

### Synthesis and Distillation Units

Good weather and good labor relations, together with excellent supervision of the construction workers, enabled Koppers Co., Inc., to complete construction of the synthesis and distillation units (see figs. 19-23) early in the summer of 1950. Some savings were effected, and it was possible to include the erection, piping, and insulation of the synthesis carbon dioxide scrubber system and a large part of the piping and insulation of the catalyst reduction unit within the estimated cost of the plant.

### Catalyst-Fusion Unit

A catalyst-fusion unit having a capacity of 1 ton per day of sized catalyst has been designed and built by Bureau of Mines personnel. A mixer for the raw materials, crushing and sizing equipment, and a small prefabricated building to house the crushing equipment, as well as a stock of the necessary raw materials, were procured.

Mill scale (magnetic iron oxide) is used as the base for the fused catalyst. It is a cheap material, readily obtainable from steel-rolling mills. After addition of the desired activators, the mixture is electrically fused. After cooling, the pig of fused catalyst is broken and crushed to the desired particle size. The fines resulting from sizing are returned to the fusion step.

Several trial fusions have been made, and active catalyst has been produced at a very reasonable cost.

Figure 24 is a flow diagram of the catalyst-fusion unit.

### Catalyst-Reduction Unit

The continuous catalyst-reduction unit has been erected, tested, and is ready for operation. Initial tests to determine the operability of the star-wheel feeder and the functioning of controls were completed during early autumn. Initial operations are dependent on adequate supply of sized raw catalyst. Arrangements are made for storage of the reduced catalyst under oil to minimize deterioration in activity until needed for operations of the synthesis unit.

### General

The preparation of operating directives for the various units of the Gas-Synthesis Demonstration Plant were completed. These directives present a detailed description of the equipment and a suggested method of operation.

Many special studies were made relating to design and operation of the catalyst fusion, catalyst reduction, synthesis, distillation, product storage, and other units of the Demonstration Plant. The most important of these are described below:

1. Coolant oil for the synthesis is essentially a specialty product. It must contain very little or no sulfur compounds and should boil within the range of 500° to 800° F. Such an oil has been very hard to find, as most petroleum oils contain more than the allowable sulfur content. A survey revealed that a few unusual low-sulfur crude petroleum oils are being produced. One of these, from the East White Lake Field in Vermillion Parish, La., was found to be a suitable material from which a satisfactory coolant oil can be produced by fractional distillation. Two tank cars

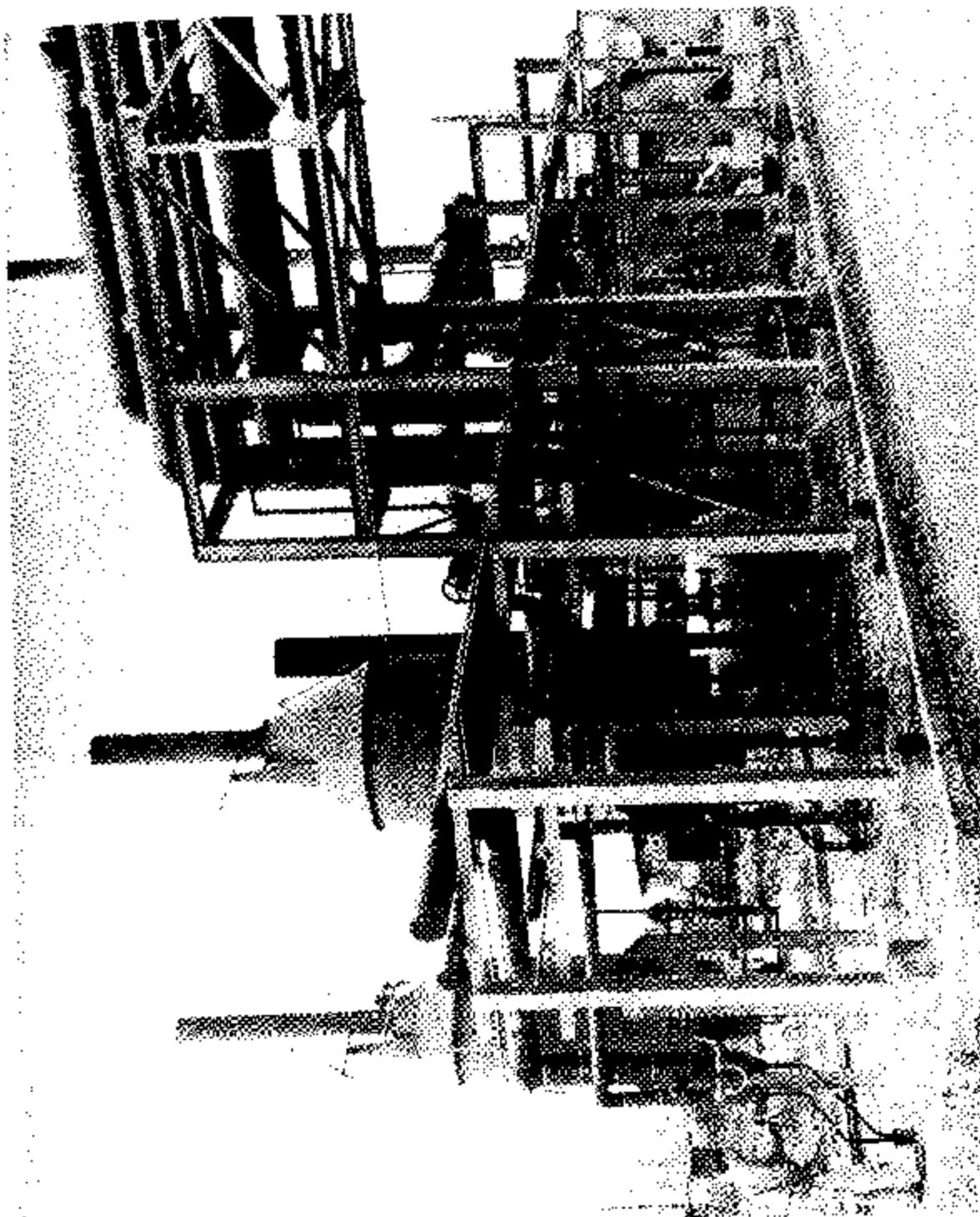


Figure 19. - Gas-fired heaters for preheating feed streams to synthesis and distillation units,  
Gas-Synthesis Demonstration Plant.

