V. APPENDIX B - REMAINING PROBLEMS

A. PRESENT STATUS

Bench-scale experimentation has proceeded far enough to show that the Kellogg Coal Gasification Process may be operated within economic ranges of the major process variables and that the molten salt system can be contained, circulated and controlled in equipment of economically attractive cost. Some areas of the process have been carried as far as is technically and economically feasible on the bench-scale and should now be investigated in the pilot plant. Other areas do remain, however, which merit further bench-scale investigation. These areas are of lesser significance in the overall economics of the process and have, therefore, been deferred while the primary areas were being investigated. Nevertheless, additional information developed in these areas on the bench-scale is expected to minimize the time required for pilot plant operation, and should, therefore, result in substantial savings for the overall program.

This section of the report represents the major areas requiring further bench-scale and process design work, and also presents the general outline of the pilot plant and its program.

B. CONTINUED BENCH-SCALE WORK

1. Chemical Process Research

The major factors governing gasification and combustion have been studied extensively and no major additional work is planned on the bench-scale in this area. On the other hand, the equipment will remain available to provide any extension or confirmation of the picture now available in these areas of the process.

The chemistry of the ash-molten salt system, however, has been studied less extensively than that of gasification and combustion. It is planned to study the effect of variables such as temperature, contact time, gas composition, ash composition, etc., to gain a better detailed understanding of this chemistry. Such information is necessary to provide optimum operating conditions for pilot plant design and operation and particularly to determine what process flexibility may be required, if any, to permit handling the variety of ashes to be encountered in the various fossil fuels to be used in the process. This part of the program will include preliminary studies of the interaction between sodium carbonate and selected ash components (silica, alumina, iron oxide, etc.) to provide a sound basis for planning the work on ash removal and for interpreting the results obtained therein.

The recovery of sodium carbonate values from quenching of the molten salt-ash-coal stream also requires further study to confirm and extend the currently available data and to study factors such as solution temperature, pH, ratio of salt to ash, contact time of extraction and further grinding of ash, Such information would again be expected to lead to a more efficient pilot plant design and test program.

Recent findings that settling of the reactor mixture quickly permits separation of an ash-rich bottoms (perhaps up to 15-20% ash by weight) makes it possible to make substantial decreases in the circulation of melt through the quench system necessary for ash rejection. The possible effects of resulting changes in the melt chemistry of gasification and wet chemistry of ash filtration and bicarbonate precipitation must also be investigated on the bench-scale.

2. Mechanical Development

Corrosion test work will be continued to explore further the effect of variables on the performance of Monofrax A and to evaluate any new refractory materials that may become available. Since Monofrax A cannot provide the insulating characteristics needed in design of the process vessels, high temperature insulating materials such as Insulag must be tested to ensure satisfactory performance under the stress and gaseous environment conditions that will be encountered in operating the process.

Melt circulation to be expected within the reactor vessels and the effect of vessel and nozzle configuration will be studied in a simulated system to aid in design of the pilot plant and in eventual scale-up of pilot plant data.

Entrainment of actual melt as a function of gas velocity and gas properties as well as melt properties will be investigated to provide design data and to define the scope of the entrainment return problem. The reverse problem of disengaging gases from the melt withdrawn from one vessel for circulation to the other will be studied either in a simulated system or in the molten salt-ash-coal system itself.

While molten salt transfer lines can be protected by Monofrax A in a full-scale plant, this is not possible in the small sizes needed in the pilot plant. Accordingly, the pilot plant will use short-life metallic alloy pipes heated electrically, provided with slip joints and housed in a pressure shell. Mechanical testing of the high temperature heaters for this service and of slip joints is planned.

3. Process Design

As bench-scale experimentation proceeds in the process research and mechanical development areas it is expected that flow sheet modifications will become of interest and some work in this area is planned as the need might arise.

C. PILOT PLANT PROGRAM

1. General Objectives

The pilot plant now needed for further development of the process will have the following overall objectives.

a. To demonstrate feasibility of the chemistry involved on a sufficiently larger scale to permit scale-up to commercial sizes.

b. To demonstrate operability and control of the equipment needed for the process.

c. To confirm the economic projections of the process.

The pilot plant and its detailed objectives and program are presented in this section of the report.

