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RESEARCH AND DEVELOPMENT DEPARTMENT



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DEPT OF THE INTERIOR

DEVELOPMENT OF KELLOGG COAL GASIFICATION PROCESS

Contract No. 14-01-0001-380

September 30, 1965

Progress Report No. 14

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THE M. W. KELLOGG COMPANY  
A DIVISION OF PULLMAN INCORPORATED



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RESEARCH & DEVELOPMENT DEPARTMENT

REPORT NO. 14

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## I. INTRODUCTION

The objective of this contract with the Office of Coal Research is to develop the Kellogg Gasification Process to the point where it will be able, on a commercial scale, to convert coal into pipeline gas at a cost of 50¢/MSCF or hydrogen at 25¢/MSCF. Five raw materials are to be studied -- an anthracite, a high-volatile bituminous coal, a sub-bituminous coal, a lignite, and a char. Although Kellogg's experimental work will not extend beyond the production of raw synthesis gas, the overall project must make engineering evaluations for four ultimate end products -- pipeline gas, hydrogen, synthesis gas, and transport gas.

Basis for the Kellogg Gasification Process is the reaction between steam and fine coal in a molten salt bath to form synthesis gas, a mixture of hydrogen and carbon monoxide, according to the reaction:



The necessary heat of reaction is supplied by circulating a heated molten salt stream. In addition, the molten salt mixture is chosen to catalyze the gasification reaction so that it may be carried out at a relatively low temperature.

The program is divided into three phases of study extending over a five-year period. Phase I, which is now in progress, involves several concurrent efforts:

1. Bench-scale process research -- to investigate melt properties, reaction kinetics, and the effect of process variables.
2. Chemical engineering studies and development -- to determine the optimum process flowsheet and operating conditions and to coordinate experimental work with overall project objectives.
3. Mechanical development -- to find acceptable materials of construction and develop techniques for handling the molten salt and powdered coal.



Phase I will be concluded by the design of a pilot plant to gasify 24 tons of coal per day, if it is found that a pilot plant program is justified by the bench-scale experimentation and economic studies.

Phase II will be devoted largely to the construction and operation of a pilot plant to convert a variety of raw materials into raw synthesis gas. The effect of operating variables found to be significant in Phase I will be investigated to obtain data for design of a commercial plant.

Phase III will involve the detailed process design of a commercial plant to produce 250 million standard cubic feet a day of product gas, including cost estimates and projected economics for those areas of the country that appear to offer commercial possibilities.



## 11. SUMMARY AND PROJECTIONS

This monthly summary is the fourteenth since the awarding of the contract. It is concerned with the first phase of the contract and outlines the progress that has been made in the three main areas now being studied: process research, chemical engineering studies, and mechanical development.

The study of combustion in the molten salt was continued during the month. A specially-prepared coke was used as fuel because the material actually burned in the combustor of a commercial process will be ungasified coal residue, or coke. As expected, no effect of initial carbon concentration in the melt on the combustion rate constant was discovered. That is, given enough oxygen, if twice as much coke is present in the melt, twice as much will be burned in a stated time interval.

Mere aging of the coke in the melt showed no apparent effect on the rate of combustion. On the other hand, not enough data are available yet to determine whether combustion (or, for that matter, gasification) consumes the more-reactive portions of the coal particle first, leaving a less-reactive residue as reported by other researchers.

Since gasification rates have been found, in general, to be higher than combustion rates, the possibility of improving the latter by adding steam to the combustion air was suggested. An experiment showed that combustion rate is in fact increased about 40% by steam addition. This device could be used in a commercial process to decrease combustor volume appreciably at the cost of a fairly small quantity of steam. The economics must still be investigated.

The effect of coal ash in the melt on combustion rate was explored further. The rate is found to increase with increasing ash concentration, at least doubling as the ash rises from 0 to 10%. Further, the salt volatility is reduced by about 70% when ash concentration is increased from 0 to 2.5%. Both of these factors are, of course, favorable to commercial feasibility.



The combustion rates set as objectives in earlier process evaluation work (Progress Report No. 8, page 6) have been exceeded, even without the advantage expected to be gained by high-pressure operation. It was pointed out in that evaluation that combustion must produce  $CO_2$ , not  $CO$ , for economic reasons, even though excess carbon is present in the melt. This has been achieved in all experiments to date, except when not enough air was supplied to keep up with the rapid combustion. It is further necessary, though, that combustion to  $CO_2$  occur in the melt, where the heat is needed, not in the gas space above the melt. Whether this has been achieved is still uncertain, and a long-probe sampler is being constructed to extract a portion of the gas just above the melt surface for rapid cooling and analysis to try to answer this question.

Further combustion experiments will be made to investigate the effect of melt temperature, coal rank, and preheating of combustion air.

Process design calculations based on recent experimental data have been completed for a plant to produce 250 MM SCFD of pipeline gas from sub-bituminous coal. A process flowsheet and capital cost estimate are now being prepared. The overall plant energy balance resulting from this design has shown that if the Fluor gas purification process previously adjudged most attractive is employed, the plant will have a large excess of low-pressure steam that just can't be used. The net result is the selection of the Shell Sulfinol process, which does have a greater steam requirement but a smaller capital investment. This process is more economical when steam is virtually free, as it is in these circumstances.

Study of the effect of several design factors and operating parameters on economics, and the calculation of a similar case based on bituminous coal will be continued.