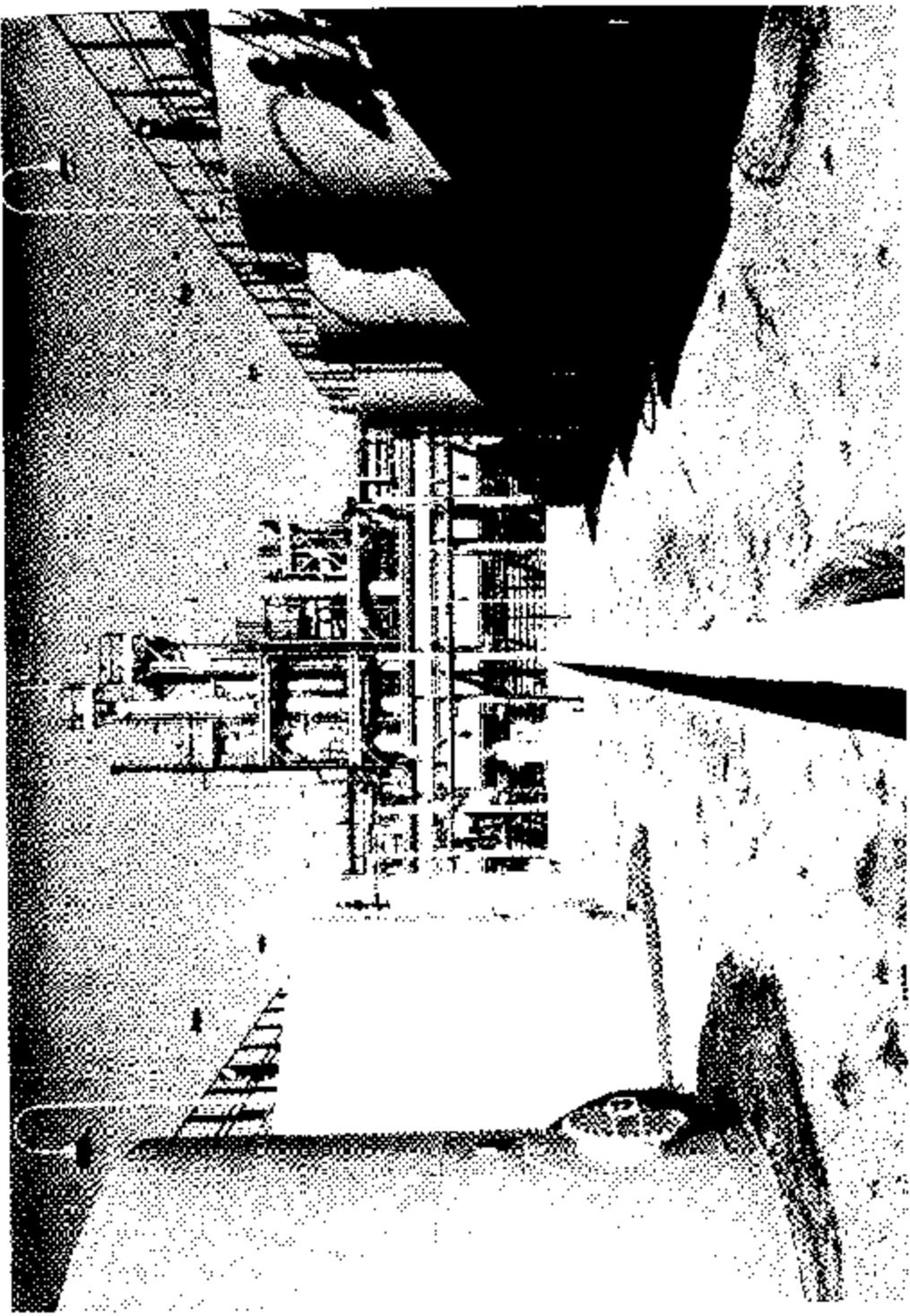


Figure 20. - View (looking north) of distillation unit, with tankage in foreground,  
Gas-Synthesis Demonstration Plant.

