 M	4575.0 M	4815.0 M	4975.0 M	5435.0 M
1732.9 M	1752.7 M	1766.4 M	1777.4 M									

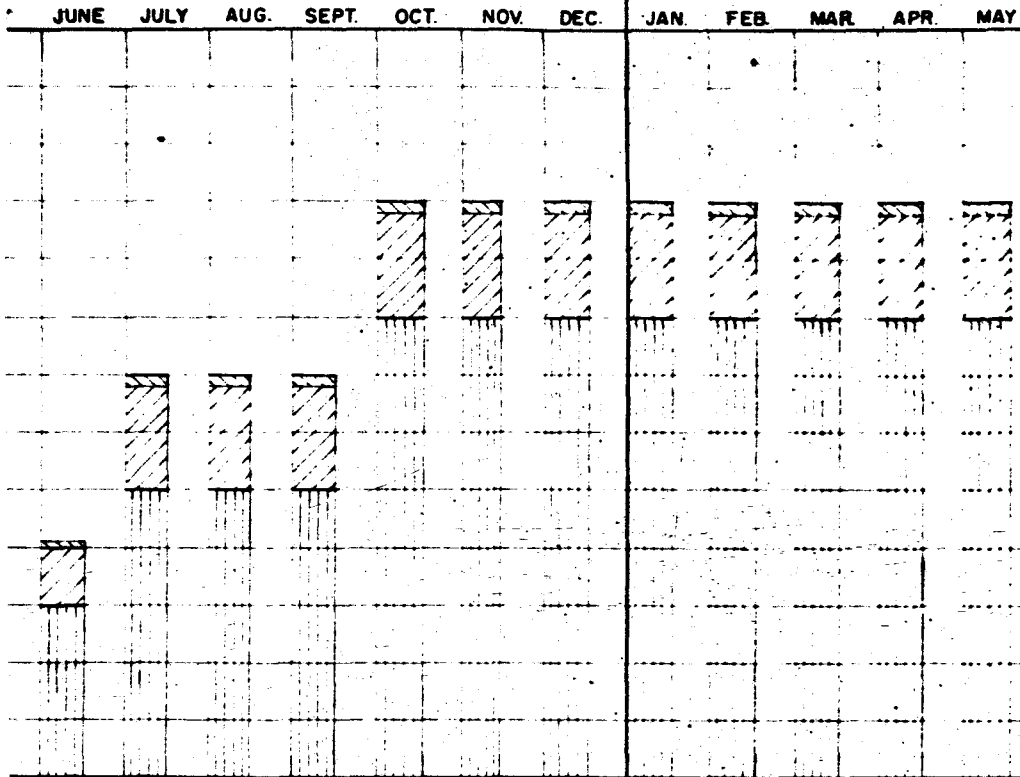
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MONTHLY PROGRESS CHART

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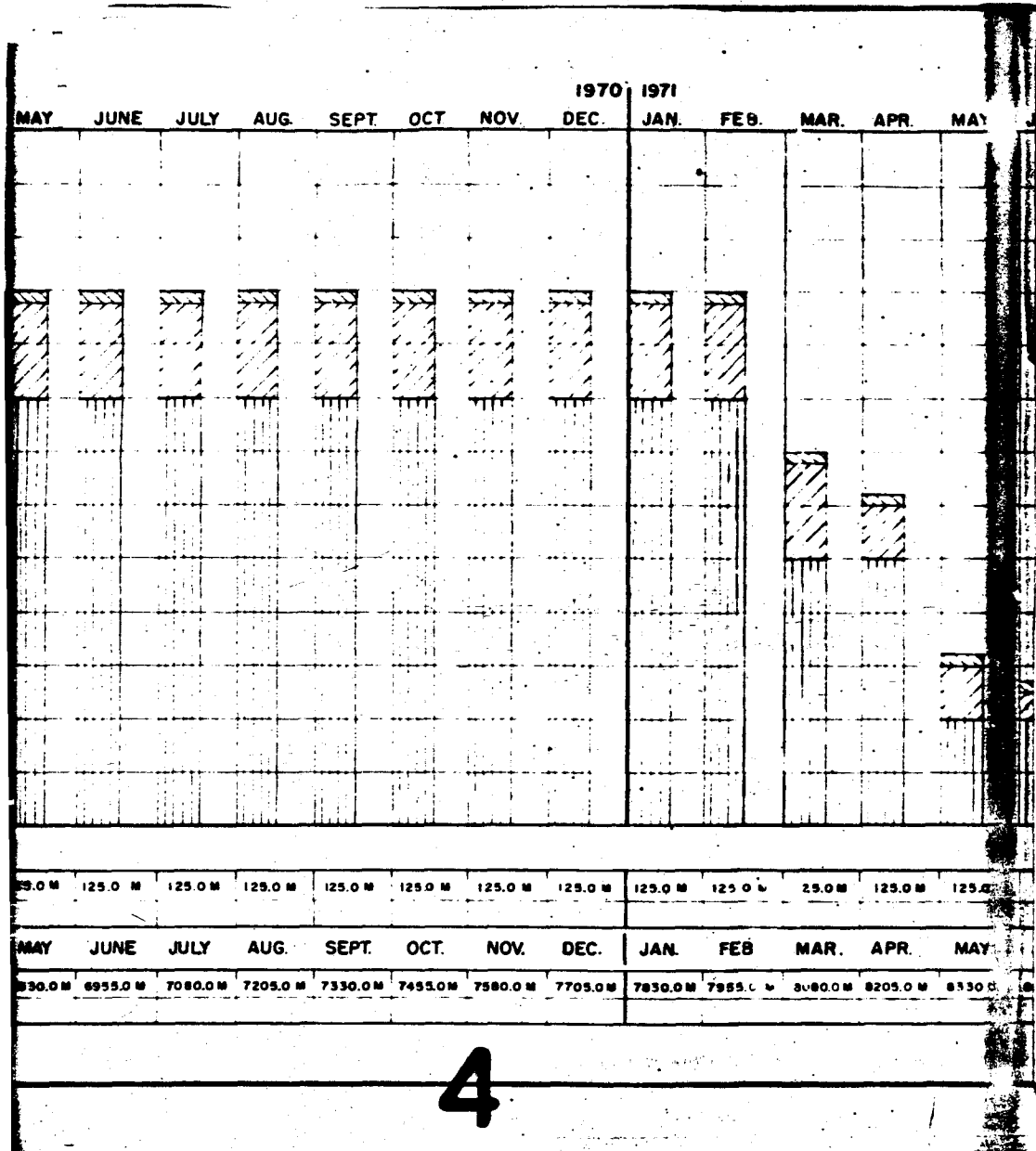
1969 | 1970



480.0 M	100.0 M	100.0 M	100.0 M	150.0 M	150.0 M	150.0 M	125.0 M	125.0 M	125.0 M	125.0 M	125.0 M	125.0 M	125.0 M
JUNE	JULY	AUG.	SEPT.	OCT.	NOV.	DEC.	JAN.	FEB.	MAR.	APR.	MAY		
5485.0 M	5585.0 M	5685.0 M	5785.0 M	5905.0 M	6055.0 M	6205.0 M	6330.0 M	6455.0 M	6580.0 M	6705.0 M	6830.0 M		

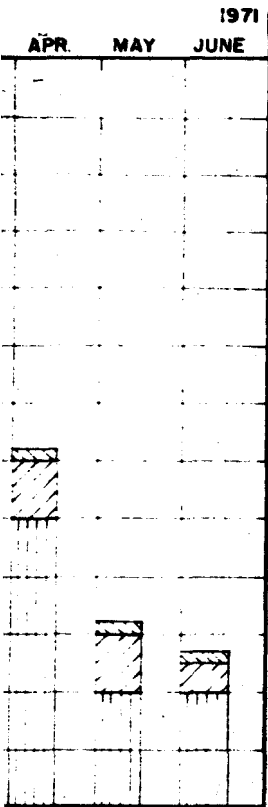
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DATE: September, 1968



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SUPERVISING		
PROFESSIONAL		
TECHNICIAN		

⊕ Includes Supervisory Exempt Technicians.
 ⊗ Includes Clerical.

