WADD TR 61-72

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The following corrections are applicable to WADD TR 61-72, Research and Development on Advanced Graphite Materials:

Volume XXV - Lamellar Compounds of Nongraphitized Petroleum Cokes, by H. F. Volk, will be distributed to Government Offices only.

Volume XXIX - Evaluation of Graphite Materials in a Subscale Solid-Propellant Rocket Motor, by D. C. Hiler and R. B. Dull, will not be published.

Volume XXIX - Supplement - Title same as above, by S. O. Johnson and R. B. Dull, will not be published.

Volume XXXI - High Performance Graphite by Liquid Impregnation, will be published as Impregnation of Graphite.

Air Force Materials Laboratory
Research and Technology Division
Air Force Systems Command
United States Air Force
Wright-Patterson Air Force Base, Ohio

FOREWORD

This volume is the thirty-sixth of the WADD Technical Report 61-72 series describing various phases of research and development on advanced graphite materials conducted by Union Carbide Corporation, Carbon Products Division, under USAF Contract No. AF 33(616)-6915.

The work covered in this report was conducted from July 1962 through April 1963 at the Advanced Materials Laboratory, Lawrenceburg, Tennessee and Marathon Oil Company Refinery, Robinson, Illinois under the management of R. M. Bushong, Director of the Advanced Materials Project, J. C. Bowman, Director of Research, W. P. Eatherly, Assistant Director of Research, and R. C. Stroup, Manager of the Advanced Materials Laboratory.

The authors wish to acknowledge the help of the Marathon Oil Company, Findlay, Ohio, and Robinson, Illinois, who engineered and contracted the experimental unit on a nonprofit basis and the M. W. Kellogg Company, New York, New York, who engineered the basic design.

Acknowledgment is also given to the many technical and hourly workers at Marathon Oil Company, Robinson, Illinois; Marathon Research Laboratory, Littleton, Colorado; Parma Research Center, Union Carbide Corporation, Parma, Ohio; and at the Advanced Materials Laboratory, Lawrenceburg, Tennessee for the operation of this unit and the evaluative efforts on its products.

The contract for this R&D program was initiated under Project No. 7350, "Refractory Inorganic Nonmetallic Materials," Task No. 735002, "Refractory Inorganic Nonmetallic Materials; Graphitic;" Project No. 7381, "Materials Application," Task No. 738102, "Materials Processes," and Project No. 7-817, "Process Development for Graphite Materials." The work was administrated by the Air Force Materials Laboratory, Aeronautical Systems Division, with Major R. H. Wilson, L. J. Conlon and W. P. Conrardy acting as Project Engineers.

Other volumes in this WADD Technical Report 61-72 series are:

Volume	I -	Observations by Electron Microscopy of Dislocations in Graphite, by R. Sprague.
Volume	II -	Applications of Anisotropic Elastic Continuum Theory to Dislocations in Graphite, by G. B. Spence.
Volume	III -	Decoration of Dislocations and Low Angle Grain Boundaries in Graphite Single Crystals, by R. Bacon and R. Sprague.
Volume	IV -	Adaptation of Radiographic Principles to the Quality Control of Graphite, by R. W. Wallouch.

Volume	V -	Analysis of Creep and Recovery Curves for ATJ Graphite, by E. J. Seldin.
Volume	VI -	Creep of Carbons and Graphites in Flexure at High Temperature, by E. J. Seldin.
Volume	VII -	High-Density Recrystallized Graphite by Hot-Forming, by E. A. Neel, A. A. Kellar and K. J. Zeitsch.
Supplement	-	High-Density Recrystallized Graphite by Hot-Forming, by G. L. Rowe and M. B. Carter.
Volume	VIII -	Electron Spin Resonance in Polycrystalline Graphite, by L. S. Singer and G. Wagoner.
Volume	IX -	Fabrication and Properties of Carbonized Cloth Composites, by W. C. Beasley and E. L. Piper.
Volume	X -	Thermal Reactivity of Aromatic Hydrocarbons, by I. C. Lewis and T. Edstrom.
Supplement	-	Thermal Reactivity of Aromatic Hydrocarbons, by I. C. Lewis and T. Edstrom.
Volume	XI -	Characterization of Binders Used in the Fabrication of Graphite Bodies, by E. deRuiter, A. Halleux, V. Sandor and H. Tschamler.
Supplement	-	Characterization of Binders Used in the Fabrication of Graphite Bodies, by E. deRuiter, J. F. M. Oth, V. Sandor and H. Tschamler.
Volume	XII -	Development of an Improved Large-Diameter, Fine-Grain Graphite for Aerospace Applications, by C. W. Waters and E. L. Piper.
Supplement	-	Development of an Improved Large-Diameter, Fine-Grain Graphite for Aerospace Applications, by R. L. Racicot and C. W. Waters.
Volume	XIII -	Development of a Fine-Grain Isotropic Graphite for Structural and Substrate Applications, by R. A. Howard and E. L. Piper.
Supplement	-	Development of a Fine-Grain Isotropic Graphite for Structural and Substrate Applications, by R. A. Howard and R. L. Racicot.
Volume	XIV -	Study of High Temperature Tensile Properties of ZTA Grade Graphite, by R. M. Hale and W. M. Fassell, Jr.

Volume	XV -	Alumina-Condensed Furfuryl Alcohol Resins, by C. W. Boquist, E. R. Nielsen, H. J. O'Neil and R. E. Putcher.
Volume	XVI -	An Electron Spin Resonance Study of Thermal Reactions of Organic Compounds, by L. S. Singer and I. C. Lewis.
Volume	XVII -	Radiography of Carbon and Graphite, by T. C. Furnas, Jr. and M. R. Rosumny.
Volume	XVIII -	High Temperature Tensile Creep of Graphite, by E. J. Seldin.
Volume	XIX -	Thermal Stresses in Anisotropic Hollow Cylinders, by Tu-Lung Weng.
Volume	XX -	The Electric and Magnetic Properties of Pyrolytic Graphite, by G. Wagoner and B. H. Eckstein.
Volume	XXI -	Arc Image Furnace Studies of Graphite, by M. R. Null and W. W. Lozier.
Volume	XXII -	Photomicrographic Techniques for Carbon and Graphite, by G. L. Peters and H. D. Shade.
Volume	XXIII -	A Method for Determining Young's Modulus of Graphite at Elevated Temperatures, by S. O. Johnson and R. B. Dull.
Volume	XXIV -	The Thermal Expansion of Graphite in the c-Direction, by C. E. Lowell.
Volume	XXV -	Lamellar Compounds of Nongraphitized Petroleum Cokes, by H. F. Volk.
Volume	XXVI -	Physical Properties of Some Newly-Developed Graphite Grades, by R. B. Dull.
Volume	XXVII -	Carbonization Studies of Aromatic Hydrocarbons, by I. C. Lewis and T. Edstrom.
Volume	XXVIII -	Polarographic Reduction of Polynuclear Aromatics, by I. C. Lewis, H. Leibecki and S. L. Bushong.
Volume	XXIX -	Evaluation of Graphite Materials in a Subscale Solid-Propellant Rocket Motor, by D. C. Hiler and R. B. Dull.
Supplement	-	Evaluation of Graphite Materials in a Subscale Solid-Propellant Rocket Motor, by S. O. Johnson and R. B. Dull.

NOTE - Volume XXV - Lamellar Compounds of Nongraphitized Petroleum Cokes, by H. F. Volk, will be distributed to Government Offices only.

Volume XXX - Oxidation-Resistant Graphite-Base Composites, by K. J. Zeitsch and J. Criscione.

Volume XXXI - Impregnation of Graphite; by C. E. Waylett, M. A. Spring and M. B. Carter.

Volume XXXII - Studies of Binder Systems for Graphite, by T. Edstrom, I. C. Lewis, R. L. Racicot and C. F. Stout.

Volume XXXIII - Investigation of Hot Worked Recrystallized Graphite, by J. H. Turner and M. B. Carter.

Volume XXXIV - Oxidation Resistant Coatings for Graphite, by D. A. Schulz, P. H. Higgs and J. D. Cannon.

Volume XXXV - Methods of Measuring Mechanical Properties of Graphite in the 20° to 2700°C Temperature Range, by M. B. Manofsky and R. B. Dull.

ABSTRACT

This report describes the design, construction, and operation of an experimental delayed coking unit for the conversion of petroleum residues to coke for use as the filler component in graphite for aerospace applications. The effect of changes in operating conditions of the experimental coker, e. g., time-temperature, pressure, and recycle ratio was investigated with respect to the physical properties of graphitized extruded and molded samples. Four basic charge stocks were studied: vacuum residuum, slurry (decant) oil, thermal tar, and a low sulfur thermal tar. Variations in raw coke bulk density are reported; in addition, data concerning kerosene density, ash, and sulfur content of the 1000°C calcined cokes are also included. Changes in bulk density, weight, and volume of the extruded and molded test samples resulting from the processing steps of baking and graphitizing are reported. The CTE (coefficient of thermal expansion), specific resistance, and flexural strength of the graphitized molded plugs in both the with- and across-grain directions are reported as well as the same properties for the with-grain direction of extruded rods.

High coke yields from cracked feed stocks were obtained by operating at high recycle ratios and higher-than-normal drum pressures. Thermally and catalytically-cracked charge stocks were coked to produce cokes yielding fabricated graphites exhibiting low CTE. Super-heated steam introduced into the hot oil stream between the furnace and the coking drum resulted in a marked effect on the coke produced from vacuum residuum; the graphite formed using this coke as filler material was higher in CTE and more isotropic than conventional graphites, making it useful as a substrate for coatings.

This technical documentary report has been reviewed and is approved.

W. G. RAMKE
Chief, Ceramics and Graphite Branch
Metals and Ceramics Division
Air Force Materials Laboratory

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1. INTRODUCTION

Uniformity and reproducibility of fabricated graphite are dependent upon raw materials which will yield (a) filler components, (b) binder components, and (c) impregnants which are uniform in their general composition and properties. The properties of the finished graphite can be strongly influenced by the characteristics of the raw materials, particularly those of the filler component. Thus, major effort must be directed towards investigation of reproducible raw materials which yield graphite-based materials with desirable combinations of thermal and mechanical properties for aerospace applications.

In work reported elsewhere [1], effort has been directed toward achieving a basic understanding of the mechanisms and kinetics of thermal processing whereby relatively low molecular weight hydrocarbons are converted to a stage of polymerization suitable for use as a binder, or, further, to a stage ("coke") suitable for use as a filler component. Bench-scale, batch experimentation has demonstrated that the characteristics of the coke can be varied over a wide range, not only by varying the initial composition of the material processed, but also by varying time, temperature, and pressure during the coking process.

The present report discusses the design and construction of an experimental delayed coking apparatus and the results of the first phase of an experimental program using selected petroleum residues as feed material. The experimental delayed coker employs a "flow" process in contrast to the "batch" operation used in the bench-scale work and, hence, supplies information useful for design of production scale equipment.

2. OBJECTIVES

Petroleum coke has long been the major raw material employed as the filler component in the manufacture of graphite. The petroleum refining industry, however, considers petroleum coke a by-product; coking is carried on only to obtain an increased yield of light products, either motor fuel or gas oil, which, in turn, can be readily converted to motor fuel. Coking of high boiling petroleum stock is a process involving depositing in solid form a material of low hydrogen-to-carbon ratio in order to obtain the desired overhead products of higher hydrogen-to-carbon ratio.

The dominant interest, on the other hand, for the manufacture of graphite, and thus for the present program, lies in the coke rather than the overheads, particularly in those characteristics of the coke which strongly influence the properties of graphite. These characteristics include the amount of volatile matter, ash, and sulfur, but more importantly the spatial structure is largely planar or substantially cross-linked in three dimensions.

The experimental unit discussed in this report has been designed with the purposes of achieving a high degree of flexibility and permitting the use of a much wider range of pressures and temperature than is possible in present commercial units. Pipe-coil furnaces, therefore, are rated for 1000 lbs/in^2 gauge at 1000°F, and the coke drums for 400 lbs/in^2 gauge at 950°F. Four helical-coil furnaces are available, which can be employed in pairs of two singly, pairs of two in parallel, or as a unit in series. The temperature of the oil at the outlet of each furnace is separately controlled. The heater volume and temperature profile can be varied over a wide range. The unit can be employed not only for coking but also, through a by-pass of the coke drum, for thermal cracking. In this latter mode of operation, a heavy liquid tar is obtained, rather than a solid polymer, as in coking. The experimental coker is highly instrumented, permitting fine control of the unit and accurate material balances.

The experimental program was designed to obtain a maximum amount of information in the time available. Parameters which were studied included the following variations: the nature of the fresh feed stock (four types); the degree of severity of cracking in the pipe-coil furnaces by changes in temperature profile, furnace feed rate, and outlet pressure; in coke drum pressure from 50 to 350 lbs/in^2 gauge; in recycle ratio from 0.2 to 3.1; and in the amount of steam injected into the hot oil stream entering the coke drum.

3. HISTORY

Early work by Union Carbide Corporation, Carbon Products Division (formerly National Carbon Company) indicated that the dominant variable in a relatively rapid batch coking process (at atmospheric pressure), insofar as the with-grain coefficient of thermal expansion (CTE) of the finished graphite is concerned, was the chemical composition of the charge material. Table 1 illustrates this point by comparing the CTE of graphites made of cokes from 70 tests on four basic charge stocks from different refineries. The procedure for evaluation of these stocks

Table 1. CTE Measurements of Laboratory Cokes from Various Feed Stocks

	No. of Tests	CTE* 10^{-6}/°C	Variation
Atmospheric Reduced Crude(1)	19	1.54	±0.24
Vacuum Reduced Crude (2)	11	1.73	±0.15
Slurry and Decant Oils (3)	23	0.50	±0.10
Thermal Tars (4)	17	0.53	±0.11

(1) Virgin residue from atmospheric distillation of a crude oil.
(2) Virgin residue from vacuum distillation of an atmospheric reduced crude oil.
(3) Residue from distillation of a catalytically cracked distillate stock.
(4) Residue from distillation of a thermally cracked stock.

was as follows: the oils were coked at 450°C in one-liter stainless steel or glass crucibles protected in a double sagger. The resulting raw coke was then calcined to 1000°C, milled to a flour, hot mixed with a 100°C softening point coal tar pitch and extruded into rods using a 10-ton press. After the rods were baked and graphitized by conventional methods and ground into strips, CTE measurements were taken. *

Later bench scale batch coking experiments clearly demonstrated that the coke properties are strongly dependent, not only on the feed stock, but also upon the parameters of coking, such as time, temperature, and pressure. The effects of varying the time-temperature complex at atmospheric pressure are shown in Table 2.

* All CTE values in this report are mean values over the temperature range 30° to 100°C.

Table 2. Effect of Time on Coke Properties, Atmospheric Coking, Vacuum Residuum Charge

Experiment No.	Time - Hours 300°C	300°C	390°C	390°C	450°C	450°C	Yields Weight Per Cent Raw Coke	Calcined	Overall	Calcined Coke Kerosene Apparent Density g/cc	Per Cent Sulfur	Per Cent Ash	Graphite Rod Properties Bulk Density g/cc	Specific Resistance 10^{-6} ohm-cm	With-grain CTE 30 - 100 °C 10^{-6} / °C	N	Coking Equipment
1	5	0	1.5	0	2	5	13.9	92.5	12.9	2.041	1.1	0.51	1.575	8.55	1.62 ± 0.06	2	Crucible
2	0.75	0	3	13	7	5	13.9	94.5	13.1	--	---	----	1.544	8.31	1.22 ± 0.03	2	Crucible
3	0.75	0	3	13	7	5	18.1	90.9	16.5	1.941	0.62	0.62	1.537	8.95	1.22 ± 0.01	2	Autoclave

Experiments 1 and 2 were performed in beakers and experiment 3 in the batch-coking equipment illustrated in Figure 1. The lower CTE with increased hold time at 390°C is quite evident. Experiments 2 and 3 demonstrate that the same results are obtained, whether carried out in beakers or in the larger scale vessel (Figure 1).

With the latter equipment, batch coking experiments were performed at pressures up to 1000 lbs/in^2 gauge. The data of Table 3 (with Table 4 giving analysis of overhead products) and Figure 2 indicate the pronounced decrease in CTE obtained by the application of pressure during thermal processing, particularly for a vacuum residuum charge stock.

Apparently, the major effect of pressure is the retention in the liquid phase of those products of thermal cracking which would vaporize and distill from the vessel at atmospheric pressure. Retention of these materials in the liquid phase is considered to affect the properties of the coke in two ways:

a) Such materials are partially converted into solid polymer (coke) in the later stages of thermal treatment, evidenced by the substantial increase in coke yield with increasing pressure.

b) Such materials provide a diluent during the early stages of thermal treatment. In the presence of such diluent, the thermal decomposition (cracking) of high molecular weight asphaltenes (in a virgin charge stock) is favored over their polymerization to a cross-linked polymer. Cracking is an unimolecular reaction, whereas polymerization is a bimolecular reaction which is dependent upon concentration.

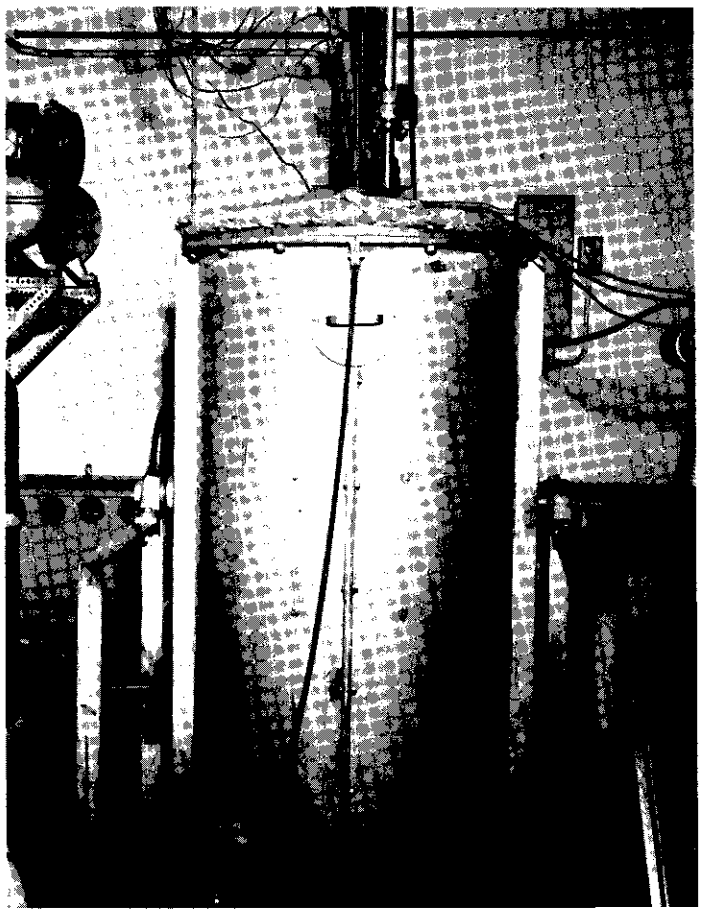

Figure 1. Pressure Autoclave at Advanced Materials Laboratory

In some of the batch coking experiments, high pressures were employed only during the early (400°C) stages of thermal treatment; the later stages simulate the flow condition of the pilot coker, with the early stages corresponding to the thermal treatment in the furnace coil and the later stages corresponding to the thermal treatment applied in the coke drum. Evidently, a substantial reduction of CTE for coke* from vacuum residuum may be obtained by achieving a severity of cracking during the "precoking" stage corresponding to the production of 15 weight per cent of gasoline (205°C end point).

Translation of these results from batch experiments to the dynamic or flow conditions of the pilot delayed coker depends in large measure on (a) the maximum degree of cracking severity which can be accomplished in the furnace without coke deposition, and (b) the pressure maintained in the coke drum. In turn, the cracking severity attainable

* Actually the CTE is measured on graphite strips fabricated from the coke as described previously.

in the furnace without coke deposition depends on the degree of dilution of the fresh charge, that is, the recycle ratio.

Table 3. Effect of Pressure on Coke Properties
Batch Coking of a Virgin Vacuum Residuum

Run No.	Charge Stock	Pressure - lbs/in²			Yields - Weight Per Cent					Extruded Graphitized Rod Data			Calcined Coke Data		
		Initial	Through 400°C	Through 470°C	Raw Coke	Distillate	Gas	Loss	1000°C Calcined Coke	Bulk Density g/cc	Specific Resistance 10^{-4} ohm-cm	With-grain CTE 30 - 100°C 10^{-6} /°C	Kerosene Apparent Density g/cc	Sulfur Per Cent	Ash Per Cent
3	Vacuum Residuum-Continental Oil Co.	0	0	0	20.6	69.0	9.9	-0.5	18.7	1.53	5.56	1.42	1.987	0.94	0.29
1	Vacuum Residuum-Continental Oil Co.	90	200	200-0	34.6	45.1	22.3	+2.0	32.1	1.50	6.02	0.70	1.975	0.78	0.34
5	Vacuum Residuum-Continental Oil Co.	380	1000	1000-0	36.2	30.0	33.9	+0.1	33.1	1.44	7.00	0.58	1.928	0.62	0.22
7	Vacuum Residuum-Continental Oil Co.	300	1000	400-0	37.0	40.5	22.5	--	33.7	1.53	8.02	0.66	2.007	0.82	0.33
8	Vacuum Residuum-Continental Oil Co.	300	1000	50-0	32.8	41.0	23.4	-2.8	28.7	1.50	9.50	0.81	2.025	0.91	0.39
32	Slurry Oil-Marathon Oil Co.	0	0	0	23.0	68.8	9.3	+1.1	21.9	1.52	6.45	0.44	2.002	0.45	0.29
33	Slurry Oil-Marathon Oil Co.	90	200	200-0	44.9	34.4	19.5	-1.2	40.2	1.52	7.20	0.33	2.043	0.49	0.12
34	Slurry Oil-Marathon Oil Co.	300	1000	1000-0	52.2	17.0	25.6	-5.2	48.2	1.54	6.38	0.15	1.881	0.52	0.07

Firing Schedule - Rush to 315, 9.5 ± 1.0 hrs. at 300-325°C, 6½ hrs. to 390-415°C, 15 hrs. ± 1.0 hrs. at 400°C ± 10°C, 5 hrs. to 460°C, 7 hrs. at 470°C ± 5°C, last hour at 0 lbs/in² gauge.

Table 4. Analysis of Laboratory Coker Overheads

Run No.	Charge Stock	Distillate per cent	Conradson Carbon per cent	Through 400°C Hold										Gas per cent
				Gasoline					Gas Oil					
				Yield per cent	S.G. g/cc	B.P.°F Ave. Molal	K(1)	M.W. (est.)	Yield per cent	S.G. g/cc	B.P.°F Ave. Molal	K(1)	M.W. (est.)	
3	Vacuum Residuum Continental Oil Co.	57.9	0.72	17.2	0.813	284	11.1	114	40.7	0.899	637	11.5	265	5.0
1	Vacuum Residuum Continental Oil Co.	40.3	0.02	16.0	0.858	259	10.4	101	24.3	0.863	483	11.4	188	13.5
5	Vacuum Residuum Continental Oil Co.	17.7	0.02	12.0	0.739	234	12.0	107	5.7	0.900	487	10.9	182	--
7	Vacuum Residuum Continental Oil Co.	18.0	0.02	12.8	0.777	272	11.6	113	5.2	0.947	506	10.4	175	17.2
8	Vacuum Residuum Continental Oil Co.	39.4	0.02	22.6	0.741	229	11.9	105	16.8	0.943	490	10.9	182	12.3
32	Slurry Oil Marathon Oil Co.	38.7	0.10	3.1	0.763	302	12.0	129	35.6	0.925	583	11.0	222	2.9
33	Slurry Oil Marathon Oil Co.	11.3	0.15	5.3	0.721	332	12.9	150	6.0	0.887	528	11.2	205	5.9
34	Slurry Oil Marathon Oil Co.	0.0	--	0.0	--	--	--	--	0.0	--	--	--	--	2.2

Table 4. Analysis of Laboratory Coker Overheads (Continued)

Run No.	Through 470°C Hold: Yield per cent	Conradson Carbon per cent	Gasoline (400°F - E.P.) B.P. °F: Yield per cent	S.G. g/cc	Ave. Molal	K(1)	M.W. (est.)	Gas Oil B.P. °F: Yield per cent	S.G. g/cc	Ave. Molal	K(1)	M.W. (est.)	Gas per cent	Total, per cent: Gasoline 400°F(E.P.)	Gas-Oil	Total, per cent: Raw Coke	Distillate	Gas
3	11.1	1.06	2.2	0.799	265	11.2	109	8.9	0.924	630	11.1	255	4.9	19.4	49.6	20.6	69.0	9.9
1	4.8	5.00	1.5	0.830	285	10.9	111	3.3	0.930	527	9.0	193	8.8	17.5	27.6	34.6	45.1	22.3
5	12.3	1.22	7.7	0.891	303	10.3	110	4.6	1.096	596	9.3	-		19.7	10.3	36.2	30.0	33.0
7	8.6	1.70	4.1	0.860	282	10.5	108	4.5	1.197	605	8.5	Off Chart	5.3	16.9	9.7	37.0	26.6	22.5
8	1.6	0.29	0.8	0.869	285	11.2	114	0.8	1.033	577	9.8	188	11.1	23.4	17.6	32.8	41.0	23.4
32	30.1	0.44	1.2	0.764	312	12.0	132	28.9	1.000	598	10.2	210	6.4	4.3	64.5	23.0	68.8	9.3
33	23.1	0.18	6.7	0.796	265	11.3	109	16.3	0.993	590	10.2	207	13.6	12.0	22.3	44.9	34.4	19.5
34	17.0	0.10	7.1	0.779	277	11.6	124	9.9	1.016	598	10.0	208	23.4	7.1	9.9	52.2	17.0	25.6

(1) K = Characterization Factor = $\sqrt[3]{\frac{\text{Molal Ave. Boiling Point (°R)}}{\text{S.G. 60°F/60°F}}}$

Molecular Weight estimated from K data

S.G. = Specific Gravity 60°F/60°F
B.P. = Boiling Point (Molal average)
M.W. = Molecular Weight

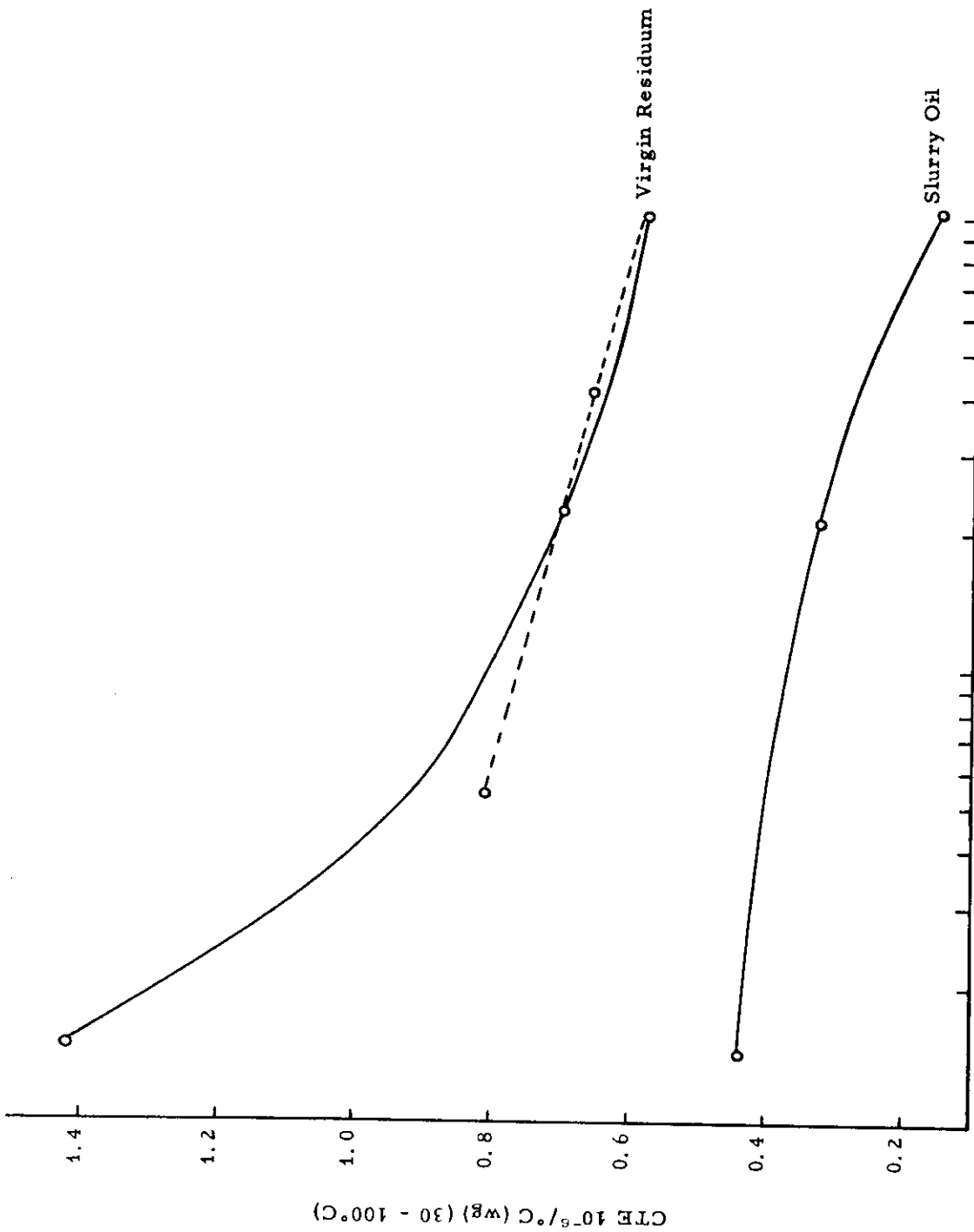

Figure 2. Coking Pressure versus CTE of Coke (Advanced Materials Laboratory)

4. PLAN OF EXPERIMENTAL PROGRAM

A four-stage preliminary schedule was prepared to cover the variables considered to be of major importance. Phases 1 - 3 involved three charge stocks where the conditions of time, temperature, and pressure were varied at a comparatively mild temperature profile in the furnace coils by a substantial expension of time at temperatures above 750°F.

The schedule was prepared on the basis of the batch experiments discussed in Section 3, combined with Marathon Oil Company's experience with commercial delayed coking operations. A total of 24 experiments was planned for the first three months of operation, with the understanding that revisions might be made during this period.

Definitions of the terms used are:

Temperature (T)	-	°F at the final furnace outlet
Pressure (P)	-	lbs/in^2 gauge in coke drum
Space Velocity (SV)	-	indication of the residence time of the feed in the furnaces $SV = \frac{\text{Flow rate (gal/hr)}}{\text{Furnace volume (gal) above a reference temperature}}$
Reference Temperature	-	a temperature established for a given charge stock above which the thermal cracking rate is rapidly increased
Recycle Ratio	-	ratio of recycled material to that of fresh feed
Profile (° F)	-	temperatures maintained on furnace outlets
Furnace Feed Rate	-	total furnace charge expressed in gal/hr
Drum Cycle	-	time in hours operating in a given coke drum

Charge Stocks

Vacuum Residuum	-	residue from the combined atmospheric and vacuum distillation of a crude petroleum oil
Slurry Oil	-	residue from the distillation of catalytically cracked, mixed gas oils
Thermal Tar	-	residue from the distillation of a thermally cracked distillate stock

EXPERIMENTAL SCHEDULE

Phase 1

FIXED CONDITIONS

Fresh Feed	- Vacuum Residuum
Drum Cycle	- 24 hours
Reference Temperature (for SV calculations)	- 750°F
Recycle	1:1

Experiment	1	2	3	4	5	6	7	8	9
Run No.	1	2	3	4	5	6	7	22	23
Temperature	900°	900°	930°	930°	930°	900°	900°	900°	900°
Pressure	50	50	50	50	50	140	140	400	400
Space Velocity	20	10	20	10	20	20	10	20	10
Furnace Feed Rate	340	170	378	189	378	340	170	340	189
*Profile	A	A	B	B	B	A	A	A	B

*Legend of Profile

Furnace	A	B
1	525 - 624°	525 - 630°
2	624 - 688°	630 - 702°
3	688 - 791°	702 - 813°
4	791 - 900°	813 - 930°

Phase 2

FIXED CONDITIONS

Fresh Feed	- Slurry Oil
Drum Cycle	- 24 hours
Reference Temperature (for SV calculations)	- 800°F
Recycle	1:1

Experiment	1	2	3	4	5	6
Run No.	8	9	10	11	18	19
Temperature	930°	950°	930°	950°	930°	950°
Pressure	50	50	140	140	400	400
Space Velocity	20	20	20	20	20	20
Furnace Feed Rate	285	310	285	310	275	310
*Profile	B	C	B	C	B	C

*Legend of Profile

Furnace	A	B
1	525 - 630°	525 - 634°
2	630 - 702°	634 - 711°
3	702 - 813°	711 - 827°
4	831 - 930°	827 - 950°

Phase 3

FIXED CONDITIONS

Fresh Feed	- Thermal Residuum
Drum Cycle	- 24 hours
Reference Temperature (for SV calculations)	- 800°F
Recycle	1:1

Experiment	1	2	3	4	5	6
Run No.	12	13	14	15	20	21
Temperature	930°	910°	930°	910°	930°	910°
Pressure	50	50	140	140	400	400
Space Velocity	10	10	10	10	10	10
Furnace Feed Rate	138	124	138	124	138	124
*Profile	B	D	B	D	B	D

*Legend of Profile

Furnace	B	D
1	525 - 630°	525 - 626°
2	630 - 702°	626 - 692°
3	702 - 813°	692 - 798°
4	813 - 930°	798 - 910°

Phase 4

FIXED CONDITIONS

Fresh Feed	- Slurry Oil
Drum Cycle	- 24 hours
Reference Temperature (for SV calculations)	- 800°F
Recycle	1:1

Experiment	1	2	3
Run No.	8	16	17
Temperature	930°	930°	930°
Pressure	50	50	50
Space Velocity	20	12.5	10
Furnace Feed Rate	285	285	285
*Profile			

*Legend of Profile

Furnace	B	E	F
1	525 - 63 °	525 - 675°	525 - 733°
2	630 - 702°	675 - 776°	733 - 871°
3	702 - 813°	776 - 930°	871 - 930°
4	813 - 930°	930 - 930°	930 - 930°

POST TREATMENT OF COKE IN ALL EXPERIMENTS

1. Drum switched off feed.
2. 600 lbs/in^2 gauge steam bled in.
3. Slow depressurization to blowdown line.
4. Continue steaming at atmospheric pressure (to blowdown line) for two hours.
5. Begin introduction of water with steam over a period of two hours.
6. Cut steam out and fill drum with water to 9-foot outage level.
7. Cool to 200°F coke temperature.
8. Drain water from drum.
9. Hydraulic decoking of drum.

5. EXPERIMENTAL PHASES

5.1. Sampling Procedures

5.1.1. Overhead and Feed Products

Samples of product gasoline and gas oil were taken at eight-hour intervals during each experiment. A sample of the product gas was taken when there was a major change in operating conditions or feed stock. A sample of combined feed (furnace charge) was taken during each experiment after steady-state conditions had been achieved, and a fresh feed sample was obtained at least once during each experiment. Fresh and combined feed samples were always checked for specific gravity and at intervals subjected to chemical characterization.

5.1.2. Coke Product

5.1.2.1. General Procedure

1) The decoking crew checked with the control room to determine if sampling technique was to be applied to that specific cycle.

2) If the sampling technique was not applied, no specific decoking procedure was required. The coke was hauled to a storage area and accumulated for later disposal.

3) If the sampling technique was applied, the decoking and sampling techniques outlined in Sections 5.2.2 and 5.2.3 were followed.

5.1.2.2. Decoking

The coke was divided into three equal portions, except when there was less than ten feet of coke in the drum, in which case the coke was divided into two equal portions. The material from the center hole and that from within one foot of the bottom was not sampled. The coke was always cut into equal sections starting from the top.

1) The center hole was cut first, which sometimes caused the bottom one or two feet of coke to fall out. If this action occurred, it was not necessary to cut any additional material off the bottom.

2) The second, third and fourth loads of coke were separated on the pad to avoid mixing.

3) Samples were obtained from loads 2, 3 and 4 after the coke had been allowed to dry overnight.

5.1.2.3. Sampling Procedure

1) Any pieces larger than approximately one foot in diameter were broken into smaller pieces.

2) The pile of coke was spread out evenly until it was no higher than one foot.

3) The pile was then divided into four quarters by marking with a shovel.

4) Two opposite quarters were discarded and the other two quarters kept. The material kept was mixed and then divided into quarters again as in step 3. Two opposite quarters were again discarded, and the remaining sample mixed and quartered until the pile was reduced to a quantity sufficient for the sample drum (100 pounds).

5) The discarded coke was removed and the pad cleaned.

5.2. Testing Procedure

5.2.1. Overheads from Coking and Feed Material

Samples of product gasoline and gas oil were taken every eight hours and tested as follows:

Method	ASTM Designation
Gasoline	
Gravity °API	D287-55
Distillation	D 86-61
Gas-Oil	
Gravity °API	D287-55
Distillation	D158-59
Conradson Carbon	D189-61

During critical experiments in which there was a radical change in operating conditions or fresh feed composition, a sample of gas was subjected to a mass spectrometric examination by ASTM Proposed Specification, "Analysis of Light Petroleum Products by Mass Spectrometry".