2. Process Description of Pilot Plant

A process flow sheet for a pilot plant capable of handling 45 tons per day of bituminous coal to produce raw synthesis gas is described below.

a. Section 100 - Coal Handling Facilities

A process flow sheet for the coal handling facilities is presented as Drawing No. P-2718-D. During eight hours a day, coal is received by rail car or truck at the rate of 5.5 tons per hour with a particle size of less than 2 inches. The raw coal travels by belt feeder and bucket elevator (101-V and 102-V) to a 150 ton capacity raw coal storage silo, 103-FA. From here, coal travels by bucket elevator 103-V to a hammer mill 103-L where its size is reduced to 3/8 inch and then to a roller mill dryer system, 104-L, where it is dryed to less than 2% moisture and ground to minus 12 mesh. The fine coal is blown into a cyclone separator and then conveyed into a 70 ton fine coal storage silo, 104-F from which it is carried by screw conveyor, 105-V and elevator, 106-V, to Section 200.

b. Section 150 - Carbonate Handling Facilities

A flow sheet for the carbonate handling facilities is shown as Drawing No. P-1169-B. In this section, either fresh sodium carbonate in bags or drums or recycle sodium bicarbonate from the ash removal section is carried by one of two bucket elevators, 151-V and 154-V, into storage hoppers, 151-F and 152-F. From storage, the carbonate is transported by elevator 154-V to Section 200.

c. Section 200 - Gasification

A process flow diagram for the gasification section is presented as Drawing No. P-2719-D.

Bituminous coal from storage, ground to approximately -12 mesh in Section 100, is fed to a set of lock hoppers, 201-F, a & b, whose purpose is to receive the coal at atmospheric pressure and to deliver it to the process at operating pressure of about 415 psia. Each of the lock hoppers operates on a 30-minute cycle comprising the following steps:

1. Filling with coal at atmospheric pressure

2. Pressurizing to about 450 psia with synthesis gas or dry inert gas

3. Discharging the coal into the steam line

4. De-pressurizing

The lock hopper system is designed so that while one hopper is being filled and pressurized, the other is discharging coal into the process stream. This provides a continuous flow of coal to the gasifier.

Coal from 201-F, a & b, flows by rotary feeders at the rate of 3,780 pounds per hour into a steam line where it is mixed with 440 psia steam superheated to 1000°F. The amount of steam used as a carrier gas is such that the coal will not be heated above about 650°F, thus preventing the coal from becoming plastic and sticky in the lines. The solids-containing steam stream then flows to the gasifier 201-D together with the remainder of the 1000°F steam. The total steam fed to the gasifier is 3,340 pounds per hour.

In the gasifier, the steam and coal are heated to 1830°F by intimate contact with melt (a mixture of molten sodium carbonate, coal ash, and coal) and react according to the following reactions to produce synthesis gas.

$$C + H_2O + \text{heat} \rightarrow H_2 + CO \qquad (1)$$

$$C + 2H_2O \rightarrow CO_2 + 2H_2 \qquad (2)$$

$$CO + H_2O \rightleftarrows CO_2 + H_2 \qquad (3)$$

$$CO + 3H_2 \rightleftarrows CH_4 + H_2O \qquad (4)$$

$$C + 2H_2 \rightleftarrows CH_4 \qquad (5)$$

$$C_mH_n + H_2 \rightarrow CH_4 + C \qquad (6)$$

The raw synthesis gas leaves the melt at about 1830°F and 415 psia at the rate of 395 mols per hour and either flows through a test exchanger, 201-C or is quenched to 600°F with water and flared. Provision has been made to pass the synthesis gas through a guard drum, 210-F, filled with alumina chips prior to the test exchanger. The purpose of this is to aid in removing any particulate matter carried over with the gas.

The primary gasification reaction (Reaction 1) is highly endothermic; the required heat of reaction is supplied as sensible heat of circulated melt from the combustor 202-D. Melt is circulated between the separate vessels by means of gas lift, whereby the density difference between the liquid in the lift leg and quiescent leg causes the liquid to flow. The melt to the gasifier is picked up by a portion of the steam while a portion of the combustion air circulates the melt to the combustor.

