MWK-MPR-38

RESEARCH AND DEVELOPMENT DEPARTMENT

KELLOGG

DEVELOPMENT OF KELLOGG COAL GASIFICATION PROCESS

CONTRACT NO. 14-01-0001-380

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PROGRESS REPORT NO. 38

APPROVED:

Project Manager

Manager

RESEARCH & DEVELOPMENT DEPARTMENT

CONTENTS

I. SUMMARY

This progress report is the thirty-eighth since the awarding of the contract. It is concerned with the first phase of the contract and summarizes the progress that has been made in the three principal areas now being studied: Process Research, Chemical Engineering Studies and Mechanical Development.

A run was performed in cooperation with Mechanical Development in order to prepare a metal-uncontaminated melt containing about 8% ash which could be used for sodium recovery studies. Difficulties were encountered in this run with plugging of the small diameter inlet tube used for continuous coal feeding. It appears that this is mainly a function of the small scale of operation used in this experiment and it should not pose a problem in pilot plant or commercial scale.

Another problem which persisted during this run was that of high salt carry-over out of the reactor. A detailed examination of the material carried over indicates that the difficulty is one of physical carry-over or entrainment rather than one of volatility. Other observations made during this run are:

1. Gasification of the volatile matter in coal appears to proceed readily when the coal is bottom-fed to the melt.

2. Sulfur in the coal reacts with and remains in the melt.

3. Gasification rates under conditions of continuous feeding appear to be greater than predicted from batch runs.

4. Bed expansions at atmospheric pressure (with gasification occurring) and in small diameter tubes may be higher than previously predicted.

Cost estimation of a plant capable of producing 250,000,000 SCFD of synthesis gas from bituminous coal has been completed. Since this design was made before the recent data on sulfur disposition were available, the process includes provisions for a separate sulfur removal step. With this added equipment, total capital investment is estimated to be $74,000,000. With this figure and assuming bituminous coal at $4 per ton, total operating expense is calculated to be 20.4¢/MSCF. On the other hand, if this extra sulfur removal equipment were eliminated and if elemental sulfur were recovered via the Claus Process and sold for $30 per ton, a

savings of about 1.0¢/MSCF could be realized. This would bring operating expenses down to 19.4¢/MSCF.

Work has continued on the pilot plant modifications dictated by the new findings concerning sulfur in the melt.

Two tests were conducted to determine the effects of thermal cycling on Insulag--proposed as the backup insulation behind Monofrax A. The first test consisted of subjecting a hollow cylindrical section of Insulag in a stainless steel pipe to three cycles from 220 to 1850°F. The results of this test indicated that the Insulag underwent severe shrinkage, checking, and cracking. In the second test, a similar test section was subjected to 13 cycles from 220 to 1500°F on the inside wall. The outside wall was exposed to the atmosphere and reached a maximum of 275°F. Thus the specimen was subjected to a radial temperature gradient similar to that which the insulation would experience in service. In this test the insulation remained in good condition thus indicating that Insulag appears tentatively to be a satisfactory insulation at temperatures of 1500°F and below. A powdering effect noted at the higher temperatures warrants further study.

Two new computer programs were successfully tested which will serve as design aids for the high-temperature vessels (and lines) in the process.

Upon receipt of the Notice of Termination all new work has been suspended on this project. Efforts will now begin to prepare the final summary report which will be completed by the end of January 1968.

II. PROCESS RESEARCH

A. Accomplishments

1. Continuous Gasification of Anthracite for Melt Preparation

A run was performed in cooperation with the Mechanical Engineering Department using a continuous coal feeder, a two inch ID alumina reactor, and an alumina inlet tube. Steam was introduced at 1500°F at a superficial gas reactor velocity of 0.5 ft./sec., carbon dioxide at 0.1 ft./sec., and 20/60 mesh anthracite was fed at the rate of 53 grams/hr. Temperature was maintained at 1840°F and the reactor was open to the atmosphere. The main objective was to prepare a metal-uncontaminated melt of about 8% ash which could be used for sodium recovery studies.