Each fresh feed used was characterized by the following methods:

Method	ASTM Designation
Vacuum Distillation	D1160-61
Gravity °API	D 287-55
Viscosity SSU and SSF	D 88-56
Conradson Carbon	D 189-61
Per Cent Aromatic and Nonaromatics	Ref: Anal. Chem. Vol. 30, No. 7, July 1958, Eby, L. T.
Per Cent Aromatic Carbons Per Cent Paraffinic Carbons Per Cent Napthenic Carbons	Ref: Anal. Chem. Vol. 25, No. 7, July 1953, Kurtz, et al

5.2.2. Coke Product Testing

5.2.2.1. Calcining of Raw Cokes for Laboratory Evaluation

The raw coke samples were first dried at 115 to 125° for 48 to 60 hours in an air circulating oven. The dried coke was then crushed in a roll crusher and the material passing through 12 mesh was discarded. The coke retained on 12 mesh was placed in an inverted double sagger protected against air infiltration by the annular space filled with layers of packing coke, wood charcoal and sand. Calcining was effected in a pot type furnace with temperature raised rapidly to 400°C, controlled at 60°C/hr. to 1000°C, and then held 6 hours at 1000°C.

5.2.2.2. Large Scale Calcining of Raw Cokes for Product Evaluation

Two types of raw cokes, slurry oil coke and vacuum residuum coke, were produced on a large scale in the experimental coker. The formulation characteristics for both cokes are discussed in Section 6.3. The slurry oil coke was the entire production of cycles 23S to 25S.

Both of these cokes are to be processed into BO graphite material, after which this material is to be used in the forming of 18-inch diameter graphite plugs. One of the necessary processing steps in the formulation of BO materials from raw coke is the 1350°C calcining operation where approximately 8 to 10 per cent of the residual volatiles in the raw coke are eliminated. A 500 KW induction heating furnace was selected for this 1350°C calcining operation in order to provide uniform heating for a reasonably large coke charge.

A cross sectional view of the induction heating assembly used in the 1350°C calcination of the slurry oil coke is presented in Figure 3. The heating susceptors were machined from a standard grade of bulk graphite to 42-inch outside diameter by 35-inch inside diameter by 30-inch length. The end plates were machined from the same material to 42-inch diameter by 3-inch length.

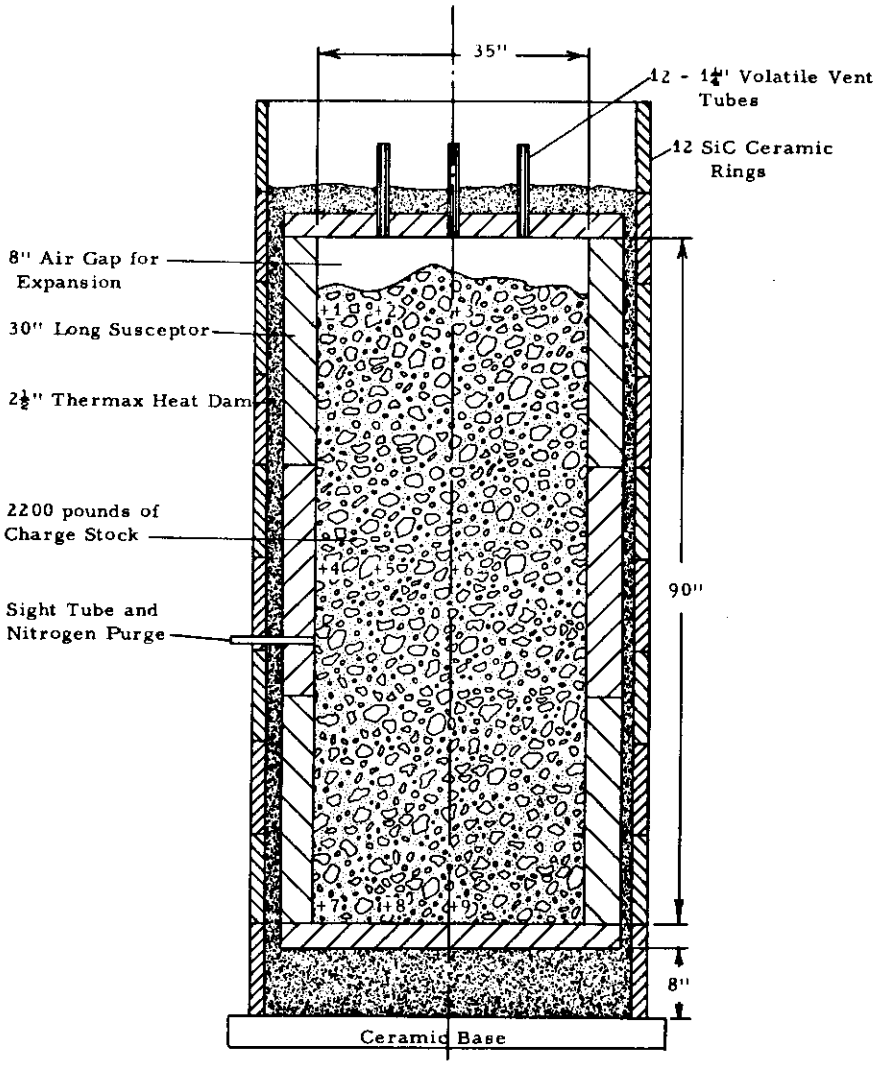

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Figure 3. Induction Calcining Assembly.

As illustrated in Figure 3, a complete assembly consisted of three susceptors and two end plates along with the insulating members. The primary insulation was a 2½-inch thermatomic heat barrier placed between the susceptors and the ceramic rings. The only element of the assembly not shown in Figure 3 is the 52-inch diameter by 108-inch length induction heating coil. The electrical power to the coil was supplied from a 500 KW/180 CPS source. This assembly allowed a maximum charge of 2250 pounds of raw coke with a yield, after calcining, of 2000 pounds.

The firing schedule for this calcining operation was as follows:

First day - Room Temperature to 900°C in 6 hours
Second day - 400°C (approximate) to 1350°C in 6 hours
Hold at 1350°C for 1 hour.

The heating schedule was regulated essentially by the evolution of volatile gases during the operation. If the charge were carried directly to 1350°C, the volatile evolution would greatly exceed the capacity of the fume system.

The one hour hold time was necessary to minimize the temperature gradient within the charge. A 13 mm sight tube was placed at the center of the charge and another at the surface of the susceptor during one heating cycle in order to measure optically the radial gradient. After a one hour hold at 1350°C, there was no measurable gradient across this path.

Since specific resistance is a direct function of the temperature of heat treatment, longitudinal temperature gradients were assessed by resistance measurements on the coke. After calcining to 1350°C, nine samples of coke were selected from the positions marked 1 through 9 in Figure 3 and milled to a flour (45 per cent through 200 mesh). The specific resistance of each sample was then measured. This data, shown in Table 5, indicates that only a small temperature gradient existed from top to bottom of the assembly.

Table 5. Specific Resistance Measurements 1350°C Calcine - Robinson Slurry Oil Coke

Sample Number	N	Specific Resistance*, ohm-in.
1	3	0.03425
2	3	0.03398
3	3	0.03474
4	3	0.03433
5	3	0.03550
6	3	0.03622
7	3	0.03520
8	3	0.03686
9	3	0.03664

N = Number of determinations per sample
* = Average of N determinations

5.2.3. Preparation of Samples for Laboratory Evaluation

5.2.3.1. Extruded Rods for CTE and Electrical Resistivity Determination

The 1000°C calcined coke was milled to approximately 55 per cent throuth a 200 mesh screen and hand mixed with premelted 100 to 110°C melting point coal tar pitch at 130 to 140°C. The binder level was

32 parts per hundred (pph) ± 0.5 pph.* Iron oxide (2 pph) was used as a puffing inhibitor. The hot mix was charged to an extrusion press capable of delivering ten tons per square inch on an area of two square inches and extruded at a temperature of 125°C ± 2-C into $\frac{5}{8}$-inch by 6-inch rods. The green rods were packed in 6/3 mesh calcined coke in an inverted double sagger and baked on a schedule of 25°C/hr. to 400°C, followed by 50°C/hr. to 1000°C. The rods were then heated to 3000°C in a graphite tube furnace in a nitrogen atmosphere.

5.2.3.2. Molded Plugs for Across-Grain Properties

The materials for molding 3-inch diameter by 3-inch length plugs were prepared as described for the extruded rods. Mixing was accomplished in a heated Baker-Perkins 4-AN2 type mixer. After 20 minutes mixing at 150°C, the mix was transferred to a preheated mold.

Molding was accomplished in an electrically heated mold on a Wabash 12-ton hydraulic press. This press was a laboratory type which provided for constant pressure and temperature during the entire molding operation. The plugs were molded at 115°C and a pressure of 3000 lbs/in^2 with a 15-minute hold at the molding pressure. The plugs were then baked and graphitized similarly to the extruded rods.

5.2.3.3. Molded Plugs for Puffing Determination

Two 1-inch diameter plugs were prepared for puffing examination. The batch formula was:

35/100 particles	25 g.
100/pan flour	25 g.
110°C M. P. coal tar Pitch	15.5 g.

The filler materials and the mold and rams were preheated to 200°C. The 110°C M. P. binder pitch was then melted and the heated particles and flour thoroughly mixed by hand with the molten pitch. The mix was immediately transferred to the heated mold and pressed to a pressure of 2500 lbs/in^2 for a one-minute hold time. The mold was then placed in cold water for five minutes, after which the molded plug was pressed out. After the plugs were weighed and measured for bulk density determination, they were packed in 6/3 mesh calcined coke inside an inverted double sagger and baked on a schedule of 25°C/hr. to 400°C followed by 50°C/hr. to 1000°C with a 6-hour hold. The plugs were again weighed and measured for bulk density, machined to $\frac{21}{32}$-inch diameter by 2-inch length, reweighed and measured and set aside for the puffing evaluation.

* pph = parts in one hundred parts of coke flour, thus 32 parts binder pitch per 100 parts coke flour.

5.2.4. X-ray Ratio Sample Preparation

The samples were calcined to 1500°F (816°C) for 30 minutes, followed by 2500°F (1370°C) for 30 minutes, cooled, and then crushed to a minus 325 mesh flour. This material was then examined by standard x-ray diffraction techniques.

5.2.5. Apparatus

1. Thermal Expansion Apparatus

The CTE was determined by measuring the linear expansion of the graphite sample when the temperature was raised from 30 to 100°C. The measurement was made parallel to the direction of preferred orientation in the case of the extruded sample, i. e., with-grain (w. g.) and both parallel and perpendicular to the molding direction in the 3-inch plug.

In the CTE apparatus, a $\frac{3}{8}$- by $\frac{1}{4}$ - by 3-inch sample was clamped in a jig mounted in an Invar bar base. The difference in expansion between the sample and the Invar standard was measured by means of an optical lever.

2. Flexural Strength Apparatus

Flexural strength was measured using a Wiedeman-Baldwin testing machine capable of regulated rates of loading and equipped with a variable range load indicator. The samples tested were rectangular beams, one-half inch in width and thickness, and $2\frac{1}{2}$ inches to 3 inches in length. Single point loading was used. The value for flexural strength was then calculated according to the following formula:

$$R = \frac{3\,PL}{2\,WT^2}$$

where
R = Flexural strength (lbs/in^2)
P = Maximum load (lbs.)
L = Lower span (inches)
W = Average width (inches)
T = Average thickness (inches)

3. Electrical Resistivity Apparatus

Specific electrical resistance is the resistance of a material one square cm in cross section and one cm in length and is expressed as 10^{-4} ohm-cm. The resistance measurements were made at room temperature on the same type of samples as listed under the CTE determination. A sample is placed on two contact points having a 3 centimeter span. The two terminals of a power supply of constant voltage are applied to the end faces of the sample. The apparatus contains a built-in Wheatstone bridge and the resistance of the bridge is adjusted to match the voltage drop be-

tween the two contact points. The resistance of the sample is read directly. Using the dimensions of the rectangular sample, the specific resistance can be calculated from the formula $P = \frac{RA}{L}$, where P is the specific resistance, R is the resistance in ohms, A is cross sectional area in cm^2, and L is the length in CM.

4. Puffing Apparatus

The percentage shrinkage or puffing was taken directly from a graphic representation of the length change of 1-inch molded plugs during heating from 1100 to 3000°C. The basic equipment included a 3-inch I. D. graphite tube furnace, a graphite dilatometer assembly and an optical pyrometer. The dilatometer assembly consisted of a graphite tube, in which the sample was placed, and a graphite follower rod which acted as an extension of the sample and moved with the sample in shrinkage or expansion. The follower rod pressed against a 0.0001-inch graduated dial gauge which was attached to a water cooled head assembly. During the heating cycle an inert gas was passed through the furnace to prevent oxidation. The furnace was adjusted to maintain a 15°C/min. temperature rise with a 30 minute hold at 3000°C. The time, temperature and dial readings were recorded every five minutes during the rise and hold periods. The results were recorded as per cent length change versus temperature from 1100 to 3000°C. The total length change was determined by micrometer measurements on the plug at room temperature before and after heating to 3000°C.

5.3. Tabulation of Results - Coking Phase

Tables 6, 7 and 8 are condensed from the test data compiled in Appendix 9.2. The experiments are grouped in order of increasing thermal cracking severity for each fresh feed material and list only those operating parameters which are considered to be significant, i.e., time in furnace (SV), temperature (furnace outlet and profile), and pressure (coke drum).

The with-grain CTE values were determined on duplicate samples at the Advanced Materials Laboratory, Lawrenceburg, Tennessee, and the Carbon Products Division Research Center, at Parma, Ohio. Identical methods as outlined in Section 6.2.6 were used. The x-ray ratio was determined at the Marathon Oil Research Center, Littleton, Colorado and is the ratio of peak height to peak width at half-height of the 002 line.

5.4. Data Evaluation

The data of Tables 6, 7 and 8 demonstrate that the parameters of the coking process have not yet been varied sufficiently to produce the changes in coke properties that were found in the batch coking experiments (Section 3).

Table 6. Results of Coking Vacuum Residuum

Cycle No.	Furnace Outlet °F	Furnace Profile No.	Drum Pressure lbs/in²	Combined Feed b/d	Space Velocity hr.-1	Steam in Transfer Line	Coke CTE 10^{-6}/°C AML	Coke CTE 10^{-6}/°C PRC[1]	Coke X-ray Ratio H/β MORC[a]	Yields - Weight Per Cent Coke By Outage	Coke By Difference	Gas Oil	Gasoline	Gas
Recycle Ratio ≅ 0.2														
26S	930	B[1]	50	150	13	yes	-	-	-	29.7	16.8	30.6	43.9	8.7
27N	930	B[1]	50	150	13	yes	1.90	2.44	19.4	28.5	10.0	36.4	42.7	10.9
27S	930	B[1]	50	150	13	yes	1.27	1.47	26.4	32.4	23.0	26.0	41.5	9.5
28N	930	B[1]	50	150	13	yes	2.27	2.50	17.8	27.5	19.5	25.9	43.6	11.1
30S	930	B[1]	50	150	13	no	1.55	1.79	20.8	28.9	44.3	14.9	29.0	11.8
Recycle Ratio ≅ 1.0														
6N	935	B	50	201	18	no	1.72	1.79	19.5	34.3	22.1	30.6	35.1	12.1
8S	935	B	100	201	18	no	1.55	1.73	22.9	33.5	26.7	22.7	38.5	12.1
10S	935	B	50	101	8	no	1.57	1.80	22.8	32.3	37.3	26.6	20.7	15.4
9S	935	B	100	101	8	no	1.42	1.56	22.9	32.6	47.2	6.7	33.6	12.5
11S	950	C	50	201	17	no	1.38	1.68	22.8	30.6	32.8	27.5	25.9	13.8
12S	950	C	100	101	9	no	1.67	1.66	22.5	27.3	35.0	18.1	28.2	18.7
40S	935	H	50	100	7	no	1.30	1.43	24.1	30.5	35.6	10.8	36.6	17.0
41S	945	J	50	125	10	no	1.27	1.42	26.1	28.4	36.8	20.6	22.9	19.7
31S	935	B	250	200	18	no	1.36	1.64	22.2	41.1	44.8	15.1	20.8	19.3
32S	935	B	350	200	18	no	1.22	1.68	23.3	39.6	47.0	1.8	29.4	21.8

(1) Parma Research Center, National Carbon Company, Parma, Ohio
(a) X-ray Ratio, Marathon Oil Research Center, Littleton, Colorado

Table 7. Results of Coking Slurry Oil

Cycle No.	Furnace Outlet °F	Furnace Profile No.	Drum Pressure lbs/in²	Combined Feed b/d	Space Velocity hr.-1	Coke CTE 10^{-6}/°C AML	Coke CTE 10^{-6}/°C PRC[1]	Coke X-ray Ratio H/β MORC[a]	Yields - Weight Per Cent Coke By Outage	Coke By Difference	Gas Oil	Gasoline	Gas
Recycle Ratio ≅ 1.0													
16S	935	B	50	201	27	0.42	0.30	35.2	44.3	40.6	32.8	13.2	13.4
15S	935	B	100	201	25	0.44	0.29	31.6	51.6	43.2	29.0	14.1	13.7
13S	950	C	50	201	23	0.19	0.51	33.9	47.2	45.9	29.7	8.6	15.8
17S	950	C	50	201	24	0.41	0.30	31.8	46.3	44.3	31.4	11.1	13.2
18N	950	C	50	201	24	0.41	0.34	34.4	43.6	44.3	29.6	12.3	13.6
18S	950	C	50	201	24	0.24	0.34	31.7	46.5	41.9	29.0	11.4	17.6
14S	950	C	100	200	23	0.40	0.31	35.6	55.5	55.4	21.8	8.7	14.1
19S	965	C	100	202	23	0.37	0.37	29.6	64.0	53.5	9.6	17.2	19.9
38N	935	E	50	150	11	0.21	0.38	34.0	49.2	55.1	16.9	8.6	19.4
39N	935	F	50	150	9	0.24	0.41	32.4	55.9	57.5	10.5	13.7	18.3
22N	950	C	150	150	17	0.21	0.26	30.1	57.8	50.3	13.8	16.2	19.7
23S	950	C	260	150	17	0.36	0.49	--	71.3	61.2	0.0	14.8	24.0
25S	935	B	350	150	18	0.20	0.44	30.6	78.2	63.4	0.0	15.2	21.4
24S	950	C	350	150	16	0.22	0.37	30.7	71.5	62.7	0.0	13.7	23.6
Recycle Ratio ≅ 3.2													
20S	950	C	100	201	23	0.27	0.34	34.1	71.4	44.5	0.0	33.0	22.5

(1) Parma Research Center, National Carbon Company, Parma, Ohio
(a) X-ray Ratio, Marathon Oil Research Center, Littleton, Colorado

Table 8. Results of Coking Thermal Tar (Marathon Oil)

Cycle No.	Furnace Outlet °F	Furnace Profile No.	Drum Pressure lbs/in^2	Combined Feed Rate b/d	Space Velocity hr.$^{-1}$	Coke: CTE 10^{-6}/°C AML	Coke: CTE 10^{-6}/°C PRC[1]	Coke: X-ray Ratio H/β MORC[2]	Yields - Weight Per Cent: Coke By Outage	Coke By Difference	Gas Oil	Gasoline	Gas
Results of Coking Thermal Tar (Marathon Oil)													
Recycle Ratio ≅ 1.0													
42S	935	B	50	150	17	0.37	0.43	35.1	64.0	59.9	20.8	1.9	18.3
43S	935	E	50	150	11	0.39	0.54	42.1	68.0	59.9	21.1	0.9	18.1
44S	935	F	50	124	8	0.27	0.41	40.7	61.0	59.6	19.9	0.0	20.5
Results of Coking Thermal Tar (Pure Oil)													
Recycle Ratio ≅ 1.0													
33S	950	C	100	150	18	0.25	0.58	35.4	45.5	58.1	21.2	4.2	16.5
Recycle Ratio ≅ 0.6													
34S	950	C	350	125	15	0.34	0.47	36.0	59.6	80.6	0.0	3.0	16.4

(1) Parma Research Center, National Carbon Company, Parma, Ohio
(2) X-ray Ratio, Marathon Oil Research Center, Littleton, Colorado

In particular, the properties of the cokes from the charge stocks from catalytic or thermal cracking processes (Tables 7 and 8) are insensitive to the variations in coking parameters so far imposed. The most extensive data are available for a slurry oil charge stock (Table 7). These data are a positive indication that exacting control of factors such as furnace outlet temperature, space velocity, etc., are not needed for reproducibility of coke properties. Coke yields, on the other hand, are substantially increased at the higher drum pressures or higher recycle ratios. Very little foam was encountered in coking of the cracked feed stocks, permitting an excellent utilization of drum volume.

With a vacuum residuum charge stock, there is a trend toward lower CTE's at higher operating drum pressure, evidenced by the data for cycles 10S and 32S in Table 6. There is also a correlation between lower CTE's and an increase in cracking severity in the furnaces. A pronounced increase in gas production is noted in those experiments which involve greater cracking severity and higher drum pressures. Increased coke yields, however, are evident only at the highest drum pressures, a condition which supports the results obtained from batch coking experiments.

A significant effect was obtained with vacuum residuum feed stock in experiments where superheated steam was introduced into the hot oil stream between the furnaces and the coke drum. In some cases the CTE was increased by 0.5 to 0.7 (10^{-6}/°C). The effect of steam may be to strip from the feed entering the drum those materials which, if allowed to remain in the drum, would crack to coke and result in lowering the CTE of the combined mass. Admittedly this explanation is not consistent with the coke and gas oil yields. These should be both lower and higher respectively when compared with the control experiment (no steam). The effect might be explained by the turbulent condition (higher vapor velocity) existing at the time of spatial structure formation. Coke from the steam injection experiments not only has a higher CTE but is more isotropic, and as such should yield a graphite of desired properties for certain missile applications where a coating must be employed for oxidation resistance.

The determination of coke yields presented in Tables 6 to 8 was previously discussed in Section 5.3. In a number of experiments the gasoline product contained entrained water which came from either the steam injected in certain experiments, or from a steam valve leak into the vapor and/or feed lines. The gasoline accumulator did not efficiently separate this excess water with the result that gasoline yields were higher than the true value, and coke yields (by difference) were consequently lower than the true value.* Since this condition existed for an unknown period of time, coke yields were also calculated using drum outage (coke volume) and a bulk density averaged from Table 16, Appendix II.

Puffing is an irreversible volume expansion of a baked carbon article during graphitization. In some cases puffing may fracture the piece. Such puffing is due to decomposition and evolution of sulfur from carbon-sulfur complexes in the coke. Generally speaking, there is no definite relationship between the amount of puffing and sulfur content. All cokes tested in these experiments, with the exception of Pure Oil thermal tar, had similar sulfur contents. The data in Table 21, Appendix II, indicates that with slurry oil and thermal tar (Marathon Oil) more puffing occurs and it occurs over a wider range of temperature than with the virgin stock (vacuum residuum). The exceptionally low sulfur coke from Pure Oil thermal tar gave no indication of puffing. In all cases, however, the extent of puffing was not considered to be great enough to interfere with fabrication of large graphite shapes.

* The construction of the separator has since been modified to avoid this difficulty.

6. UNIT PERFORMANCE

6.1. Engineering and Process Design

The experimental unit has performed within the limits included in the original design.

Various small changes were made as the program progressed to simplify operations, provide additional and more reliable data, prevent costly shutdowns due to inclement weather, and to provide a more satisfactory environment and a higher degree of safety for the operators.

The changes were as follows:

1) Simplification of operations:

   a) Automatic temperature controllers were installed on the outlets to the first and third furnace, replacing manual controls.

   b) A heat exchanger (600 pound steam) was installed on the fresh feed line in order to decrease the thermal load on the furnaces.

2) Provision of additional and more reliable data:

   a) A thermocouple was installed in the transfer line leading to the coke drum for measurement of ΔT between bottom and top of drum.

   b) The thermocouple on the vapor line at the top of the coke drum was repositioned for better temperature measurement.

   c) Positive displacement meters (PDM) were installed at the fresh feed inlet and product lines for direct measurement of volume flows rather than using orifice meter charts which must be interpolated. This procedure was necessary for the fresh feed flow when it was found that the orifice meter did not correctly read the volume flow of the high viscosity vacuum residuum. The control of flow was subsequently based on the positive displacement meter readings.

   d) The feed pipe into the coke drum was extended with a diffuser head. A head of coke was built into the bottom cover, and the top and bottom covers were insulated to prevent heat losses.

3) Prevention of costly shutdowns:

a) Check valves were installed in the steam inlet to the furnaces to prevent backing up of feed into the steam coils.

b) Steam tracing and more insulation were added to pipe lines to prevent freezing during subzero weather.

c) An automatic gas shutoff on the steam superheater was installed which would operate in the event of refinery steam loss.

4) Provision of more satisfactory environment and safety precautions:

a) The decoking platform was enclosed for weather protection of the decoking crew.

b) An escape line was installed as an emergency exit from the drilling deck.

The major engineering limitations of this unit involve the transfer lines between furnaces and those lines running from the furnaces to coke drum. Examination during periodic shutdowns revealed that coke deposits had formed in transfer lines. The furnace coils are $\frac{1}{2}$-I. D. schedule 80 pipe for the Nos. 1 and 2 furnaces (1BA and 1BB) and $\frac{3}{4}$-inch I. D. schedule 80 pipe for Nos. 3 and 4 furnaces (2BA and 2BB), respectively. The transfer lines range from $1\frac{1}{2}$- to $2\frac{1}{2}$-inch pipe (schedule 80) up to 4-inch pipe near the coker drums. The linear velocity of the feed stocks decreases by a factor of eight in traveling through this system.

In areas of critical temperature, the linear velocity drops, resulting in coke laydown. A typical x-ray picture of the coke deposit experienced is shown in Figure 4.

6.2. Operation Techniques

Early in the program, the fractionator went through a slump immediately after the switch from one coke drum to the other, resulting in loss of level in the fractionator bottoms (feed to furnace) and in the gas oil tray supplying reflux liquid. Coking in the new, relatively cold drum had not proceeded far enough to supply cracked products for level control.

Various corrections were attempted, such as steam injection before switch to build up a high level and steam blowing after switch for various lengths of time in order to maintain a high level. During the latter tests, the effect of steam on CTE was discovered. The eventual solution of the problem was a simple one which served a dual purpose. After the switch, the vapor lines of the two drums were interconnected and the old

drum (filled with coke) was blown with superheated steam for a period of one to two hours before the introduction of cooling steam which was then routed to blowdown. The superheated steam stripped the coke of absorbed gas oil which was then used for level control in the fractionator. The steam also had the effect of finishing coking those last increments of charge stock which did not have sufficient residence time in the old drum to coke by incipient heat. Theoretically, the steam treatment should reduce variation of coke quality from top to bottom of the drum by decreasing the volatile content of the top portion.

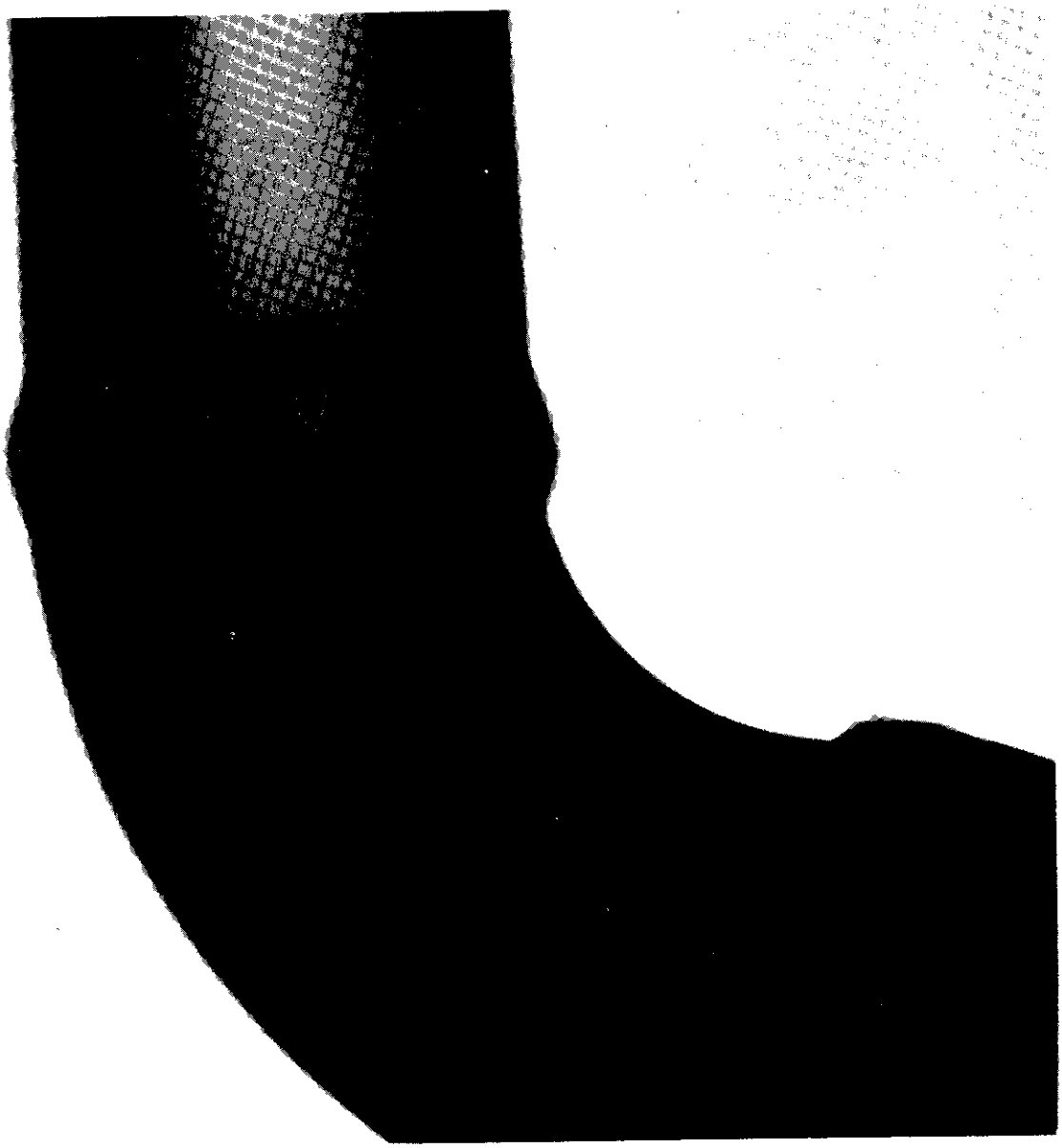

Figure 4. Coke Deposit in Transfer Line

7. CONCLUSIONS

1. Cokes have been prepared showing a high degree of reproducibility of properties from run to run, indicative of the degree of control attained during the coking process.

2. The properties of cokes prepared from charge stocks derived from catalytic or thermal cracking operations are relatively insensitive to moderate changes in furnace temperature profile, furnace outlet temperature, furnace feed rate and coke drum pressure. Thus, for these charge stocks, precise control of these parameters are not required to obtain coke of reproducible properties.

3. Virgin residue charge stock is more sensitive to changes in these parameters of the coking process and a closer control is required to attain reproducible coke properties.

4. Steam injection into the transfer line in conjunction with a virgin residue charge stock produces coke leading to graphite of higher coefficient of thermal expansion and more nearly isotropic properties, as required for use as substrate for coatings.

5. Coke prepared from a low-sulfur charge stock from thermal cracking shows no puffing when processed to graphite.

6. Extremely high yields of dense, low CTE cokes (in excess of 70 weight per cent of the feed) are obtained at high drum pressures (250 to 350 lbs/in^2 gauge), using slurry oil as the feed stock. Coke yields in excess of 60 weight per cent are obtained at only 50 lbs/in^2 gauge drum pressure using a thermally cracked slurry oil, i.e., thermal tar.

7. Two lots of coke, representing the widest range of properties so far obtained, are being processed to 18-inch diameter graphite for evaluation of the influence of coke variations on final graphite properties.

8. The work has indicated the direction of future effort toward the objective of reproducibly preparing raw materials with the wide range of properties required for the various aerospace applications of graphite.

8. RECOMMENDATIONS

8.1. Unit Revisions

Only two major revisions are contemplated for the unit. They are:

1) Replacement of transfer lines between furnaces 1BA-1BB and 1BB-2BA with $\frac{1}{2}$-inch pipe, and between 2BA-2BB and 2BB-switch valve with $\frac{3}{4}$-inch pipe. Concurrently, some 90° bends will be eliminated with sweeping bends, and the large thermocouples will be replaced with a miniature type.

2) Installation of a pipe line to permit introduction of hydrogen into the fresh feed in the furnace coils. This refinement will serve the dual purpose of (a) possible upgrading of the virgin stock for lowering CTE and (b) hydrocracking of thermal tars for exploratory work on petroleum pitches.

8.2. Future Efforts

The results obtained to date (Tables 6, 7 and 8) have indicated the directions for further work, specifically to extend the range of parameters of time and temperature either in the furnace or in the coke drum.

To gain information concerning the furnace effects and to separate the coke drum effects, the next phase of this program will be to bypass the coke drum in order to operate the unit as a thermal cracker using the same furnace profiles as in the coking phase but with various back pressures. The flow diagram for thermal cracking is shown in Figure 5. These experiments will indicate the amount of furnace cracking that can be achieved without coke formation in the furnaces. These tests will be carried out on a recycle basis, the same as in coking. Thermal tar products will be characterized by gravity, distillation, viscosity, silica gel chromatography [2] and nuclear magnetic resonance. [4] In addition, the thermal tar will be coked in the laboratory, calcined, formed, graphitized and tested using the techniques as outlined in Section 5.2.2.

The preliminary schedule is shown in Table 9 and the results will be reported at a future date. The experiments are arranged to give the maximum degree of safety in avoiding premature shutdown. The effect of the furnace back pressure is to suppress vaporization and prevent segregation of liquid and vapor phases. The mixtures of oil and vapor at cracking conditions tend to deposit coke because the liquid segregates and collects on the hot walls of the furnace tubes where it is cracked more extensively than is the main body of the feed. High pressure produces a vapor having density similar to the liquid phase. The critical pressure of most hydrocarbons is less than 400 lbs/in^2. The schedule in Table 9 is designed to investigate the effect of the time temperature complex.

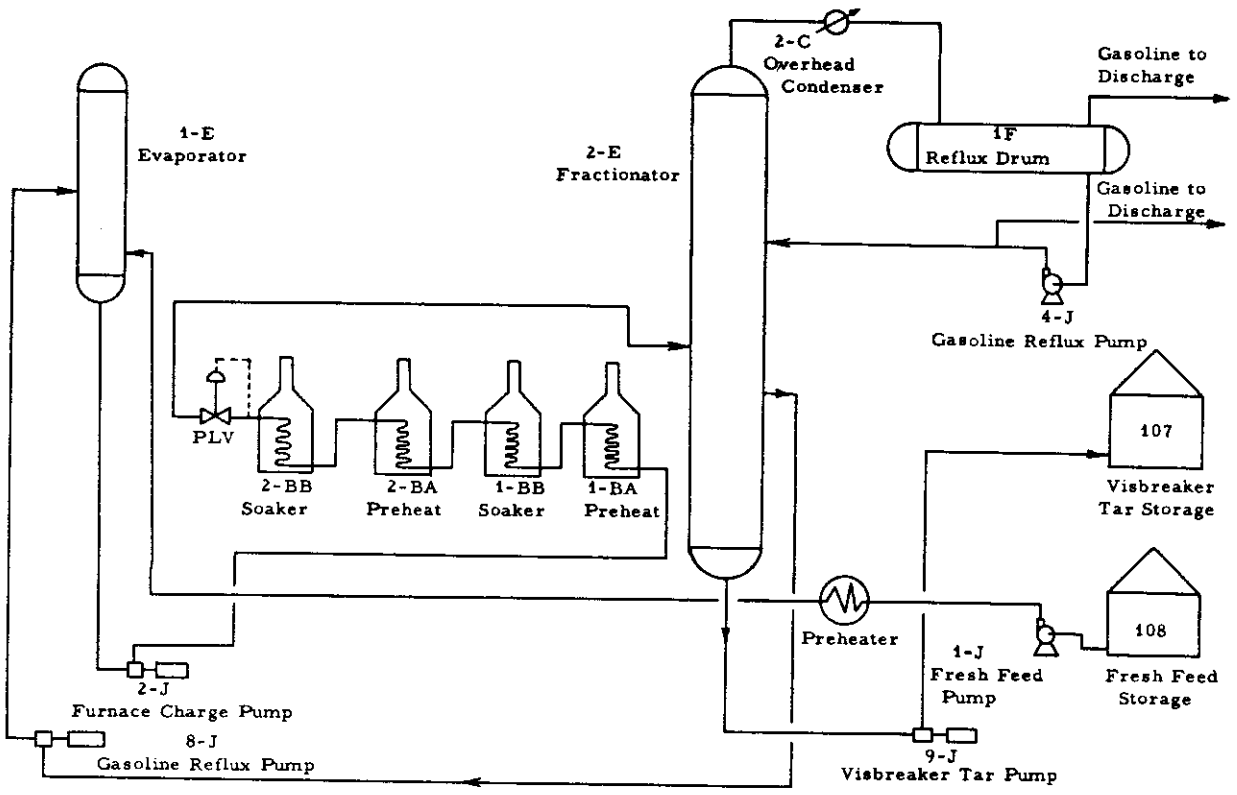

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Figure 5. Flow Diagram of Experimental Unit When Operated as a Thermal Cracker

If pressure drops indicate coke deposition in the furnace tubes, the experiment will be discontinued.