The flue gases which supply heat to the combustor are generated by combustion of coal with air. Air for this purpose is compressed at the rate of 15,500 pounds per hour to 440 psia by air compressor 201-J, is preheated to 1500°F in air preheater 201-B, and is split into three portions. One small stream is used to transport sodium bicarbonate recycle from one of a pair of lock hoppers, 202-F, a & B, to the combustor, another portion lifts melt into the combustor, while the major portion is injected directly into the combustor. Combustion occurs in direct contact with the melt, transferring heat to the reacting system efficiently.

The flue gas leaves the combustor at about 2200°F and 415 psia at the rate of 557 moles per hour. From here, the flue gas is quenched to 1500°F with steam and then either flows through a test exchanger, 203-C, a test expander, 203-J, or is quenched to 600°F with water and vented. As was the case for the synthesis gas a guard chamber, 211-F, can be used to remove particulate matter from the gas.

Provision has been made to add superheated steam at the bottom of the combustor in order to strip dissolved (or entrained) flue gas from the circulating melt in the event that such carry-over becomes excessive.

The ash left in the melt by the combustion and gasification of the coal is allowed to build up to a level of 8 weight percent. A slip-stream of the ash-carbon-Na_2CO_3 mixture is continuously withdrawn from 202-D and flows to 201-E, where it is quenched to 400°F with a portion of a recycle solution saturated with $NaHCO_3$ at 100°F and leaves the gasification section.

d. Section 300 - Ash Removal

A process flow sheet for the ash removal section is shown as Drawing No. P-2720-D. The 400°F slurry enters the ash removal section and is quenched to 245°F with the remainder of the recycle $NaHCO_3$ stream. Solid melt particles in the resulting slurry are ground in 301-L to facilitate dissolution of the melt stream.

This stream is then flashed to 31.8 psia in 301-F, where sufficient holding time is provided to dissolve the Na_2CO_3. The bottoms slurry from 301-F is filtered in 304-L to separate the ash and carbon (and some undissolved Na_2CO_3) from the solution. This residue is sent to disposal.

The filtrate from 304-L is pumped up to 30 psia in 302-J and is fed to carbonation tower 301-E. In this tower the Na_2CO_3 is reacted with CO_2 according to Reaction (7).

$$Na_2CO_3 + CO_2 + H_2O \rightleftarrows 2NaHCO_3 \quad (7)$$

Overhead gas from the tower at 200°F is cooled to 95°F in 301-C to condense water and is recycled through 301-J to the tower. Fresh CO_2 is added to the tower at the rate of 25 moles per hour.

The operating temperature at the bottom of 301-E is 100°F. At this temperature the $NaHCO_3$ (along with some ash) is precipitated. Provision has been made to allow more time for crystal growth in stirred crystallizers 304-F, a & b, and in holding drum 302-F if needed. The resulting slurry is filtered in 306-L, the $NaHCO_3$ solution being pumped up to 400 psia and recycled to 201-E. The filter residue ($NaHCO_3$) is conveyed on 301-V to a salt cake dryer 307-L where the moisture content of the $NaHCO_3$ cake is reduced from 15% to 1%. The dried salt is then returned to Section 150 to be recycled through the carbonate lock hoppers 202-F, a & b, to the combustor.

3. Design Rationale

It should be noted here that the preceding description applies to the pilot plant operating at normal conditions with bituminous coal feed. However, since the pilot plant must operate on other feeds than bituminous (anthracite, lignite, subbituminous and char), considerable flexibility, of necessity, has been incorporated in the design.

a. Section 100 - Coal Handling Facilities

The limiting equipment in the coal handling facilities is the Roller Mill-Dryer System, 104-L. When handling bituminous coal, it is only necessary to operate this equipment one shift per day in order to satisfy the gasification requirements. Lignite, with its potentially high moisture content, requires operation of 104-L for two shifts per day in order to have sufficient dry feed to satisfy gasification. Anthracite feed gives best performance (based upon bench-scale work) when ground to a much finer mesh than any of the other feeds. Since throughput in the roller mill is a function of grind size, it has a much lower capacity with anthracite and must be operated for two shifts a day to satisfy gasification requirements on an around-the-clock basis.

b. Section 150 - Carbonate Handling Facilities

The equipment in Section 150 handling fresh sodium carbonate (152-F, 153-F, 151-H, 152-L) is designed to accomodate carbonate requirements on a once-through basis resulting when char feed (containing the highest ash content of all the feeds) is employed. Such a system enables the gasification section to operate without the necessity of the ash removal section—the melt slipstream is continuously withdrawn but no bicarbonate is recycled.