Corrosion Test #4, conducted at 1800° F for 500 hours, was completed during the month. An unexpected and discouraging result, at least for the time being, was the complete disappearance of all nine zirconia specimens tested (three samples of each of three grades of zirconia). However, because similar samples of zirconia appeared to be completely unaffected in Test #3, conducted at the same conditions but for 200 hours instead of 500, the poor performance in Test #4 might be attributable to the effects of corrosion products from other materials being co-tested. Silicon nitride, molybdenum, and Incolloy 800 are the suspect materials, and bench-scale experiments are in progress to sort out any interactions. When these effects, if any, have been discerned, another 500-hour run at 1800° F is planned to test zirconia and a new cobalt-base alloy obtained from the Stellite Division of Union Carbide.



High-purity alumina (99.3%  $Al_2O_3$ ) survived Tests #3 and #4 in good condition and appears to be a satisfactory alternative to zirconia if the latter's poor performance in Test #4 cannot be explained. The higher thermal conductivity of alumina may require special design precautions for small experimental equipment to avoid excessive heat losses. On the other hand, the lower cost of alumina (by an order of magnitude) is a definite advantage for a commercial plant.

A bench-scale coal feeder has been completed and tested at 1 atmosphere and 400 psig for 10- to 200-mesh particles. Good operation was obtained over the complete range of feed rate, pressure, and particle size. It is planned to incorporate this system into the high-pressure test unit for which materials are now being procured.

Because of the importance of bubble size to heat transfer and efficiency of gas-melt contacting, a room-temperature simulation test is being set up in a 6" x 6" transparent tower to study the effect of melt viscosity and type of gas distributor on bubble size and pattern. Melt density will be simulated by an aqueous zinc chloride solution; viscosity will be varied by additions of Cab-O-Sil.



### III. PROCESS RESEARCH

#### A. Accomplishments

##### 1. Combustion Studies

Eight combustion runs were completed, seven using coke III (Island Creek bituminous coal coked at 950°C) and one with anthracite coal, in which concentration of carbon and ash in melt, aging of coke in melt, presence of steam in air, and air rate were studied. The conditions and results of these runs are presented in Table I and discussed below.

As in last month's report, the first-order combustion rate constant, consistent with the gasification rate constant previously employed, was used to compare the data. In addition three runs were calculated based on a pseudo second-order rate expression, and although the data plotted better, i.e. a straight line was obtained out to about 75% of the carbon consumed, the constant added nothing more to the data than the one from the first order. More mathematical work is needed to arrive at a better representation of the combustion data.

Concentration of carbon in the bed was further investigated in run J-9835 where 2.5% carbon was present initially. Comparison with two previous runs at 5 and 10% carbon concentration shows no effect of initial carbon concentration on the rate constant, as illustrated by the following tabulation:

<u>Run J-</u>	<u>% Carbon</u>	<u>k - spec. rate const.</u>
9835	2.5	0.41
9823	5.0	0.43
9825	10.0	0.38

With each increase in carbon level, however, the amount of carbon burned is doubled. This is reflected in the analysis of the product gas.





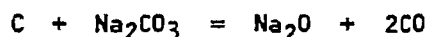
Table I  
Combustion of Coke III and Anthracite Coal in Molten  $\text{Na}_2\text{CO}_3$  /1/

Run No. J-	9835	9836	9837	9838	9839	9840	9841	9842
Date - 1965	8/31	9/1	9/6	9/8	9/13	9/13	9/21	9/23
<b>Feed</b>								
% Fixed Carbon	Coke III 92.2				Anthracite 80.9		Coke III 92.2	
% Vol. Matter	2.0				4.0		2.0	
% Ash	5.8				14.1		5.8	
gas. charge	11.4	24.0	24.0	23.0	24.0	27.0	24.0	24.0
mesh size	12/20							
$\text{Na}_2\text{CO}_3$ - gms.	414							
Bed ht. - inches	4							
ash - gms.	-	-	10.5	1.38	103.5	-	-	46.0
% in melt /2/	-	-	2.47	0.33	20.0	-	-	10.0
<b>Conditions</b>								
Temperature °F.	1840							
% C in melt at start	2.47	5.07	4.95	4.75	4.10	5.00	5.07	4.57
Air Rate-cc/min.	4230							
Run Time - min.	105	115	85	105	85	105	115	35
Devolatilization-min.	10	60	5	5	5	5	5	5
Steam-cc liq. $\text{H}_2\text{O}$ /hr.	-	-	-	-	-	-	10	-
<b>Results</b>								
<b>Product Gas</b>								
% $\text{CO}_2$ - 5 min.	5.0	8.0	20.0	16.5	23.0	14.5	17.5	21.5
- 35 min.	4.5	10.0	18.0	11.0	11.0	10.5	13.5	3.0
- 65 min.	4.5	8.0	2.5	8.0	3.0	8.0	7.0	-
- end	0.6	0.8	0.8	0.8	1.5	1.0	1.0	1.0
% $\text{O}_2$ - 5 min.	16.0	12.0	1.0	7.0	0.8	8.0	7.5	1.5
- 35 min.	16.5	11.0	5.0	10.0	13.0	11.0	7.5	17.5
- 65 min.	16.0	12.5	19.5	13.0	17.5	14.0	-	-
- end	20.0	19.5	19.5	19.0	19.0	19.0	19.0	19.5
% $\text{CO}$ - 5 min.	27	0	1.3	27	4.0	0	0	0.9
Gms. Volatile Salt	15.2	17.7	3.1	14.6	2.6	24.8	24.0	4.8
Minutes to 50% C left	44	44	25	33	17	35	30	13
Rate Const. Combustion /3/	0.41	0.41	0.72	0.55	1.06	0.51	0.60	1.50
Total % C Consumed	92	103	104	103	116	97	104	103
Notes	/4/							