Beyond this schedule and based on small scale laboratory results, the effort will lie toward producing:

1) A high CTE, isotropic coke based on variations of charge stocks.

2) Petroleum pitches by thermal (and possibly hydro)cracking of aromatic stocks.

Table 9. Schedule of Thermal Cracking Experiments

Experiment No.	Feed Stock	Furnace[1] Profile No.	Furnace Outlet Pressure	Combined Feed b/d	Maximum[2] Recycle Ratio
36	Coker Gas Oil	B	50	100	Max.
39	Coker Gas Oil	B	150	150	Max.
40	Coker Gas Oil	B	400	150	Max.
37	Vacuum Residuum	B	50	100	Max.
38	Vacuum Residuum	B	400	100	Max.
41	Vacuum Residuum	B	150	150	Max.
42	Vacuum Residuum	H	150	150	Max.
44	Vacuum Residuum	L	50	100	Max.
45	Vacuum Residuum	M	50	100	Max.
46	Vacuum Residuum	N	50	100	Max.

(1)

Furnace Profiles	B	H	L	M	N
1BA outlet of	635	680	725	750	775
1BB outlet of	707	781	850	875	900
2BA outlet of	817	875	850	875	900
2BB outlet of	935	935	850	875	900

(2) Maintain combined feed rate with recycle material

9. LIST OF REFERENCES

1. WADD Technical Report 61-72, Volume X, "Thermal Reactivity of Aromatic Hydrocarbons," by I. C. Lewis and T. Edstrom.

2. Eby, L. T., Anal. Chem., Volume 25, No. 7, July 1953.

3. Kurtz, S. S., et al, Anal. Chem., Volume 30, No. 7, July 1958.

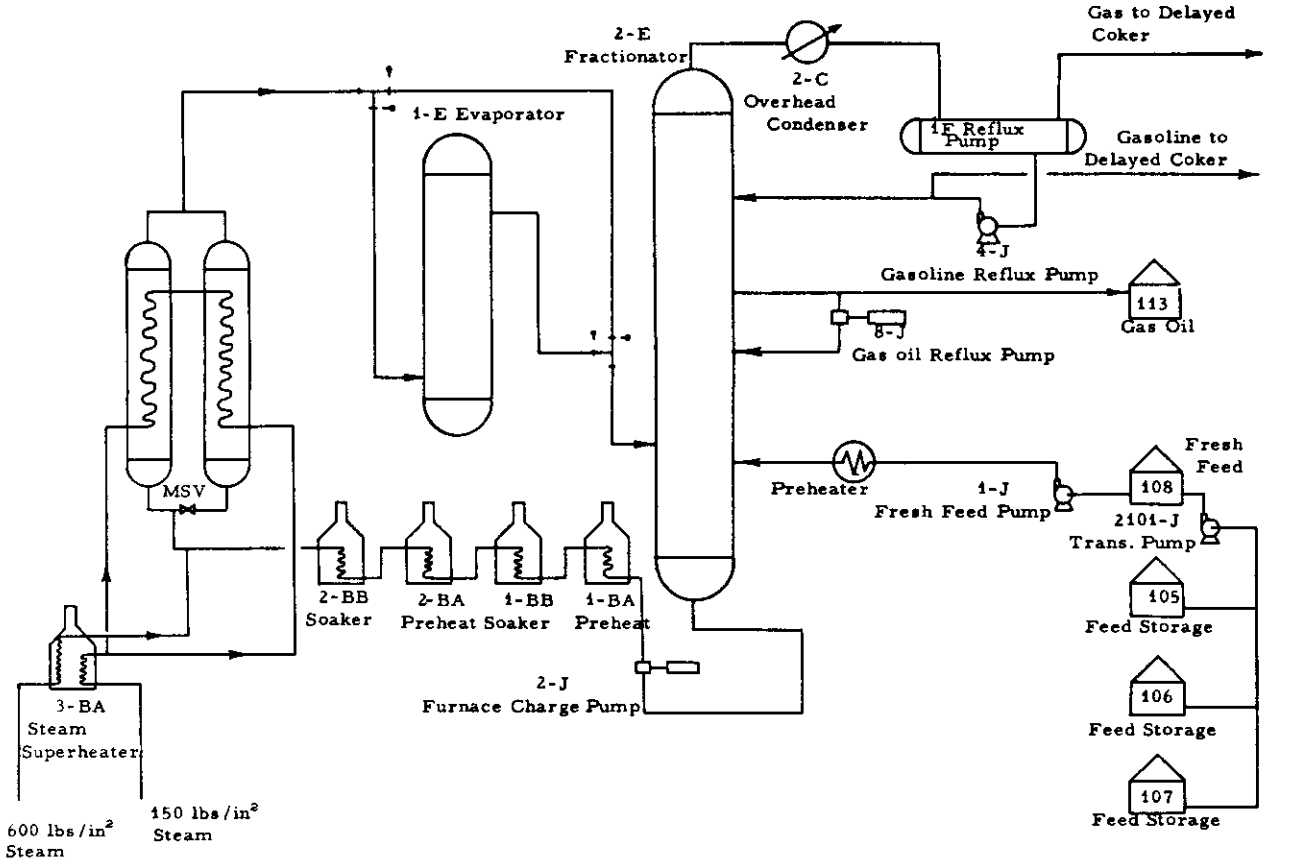
4. Willisms, R. B., ASTM Spec., Publication No. 224.

APPENDIX I

ENGINEERING CONCEPT

I. 1. Process Design Description Introduction

The $3\frac{1}{2}$ ton-per-day experimental coking unit was designed to process a wide variety of charge stocks, such as virgin residuum, thermal tar and slurry oil, at through-put rates ranging from 50 to 350 barrels per day. The unit was designed for a coke drum outlet temperature and pressure of 950°F and 400 lbs/in^2, respectively, and consists of two coke drums, four furnaces, evaporator tower, fractionator tower, steam superheater and attendant equipment. Coke is removed from the drums by means of hydraulic decoking equipment. In addition to the coking operation, the unit can also operate as a gas oil cracker and residuum visbreaker. The flow diagram for the unit operated as a delayed coker is shown in Figure 6.



L-435

Figure 6. Flow Diagram of Experimental Unit When Operated as a Delayed Coker

I. 1. 1. Low Pressure Coking Process Flow (0-100 lbs/in^2 gauge)

The charge stock, i. e., fresh feed, is pumped from the storage tank through a preheater into the bottom of the fractionator where the fresh feed is diluted with recycle to obtain the desired mixture of combined feed. The combined feed is then pumped through the heaters into the bottom of the coke drum where coking occurs. The vapors from the top of the coke drum are returned to the bottom of the fractionator, where the heavier vapors are condensed as recycle and the lighter vapors pass up through the tower and are drawn off as gas oil product, gasoline product and gas. The gas oil goes to a storage tank, where it is available to be returned to the unit if needed or pumped from the tank into the gas oil product line from the commercial delayed coking unit. The gasoline produced is used for reflux with the excess pumped into the gasoline reflux systems. Gas from the experimental unit is fed into the refinery fuel system, if the pressure on the fractionator is high enough; otherwise, the gas is directed into the refinery flare system.

The coke made in each drum is steam stripped, water cooled and then hydraulically decoked from the drum. If the drum is a sample drum, the coke is hauled to a concrete pad, spread out and sampled. If the drum is not a sample drum, the coke is hauled to the field and stored.

I. 1. 2. High Pressure Coking Process Flow (100-400 lbs/in^2 gauge)

When coking at high pressure, an evaporator, a pressure letdown valve and liquid level control are put into service between the coke drum and the fractionator. This procedure is necessary because the fractionator has an upper operating limit of 95 lbs/in^2 gauge whereas the coke drums are operable to 400 lbs/in^2 gauge. The evaporator provides an initial separation of gaseous and liquid products before passage through the pressure letdown valve and liquid level control to the fractionator. Without the evaporator, control of the drum pressure would be difficult, and coking of the pressure letdown valves would be increased.

I. 1. 3. Furnaces

The unit is equipped with four coil furnaces which may be used in pairs (either singly or in parallel) or in series. One pair of furnaces is designed for a maximum feed rate of 120 barrels per day. The second pair of furnaces is designed for a maximum feed rate of 197 barrels per day. The first furnace in each pair is used as a preheater. The second furnace is used as a soaker. All four furnaces have been designed for a maximum coil outlet temperature of 1000°F at 1000 lbs/in^2 gauge and are operated on automatic temperature control. Each preheater furnace has a 600 pound steam inlet which may be used to increase vapor velocity through the furnaces. The amount of 600 pound steam used can be measured with a rotameter.

I. 1.4. Coke Drums

Two 4-feet I. D. by 30-feet height coke drums are designed for a maximum temperature of 950°F at 400 lbs/in^2 gauge with an alternate temperature of 1000°F at 125 lbs/in^2 gauge. The alternate conditions are provided for use during the final stages of the coking operations to permit drying out the coke bed if necessary. Since the coke drum surface area is large compared with the volume of the oil flow, the coke drums are wrapped with steam coils to minimize heat loss. There are four sections of coils around each drum. Each section has its own control valve to regulate flow of steam through the coils. A block valve is installed on the common outlet to hold steam pressure on the coils.

I. 1.5. Fractionator

The fractionator is used as a mixing vessel in addition to being used to separate the overhead gas, gasoline and gas oil products. During the coking operation, the fresh feed is pumped into the bottom section of the fractionator. The desired amount of gas oil is condensed or blended with the fresh feed to provide the required amount of recycle in the combined feed. During the visbreaker operation, the visbreaker tar collects in the bottom of the fractionator where this material is steam stripped before being pumped to storage.

I. 1. 6. Steam Superheater

The steam superheater provides superheated steam for preheating the coke drums and for holding temperature of the coke drum during operations. An extra high pressure coil is included in the superheater to provide high pressure steam for steam testing the coke drums and to allow steam injection into the coke drums and transfer lines at high pressure.

I. 1. 7. Evaporator

The evaporator tower is used when coking at pressures of 95 lbs/in^2 gauge or higher. The purpose of this tower is to provide clean vapors to the fractionator by condensing out the heavy ends of the coke drum vapors. The vapors leaving the evaporator tower pass through a pressure controller before going to the fractionator tower. Without the use of the evaporator tower, the coke drum vapors would pass directly through the pressure control valve before flowing to the fractionator tower. At the operating conditions contemplated in the process design, the unit would soon become inoperable due to the formation of coke in the pressure control valve. Gas oil from the fractionator can be used as reflux to the evaporator if the coke drum vapors need to be cooled or quenched. During the thermal cracking operation, the fresh feed is pumped into the evaporator tower together with the required amount of gas oil to make the correct mixture of combined feed. The furnace feed pump transfers this combined charge from the evaporator to the heaters.

I.1.8. Antifoam Injection

Provisions have been made for the injection of antifoam solution into the coke drums to reduce foaming. Antifoam solution may also be injected into the suction of the combined feed pump to reduce foaming in the charge.

I.1.9. Foam Detectors

Each coke drum is equipped with a foam indicating device (Ohmart gauge) located nine feet from the top of the drum. This device detects passage of the foam front and alerts the operators to inject antifoam for prevention of drum overflow.

I.1.10. Hand Bypasses on Pumps

Due to the wide variation in flow rates, hand bypasses are installed on all pumps. This precaution is necessary because the reciprocating pumps will not run slowly enough to handle the low flow rates. Hand bypasses permit the pumps to run a little faster, thereby giving better control.

I.1.11. Startup Lines

A gas oil startup line is installed in the unit with a level control valve. During startup the necessary amount of gas oil is pumped into the fractionator bottom. When the gas oil level has increased until it reaches the desired height, the level control valve shuts off on the incoming gas oil.

A connection is also made so that hot gas oil from the commercial delayed coking unit can be put directly into the fractionator on the gas oil pan. It is desirable that some oil be available for quench during the thermal cracking startup before quench oil is made in the unit. Otherwise, vapors from the heater outlet would go to the fractionator at a temperature which would cause coking in the vapor line connecting the heater outlet to the factionator.

I.1.12 Relief System

Pressure relief valves are incorporated into the outlet of each heater. Connections are also made so that each heater can be routed through relief valves to the blowdown system. Each pump has a valve that relieves from the discharge side to the suction side if the set pressure is exceeded. All vessels also have relief valves.

I.2. Mechanical Design

The general climatic and seismatic data used in the design of the experimental coker at Robinson, Illinois included the following:

Winter minimum temperature	-20°F
Summer maximum temperature	110°F
Maximum wind velocity	80 MPH
Maximum rainfall	2.5 in/hr.
Earthquake zone	2
Frost line	33 inches
Soil bearing	4000 lbs/sq. ft.

I.2.1. Tankage

To provide charge and product storage for the experimental coker, the following existing refinery storage tanks were selected and moved to the construction site.

Tank No.		
105	Clarified Slurry	1250 bbl.
106	Thermal Residuum	1250 bbl.
107	Vacuum Column Bottoms	1250 bbl.
108	Mixed Charge Stock	1240 bbl.
113	Gas Oil Product	497 bbl.

A sand pad 75 feet wide by 175 feet long was prepared for these tanks.

I.2.2. Heaters

Four process heaters were selected, two each in a size. To permit any desired combination of series or parallel operation, all heater coils were designed for 1000 lbs/in^2 operating pressure at 1000°F. Because of the possibility of coke formation, this design specification was carried through the interconnecting process piping up to and including the switch valve. Test pressure in this system was to be 2000 lbs/in^2.

Because of the relatively short life of the heaters (ten years design), maximum corrosion protection was not thought to be necessary. A 5 per cent Cr - 0.5 per cent Mo tube material and castable heater lining were selected. The coils were designed as a continuous spiral of 534 feet in the two smaller heaters, and 550 feet in the two larger heaters. Tube diameters were 0.840 O.D. by 0.147 wall and 1.05 O.D. by 0.154 wall. Because of its relatively small size, each heater was supplied with its individual self-supporting stack. The heater shells were designed for easy removal of the breeching and stack to permit changing of the spiral tubes through the top of the heaters. An adjustable reradiation baffle was placed in the breeching of each heater to increase the heat transfer rate and permit firing and draft control. John Zinc burners were selected to fire a 1000 BTU/cu. ft. fuel gas from the floor.

A fifth heater was provided to supply superheated steam at 150 lbs/in^2 and 1200°F to the coils around each coke drum. To maintain adiabatic conditions, an estimated duty of 1,930,000 BTU/hr. was required. Because of the temperature profile in this heater, 4-inch O.D.

tube material was divided into three sections, beginning with A-53 carbon steel, then 2 per cent Cr - 1 per cent Mo and finally 316 stainless steel. . An additional 1-inch O. D. -316 S. S. tube was provided in the steam superheater to supply process injection steam at 600 lbs/in^2 and 1200°F.

I.2.3. Coke Drums

To accommodate a variety of operating conditions, the two 4-feet O. D. by 30-feet long coke drums were designed for 400 lbs/in^2 at 950°F and 125 lbs/in^2 at 1000°F. Because of the relatively short life expectancy of the unit, no alloy lining was provided for the drums. Shell thickness was 1 $\frac{3}{16}$ inches allowing $\frac{1}{4}$-inch corrosion protection. Shell and head material for the drums was 1 per cent Cr - 0.5 per cent Mo A387 steel. A 48-inch R. J. flanged opening was provided at top and bottom of both drums.

I. 2.4. Fractionator

The original Kellogg design specification called for a fractionator of 2 feet and 8 inches I. D. by 40 feet long with a design pressure of 105 lbs/in^2. Instead, an existing refinery tower 2 feet and 8 inches I. D. by 50 feet long with a design pressure of 368 lbs/in^2 was selected.

Eleven new 11 to 13 per cent Cr bubble cap trays were selected for this column. To permit a wide variety of operating conditions, blanking equipment was provided to blind off portions of the tray deck for reduced flow. Six 11 to 13 per cent Cr segmental weir flow baffles were selected for the lower section of the tower.

I. 2. 5. Evaporator

A 2-feet I. D. by 10 feet and 6 inches length evaporator designed for 400 lbs/in^2 at 760°F was purchased from Terre Haute Boiler Works. Head flanges were designed for the full diameter of the tower to accept three 11 to 13 per cent Cr slip in bubble cap trays. Support for the vessel consisted of a 2-feet I. D. skirt, 14 feet in length.

I. 2. 6. Reflux Drum

Kellogg proposed a reflux drum 2-feet O. D. by 10 feet in length designed for 105 lbs/in^2 and 110°F. As an alternate, an existing refinery vessel, 2-feet O. D. by 14 feet in length, designed for operation up to 120 lbs/in^2 and 150°F, was selected.

I. 2. 7. Pumps

Because of the low flow, high head requirements of most of the streams, positive displacement pumps were chosen. The only exception was a centrifugal pump to move the fractionator gasoline product to the delayed coker. To obtain a controlled uniform flow for fresh feed, a

screw pump with automatic speed changes was installed.

For furnace charge, a 1200 lbs/in^2 design reciprocating pump was selected to operate in conjunction with a pneumatic pulsation chamber in order to reduce flow surges.

Because of the high viscosity of the sundry feeds, a gear pump was installed in the tank area for feed transfer.

A vertical sump pump was provided for decoking water return from the pit to the sluiceway.

To provide condensate to desuperheat the 1200°F steam leaving the coke drum coils, a Westco turbine pump was installed.

A Hills-McCanna chemical injection pump was installed to handle antifoam.

I. 2. 8. Piping

To permit a maximum process operation of 1000°F at 1000 lbs/in^2, 1500 pound ASA flanges with 1.5 per cent Cr pipe were used in the hot oil system. To obtain high temperature strength for the 1200°F superheated steam system, 316 stainless steel piping was used. All other utility and process piping was carbon steel using ASA flange design.

I. 2. 9. Insulation

Since the highest temperature encountered in the experimental coker was 1200°F, a preformed calcium silicate insulation was thought to be satisfactory for both lines and vessels. A 4-inch thickness was used on the coke drums to conserve as much heat as practical in order to maintain adiabatic conditions. Quotations indicated aluminum jacketing to be the most economic covering for the towers. Since several piping changes might be made during operation or decoking, a prefabricated, aluminum-jacketed pipe covering was selected. This insulation can be removed and reinstalled when necessary.

I. 2. 10 Utilities

Utilities supplied for the pilot plant consisted of 110 volt, 60 cycle power for lighting and fractional H. P. drivers, and 440 volt power for electric pump drives. Steam at 140 lbs/in^2 was supplied for reciprocating pump drives, steam superheater, and service hoses. Steam was supplied at 600 lbs/in^2 for process injection and relief valve flushing. Treated circulating water at 90°F was provided for process coolers and service water. Air at 90 lbs/in^2 was provided for hoisting, rotation and service air for decoking equipment. Jet water at 1500 lbs/in^2 was provided for the decoking equipment.

I.3. Construction

Arrow Diagram Planning was used for planning and sequencing in the construction of the coker. This procedure enabled the contractor (Marathon Oil Company) to plan the purchase and installation of the major components of the experimental coking unit which would best utilize the time, equipment and manpower available.

Arrow Diagram Planning, an accepted tool in the petroleum industry, permits efficient planning of construction and maintenance work. This method was used for all design and construction. Each job, from preliminary design to completion, is broken down and each step identified. Logic then establishes the order in which the work can be performed and the amount of time required for each job. This information is then fed into a computer in order to obtain a list of the jobs and the order of their performance. The critical jobs are also pointed out by the computer, after which a bar chart can be constructed which may be used as a work sheet to show where the most effort must be concentrated and, later, the progress accomplished.

Construction of the experimental coking unit began at the Marathon Oil Company's Robinson Refinery on March 5, 1962. Four subcontractors were employed by Marathon for the actual construction work. These were as follows:

Company	Work Done
A&K Midwest Insulation Company	Insulating vessels
Johns-Manville Sales Corporation	Insulating piping
Potter Electrical Engineering and Construction Company, Inc.	Electrical work
Sheehan Pipeline Construction Company	All other work

Grading of the area began on March 5, followed by excavating for underground sewer connections, sewer lines, footers for the pumps, exchangers, towers and furnaces. Also, the excavating, forming and pouring the reinforced concrete footers and sluiceway for the coke drum structure were accomplished at the same time. During this time, work was being accomplished in other areas. The purchase of new material or major components of the unit was made as soon as its design was complete.

Surplus equipment at Marathon's Robinson Refinery included the fractionation tower, the reflux drum, overhead condenser, gas oil cooler, visbreaker tar cooler, six pumps and five storage tanks. Reconditioning of these existing pieces of equipment began in January 1962.

The fractionation tower was removed from its existing location to facilitate reconditioning. The tower is 65 feet tall and $2\frac{1}{2}$ feet in diameter. The old trays were of necessity moved, a new bottom installed and two new manways installed. Later the new trays, which had been ordered previously, were installed and complete prime coat of paint applied.

A baffle and various new nozzles were installed on the reflux drum.

The overhead condenser, gas oil cooler and visbreaker tar cooler were dismantled, cleaned and repaired.

The six pumps to be used were renovated at the machine shop. They were completely dismantled, cleaned, repaired, repacked and reassembled for use.

Several items were purchased new from various suppliers, each being selected on the basis of quotation. Quotations were evaluated primarily on the basis of delivery time and cost.

Collins and Rice, consulting engineers from Springfield, Illinois were employed for the purpose of designing the coke drum structure and foundation. Once fabrication and erection drawings were completed, the consultants invited bidders for the selection, with Marathon's approval, of a suitable fabricator for the structural steel. Vincennes Steel Corporation was selected to fabricate and afterwards deliver the structure to the job site. The lower half of the structure was to be delivered complete on April 1, 1962 and the upper half on May 1, 1962 in order to allow the coke drums to be set on the lower half of the structure and all the related piping and instrument work to proceed without waiting for the remaining structure. This procedure also offset the effect of the price increase in steel. The fabricator was able to make the deliveries on schedule.

The coke drums were fabricated by Nooter Corporation at St. Louis, Missouri, delivered to the job site and assembled April 1, 1962.

An unusual feature of the coke drums was the four banks of 1-inch superheater steam coils around the outside of each drum. The coils, installed by Nooter Corporation, were spaced on 4-inch centers. The drums were set in the structure on schedule.

The hydraulic decoking equipment design, fabrication and delivery were accomplished by Powered Equipment, Incorporated, Terre Haute, Indiana. The decoking equipment was partly a new design and partly a miniaturized copy of the existing commercial delayed coking unit at Marathon's Robinson Refinery. The decoking system utilizes the same turbine driven hydraulic water pump as the commercial coking unit. This system also demonstrated the advantage of the large capacity and pressure reserve in handling some of the extremely hard cokes produced. The rotary drilling head for the decoking equipment was secured from the Worthington Corporation.

The furnaces for the experimental coking unit consisted of four process heaters and one steam superheater. The plot plan location of all five heaters nested them together in one corner of the unit area. The foundations for the heaters were put in along with the other foundation work. These heaters were of a vertical, petrochemical type design, with

the burner in the bottom, requiring supports high enough to walk under. These supports were fabricated on the job site and put in place to be ready for the heaters upon delivery.

The evaporator in the experimental coking unit, a small tower to be used during high pressure operation, was purchased from Terre Haute Boiler Works, Terre Haute, Indiana, who were responsible for fabrication, testing and delivery to the job site. The tower was delivered on schedule, allowing time for the trays to be installed while it was on the ground. The trays for both the evaporator and fractionator were purchased from Koch Engineering Company.

A small but very important item for any coking unit is the master switch valve. Since only one supplier, the Oil Well Supply Company, was interested in making a special switch valve of this type in such a small size, two of these valves were purchased from this supplier. A spare was procured to assure continued operation of the unit.

Four pumps were purchased new. Two of these, the residuum feed pump and the tank transfer pump were supplied by Sier-Bath Gear and Pump Company. The furnace charge pump, an American Marsh pump, was supplied by Powered Equipment Company. The sump pump for the sluice was a Fairbanks Morse, also furnished by Powered Equipment Company.

The experimental coking unit was located adjacent to the commercial coking unit. The off product streams and the utilities of the experimental unit were tied into the comparable streams and utilities to the commercial coker. This arrangement afforded considerable saving over situating the unit at a remote location. However, "A" frame supports were required to carry the pipe and conduit to and from the two units. The "A" frames were fabricated on the job site and installed on their foundations as the job progressed.

It was necessary to move the five product storage tanks from their location in the refinery to their particular location on the plot plan of the experimental coking unit. Four of the tanks are 1250 barrel capacity while the fifth one is 500 barrels. The tanks were moved to the new location and mounted on a previously prepared sand pad. The viscosity of the materials to be stored in these tanks made it necessary to install steam lances in the tanks. Other minor modifications, including gauges and vents, were also required. Moving the tanks was started in January 1962, and installation of steam lances, vents and gauges followed as soon as the tanks were in place.

Since the major portion of the work involved the piping, both welded and screwed construction, a shop was set up to prefabricate as much welded pipe as possible. As soon as the prefabricating materials began to arrive, qualified pipefitters and welders were employed.

The alloy piping for the unit consisted mostly of the furnace

transfer lines from the final furnace outlet to the two coke drums. Since the welds on this pipe had to be stress relieved before it could be put in service, all of the prefabricated alloy pipe was taken to a large furnace and the relief stress performed in one lot.

Steam tracing preceded the insulation of the piping on lines handling heavy products or where a temperature loss could not be tolerated. Copper tubing was either tied on as a companion line or coiled around the line to be kept warm.

Insulation of the unit was handled in two parts. The large vessels, coke drum, fractionator and evaporator were insulated by the A&K Insulation Company, whereas the piping and all related equipment were insulated by the Johns-Manville Sales Corporation and Refinery maintenance group.

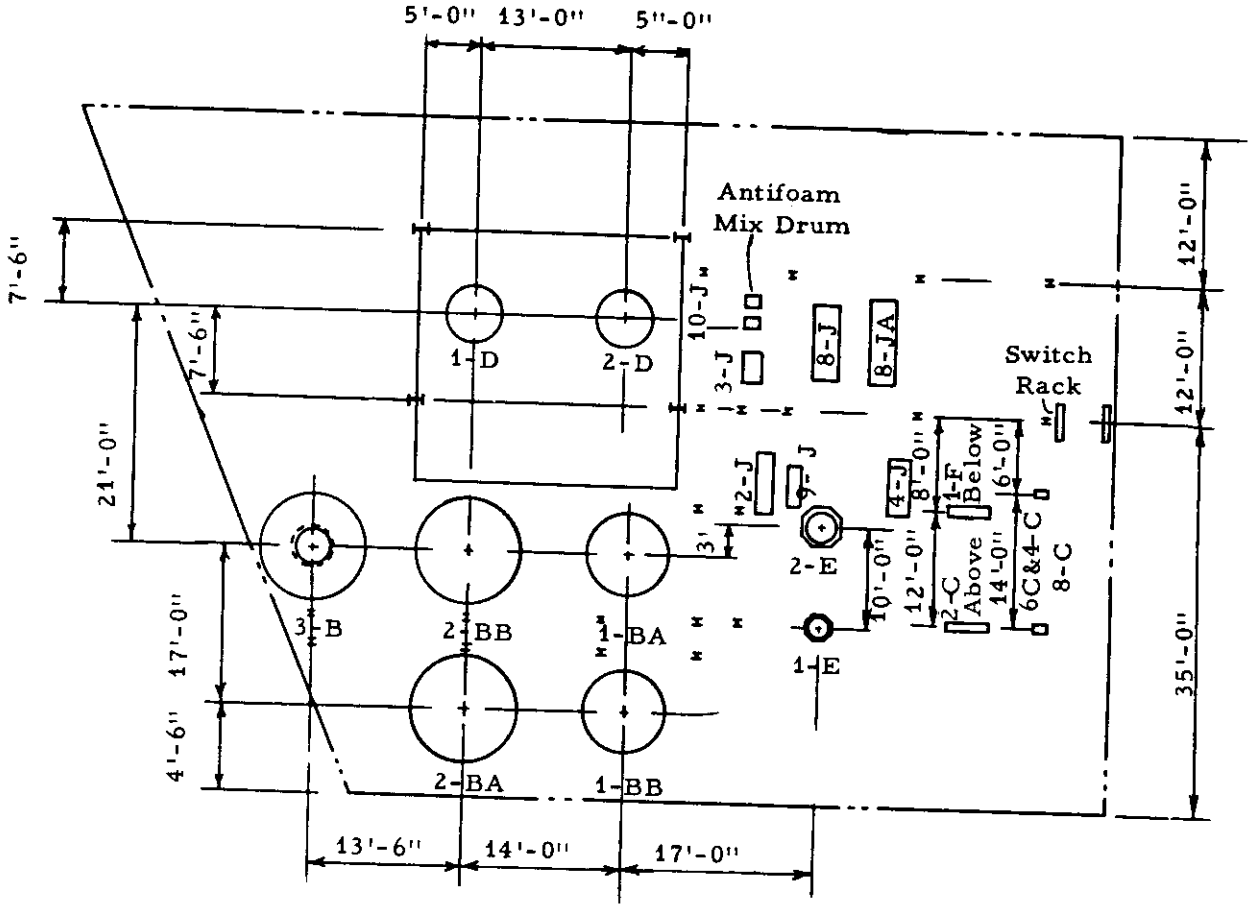
Painting was carried on during most of the construction period. The coke drum structure was delivered with a prime coat already applied. All other structural steel and pipe was prime coated as it was installed. All exposed uninsulated parts of the units were finished with a coat of aluminum paint. The painting work was done by Sheehan Pipe Line Construction Company as a part of the general contract.

Comment on instrumentation is omitted here, since a complete summary of this phase of the work is written as a separate portion of this report.

A test schedule for the unit was prepared by the Marathon Oil Company inspection department. This schedule outlines the hydrostatic test pressures applied to each part of the unit. The testing conditions outlined by the inspection department were taken from the A. S. M. E. Unfired Pressure Vessel Coke, Section VIII, 1959. Figure 7, attachment "A", is a copy of the testing procedures.

The construction phase of the experimental coking unit was completed June 29, 1962. The construction of the unit was completed according to the M. W. Kellogg Company preliminary design and Marathon Oil Company plans and specifications. All mechanical and electrical components of the unit were checked out and found operative at this time.

During construction, the following weekly pictures were taken to relate the progress of the work to the various agencies concerned with this project.



1-BA	Preheater	1-E	Evaporator	10-J	Antifoam Pump
1-BB	Cracking Furnace	2-E	Fractionator	11-J	Coke Fines and Water Pump
2-BA	Preheater				
2-BB	Cracking Furnace	1-F	Reflux Drum		
3-B	Steam Superheater				
		2-J	Furnace Charge Pump		
2-C	Overhead Condenser	3-J	Condenser Pump		
4-C	Gas Oil Product Cooler	4-J	Gasoline & Reflux Pump		
6-C	Visbreaker Tar Cooler	8-J	Flushing Oil and Evaporator Quench		
8-C	Fresh Feed Preheater				
		8-JA	Gas Oil Reflux Pump		
1-D	Coke Drum	9-J	Visbreaker Tar Pump		
2-D	Coke Drum				

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Figure 7. Equipment Plot Plan