125.0 M	125.0 M	170.0 M
APR.	MAY	JUNE
8205.0 M	8330.0 M	8500.0 M

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MONTHLY PROGRESS CHART
 OFFICE OF COAL RESEARCH
 DEPARTMENT OF THE INTERIOR
CONSOLIDATION COAL CO.
 RESEARCH DIVISION LIBRARY, PA.
 CONTRACT No. 14-01-0001-415

CEVC-ATA--
 October 1968

PROGRESS REPORT NO. 53
 OCR CONTRACT NO. 14-01-0001-415(2)
CONSOL LIGNITE GASIFICATION PROCESS DEVELOPMENT

A. Summary

Phase I - Feasibility Study

Complete.

Phase II - Bench-Scale Experiments

All effort in this area is devoted to the preparation of a final report on Phase II activities.

Phase III - Pilot Plant Engineering

Proposals were received in early October from four major contractors for the final engineering, construction, and operation of the Rapid City pilot plant. The original proposals have been supplemented by meetings with each of the contractors, and recommendations concerning contractor preference will be submitted to OCR in early November.

Financial

Estimated expenditures in October were \$10,000. Cumulative expenses to date are \$1,786,270.

B. Technical Results and Program

Phase II - Bench-Scale Research

1. Experimental Projects (Project 530, 532, etc.)

All experimental work has ceased and efforts continue to prepare a final report on this phase of the CO₂ Acceptor development. It is now projected that the final report will be submitted to OCR for approval on or before February 1, 1969.

2. Supporting Patent and Legal Activities (Project No. 534.00)

The current laboratory reports and memoranda were reviewed for determination of Subject Inventions.

3. Supporting Process and Economic Analysis (Project No. 533.00)

No work was done on this project during the report period. It is planned in mid-November to initiate the preparation of an economic section in the final report which will define the impact on potential commercial economics of the results achieved in the bench-scale experiments.

Phase III - Pilot Plant Engineering

4. Catalytic Design (Project No. 533.30)

Detailed proposals for pilot plant construction and operations were received from Catalytic Construction, Dravo, Procon, and Stearns-Roger. During the month, clarification meetings were held with each of the contractors to review the proposals, to solicit opinions concerning potential savings or improvements in the present design, and to define the basis whereby the contractors would assume responsibility for the structural and mechanical integrity of the plant. Recommended contractor preference for further negotiations will be submitted to OCR for approval in early November.

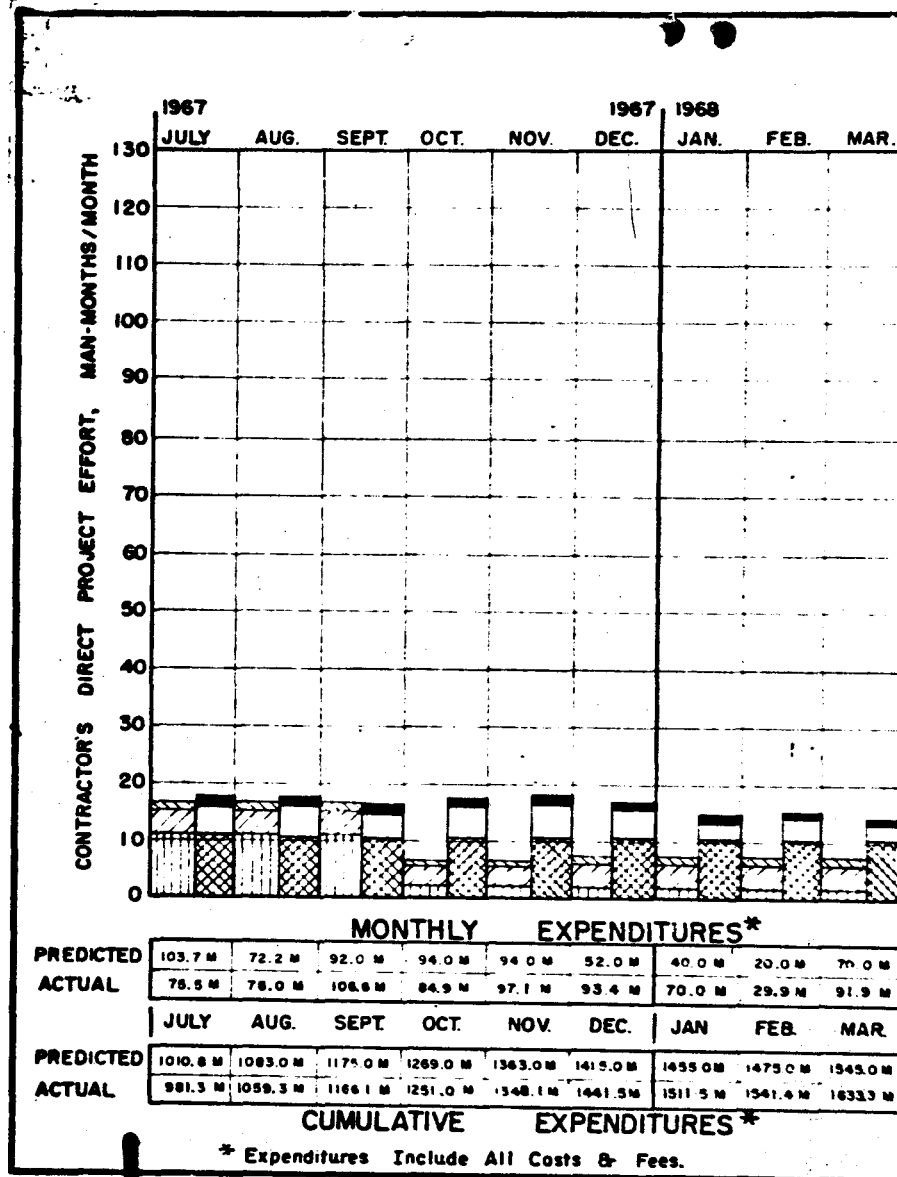
C. Subject Inventions

None.