- /1/ Atmospheric pressure, 0.5 ft./sec. superficial air velocity, devolatilization period employed 2 liters/min of  $\text{N}_2$ .  
 /2/ Miscellaneous coal ash from Island Creek 27 coal before coke addition.  
 /3/ Same kinetic basis as used for gasification runs.  
 /4/ During devolatilization, 18.4% of carbon in coke reacted with  $\text{Na}_2\text{CO}_3$  at a reaction rate constant of 0.07.



The increased rate of carbon disappearance with time during combustion led to the suggestion that perhaps aging of the coke in the melt was enhancing its reactivity. Run J-9836 tested this hypothesis in a one-hour aging period and proved that this is not the case. The same rate constant was obtained as for a normal unaged run under the same conditions. Notably, during the aging period further proof of carbon reaction with sodium carbonate was obtained. Nitrogen sweep at 2 liters/min. was used during the 60-minute aging period. After an initial burst of carbon dioxide, carbon monoxide appeared to be the primary product. This can be accounted for by the following equation:



A reaction rate constant of 0.07 for the aging period was obtained and although it is low, about 18% of the carbon was consumed during this period.

Evaluation of the combustion rate of anthracite coal on a first-order plot indicates it to be equivalent or slightly more reactive than coke III, 0.51 versus 0.43 rate constant, respectively. On a second-order plot, the reaction rate constants are -0.18\* for anthracite and -0.22 for the coke, essentially equivalent.

Addition of steam to the combustion air at a concentration of about 5 mole percent gave in run J-9841 an improved rate constant of 0.60 versus the 0.43 constant for the air-only run. Surprisingly, the amount of salt carryover, as found in the transfer line and condenser system, was one of the highest to date. About 24 grams of dry salt was obtained in this run as against 17 grams in an air-only run.

The favorable effect on combustion rate of bituminous coal ash in the melt, as reported in the last summary, was further explored through several runs in which the ash content was varied from 0.33 to 20% in the melt (runs J-9837-9). The results are summarized in the following tabulation. The amount of coke added was held constant.

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\* Units are liters per mole min.



<u>Run No. J-</u>	<u>% Ash</u>	<u>Air - cc/min</u>	<u>% O<sub>2</sub> at 5 min.</u>	<u>k</u>
9823	0	4230	6.5	0.43
9838	0.33	4230	7.0	0.55
9837	2.47	4230	1.0	0.72
9829	10.0	4230	tr	0.86
9839	20.0	4230	0.8	1.06
9842	10.0	8460	1.5	1.50

Addition of the coke produces about 0.33% ash in the melt at the end of a normal run. Thus run 9838, made with only 0.33% ash added before the coke, had a noticeable effect on the rate constant. The remaining runs at 2.47% ash and higher showed not only higher rate constants but less than one percent oxygen in the 5-minute gas sample, which indicates that perhaps insufficient oxygen was present for complete combustion. To verify this the air rate was doubled in run 9842, and the rate constant increased from 0.86 in run 9829 to 1.50 in this run. The low 1.5% oxygen content at 5 minutes in this last run also indicates that there still may not have been enough oxygen present. It was noticed in this run that the heat of combustion in the salt zone in the reactor was sufficient to keep the Kanthal furnace off for 13 minutes and maintain temperature, an extraordinary condition for this reactor-furnace system.

A k of 1.5 corresponds to 22 lbs. of carbon consumed per hour per cubic foot of melt at a concentration of 5% carbon in the melt or 47 lbs. carbon at 10% carbon in the melt. Thus it appears that satisfactory combustion rates can be achieved. As in gasification, the presence of ash lowered salt volatility significantly, only about one-quarter as much salt being volatilized when ash at 2.5% or higher was present.

An attempt was made to try to determine whether carbon monoxide, if made from carbon and oxygen directly in the melt, would be released from the melt as such. In most of the runs only carbon dioxide appears in the exit gas stream. In a few runs practically all the oxygen was consumed initially which allowed 1 to 4% carbon monoxide to appear in the exit gas. A mixture of 2% carbon monoxide in air was passed through



a reactor at 1800°F with and without molten sodium carbonate present. The exit gas showed all the carbon monoxide had been oxidized. Thus the experiment does not answer the question. A long-probe sampler is being constructed at present so that a sample of the combusted gas as it is released from the melt can be instantly cooled and taken for analysis. Thus oxidation of any carbon monoxide to dioxide as it travels away from the melt and up the hot reactor before sampling can be avoided.

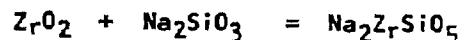
## 2. Corrosion Tests

Pieces of zirconia were tested for corrosion resistance at 1800°F in sodium carbonate melts containing coupons of silicon nitride, Incolloy 800 and molybdenum. A blank of zirconia in sodium carbonate was also run. Inconel crucibles were used and the test was run for 112 hours in an atmosphere of carbon dioxide.

