FE-1734-45(Vol.12)(Bk.1)
Distribution Category UC-90c

PLANT OPERATIONS FINAL REPORT

CO_2 ACCEPTOR PROCESS GASIFICATION PILOT PLANT

FINAL REPORT
VOLUME 12, BOOK 1 OF 4
PERIOD: JANUARY 1972 - OCTOBER 1977

CONOCO COAL DEVELOPMENT COMPANY
RESEARCH DIVISION
LIBRARY, PENNSYLVANIA 15129
AND
STEARNS-ROGER INCORPORATED
P.O. BOX 5888
DENVER, COLORADO 80217

PREPARED FOR

UNITED STATES DEPARTMENT OF ENERGY
AND
AMERICAN GAS ASSOCIATION
UNDER CONTRACT EX-76-C-01-1734

FOREWORD

This final report (Volume 12) on the operation of the CO_2 Acceptor Process Gasification Pilot Plant is submitted in fulfillment of the reporting requirements (Article V-Reports) of the prime Contract (EX-76-C-01-1734) between Continental Oil Company and the U. S. Department of Energy.

The work described here was carried out in part under the subject Contract and also under a prior Contract (14-001-0001-415) between Consolidation Coal Company (now a subsidiary of Conoco) and the Department of Interior. In both Contracts, Stearns-Roger, Incorporated was the operating subcontractor for Conoco Coal Development Company (previously the Research Division of Consolidation Coal Company).

Initial effort on the project was monitored by Mr. George I. Staber. Since 1972, the project monitor has been Dr. C. Lowell Miller, Chief Project Engineer, Fossil Energy Division, Department of Energy.

This project was also monitored by the Advisory Committee of American Gas Association.

Contributors to the present report include the following:

Conoco Coal Development Company

Vicki L. Bartos
George P. Curran
Louis A. Evans
Chester L. Krcil
Michael S. Lancet
Donald R. Lindahl
Duane C. McCoy
Barbara J. Minor
Pete F. Moran
Clifford J. Moskal
Louella M. Packard

Stearns-Roger Incorporated

Raymond W. Bain
Frank J. Batug
Eugene R. Casteel
Richard E. Davis
Donna M. Donovan
Robert A. Fortman
Denis A. Gautheir
Glenn F. Goreley
Dennis W. Hinders
M. Thomas Holen
Wyann E. Kaiser
Ralf P. Keller
Craig K. Knock
Blanche F. Messer
Richard L. Naugle
H. Dale Pettapiece
Larry C. Pierce
Alan P. Spragens
Robert W. Stansfield
Marion H. Vardaman

The CO_2 Acceptor Process development was initiated in 1964; subsequently a number of reports on laboratory research, pilot plant construction, interim operating results, and potential commercial process economics have been written. For reference by the reader, these are listed below.

Pipeline Gas from Lignite Gasification-A Feasibility Study
Issued: February, 1965
OCR R&D Report No. 16-Interim Report No. 1
Contractor: Consolidation Coal Company-Contract 14-01-0001-415
Available From:

NTIS: PB-166 817/AS (Feasibility Study)
PB-166 818/AS (Appendix)

Low-Sulfur Boiler Fuel Using the Consol CO_2 Acceptor Process
Issued: November, 1967
OCR R&D Report No. 16-Interim Report No. 2
Contractor: Consolidation Coal Company-Contract 14-01-0001-415
Available From:

NTIS: PB-176 910/AS

Phase II, Bench-Scale Research on CSG Process-Studies on Mechanics of Fluo-Solids Systems
Issued: January, 1970
OCR R&D Report No. 16-Interim Report No. 3, Book 1
Contractor: Consolidation Coal Company-Contract 14-01-0001-415
Available From:

GPO: Stock No. 163.10:16/Int. 3/Book 1
NTIS: PB-184 718/AS

Phase II, Bench-Scale Research on CSG Process-Laboratory Physico-Chemical Studies
Issued: January, 1970
OCR R&D Report No. 16-Interim Report No. 3, Book 2
Contractor: Consolidation Coal Company-Contract 14-01-0001-415
Available From:

NTIS: PB-184 719/AS

Phase II, Bench-Scale Research on CSG Process-Operation of the Bench-Scale Continuous Gasification Unit
Issued: January, 1970
OCR R&D Report No. 16-Interim Report No. 3, Book 3
Contractor: Consolidation Coal Company-Contract 14-01-0001-415
Available From:

GPO: Stock No. 163.10:16/Int. 3/Book 3
NTIS: PB-184 720/AS

Pipeline Gas From Lignite Gasification-Current Commercial Economics
Issued: January, 1970
OCR R&D Report No. 16-Interium Report No. 4
Contractor: Consolidation Coal Company-Contract 14-01-0001-415
Available From:

GPO: Stock No. 163.10:16/Int. 4
NTIS: PB-235 856/AS

Phase III and Phase VI-a-Design and Construction of the Consolidation Synthetic Gas Pilot Plant, Rapid City, South Dakota
Issued: May, 1974
OCR R&D Report No. 16-Interim Report No. 5
Contractor: Consolidation Coal Company-Contract 14-01-0001-415
Available From:

NTIS: PB-236 504/AS (PC not available)

Operations-CO_2 Acceptor Process Gasification Pilot Plant-January, 1972-January, 1973
Issued: April, 1975
DOE Final Report No. FE/1734-39: Volume 6, Book 1
Contractor: Conoco Coal Development Company-Contract EX-C-01-1734
Available From:

NTIS: (Identification number not yet available)

Operators-CO_2 Acceptor Process Gasification Pilot Plant-January, 1972-June, 1973
Issued: April, 1978
DOE Final Report No. FE/1734/39: Volume 6, Book 2
Contractor: Conoco Coal Development Company-Contract EX-76-C-01-1734
Available From:

NTIS: (Identification number not yet available)

Operations-CO_2 Acceptor Process Gasification Pilot Plant-July, 1973-July, 1974
Issued: April, 1978
DOE Final Report No. FE/1734/40: Volume 7
Contractor: Conoco Coal Development Company-Contract EX-76-C-01-1734
Available From:

NTIS: (Identificiation number not yet available)

Run Reports-CO_2 Acceptor Process Gasification Pilot Plant-Runs 1-11:
Period: January, 1972-July, 1973
Issued: April, 1978
DOE Final Report No. FE/1734-41: Volume 8, Book 1
Contractor: Conoco Coal Development Company-Contract EX-76-C-01-1734
Available From:

NTIS: (Identification number not yet available)

Run Reports-CO_2 Acceptor Process Gasification Pilot Plant-Runs 12-18:
Period: August, 1973-April, 1974
Issued: April, 1978
DOE Final Report No. FE/1734-41: Volume 8, Book 2
Contractor: Conoco Coal Development Company-Contract EX-76-C-01-1734
Available From:

NTIS: (Identification number not yet available)

Run Reports-CO_2 Acceptor Process Gasification Pilot Plant-Runs 19-27:
Period: June, 1974-August, 1975
Issued: April, 1978
DOE Final Report No. FE/1734-41: Volume 8, Book 3
Contractor: Conoco Coal Development Company-Contract EX-76-C-01-1734
Available From:

NTIS: (Identification number not yet available)

Run Reports-CO_2 Acceptor Process Gasification Pilot Plant-Runs 28-36:
Period: August, 1975-May, 1976
Issued: April, 1978
DOE Final Report No. FE/1734-41: Volume 8, Book 4
Contractor: Conoco Coal Development Company-Contract EX-76-C-01-1734
Available From:

NTIS: (Identification number not yet available)

Run Reports-CO_2 Acceptor Process Gasification Pilot Plant-Runs 37-42:
Period: May, 1976-February, 1977
Issued: April, 1978
DOE Final Report No. FE/1734/41: Volume 8, Book 5
Contractor: Conoco Coal Development Company-Contract EX-76-C-01-1734
Available From:

NTIS: (Identification number not yet available)

Run Reports-CO_2 Acceptor Process Gasification Pilot Plant-Runs 43-47:
Period: March, 1977-October, 1977
Issued: April, 1978
DOE Final Report No. FE/1734-41: Volume 8, Book 6
Contractor: Conoco Coal Development Company-Contract EX-76-C-01-1734
Available From:

NTIS: (Identification number not yet available)

Support Studies By South Dakota School of Mines-CO_2 Acceptor Process Gasification Pilot Plant
Period: March, 1971-June, 1977
Issued: April, 1978
DOE Final Report No. FE/1734-42, Volume 9, Book 1
Contractor: Conoco Coal Development Company-Contract EX-76-C-01-1734
Available From:

NTIS: (Identification number not yet available)

Support Studies By South Dakota School of Mines-CO_2 Acceptor Process Gasification Pilot Plant
Period: March, 1971-June, 1977
Issued: April, 1978
DOE Final Report No. FE/1734-42, Volume 9, Book 2
Contractor: Conoco Coal Development Company-Contract EX-76-C-01-1734
Available From:

NTIS: (Identification number not yet available)