D. Financial Status

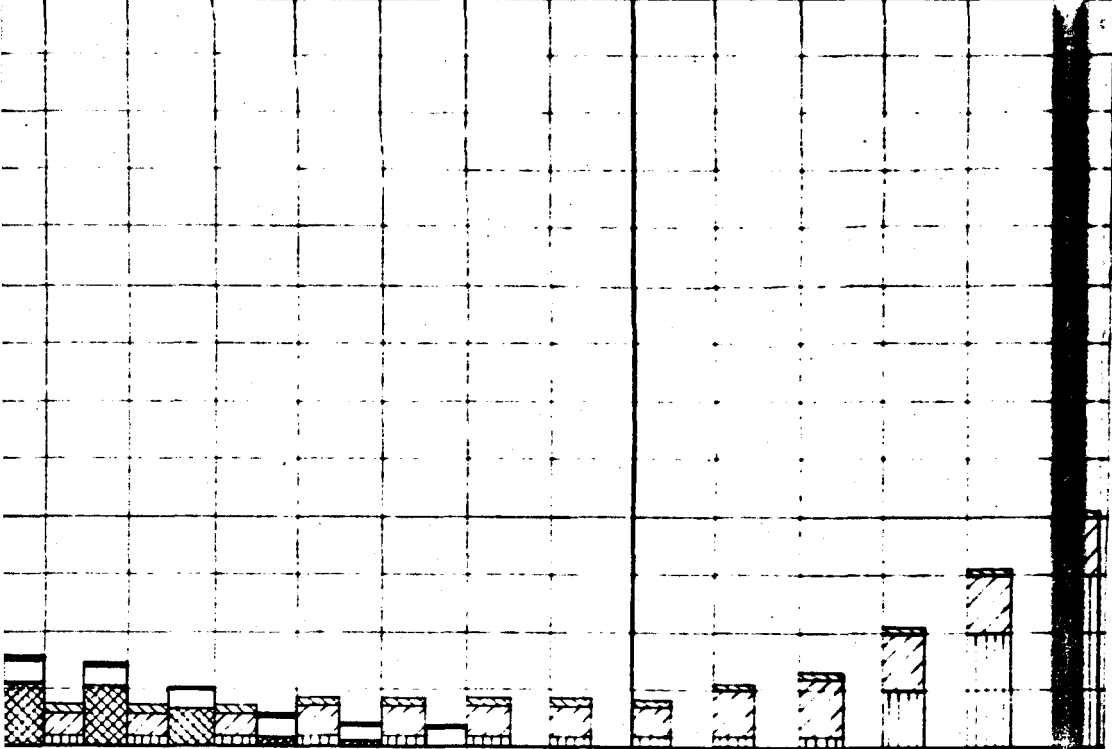
Expense	Actual	Budget
Cumulative through September	\$ 1,776,270	\$ 2,615,000
Expended in Period (estimate)	10,000	320,000
Cumulative through October	\$ 1,786,270	\$ 2,935,000

11/11/68
Att. - Monthly Progress Chart



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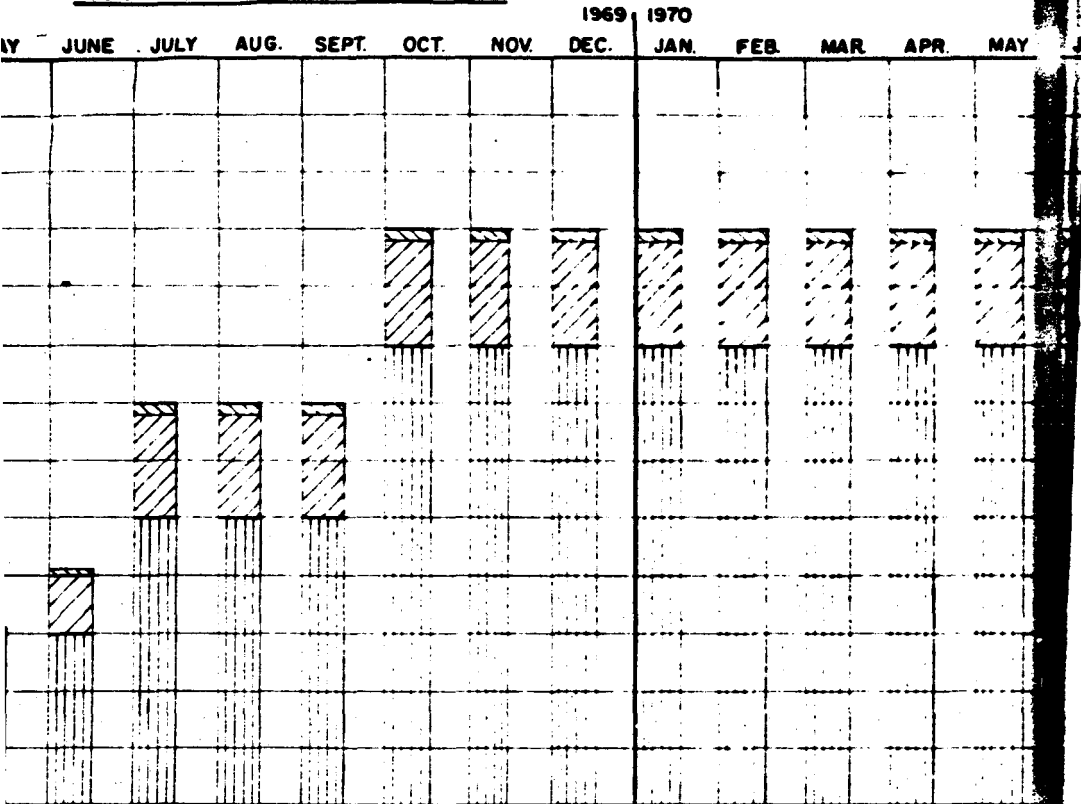
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M	31.6 M	19.8 M	13.7 M	9.9 M	10.0 M								
Y	JUNE	JULY	AUG.	SEPT.	OCT.	NOV.	DEC.	JAN.	FEB.	MAR.	APR.	MAY	JUNE
OM	1955.0 M	2175.0 M	2395.0 M	2615.0 M	2935.0 M	3255.0 M	3575.0 M	3995.0 M	4265.0 M	4575.0 M	4815.0 M	4975.0 M	5185.0 M
M	1732.9 M	1752.7 M	1766.4 M	1776.3 M	1786.3 M								

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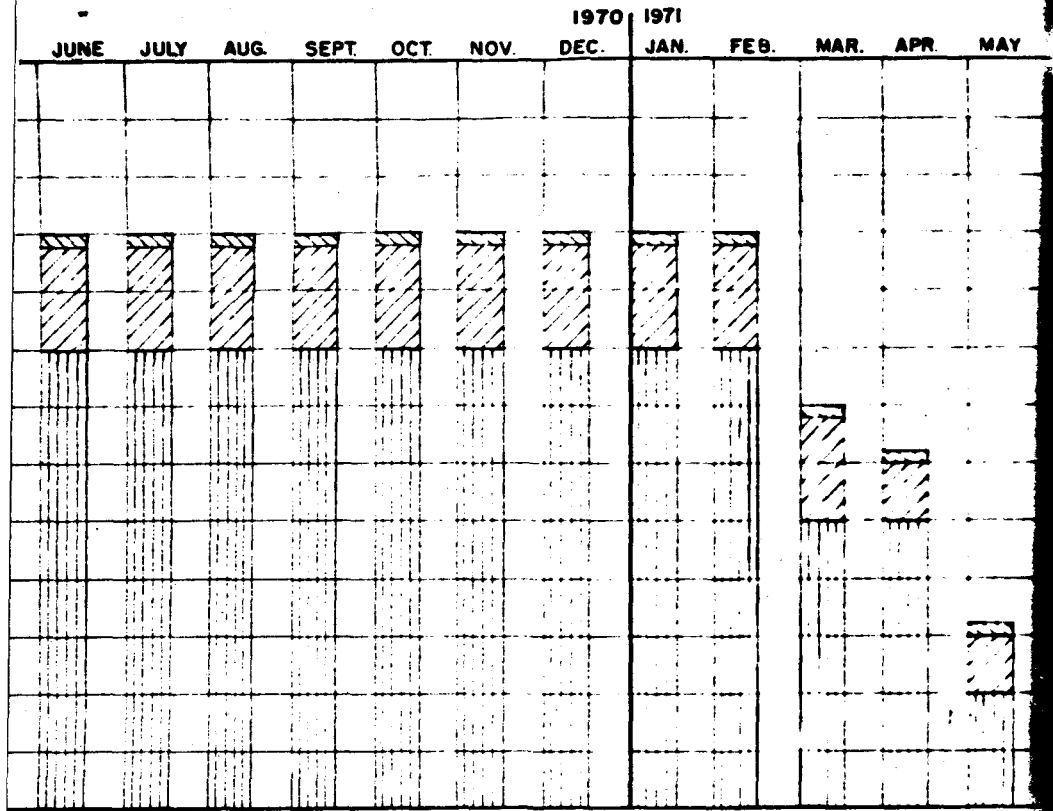
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JUNE	JULY	AUG.	SEPT.	OCT.	NOV.	DEC.	JAN.	FEB.	MAR.	APR.	MAY
8855.0 M	7080.0 M	7205.0 M	7330.0 M	7495.0 M	7580.0 M	7705.0 M	7830.0 M	7955.0 M	8080.0 M	8205.0 M	8330.0 M