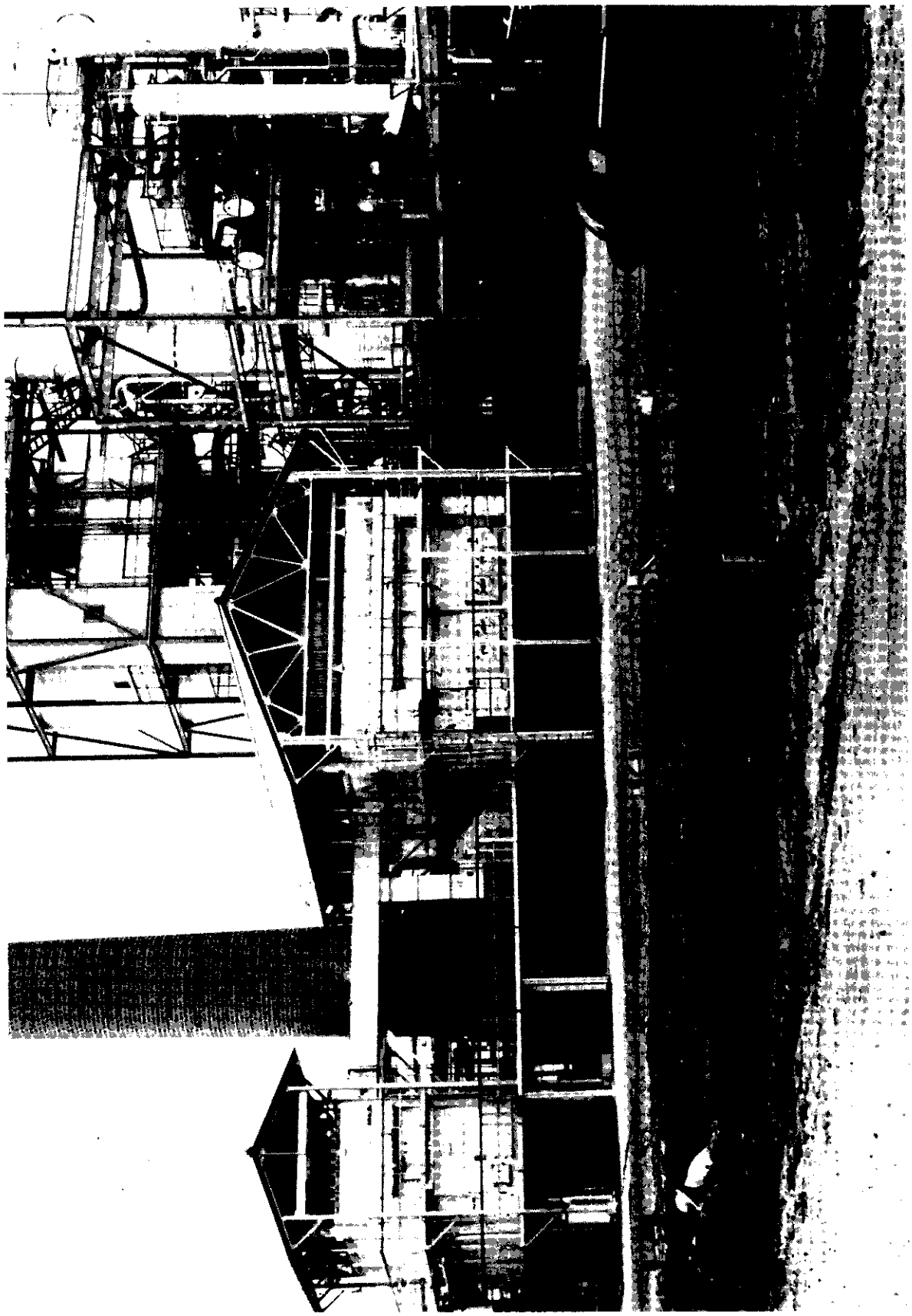

Figure 8. Construction as of March 9, 1962

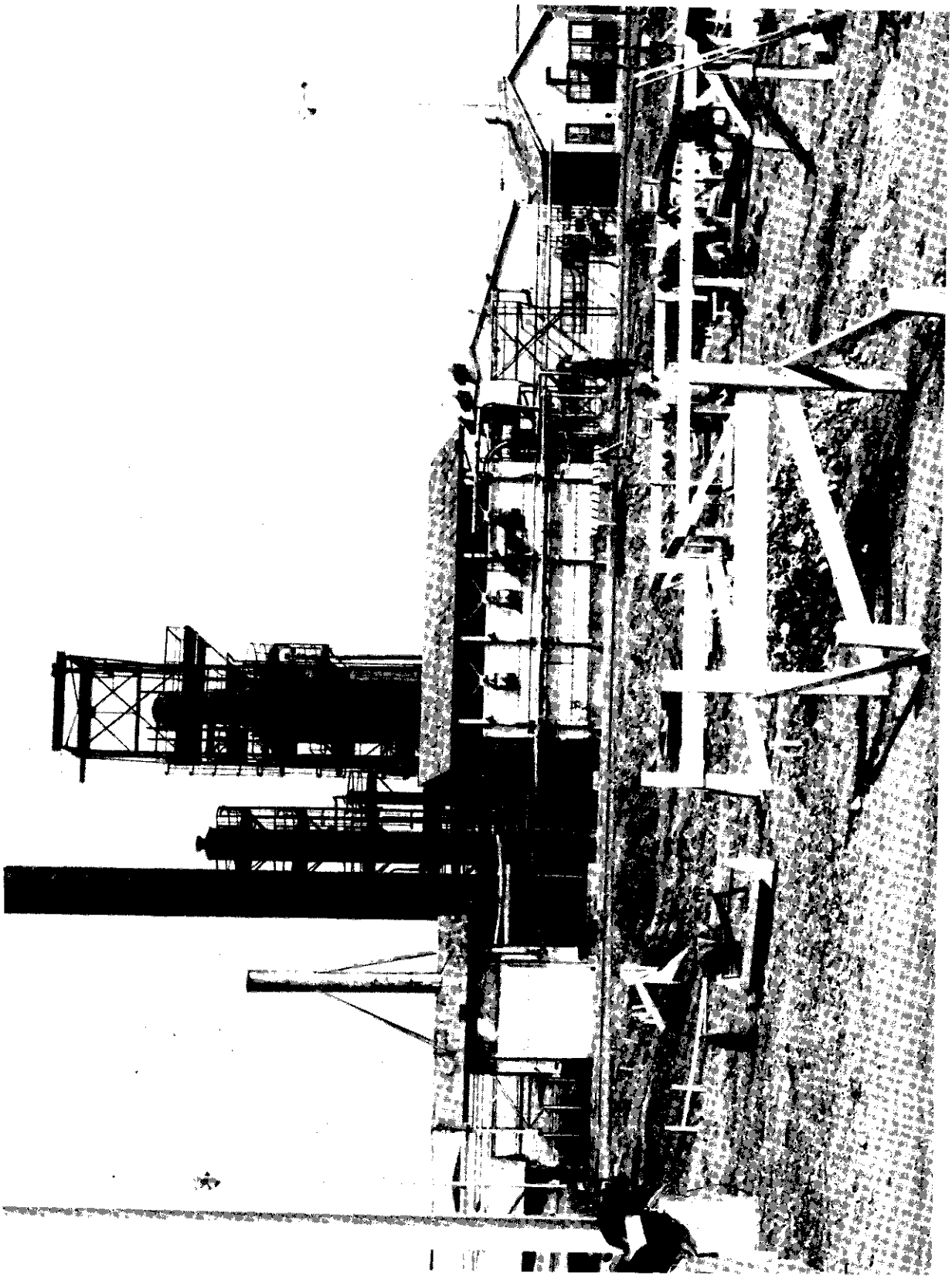

Figure 9. Construction as of March 19, 1962

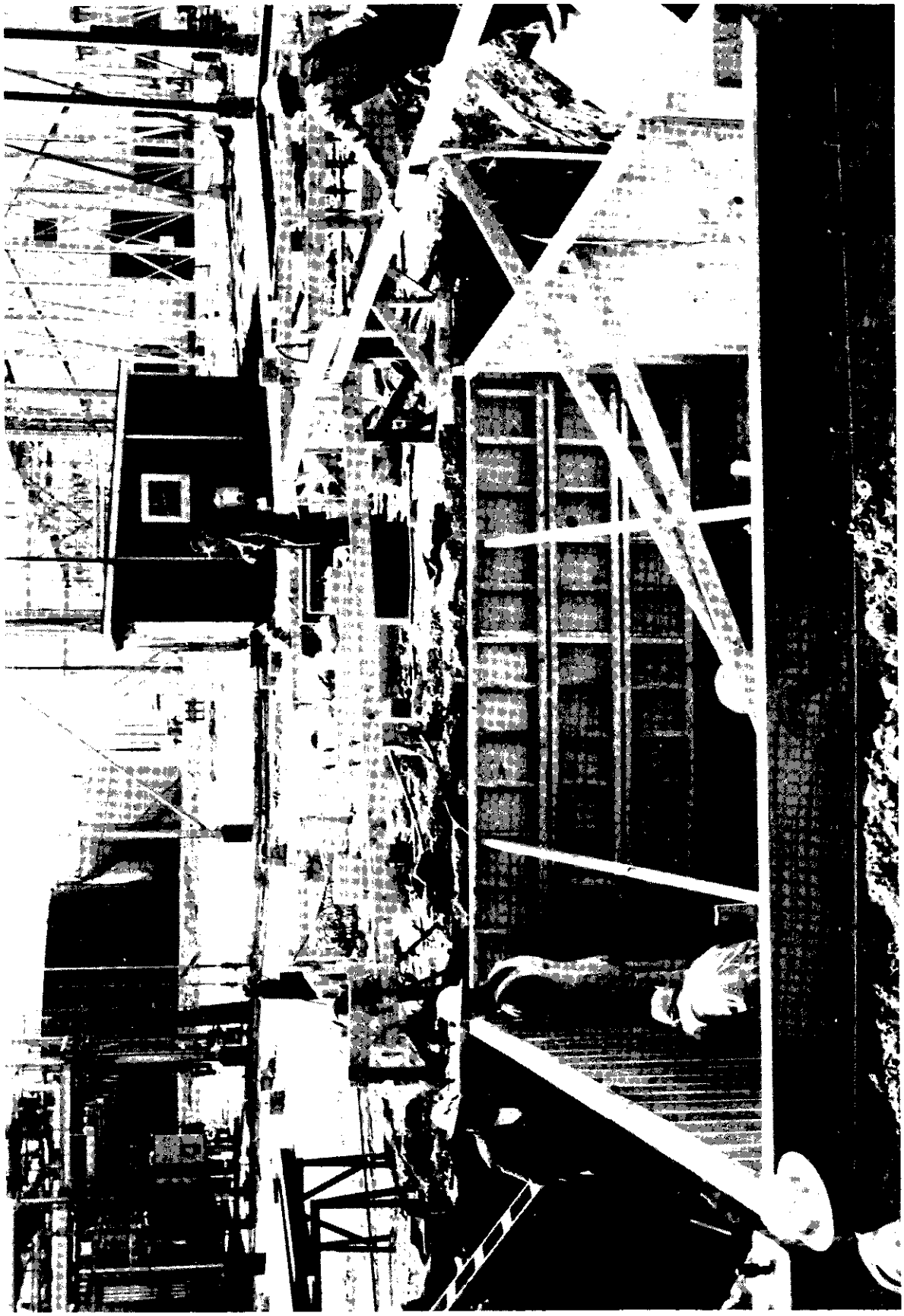

Figure 10. Construction as of March 27, 1962

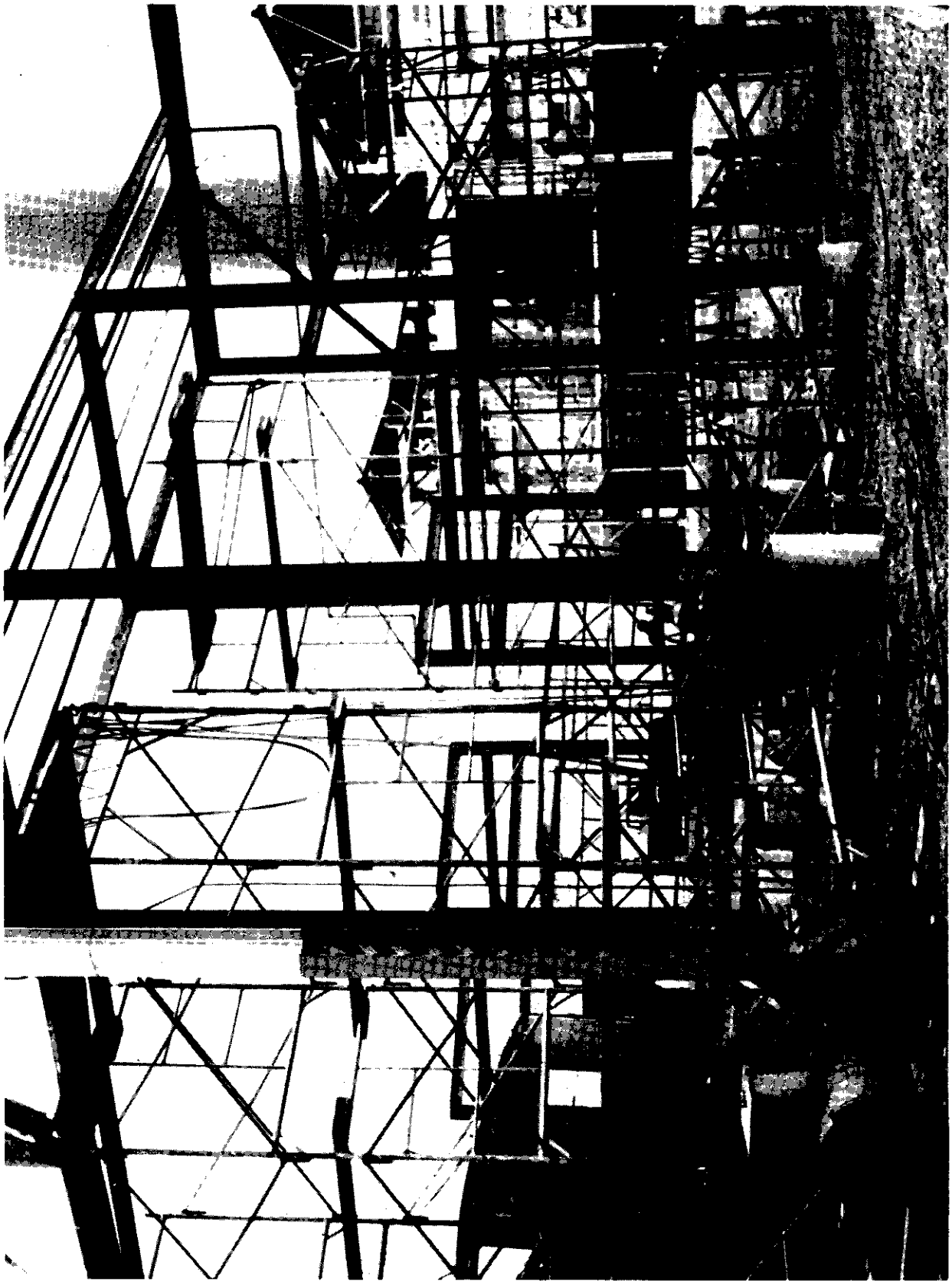

Figure 11. Construction as of April 2, 1962

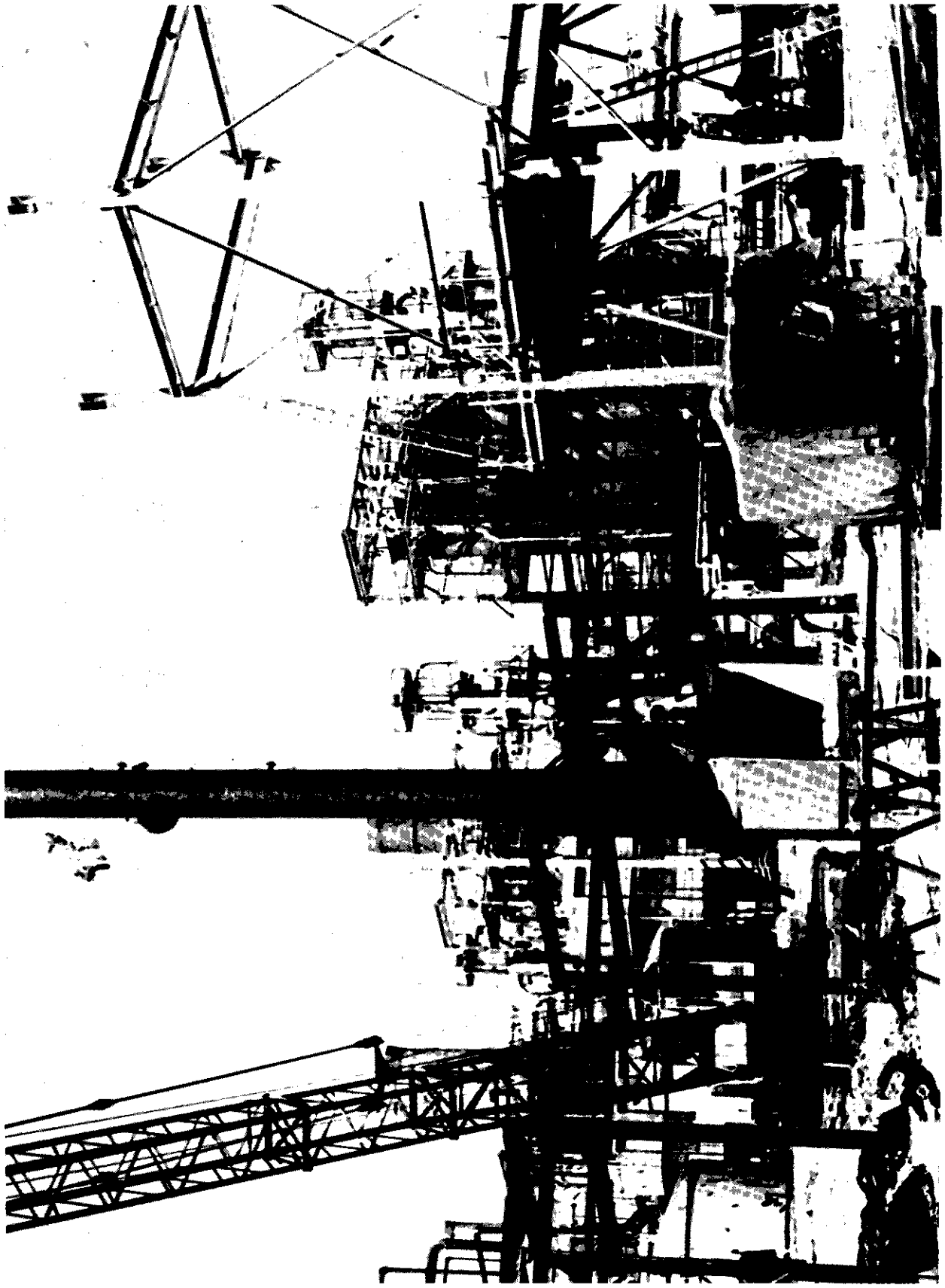

Figure 12. Construction as of April 9, 1962

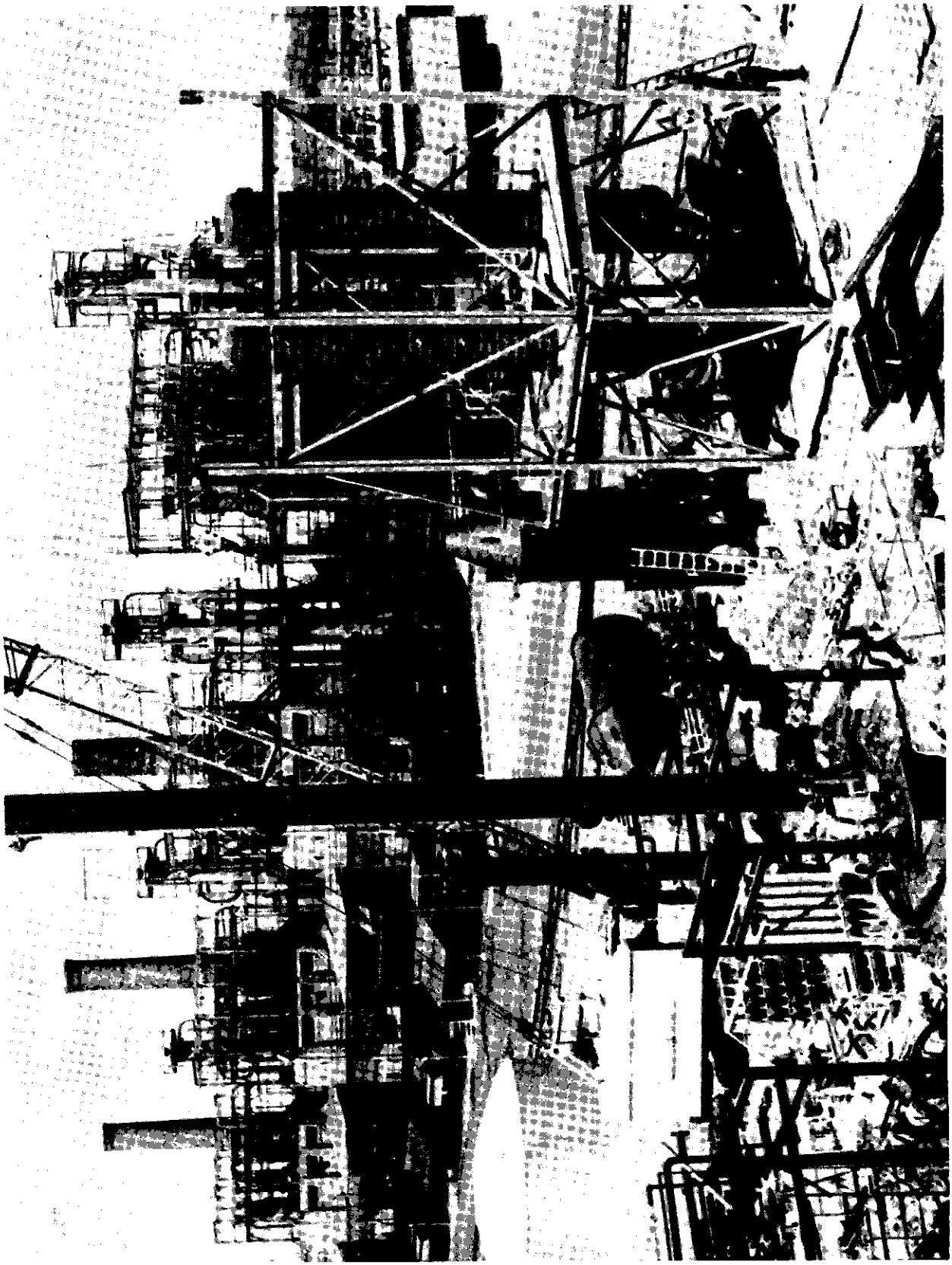

Figure 13. Construction as of April 16, 1962

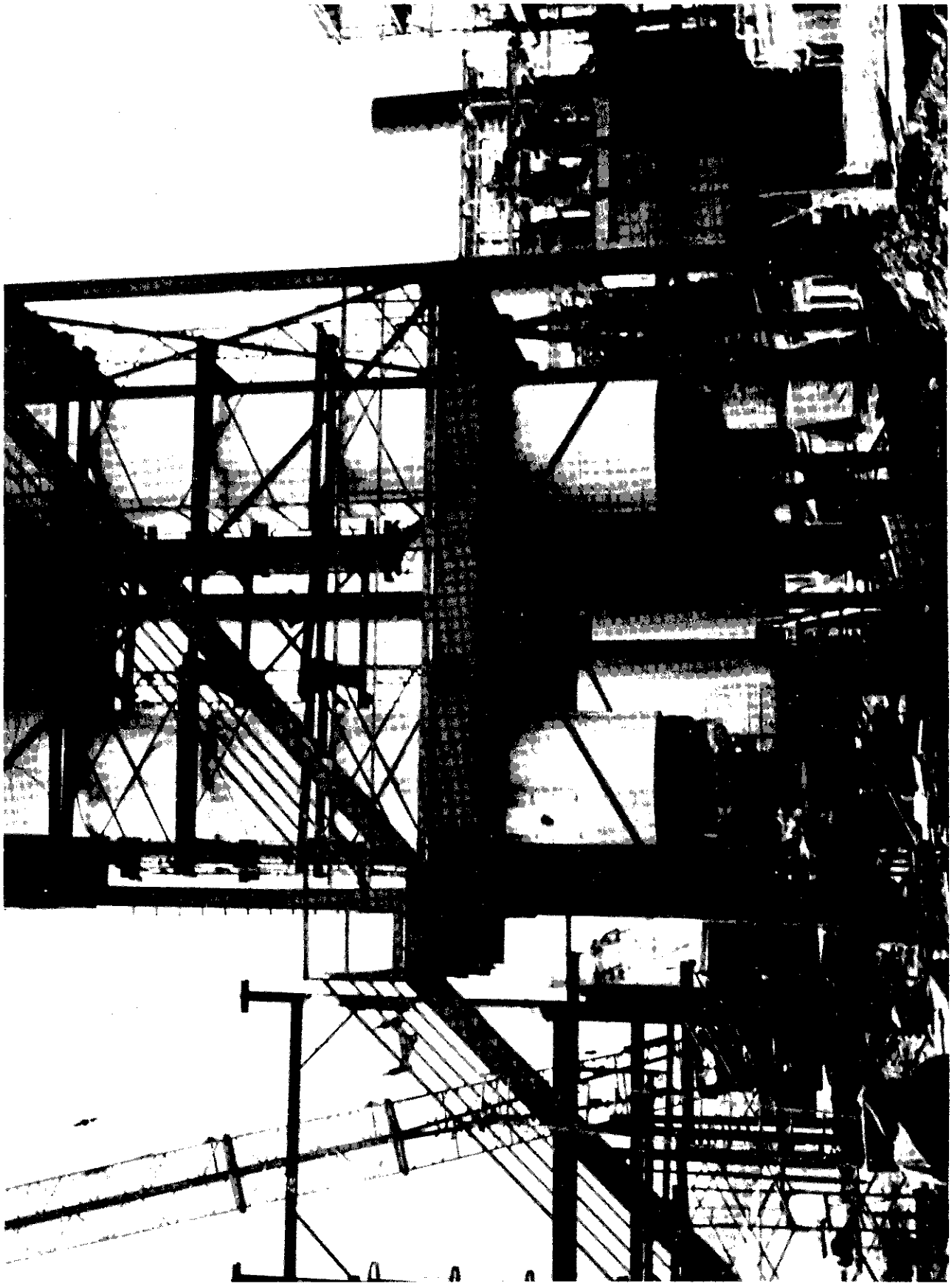

Figure 14. Construction as of April 23, 1962

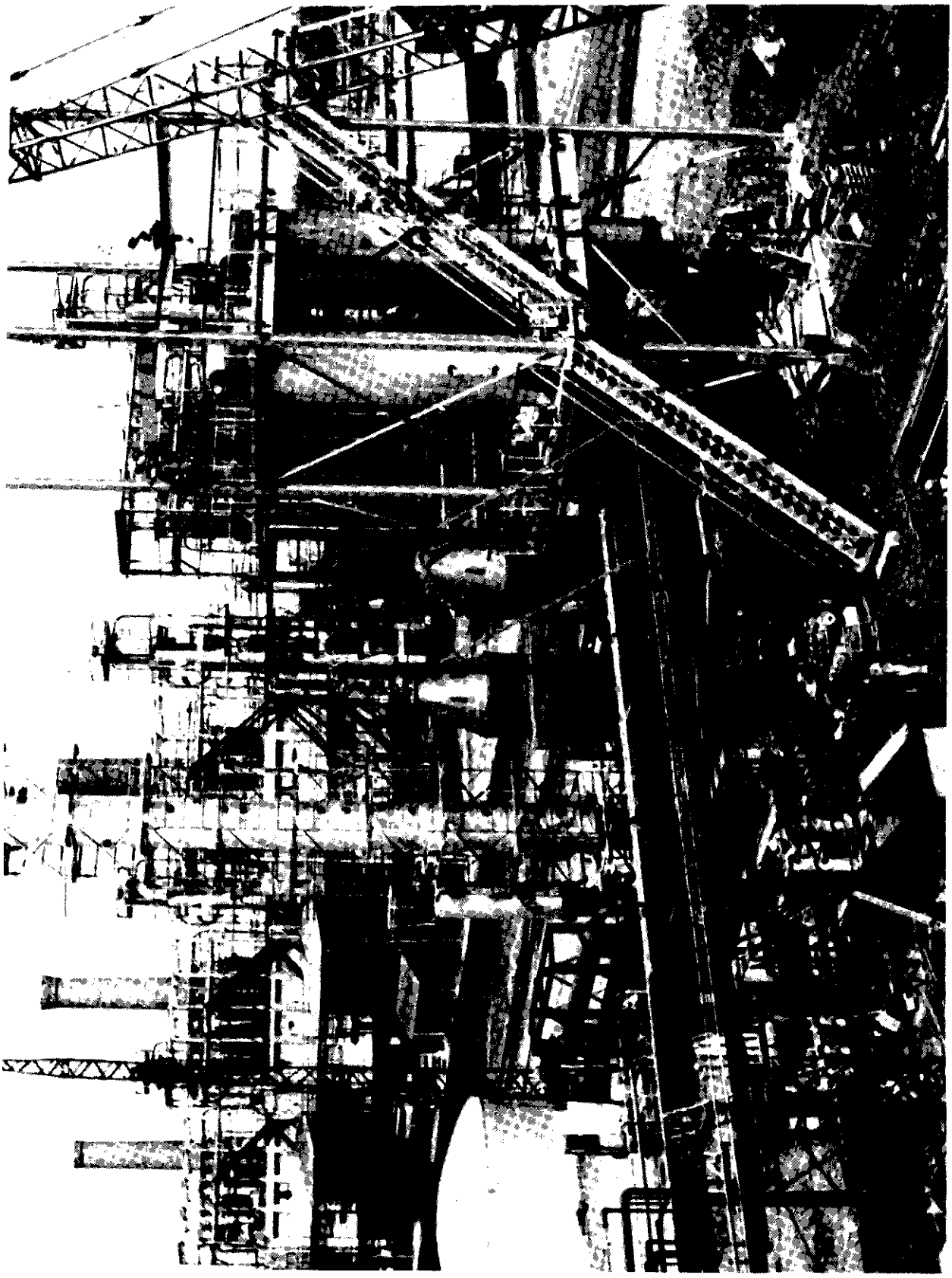

Figure 15. Construction as of May 2, 1962

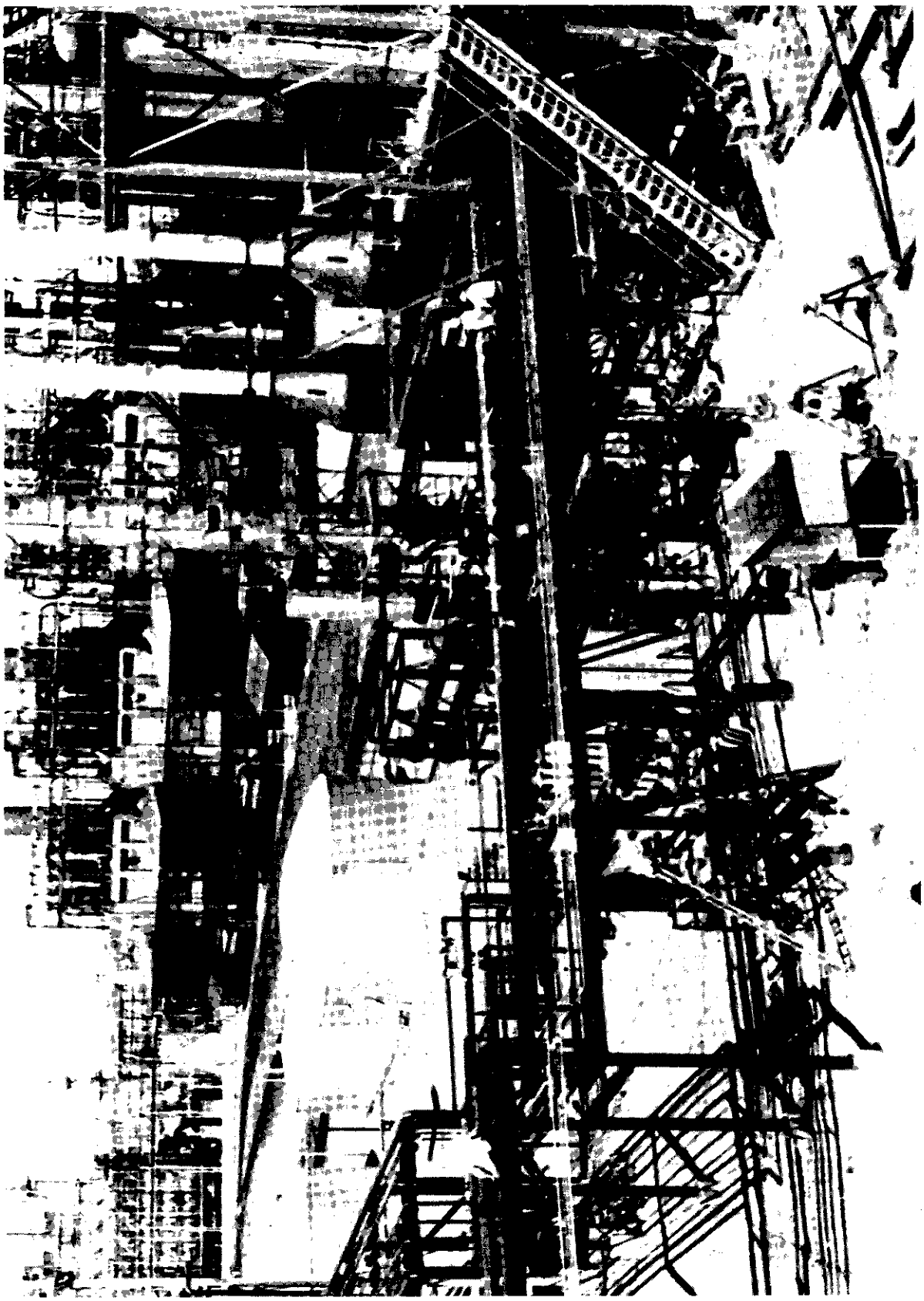

Figure 16. Construction as of May 7, 1962

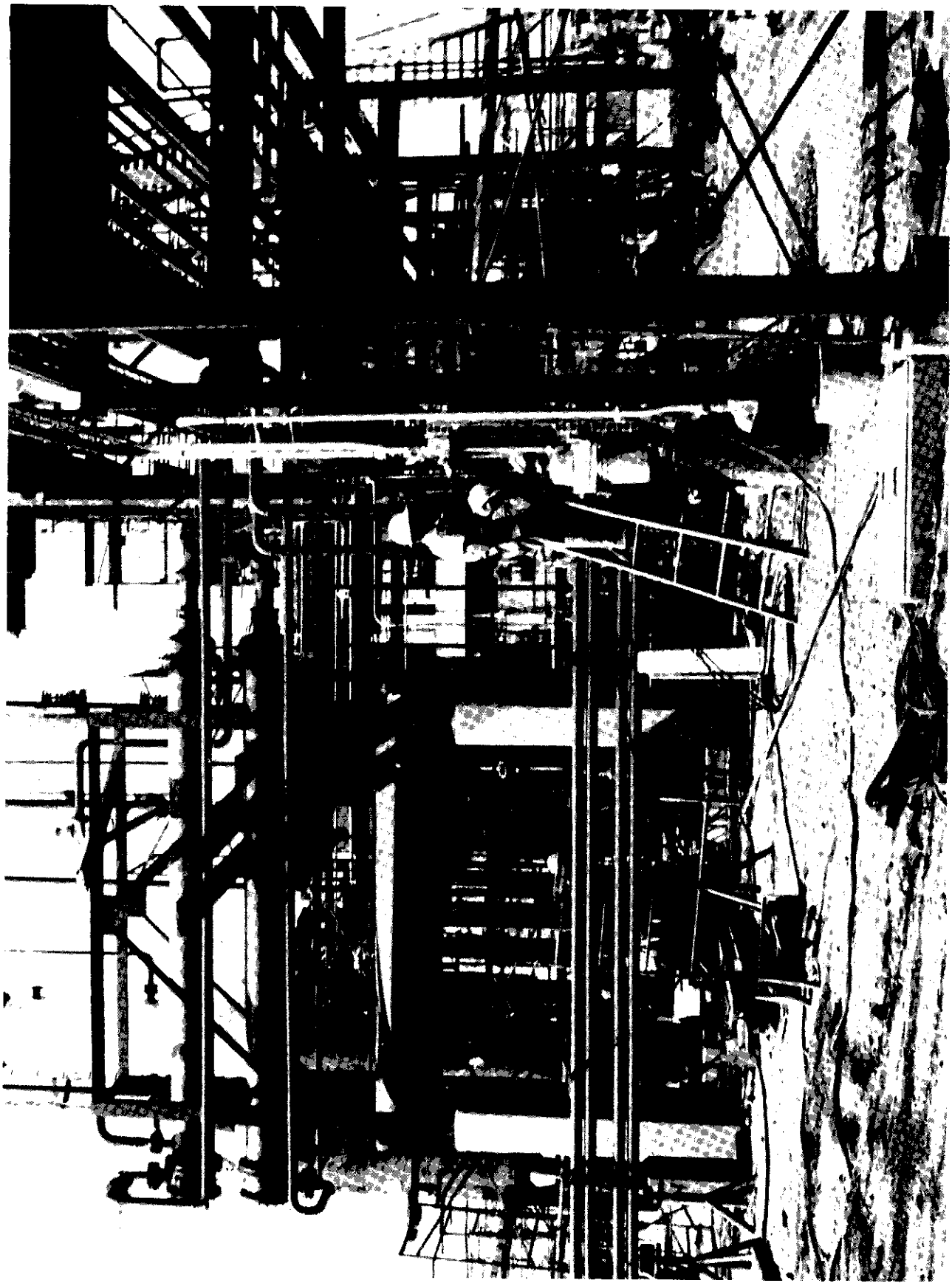

Figure 17. Construction as of May 14, 1962

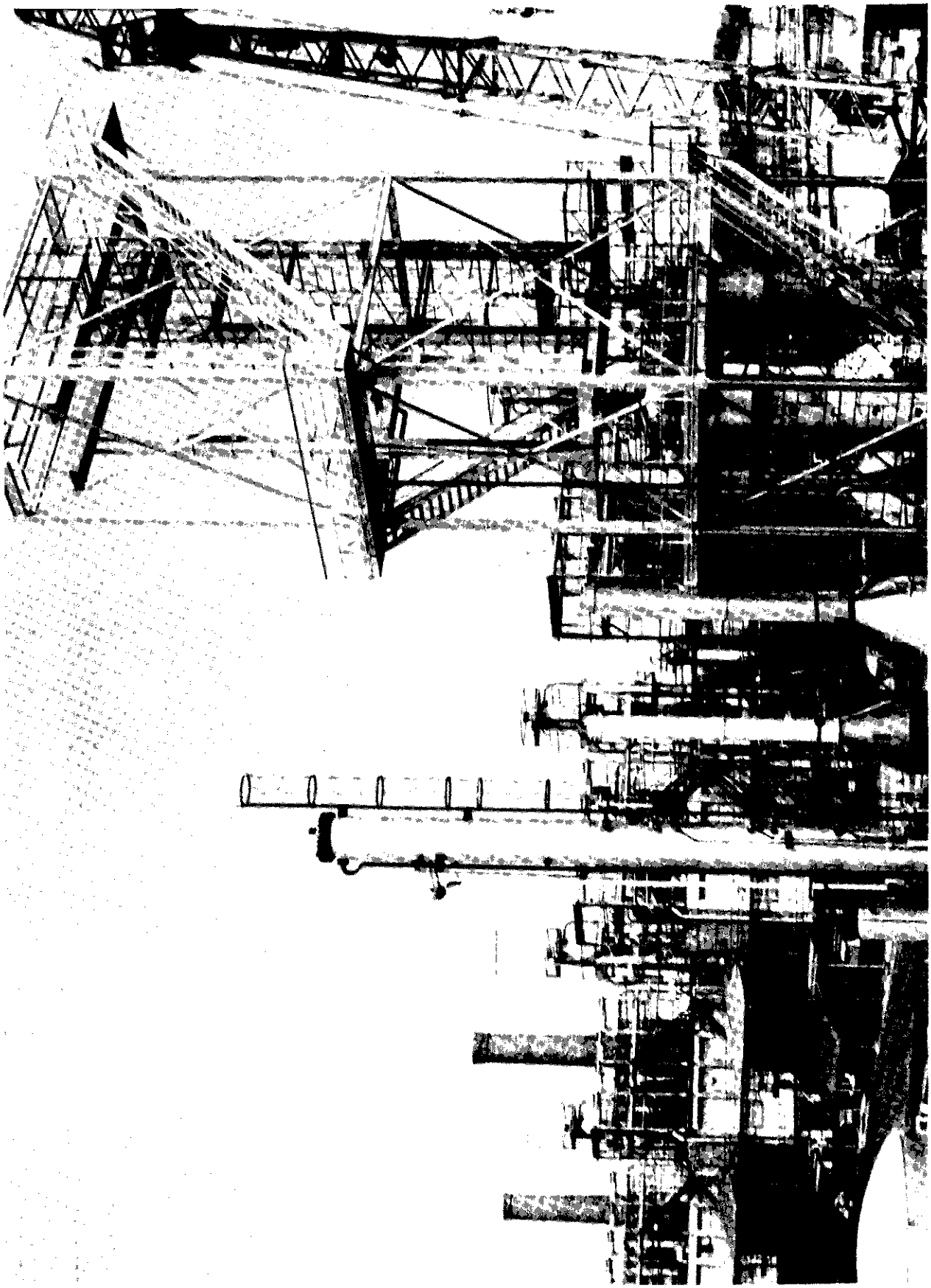

Figure 18. Construction as of May 21, 1962

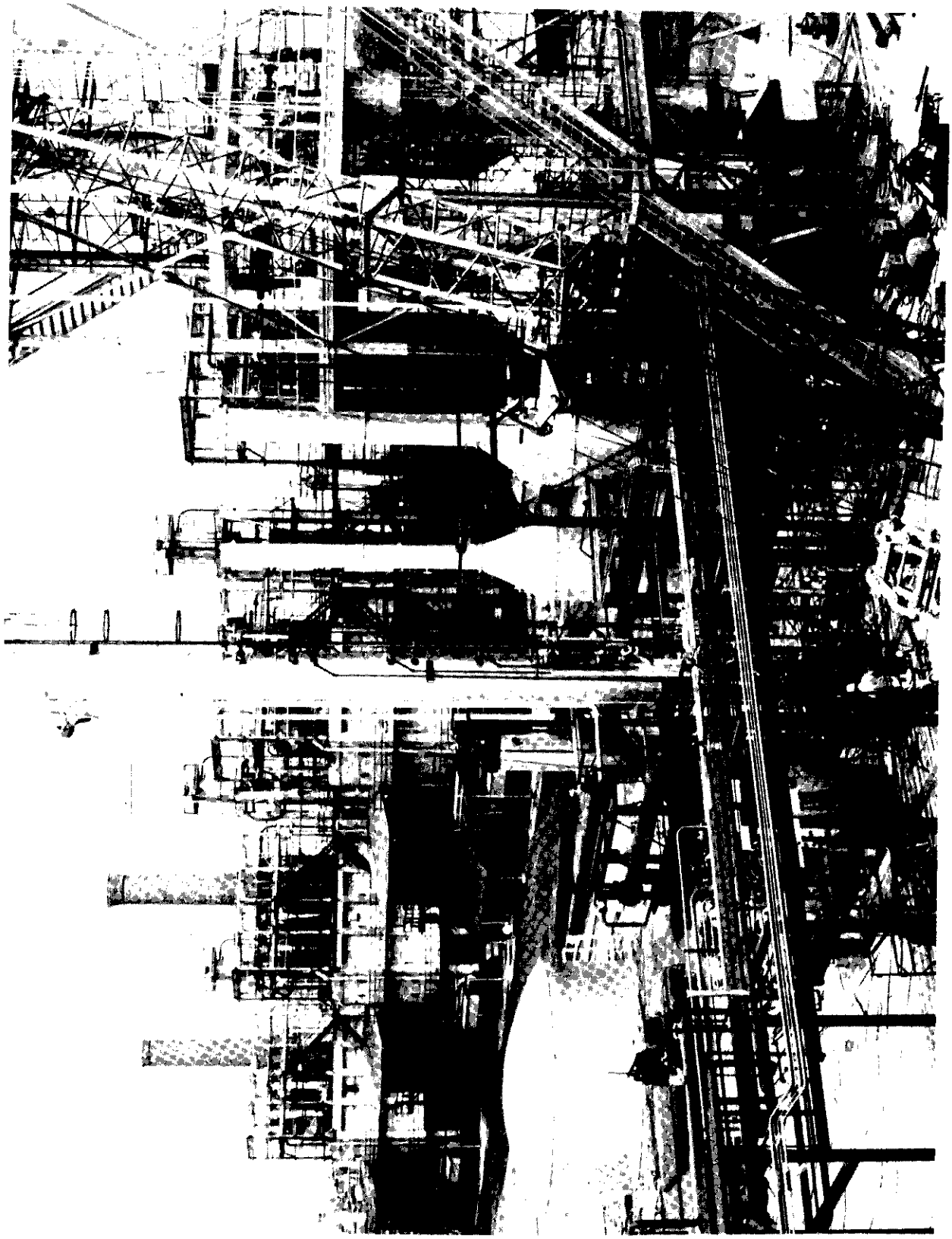

Figure 19. Construction as of May 28, 1962

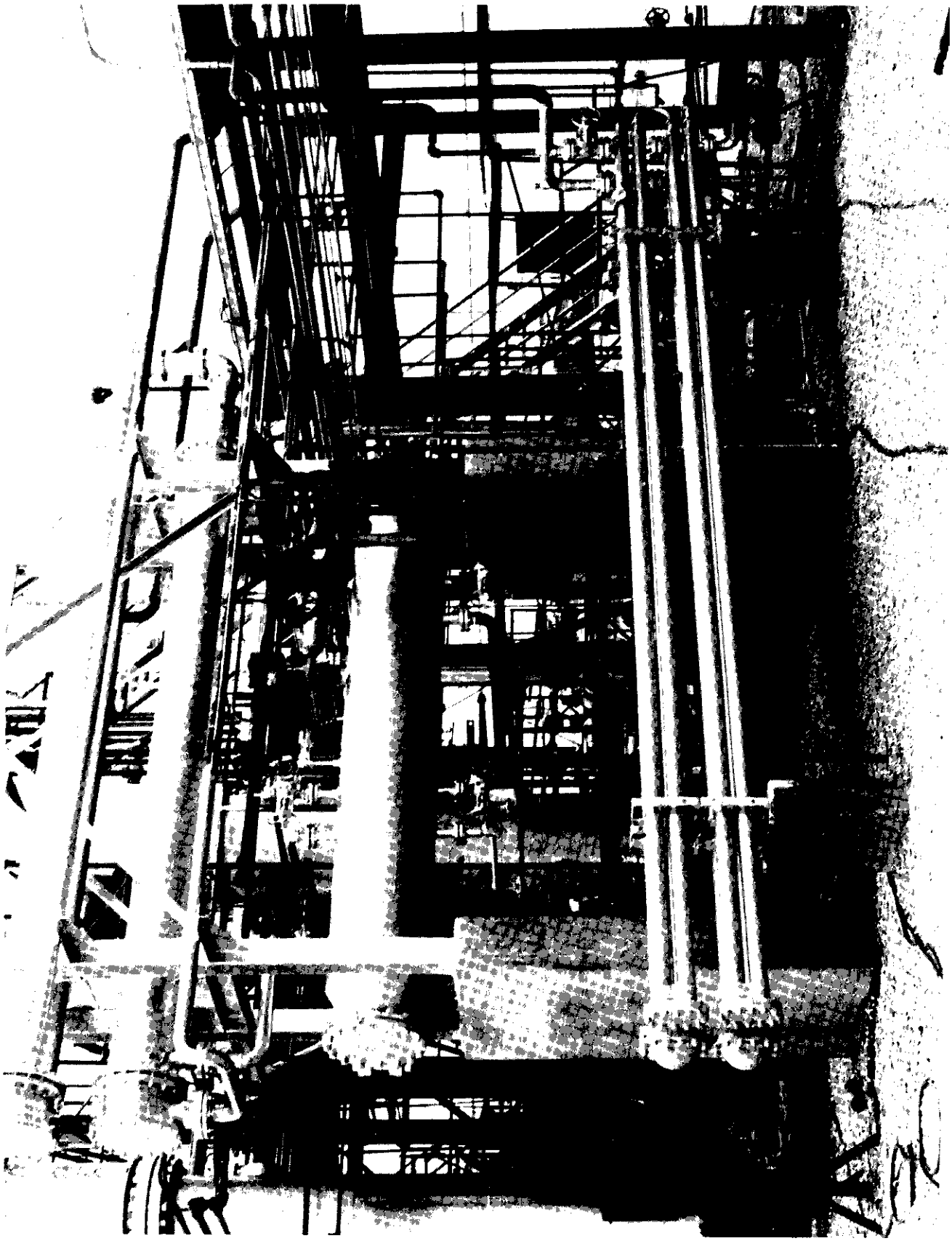

Figure 20. Construction as of June 4, 1962

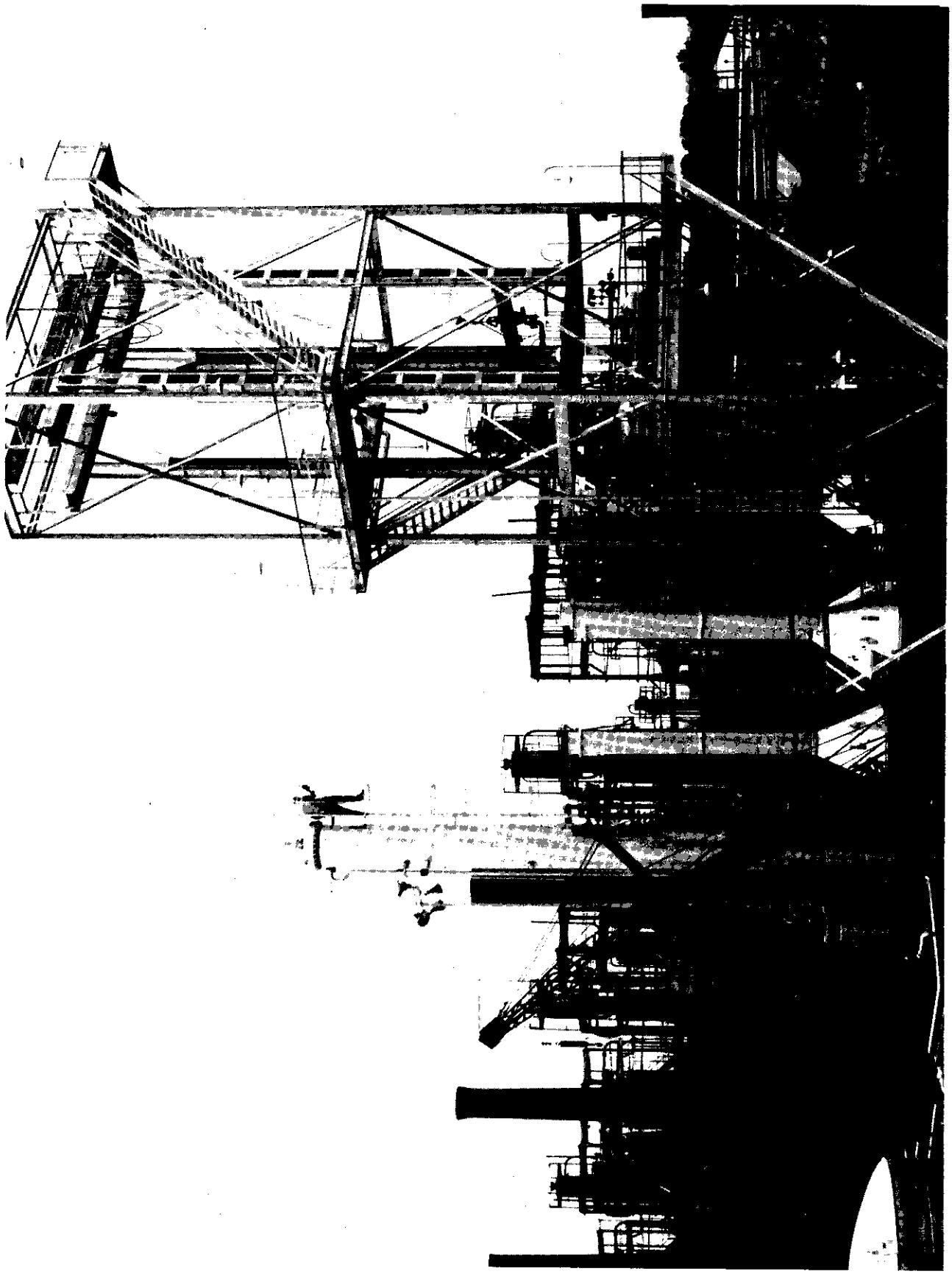

Figure 21. Construction as of June 11, 1962

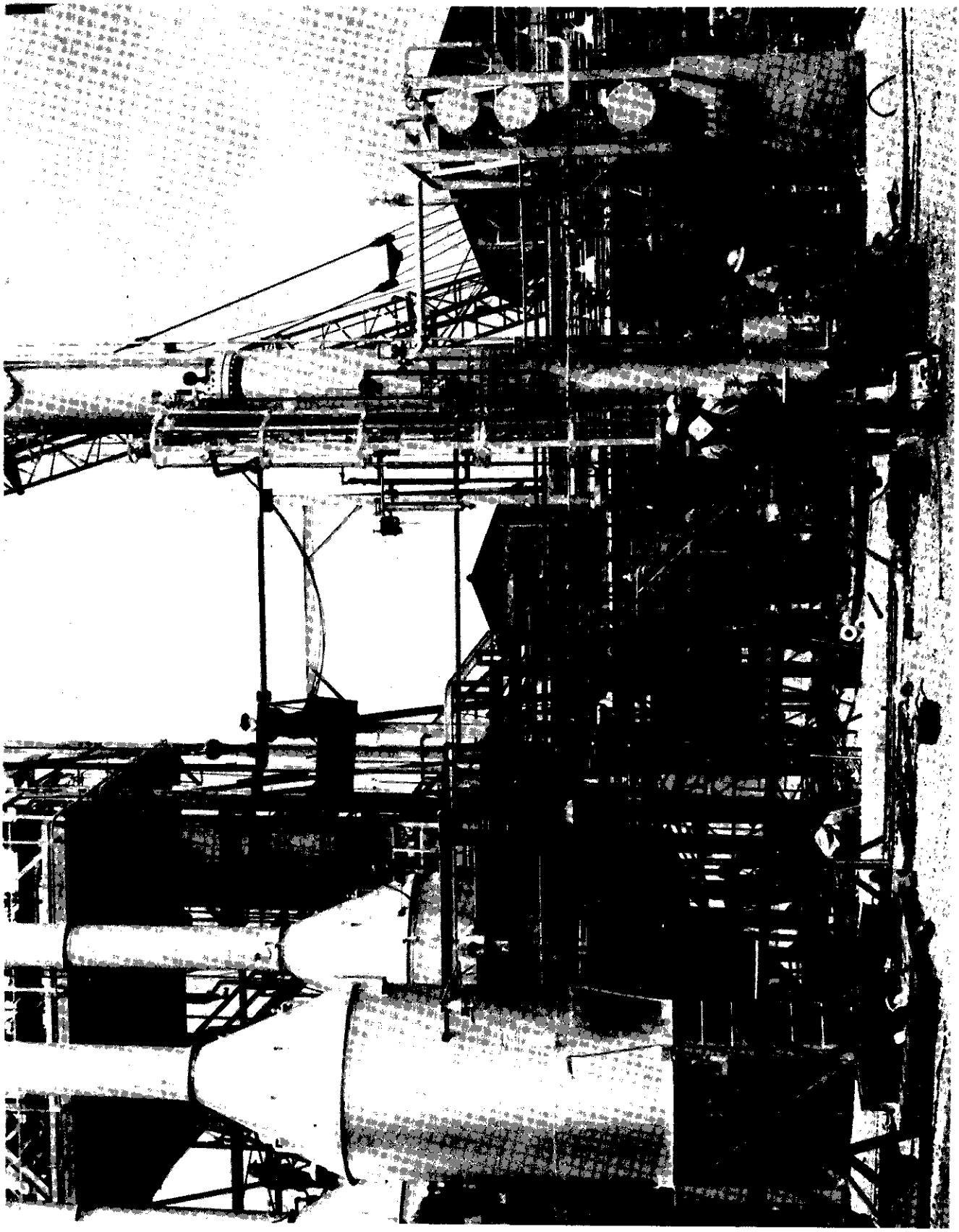

Figure 22. Construction as of June 18, 1962

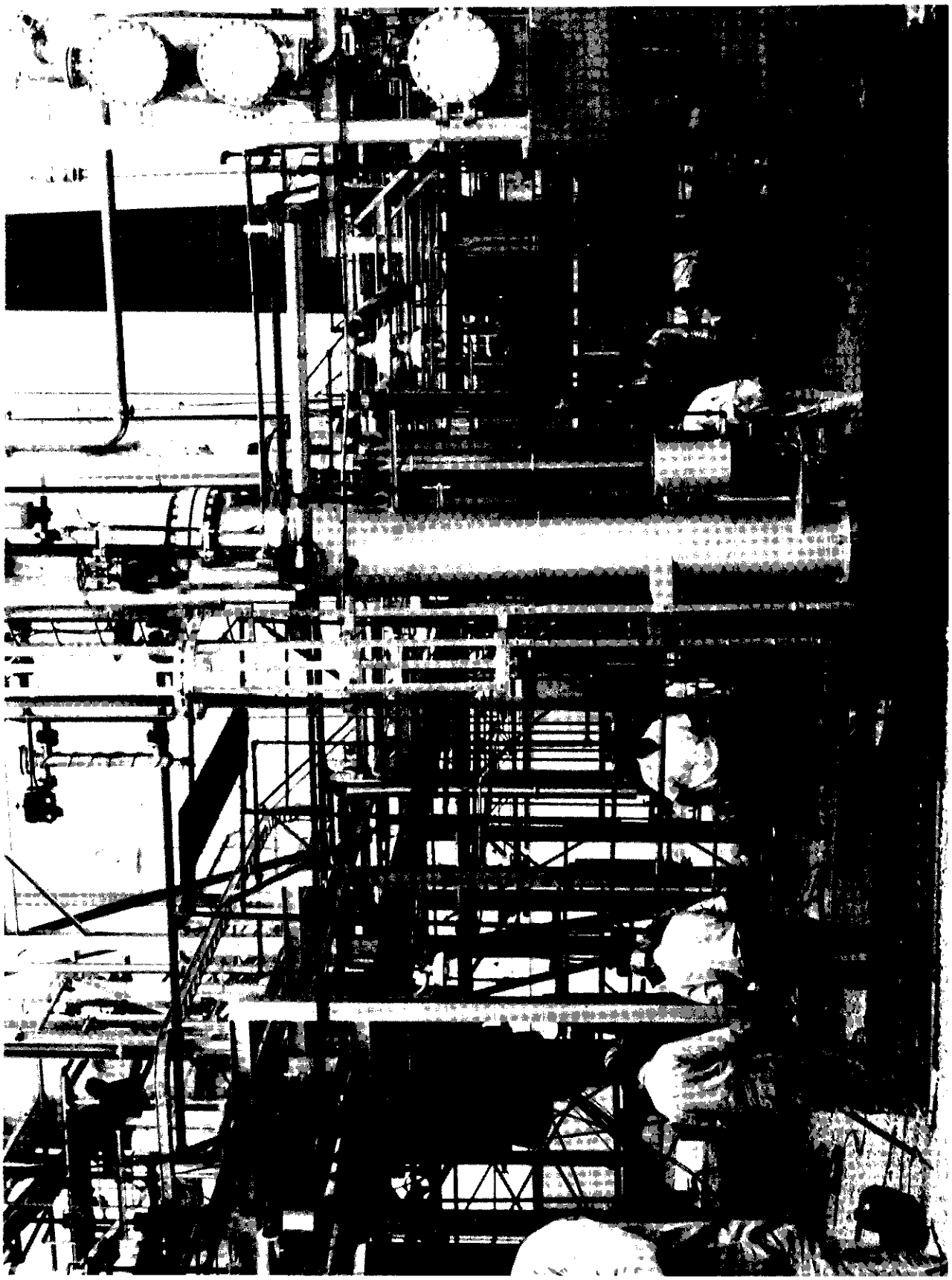

Figure 23. Construction as of June 25, 1962

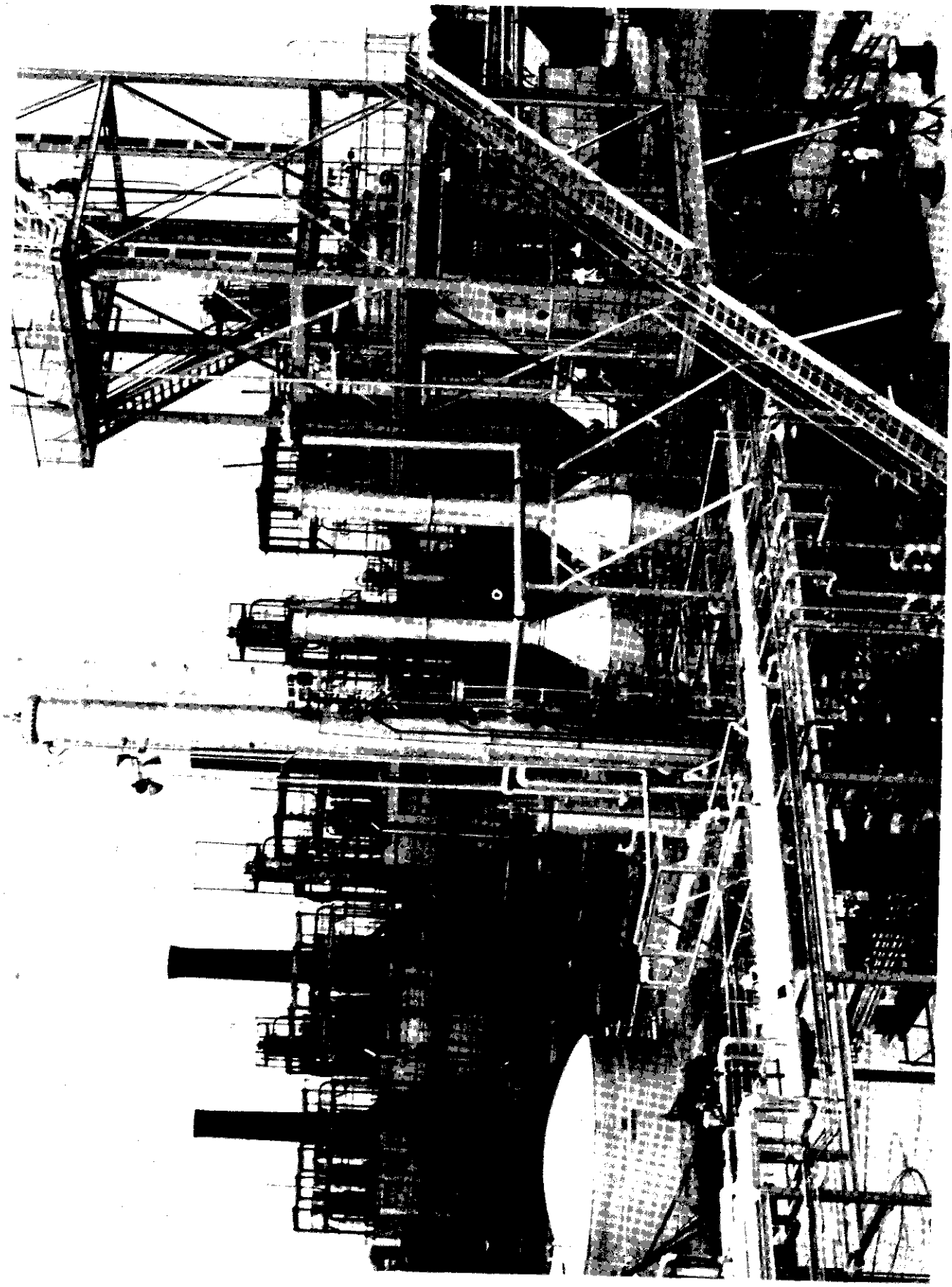

Figure 24. Construction as of July 3, 1962

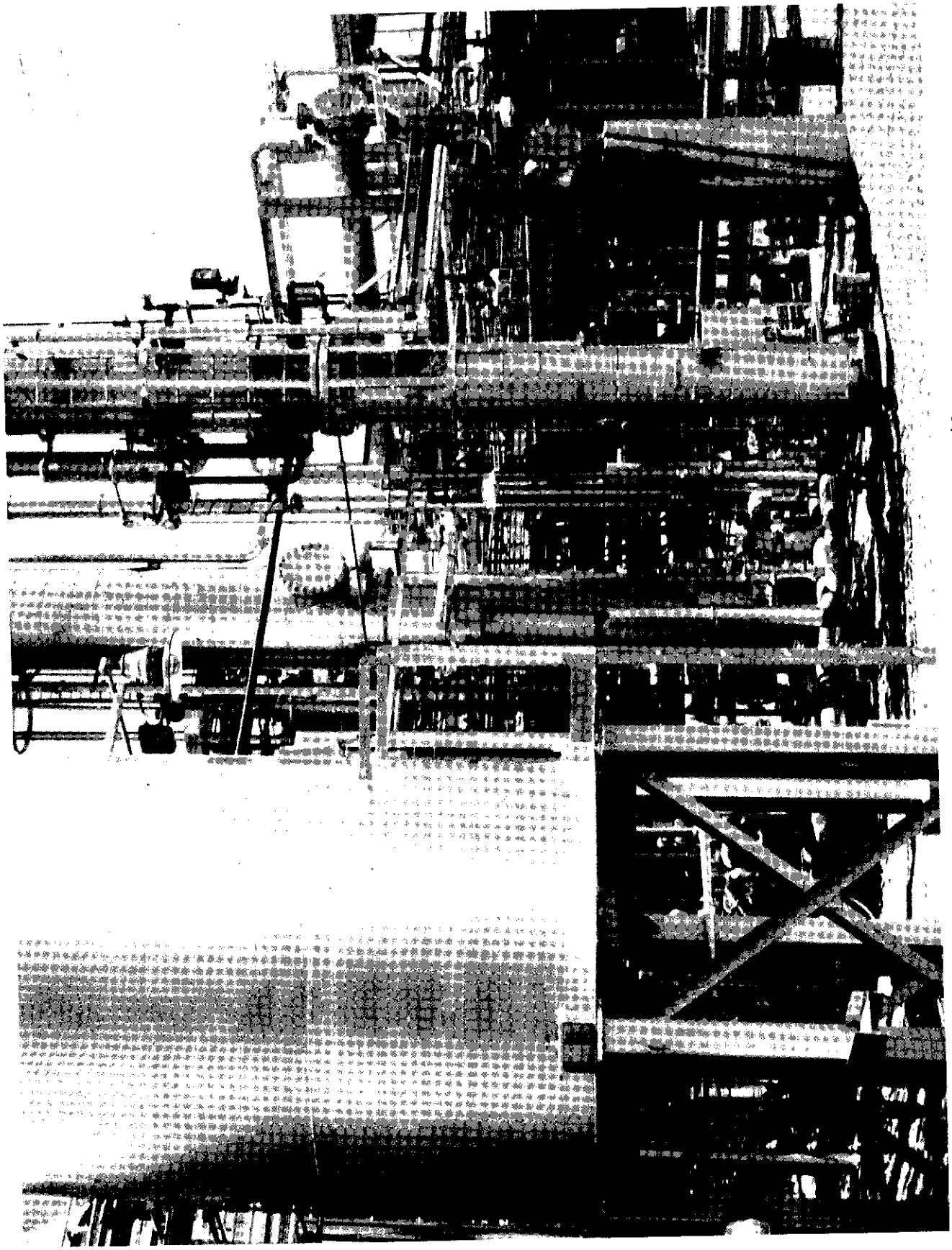

Figure 25. Construction as of July 10, 1962

A number of small revisions and additions were necessary during the early weeks of operation. Most of these were changes in instrumentation and will be explained in another phase of this report.

A major mechanical change was the addition of a preheater in the charge stream to the fractionator from the residuum feed pump in order to maintain a 500°F inlet temperature to the fractionator tower. Another change was the addition of a water boot on the bottom of the reflux drum which permitted a longer water column for separation of water and gasoline. The boot was added and the level controller piping on the boot changed during a scheduled shutdown.

An equipment maintenance and lubrication manual was prepared listing all equipment and the individual maintenance requirements. The frequency of lubrication and the type of lubricant required for each piece of equipment is described. The maintenance manual is outlined in the latter part of this Appendix.

I. 4. Instrumentation

I. 4. 1. Engineering Design Concepts

The instrumentation involved in the experimental coking unit is to a large degree unconventional. The design was based primarily upon need for extreme flexibility consistent with operation over a limited period of time. A relatively large number of special items were necessary, due to extremely low flow rates and the coking characteristics of some of the process streams. An unusually large number of measurements were necessary to obtain the experimental data required.

Due to the wide range of process variables for the tests proposed, it was not possible to design a single set of instruments capable of operating over the full range of conditions. The use of unconventional instrumentation minimized the need for reinstrumenting, and the use of alternate ranges and trim was expected to handle the remainder. The basic instrumentation design philosophy was founded on maximum utilization of unconventional features, since normal practices would require extremely high cost and would not be suitable for the abnormal process requirements.

Continuous process type instruments were selected rather than pilot plant instruments. All instruments necessary for the continuous safe operation of the unit were installed in the control room. The original idea of providing a separate control house for the unit was abandoned in favor of installing the control panel in the control room of the commercial unit. Many advantages were gained by combining the control rooms for the commercial and experimental units. The need to add one board man per shift was eliminated. Coordination was improved for equipment shared by both units such as the high pressure drilling water system and all utilities. Duplication of instrumentation for the items mentioned was avoided. Better coordination was obtained in sharing the board man, the foreman, the stillman, the coke drilling crew, maintenance men, and in the exchange of man-

power for emergencies. A combination of all of these items permitted further reduction of operating labor for the experimental unit.

An added instrumentation design problem arose in altering the location of the control room to an average distance of 350 feet from the control elements. Adverse frequency response characteristics are normally encountered with long lengths of pneumatic control tubing. Electronic control could not be used, because none of the special transmitting elements were available in electronic equipment. The normal adverse effects were compensated for by the small volume diaphragms on the control valves used on extremely small flows. Piston operators with positioners were selected for valves with more demanding requirements. Due to the limited anticipated life of the project, plastic tubing and plastic jacketed thermocouple wiring bundles were substituted for copper tubing.

I. 4. 2. Control Valves and Hand Control Valves

The extreme variations in process operations made it impossible to size many of the control valves to cover all conditions. Two valve types were used and in each case only one manufacturer could be found to meet all of the requirements. Twenty Foxboro type V-4 needle valves were used for which trim is available in 12 sizes from $\frac{1}{64}$ inch to 1 inch. A selection of alternate trim was purchased that could, by changing trim, meet various process conditions. An equal percentage plug with a 35:1 ratio of flow coefficient permits a wide range of operation. The flexibility of this valve permits operation over a much wider range of conditions than is normally possible. Nine special chrome-moly piston operated valves were used for 1200°F superheated steam and for the visbreaking pressure letdown valve.

Thirty Hancock manual hand control valves of various types were used. Twenty of these were used as bypasses around the Foxboro type V-4 control valves to permit changing trim during operation. Each bypass valve was individually engineered for good hand control because of the extremely low flows and relatively high pressure drops. Since the 1200°F superheated steam valves are not in continuous operation, bypass valves were eliminated, and a block valve was provided to isolate as a group the four valves on each drum. This approach greatly simplified the stainless steel piping. The bypass for the thermal cracker letdown valve was eliminated to lessen possibilities of coking, since the bypass facilities would probably be inoperable due to coking in the normally static line.

I. 4. 3. Flow Measurement

The measurement of flow presented special problems, and several individual approaches were necessary. One problem typical of most of the flow measurements was the wide variation of flow rates. Indicating linear flow transmitters were used for all transmitted flow signals. This type of transmitter extracts the square root of the differential pressure measurement and indicates and transmits a linear signal proportional to flow. The significant advantage of this instrument system is that the flow measurement

is readable over about 20 to 1 range compared with about a 3 to 1 range for the normal square root measurement. For pulsating flows on reciprocating pumps, any attempt to dampen the flow signal introduces a very large error, whereas the linear transmitted signal can be dampened without introducing error. The large integral linear flow indicators on the Taylor transmitters aid the field operators and eliminate the normal cost of running piping to eye level to make small separately mounted indicators readable. They also facilitate field calibration of the transmitter.