Commercial Plant Conceptual Design and Cost Estimate-CO_2 Acceptor Process Gasification Pilot Plant
Period: August, 1976-December, 1977
Issued: April, 1978
DOE Final Report No. FE/1734-43, Volume 10, Book 1
Contractor: Conoco Coal Development Company-Contract EX-76-C-01-1734
Available From:

NTIS: (Identification number not yet available)

Commercial Plant Conceptual Design and Cost Estimate-CO_2 Acceptor Process Gasification Pilot Plant
Period: August, 1976-December, 1977
Issued: April, 1978
DOE Final Report No. FE/1734-43, Volume 10, Book 2
Contractor: Conoco Coal Development Company-Contract EX-76-C-01-1734
Available From:

NTIS: (Identification number not yet available)

Commercial Plant Conceptual Design and Cost Estimate-CO_2 Acceptor Process Gasification Pilot Plant
Period: August, 1976-December, 1977
Issued: April, 1978
DOE Final Report No. FE/1734-43, Volume 10, Book 3
Contractor: Conoco Coal Development Company-Contract EX-76-C-01-1734
Available From:

NTIS: (Identification number not yet available)

Environmental Characterization-CO_2 Acceptor Process Gasification Pilot Plant
Period: July, 1976-March, 1978
Issued: April, 1978
DOE Final Report No. FE/1734/44, Volume 11, Book 1
Contractor: Conoco Coal Development Company-Contract EX-76-C-01-1734
Available From:

NTIS: (Identification number not yet available)

Environmental Characterization-CO_2 Acceptor Process Gasification Pilot Plant
Period: July, 1976-March, 1978
Issued: April, 1978
DOE Final Report No. FE/1734/44, Volume 11, Book 2
Contractor: Conoco Coal Development Company-Contract EX-76-C-01-1734
Available From:

NTIS: (Identification number not yet available)

Environmental Characterization-CO_2 Acceptor Process Gasification Pilot Plant
Period: July, 1976-March, 1978
Issued: April, 1978
DOE Final Report No. FE/1734/44, Volume 11, Book 3
Contractor: Conoco Coal Development Company-Contract EX-76-C-01-1734
Available From:

NTIS: (Identification number not yet available)

Final Report On Pilot PLant Operations-CO_2 Acceptor Process Gasification Pilot Plant
Period: January, 1972-October, 1977
Issued:
DOE Final Report No. FE/1734/45, Volume 12, Book 1
Contractor: Conoco Coal Development Company-Contract EX-76-C-01-1734
Available From:

NTIS: (Identification number not yet available)

Final Report On Pilot Plant Operations-CO_2 Acceptor Process Gasification Pilot Plant
Period: January, 1972-October, 1977
Issued:
DOE Final Report No. FE/1734/45, Volume 12, Book 2
Contractor: Conoco Coal Development Company-Contract EX-76-C-01-1734
Available From:

NTIS: (Identification number not yet available)

Final Report On Pilot Plant Operations-CO_2 Acceptor Process Gasification Pilot Plant
Period: January, 1972-October, 1977
Issued:
DOE Final Report No. FE/1734/45, Volume 12, Book 3
Contractor: Conoco Coal Development Company-Contract EX-76-C-01-1734
Available From:

NTIS: (Identification number not yet available)

Final Report On Pilot Plant Operations-CO_2 Acceptor Process Gasification Pilot Plant
Period: January, 1972-October, 1977
Issued:
DOE Final Report No. FE/1734/45, Volume 12, Book 4
Contractor: Conoco Coal Development Company-Contract EX-76-C-01-1734
Available From:

NTIS: (Identification number not yet available)

Executive Summary Commercial Plant Conceptual Design and Cost Estimate-CO_2 Acceptor Process Gasification Pilot Plant
Period: August, 1976-December, 1977
Issued: April, 1978
DOE Final Report No. FE/1734/46, Volume 13
Contractor: Conoco Coal Development Company-Contract EX-76-C-01-1734
Available From:

NTIS: (Identification number not yet available)

TABLE OF CONTENTS

TABLE OF CONTENTS (continued)

SECTION 3
PILOT PLANT PRACTICES

SECTION 4
MECHANICAL PERFORMANCE

LIST OF FIGURES AND TABLES

SECTION 2

SECTION 3

SECTION 4

SECTION 4 (continued)

SECTION 4 (continued)

ABSTRACT

For the purpose of demonstrating the feasibility of the CO_2 Acceptor Gasification Process, a pilot plant was constructed at Rapid City, South Dakota, in 1971 and operated until 1977. The details of the plant design and of the operating program have been presented in previous Volumes 5 through 8. Ancillary support programs are detailed in Volumes 9 through 11.