After charging the crucibles to the furnace, it was noticed that the one containing silicon nitride was evolving a gas. Initially this gas burned in the furnace before CO<sub>2</sub> displaced the air. This indicates that it was carbon monoxide. The percent change in weight of the zirconia for the four tests are shown in the following:

<u>Test</u>	<u>ZrO<sub>2</sub></u> <u>% wt. Change</u>	<u>Notes</u>
Na <sub>2</sub> CO <sub>3</sub> + ZrO <sub>2</sub>	+0.27	-
Na <sub>2</sub> CO <sub>3</sub> + ZrO <sub>2</sub> + Si <sub>3</sub> N <sub>4</sub>	-0.72	70% of Si <sub>3</sub> N <sub>4</sub> gone
Na <sub>2</sub> CO <sub>3</sub> + ZrO <sub>2</sub> + Mo	+0.44	Mo dissolved
Na <sub>2</sub> CO <sub>3</sub> + ZrO <sub>2</sub> + Incolloy	+0.42	Incolloy corroded

As shown, only the test in the presence of silicon nitride showed a weight loss for the zirconia (0.06 inches per year), the others apparently picked up some of the salt in the pores. It is postulated that the following reactions may have occurred.



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A sample of zirconia was placed in a nickel crucible containing sodium hydroxide and heated at 1200-1400° F for a one-day period before removing and washing. In this case a 2.4 inches-per-year corrosion rate was obtained. Thus if a lot of sodium hydroxide or oxide becomes available, zirconia will dissolve.

B. Projections

The effect of preheat temperature of the incoming steam on the rate of gasification of bituminous coal will be determined. A preheat furnace will be placed between the steam generator and the reactor inlet.

Additional combustion runs will be made at about 5% ash and two temperatures. Other feedstocks, i.e. sub-bituminous coal and lignite, are available for evaluation by gasification and by combustion.



#### IV. CHEMICAL ENGINEERING STUDIES AND DEVELOPMENT

##### A. Accomplishments

Process design calculations have been completed for a plant to produce 250 MM SCFD of pipeline gas from sub-bituminous coal. Recent experimental data in the area of gasification, combustion, and ash removal were incorporated in the design. The overall energy balance for this design has confirmed a suspicion that arose several months ago: If the most attractive gas purification process is used, one that requires virtually no steam for solvent regeneration (Fluor), the plant will have a large excess of low-level heat (i.e. low-pressure steam) which just can't be used because there is no need for it. The fundamental reason for this is that the oxygen plant which is a very large steam consumer for other coal gasification processes is not required for the Kellogg process. The net result is that the Shell Sulfinol process, which needs a large quantity of steam for solvent regeneration but has a lower capital investment than the Fluor process, is more economical when the steam is virtually free, as it is in the present circumstances. The Sulfinol process has thus been chosen in the evaluation studies.

This state of affairs (excess low-level heat) will probably not exist in a plant producing hydrogen as final product because the enormous quantity of heat liberated by the synthesis of methane will not be available. In that case the Fluor gas purification process should still be the most economical.

An estimate of capital investment is now in progress and the effect on economics of several parameters (operating temperature, gasification and combustion rates, concentration of ash and coal in melt, excess air in combustor) is being investigated.

##### B. Projections

A process flowsheet will be prepared for pipeline gas from sub-bituminous coal. The capital cost estimate will be completed and gas production costs will be calculated, including the effect of the parameters mentioned.

Calculation of a similar case based on bituminous coal will be continued.



## V. MECHANICAL DEVELOPMENT

### A. Accomplishments

#### 1. Environmental Testing of High-Temperature Materials

Test #4, conducted at 1800°F for a duration of 500 hours, has been completed. The results of the test samples are included in Table II, an up-to-date summary of all four completed tests. As noted in the table the metal alloy test samples have been sent to the INCO Corrosion Test Laboratory for corrosion rate evaluation. The disintegration of the three grades (three specimens of each grade) of zirconia was an unpredicted result since this material had proved to be satisfactory in three previous tests. In discussing this result with a representative of the Zirconium Corporation of America, we were advised that the deleterious effect on zirconia may be due to the presence of silicon nitride in the same test. It is known that silicon reacts with zirconia forming a new phase at the interface. Bench-scale tests to determine what effect silicon nitride may have on zirconia are underway and preliminary results are reported in Section III.

As noted in the table, Inconel 600 (and RA600) has shown the best corrosion resistance of all the alloys tested to date. Although the corrosion rate is far in excess of that for a commercially-acceptable material, this alloy can be considered suitable for short-term experimental service.

#### 2. Coal Feeding Studies

Fabrication of the bench-scale coal feeder has been completed. Calibration tests have been conducted for various coal particle sizes ranging from 10 to 200 mesh (74 to 2000 microns) at atmospheric pressure and 400 psig pressure. Results of these tests are shown on Figure 1.