The items handling recycle bicarbonate (151-F, 151-L, 151-V, 152-V) are sized for the normal flows for the anthracite case. The rationale used here is the same as for Section 300 and is discussed later.

c. Section 200 - Gasification

Mechanical design limitations such as physical installation of Monofrax lining and heat loss minimization as a function of surface to volume ratio predetermined the minimum inside diameter of the Gasifier, 201-D and the Combustor, 202-D. This in turn established the normal feed rate of bituminous coal at 45 tons per day to maintain desired gasification and combustion rates. Combustor Air Compressor, 201-J and Air Preheater, 201-B were sized to accomodate the normal bituminous coal feed rate. These limitations, therefore, have dictated the flow rates of the other feed materials which can be handled. These maximum feed rates are 37, 53, 55, and 65 tons per day for anthracite, subbituminous, char and lignite (dry), respectively. Thus, the coal lock hoppers, 201-F, a & b, have been sized to handle the maximum coal feed rate; namely, lignite. On the other hand, the carbonate lock hoppers, 202-F, a & b, and the melt quench tower, 201-E, have been sized for the case wherein the maximum amount of ash is treated; namely, the case for the FMC char.

d. Section 300 - Ash Removal

All of the equipment in Section 300 has been sized to accommodate the flows required when anthracite is used as feed. These sizes are sufficient to handle the normal flows for the bituminous and subbituminous cases as well. When lignite or char is used, however, the solution fed to this section is larger than can be treated, and so a portion of it is rejected from the system following the quench tower, 201-E. Make-up carbonate solution, corresponding to the amount purged, is added from make-up tank 306-F and pump 307-J.

4. Specific Objectives and Pilot Plant Program

The many specific objectives of the pilot plant program together with a brief outline of the various steps in the program are presented in the following section of the report. For this purpose the pilot plant is divided into several sections including: coal and carbonate handling; gasification; and ash removal. In each section the problems are presented in approximately the order of their priority.

The final order in which the various factors listed below will be studied remains to be determined but the pilot plant program will be divided into three consecutive phases. First, there will be a shakedown period during which the operating crew will be trained. This will be followed by the necessary study of the variables which will provide final data for selection of the preferred operating conditions for each coal. Finally, there will be a sustained operating period to demonstrate the stability and economics of the process.

a. Sections 100 and 150 - Coal and Carbonate Handling

1. Objectives

(a) Provide constant supply of coal and carbonate to the gasification and ash removal sections.

(b) Provide a fair degree of flexibility with regard to the coal particle size supplied to the gasifier.

(c) Provide adequate carbonate storage to enable the ash removal section to be studied independently from gasification (once-through basis).

(d) Determine which fuels will require drying before they can be easily handled and transported.

2. Program

(a) Operate roller mill over as wide a range as possible in order to vary the particle size of the various feeds. Test the validity of the effect of particle size on kinetics which has been proposed based on bench-scale data.

(b) Vary transport gas (air and steam) flow rates and temperatures if design conditions result in difficult coal and/or carbonate feeding.

b. Section 200 - Gasification

1. Objectives

(a) Determine acceptability of Monofrax A as the material of construction in contact with the melt at the proposed commercial operating conditions.

(b) Determine the effect of commercial bed depths on gasification and combustion kinetics.
(Design gasification rate ~20#C/hr-CF)
(Design combustion rate ~12#C/hr-CF)

(1) These high bed depths should enable high (~70%) steam conversions to be obtained at 400 psi. Determine how such conversions will affect reaction kinetics.

(c) Determine the extent of sodium, silica and tar entrainment and/or volatility from gasifier and combustor. Determine required means of cleaning up synthesis and flue gases in order that energy can be recovered from them as shown in the conceptual designs.