Difficulties with continuous coal feeding through a small diameter inlet tube, higher than expected gasification rate, and high salt carry-over out of the reactor all contributed to a poorly controlled gasification run. Some of the quantative information obtained is presented in the following.

Anthracite of 20/60 mesh was found to have 18.3% ash instead of 11.7% as originally obtained early in the program. Thus 73 grams of ash went into the reactor along with 400 grams of Na_2CO_3 charged initially and 300 grams charged later in the run. Only 355 grams of melt was recovered from the alumina reactor representing 46% recovery of ash and melt--the rest was carried out of the reactor during the run. This was obvious by the intense yellow color of the flame at the mouth of the reactor and the deposition of white solids on the cool surfaces above the reactor.

The 355 grams of recovered melt was ground to powder and a 30 gram sample treated in the standard manner with 9% sodium bicarbonate solution to yield Residue 1. Weight of Residue 1 corresponded to 51.5 grams of ash (and some Na) in the 355 grams of melt and it analyzed 42% silica, 34% alumina, 7.9% ferric oxide and 13.5% sodium. The assumption of 20% silica loss in the filtrate from Residue 1, as based on prior work, leads to the conclusion that the final melt contained 16% ash instead of the desired 8%. Thus this final melt contained 7.6% silica and 6.1% alumina plus ferric oxide.

From the amount of ash fed to the reactor and the original analysis of anthracite ash, the percent recovery in the melt of the ash and oxides of aluminum, silicon, and iron as well as the sodium carbonate can be calculated.

	% Recovery
Na_2CO_3	43
Ash	78
SiO_2	71
Al_2O_3	75
Fe_2O_3	82

Within analytical accuracy, the results appear to be consistent and do not indicate any preferential loss of ash components. The higher loss of sodium carbonate is as expected since it was being carried out of the unit from the beginning of the run while ash was building up from the continuous addition of anthracite.

A sample of some of the melt which escaped from the reactor and collected on a cool surface above the reactor outlet was analyzed and found to contain 6.36% silica and 1.06% iron and aluminum oxides. The silica appears consistent but the low figure for iron and aluminum is not consistent with the silica. Since the melt at the end was completely ground and carefully sampled, greater reliance on the analytical results from this sample leads to the tentative conclusion that the salt carry-out of the unit was due mainly to entrainment and not to any volatility or sublimation problem with sodium compounds or with silica.

Although the sodium content of Residue 1 was determined and a sodium loss can be calculated (4.0% basis 400g. Na_2CO_3 charged initially or 2.3% basis 700 g Na_2CO_3 total charge), very little significance can be assigned to this because of the erratic control and high carry-over characteristic of the total run.

Experience gained in these attempts to prepare an uncontaminated melt of 8% ash content has led to some important observations.

1. Gasification of the volatile matter in a coal appears to proceed readily by subsurface feeding of the coal to a melt.

2. Sulfur in the coal reacts with and remains in the melt.

3. Melt entrainment is a problem when operating at atmospheric pressure in small diameter vessels.

4. Bed expansion at atmospheric pressure and in small diameter tubes may also be a problem.

5. Gasification rate under continuous feeding appears to be greater than predicted from batch runs.

Previous work has established that gasification under 10 atmospheres pressure led to lower salt carry-over and decreased bed expansion. However, the importance of these problems leads to the recommendation that, if at all possible, a study of bed expansion under gasification and combustion conditions should be done in larger diameter equipment than a 2 inch reactor.

B. Projections

Due to the Notice of Termination received during the month, no further work is planned.