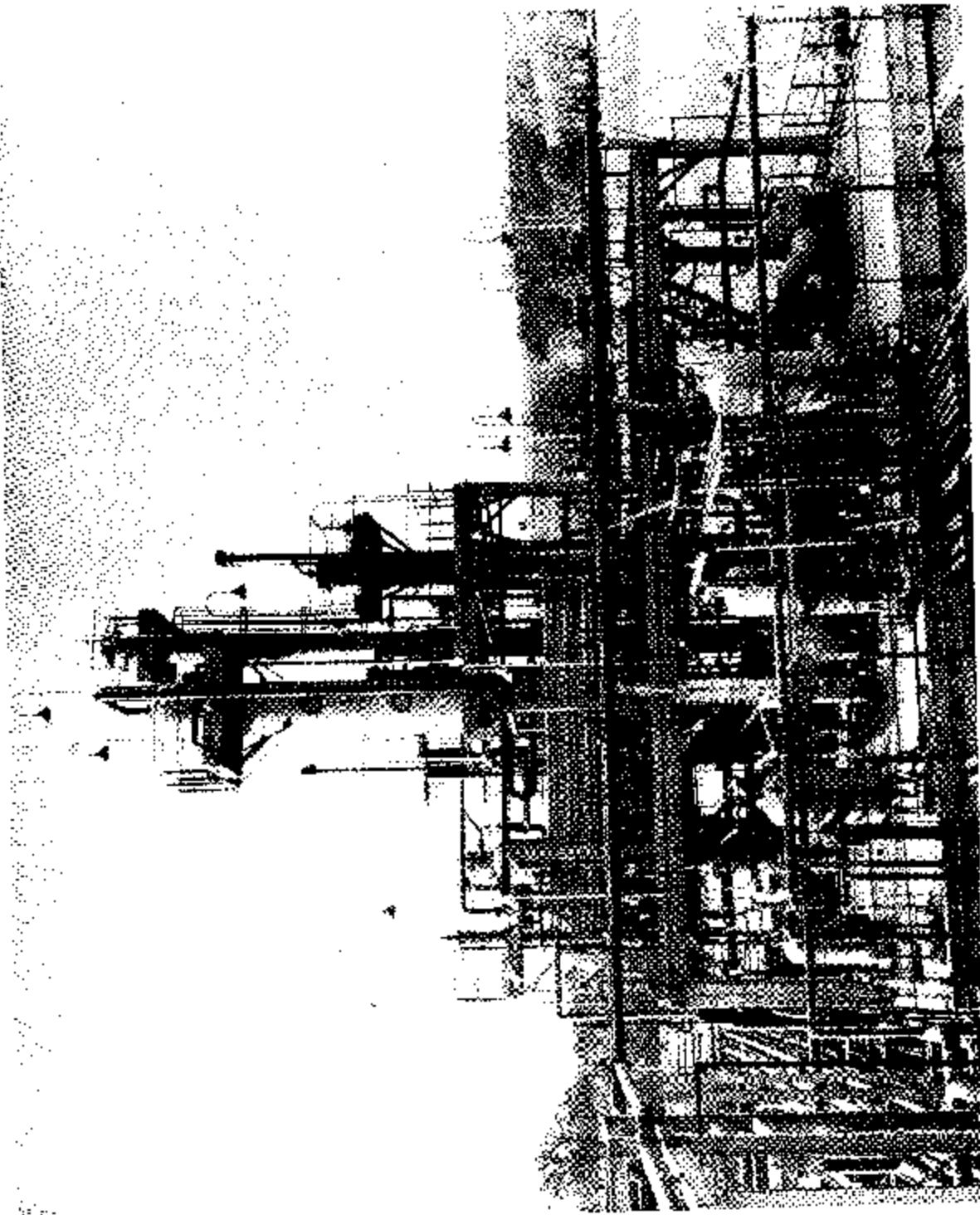


Figure 21. - View (looking south) of distillation columns for refining synthesis crude into finished products, Gas-Synthesis Demonstration Plant.