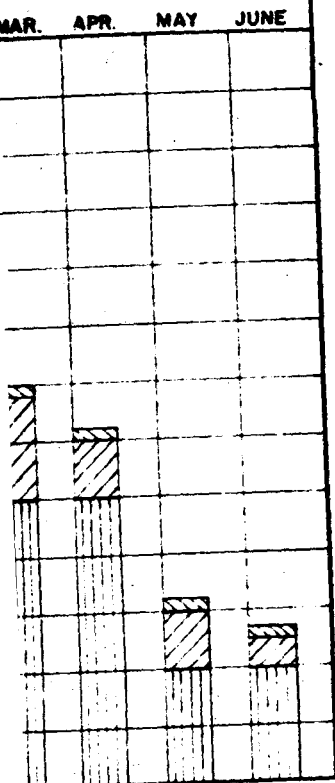
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 NOVEMBER 1968

DATE: October, 1968

1971



PREDICTED **ACTUAL**

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PROFESSIONAL [diagonal lines] [white with circle]

TECHNICIAN [vertical lines] [diagonal lines with circles]

⊙ Includes Supervisory Exempt Technicians.
 ⊙⊙ Includes Clerical.

1971	MAR.	APR.	MAY	JUNE
100.0 M	125.0 M	129.0 M	170.0 M	

MONTHLY PROGRESS CHART

OFFICE OF COAL RESEARCH
 DEPARTMENT OF THE INTERIOR

CONSOLIDATION COAL CO.
 RESEARCH DIVISION LIBRARY, PA.

CONTRACT No. 14-01-0001-415

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PROGRESS REPORT NO. 54
 OCR CONTRACT NO. 14-01-0001-415(2)
 CONSOL. LIGNITE GASIFICATION PROCESS DEVELOPMENT

A. Summary

Phase I - Feasibility Study

Complete.

Phase II - Bench-Scale Experiments

All effort in this area is devoted to preparation of a final report on Phase II activities.

Phase III - Pilot Plant Engineering

RM-11990, submitted to OCF on November 11, 1968, contains an evaluation of construction and operation proposals for the Rapid City pilot plant. It was recommended that negotiations be initiated with Stearns-Roger to arrive at an acceptable guaranteed maximum, split savings contract.

Financial

Estimated expenditures in November were \$9,000. Cumulative expenses to date are \$1,794,632.

B. Technical Results and Program

Phase II - Bench-Scale Research

1. Experimental Projects (Projects 530, 532, etc.)

Work continues on the preparation of a final report on this phase of CO₂ Acceptor development. It is projected that the final report will be submitted to OCR for approval on or about February 1, 1969.

2. Supporting Patent and Legal Activities (Project No. 534.00)

The current laboratory reports and memoranda were reviewed for determination of Subject Inventions.

3. Supporting Process and Economic Analysis (Project No. 533.00)

Work was started in late November on the preparation of an economics section for the final report.

Phase III - Pilot Plant Engineering

4. Catalytic Design (Project No. 533.30)

Consol's appraisal of the construction and operation proposals for the Rapid City pilot plant were submitted to OCR on November 11, 1968, with the recommendation that negotiations be initiated with Stearns-Roger for a maximum price, split savings contract. It would appear that the cost of the Rapid City pilot plant construction will be between \$7.8 - 8.0 MM. The estimated cost for the 30-month operating program as set forth in our Invitation for Operating Proposals is approximately \$4.5 MM.

During November, a further review of the hot piping, valves, and associated insulation in the dolomite transfer lines was initiated. This study is continuing with the help of equipment vendors.

The CSG pilot plant model was shown at the AGA Meeting in Pittsburgh on November 21 and 22. The model is now stored in Library.

C. Subject Inventions

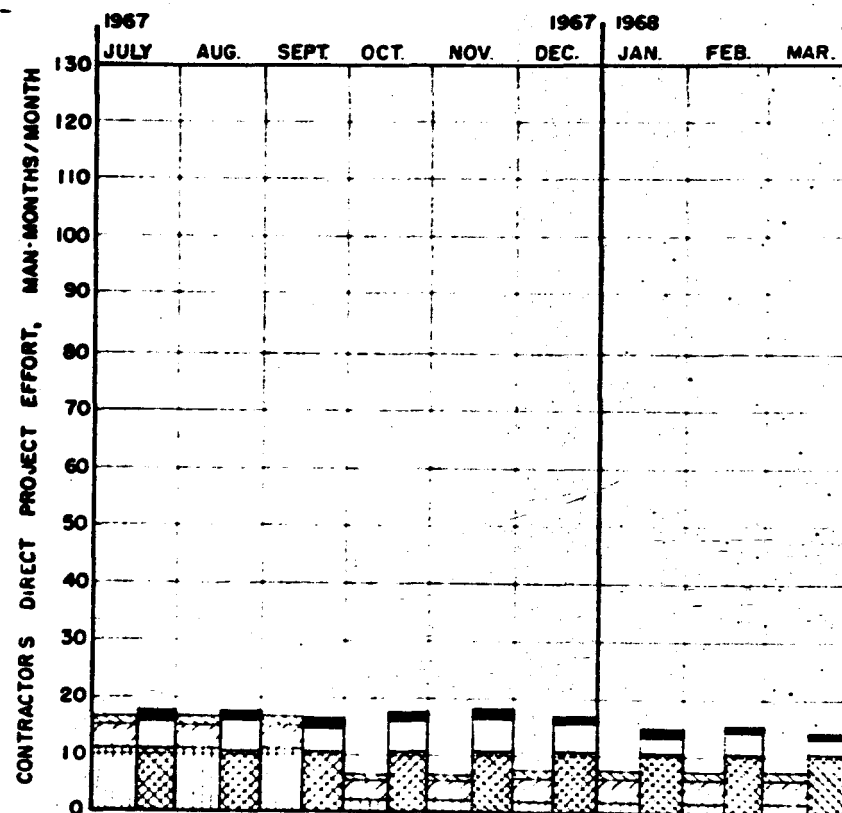
None.