III. CHEMICAL ENGINEERING STUDIES AND DEVELOPMENT

A. Accomplishments

1. Flow Sheet Studies:

The cost estimate of the synthesis gas-from-bituminous plant has been completed. Briefly, the process consists of a coal preparation section where the coal is received and ground to a size (-12 mesh) acceptable for good gasification and combustion rates. The coal then flows to a gasification section where it is reacted with steam in the presence of molten salt to produce synthesis gas. Ash brought in with the coal is continuously removed in an ash removal section by processing a melt slipstream withdrawn from the gasifier. The raw synthesis gas undergoes a single stage of high temperature shift conversion and is then cooled down in preparation for gas purification. The scheme used for CO_2 removal is the Fluor Process which reduces the CO_2 level to about 1 percent. Since the data concerning the ability of the melt to remove sulfur compounds were not available when this flow sheet was calculated, sponge iron and activated carbon units have also been included for H_2S and organic sulfur removal, respectively. However, the effect of eliminating this equipment has been considered and is included below. Effluent gas leaves the plant at about 340 psia at the rate of 250,000,000 SCFD. Gas specifications are given below:

Quantity, SCFD	250,000,000
Temperature, °F	80
Pressure, psig	325
Composition (Mole Percent):	
H_2	61.8
CH_4	9.1
N_2	0.4
CO	27.7
CO_2	1.0
S Compounds	1 ppm max.

The cost of producing 250,000,000 SCFD of synthesis gas from bituminous coal according to the sequence just described is calculated in Tables 1 and 2, assuming 90 percent stream efficiency. Since no precedent has yet been set for the method to be used in calculating synthesis gas costs, the procedure used here is in accordance with the standards used for estimating pipeline gas costs.

Estimated capital investment is summarized in Table 1. Shift catalyst as well as activated carbon have been included in fixed investment because they have long lifetimes. Total capital investment is about $74,000,000.

Estimated operating expenses are shown in Table 2. Bituminous coal is charged at $4 per ton. Total operating expense is calculated to be 20.4¢/MSCF.

As was mentioned in Progress Report No. 37, recent laboratory findings on the ability of the melt to retain sulfur have eliminated a potential pollution problem (SO_2) as well as reducing the sulfur removal costs downstream. For the synthesis gas plant, the estimated reduction in sulfur removal equipment amounts to approximately $2,800,000. This in turn would result in a decrease in manufacturing cost of about 0.6¢/MSCF. In addition, if the sulfur were recovered via the Claus Process and could be sold at $30/ton, an additional savings of 0.4¢/MSCF could be realized. The combination of these reductions would result in a synthesis gas manufacturing cost of about 19.4¢/MSCF.

2. Pilot Plant Design

Work continued on the pilot plant flow sheet modifications dictated by the new findings concerning sulfur disposition as outlined last month. However, a final design had not yet been attained when the Notice of Termination was received.

B. Projections

Work in this area has been stopped upon receipt of the termination notice.

TABLE I

INVESTMENT SUMMARY

SYNTHESIS GAS FROM BITUMINOUS COAL

Basis: 250,000,000 SCFD of Synthesis Gas
90% Stream Efficiency

Section	Title	Bare Cost*
100	Coal Storage and Preparation	$ 3,394,000
200	Gasification	32,919,800
300	High Temperature Shift Conversion	1,808,200
400	Gas Purification	8,572,000
600	Ash Removal	3,776,800
1100	Offsite Facilities	10,288,500
	Total Bare Cost	$60,759,300
	Interest During Construction and Contractor's Overhead and Profit	10,693,700
	TOTAL FIXED INVESTMENT	$71,453,000
Working Capital		
	30 days Coal Inventory	527,700
	30 days Carbonate Inventory	69,800
	30 days Catalyst Inventory	5,300
	Catalyst Charge	43,000
	Accounts Receivable at 11% of Total Operating Expense	1,870,000
	Total Working Capital	2,515,800
	TOTAL CAPITAL INVESTMENT	73,968,800

* Bare Cost includes materials, freight, construction labor, field administration and supervision, insurance during construction, cost of tools, field office expense, and cost of home office engineering and procurement.