TABLE II  
CORROSION TEST SUMMARY

<u>1. Metal Alloys</u>	<u>Source</u>	<u>Test Number</u>	<u>Results</u>	<u>Remarks</u>
Inconel 600	INCO	1 <sup>/1/</sup>	U	Of all the metals and alloys tested to date, this one has shown up best of all. Corrosion rate in Test #1 was .1403 ipy <sup>/2/</sup> (based on thk. of metal remaining) or 1.589 ipy <sup>/3/</sup> (based on metal unaffected)
		4 <sup>/4/</sup>		See Note 5.
Inconel 702	INCO	1	U	1.612 ipy <sup>/2/</sup> and 1.812 <sup>/3/</sup>
Incoloy 800	INCO	1	U	1.401 ipy <sup>/2/</sup> and 3.510 <sup>/3/</sup>
Incoloy 800 w/ 1.5 Al	INCO	4	U	Severely corroded. See Note 5.
Incoloy 800 w/ 2.4 Al	INCO	4	U	Severely corroded. See Note 5.
Incoloy 800 w/ 3.6 Al	INCO	4	U	Severely corroded. See Note 5.
Incoloy 804	INCO	1	U	.895 ipy <sup>/2/</sup> and 1.850 ipy <sup>/3/</sup>
Type 310	INCO	1	U	1.121 ipy <sup>/2/</sup> and 3.880 ipy <sup>/3/</sup>
50 Cr.-50 Ni <sup>/6/</sup>	INCO	1	U	.984 ipy <sup>/2/</sup> and 1.990 ipy <sup>/3/</sup>
60 Cr.-40 Ni <sup>/6/</sup>	INCO	4		See Note 5.





TABLE I<sub>r</sub>(CONT.)

<u>Metal Alloys</u>	<u>Source</u>	<u>Test</u>	<u>Results</u>	<u>Remarks</u>
Hastelloy B	UC	1	U	Severely corroded.
Hastelloy C	UC	1	U	Severely corroded.
RA 600	Rolled Alloys, Inc.	1	U	Same as Inconel 600.
RA 333	Rolled Alloys, Inc.	1	U	
RA 446	Rolled Alloys, Inc.	3 <sup>/7/</sup>	U	Badly corroded.
Thermalloy 47	Electro-Alloys Div.	3	U	Centrifugally-cast 25-20 Disintegrated during test.
Thermalloy 38	Electro-Alloys Div.	3	U	Centrifugally-cast 28-11. Disintegrated during test.
Thermalloy 28	Electro-Alloys Div.	3	U	Centrifugally-cast 29 Cr-Fe. Disintegrated during test.

<u>2. Pure Metals</u>	<u>Source</u>	<u>Test</u>	<u>Results</u>	<u>Remarks</u>
Platinum (Wire)	Lab	4	U	Wire maintained structural shape but surface seemed severely attacked.
Tungsten	Fansteel	4	U	Not recovered.
Molybdenum	Lab	4	U	Not recovered.

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TABLE I. (CONT.)

3. Ceramics	Source	Test	Result	Remarks
Zircorax O	Carborundum	2/8/	S	Lime stabilized $ZrO_2$ (94.4%).
		3	U	Disintegrated. Attributed to poor structure of samples rather than material.
Mullfrax H	Carborundum	2	U	90% $Al_2O_3$ - 9% $SiO_2$ . Disintegrated.
Mullfrax W	Carborundum	2	U	78% $Al_2O_3$ - 20% $SiO_2$ . Disintegrated.
KT SiC	Carborundum	2	U	~100% SiC. Disintegrated.
Harbide	Harbison-Walker	2	U	89% SiC - 7% $SiO_2$ . Disintegrated.
Varnon BF	"	2	U	42% $Al_2O_3$ - 53% $SiO_2$ . Disintegrated.
Korundal XD	"	2	U	89% $Al_2O_3$ - 10% $SiO_2$ . Disintegrated.
Harklase	"	2	U	97% MgO. Recovered from test but crumbled easily.
Chromex B	"	2	S	34% MgO-26% $Cr_2O_3$ -21% $Al_2O_3$ .
		3	U	Disintegrated.
Ritex CB	Grefco	2	S	> 50% $Cr_2O_3$ - Approx. 32% MgO.
		3	U	One (fully submerged) of three samples recovered but crumbled easily.



TABLE I. (CONT.)

<u>Ceramics</u>	<u>Source</u>	<u>Test</u>	<u>Result</u>	<u>Remarks</u>
Monofrax A	Harbison-Carb.	3	S	99.3% Al <sub>2</sub> O <sub>3</sub> . Relatively unaffected.
		4	S(?)	All three samples recovered. Corrosion effects will be evaluated at later date.
Monofrax K-3	"	3	U	60% Al <sub>2</sub> O <sub>3</sub> - 27% Cr <sub>2</sub> O <sub>3</sub> . Disintegrated.
Silicon Nitride	Carborundum	4	U	Disintegrated.
Zirconia Y-1027	Zircoa	3	S	Appeared unaffected.
		4	(?)	Not recovered. See Note 9.
Zirconia RMF-SP	"	4	(?)	Ram Mix. Not recovered. See Note 9.
Zirconia 60D	"	4	(?)	Castable. Not recovered. See Note 9.
<u>4. Misc. Matls.</u>				
RA 600 w/ .040/.050 thk. Zirconia Spray Coat	Rolled Alloys, Inc. Alloy sprayed by Metco System	3	U	Zirconia coat lost during test. Basic alloy showed minor corrosion effects.