D. Financial Status

Expense	Actual	Budget
Cumulative through October	\$1,785,682	\$2,935,000
Expended in Period (estimate)	9,000	320,000
Cumulative through November	\$1,794,682	\$3,255,000

12/9/68

Att. - Monthly Progress Chart



	MONTHLY EXPENDITURES*								
	1967	1967			1967			1968	
	JULY	AUG.	SEPT.	OCT.	NOV.	DEC.	JAN.	FEB.	MAR.
PREDICTED	103.7 M	72.2 M	92.0 M	94.0 M	94.0 M	92.0 M	40.0 M	20.0 M	70.0 M
ACTUAL	78.5 M	78.0 M	106.6 M	84.9 M	97.1 M	93.4 M	70.0 M	29.9 M	91.9 M
PREDICTED	1010.8 M	1083.0 M	1175.0 M	1269.0 M	1363.0 M	1415.0 M	1455.0 M	1479.0 M	1545.0 M
ACTUAL	981.3 M	1059.3 M	1166.1 M	1251.0 M	1348.1 M	1441.5 M	1511.5 M	1541.4 M	1633.7 M

CUMULATIVE EXPENDITURES*

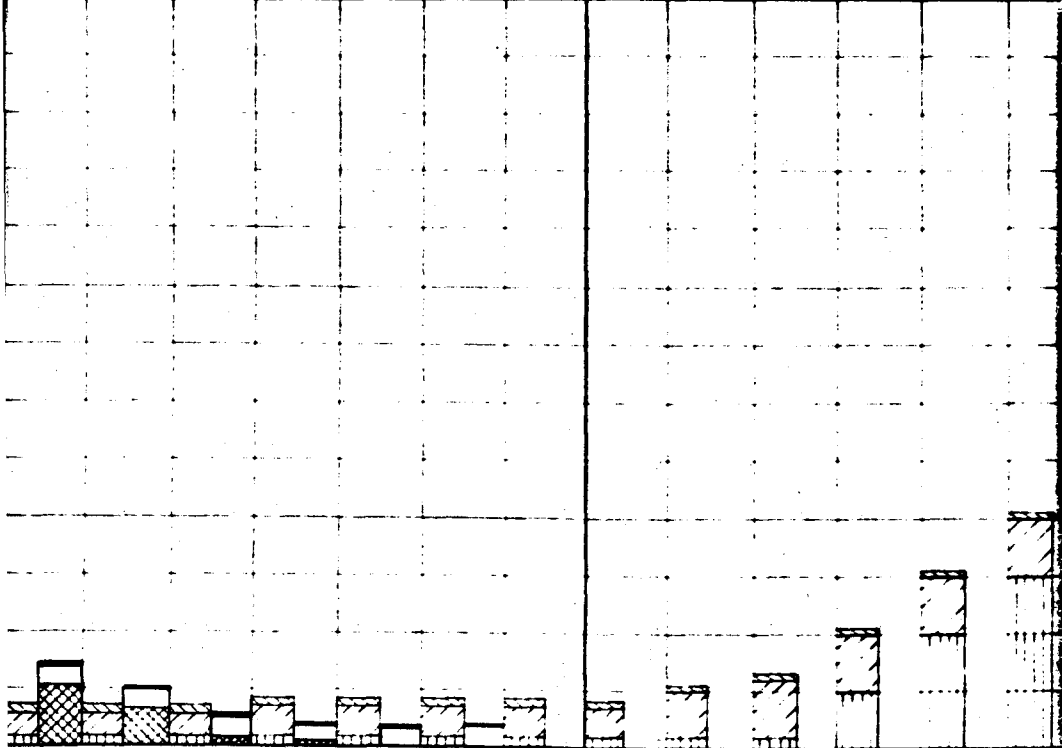
* Expenditures Include All Costs & Fees.

MONTHLY

CITY

1968 1969

JUNE JULY AUG. SEPT. OCT. NOV. DEC. JAN. FEB. MAR. APR. MAY JUN



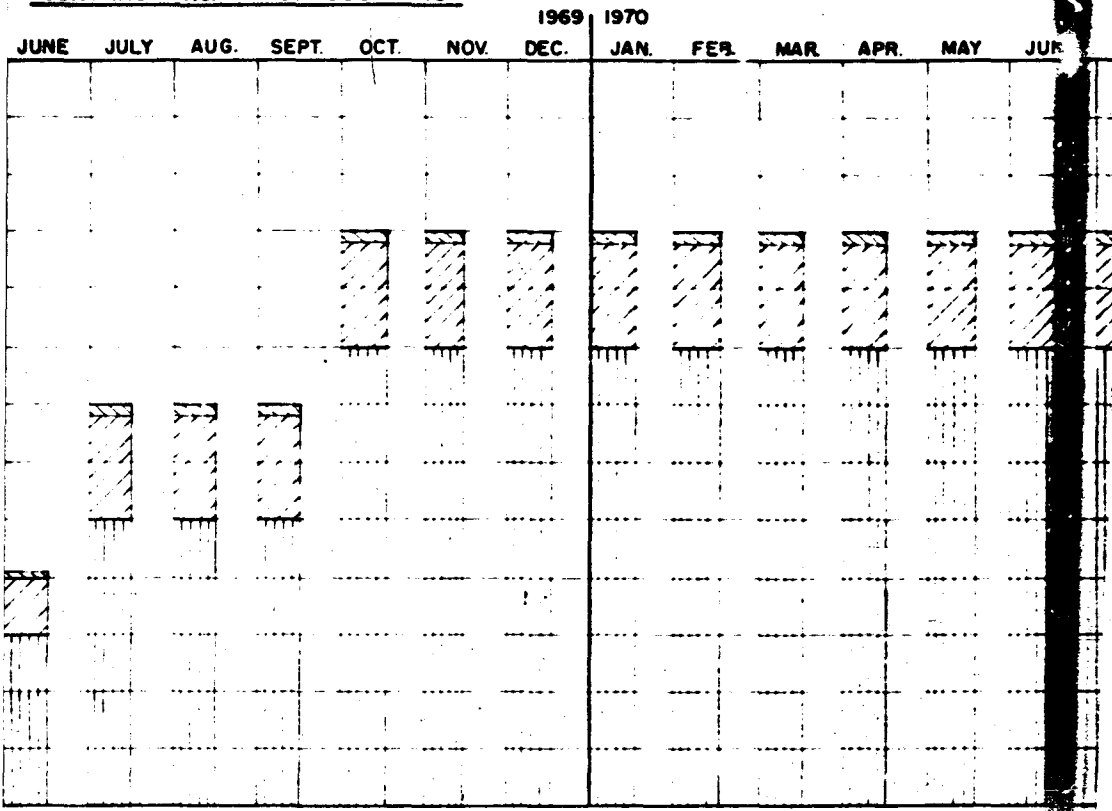
220.0 M	220.0 M	220.0 M	220.0 M	320.0 M	320.0 M	320.0 M	420.0 M	290.0 M	290.0 M	240.0 M	160.0 M	480.0 M
31.6 M	19.8 M	13.7 M	9.9 M	34 M	90 M							
JUNE	JULY	AUG.	SEPT.	OCT.	NOV.	DEC.	JAN.	FEB.	MAR.	APR.	MAY	JUNE
1955.0 M	2175.0 M	2395.0 M	2615.0 M	2935.0 M	3255.0 M	3575.0 M	3995.0 M	4265.0 M	4575.0 M	485.0 M	4975.0 M	5455.0 M
732.9 M	1752.7 M	1766.4 M	1776.3 M	1785.7 M	1794.7 M							