Primary flow elements used for the above transmitters were orifice plates. Two orifice flanges, four 1 $\frac{1}{2}$-inch flow tubes, four 1-inch flow tubes and two integral orifices were used. Spare blank plates were provided with the flow sections, and one set of six special orifices for use with the integral orifice assemblies were provided for flows outside the range of the transmitters. To insure good accuracy for low flow rates of orifice measurement, honed flow tubes were necessary because of increased effects of tolerance and roughness. For very low flows the flow section was built as an integral part of the differential pressure transmitter with orifice bores ranging from 0.020 to 0.340 inch. The two integral orifice transmitters came equipped with a bypass to permit changing orifices during operation.

Four high pressure armored rotameters were installed for flow indication.

I.4.4. Radioactive Foam Level Measurement of Coke Drums

Delayed coking units ordinarily generate a large level of foam above the coke in the coke drums. The foam presents a problem because the level is unpredictable, and if it builds up high enough in the drum, it will carry over into the vapor line and plug off causing a shutdown. Although the solid coke in the experimental unit does not build up to any appreciable level, the height of the foam layer is much the same as for a larger commercial unit but is even more unpredictable because of changes in feed composition and operating conditions. Radioactive equipment only is reliable for detection of foam level.

The radioactive level detection equipment (Ohmart level recorder) consists of one point of detection in each drum, nine feet below the upper flange. Antifoaming agents can be added to reduce the foam level. The time and amount of injection of antifoaming agent is based on the information from the Ohmart level recorder.

I.4.5. Level Measure and Control

With the exception of the radioactive level detector, all other levels are measured and controlled by conventional process equipment. Five external displacement type level controllers are used. All level controllers are located in the unit area. Controls and level indication are installed at ground level if the float chambers are in fairly inaccessible

locations. Gauge glass type level indication was provided with level controls, but lighting was omitted. The omission of platforms and gauge glass lighting has caused considerable inconvenience. Three levels signals are transmitted to the control room for recording or indication and for alarms.

The five storage tanks were fitted for level gauges of an obsolete type. Although these gauges are not very accurate, they eliminate the necessity for hand gauging and are adequate for the requirements.

I.4.6. Temperature Measurement

A total of 56 temperature measurements were made by means of iron-constantan thermocouples using a 56 pair extension wire bundle as the main source of wiring. Special skin temperature thermocouples were made for 16 points on the coke drums, 24 special flanged thermocouple assemblies of seven different types were made up for special requirements and the remainder of the thermocouples were standard items. Five duplex thermocouple assemblies were used to combine the indicating and controlling functions and thus reduce the cost of special wells. Flanged thermocouple assemblies were arranged to double as coke knocking openings. The multipoint precision indicator, a key switch assembly, and 24 point recorder were used to monitor the temperatures at vital points. Six standard dial thermometers and one special thermometer assembly were installed.

I.4.7. Temperature Control

Specifications include five temperature controllers mounted on the control board for fractionator top, fractionator chimney, soaker No. 1 outlet, soaker No. 2 outlet and steam superheater outlet.

Another temperature controller was added to provide a steam desuperheating station to cool superheated steam exhausted from the adiabatic heating coils to a safe temperature before allowing it to enter temperature piping of the 10 pound steam system.

Fast firing temperature control valves for process furnaces are normally pressure balanced regulators which are not available in the very small sizes required by the experimental unit. Needle control valves were substituted, and a gas pressure regulator was added on the Refinery fuel gas supply to the experimental unit. This system offers an unusual degree of flexibility, since the gas supply to the five gas firing valves can be lowered or increased to keep the control valves within control range. Also, the large gas pressure changes due to switching drums on the commercial coker are minimized.

Another addition was made to satisfy the requirements of the Refinery insurance underwriters. Safety devices were installed to automatically shut down the gas firing valve to the steam superheater for low flows in either the 150 pound or the 600 pound steam coils. A safety

hazard is associated with fired steam superheaters in that if the steam flow in the coil is reduced to a low level, due to coking for example, the pressures and temperatures in the tube could build up to the point of rupture. If such a rupture occurred, the loss of steam from the Refinery steam header could be great enough to endanger the entire Refinery.

I. 4. 8. Pressure Measurement

Combinations of high temperature, high pressure, and tendency of the process material to coke required "NAK" wafer type volumetric pressure transmitters in all cases except for fractionator pressure and the inlet pressure to both preheaters. Extreme variations of process pressures required duplication of all the "NAK" pressure elements for alternate ranges. All of the transmitters are indicating. The pressure transmitter for the pressure letdown valve was eliminated by manifolding the transmitted signal of the outlet of both soakers to this controller. In most cases coke knocking flanges were used to attach the "NAK" pressure measurement capsules to reduce coking effects. Standard bourdon tube pressure elements and standard pressure gauges were used wherever possible. Taylor Instrument Company was the only supplier to offer a proposal for pressure elements suitable for the operating conditions.

Draft gauges of a light duty type were selected in preference to the normal industrial quality.

I. 4. 9. Instrument Panel and Panel Mounted Instruments

Although the instrument panel and panel mounted instruments are more or less conventional, numerous deviations from normal practice were made in order to reduce the cost and to make the installation more flexible. The panel front is removable in three sections to permit major changes without disturbing the rest of the panel. The panel is piped with plastic tubing to facilitate changes. The key switch assembly doubles as a junction box for the 58 pair thermocouple bundle. The use of linear flow charts facilitates the fullest possible employment of two and three pen recorders, thus reducing the total number of instruments required. The 12 manual loading controls selected occupy a minimum panel space. Since available panel space was not large enough to include the coke drum level instruments, they were mounted on the commercial coker panel.

The instruments mounted on the panel must be flexible enough to accommodate any possible combinations of process equipment plus all operating process variables. Alternate charts and scales, selector switches for changing control points, replacement control modes and other items have been provided to satisfy some of these requirements. Controllers have also been added, relocated, switched services revised and, in some cases, the method of control can be varied to meet changing process conditions. If all else should fail, manual control is provided.

The instrument panel is shown in Figures 26 and 27.