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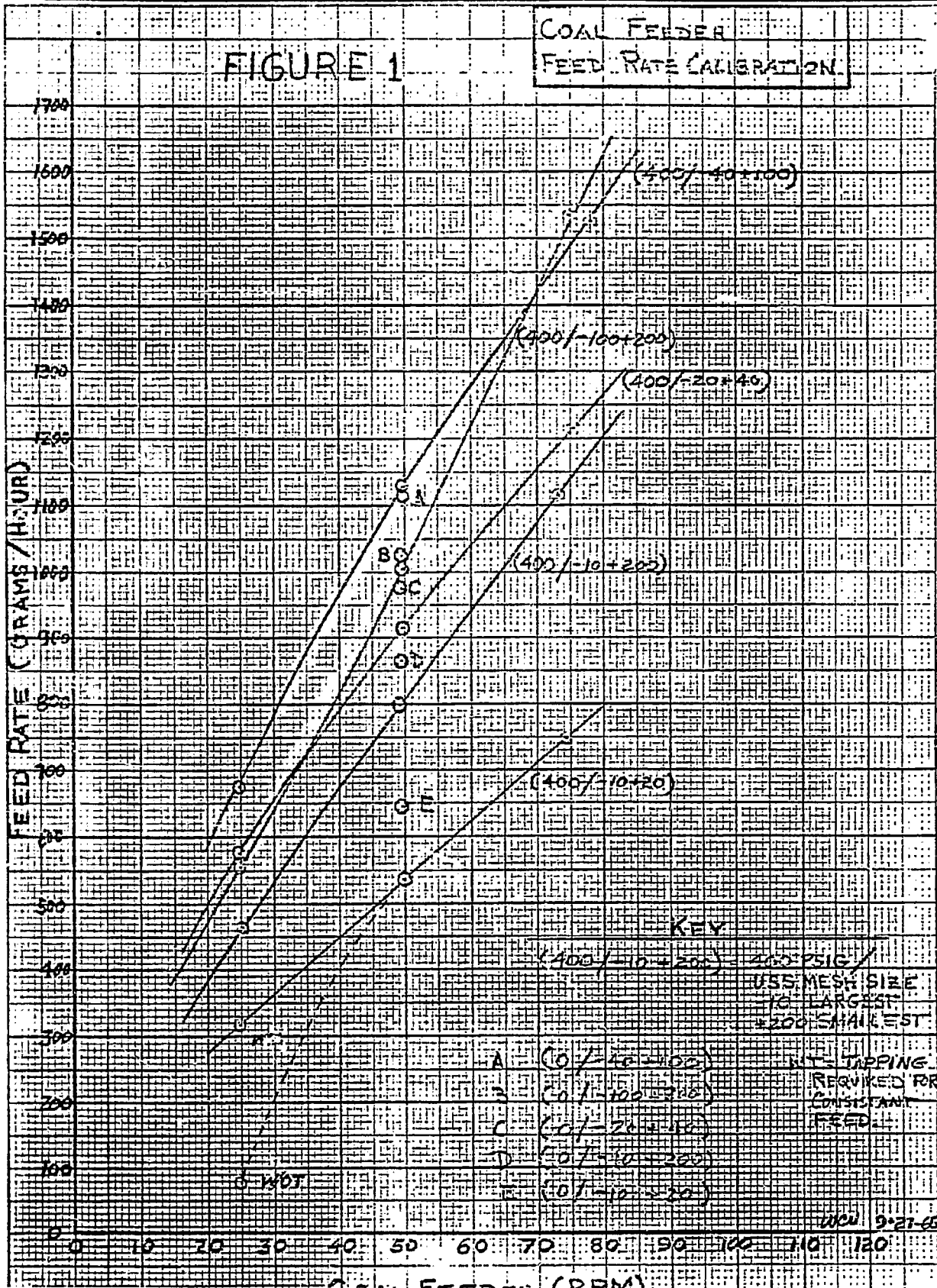
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TABLE 11 (Cont.)

NOTES

1. Test #1: 100 hours @ 1650°F.
2. Corrosion rate based on a micrometer measurement before test and after test.
3. Corrosion rate based on intergranular corrosion at surface determined by metallographic examination.
4. Test #4: 500 hours @ 1800°F.
5. Metal alloy samples of Test #4 have been sent to the INCO Corrosion Laboratory for corrosion rate evaluation.
6. This alloy is available in plate and cast form from Universal Cyclops.
7. Test #3: 200 hours @ 1800°F.
8. Test #2: 200 hours @ 1650°F.
9. It is suspected that the disintegration of the zirconia samples in Test #4 was due to the co-presence of silicon nitride. Tests are planned to confirm this assumption.





The feeder appeared to operate satisfactorily at all speeds and with all particle sizes tested. However, with the largest particle sizes tested (10- to 20-mesh) occasional hanging-up was experienced and some tapping of the feeder was required to assure continuous flow. The loss of flow probably results from the fact that the slots in the feeder rotor are approximately 0.250 inches wide and 0.080 inches deep, and in screening oblong coal particles with a major dimension in excess of 0.20 inches were included in the 10- to 20-mesh feed.

In general, the feed rates at atmospheric pressure were very close to or slightly higher than those obtained at 400 psig pressure. It is felt that, as long as a uniform pressure is maintained upstream and downstream of the feeder, pressure should have little effect on the feed rate.

Several tests were conducted to determine some of the flow properties of coal beds of various particle sizes. The bulk density, angle of repose in a packed and unpacked condition, and minimum tube size required for flow in a packed and unpacked condition were determined. These data are presented in Table III.

In screening and handling the coal samples it was noted that moisture in the air affected the flow properties of the coal samples. The finer particle size cuts seemed more subject to packing with high humidity.