TABLE 2

ESTIMATED OPERATING EXPENSE

SYNTHESIS GAS FROM BITUMINOUS COAL

Basis: 250,000,000 SCFD of Synthesis Gas
90% Stream Efficiency

ITEM	$/YEAR	¢/MSCF
Bituminous Coal at $4 per ton	$ 5,804,000	7.0
Sodium Carbonate make-up at 1.55¢ per pound	768,000	0.9
Miscellaneous Chemicals	54,000	0.1
Sponge Iron Make-up	25,000	0.03
Direct Operating Labor at $3.20 per man-hour	608,000	0.7
Maintenance at 3% of bare cost	1,823,000	2.2
Supplies at 15% of Maintenance	273,000	0.3
Supervision at 10% of Operating Labor	61,000	0.1
Payroll Overhead at 10% of Operating Labor and Supervision	67,000	0.1
General Overhead at 50% of Maintenance + Supplies + Operating Labor + Supervision	1,383,000	1.7
Plant Operating Expenses	$ 10,866,000	13.1
Depreciation at 5% of Fixed Investment	3,573,000	4.3
Local Taxes and Insurance at 3% of Fixed Investment	2,144,000	2.6
Sub-Total	$ 16,583,000	20.0
Contingencies	332,000	0.4
TOTAL OPERATING EXPENSE	$ 16,915,000	20.4

IV. MECHANICAL DEVELOPMENT

A. Accomplishments

1. Corrosion Testing of High Temperature Materials

Two temperature effect tests were conducted on Insulag insulating material which had been proposed as a backup insulation behind Monofrax A in melt contact areas.

Test #1 subjected a hollow cylindrical section of Insulag encased in stainless steel pipe (except for the ends) to three temperature cycles of from 220 to 1850°F. Figure 1 shows the test section configuration utilized in Test 1 and Test 2. In Test 1, the entire assembly was heated in an electric furnace. Upon completion of the test, it was found that the Insulag had undergone severe shrinkage, checking and cracking. One longitudinal crack extended completely through the section of Insulag, thus significantly impairing its insulating capacity. The insulation itself had a hard crusty texture.

Test #2 subjected a hollow cylindrical section (similar to the test section in Test 1) to 13 cycles of from 220 to 1500°F on the outside wall. The outside wall in this case was exposed to the atmosphere and reached a maximum temperature of 275°F, thus subjecting the insulation to a radial temperature gradient similar to that which insulation would experience in service. In this case the insulation remained in good condition except for a definite powdering of the insulation extending into the insulation for 1/4" from the hot surface. The outsi appeared sound and, although not quite as hard as in Test 1, it was uncracked except for a closed circumferential crack which may have been produced mechanically on disassembly.

In summary, Insulag appears to be a satisfactory insulation at temperatures of 1500°F and below if the powdering can be tolerated.

2. Design Analysis

Two computer programs were tested and debugged during this period.

a. Stress and Strain Analysis of a Short Cylindrical Shell

b. Stress and Strain Analysis of a Short Conical Shell

Both programs were successfully tested against known solutions given in the literature.

The analysis determines influence coefficients or the stresses and strains in short conical or cylindrical shells due to pressure, end loads (shears and bending moments) and thermal effects namely, longitudinally variable temperature and temperature gradient across the shell wall.

B. <u>Projections</u>

No further work is planned in this area due to termination of the contract.