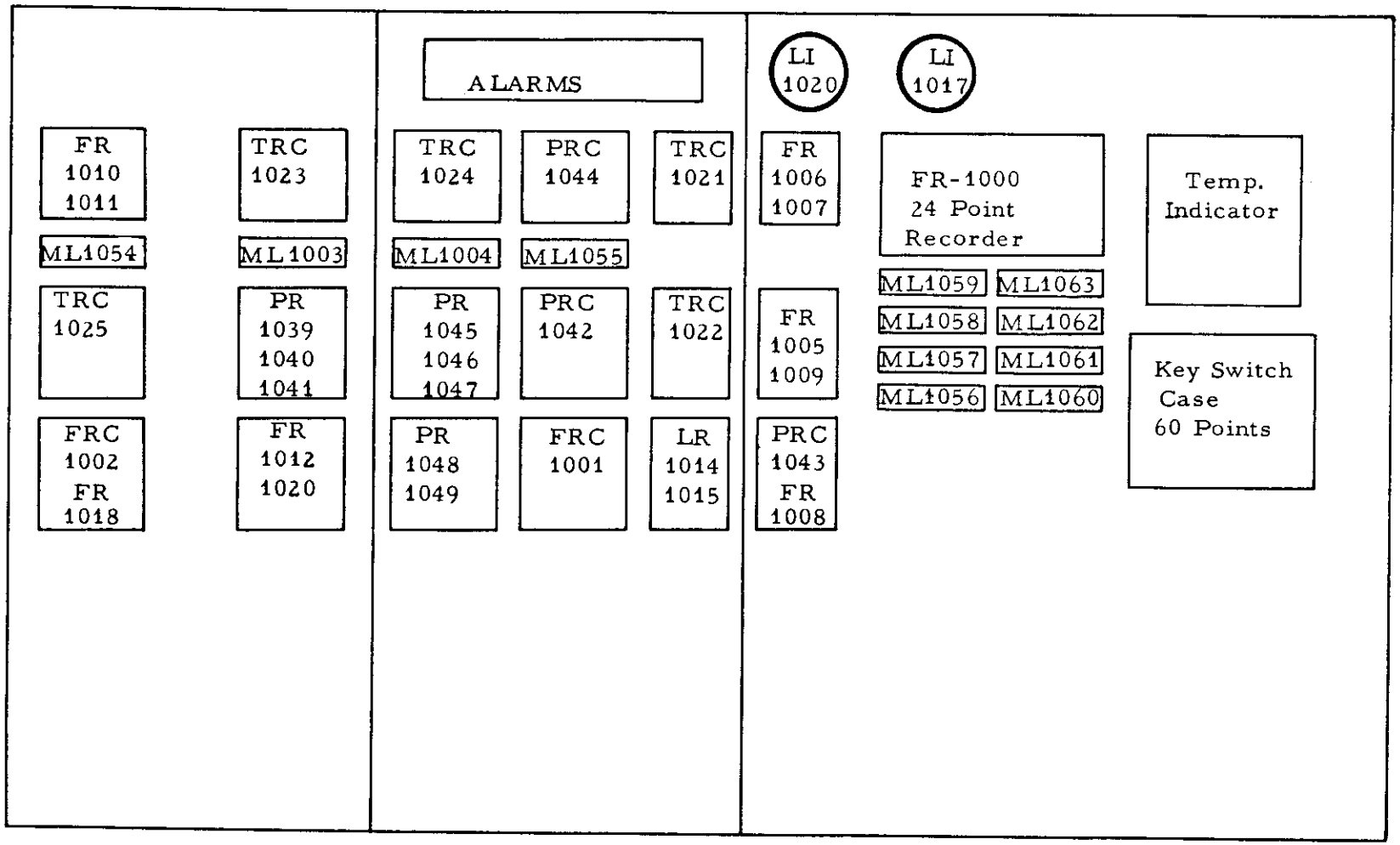

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Figure 26. Schematic Drawing of Instrument Panel

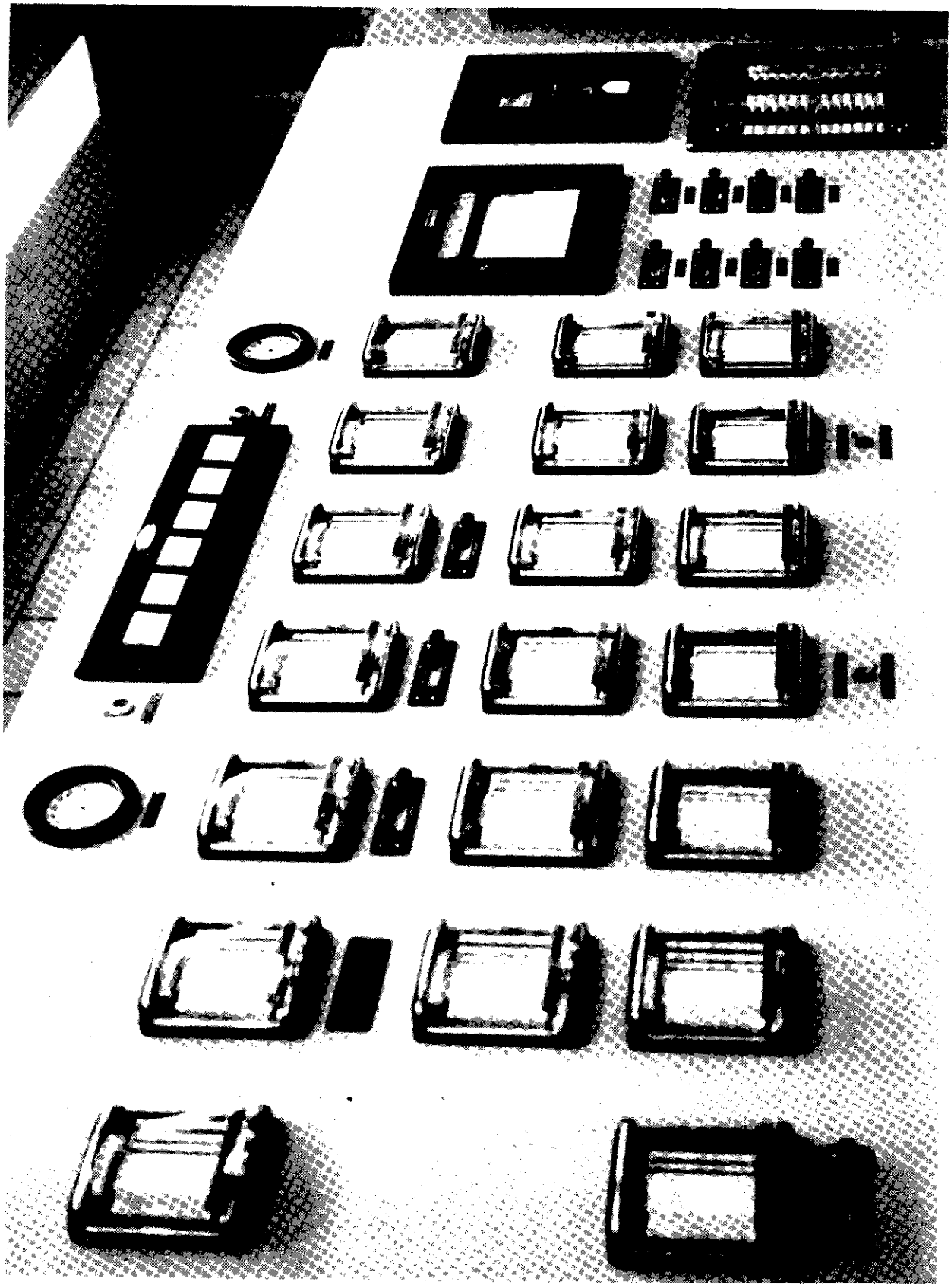

Figure 27. Photograph of Instrument Panel

I.5. Operational Procedures

I.5.1. Start Up Procedure (Using Evaporator)

After the various unit sections have been hydrostatically tested, the water is drained from the unit at all low points. All blinds are removed at the area limits except the fuel gas to heaters blind.

The unit is purged by introducing steam into the bottom of the fractionating tower. The 1-inch vent at the top of the evaporator is cracked.

The vent on the fuel gas line from the reflux accumulator to the knockout drums is then opened.

The pressure control manifold on fuel gas line is bypassed.

The vent on the experimental coker side of the fuel gas knockout drum is opened and the block valve on the gas knockout drum at the commercial coker is closed.

The vent on the gasoline reflux pump is opened.

Steam is backed down the gas oil takeoff line to the flushing oil and gas oil reflux pump.

The valve on gas oil product line to cooler is blocked.

All fresh feed inlets to the fractionator and the fresh feed pump are steam flushed. The drain at the discharge side of the fresh feed pump is opened.

The fractionator suction line to the thermal tar pump is steam purged. The discharge side of the thermal tar pump is blocked so steam will not enter the thermal tar cooler.

The line to the heater charge pump is steam purged with the block valve on the discharge side cracked to blow down.

Evaporator

If the evaporator is used the following procedure is followed:

With the vent on top of the evaporator cracked, steam is introduced into the bottom with the drain opened enough for condensate removal.

The steam is allowed to purge the quench oil line from the evaporator to the discharge side of the quench oil pump.

The steam is slowly turned off at the points leading to the evapo-

rator and fractionator while gas is introduced by closing the bleeder on the line to coker fuel gas knockout drum and opening the 1½-inch fuel gas line. As soon as a gas odor is detected at the vent on the reflux drum, the vent is closed so fuel gas will back further into the unit through the 3-inch line to the top of the fractionator tower. As soon as gas is detected at the vent on the fractionator tower overhead, the vent is closed to allow gas to back down through the tower and into the suction line of the gasoline reflux pump.

When the gas pressure begins to build on the tower and gas is detected on all bleeders, they are then blocked off and the unit is pressurized to fuel line pressure. The purging then continues from the fractionator bottom through the furnace charge pump through all four heaters to the master switch valve line.

With the vapor lines to the coke drums closed, the master switch valve is opened, allowing passage of purge gas to the common vapor line and to the evaporator. The evaporator and fractionator are interconnected by opening an angle valve to bypass the evaporator level control valve.

Gas oil is then introduced into the fractionator through the fractionator level control valve, and circulated through the unit, with the master switch valve set to bypass the coke drum.

The furnace housings are then purged with steam and the fires lighted. The temperatures (outlet) are raised 150 to 175°F per hour until oil at the bottom of the fractionator is holding at 700°F.

The coke drums are heated with superheated steam, which is introduced around the coke drum in coils (150 pound steam) and also through the coke drum (600 pound steam). Each coil has a manual loader for regulating the temperature of each coil. The 600 pound superheated steam passes up through the coke drum and out through the vapor line to the manifold, where it is blocked by two valves in the line and is routed to the blowdown system. The condensate from the drum is drawn off to the sewer.

When all temperatures on the coke drum show above 800°F, the furnace outlets are raised to at least 850°F and the charge stock circulation is started.

As the pressure rises on the unit, the pressure control valve is set on vapor out of the evaporator to hold back pressure on the coke drum. The pressure control valve on the line leaving the gasoline reflux drum is set to hold back pressure on the fractionating tower. The gas goes either to flare or to gas knockout (FA-7) at the commercial delayed coker.

If the coke drum is to operate at above 100 lbs/in^2 gauge, the pressure control valve is pinched to raise pressure on the evaporator before switching heater outlets into bottom of coke drum.

If light material starts to accumulate in the reflux drum, all moisture is drawn off to sewer and the gasoline reflux pump started to pump reflux back into the fractionator above the top tray.

When level shows in the gas oil pan on the level gauge, the suction line is opened to draw out any moisture on the quench pump. The suction line is flushed out to the evaporator line before too high a temperature has been reached. The gas oil reflux line into the fractionator is also flushed out.

When the pressures are up and heater outlets are up to temperature, the test is started by switching into the active coke drum.

Steam is cut under all relief valves in operation.

The blowdown valve is pinched down to raise the drum pressure (if it is below the desired point) well above the operating point and the 600 pound steam is cut out at the coke drum bottom (to pull pressure down to operating point) through the line to sewer or coke sluice, which allows any condensate to be removed from the coke drum before switching in with hot oil.

The inside and outside vapor line valves are opened slowly until wide open.

The switch valve is then slowly turned towards the drum that has been steam heated, the vapor manifold valve is turned wide open, and the block valve on the bypass line is closed.

The unit is then lined out on the desired flows, temperatures and pressures.

At this point, gas oil is being produced in the unit and the level control valve on gas oil to storage is lined up.

The level control valve on the evaporator (if being used) is adjusted to hold level.

Fresh feed flow to the unit is set by the flow rate controller and measured through the positive displacement meter.

I.5.2. Test Procedure

As soon as the unit is lined out(lining out usually covers a 24 hour period), the drums are switched in the procedure outlined above and the test begins. The test is made on either the north or south drum and a cycle number is given to the specific test. The cycle numbers are listed consecutively.

I. 5. 3. Shutdown Procedure on Coking Cycles

The coke drums are switched before starting procedure to bring the unit down.

While the heater outlets are riding on test temperatures, the combined feed is lowered 35 per cent by pinching hand control valves 1070 and 1071. The coke drum pressure is then lowered. If using the evaporator, the pressure is lowered on the evaporator through PCV-1044 in the control room until it is low enough so 150 pound steam can be used in the spool at the master switch valve.

The outlet heater temperatures are slowly lowered to 850°F, requiring 30 to 45 minutes.

The fresh feed is then switched to gas oil used in startup while the outlet heater temperatures are lowered to 750°F.

With temperatures riding at 750°F, a switch is made from the coke drum into the bypass line. If the evaporator is used, the switch is through bypass line from the back side of the switch valve into the vapor manifold and into the evaporator. Then the 1-inch drain line (to blowdown) on the bypass line is cracked and the block valve on the bypass line at the vapor manifold is slowly opened so that vapor will start to back down the line to blowdown. When this line is warm, the 1-inch line is closed and the block valve is fully opened. The master switch valve is moved toward bypass and out of the coke drum.

Raw 150 pound steam is now added to the coke drum through spool.

The operator on the vapor deck pinches down on the vapor line valves and starts opening valves to blowdown. The drum is completely depressurized through the line to blowdown.

With only gas oil circulating in the system, the outlet temperature on the furnaces is lowered toward 600°F.

At the lower temperature, the gas oil and gasoline product will fade out. The gasoline in the accumulator is then pumped back into the fractionator and the gas oil pan is pumped dry (with reflux pump 8JA) to the fractionator tray below the pan.

The thermal tar pump is lined up to take suction on the fractionator bottom and pump to blowdown in order to pull in fresh gas oil for circulation.

With temperatures holding at 600°F, the flow rates through the heaters are increased to original feed rate in order to wash out heater tubes and towers.

When the gravity of the gas oil fresh feed is equal to gravity in the fractionator (or evaporator), the unit is pumped to blowdown and the heater fires are cut.

The superheater steam is slowly cut out of the coils around the coke drum in order to cool evenly and minimize warpage.

All steam is cut out of unit.

All pumps are shut down and 150 pound steam is introduced into all furnace coils at 1-BA and 2-BA inlets, through to blowdown to the by-pass line below the block valve, back through the master switch valve to 1-BB outlet to blowdown, and to bottom of the fractionator.

The outside vapor line of each coke drum is opened at the manifold and steam is cut into the spools between the valves.

All water is shut off to condensers and coolers.

The water and gasoline are drawn out of the accumulator to the sewer.

The block valve at knockout drum is closed and the steam line connected to flush back through the accumulator.

Gas oil lines to the gas oil cooler and back to the suction side of the reflux pump are steamed down.

Steam is connected to the fresh feed line at the tank and flushed back to the fractionator.

Condensate is bled at all low points.

600 pound Steam Injection Points to Experimental Coker

1) Relief valve No. 1079 outlet of 2-BA heater.
2) Relief valve No. 1080 outlet of 2-BB heater.
3) Relief valve No. 1072 outlet of 1-BA heater.
4) Relief valve No. 1073 outlet of 1-BB heater.
5) Spool from master switch valve to coke drum 2-D.
6) Spool from master switch valve to coke drum 1-D.
7) Block valves from master switch valve to both coke drums.
8) Block valves above steam and water manifold on bottom of both coke drums.
9) Master switch valve lantern gland.
10) Coke drum bypass line valve.
11) Common vapor line valve.
12) Overhead vapor line block valves from both coke drums, all four valves.
13) Relief valve No. 1074 overhead vapor line from coke drum 1-D.

600 pound Steam Injection Points to Experimental Coker (Cont'd)

14) Relief valve No. 1075 overhead vapor line from coke drum 2-D.
15) Inside block valves on blowdown lines from both coke drums.
16) Sample draw block valve on coke drum overhead vapor line.
17) Relief valve No. 1070 on evaporator 2-E.
18) Sample cooler on outlet line on bottom of evaporator.
19) Both silicone injections.

150 pound Steam Injection

1) Sample cooler block valve on outlet of bottom of fractionator.
2) Both spools from master switch valve to coke drums.
3) Between coke drum overhead vapor line block valves from both drums.
4) Sample draw on vapor line from evaporator 1-E to fractionator 2-E.

I. 6. Maintenance Manual

A Plot Plan (Inspection Department)
B
C Equipment Summary and Item Numbers (Maintenance Department)
D Vessel Data (Area Engineer)
E
F Trolleys, Hoists and Slings (Area Engineer)
G Vessel Drawings (Inspection Department)
H Flange Data - 12 Inches and Larger (Area Engineer)
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L Heater Data (Inspection Department)
M Gasket Drawings (Area Engineer)
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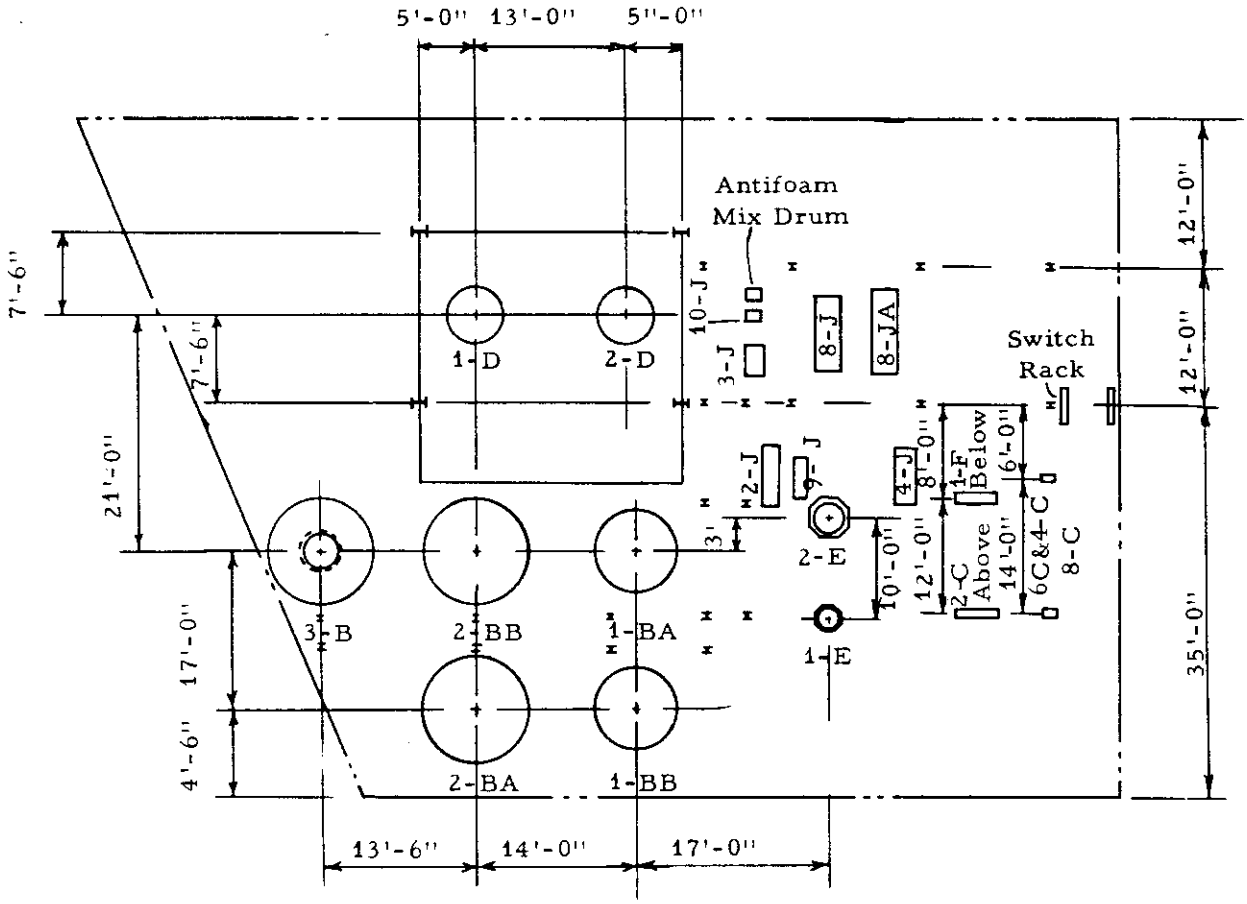
P Relief Valve Data (Inspection Department)
Q Blinding Schedule (Unit Foreman)
R Lubrication Charts (Inspection Department)
S
T Decoking Procedure
U Test Procedure (Inspection Department) See Section 4. 3.
V Inspection of Equipment (Inspection Department)
W Maintenance of Equipment (Inspection Department)
XYZ



1-BA	Preheater	1-E	Evaporator	10-J	Antifoam Pump
1-BB	Cracking Furnace	2-E	Fractionator	11-J	Coke Fines and Water Pump
2-BA	Preheater				
2-BB	Cracking Furnace	1-F	Reflux Drum		
3-B	Steam Superheater				
		2-J	Furnace Charge Pump		
2-C	Overhead Condenser	3-J	Condenser Pump		
4-C	Gas Oil Product Cooler	4-J	Gasoline & Reflux Pump		
6-C	Visbreaker Tar Cooler	8-J	Flushing Oil and Evaporator Quench		
8-C	Fresh Feed Preheater	8-JA	Gas Oil Reflux Pump		
		9-J	Visbreaker Tar Pump		
1-D	Coke Drum				
2-D	Coke Drum				

A. Equipment Plot Plan

C. Equipment Summary and Item Numbers

Vessels

1-D	North Coke Drum	6501
2-D	South Coke Drum	6502
1-E	Evaporator	6505
2-E	Fractionator	6506
1-F	Reflux Drum	6510

Exchangers

2-C	Overhead Condenser	6520
4-C	Gas oil Product Cooler	6522
6-C	Visbreaker Tar Cooler	6524
8-C	Fresh Feed Preheater	6526

Pumps

2-J	Furnace Charge Pump	6531
3-J	Condensate Pump	6532
4-J	Gasoline and Reflux Pump	6533
8-J	Flushing Oil and Evaporator Quench	6534
8-JA	Gas Oil Reflux Pump	6535
9-J	Visbreaker Tar Pump	6536
10-J	Antifoam Pump	6537
11-J	Coke Fines and Water Pump	6538

Heaters

1BA	Southeast Preheater	6541
1BB	Southwest Cracking Furnace	6542
2BA	Northwest Preheater	6543
2BB	Northeast Cracking Furnace	6544
3D	Steam Superheater	6545
Coke Drilling Equipment		6549
Large Piping		6550
Large Valves		6559
Flow Control Valves		6560
Flow Orifices and Measuring Tubes		6561
Blinding		6565

Relief Valves

Machine work only	6599
To be checked and inspected in place	6568
To be removed at turnaround, cleaned and tested	6569
To be cleaned and tested at turnaround and, if not operating properly the first time, replaced	6570
To be cleaned and tested (other than turnaround)	6571
Miscellaneous	6600
Requiring engineering and layout	6601
Testing vessels, lines or other test work not chargeable against any one definite item. Testing after unit is buttoned up comes under this item	6602
After unit is ready to start up, such work as tightening leaks on lines and vessels as required during startup period. Charges against this item should terminate immediately when unit is lined out	6603
Valve packing (all lines)	6613
Steam lines	6604
Oil lines	6605
Gas lines	6606
Water lines	6607
Air lines	6608
Electric utilities	6609
Chemical lines	6610
Insulation required to start up unit caused by TA. (Do not use this after unit is on stream)	6611
Charge to be used for supervision when unit is down. This is to include outside inspectors or any other charges that involve supervision	6612

D. Vessel Data

Item No.	6501	6502	6505	6506	6510
Vessel No.	1-D	2-D	1-E	2-E	1-F
Service	Coke Drum	Coke Drum	Evaporator	Fractionator	Reflux Drum
Inside Diameter, feet	4	4	24	2.67	1.94
Length Overall, feet	-	-	11.25	50	13.99
Length T to T feet	30	30	9.5	49.5	12
Design Pressure, lbs/in²	400	400	400	368	120
Design Temperature, °F	950	950	760	415	150
Shell Material	1 per cent Chromium ½ per cent Molybdenum A-387	1 per cent Chromium ½ per cent Molybdenum A-387	Steel A-204-B	Steel A-285 Grade C	Steel A-106
Shell Thickness	1.1875	1.1875	0.5625	0.3125 0.6875	0.375
Corrosion Allowance	0.25	0.25			

D. Vessel Data (Continued)

Item No.	6501	6502	6505	6506	6510
Vessel No.	1-D	2-D	1-E	2-E	1-F
Service	Coke Drum	Coke Drum	Evaporator	Fractionator	Reflux Drum
Code Stamp	Yes	Yes	Yes	No	Yes
Code	ASME	ASME	ASME	No	API-ASME
Stress Relieved	Yes	Yes	No	No	No
X-rayed	Yes	Yes	No	No	No
Heads - Type	2:1 Elliptical	2:1 Elliptical	Top-Blind Flange Bottom-2:1 Elliptical	Top-Blind Flange Bottom-2:1 Elliptical	Cone W/blind flange
Heads - Thickness, inches	1.14	1.14	Bottom 0.5625	Bottom 0.875	0.375 Cone
Heads -Material	1 per cent Chromium ½ per cent Molybdenum A-387	1 per cent Chromium ½ per cent Molybdenum A-387	Steel A-204-B	Steel A-285 Grade C	Steel A-106 Grade A
Number of Manholes	2	2	2	3	2
Size and Rating	48-inch 400 lbs/in² RJ	48-inch 400 lbs/in² RJ	24-inch 400 lbs/in² RF	2-18-inch 300 lbs/in² RF 1-36-inch 300 lbs/in² RF	16-inch 150 lbs/in² RF
Gasket Material	4-6 per cent Chromium ½ per cent Molybdenum	4-6 per cent Chromium ½ per cent Molybdenum	Corrugated Asbestos Filled	Corrugated Asbestos Filled	Spirotallic
Bolt Material	A-193-B-14	A-193-B-14			

F. Trolleys, Hoists and Slings

Coke Drum Heads

4) Wright Safeway hand hoists - 1¼ ton capacity with safety hook.
2) Wright geared trolleys - 2 ton capacity for 7-inch I beam.
2) Wright geared trolleys - 2 ton capacity for 12-inch I beam.
4) Rod type slings, 2 rods per unit with safety hook and turnbuckles on rods, 1½ ton capacity.

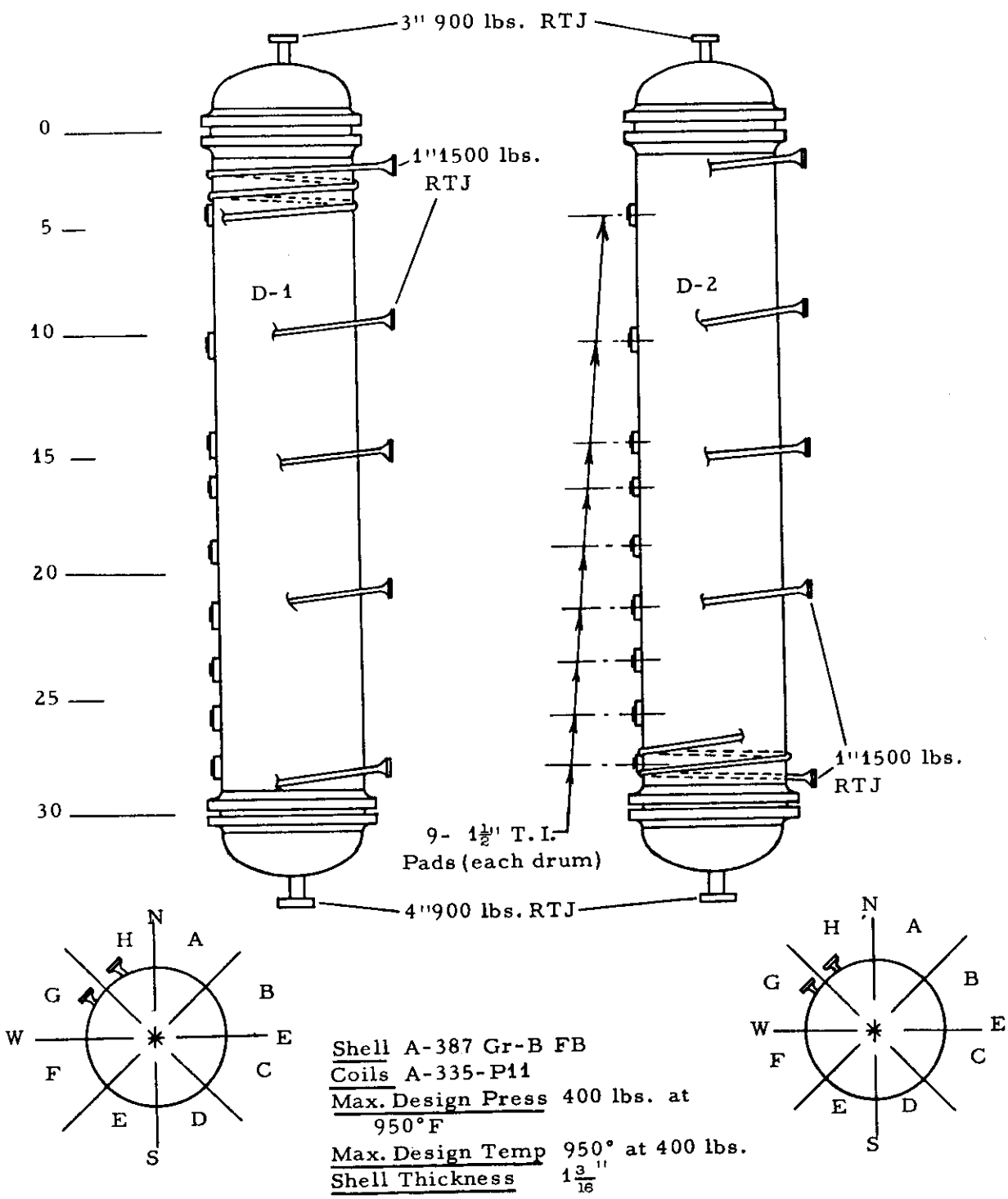


Figure 28. Schematic Drawing of Coke Drums - D1 and D2

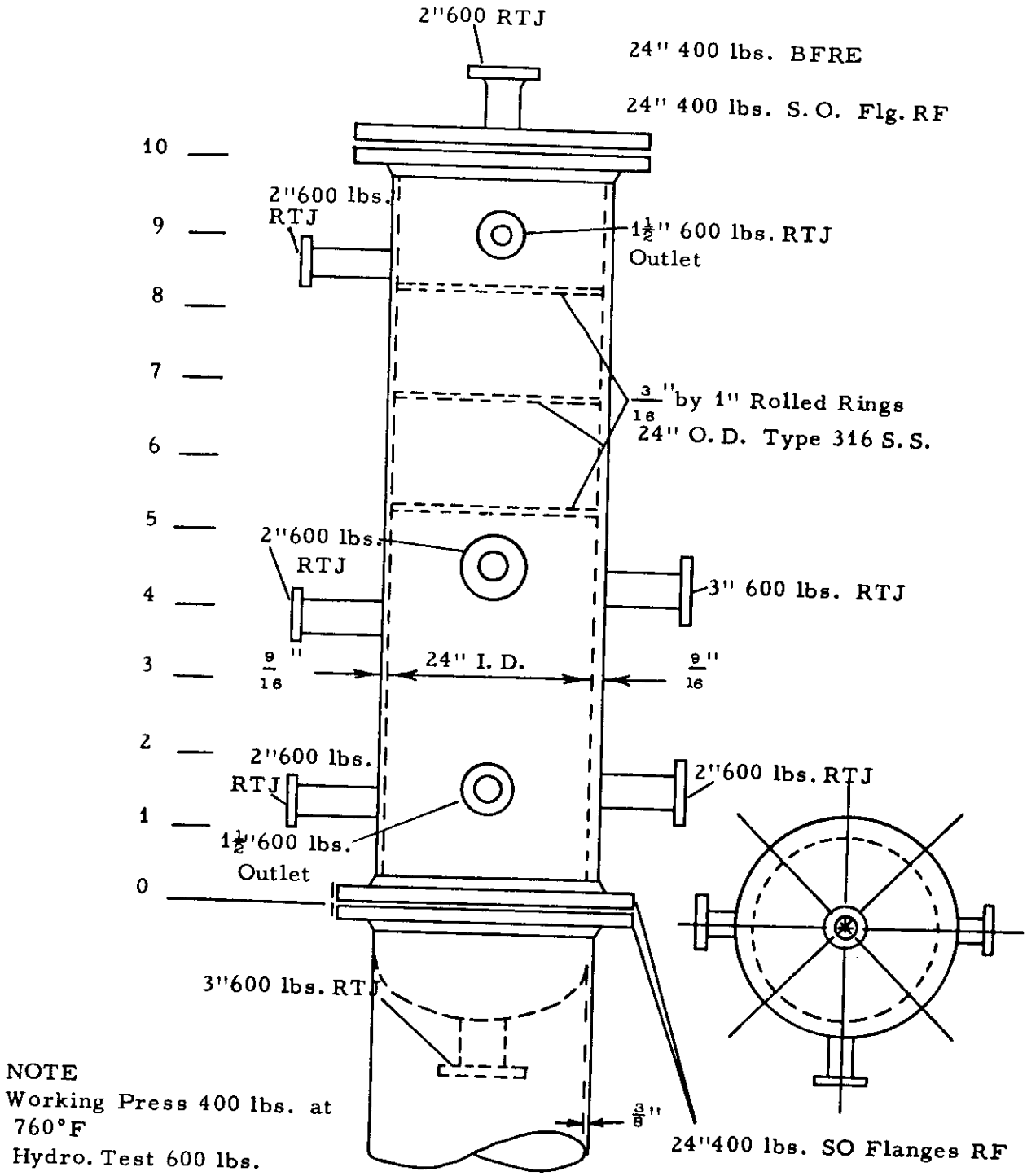


Figure 29. Schematic Drawing of Evaporator

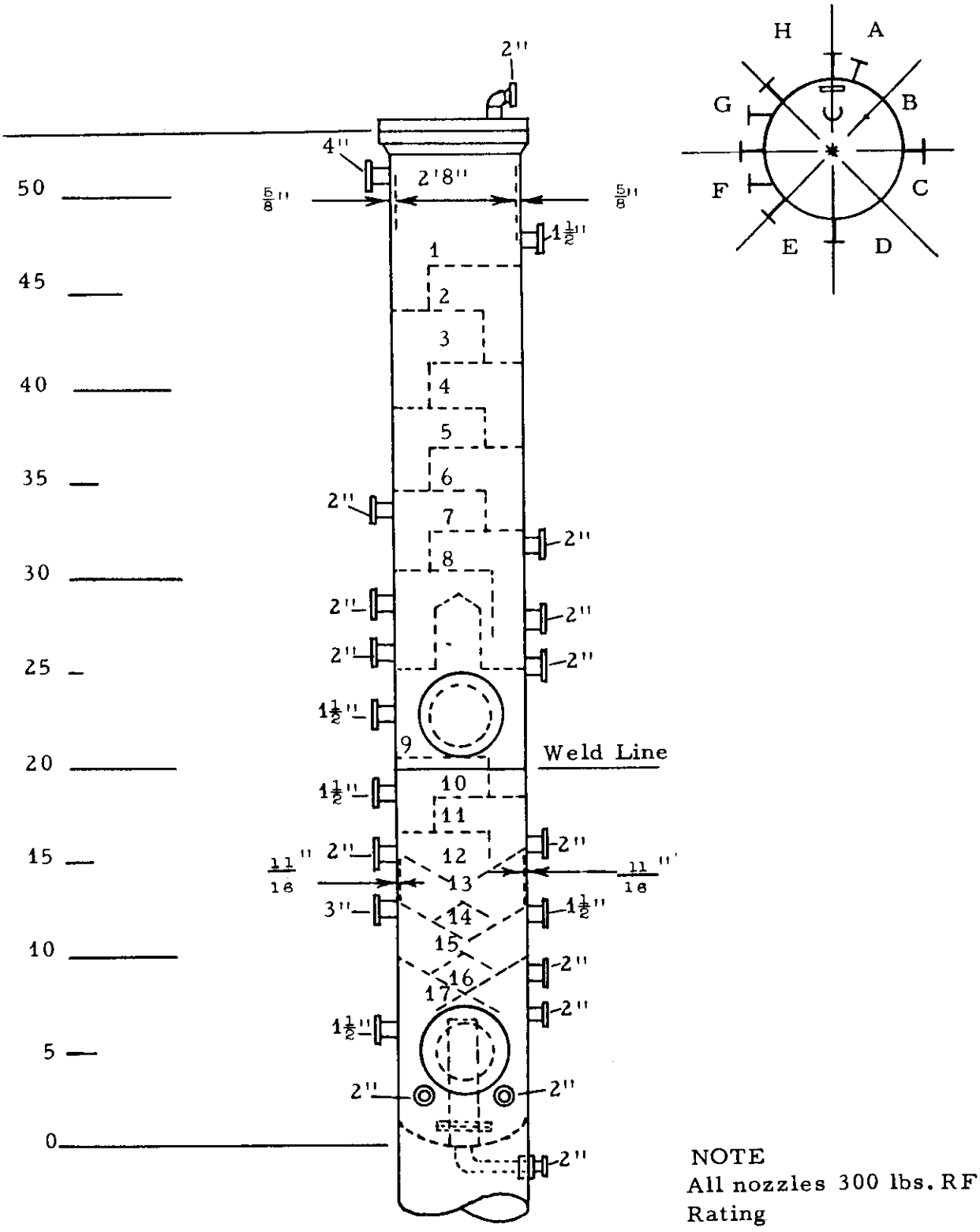


Figure 30. Schematic Drawing of Fractionator

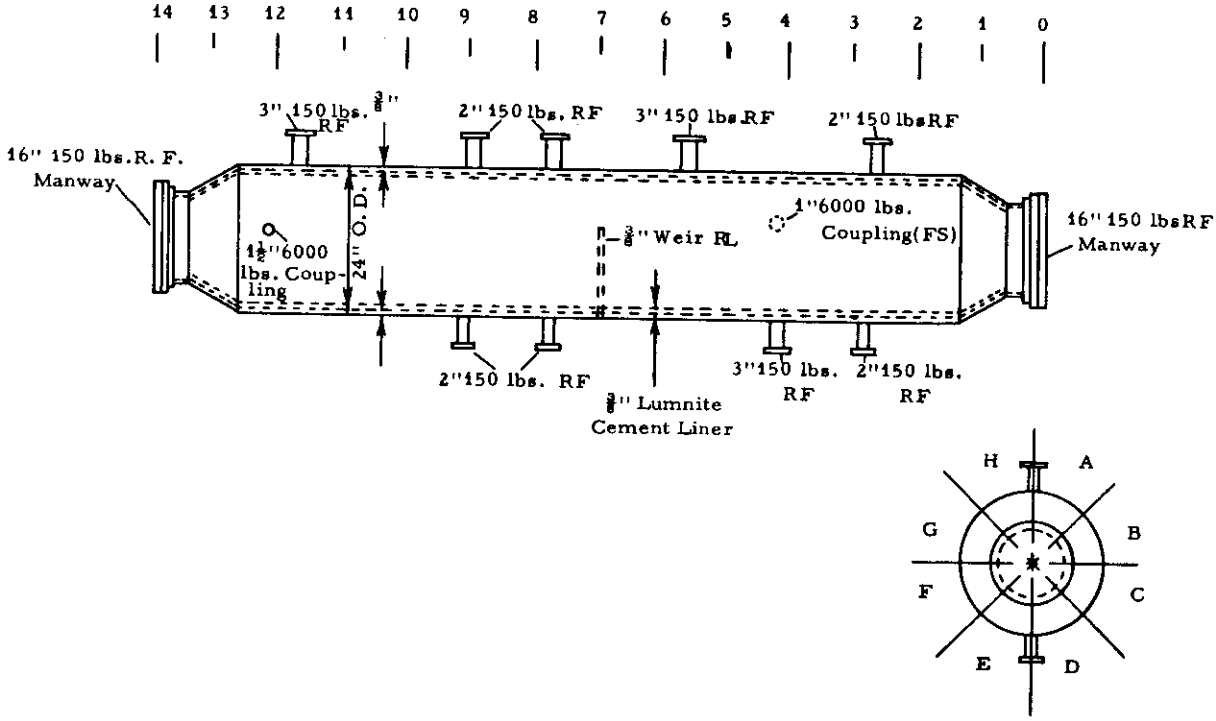
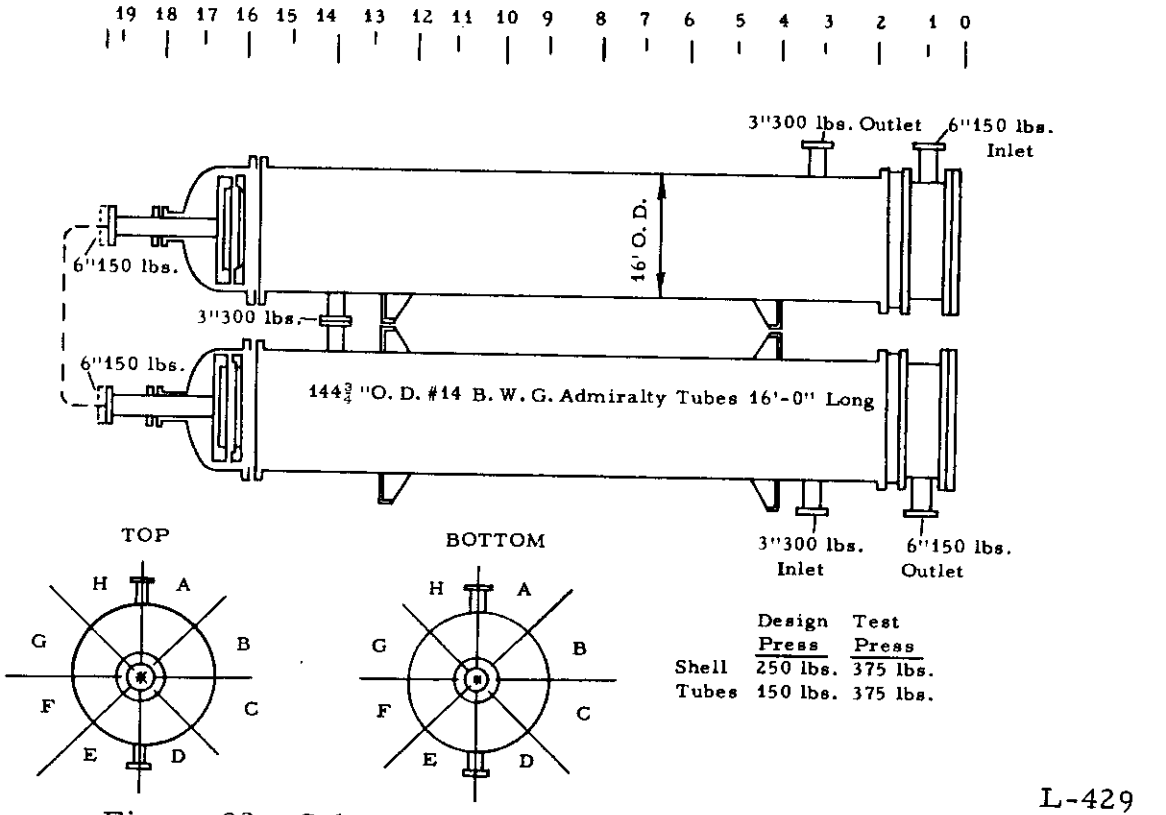