### 3. Effect of Physical Properties on Bubble Size

In order to study the effect of viscosity, density, and type of sparger on bubble size in the melt, a simulation test has been set up. This test setup consists of a 6-inch x 6-inch square transparent tower to allow visual observation, with associated equipment for introducing air through various spargers at the bottom of the tower. A zinc chloride-H<sub>2</sub>O mix, density = 1.8, has been prepared as the test medium and can be treated with Cab-O-Sil to give the required viscosities. Provision has also been made to photograph the bubble patterns if so desired.



Table III

Flow Properties of  
 Bituminous Coal  
 (F-5920)

Sample	Bulk Density #/Cu. Ft. As Received	Angle of Repose, °		Min. Tube Size* Req'd for Flow (Nearest 1/8")	
		As Received	Packed	As Received	Packed
As Received	46.8	55	70	1/2"	1/2"
USS Mesh Cut					
0 + 8	39.6	50	60	3/4"-	3/4"-
-8 + 10	40.2	44	50	3/8"+	1/2"+
-10 + 12	42.0	50	55	3/8"-	3/8"+
-12 + 20	40.5	43	51	1/4"-	1/4"-
-20 + 60	38.7	41	42	1/4"-	1/4"-
-60 +100	33.4	37	90	1/4"-	5/8"
-100 Fines	35.8	75	90	1/4"	1"+

\*Smooth ID Lucite Pipe.



## B. Projections

### 1. Environmental Testing of High Temperature Materials

A new cobalt-base alloy developed by the Stellite Division of Union Carbide has been publicised as a good corrosion-resistant material in high-temperature corrosive environments. The alloy, designated Haynes Alloy No. 150, is composed of 28 Cr - 50 Co - 20 Fe. It is planned to test this metal alloy in our apparatus in the near future.

If the suspected reaction of silicon nitride with zirconia is confirmed, it is planned to retest zirconia in an 1800°F environment for 500 hours without silicon nitride present.

### 2. Coal Feeding Studies

The bench-scale feeder will be further tested at pressure (400 psig) and at elevated temperature (400°F). The conditions of actual operation of the high-pressure test unit will be approximated as nearly as possible and full-range calibration tests will be completed.

### 3. Effect of Physical Properties on Bubble Size

Initially, the test facility will be used to study bubbles in a simulated "melt" of 1.8 density and a viscosity of 600 c.p. (Brookfield Spindel #4 and speed of 20 RPM.) This corresponds to a 10%-ash melt. Later, other viscosities and spargers will be investigated.



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## VI. MANPOWER AND COST ESTIMATES

Figure 2 shows the projected manpower breakdown for Phase I for 1965 as well as the actual effort that was made. It can be seen that a 14.2-man effort was made during September.

Figure 3 shows the expenditures during September. For the month \$26,877 was expended, not including fee and G & A. The total expenditures through September were \$243,187. Including fee and G & A the total expenditures were \$282,129. This is 47% of the encumbered funds.