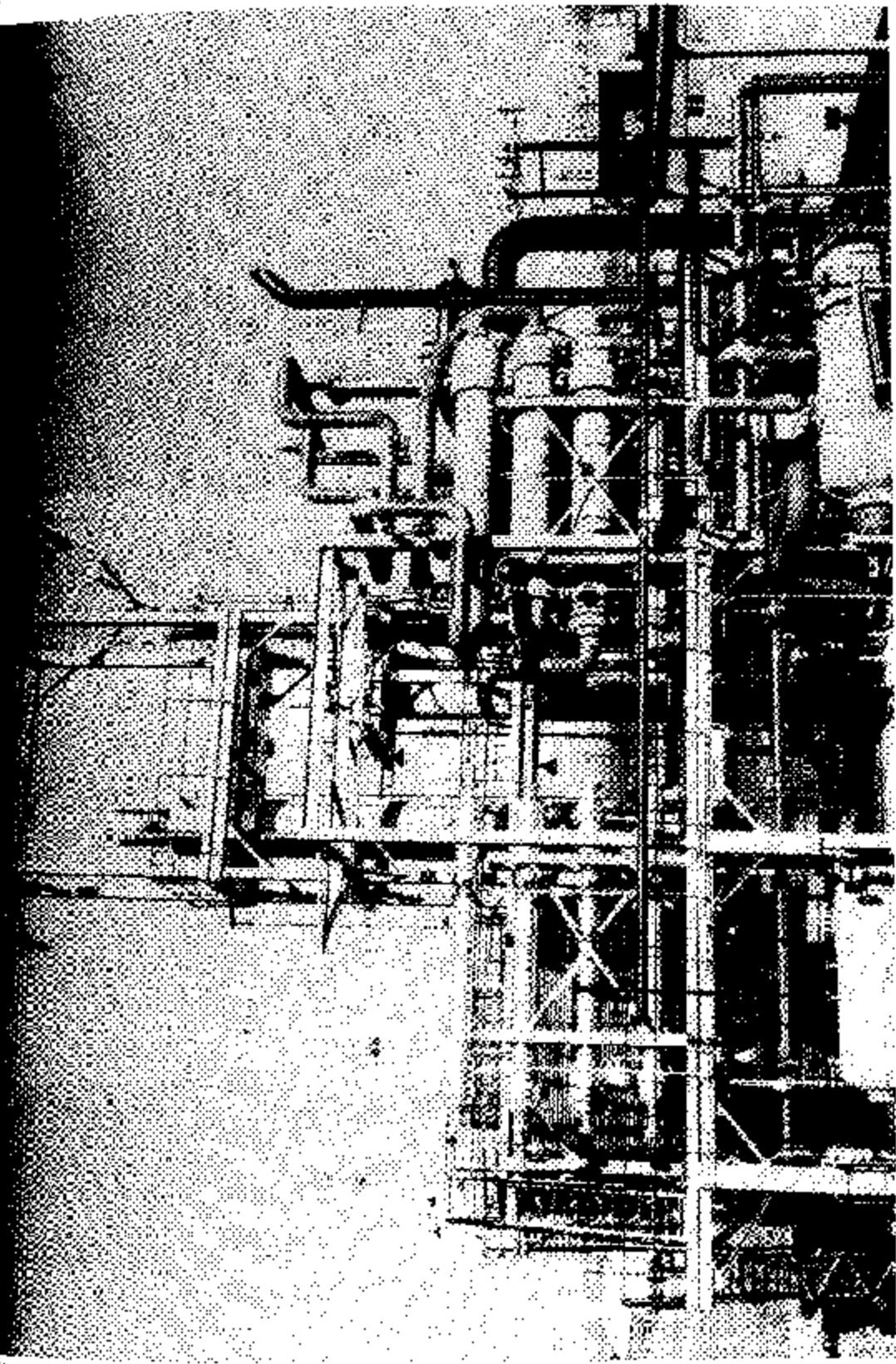


Figure 22. - View (looking north) of auxiliary equipment and piping of synthesis reactor.  
Gas-Synthesis Demonstration Plant.

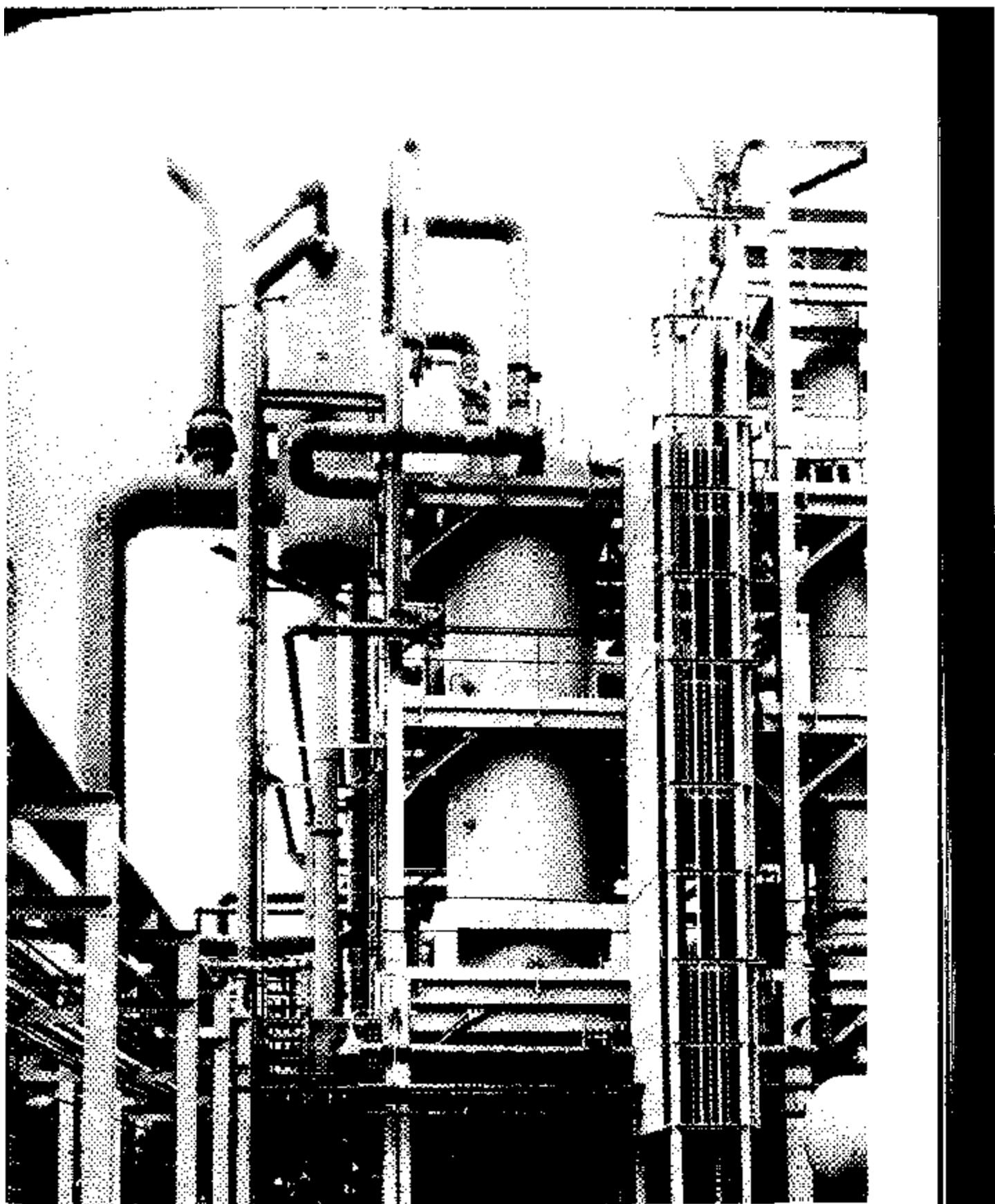


Figure 23. - Gas-synthesis reactor, Gas-Synthesis Demonstration Plant.