(d) Determine conditions required to circulate melt at rates sufficient to supply heat to the gasifier in order to obtain at least the gasification rates assumed for the conceptual design. Determine lift gas requirements for such circulation. Determine maximum ΔT between gasifier and combustor (minimum circulation rates) to obtain design kinetics.

(e) Determine the extent to which melt mineralization, (conversion to insoluble sodium compounds), occurs at 400 psi. (Total sodium loss assumed in conceptual design = 2.4% of melt circulated to ash removal.)

(f) Determine the efficiency with which carbon is burned to CO_2 in the combustor with excess carbon present at 400 psi. (Design combustion efficiency ≈ 85% C → CO_2.) Also determine the efficiency at which heat is transferred from the flue gas to the melt. (Design flue gas temperature = 2200°F with melt at 1900°F.)

(g) Determine the extent to which gases (particularly N_2) will be entrained (or dissolved) in circulating melt and thereby transported between combustor and gasifier. Study effectiveness of steam stripping of melt to remove N_2 from combustor effluent melt. (Conceptual design assumes no N_2 carryover. At design circulation rates, a maximum of about 0.02 CF N_2/CF melt can be carried over to maintain specified product gas purity.)

(h) Determine optimum conditions (residence time, temperature, CO_2 pressure) for melt quench tower. (Conceptual design based on minimum holding time and steam disengaging space.)

(i) Determine effects of ash and carbon concentrations in the melt on operability and reaction kinetics. (Design conditions are 8% ash and 4% C in melt.)

(j) Determine effect of reaction temperatures on operability and kinetics. (Design gasification temperature = 1830°F; combustion temperature = 1900°F.)

(k) Determine effect of gas velocities on operability and kinetics. (Design outlet gasifier velocity = 1.2 fps; combustor outlet velocity = 1.75 fps.)

(l) Determine effect of steam/carbon ratio on operability and kinetics (Design steam/carbon ratio ≐ 2 lb/lb).

(m) Determine effect of coal particle size on operability and kinetics.

(n) Determine bed expansions for each set of operating conditions (Assumed bed expansion for conceptual design = 25%.)

(o) Determine whether coal can be adequately bottom fed to gasifier. Establish minimum required steam for transporting coal from lock hoppers to gasifier.

(p) Determine whether carbonate/bicarbonate recycle can be adequately transported to combustor without sticking. Establish minimum amount of transport gas for this duty.

(q) Establish operability and kinetics for each of the fuels to be considered in addition to bituminous (anthracite, subbituminous, lignite, char and waste.)

(r) Determine most desirable means of starting up, shutting down and normally operating to insure a maximum degree of safety.

2. Program

For each gasification run, the following measurements should be taken:

(a) Coal and carbonate feed rates.

(b) Steam and air feed rates, pressures and temperatures (including lift gas and transport gas rates.)

(c) Synthesis and flue gas compositions, pressures, flow rates and exit temperatures from gasifier and combustor.

(d) Melt temperature in both gasifier and combustor.

(e) Concentration and nature of volatilized or entrained material in effluent gases.

(f) Melt bed density - preferably as a function of depth.

(g) Melt height.

(h) When quench is used on effluent gases, measure quench rate and temperature and temperature of quenched gas to determine efficiency of heat transfer.

(i) When studying expander, measure inlet and outlet gas conditions and flow rate in order to ascertain the effect of time on efficiency.

(j) When studying exchangers on effluent gases, measure flow rate, inlet and outlet temperatures, coolant temperatures and rate in order to detect any change in heat transfer rate with time.

The following variables should be investigated during the pilot plant program:

(a) Bed depth—at least two levels.

(b) Excess air in the combustor.

(c) Quench level of synthesis and flue gases before feeding to the test exchangers and expander.

(d) Stripping gas (steam) rate to combustor for tolerable N_2 carryover.

(e) Temperature difference (hence, melt circulation rate) between gasifier and combustor.

(f) Holding time in melt quench vessel.

(g) Ash and carbon concentrations in melt.

(h) Gasifier and combustor temperature.

(i) Superficial velocities in gasifier and combustor.

(j) Steam/carbon ratio.

(k) Coal particle size.