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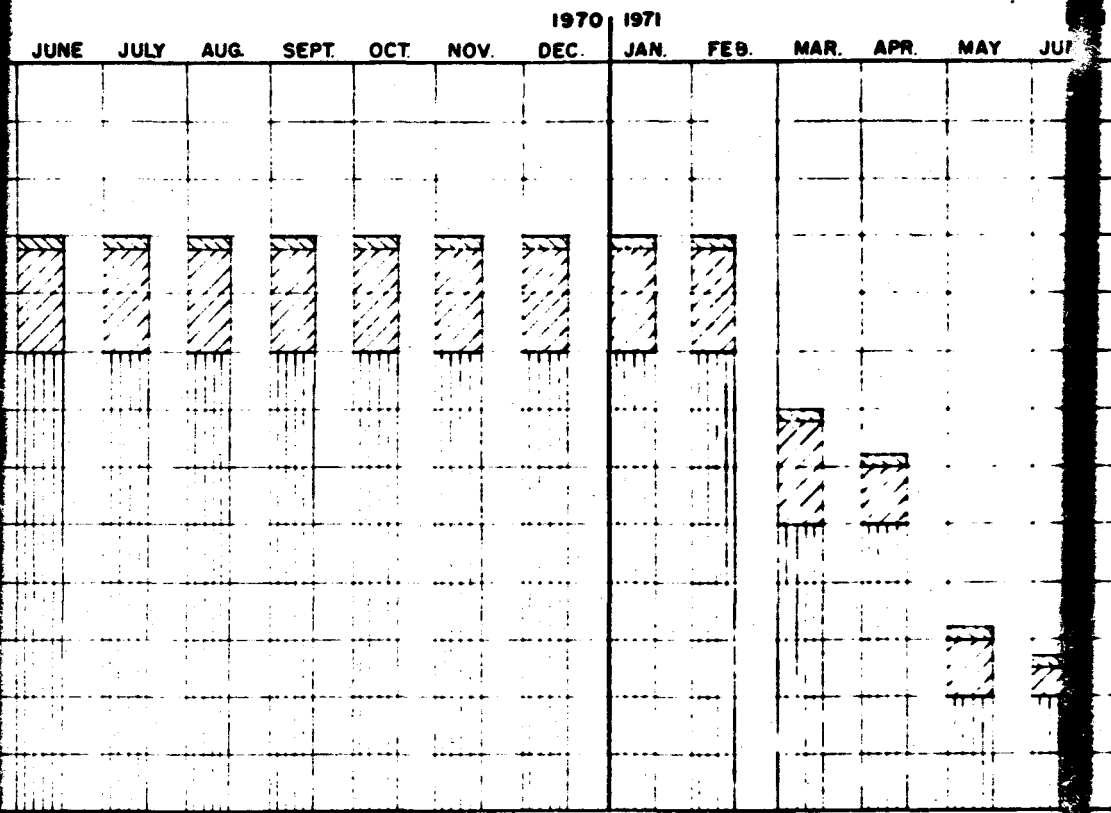
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CONTRACT No. 14-01-0001-415



400.0 M	500.0 M	600.0 M	700.0 M	800.0 M	900.0 M	1000.0 M	1100.0 M	1200.0 M	1300.0 M	1400.0 M	1500.0 M	1600.0 M	1700.0 M	1800.0 M	1900.0 M	2000.0 M
JUNE	JULY	AUG.	SEPT.	OCT.	NOV.	DEC.	JAN.	FEB.	MAR.	APR.	MAY	JUN.	JULY	AUG.	SEPT.	OCT.
5495.0 M	5555.0 M	5655.0 M	5755.0 M	5905.0 M	6055.0 M	6205.0 M	6330.0 M	6455.0 M	6580.0 M	6705.0 M	6830.0 M	6955.0 M	7080.0 M	7205.0 M	7330.0 M	7455.0 M

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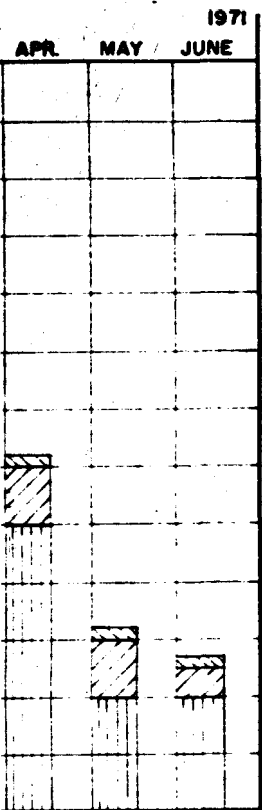


125.0 M	125.0 M	125.0 M	125.0 M	125.0 M	125.0 M	125.0 M	125.0 M	125.0 M	25.0 M	125.0 M	125.0 M	125.0 M	125.0 M
JUNE	JULY	AUG.	SEPT.	OCT.	NOV.	DEC.	JAN.	FEB.	MAR.	APR.	MAY	JUN.	JULY
6955.0 M	7060.0 M	7205.0 M	7330.0 M	7455.0 M	7580.0 M	7705.0 M	7830.0 M	7955.0 M	8080.0 M	8205.0 M	8330.0 M	8455.0 M	8580.0 M

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December 1968
 CEDC-MPR--55

DATE: Dec 1968



PREDICTED **ACTUAL**

SUPERVISING [diagonal lines] [solid black]

PROFESSIONAL [diagonal lines] [white with circle]

TECHNICIAN [vertical lines] [diagonal lines with circles]

⊕ Includes Supervisory Exempt Technicians.
 ⊕⊕ Includes Clerical.

MONTHLY PROGRESS CHART

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PROGRESS REPORT NO. 55
 OCR CONTRACT NO. 14-01-0001-415(2)
CONSOL LIGNITE GASIFICATION PROCESS DEVELOPMENT

A. Summary

Phase I - Feasibility Study

Complete.

Phase II - Bench-Scale Experiments

All effort in this area is devoted to preparation of a final report on Phase II activities. This includes work to update the potential commercial economics of the lignite gasification process.

Phase III - Pilot Plant Engineering

No work was done in this area.

Financial

Estimated expenditures in December were \$9,900. Cumulative expenses to date are \$1,803,949.

B. Technical Results and Program

Phase II - Bench-Scale Research

1. Experimental Projects (Projects 530, 532, etc.)

Work continues on the preparation of a final report on this phase of the CO₂ Acceptor development. The completion of this report will be delayed, because of the continued absence of Mr. Curran for personal reasons.

2. Supporting Patent and Legal Activities (Project No. 534.00)

The current laboratory reports and memoranda were reviewed for determination of Subject Inventions.

3. Supporting Process and Economic Analysis (Project No. 533.00)

Work consists of the updating of potential commercial economics of the lignite gasification process. The design bases have been reviewed in light of the experimental work completed since 1966 and equipment resized accordingly.

The correlated results from the experimental program suggest that the optimum commercial operating conditions may be different than those set forth in the original Feasibility Study (Fourth Quarter, 1964). Accordingly, consideration is being given to an engineering and economic study to redefine the preferred commercial operating conditions.

Phase III - Pilot Plant Engineering

4. Catalytic Design (Project No. 533.30)

Discussion with vendors regarding the hot piping, valves, etc., in the Rapid City pilot plant is essentially completed. No negative conclusions were drawn from these discussions, although it is apparent that it will be necessary to expend additional funds for consulting, detailed design work, and possibly experimental development to arrive at the precise definition of specific, novel items for the plant. Such effort has been deferred until the decision to proceed with the pilot plant design and construction is made.