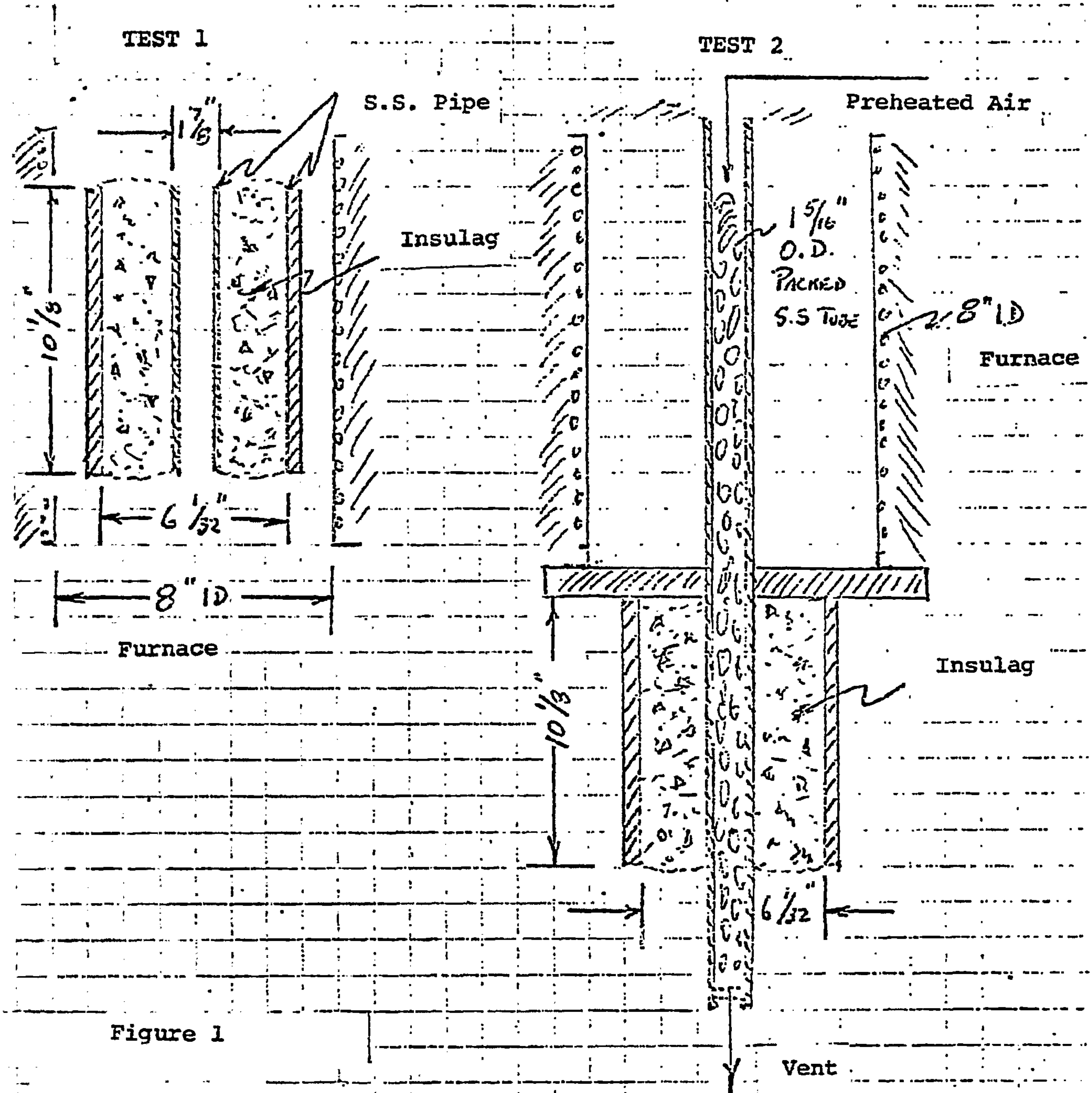

Figure 1

INSULAG TEST

V. MANPOWER AND COST ESTIMATES

Figure 2 shows the projected breakdown for Phase I for 1967 as well as the actual effort that was made. It can be seen that an 11.2 man-effort was made during September.

Figure 3 shows expenditures during September. For the month $20,638. was expended, not including fee and G & A. The total expenditures through September were $756,131. Including fee and G & A the total expenditures were $861,482.