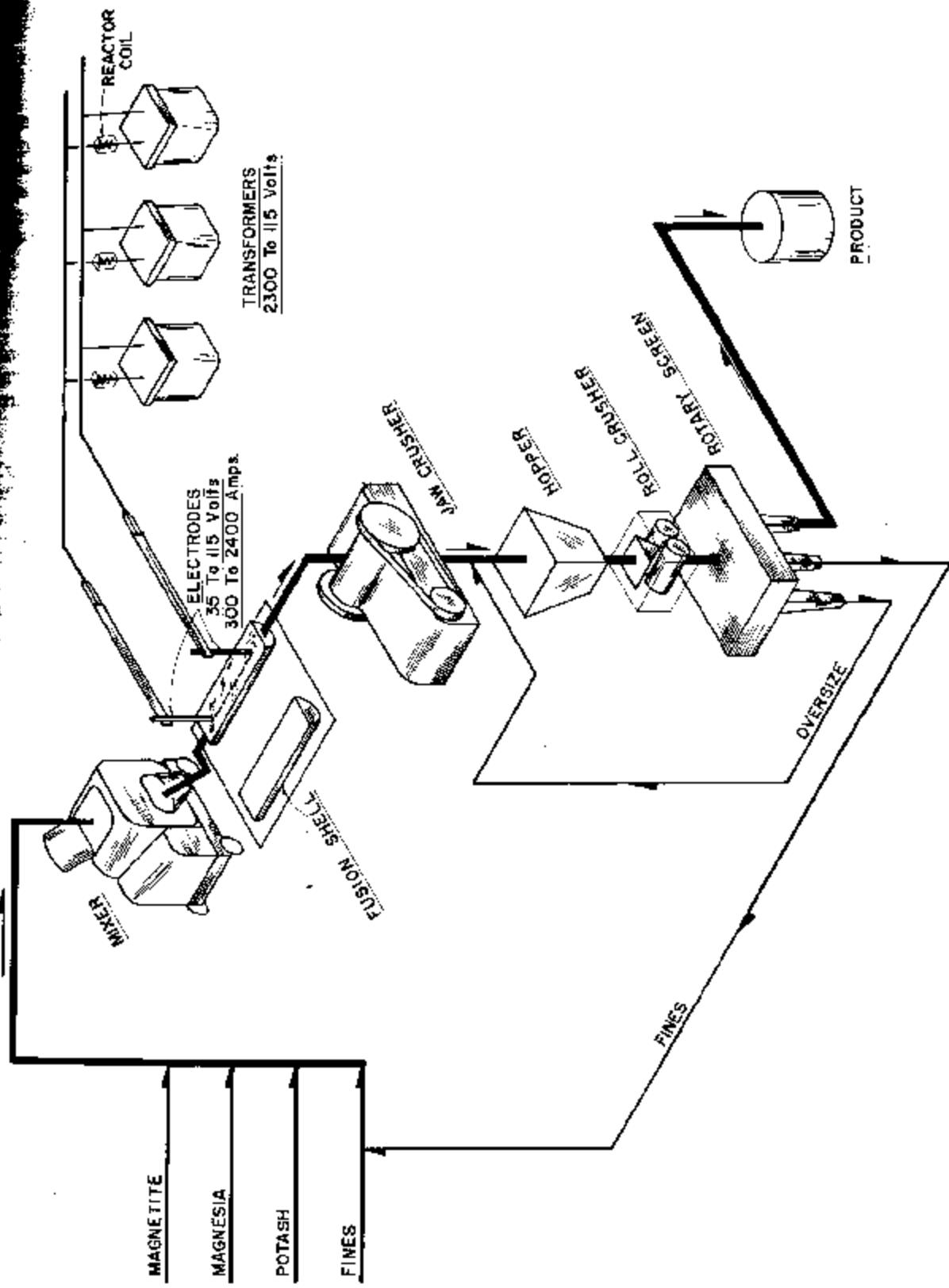


Figure 24. ~ Flow diagram of catalyst fusion unit.

(20,000 gallons) of this crude oil have been obtained and will be distilled to produce coolant oil for the synthesis unit and absorber oil for use in the distillation unit.

2. An economic study was made showing the effect of variations in synthesis operating conditions and process yields on the relative costs of producing a liquid product. The effects of such factors as (a) single- or multi-stage operation, (b) gas recycle, (c) carbon dioxide removal, (d) catalyst life, (e) space velocity, and (f) degree of conversion of the reacting gases have been evaluated for a basic case and fifteen variations thereof. Comparative cost figures developed in this study will be employed as a guide in planning demonstration operations and for further cost studies.

3. Special consideration was given to possible changes in operating variables that might affect the functioning of the interstage carbon dioxide removal unit. Gas flows, variations in carbon dioxide content of the feed gas under different operating conditions, rates of flooding under various pressure and flow conditions, and anticipated carbon dioxide absorption efficiencies were evaluated. These calculations indicate that it will be possible to remove the carbon dioxide contained in the reactor gases at expected flows and at pressures of 300 to 450 p.s.i.g.

4. Process studies have been made to explore the probable limitations of the demonstration plant in terms of flows, temperatures, pressures, and yields. Of these, the variation in the flows of reacting gas mixtures is most important. The flow of synthesis gas of variable H<sub>2</sub>/CO ratio, the influence of variable recycle gas ratios, and the removal of carbon dioxide from the recycle gas change markedly the capacity of the synthesis unit.

#### Engineering and Economic Studies for Commercial-Size Operations

Various engineering and economic studies were made in connection with the design of commercial-scale plants to indicate the direction of research activities as well as to make preliminary determinations of the cost of various synthetic fuel products.

#### Shale-Oil Hydrogenation

In connection with shale-oil hydrogenation, the possibilities of making hydrogen from retort gas and coker tail gas produced in shale-oil refining were reviewed. It was determined that both of these gases will serve as feed to the hydrocarbon-steam cracking operation, whereupon 96 percent hydrogen could be produced by shifting and carbon-dioxide scrubbing of the effluent gases. From 21 million std.c.f. of retort gas, 34.2 million cu. ft. of hydrogen gas can be produced. Similarly, 3.25 million cu. ft. of coker tail gas will produce 8.7 million cu. ft. of hydrogen. Of the two gases, coker tail gas is preferred, owing to its lower total sulfur content and its lower content of inert materials, which make for smaller equipment sizes.

#### Effect of Octane Ratings on Production Costs

A study was made showing the effect of octane ratings on the production costs of coal hydrogenation plants producing various grades of gasoline. Results of this study are shown in graph form in figure 25.