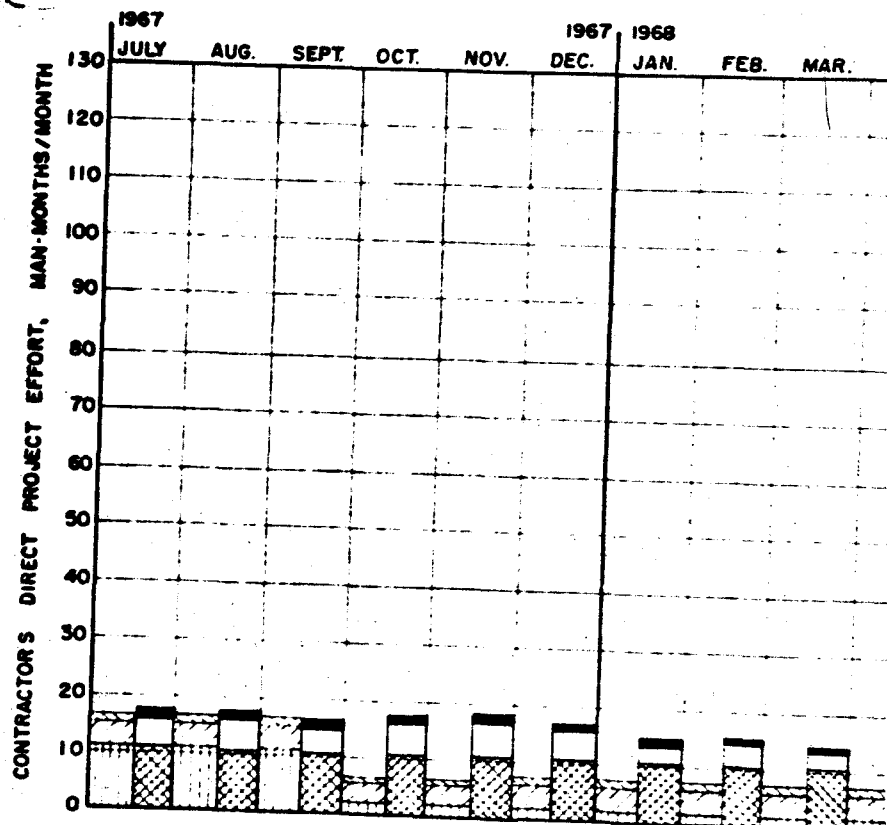
C. Subject Inventions

None.

D. Financial Status

<u>Expense</u>	<u>Actual</u>	<u>Budget</u>
Cumulative through November Expended in Period (estimate)	\$ 1,794,049 9,900	\$ 3,255,000 320,000
Cumulative through December	\$ 1,803,949	\$ 3,575,000

Att. - Monthly Progress Chart
1/13/69



	<u>MONTHLY EXPENDITURES*</u>											
	1967						1968					
	JULY	AUG.	SEPT.	OCT.	NOV.	DEC.	JAN.	FEB.	MAR.	APR.	MAY	JUN.
<u>PREDICTED</u>	103.7 M	72.2 M	92.0 M	94.0 M	94.0 M	92.0 M	40.0 M	20.0 M	70.0 M	70.0 M	70.0 M	70.0 M
<u>ACTUAL</u>	75.5 M	76.0 M	106.6 M	84.9 M	97.1 M	93.4 M	70.0 M	29.9 M	91.9 M	37.5 M	37.5 M	37.5 M
<u>PREDICTED</u>	1010.8 M	1085.0 M	1175.0 M	1269.0 M	1363.0 M	1415.0 M	1455.0 M	1475.0 M	1545.0 M	1615.0 M	1675.0 M	1715.0 M
<u>ACTUAL</u>	981.3 M	1059.3 M	1166.1 M	1251.0 M	1348.1 M	1441.5 M	1511.5 M	1541.4 M	1633.9 M	1671.0 M	1715.0 M	1715.0 M

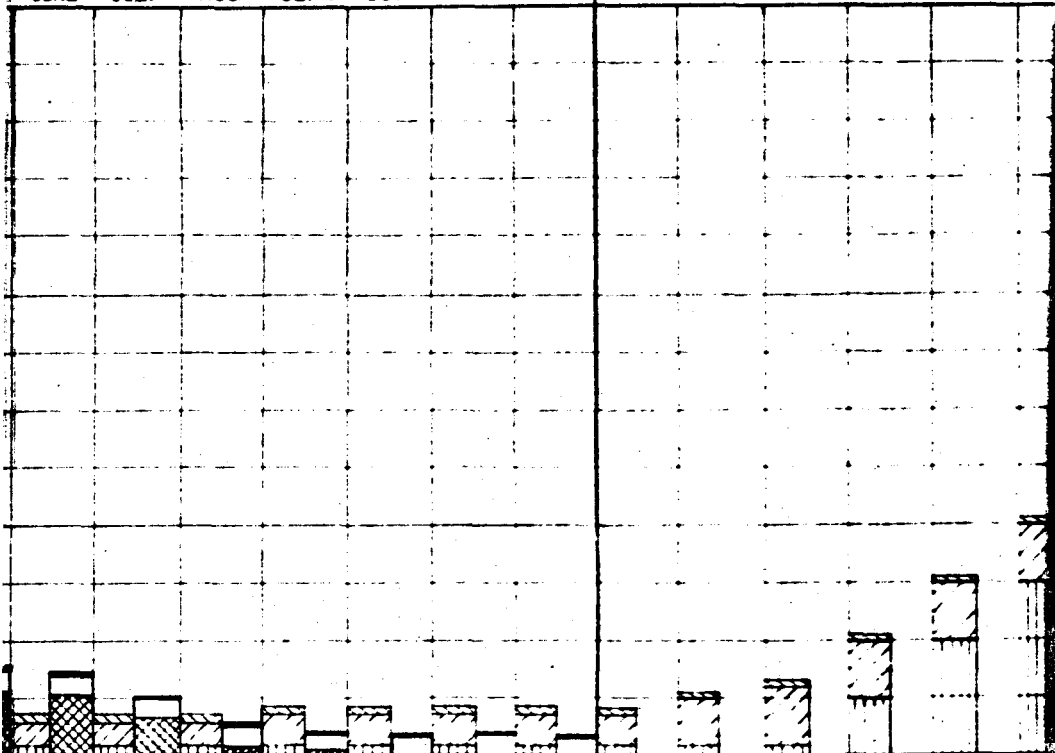
CUMULATIVE EXPENDITURES*

* Expenditures Include All Costs & Fees.