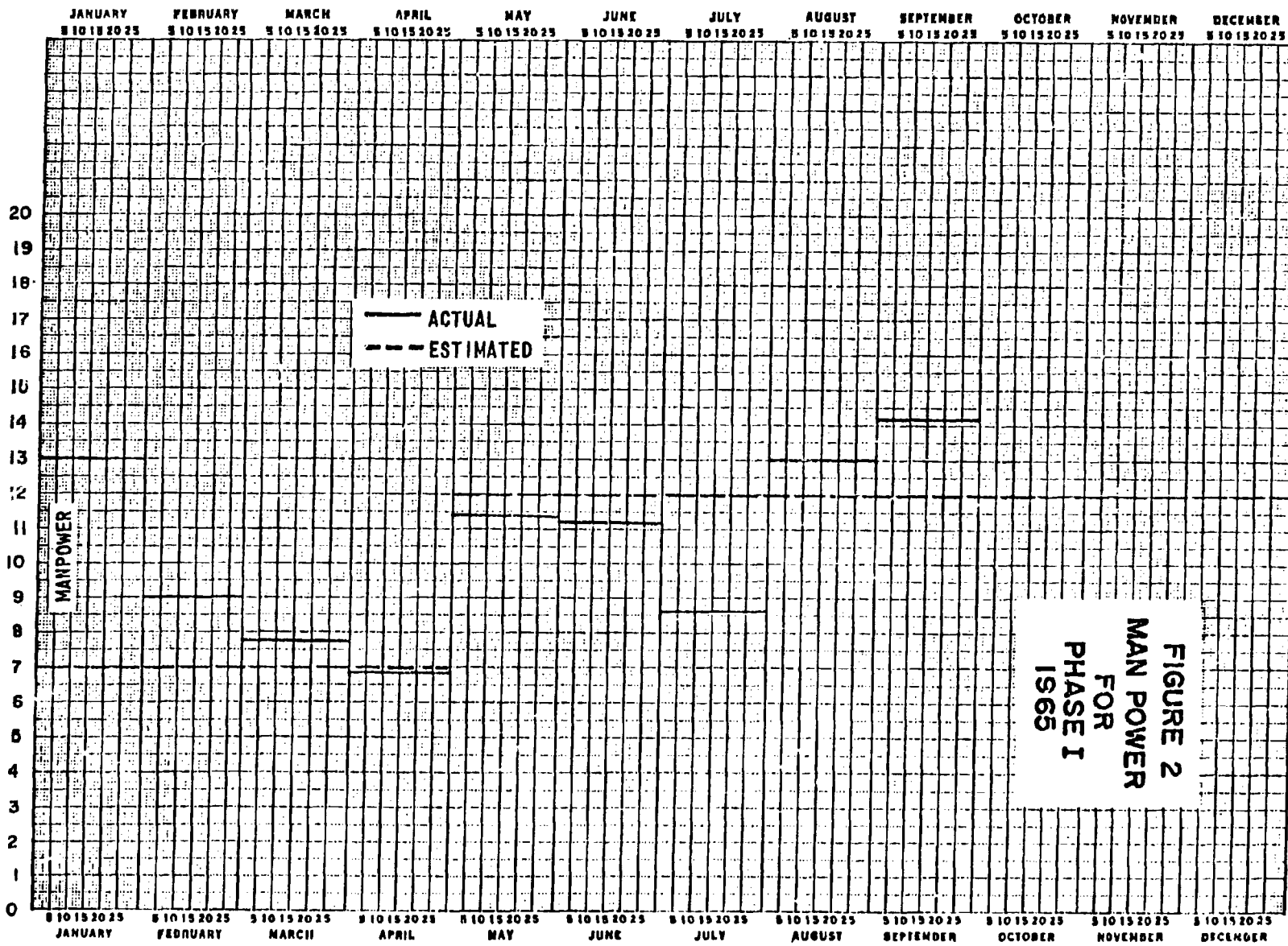


FIGURE 2  
 MAN POWER  
 FOR  
 PHASE I  
 1965

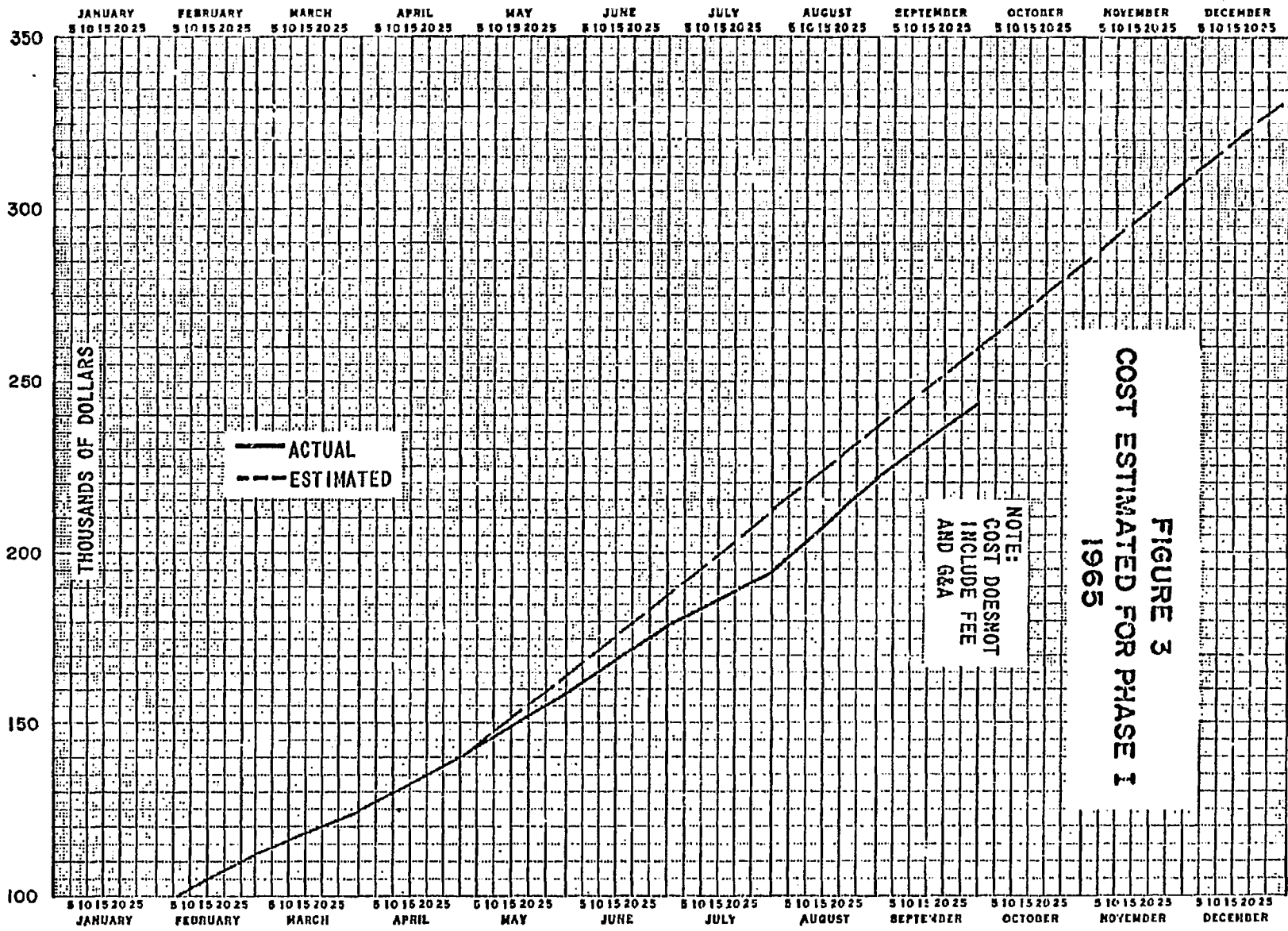


FIGURE 3  
COST ESTIMATED FOR PHASE I  
1965

NOTE:  
COST DOES NOT  
INCLUDE FEE  
AND G&A