Figure 31. Schematic Drawing of Reflux Drum

L-428

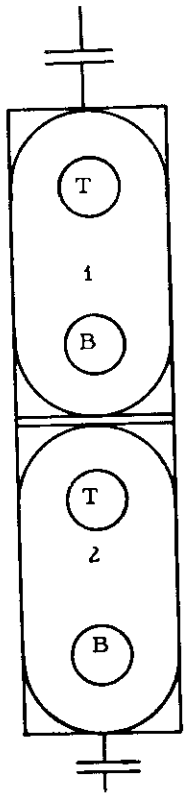


L-429

Figure 32. Schematic Drawing of Overhead Condenser

H. Vessel Flanges - 12-inch and Larger

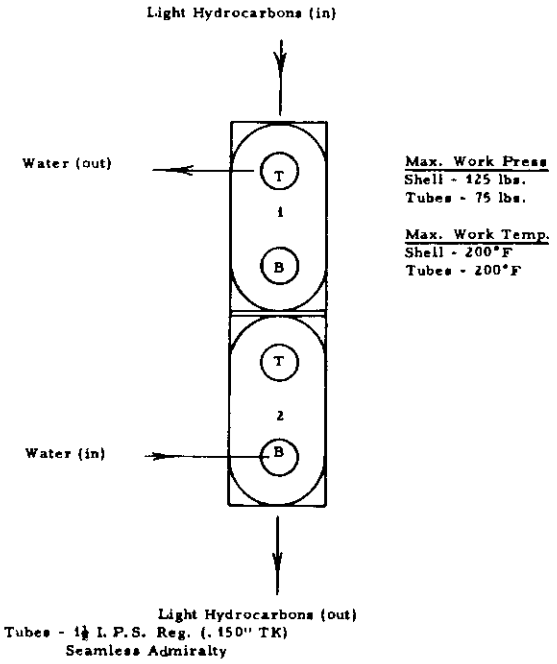
Vessel No.	Description	Item No.	No. of Manways	Manway Description	Gasket Size or Number	Gasket Material	Bolt Size: Number, Diameter, Length and Material (each manway)
1-D	North Coke Drum	6501	2	48-inch 400 lbs/in^2 RJ	9800-61	4-6 per cent Chromium $\frac{1}{2}$ per cent Molybdenum	(56) $1\frac{1}{4}$ by $13\frac{1}{2}$, A-193, B-14 (Nuts-A-194, Gr. 6)
2-D	South Coke Drum	6502	2	48-inch 400 lbs/in^2 RJ	9800-61	4-6 per cent Chromium $\frac{1}{2}$ per cent Molybdenum	(56) $1\frac{1}{4}$ by $13\frac{1}{2}$, A-193, B-14 (Nuts-A-194, Gr. 6)
1-E	Evaporator	6505	2	24-inch 400 lbs/in^2 RF	$25\frac{1}{4}$ by $30\frac{1}{4}$	Corrugated Asbestos Filled	(48) $1\frac{3}{4}$ by $10\frac{3}{4}$
2-E	Fractionator	6506	2	18-inch 300 lbs/in^2 RF 36-inch 300 lbs/in^2 RF	$17\frac{1}{4}$ by $23\frac{3}{8}$ $37\frac{17}{32}$ by $38\frac{16}{32}$	Corrugated Asbestos Filled	(24) $1\frac{1}{4}$ by $7\frac{3}{4}$
1F	Reflux Drum	6510	2	16-inch 150 lbs/in^2 RF	$16\frac{1}{4}$ by $20\frac{3}{16}$	Spirotallic	(16) 1 by $5\frac{1}{2}$



	O. D.	I. D.	Material	Design Press	Test Press	Design Temperature
Shell	3.5	3.068	Steel	500 lbs.	750 lbs.	700°F
Tube	1.9	1.610	Seamless Steel	500 lbs.	750 lbs.	700°F

L-430

Figure 33. Schematic Drawing of Gas Oil Product Cooler



L-431

Figure 34. Schematic Drawing of Fuel Oil Cooler

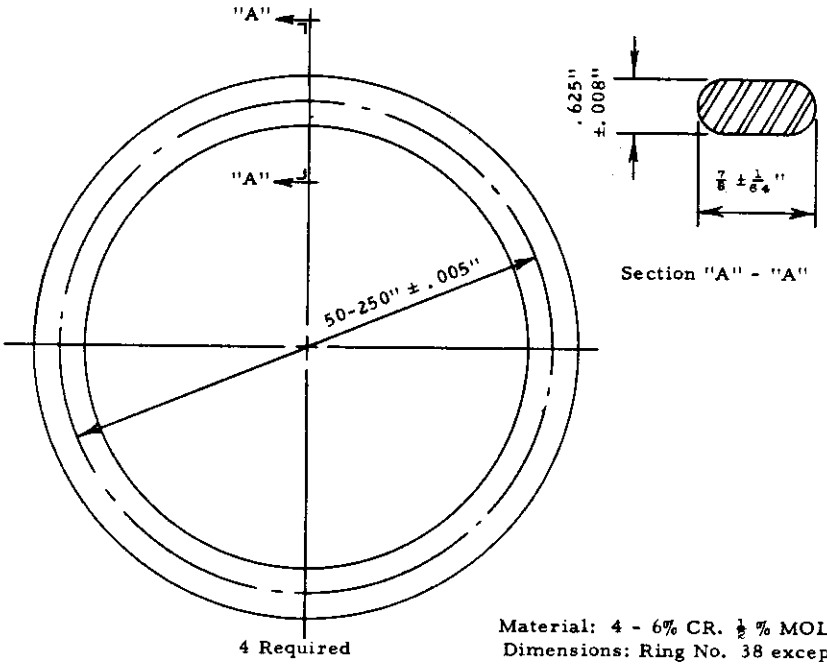
J. Exchanger Data

Name of Exchanger	Overhead Condenser	Visbreaker Tar Cooler	Gas Oil Product Cooler	Fresh Feed Preheater
Number	2-C	6-C	4-C	8-C
Number of Sections	2	2	2	1
Manufacturer	M. W. Kellogg Co.	Griscom Russell	Brown Fintube	Griscom Russell
Serial Number	1967 and 1977	63103A, 63104A	1293-17 and 22	85769
Shell				
Design Temperature, °F	670	200	750	650
Design Pressure, lbs/in²	250	125	500	1200
Test Pressure, lbs/in²	200	150	200	200
Size				
Diameter	16-inch OD	3½-inch OD	3½-inch OD	3⅝-inch OD
Length, feet	15.65	22.43	21	20
Thickness, inches	0.375	0.216	0.216	0.284
Material	16-inch OD by ⅜-inch L. W. Steel Pipe	Steel Pipe	Steel Pipe	Steel
Tube Bundle				
Design Temperature, °F	150	200	150	406
Design Pressure, lbs/in²	150	75	500	990
Test Pressure, lbs/in²	75	75	75	900
Tube Size				
Number Required	144	2	2	1
OD by Gauge	¾-inch by 14-inch Birmingham Wire Gauge	1.9-inch by 0.145-inch thickness	1.9-inch by 0.290-inch thickness	1½-inch Schedule 80
Length	16 feet	21 feet, 11 inches	21 feet	20 feet
Material	Admiralty	Admiralty	Seamless Steel	Admiralty(CAA-248)
Number of Fins		32	28	
Material		Admiralty	½-inch by 0.035-inch Steel	Admiralty
Gasket Drawings	133512-KA 133512-LA			9800-71

L. Heater Data

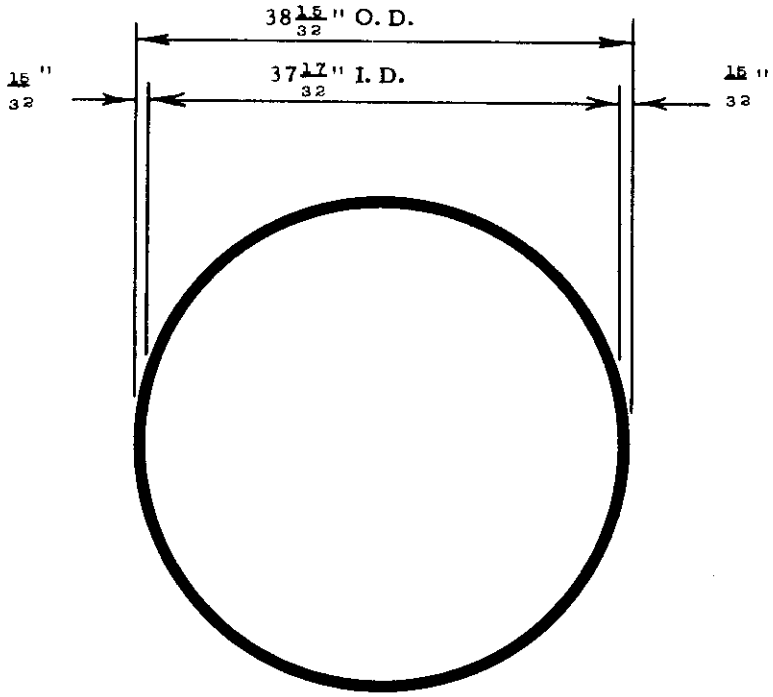
Item Number	6541	6542	6543	6544
Vessel Number and Service	1-BA Preheater	1-BB Cracking Furnace	2-BA Preheater	2-BB Cracking Furnace
Manufacturer	Yuba Heat Transfer	Yuba Heat Transfer	Yuba Heat Transfer	Yuba Heat Transfer
Serial Number	62-F159-1BA	62-F-159-1BB	62-F-159-2BA	62-F 159-2BB
Year Built	1962	1962	1962	1962
Purchase Order	RX 45651	RX 45651	RX 45651	RX 45651
Design Pressure lbs/in^2 gauge	1000	1000	1000	1000
Design Temperature °F	1000	1000	1000	1000
Tube Size and Material	0.840-inch OD by 0.147-inch Wall Alloy Steel A-335-P-5	0.840-inch OD by 0.147-inch Wall Alloy Steel A-335-P-5	1.05-inch OD by 0.154-Wall Alloy Steel A-335-P-5	1.05-inch OD by 0.154-inch Wall Alloy Steel A-335-P-5
Test Pressure lbs/in^2	2000	2000	2000	2000
Code	No	No	No	No
Stamped	No	No	No	No
Burner	John Zink Co. VBMR-12	John Zink Co. VBMR-12	John Zink Co. VBMR-14	John Zink Co. VBMR-14
Header Inlet	½-inch 1500 pound RJ A-182-F-5	½-inch 1500 pound RJ A-182-F-5	¾-inch 1500 pound RJ A-182-F-5	¾-inch 1500 pound RJ A-182-F-5
Header Outlet	½-inch 1500 pound RJ A-182-F-5	½-inch 1500 pound RJ A-182-F-5	¾-inch 1500 pound RJ A-182-F-5	¾-inch 1500 pound RJ A-182-F-5

Item Number	6545
Vessel Numb and Service	3-B Steam Superheater
Manufacturer	Yuba Heat Transfer
Serial Number	62-F-159-3
Year Built	1962
Purchase Order	RX 45651
Design Pressure lbs/in^2 gauge	600 pound Coil - 675 150 pound Coil - 300
Design Temperature °F	600 pound Coil - 1200 150 pound Coil - 1200
Tube Size and Material	600 pound Coil - 1.05-inch OD by 0.114-inch Wall Stainless-SA-213-TP-316, 11 square feet 150 pound Coil - (5 coils from inlet, 4-inch OD by 0.318-inch Wall, Carbon Steel, SA-53, Gr. B.)(6th through 10th coils, 4-inch OD by 0.320-inch Wall, Alloy Steel, SA-213, T-22)(11th through 17th coils, 4-inch OD by 0.400-inch Wall, Stainless, SA-213, TP-316) 346 square feet
Test Pressure	(DO NOT TEST SUPERHEATER)
Code	ASME
Stamped	Yes
Burner	John Zink Co., VBMR-20
Header Inlet	600 pound Coil - ¾-inch 6000 pound Threaded F.S. coupling 150 pound Coil - 3-inch 300 pound RF - Schedule 80 bore, SA-181, Gr. I.
Header Outlet	600 pound Coil - ¾-inch 6000 pound Threaded F.S. coupling 150 pound Coil - 4-inch 1500 pound RJ - SA-182, F-316



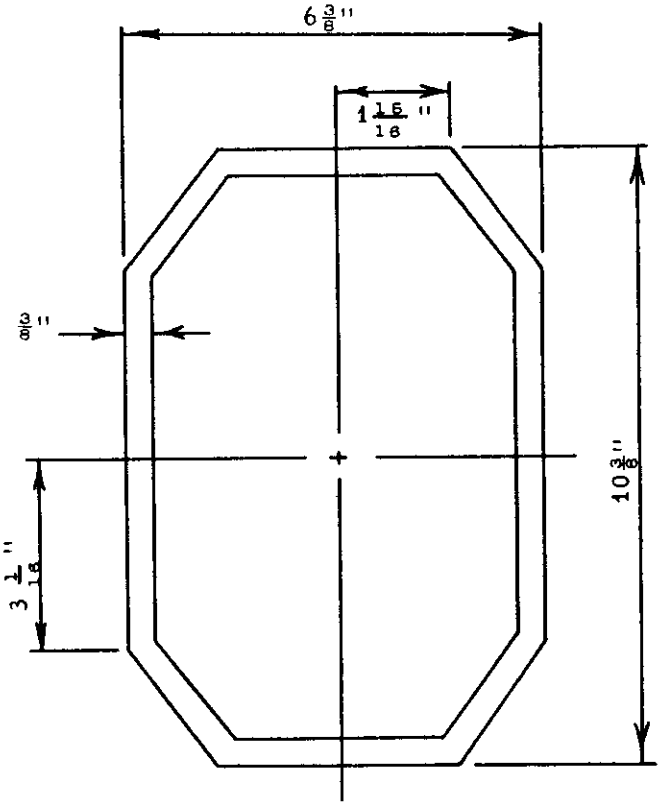
Material: 4 - 6% CR. ½ % MOLY.
Dimensions: Ring No. 38 except
20-250" ± .005" P. D.

Figure 35. Coke Drum Head Gaskets L-438



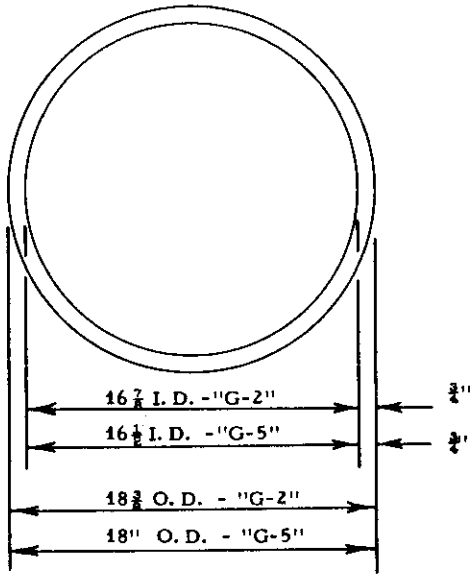
Material: ⅛" Thick Soft Steel
Maximum Hardness No. 86 Brinell - 1 Required

Figure 36. Fractionator Top Head Gaskets L-439



L-440

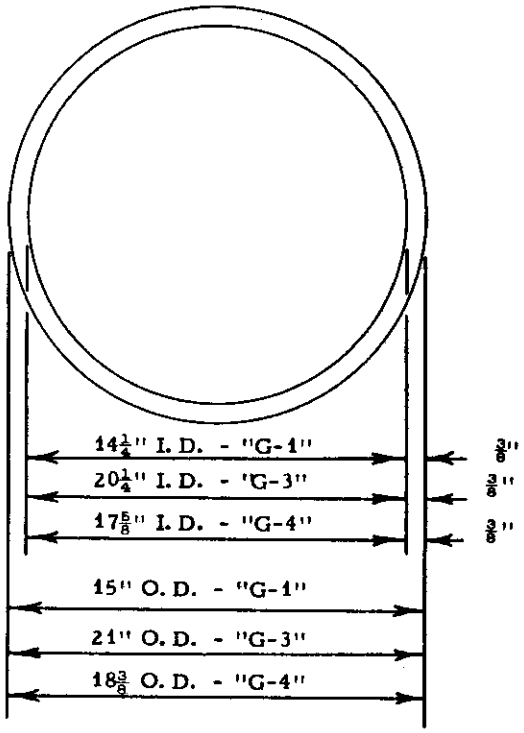
Figure 37. Fresh Feed Preheater Shell Cover Gasket



Material: 1/8" Armco-Asbestos Filled
Goetze "K" or Equal
"G-2" - For Stationary Tube Sheet and Channel-2 Required
"G-5" - For Channel and Channel Cover - 2 Required

L-441

Figure 38. Exchanger 2C (Overhead Condenser) Gaskets



Material: ⅛" Armco-Asbestos Filled
Goetze "K" or Equal
"G-1" - For Floating Tube Sheet and Cover - 2 Required
"G-3" - For Bonnet and Shell Flante - 2 Required
"G-4" - For Stationary Tube Sheet and Shell Flange - 2 Required L-442

Figure 39. Exchanger 2C (Overhead Condenser) Gaskets

P. Relief Valves

Relief Valve Number	Service	Cold Set Pressure, pounds
RV-1064	Residuum Feed Pump 1-J	309
RV-1065	Fractionator 2-E	108
RV-1067	Flushing Oil and Evaporator Quench Pump 8-J	1200
RV-1068	Gas oil Reflux Pump 8-JA	155
RV-1069	Visbreaker Tar Pump 9-J	155
RV-1070	Evaporator 1-E	412
RV-1071	Furnace Charge Pump 2-J	1250
RV-1072	Preheater 1-BA	1200
RV-1073	Cracking Furnace 1-BB	1200
RV-1074	Coke Drum 1-D	412
RV-1075	Coke Drum 2-D	412
RV-1076	150 pound Steam Superheater 3-B	210
RV-1077	600 pound Steam Superheater 3-B	709
RV-1078	Visbreaker Tar Cooler 6-C (Water Side)	75

P. Relief Valves (Continued)

Relief Valve Number	Service	Cold Set Pressure, pounds
RV-1079	Preheater 2-BA	1030
RV-1080	Cracking Furnace 2-BB	1030
RV-1081	Gas oil Product Cooler 4-C (Water Side)	75
RV-1082	Overhead Condenser 2-C (Water Side)	75
RV-1083	Circulating Pump 2101J	40
RV-1084	Condensate Injection Pump 3-J	50
RV-1085	Antifoam Pump 10-J	500

Q. Blinding

Tag Number	Description	Size and Type
Blinds used when shutting down		
2-inch JFG	Fuel Gas to Unit	2-inch 150 pound RF
2-inch J5A	Gas Oil to Unit	2-inch 150 pound RF
4-inch J22A	Glare Line	4-inch 150 pound RF
3-inch J3A	Visbreaker Tar	3-inch 150 pound RF
	Gasoline Product -	No blind will be used. Part union at big coker.
	Water to H2S Stripper -	No blind will be used. Part union at bottom of absorber water draw.
2-inch J18A	Gas to Coker Fuel Gas Knockout Drum	2-inch 150 pound RF
4-inch J11A	Blowdown	4-inch 300 pound RTJ

List of Goggle Eye Blinds

Tag Number	Number	Rating
3-inch J-7B	1	300 pound RTJ
3-inch J-7D	1	300 pound RTJ
1½-inch J-9	5	1500 pound RTJ
1½-inch J-8	1	1500 pound RTJ
2-inch J-8	1	1500 pound RTJ
3-inch J-10	5	300 pound RTJ

R. Lubrication Charts (Inspection Department)

Pumps

Equipment Service	Item No.	Equipment Lubricant	Driver Lubricant	Other Lubricant
Residuum Feed Pump	1-J	601 Mara. End. SAE 20W	1212 Marathon Balrax	Gear Case - 602 Marathon End. SAE 30W
Furnace Charge Pump	2-J	None	73 Marathon Cylona	
Condensate Injection Pump	3-J	1212 Marathon Balrax	1212 Marathon Balrax	
Gasoline and Reflux Pump	4-J	742 Marathon Turbine Oil	1212 Marathon Balrax	
Flushing Oil Pump	8-J	None	73 Marathon Cylona	
Gas Oil Reflux Pump	8-JA	None	73 Marathon Cylona	
Visbreaker Tar Pump	9-J	None	73 Marathon Cylona	
Antifoam Pump	10-J	1212 Marathon Balrax	1212 Marathon Balrax	Speed Reducer and Rack and Pinion Housing - 573 Mara. M. P. Lubricant
Circulating and Transfer Pump	2101-J	601 Mara. End. SAE 20W	1212 Marathon Balrax	
Sump Pump		1212 Marathon Galrax	1212 Marathon Balrax	Grease 2 times weekly

Drill Mechanism

Equipment Service	Item No.	Equipment Lubricant	Driver Lubricant	Other Lubricant
Drill Stem Motor (Swivel Joint and Crosshead)				
Motor Lubricator		741 Marathon Turbine Oil		Check Daily
Motor Crankcase		573 Mara. A. P. Lubricant		Check Daily
Reduction Gear Case		Summer-573 Mara. A. P. Lubricant Winter-571 Mara. A. P. Lubricant		Check Weekly
Water Line Swivel Joints		526 Mara. G. P. Lubricant		Grease Weekly
Air Line Swivel Joints		526 Mara. G. P. Lubricant		Grease Weekly
Crosshead		1212 Marathon Balrax		Grease Weekly
Hoisting Motor and Travel Motor				
Hoisting Motor Crankcase		601 Mara. End. SAE 20W		
Travel Motor Crankcase		601 Mara. End. SAE 20W		
Travel Motor Bearing		1212 Marathon Balrax		Grease 2 Times Per Week
Hoisting Motor Bearing		1212 Marathon Balrax		Grease 2 Times Per Week
Drill Guides		1399 Mara. D. G. Lubricant		

T. Decoking Procedure

1) Connect decoking piping to heater inlets and outlets as required.

2) Establish steam flow through all heater passes to remove possible obstructions. Steam pressure at the heater inlet should be 60 to 125 lbs/in^2.

3) Light the heater. Bring the heater temperature up at the rate of 250°F per hour for all heaters. Line out the furnace manually to obtain a uniform temperature throughout. The burner flame should be spaced over heater so that tubes are heated as uniformly as possible. The heater should be heated until skin-temperature thermocouples read 1050 to 1100°F. At this point the tubes are still dark in color but are near the temperature at which they change to a dull red.

4) Coke will begin to spall at coke temperature of 900°F with the steam in the tubes. Spalling of coke from tube should continue until the outlet looks clean. Reverse the steam flow about every thirty minutes during this operation.

5) Next, introduce air with the same steam valve setting and the same burner settings in the heater so that the heater pressure is raised 10 to 20 lbs/in^2. The usual weight ratio of steam to air is 9:1. Burning will usually start shortly and the burning progress can be observed through the heater. The temperature of the burning front should not get above 1300°F. The tube color should change from dark to dull red to cherry red as the burning front passes. If the tube gets whiter than cherry red, cut back on the air to obtain the proper air to steam ratio. As soon as burning starts, the outlet gas will be red. If the temperature rise at any point is too fast, the air should be cut back to obtain a lower air to steam ratio. About every ten minutes, the steam valve should be opened wide to blow out any loose particles of material. The flow through the heater should be reversed every thirty minutes. When no red or burning spots appear and a red gas is discharged, the heater can be assumed to be decoked.

6) The heater is then cooled at a 250°F per hour rate from 1050°F to 500°F, at which time the fires can be cut off.

7) The heater is then uncapped as required for inspection.

Precautions for Decoking

1) Be sure steam is passing through all passes of heater when

the heater is lighted. Steam should be going through all passes of heater at all times when the fires are burning.

2) Light as many burners as possible so that the tube metal is 1050°F over their entire length.

   a) Raise the temperature of the heater at a rate of 250°F per hour above 500°F.

3) Hold the tube temperature at 1050 to 1100°F with steam only through tubes.

4) Do not let tube temperature get above 1300°F when decoking is proceeding.

5) Cool heater at a rate of 250°F per hour to 500°F, then cut off fires.

V. Inspection of Equipment

1) Inspection of Process Equipment

Inspected by	Frequency
Fireman	Once per shift
Stillman	Once per day
Foreman	Once per week
Engineering	Upon request of Unit Foreman or at shutdown*

* Shutdown - Time interval varies from one to three months.

W. Maintenance of Equipment

The maintenance work performed on the experimental coking unit and its allied components is carried out by the Maintenance Department of the Marathon Oil Company at Robinson, Illinois. The scope of the work consists of machining, electrical, intrumentation, carpentry, painting,boilermaking, labor, welding and insulating, all of which are available for servicing the unit upon request of the Unit Foreman or at shutdown*.

* Shutdown - Time interval varies from one to three months.

APPENDIX II

EXTENSIVE TEST DATA

Table 10. Extensive Test Data - Flow Data

Cycle Number	Steam into Transfer Line		Time hrs.	Rate lb/hr.	Cycle Time hrs.	Fresh Feed b/d	Combined Feed b/d	Recycle Ratio
		Type						
Charge Stock - Vacuum Residuum								
6N	No				26	111	201	0.81
8S	No				24	99	201	1.03
9S	No				24	49	101	1.06
10S	No				24	51	101	0.98
11S	No				24	95	201	1.12
12S	No				24	53	102	0.93
26S	Yes	600 pound	25.75	300	25.75	127	150	0.18
27N	Yes	Superheated	26	300	26	125	150	0.20
27S	Yes	Superheated	26.25	300	26.25	120	150	0.25
28N	Yes	Superheated	24	300	24	122	150	0.23
30S	No				24	114	150	0.31
31S	No				24	103	200	0.94
32S	No				24	98	200	1.04
40S	No				24	50	100	1.00
41S	No				24	51	124	1.43
Charge Stock - Slurry Oil								
13S	No				24	92	201	1.19
14S	No				23	103	202	0.96
15S	No				21.5	102	201	0.97
16S	No				23	100	201	1.01
17S	No				24	100	201	1.01
18N	No				24	100	201	1.01
18S	No				24	99	201	1.03
19S	No				24	67	190	1.83
20S	No				20	48	201	3.17
22N	No				24	75	150	1.00
23S	No				23	75	150	1.00
24S	No				22.5	75	150	1.00
25S	No				22	74	150	1.03
38N	No				24	76	150	0.98
39N	No				24	75	150	1.00
Charge Stock - Thermal Tar (Pure Oil Co.)								
33S	No				24	77	150	0.95
34S	No				24	76	125	0.65
Charge Stock - Thermal Tar (Marathon Oil Co.)								
42S	No				24	69	150	1.17
43S	No				24	72	150	1.08
44S	No				24	62	124	1.00

Table 11. Extensive Test Data - Pressure Temperature Profiles

Cycle Number	Pressures (Outlet) 1BA	1BB	2BA	2BB	Drum	Temperatures (Outlet) 1BA	1BB	2BA	2BB	Coke Drum Inlet	Coke Drum Outlet	Calculated Space Velocity hr^{-1}
Charge Stock - Vacuum Residuum												
6N	455	280	227	75	52	630	705	817	935	893	805	18
8S	485	317	260	143	105	625	704	815	935	890	740	18
9S	265	225	220	120	102	620	710	815	935	880	790	8
10S	250	195	170	70	50	620	710	814	935	885	785	8
11S	480	320	260	75	50	660	713	225	950	906	800	17
12S	250	210	185	73	50	635	712	835	950	895	750	9
26S	394	240	210	71	60	628	710	832	930	885	806	13
27N	385	235	207	90	63	634	710	825	930	888	797	13
27S	375	226	197	70	55	624	710	819	930	873	785	13
28N	377	230	200	102	70	634	711	812	930	885	748	13
30S	376	233	200	70	60	630	704	807	930	875	790	13
31S	690	573	436	310	250	634	703	822	935	900	806	18
32S	713	517	460	360	350	631	703	816	935	900	803	18
40S	726	669	296	60	50	686	784	879	936	883	787	7
41S	847	774	333	65	51	679	703	883	947	900	787	8
Charge Stock - Slurry Oil												
13S	460	320	265	75	50	630	712	825	950	917	830	23
14S	485	340	255	130	100	630	710	827	950	912	830	23
15S	660	500	470	125	100	635	705	815	935	906	825	25
16S	500	350	300	80	50	625	705	805	935	900	825	27
17S	550	400	350	85	50	620	710	820	950	920	835	24
18N	530	380	330	85	50	624	711	820	950	920	840	24
18S	565	410	365	85	50	635	711	820	950	918	835	24
19S	590	430	345	160	100	610	718	823	965	935	825	23
20S	632	475	425	125	100	635	712	878	950	923	840	23
22N	380	278	260	160	150	630	712	830	950	913	812	17
23S	460	340	335	225	258	640	715	827	950	913	809	17
24S	553	435	415	360	350	629	715	834	950	905	798	16
25S	510	423	419	353	350	620	710	819	935	895	788	18
38N	530	439	374	79	50	681	782	937	937	903	828	11
39N	602	495	403	75	50	737	876	935	936	900	820	9
Charge Stock - Thermal Tar (Pure Oil Co.)												
33S	400	310	270	110	100	628	704	820	950	916	810	18
34S	519	453	432	370	350	625	706	810	950	916	780	15
Charge Stock - Thermal Tar (Marathon Oil Co.)												
42S	810	748	240	64	50	635	708	819	936	906	815	17
43S	940	892	363	66	50	894	783	921	936	897	814	11
44S	-	1018	396	60	50	738	877	936	936	899	811	8

Table 12. Extensive Test Data - Laboratory Examination of Feed and Overhead Products

Cycle Number	Gravity, API Fresh Feed	Gravity, API Combined Feed	GASOLINE Gravity API	GASOLINE Distillation (ASTM) °F IBP*	10 %	50 %	90 %	EP**	GAS OIL Gravity API	Flash COC °F***	Conradson Carbon, %	GAS OIL Distillation (ASTM) °F IBP*	10 %	50 %	90 %	EP**
Charge Stock - Vacuum Residuum																
6N	-	-	-	98	158	276	400	420	30.2	175	0.087	348	466	552	670	726
8S	12.8	14.8	63.0	98	162	296	410	426	31.2	155	0.010	318	470	520	580	640
9S	13.1	16.3	62.4	92	152	308	442	446	29.9	150	0.009	326	470	520	570	638
10S	12.9	16.1	64.2	94	130	228	360	384	36.7	130	0.052	324	384	444	548	644
11S	13.0	14.1	64.2	90	146	246	338	358	33.4	140	0.040	340	428	492	572	634
12S	12.6	12.1	64.5	90	146	250	358	376	30.4	175	0.021	308	438	500	598	678
26S	12.4	13.6	55.4	108	198	380	534	556	22.6	300	0.233	540	630	668	698	790
27N	13.7	13.8	56.1	100	182	346	494	528	23.5	300	0.264	492	592	696	788	814
27S	13.7	11.8	55.4	116	192	344	470	502	25.8	230	0.076	484	570	652	748	780
28N	13.8	14.3	55.1	106	204	396	538	568	24.2	350	0.070	526	628	690	770	810
30S	12.3	14.2	62.7	94	158	256	350	386	40.6	95	0.004	242	356	416	526	626
31S	13.1	14.8	62.7	88	146	230	332	402	35.3	150	0.005	300	370	404	460	530
32S	11.2	14.4	61.5	88	146	220	326	346	32.1	100	0.027	286	358	388	474	598
40S	12.1	14.4	54.3	92	154	292	436	458	23.9	230	0.030	420	500	548	642	738
41S	12.1	14.6	63.3	88	148	260	368	398	31.8	125	0.006	276	396	456	550	638
Charge Stock - Slurry Oil																
13S	9.0	4.2	60.8	102	168	260	348	384	26.1	150	0.003	340	436	528	630	676
14S	8.0	4.4	66.6	106	162	244	318	352	29.9	145	0.005	318	400	480	598	660
15S	8.4	5.8	66.4	98	156	234	308	342	24.8	130	0.151	310	392	464	570	630
16S	8.5	4.3	61.6	116	172	264	346	370	25.5	180	0.304	370	448	558	644	670
17S	7.4	2.3	61.0	100	172	267	344	378	20.7	200	0.010	388	482	598	672	724
18N	8.7	3.5	60.5	118	170	244	316	358	23.3	200	0.015	354	432	578	668	712
18S	8.3	5.0	60.4	102	168	247	318	356	22.0	185	0.021	364	452	590	672	718
19S	8.3	3.7	66.4	100	147	206	298	344	40.9	100	0.000	252	310	394	480	544
20S	8.4	4.1	67.2	94	154	256	334	352	42.3							
22N	8.7	12.0	68.9	90	144	226	340	404	30.3	90	0.009	230	354	450	590	642
23S	8.3	5.5	52.2	106	170	300	466	498	40.8	Instant	0.007	178	248	356	492	602
24S	8.5	11.3	62.7	100	156	224	338	396	27.9	120	0.002	270	346	512	616	660
25S	9.0	13.8	64.5	104	152	212	276	320	28.9	Instant	0.009	184	268	416	616	686
38N	8.4	6.7	58.5	96	162	280	420	466	19.1	200	0.004	350	474	580	674	740
39N	8.7	5.0	53.2	96	170	330	490	560	10.6	225	0.035	352	550	636	696	756
Charge Stock - Thermal Tar (Pure Oil Co.)																
33S	3.0	1.2	26.5	124	222	328	398	426	11.1	215	0.012	424	468	500	576	656
34S	3.4	2.9	27.6	154	274	412	482	508	10.9	220	0.011	408	474	506	516	660
Charge Stock - Thermal Tar (Marathon Oil Co.)																
42S	-1.5	1.0	61.1	100	176	252	332	392	34.1	130	0.008	288	360	434	560	656
43S	-1.1	-1.5	64.7	104	168	222	272	294	25.2	115	0.012	290	370	464	627	678
44S	-1.7	-1.5	65.6	106	164	216	318	382	10.2	145	0.007	300	398	604	676	748