(l) Coal type.

c. Section 300 - Ash Removal

1. Objectives

(a) Determine efficiency with which ash can be separated from the quench solution as a function of wash rate. (Conceptual design assumes 2.4% of the Na_2CO_3 is lost with this filter cake with a wash ratio of 6.6 lb. H_2O/lb solids.)

(b) Determine efficiency of carbonator and/ or stirred crystallizers for producing $NaHCO_3$.

(c) Determine capacity and performance of salt filter.

(d) Determine the extent to which SiO_2 builds up in the recycle $NaHCO_3$ and the recycle quench solution. (Conceptual design assumed 35% of the SiO_2 in the coal was recycled back with $NaHCO_3$.)

(e) Determine effect of residence time and agitation in dissolving tank on sodium loss in ash filter.

(f) Determine effect of residence time and agitation in carbonator and/or crystallizers on $NaHCO_3$ crystal formation and growth. Determine filter requirements for each condition.

(g) Determine grinder requirements (if any) for good Na_2CO_3 solution rates and high sodium recovery.

(h) Determine drying requirements for easy handling of $NaHCO_3$ recycle.

(i) Determine operability for each coal ash.

2. Program

It should be noted that the bulk of the ash removal variables studies could be done without running gasification simultaneously. When conditions for good operability have been determined, the two sections could be operated together.

For each run, the following measurements should be taken:

(a) Quench rates (to quench tower and to grinder).

(b) When running once through, simulated melt rate and composition.

(c) Liquid level, agitator speed and flows in and out of dissolving tank as well as temperature and pressure of tank.

(d) Particle size of solids leaving grinder.

(e) Ash filter cake quantity and composition.

(f) Wash water rate.

(g) Intermediate temperatures and liquid compositions, agitator speed, operating pressure of carbonation tower and agitator speeds and liquid levels in crystallizer tanks. Measure gas rates and compositions in and out of carbonation system along with liquid rates and compositions in and out (including slurry).

(h) Measure rates and compositions of filtrate (including solid $NaHCO_3$) and cake from ash filter.

The following variables should be studied in the ash removal system:

(a) Residence time, agitator speed and temperature in dissolving tank.

(b) Wash water rate in ash filter.

(c) Residence time, agitator speed, temperature and CO_2 pressure in carbonation system.

(d) Composition of melt slipstream fed to ash removal.

(e) Type of coal ash fed to ash removal.

(f) Particle size of material fed to dissolving tank (e.g., with and without grinder).

(g) CO_2 pressure in dissolving tank.

5. Cost and Schedule of Program

The overall costs of the pilot plant needed for development of the process are presented in Table E. These include the combined design, procurement and construction costs of the pilot plant facility, the estimated cost of the continuing bench-scale program and a preliminary estimate of cost for two years of operation of the pilot plant. The schedule envisioned for the development program is presented in Figure A.

A detailed evaluation of the costs of the program has been submitted to the Office of Coal Research and is titled "Development of Kellogg Coal Gasification Process - Pilot Plant Proposal", dated June 1, 1967.

TABLE E

PROJECTED COSTS(1)

KELLOGG MOLTEN SALT PROJECT

1. PILOT PLANT FACILITY (ERECTED COST)(2)

Solids Preparation	Process Units	Offsites	Total
$1,149,857	$5,166,151	$3,340,408	$9,656,416

2. CONTINUING BENCH-SCALE PROGRAM(2)

Process Research	Mechanical Development	Process Development	Total
$177,566	$145,511	$44,679	$367,756

3. COST FOR FIRST TWO YEARS OF PILOT PLANT OPERATION(3)

Utilities and Chemicals	Salaries and Wages	Maintenance and Modification Contingency	Total
$559,000	$2,184,000	$1,000,000	$3,743,000