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JUNE JULY AUG. SEPT. OCT. NOV. DEC. JAN. FEB. MAR. APR. MAY JU



220.0 M	220.0 M	220.0 M	220.0 M	320.0 M	320.0 M	320.0 M	420.0 M	290.0 M	290.0 M	240.0 M	160.0 M
31.6 M	19.8 M	13.7 M	9.9 M	4.4 M	8.4 M	9.9 M					
JUNE	JULY	AUG.	SEPT.	OCT.	NOV.	DEC.	JAN.	FEB.	MAR.	APR.	MAY
1955.0 M	2175.0 M	2395.0 M	2615.0 M	2935.0 M	3255.0 M	3575.0 M	3995.0 M	4285.0 M	4575.0 M	4815.0 M	4975.0 M
1732.9 M	1752.7 M	1766.4 M	1776.3 M	1785.7 M	1794.1 M	1804.0 M					

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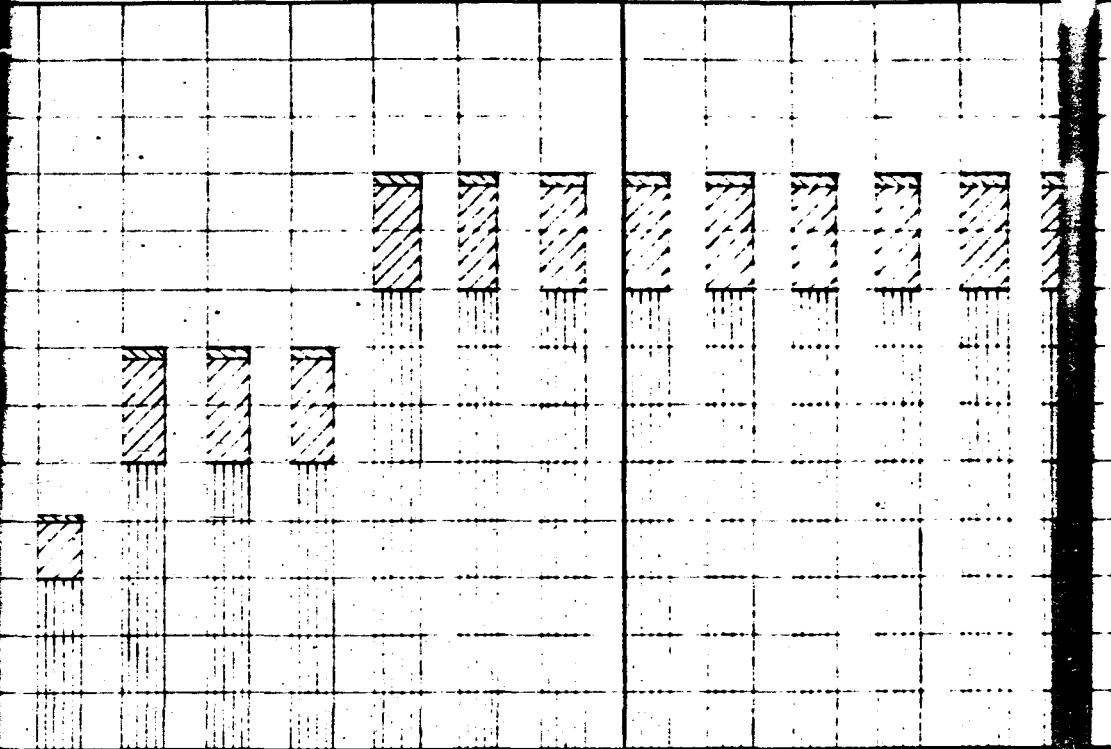
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MONTHLY PROGRESS CHART

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1969 1970

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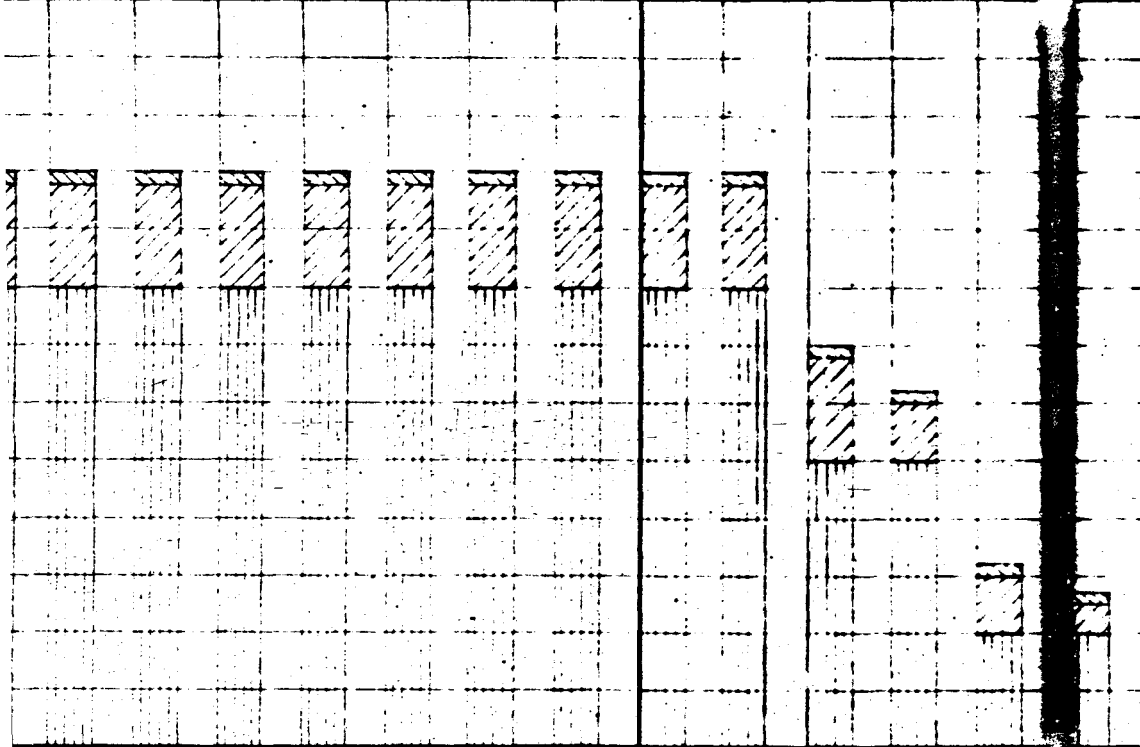
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5455.0 M 5555.0 M 5655.0 M 5755.0 M 5905.0 M 6055.0 M 6205.0 M 6330.0 M 6455.0 M 6580.0 M 6705.0 M 6830.0 M

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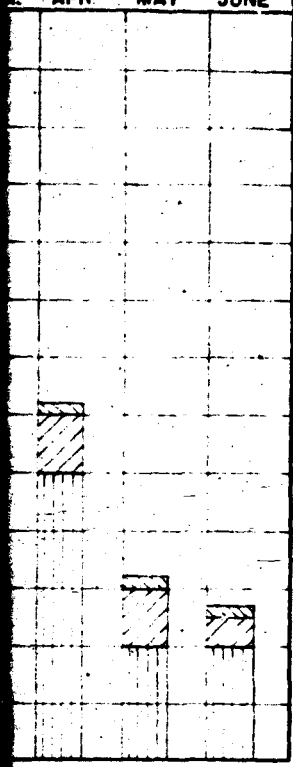


M	125.0 M	125.0 M	125.0 M	125.0 M	125.0 M	125.0 M	125.0 M	125.0 M	125.0 M	25.0 M	125.0 M	125.0 M	170.0 M
M	6955.0 M	7080.0 M	7205.0 M	7330.0 M	7455.0 M	7580.0 M	7705.0 M	7830.0 M	7955.0 M	8080.0 M	8205.0 M	8330.0 M	8400.0 M

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DATE: *December, 1968*

1971
APR MAY JUNE



PREDICTED **ACTUAL**

SUPERVISING

PROFESSIONAL

TECHNICIAN

⊙ Includes Supervisory Exempt Technicians.
⊙⊙ Includes Clerical.

125.0 M	125.0 M	170.0 M
APR	MAY	JUNE
8205.0 M	8330.0 M	8500.0 M

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