* IBP - initial boiling point
** EP - end point
*** COC - Cleveland Open Cup

Table 13. Extensive Test Data - Mass Spectrometry of Gas Product

Cycle Number	Hydrogen %	Methane %	Ethylene %	Ethane %	Propylene %	Propane %	Butenes %	iso-Butane %	n-Butane %	Pentenes %	iso-Pentanes %	n-Pentane %	Residue C6 Plus %	Nitrogen	Carbon Dioxide	Hydrogen Sulfide	Gross Heat Value BTU/cu. ft.	Specific Gravity 60°F (air=1)	Molecular Weight
Charge Stock - Vacuum Residuum																			
6N	0.5	33.1	3.2	22.4	6.2	15.5	3.4	1.2	5.9	2.3	1.2	2.0	3.2		0.2	0.6	1518	0.888	25.4
31S	0.3	19.5	1.1	15.4	5.2	17.0	5.7		13.0	5.9	3.9	4.8	6.9		1.1		1891	1.138	32.3
40S	0.5	24.7	3.3	19.4	6.9	16.6	5.5	1.9	8.2	4.2	2.5	2.5	3.6		0.1		1679	0.991	28.2
41S	0.5	26.3	3.4	18.8	8.0	16.5	6.2	2.1	7.6	3.8	1.9	2.3	2.4	0.2			1639	0.964	27.5
Charge Stock - Slurry Oil																			
13S	1.1	46.1	2.3	16.7	3.6	11.8	2.6	1.1	5.6	2.3	2.3	1.8	2.5			0.1	1303	0.740	21.2
14S	2.0	45.4	1.8	14.2	3.1	10.6	2.6	1.1	6.0	3.0	2.0	3.0	4.0	0.9	0.1	0.2	1213	0.688	19.6
18S	1.7	44.0	1.5	13.6	4.1	9.3	3.2	0.7	5.5	4.5	2.4	2.9	6.3				1286	0.727	20.8
22N	0.9	40.4	1.4	17.2	4.0	13.2	3.2	0.8	7.7	3.0	1.8	2.9	3.2		0.3		1396	0.803	22.9
23S	0.6	36.4	1.0	17.3	2.8	14.0	2.9	1.5	8.8	3.2	2.5	4.6	4.2		0.3		1513	0.880	25.1
24S	1.4	33.7	0.6	14.0	2.1	12.4	3.3	1.6	9.5	4.5	2.4	5.9	8.1		0.4		1460	0.845	24.0
25S	1.0	32.4	0.7	13.9	1.2	12.7	2.2	2.1	9.2	4.2	3.5	6.2	10.4		0.2		1553	0.845	24.0
39N	0.6	39.6	1.0	17.7	2.4	14.2	2.5	2.5	8.0	2.6	2.7	2.9	3.1				1457	0.841	24.0
Charge Stock - Thermal Tar (Pure Oil Co.)																			
34S		1.2	50.1	18.4		9.5	1.1	1.8	4.7	2.5	2.5	2.3	5.8		0.1		1265	0.717	20.5
Charge Stock - Thermal Tar (Marathon Oil Co.)																			
42S	0.3	28.1	2.0	17.9	6.7	15.5	6.0	1.6	8.2	4.6	1.8	2.9	4.1		0.2		1663	0.981	28.0
44S	0.5	27.1	1.3	10.5		7.0		2.1	5.0	0.0	6.2	3.3	27.7		0.2		1867	1.112	31.3

Table 14. Extensive Test Data - Product Yields

Cycle Number	Gas Oil	Gasoline	Gas	Total	Coke, % (by difference)
Charge Stock - Vacuum Residuum					
6N	30.6	35.1	12.2	77.9	22.1
8S	22.7	38.5	12.1	73.3	26.7
9S	6.7	33.6	12.5	52.8	47.2
10S	26.6	20.7	15.4	62.7	37.3
11S	27.5	25.9	13.8	67.2	32.8
12S	18.1	28.2	18.7	65.0	35.0
26S	30.6	43.9	8.7	83.2	16.8
27N	36.4	42.7	10.9	90.0	10.0
27S	26.0	41.5	9.5	77.0	23.0
28N	25.9	43.6	11.1	80.5	19.5
30S	14.9	29.0	11.8	55.7	44.3
31S	15.1	20.8	19.3	55.2	44.8
32S	1.8	29.4	21.8	53.0	47.0
40S	10.8	36.6	17.0	64.4	35.6
41S	20.6	22.9	19.7	63.2	36.8
Charge Stock - Slurry Oil					
13S	29.7	8.6	15.8	54.1	45.9
14S	21.8	8.7	14.1	44.6	55.4
15S	29.0	14.1	13.7	56.8	43.2
16S	32.8	13.2	.314	59.4	40.6
17S	31.4	11.1	13.2	55.7	44.3
18N	29.6	12.3	13.6	55.5	44.5
18S	29.0	11.4	17.6	58.0	41.9
19S	9.6	17.2	19.9	46.5	53.5
20S	0.0	33.0	22.5	55.5	44.5
22N	13.8	16.2	19.7	49.7	50.3
23S	0.0	14.8	24.0	38.8	61.2
24S	0.0	13.7	23.6	37.3	62.7
25S	0.0	15.2	21.4	36.6	63.4
38N	16.9	8.6	19.4	44.9	55.1
39N	10.5	13.7	18.3	42.5	57.5
Charge Stock - Thermal Tar (Pure Oil Co.)					
33S	21.2	4.2	16.5	41.9	58.1
34S	0.0	3.0	16.4	19.4	80.6
Charge Stock - Thermal Tar (Marathon Oil Co.)					
42S	20.8	1.0	18.3	40.1	59.9
43S	21.1	0.9	18.1	40.1	59.9
44S	19.9	0.0	20.5	40.4	59.6

Table 15. Extensive Test Data - Coke Yields

Cycle Number	Fresh Feed, lbs.	Drum Outage, ft.	Coke Volume cu. ft.	Apparent Bulk Density lbs / ft^3	Coke lbs.	Yield %	Yield % (by difference)
Charge Stock - Vacuum Residuum							
6N	35300	12.50	220	55	12100	34.3	22.1
8S	34000	13.77	208	55	11400	33.5	26.7
9S	16900	22.75	100	55	5500	32.6	47.2
10S	17500	21.77	103	55	5660	32.3	37.3
11S	32500	16.10	179	55	9840	30.6	32.8
12S	18100	23.00	90	55	4950	27.3	35.0
26S	40750	12.46	220	55	12100	29.7	16.8
27N	39700	13.58	206	55	11350	28.5	10.0
27S	37800	12.35	223	55	12250	32.4	23.0
28N	41400	13.79	207	55	11400	27.5	19.5
30S	39300	13.88	206	55	11350	28.9	44.3
31S	34250	10.75	244	58	14150	41.1	44.8
32S	33800	11.58	232	48	13450	39.6	47.0
40S	17300	22.38	96	55	5280	30.5	35.6
41S	17600	22.73	91	55	5000	28.4	36.8
Charge Stock - Slurry Oil							
13S	32500	11.52	234	66	15350	47.2	45.9
14S	32800	8.31	276	66	18200	55.9	55.4
15S	34000	8.85	266	66	17550	51.5	43.2
16S	35350	11.83	237	66	15650	44.3	40.6
17S	35100	10.31	246	66	16250	46.3	44.3
18N	34700	12.09	229	66	15130	43.6	44.5
18S	15100	10.62	247	66	16300	46.5	41.9
19S	23750	13.19	230	66	15300	64.0	53.5
20S	11950	19.56	131	66	8640	71.4	44.5
22N	26820	11.29	235	66	15500	57.8	50.3
23S	25500	8.00	276	66	18200	71.3	61.2
24S	24950	8.54	270	66	17850	71.5	62.7
25S	24100	9.52	257	66	16950	78.2	63.4
38N	26850	14.08	200	66	13200	49.2	55.1
39N	26430	13.50	224	66	14800	55.9	57.5
Charge Stock - Thermal Tar (Pure Oil Co.)							
33S	28140	14.35	197	63	12800	45.5	58.1
34S	27700	10.42	246	67	16500	59.6	80.6
Charge Stock - Thermal Tar (Marathon Oil Co.)							
42S	26400	8.65	270	63	17000	64.5	59.9
43S	27400	6.12	302	63	19000	65.6	59.9
44S	23850	11.63	231	63	14550	61.0	59.6

Table 16. Extensive Test Data - Coke Properties

Cycle Number	Sampled BD lbs/ft³	Drum Average lbs/ft³	Calcine Yield - Weight, Per Cent						Calcined Coke			Remarks (AML)
						Drum Average			Real Density	Ash	Sulfur	
			AML	PRC*	Marathon**	AML	PRC*	Marathon**	g/cc	%	%	
Charge Stock - Vacuum Residuum												
6N (Comp.)	40.0	40.0	87.1	90.9	90.3	-	-	-	1.959	0.09	0.59	
8S-T	53.1		88.8	90.5	91.3				2.026	0.18	0.64	
M	59.3		90.6	91.1	92.8				1.990	0.24	0.64	
B	55.6	56.0	91.0	92.3	-	90.1	91.3	92.0	1.934	0.23	0.64	
9S-T	51.8		89.0	90.6	89.8				2.072	0.18	0.59	
B	56.2	54.0	91.6	91.9	92.8	90.2	91.3	91.3	2.094	0.13	0.64	Calcined temperature to 1200°C
10S-T	50.6		90.4	90.0	90.3				2.055	0.24	0.61	
B	44.7	47.7	91.0	90.0	90.9	90.7	90.0	90.6	1.990	0.27	0.59	
11S-T	55.6		92.0	91.7	92.1				2.041	0.14	0.65	
M	55.6		89.1	92.1	93.0				2.070	0.10	0.76	
B	56.8	56.0	93.9	92.1	90.8	91.7	92.0	92.0	2.027	0.15	0.57	
12S-T	61.8		91.0	90.7	90.1				1.994	0.13	0.68	
B	62.4	62.1	88.1	91.7	90.9	89.6	91.2	90.5	1.953	0.16	0.75	Oxidized while drying
26S-T	-		-	-	90.8				-	-	-	Oxidized while drying
M	-		-	-	91.1				-	-	-	Oxidized while drying
B	-		-	-	91.5			91.2	-	-	-	Oxidized while drying
27N-T	56.9		93.0	-	91.6				1.973	0.39	0.64	
M	55.0		92.0	-	92.4				1.965	0.22	0.72	
B	57.5	56.5	91.0	-	92.3	91.0	91.4	92.1	1.933	0.21	0.70	
27S-T	53.1		89.0	-	92.4				1.902	0.24	0.63	
M	51.8		89.0	-	92.1				1.868	0.16	0.61	
B	54.4	53.1	91.0	-	92.3	89.7	90.4	92.3	1.958	0.20	0.70	
28N-T	41.9		89.0	-	92.1				1.912	0.19	0.66	
M	55.6		91.0	-	91.8				1.893	0.17	0.66	
B	59.4	52.3	90.1	-	78.4	90.0	91.0	87.5	1.961	0.21	0.61	
30S-T	51.9		84.0	-	90.3				1.986	0.15	0.64	
M	60.0		90.0	-	91.6				1.997	1.14	0.63	
B	56.8	56.2	90.0	-	89.2	87.0	91.1	90.4	1.979	0.15	0.56	
31S-T	54.4		92.0	-	92.5				1.992	0.06	0.49	
M	58.1		92.0	-	93.3				1.971	0.06	0.55	
B	58.8	57.1	93.0	-	93.9	92.3	91.7	93.2	1.985	0.05	0.60	
32S-T	59.3		89.1	-	92.9				1.915	0.06	0.51	
M	58.1		91.0	-	93.6				1.933	0.04	0.58	
B	60.0	59.1	90.0	-	92.6	90.0	92.3	93.0	1.962	0.12	0.58	
40S-T	63.1		90.0	-					1.912	0.25	0.67	
B	52.4	57.8	92.0			91.0	90.9		1.906	0.20	0.70	
41S-T	63.8		94.0	-	93.4				1.967	0.06	0.66	
B	60.0	61.9	93.0	-	91.9	93.5	91.5	92.6	1.965	0.09	0.70	
Charge Stock - Slurry Oil												
1 4S-T	-		90.0	93.3	92.8				2.005	0.53	0.55	
M	55.2		92.3	94.2	93.5				2.064	0.17	0.65	
B	68.7	55.2(est.)	93.0	94.1	90.8	91.8	93.9	92.4	2.026	0.31	0.54	
14S-T	70.0		96.0	93.6	93.4				1.975	0.06	0.61	
M	66.8		93.0	94.1	93.4				2.046	0.15	0.51	
B	66.2	67.3	96.8	93.8	93.8	95.3	93.8	93.5	1.961	0.10	0.50	
15S-T	-	-	-	-	92.8				-	-	-	
M	62.1		92.0	-	92.9				2.011	0.09	0.64	
B	63.7	62.4(est.)	97.0	-	93.1	94.0 (est.)	92.9	93.0	1.980	0.06	0.52	
16S-T	68.6		93.9	92.6	92.0				1.984	0.09	0.54	
M	65.5		94.9	93.7	92.8				1.996	0.06	0.50	
B	61.2	65.1	92.3	93.3	91.5	93.4	93.2	92.1	1.876	0.03	0.55	
17S-T	66.8		92.0	93.4	91.3				1.989	0.07	0.52	
M	65.6		91.0	93.7	92.2				1.957	0.05	0.57	
B	71.6	68.0	92.0	94.0	92.7	91.7	93.7	92.1	1.949	0.07	0.57	
18N-T	63.1		94.7	93.9	94.5				1.972	0.14	0.52	
M	65.0		89.2	94.5	94.4				2.000	0.48	0.57	
B	67.4	65.2	92.8	93.6	95.1	92.2	94.0	94.7	1.861	0.12	0.63	
18S-T	66.4		94.0	93.4	93.7				1.974	0.04	0.56	
M	70.8		92.0	93.5	94.3				1.964	0.04	0.67	Oxidized while drying
B	70.8	69.3	91.0	93.5	94.5	92.3	93.5	94.2	1.962	0.14	0.56	Oxidized while drying
19S-T	68.8		-	93.3	93.5				-	-	-	Burned in calcining
M	67.8		-	93.3	93.8				-	-	-	Burned in calcining
B	66.0	67.5	94.0	93.4	94.7	-	93.3	94.0	1.846	0.03	0.64	
20S-T	62.4		94.0	93.0	93.4				1.969	0.09	0.68	Oxidized while drying
B	68.8	65.6	92.0	91.8	93.2	93.0	92.4	93.3	1.956	0.02	0.59	Oxidized while drying
22N-T	72.5		92.1	-	88.6				1.909	0.12	0.63	
M	68.8		94.0	-	94.2				1.953	0.04	0.61	
B	68.8	70.0	94.0	-	94.6	93.3	92.6	92.5	1.971	0.06	0.61	
23S-T	58.1		95.1	93.6	92.5				1.963	0.34	0.67	
M	63.1		93.1						1.944	0.10	0.56	
B	62.4	61.2	93.9		92.2	94.0	-	92.4	1.727	0.05	0.62	
24S-T	-		96.0	-	94.3				1.932	0.21	0.65	Oxidized while drying
M	59.9		94.0	-	94.1				1.954	0.43	0.64	
B	63.8	60.0(est.)	93.0	-	93.8	94.3	93.2	94.1	1.951	0.17	0.51	
25S (Comp.)	-		94.0	-	92.4				1.934	0.27	0.57	
38N-T	66.2		91.0	-	92.4				1.990	0.10	0.65	
M	66.8		92.0	-	90.5				1.928	0.06	0.63	
B	60.6	64.5	94.0	-	94.3	92.3	93.6	92.4	1.942	0.07	0.61	
39N-T	63.1		93.0	-	91.9				1.969	0.16	0.65	
M	70.0		92.0	-	93.7				1.971	0.09	0.63	
B	65.6	66.2	94.0	-	93.8	93.0	93.9	93.1	1.946	0.09	0.58	

* Parma Research Center, National Carbon Company, Parma, Ohio
** Marathon Oil Company, Robinson, Illinois

Table 16. Extensive Test Data - Coke Properties (Continued)

Cycle Number	Sampled BD lbs/ft³	Drum Average lbs/ft³	Calcine Yield - Weight, Per Cent			Drum Average			Calcined Coke			Remarks (AML)
			AML	PRC*	Marathon**	AML	PRC*	Marathon**	Real Density g/cc	Ash %	Sulfur %	
Charge Stock - Thermal Tar (Pure Oil Co.)												
33S-T	58.7		91.0	-	92.9				1.985	0.18	0.07	
M	65.0		92.0	-	94.0				1.983	0.01	0.12	
B	70.0	64.6	92.0	-	95.3	91.7	93.7	94.1	1.963	0.03	0.15	
34S-T	66.8		94.0	-	94.6				1.917	0.05	0.17	
M	69.3		94.0	-	94.5				1.963	0.03	0.13	
B	64.9	67.0	90.0	-	91.7	92.7	93.4	93.6	1.968	0.03	0.15	
Charge Stock - Thermal Tar (Marathon Oil Co.)												
42S-T	66.2		94.0	-	92.7				1.998	0.11	0.65	
M	67.1		92.0	-	91.9				1.929	0.13	0.62	
B	61.2	64.8	93.0	-	89.9	93.0	92.5	91.5	1.977	0.08	0.67	
43S-T	53.1		93.0	-	93.7				1.941	0.13	0.54	
M	61.8		93.0	-	92.9				1.932	0.06	0.62	
B	62.4	59.1	93.0	-	92.0	93.0	93.3	92.9	1.986	0.06	0.55	
44S-T	63.4		93.0	-					1.905	0.10	0.65	
M	68.5		92.0	-					1.950	0.13	0.64	
B	62.6	64.8	92.0	-		92.3	93.1		1.985	0.10	0.64	

* Parma Research Center, National Carbon Company, Parma, Ohio
** Marathon Oil Company, Robinson, Illinois

Table 17. Extensive Test Data - Extruded Rod Data (Physical Properties) Average Values

Cycle Number	Bulk Density - g/cc Green	Baked	Graph	Weight Loss % Bake to Graph	Volume Change % Bake to Graph
Charge Stock - Vacuum Residuum					
6N	1.62	1.53	1.60	3.0	7.2
8S	1.62	1.53	1.60	4.6	8.4
9S	1.64	1.56	1.60	3.5	5.9
10S	1.63	1.55	1.61	4.3	7.6
11S	1.61	1.56	1.61	3.9	6.5
12S	1.63	1.56	1.59	4.8	6.7
27N	1.66	1.57	1.64	4.5	8.6
27S	1.63	1.55	1.60	4.5	7.5
28N	1.64	1.54	1.64	4.5	10.4
30S	1.64	1.55	1.63	4.3	8.6
31S	1.61	1.54	1.62	4.2	8.1
32S	1.59	1.52	1.58	4.7	7.9
40S	1.65	1.53	1.57	4.5	9.7
41S	1.70	1.54	1.58	4.5	6.7
Charge Stock - Slurry Oil					
14S	1.56	1.52	1.54	4.2	5.1
15S	1.60	1.50	1.53	3.6	6.0
16S	1.61	1.50	1.53	3.4	5.5
17S	1.59	1.49	1.52	3.3	5.3
18N	1.61	1.53	1.55	3.6	4.9
18S	1.60	1.52	1.56	3.0	5.7
19S	1.59	1.51	1.53	4.9	6.4
20S	1.60	1.51	1.54	3.1	5.2
22N	1.60	1.45	1.49	3.1	5.4
23S	1.59	1.46	1.49	3.3	5.2
24S	1.62	1.50	1.53	3.8	6.3
25S	1.59	1.49	1.53	3.3	6.0
38N	1.60	1.49	1.53	4.2	5.2
39N	1.61	1.52	1.52	3.6	3.2
Charge Stock - Thermal Tar (Pure Oil Co.)					
33S	1.60	1.51	1.55	3.3	5.5
34S	1.57	1.53	1.57	2.6	4.8
Charge Stock - Thermal Tar (Marathon Oil Co.)					
42S	1.64	1.51	1.54	3.2	5.4
43S	1.65	1.51	1.53	2.9	4.6
44S	1.59	1.51	1.55	4.2	6.3

Table 18. Extensive Test Data - Extruded Rod Data
CTE and Specific Resistance

Cycle Number	Specific Resistance, 10^{-4} ohm-cm				CTE, 10^{-6}/°C		H/β	CTE, 10^{-6}/°C		H/β
			Drum Average (of Composite)					Drum Average (of Composite)		
	AML	PRC*	AML	PRC*	AML	PRC*	MORC**	AML	PRC*	MORC**
Charge Stock - Vacuum Residuum										
6N (Composite)	7.88	10.35	7.88	10.35	1.48	1.79	19.5	1.48	1.79	19.5
8S-T	6.84	8.48			1.32	1.63	24.1			
M	7.06	8.74			1.61	1.62	22.1			
B	7.08	8.57	6.99	8.66	1.70	1.94	22.6	1.51	1.73	22.9
9S-T	6.82	8.35			1.14	1.31	24.5			
B	8.27	9.10	7.55	8.73	1.71	1.81	21.3	1.42	1.56	22.9
10S-T	7.23	9.63			1.03	1.33	25.4			
B	7.91	9.30	7.57	9.48	2.11	2.27	19.2	1.57	1.80	22.8
11S-T	8.27	10.16			1.50	1.62	21.3			
M	7.57	9.55			1.35	1.79	22.1			
B	7.16	9.75	7.97	9.49	1.27	1.64	24.8	1.38	1.68	22.8
12S-T	9.13	7.78			1.51	1.60	22.1			
B	9.23	8.33	9.18	8.06	1.83	1.72	23.2	1.67	1.66	22.5
27N-T	6.42	-			1.67	-	-			
M	7.04	-			2.14	-	-			
B	7.11	-	6.86	7.65	1.89	-	-	1.90	2.44	19.4
27S-T	6.28	-			0.85	-	-			
M	6.72	-			1.19	-	-			
B	6.83	-	6.61	7.85	1.78	-	-	1.27	1.47	26.4
28N-T	7.22	-			2.18	-	-			
M	7.97	-			2.21	-	-			
B	8.19	-	7.79	8.25	2.35	-	-	2.25	2.50	17.8
30S-T	7.38	-			1.43	-	-			
M	7.74	-			1.53	-	-			
B	7.56	-	7.56	8.10	1.69	-	-	1.55	1.79	20.8
31S-T	7.96	-			1.23	-	-			
M	8.90	-			1.30	-	-			
B	8.29	-	8.38	8.40	1.56	-	-	1.36	1.64	22.2
32S-T	7.69	-			1.18	-	-			
M	7.78	-			1.02	-	-			
B	7.30	-	7.59	8.47	1.47	-	-	1.22	1.68	23.3
40S-T	7.13	-			0.60	-	-			
B	7.04	-	7.09	8.71	2.00	-	-	1.30	1.43	24.1
41S-T	7.16	-			0.78	-	-			
B	7.46	-	7.31	7.74	1.76	-	-	1.27	1.42	26.1
Charge Stock - Slurry Oil										
13S-T	7.66	12.20			0.07	0.45	32.3			
M	7.54	11.30			0.24	0.48	34.5			
B	7.19	10.06	7.46	11.19	0.34	0.60	34.8	0.22	0.51	33.9
14S-T	8.90	7.52			0.37	0.27	37.6			
M	8.38	7.62			0.37	0.32	35.6			
B	8.84	8.26	8.71	7.80	0.47	0.35	-	0.40	0.31	35.6

* Parma Research Center, National Carbon Company, Parma, Ohio
** Marathon Oil Research Center, Denver, Colorado (X-ray ratio)

Table 18. Extensive Test Data - Extruded Rod Data
CTE and Specific Resistance (Continued)

Cycle Number	Specific Resistance, 10^{-4} ohm-cm		Drum Average (of Composite)		CTE, 10^{-6} /°C		H/β	CTE, 10^{-6} /°C Drum Average (of Composite)		H/β
	AML	PRC*	AML	PRC*	AML	PRC*	MORC**	AML	PRC*	MORC**
Charge Stock - Slurry Oil (Continued)										
15S-T	8.89	-			0.37	-	-			
M	8.68	-			0.50	-	-			
B	9.31	-	8.96	7.72	0.49	-	30.4	0.45	0.29	31.6
16S-T	8.68	7.05			0.55	0.42	31.8			
M	8.82	7.52			0.34	0.26	37.7			
B	8.91	7.36	8.80	7.31	0.37	0.23	36.1	0.42	0.30	35.2
17S-T	8.75	7.30			0.42	0.28	-			
M	8.85	7.06			0.30	0.25	31.8			
B	8.24	7.04	8.61	7.13	0.43	0.38	-	0.41	0.30	31.8
18N-T	8.40	7.66			0.39	0.38	31.7			
M	8.39	7.34			0.42	0.38	38.3			
B	8.51	7.62	8.43	7.54	0.43	0.31	33.2	0.41	0.34	34.4
18S-T	6.65	7.38			0.25	0.32	38.2			
M	7.04	-			0.22	0.28	37.8			
B	6.38	7.57	6.69	7.48	0.25	0.42	29.2	0.24	0.34	31.7
19S-T	-	7.46			-	0.28	28.7			
M	-	8.18			-	0.50	28.9			
B	6.87	7.86	-	7.83	0.42	0.53	31.2	0.36	0.37	29.6
20S-T	6.58	7.07			0.24	0.31	34.1			
B	6.51	7.28	6.55	7.18	0.30	0.37	34.1	0.27	0.31	34.1
22N-T	6.91	-			0.25	-	-			
M	6.81	-			0.17	-	-			
B	7.47	-	7.07	7.08	0.20	-	-	0.21	0.26	30.1
23S-T	6.60	-			0.30	-	-			
M	7.03	-			0.40	-	-			
B	7.49	-	7.04	7.34	0.40	-	-	0.36	0.49	-
24S-T	7.21	-			0.33	-	-			
M	6.77	-			0.23	-	-			
B	6.10	-	6.69	7.34	0.12	-	-	0.23	0.37	30.7
25S (Composite)	-	-	6.02	7.56	-	-	-	0.20	0.44	30.6
38N-T	8.71	-			0.16	-	-			
M	7.48	-			0.20	-	-			
B	7.56	-	7.92	7.40	0.27	-	-	0.21	0.38	34.0
39N-T	6.94	-			0.08	-	-			
M	7.01	-			0.32	-	-			
B	7.37	-	7.11	7.44	0.34	-	-	0.24	0.41	32.4
Charge Stock - Thermal Tar (Pure Oil Co.)										
33S-T	6.51	-			0.19	-	-			
M	6.60	-			0.16	-	-			
B	6.97	-	6.69	8.32	0.40	-	-	0.25	0.58	35.4
34S-T	6.14	-			0.25	-	-			
M	6.67	-			0.39	-	-			
B	6.44	-	6.42	8.28	0.37	-	-	0.34	0.47	36.0

* Parma Research Center, National Carbon Company, Parma, Ohio
** Marathon Oil Research Center, Denver, Colorado (X-ray ratio)

Table 18. Extensive Test Data - Extruded Rod Data
CTE and Specific Resistance (Continued)

Cycle Number	Specific Resistance, 10^{-4} ohm-cm				CTE, 10^{-6}/°C			H/β			H/β
			Drum Average (of Composite)					Drum Average (of Composite)			
	AML	PRC*	AML	PRC*	AML	PRC*	MORC**	AML	PRC*	MORC**	
Charge Stock - Thermal Tar (Marathon Oil Co.)											
42S-T	7.85	-			0.33	-	-				
M	8.42	-			0.43	-	-				
B	8.37	-	8.21	8.86	0.34	-	-	0.37	0.43	35.1	
43S-T	8.70	-			0.49	-	-				
M	7.81	-			0.37	-	-				
B	8.08	-	8.20	8.71	0.31	-	-	0.39	0.54	42.1	
44S-T	6.19	-			0.30	-	-				
M	5.93	-			0.25	-	-				
B	5.79	-	5.97	8.41	0.25	-	-	0.27	0.41	40.7	

* Parma Research Center, National Carbon Company, Parma, Ohio
** Marathon Oil Research Center, Denver, Colorado (X-ray ratio)

Table 19. Extensive Test Data - Molded Plug Data, Physical Properties

Cycle Number	Binder Level PPH	Bulk Density - g/cc Green	Baked	Graph	Weight Loss % Bake to Graph	Volume Loss % Bake to Graph
Charge Stock - Vacuum Residuum						
6N	32	1.63	1.54	1.57	4.4	6.3
8S	32	1.61	1.53	1.53	3.8	4.0
11S	32	1.59	1.52	1.57	3.8	6.6
28N	32	1.58	1.55	1.58	4.1	6.8
30S	32	1.58	1.54	1.58	3.9	7.7
32S	32	1.53	1.52	1.54	4.1	5.4
40S	32	1.54	1.52	1.54	4.3	5.5
41S	32	1.62	1.55	1.59	4.5	6.6
Charge Stock - Slurry Oil						
13S	32	1.61	1.53	1.52	3.3	2.7
15S	32	1.59	1.50	1.47	2.7	1.3
16S	32	1.58	1.56	1.53	4.7	2.9
17S	32	1.60	1.54	1.53	3.5	2.8
19S	32	1.53	1.48	1.49	3.0	4.0
24S	32	1.52	1.49	1.49	3.0	2.9
25S	32	1.53	1.44	1.45	3.1	3.2
39N	32	1.50	1.46	1.47	1.6	3.8
Charge Stock - Thermal Tar (Pure Oil Co.)						
33S	32	1.51	1.46	1.51	2.7	4.3
34S	32	1.56	1.47	1.51	4.1	6.8
Charge Stock - Thermal Tar(Marathon Oil Co.)						
42S	32	1.58	1.52	1.51	3.6	3.3
43S	32	1.56	1.53	1.53	3.9	4.0
44S	34	1.56	1.52	1.54	3.1	4.6

Table 20. Extensive Test Data - Molded Plug Data, CTE, Specific Resistance and Flexural Strength

Cycle Number	Specific Resistance, 10^{-4} ohm-cm				CTE, 10^{-6} (30 - 100°C)				Flexural Strength, lbs/in^2			
	A.G.*	W.G.**	Ratio (a.g./w.g.)	n (a.g./w.g.)	A.G.	W.G.	Ratio (a.g./w.g.)	n (a.g./w.g.)	W.G.	A.G.	Ratio (w.g./a.g.)	n (w.g./a.g.)
Charge Stock - Vacuum Residuum												
6N	13.24	9.82	1.3	6/6	3.00	2.02	1.5	4/3	1856	1559	1.2	3/3
8S	10.32	8.80	1.2	2/8	3.53	2.22	1.6	2/2	1725	1260	1.4	3/4
11S	13.75	8.84	1.6	4/3	3.50	1.95	1.8	2/2	-	-	-	-
28N	10.10	9.08	1.1	2/7	3.62	3.09	1.2	2/2	1515	1700	0.9	3/1
30S	11.42	8.84	1.3	4/8	3.52	2.59	1.4	2/2	1197	1016	1.2	4/2
32S	12.92	9.91	1.3	7/7	2.88	1.93	1.5	3/2	995	768	1.3	3/3
40S	12.21	9.45	1.3	6/6	3.04	1.95	1.6	4/4	1225	945	1.3	6/6
41S	11.93	7.86	1.5	6/6	3.19	1.91	1.7	4/4	1550	1175	1.3	5/6
Charge Stock - Slurry Oil												
13S	25.18	13.45	1.9	8/8	2.30	0.80	2.9	2/2	662	482	1.3	8/6
15S	14.83	9.97	1.4	8/8	2.29	0.79	2.9	2/2	1005	851	1.2	3/3
16S	15.23	10.68	1.4	4/4	2.40	0.79	3.0	2/2	1109	789	1.4	4/4
17S	-	-	-	-	2.26	0.81	2.8	2/2	921	800	1.2	4/4
19S	17.81	10.51	1.7	4/3	2.70	1.04	2.6	2/2	1031	783	1.3	2/4
24S	16.54	11.47	1.4	8/8	2.32	0.83	2.8	2/2	1032	1000	1.0	8/6
25S	18.04	10.55	1.7	8/7	1.70	0.79	2.2	2/2	1010	939	1.1	7/6
39S	25.99	15.69	1.7	4/6	2.28	0.72	3.0	2/2	569	289	2.0	4/2
Charge Stock - Thermal Tar (Pure Oil Co.)												
33S	17.37	11.64	1.5	3/4	2.26	1.01	2.2	1/1	1198	1052	1.1	4/2
34S	15.62	9.10	1.7	6/8	1.76	0.83	2.1	2/2	1237	953	1.3	8/6
Charge Stock - Thermal Tar (Marathon Oil Co.)												
42S	15.42	10.40	1.5	4/3	2.44	0.91	2.7	2/2	1139	794	1.4	3/3
43S	16.95	11.22	1.5	4/4	2.31	0.90	2.6	2/2	1270	875	1.4	3/3
44S	16.69	10.39	1.6	4/6	2.54	0.79	3.2	3/4	1329	934	1.4	3/4

* Across grain
** With grain

Table 21. Extensive Test Data - Coke Puffing Tests

Cycle Number	Puffing Range Temperature °C Max.	Puffing Range Temperature °C Min.	Puffing Range Length Change % Max.	Puffing Range Length Change % Min.	Total Puffing %	Total Length Change, % Room Temperature
Charge Stock - Vacuum Residuum						
11S	2250	1700	+0.04	-0.14	0.18	-1.98
28N	2150	1770	-0.15	-0.26	0.11	-1.61
30S	2200	1600	-0.22	-0.40	0.18	-1.98
32S	2070	1700	-0.50	-0.60	0.10	-1.15
40S	2215	1700	-1.06	-1.23	0.17	-2.33
41S	2335	1680	-0.41	-0.15	0.26	-1.87
Charge Stock - Slurry Oil						
15S	3000	1870	+0.27	-0.50	0.77	-0.48
17S	2850	1650	+1.07	-0.28	1.35	+0.10
19S	2800	1750	+0.56	-0.56	1.12	-0.30
20S	2770	1670	+1.66	-0.56	2.22	-0.41
24S	2500	1770	+0.0	-0.26	0.26	-0.65
39N	2810	1740	+0.17	-0.76	0.93	-0.91
Charge Stock - Thermal Tar (Pure Oil Co.)						
33S					0.0	-1.77
34S					0.0	-2.87
Charge Stock - Thermal Tar (Marathon Oil Co.)						
42S	2850	1750	+0.96	-0.33	1.29	+0.08
44S	2900	1800	+1.22	-0.26	1.48	+0.20

Table 22. Extensive Test Data - Chemical and Physical Characterization of Feed Stocks

Charge Stock	Aromatics % (1)		Paraffins % (1)	Napthenes % (1)	Total	Nonaromatics % (2)	Oxy % (2)	Loss % (2)	Total	Molecular Weight (3)	Atomic Carbon Hydrogen Ratio	K (4)	Sulfur %	Conradson Carbon, % (5)
Vacuum Residuum	35.2	34.8	10.9	53.9	64.8	58.7	4.4	2.1	65.2	833	0.65	—	0.38	11.3
Slurry Oil	62.0	61.4	0.0	38.0	38.0	33.0	1.1	4.5	38.6	303	0.80	10.6	0.48	5.7
Thermal Tar (Pure Oil Co.)	Off Scale	89.8	Off Scale	-	-	5.0	4.1	1.1	10.2	270	0.95	10.0	0.07	9.4
Thermal Tar (Marathon Oil Co.)	68.5	63.6	6.7	24.8	31.5	32.1	3.4	0.9	36.4	370	0.88	9.6	0.56	8.6

(1) Retractivity Intercept (Ref. 3)

(2) Silica Gel Chromatography (Ref. 2)

(3) Mechrolab - Vapor Pressure Lowering

(4) K = Characterization Factor = $\frac{\sqrt[3]{T_b}}{S.G.\ \frac{60°F}{60°F}}$

T_b = Molal Average Boiling Point - ° Rankine

(5) Conradson Carbon ASTM D189-61

Table 23. Extensive Test Data - Furnace Profiles

Profile Number	B	B^1	C	C^1	E	F	H	J
1 BA outlet °F	635	635	634	649	680	738	680	680
1 BB outlet °F	707	702	711	726	781	876	781	781
2 BA outlet °F	817	813	827	842	935	935	875	880
2 BB outlet °F	935	930	950	965	935	935	935	945