PRODUCTS, BARRELS-PER-DAY	23,000	23,000	21,491	13,578
MOTOR GASOLINE				11,920
AVIATION GASOLINE, 91 O.N.				
AVIATION GASOLINE, 100 O.N.				
JET FUEL, JP-3				25,670
L.P.G.	<u>7,130</u>	<u>7,130</u>	<u>8,639</u>	<u>2,582</u>
TOTAL	<u>30,130</u>	<u>30,130</u>	<u>30,080</u>	<u>4,465</u>
				<u>30,135</u>

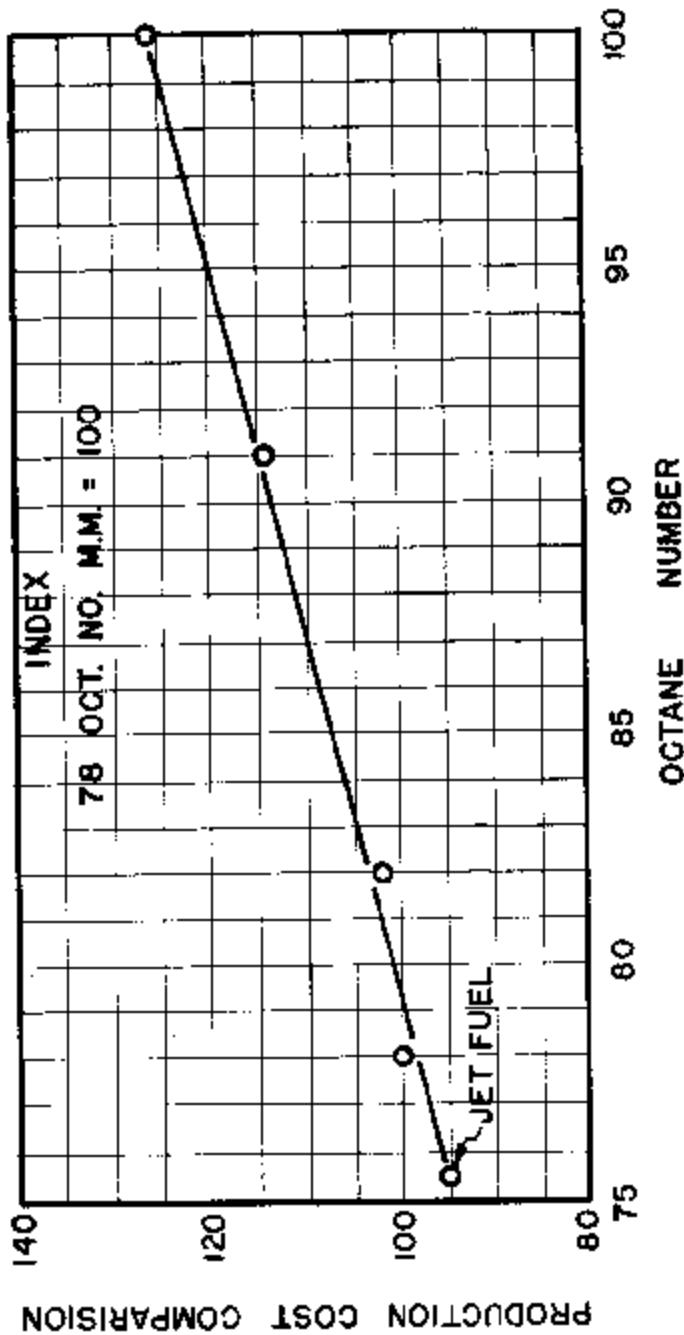


Figure 25. - Effect of octane ratings on production costs.

### Anthracite Silt Gasification

A preliminary engineering and economic study was carried out to determine the cost of manufacturing a 900 B.t.u. per cu. ft. gas, using silt washing from anthracite coal-cleaning operations. The process selected was the gas-synthesis or Fischer-Tropsch process, using two fluidized bed reactors. Two alternates for the gasification of the silt were selected: (a) pressure-dust gasification and (b) Lurgi gasification of pellets made from the silt. In each alternate, the basic process units consist of a coal-preparation plant, gasification and oxygen plants, a dust-removal system, a water-gas shift for adjustment of hydrogen and carbon monoxide ratio, a water-washing step for removal of carbon dioxide and hydrogen sulfide, a final sulfur-removal plant, and the synthesis step wherein hydrogen and carbon monoxide react over a suitable catalyst producing water, which condenses out, leaving the finished city gas.

The cost of production, including no profit but amortizing the total investment cost over a period of 15 years, is 54.5 cents per 1,000 cu. ft. of 900 B.t.u. per cu. ft. gas when using the pressure-dust gasification and 63.5 cents when using the Lurgi gasification.

Capital investment cost for the two alternates is \$57,955,000 and \$59,950,000, respectively, for 65,650,000 std.a.f. per calendar day of 900 B.t.u. city gas.

An engineering and cost comparison is shown in table 7.

TABLE 7. - Comparison of pressure-dust and Lurgi methods in gasifying anthracite silt.

	Pressure-dust gasification	Lurgi gasification
Process coal, tons/calendar day.....	2,760	4,194
Coal for power and steam, tons/calendar day.....	464	2,070
Coal for gasifier preheater, tons/calendar day.....	711	-
Coal for steam superheater, tons/calendar day.....	-	270
Total coal, tons/calendar day.....	6,935	6,534
Oxygen required, tons/calendar day.....	3,690	1,482
Operators per shift.....	16	59
Total plant investment including working capital and interest during construction.....	\$60,155,000	\$62,200,000
Operating costs, \$/calendar day. Total direct costs (silt coal @ \$1.25 and pellets @ \$2.50/ton).....	\$19,194	\$24,424
Total indirect costs.....	3,900	4,285
Total fixed costs.....	12,590	12,930
Total daily costs.....	35,684	41,639
Gas production, cu.ft./calendar day (900 B.t.u./cu. ft.).....		65,650,000
Cost, ¢/1,000 cu. ft. ....	54.5	63.5

### Coal Gasification with Air

A rough cost estimate was made for a Fischer-Tropsch plant using air instead of oxygen. It was found that the capital cost of a 10,000-barrel-per-day plant using air is \$23 million greater and that it would consume 1,388 tons per day more coal because of greater heating and power requirements.