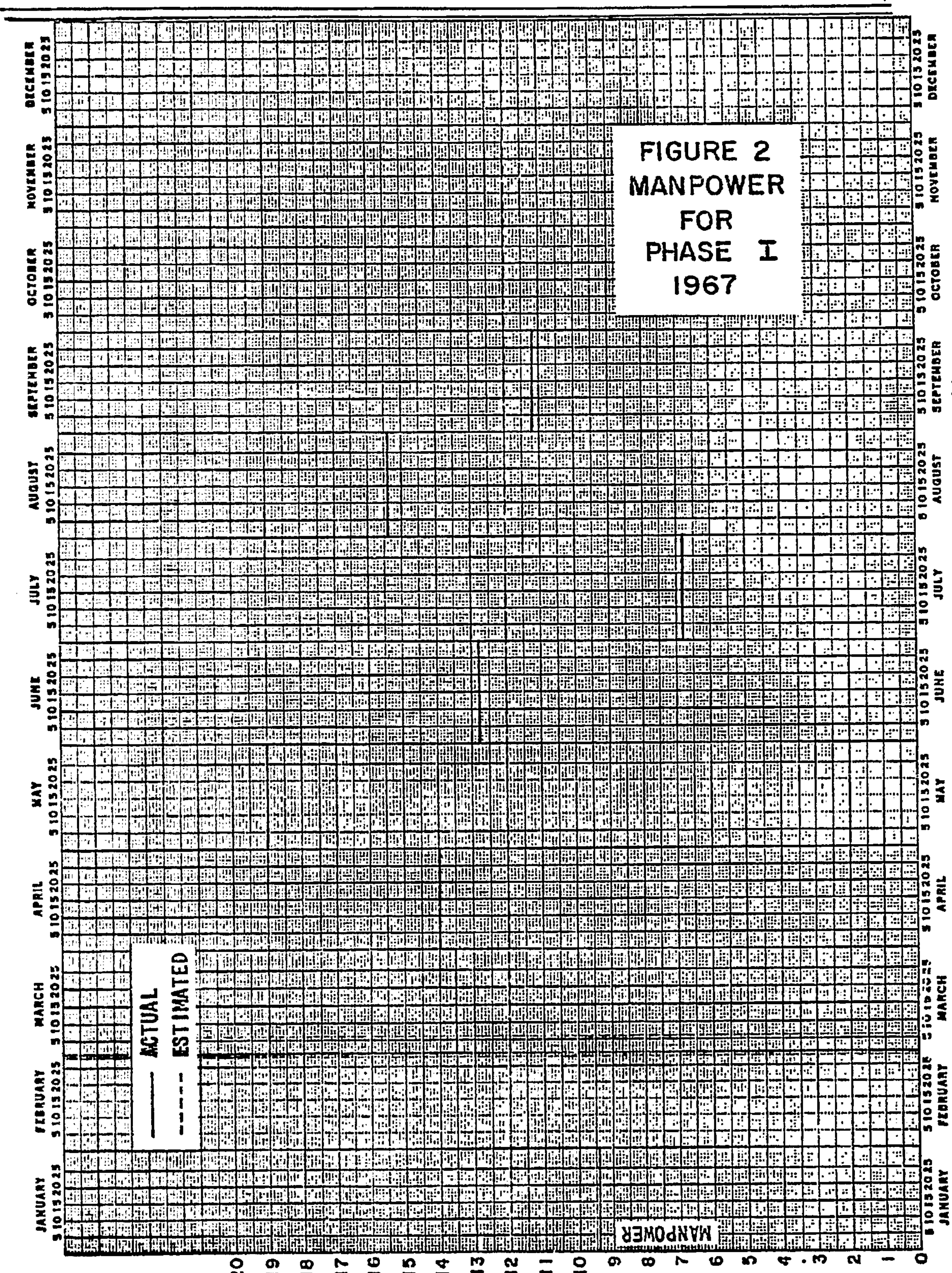

FIGURE 2
MANPOWER
FOR
PHASE I
1967

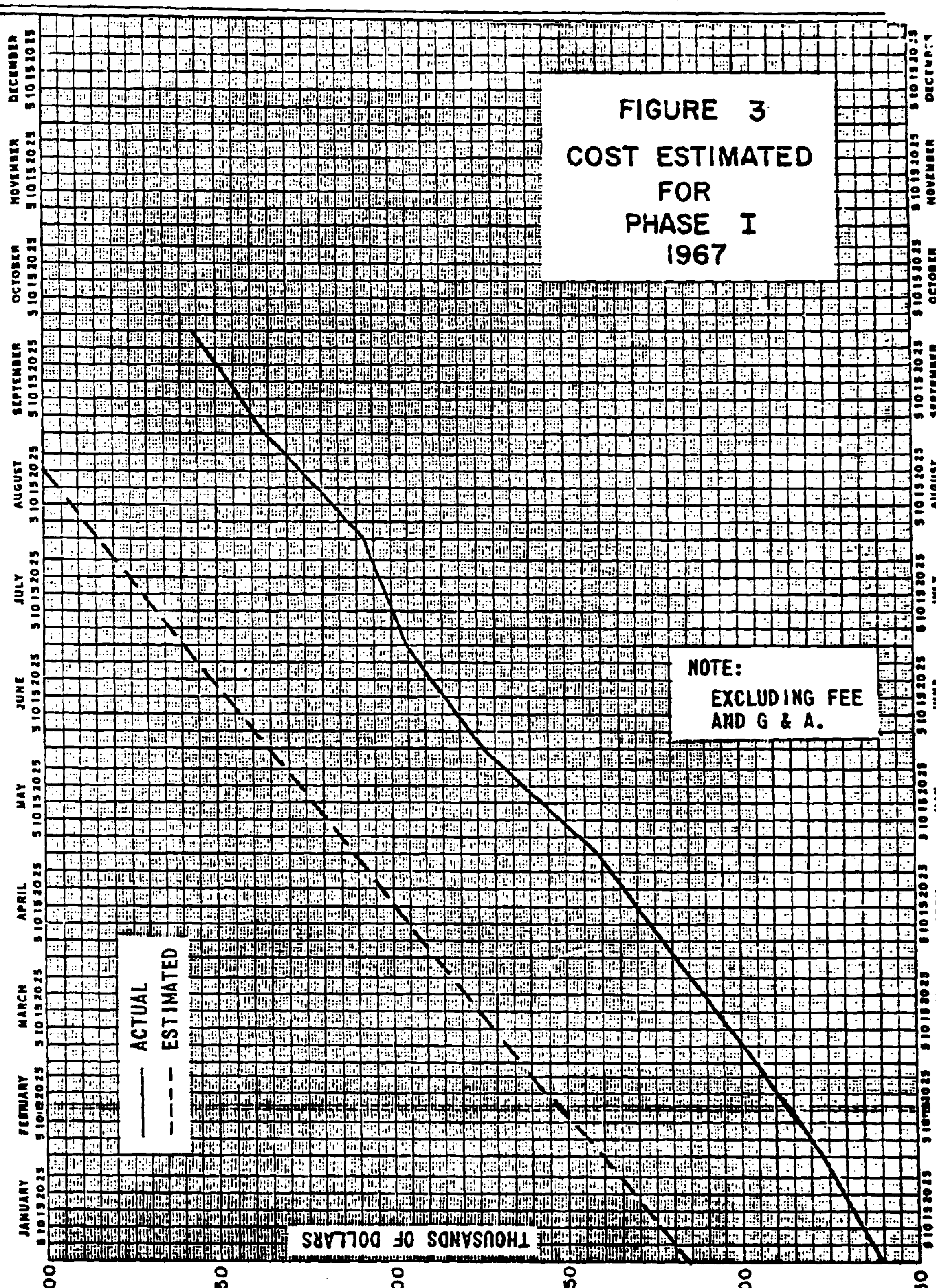
FIGURE 3
COST ESTIMATED
FOR
PHASE I
1967
NOTE:
EXCLUDING FEE
AND G & A.
ACTUAL
ESTIMATED
THOUSANDS OF DOLLARS
800
750
700
650
600
550
JANUARY
FEBRUARY
MARCH
APRIL
MAY
JUNE
JULY
AUGUST
SEPTEMBER
OCTOBER
NOVEMBER
DECEMBER

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