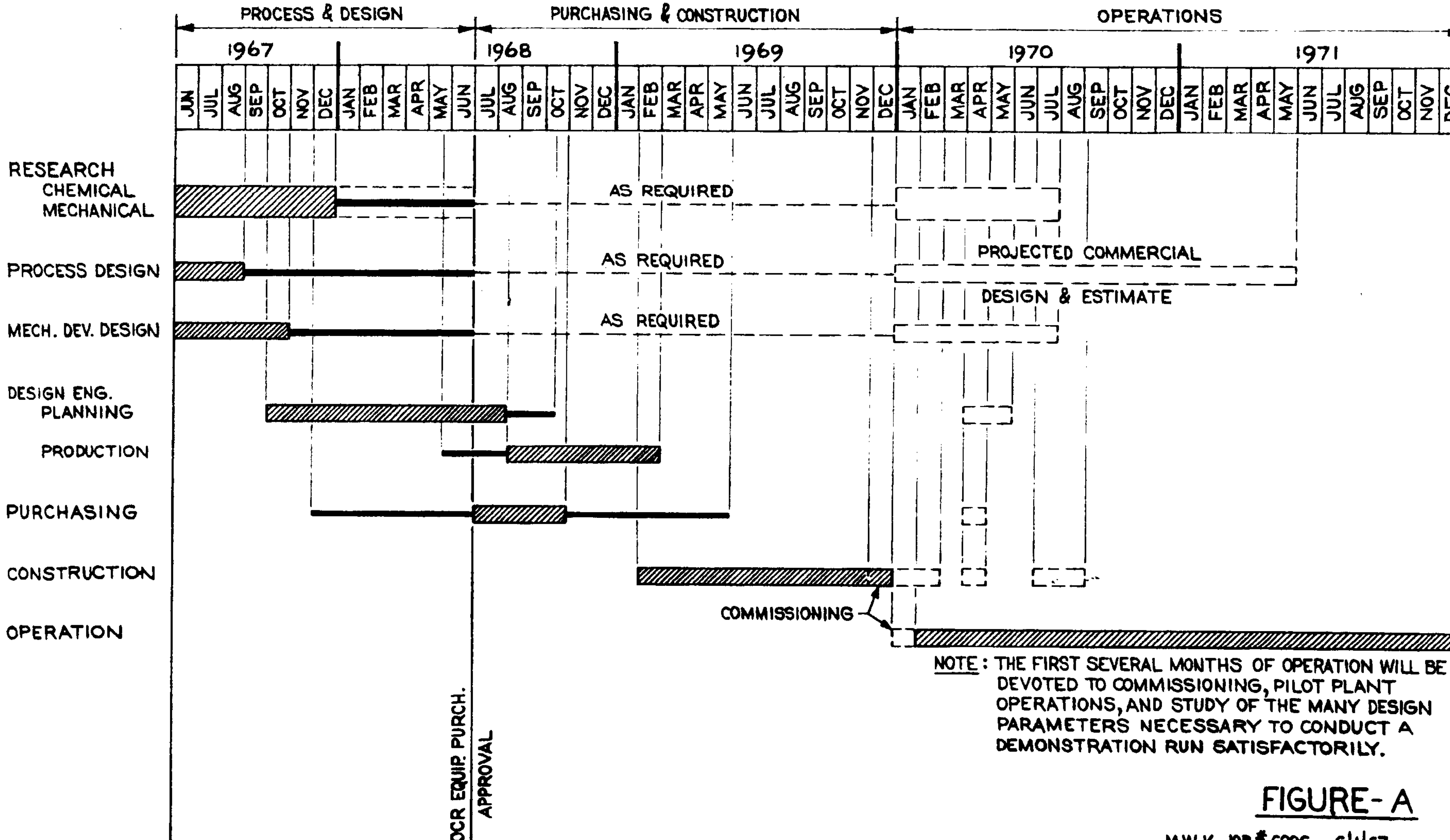
(1) For details and assumptions see "Development of Kellogg Coal Gasification Process - Pilot Plant Proposal", dated June 1, 1967.

(2) Includes Fee.

(3) Preliminary estimate exclusive of fee.

143-A

ANTICIPATED DESIGN-CONSTRUCTION-OPERATIONS SCHEDULE
M.W.K. MOLTEN SALT COAL GASIFICATION PILOT PLANT



NOTE: THE FIRST SEVERAL MONTHS OF OPERATION WILL BE DEVOTED TO COMMISSIONING, PILOT PLANT OPERATIONS, AND STUDY OF THE MANY DESIGN PARAMETERS NECESSARY TO CONDUCT A DEMONSTRATION RUN SATISFACTORILY.

FIGURE-A

M.W.K. JOB # 6026 6/1/67