### Gasification and Synthesis Plant (10,000-Bbl./Day)

Considerable effort was expended on this project during the past year. Three complete estimates of the plant actually were made for the purpose of orientation and economic analyses. This work resulted in the change or elimination of certain process steps as follows:

1. The production of synthesis gas from tail gas was found to be uneconomical and, instead, it was decided to utilize this gas as fuel.

2. Low-temperature off-gas separation was found impractical and eliminated.

The latest estimate on this plant is now being reviewed by a committee appointed by the National Petroleum Council at the request of the Secretary of the Interior.

### Coal-Hydrogenation Plant (30,000-Bbl./Day)

The cost estimate on this plant published in Report of Investigations 4584 also is being reviewed by the appropriate committee of the N.P.C. Several conferences have been held by Bureau engineers and members of this committee to familiarize the latter with the methods used, processes involved, and details of the high-pressure equipment required in this plant.

### Data for Corps of Engineers

The Bureau of Mines prepares and maintains a current chart of commercial-scale synthetic-fuel plant requirements based on the most recent data available from industry and demonstration plant findings. This chart (see fig. 26) is a compilation of raw materials, water, power, personnel, products, and wastes typical of synthetic-fuel plant operations. The information shown is made available to the Corps of Engineers to assist them in their Nation-wide survey for the selection of general locations for plant sites.

### Aromatic Hydrocarbons and Chemicals from Synthetic Liquid Fuels

A study was undertaken of the possibilities of producing benzene and other aromatic hydrocarbons, together with tar acids and other chemicals, from synthetic liquid fuels derived from coal and oil shale. The objectives of this study are manyfold and include determining the extent to which certain aromatic hydrocarbons can be obtained in the processing of synthetic liquid fuels, the processes employed for their recovery, the economics of such processes, and the effect the recovery of these substances will have upon the cost of synthetic liquid fuels.

This study is particularly timely, as the shortages of benzene and phenol have become more acute, and new sources for these materials must be found to satisfy the normal peacetime and defense needs of our country. In the past the major source of benzene has been coke ovens operated, for the most part, in conjunction with steel

**HYDROGENATION**  
Requirements for a 10,000 psi. 100% GASEOUS  
Batch at a 30,000 Btu./lb. PLANT

**SYNTHETIC USING COAL**  
Requirements for a 10,000 psi. 100% GASEOUS  
Based on a 10,000 Btu./lb. PLANT

**A. DESCRIPTION OF PROCESS**

[Include:

Brief description of flow sheet.

Names of equivalent or units proposed for use.

Significant temperatures are pressures.  
Overall efficiency [1] for average rank of coal  
and average quality of gas. 1

Coal, which is made into particles by mixing with coke,  
is hydrogenated at 10,000 psi and 500°F. in two  
continuous stages, liquid and vapor phases. Vapor  
phase products are distilled into finished gasoline  
and kerosene. With slight modification alternate  
products, such as jet fuel, diesel fuel, carbon  
oil, etc., can be produced. The gas  
is produced at 250 psi pressure by hydro-  
genation of coal with oxygen and conversion of hydro-  
genation of coal-gas with steam.

Steam and power production facilities are provided  
with the plant.  
A coal cleaning plant is included showing the use  
of run-of-mine coal.

The process data below are based on the following  
efficiency:

Over-all ..... 50.5% [1]

Cost of sulfur removal

7% of market price is taken for recovered sulfur.

High organic sulfur content in raw coal is undesirable.  
Low sulfur in process coal is preferable.  
(Chemical analysis & physical properties should  
be known.)

Cost of desulfurization does not change materially  
with percent of sulfur in coal when credit of 1/5  
of market price is taken for recovered sulfur.  
Only minor importance.

Fusible organic sulfur should be low in process  
coal.  
Fusible, normally can be removed by cleaning.

Volatile matter (raw) .... 31% minimum.  
Fixed carbon (raw) .... Not to exceed 55%  
Sulfur ..... 1% maximum.  
Moisture ..... No limit.

Rank of coal has little effect on total plant  
required.

For coals of equal rank, higher net hydrogen is  
desirable.

**OTHER CHARACTERISTICS**  
Effect of rank, coque attributes,  
moisture, etc.]

Information required for evaluation:  
1) Proximate analysis dry basis - include moisture  
as a secondary value of dry coal (face sample).  
2) Fischer Assay (dry basis).  
3) Ultimate analysis (raw basis) the content.

**SYNTHETIC USING NATURAL GAS [1]**  
Requirements for a 10,000 psi. 100% GASEOUS  
Based on a 27,000 Btu./lb. PLANT

Data presented below are based on hydrocarbon processes.  
Little information on this process is available at  
the present time. The process consists of bringing  
a stabilized natural gas with oxygen in a dy-  
namic flow system to form essentially carbon  
monoxide and hydrogen [2 parts hydrogen and 1 part  
carbon monoxide]. Diesel and gasoline hydrocarbons  
are synthesized by heating this mixture in a  
catalyzed bed of iron catalyst. Conventional oil  
refinery methods are used for separation, recovery,  
and upgrading three products. The data below are  
based on the following efficiency:

Overall efficiency ..... 47.2% [1].

Note: Inclusion to the literature of this process,  
if there is market for low pressure gas  
steam near the location of the plant, efficiency  
can be raised to about 62.5%.

\*The natural gas having a field gas pressure of  
500 psi, without compression and 1050 Btu./cu. ft.  
from selling value. [3]

FUEL REQUIREMENTS IN Btu. X 10 <sup>6</sup> /TALENDAH DAY							
	Process	N <sub>2</sub> w/o heat add.	Process heat add.	Steam	Total	Water	Product
Total	1	40	41	10	91	124	126
Natural gas	2						110
A. Maximum allowable heat content to equipment (dry basis)	4	16.5	20.5	10.5	21.0		
B. Other characteristics [Chemical analysis, etc.]	5	[1] Good desulfurizability [2] Desulfurization low sulfur in process coal is preferable. [3] Chemical analysis & physical properties should be known.					
C. Cost of sulfur removal	6						
D. Other characteristics [Chemical analysis, etc.]	7						

Data presented below are based on hydrocarbon processes.  
The process consists of bringing a stabilized natural  
gas with oxygen in a dynamic flow system to form essentially carbon  
monoxide and hydrogen [2 parts hydrogen and 1 part  
carbon monoxide]. Diesel and gasoline hydrocarbons  
are synthesized by heating this mixture in a  
catalyzed bed of iron catalyst. Conventional oil  
refinery methods are used for separation, recovery,  
and upgrading three products. The data below are  
based on the following efficiency:

Overall efficiency ..... 47.2% [1].

Note: Inclusion to the literature of this process,  
if there is market for low pressure gas  
steam near the location of the plant, efficiency  
can be raised to about 62.5%.

\*Cost of desulfurization does not change materially  
with percent of sulfur in coal when credit of 1/5  
of market price is taken for recovered sulfur.  
High organic sulfur content in raw coal is undesirable.  
as it is not easily removed by cleaning.

Fixed carbon ..... No limit.  
Volatiles matter ..... No limit.  
Sulfur ..... No limit.  
Moisture ..... No limit.

Information required for evaluation:  
1) Process steam will be provided by natural gas  
at 500 psi.

2) Availability of N<sub>2</sub>, CO<sub>2</sub>, etc. Increase the  
gas consumption and decrease the efficiency.  
and while details are not known, plant and  
operating costs will increase. 16% probable  
value increase due to addition of compressing  
plant.

**Figure 26. - Data for Corps of Engineers.**

Figure 26. - Data for Corps of Engineers (continued).

HYDROGENATION REQUIREMENTS FOR A 1,000 BBL./DAY PLANT BUILT ON A 10,000 BBL./DAY PLANT		SYNTHETIC USING NATURAL GAS 100% REQUIREMENTS FOR A 1,000 BBL./DAY PLANT BUILT ON A 10,000 BBL./DAY PLANT	
ESTIMATED QUANTITY OF PRODUCTS PER CALENDAR DAY (MARKED ON SPECIFIC QUANTITIES)			
		B.P.	BTU./LB.
F.	Non-liquefiable gases		
P.	Liquefied petroleum gases	2	Fracture - Butane - Isobutane -
R.	Gasoline	3	Gasoline - Kerosene -
S.	Diesel oil	4	Diesel oil -
T.	Kerosene	5	Kerosene -
U.	Distillate	6	Fuel oil -
V.	Residual	7	-
C.	Other (e.g., lubricants, other unpermitted water soluble compounds, etc.)	8	-
E.	Synergists	9	Monomer - Hydrogen -
G.	Total	10	10,000
H.	NATURE AND AMOUNT OF WASTES		
I.	SEWAGE	Liquids	10,000
J.	Description	Eff. by Treatment Plant	
K.	Industrial wastes	Leachate from cleaning plant	
L.	Plant and/or Household wastes	Household wastes in house	
M.	SOIL CLEANSING	Total soil category:	10
N.	PLANT AND FOR HABIT	6 Ash [100% ash]	100,000
O.	Water	100% water	100,000
P.	Land	100% land	100,000
Q.	Soil	100% soil	100,000
R.	Water	100% water	100,000
S.	Soil	100% soil	100,000
T.	Land	100% land	100,000
U.	Water	100% water	100,000
V.	Soil	100% soil	100,000
W.	Water	100% water	100,000
X.	Soil	100% soil	100,000
Y.	Land	100% land	100,000
Z.	Water	100% water	100,000
A.	AREA REQUIRED IN ACRES		
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Figure 26. - Data for Corps of Engineers (continued).

plants. In recent months benzene has been produced from petroleum, but the combined production still is insufficient, and shortages are predicted that may reach 50,000,000 gallons in 1950. Imports will ease but will not solve the problem.

A 30,000-barrel-per-day coal-hydrogenation plant could alleviate measurably the shortages of certain of these coal chemicals. Table 8 compares the total national production of benzene, toluene, xylenes, phenol, cresols, and xylencols that are obtained from coal hydrogenation and petroleum and the quantities of these chemicals available from a 30,000-barrel-per-day coal hydrogenation plant using a representative coal.

TABLE 8. - Comparison of U. S. production of aromatic chemicals and yield of a 30,000 bbl./day coal hydrogenation plant.

	1948	1949	Estimated annual yield coal hydrogenation plant
Thousands of gallons			
Benzene.....	173,795	156,620	34,000
Toluene.....	83,463	81,751	49,500
Xylenes.....	61,071	57,557	50,300
Thousands of pounds			
Phenol.....	297,330	224,544	34,500
Cresols.....	21,419	13,477	57,000
Xylencols.....	-	-	75,000

Cooperative agreements are being negotiated to determine the costs and maximum practical yields of benzene, toluene, and xylene by applying conversion processes employed in the petroleum industry to coal-hydrogenation products, and to evaluate the tar acids and other chemicals available.

As the quantities of some of these chemicals produced by coal hydrogenation are appreciably higher than present consumption, market surveys are being conducted to appraise the potential market, and a research program directed towards conversion of excess materials into usable products has been formulated. Results from these studies should be available in 1951.

#### Safety

During the extended high-pressure operations, some emergency let-downs were necessary, and a fire occurred. The emergencies were handled effectively and without any personnel injuries. Accident record for the year is as follows:

Man-hour exposure.....	830,442
Disabling injuries (lost-time accidents).....	2
Frequency rate.....	2.4
Severity rate.....	.06