The purpose of this final report is the condensation and interpretation of the accumulated technical information to provide an adequate design basis for a larger facility. Also included is a description of the demonstrated materials of construction which are suitable to provide mechanical integrity in a commercial plant. A limited description of the pilot equipment and plant operating procedures is provided to facilitate reader understanding.

SECTION 1

INTRODUCTION

The CO_2 Acceptor Gasification Process is one of the several second-generation processes which have evolved in the past fifteen years as a result of the impending need in the United States for supplemental sources of natural gas.

When applied to lignites and subbituminous coals, the CO_2 Acceptor Process appears to be more efficient, less capital intensive, and more economical with regard to water consumption than other developed processes. Since it produces no phenols or tar oils, the CO_2 Acceptor Process is at its greatest advantage where coal-derived liquids have no ready market and where pollution by liquid effluents may be of critical concern.[(1)]

Financial sponsorship for initial laboratory studies and for the construction of a 40 T/D pilot plant at Rapid City, South Dakota, was provided by the United States Department of Interior through its Office of Coal Research. Subsequent pilot plant operation during the period 1971-1977 was jointly sponsored by the American Gas Association (now the Gas Research Institute) and by the United States Government (successively through the Office of Coal Research, the Energy Research and Development Administration, and the Department of Energy). The objectives of the pilot plant program were achieved and the program was terminated in October, 1977.

Throughout the development program, the prime contractor has been Conoco Coal Development Company (formerly the Research Department of Consolidation Coal Company). Stearns-Roger, Incorporated has served as subcontractor for pilot plant construction, maintenance, and operation.

Of necessity, descriptive information on pilot plant facilities, and operating details are abbreviated in the present report; such information may be found in the many volumes enumerated in the Foreword. The primary purpose of this report is to present the technical information developed in pilot plant operations and ancillary programs, which is essential to provide a rational basis for the design of a demonstration facility or a pioneer commercial plant.

---

(1) Vol. 10, Books 1, 2, 3, "Commercial Plant Conceptual Design and Cost Estimate-CO_2 Acceptor Process Gasification Pilot Plant: Period August, 1976-December, 1977, "DOE Final Rpt. No. FE/1734-43

SECTION 2

PLANT DESCRIPTION

The CO_2 Acceptor Process Gasification Pilot Plant comprises five major process and support systems. These are: the Feedstock Preparation Area (100 Area), the Synthesis System (200 Area), the Gas Cleanup Systems (300 Area), the Methanation Unit (400 Area), and the Utilities (500 Area). The following subsections describe these systems, except for the Methanation Section which is covered in Book 3, Section 8 of this report. Process and Instrumentation diagrams for each system are included in the Appendices, Subsection 13.1 of Book 4.

2.1 FEEDSTOCK PREPARATION (100 AREA)

The Feedstock Preparation Area (100 Area) processes two raw feedstocks, acceptor and coal. Dead-burned dolomite, Tymochtee dolomite, and Minnekahta limestone have been ground and screened in the acceptor preparation area. Three different lignites, three subbituminous coals, and a lignite char have been ground, dried, and screened in the coal feedstock preparation area. A flow diagram of the 100 Area is included in Figure 2-1.

2.1.1 ACCEPTOR

The two acceptors used in the pilot plant operations were the Ohio Tymochtee dolomite and the South Dakota Minnekahta limestone. The dolomite was transported from Ohio by rail car and stored at a local quarry. It was then dried and trucked to the plant site as needed. The limestone was purchased and trucked to the plant site from a local quarry as needed. Acceptor was then conveyed to the acceptor raw storage tanks. The material was crushed by a hammer mill and sized by means of a vibrating screen. Normally, 6 and 8 Tyler mesh screens were used for sizing. Because of screening inefficiency, a 6 x 12 mesh product was recovered and put in tote bins for system feedstock. The oversize material from the vibrating screen was recycled back to the crusher feed and the fines were hauled to the local dump to be used as sanitary land fill.

Typical yields for both 6 x 9 Tymochtee dolomite and 6 x 8 Minnekahta limestone were about 1/3 product and 2/3 fines. The pilot plant mill and screening operation was adjusted to provide specification product at a rate sufficient to meet the pilot plant demands. Although higher yields would have been desirable, the efforts of plant personnel were directed toward other more critical areas of plant operation once a suitable grinding rate was achieved.

2.1.2 CHAR

The raw char, to be processed and used for run startups, was hauled by truck to the plant site from Husky Industries at

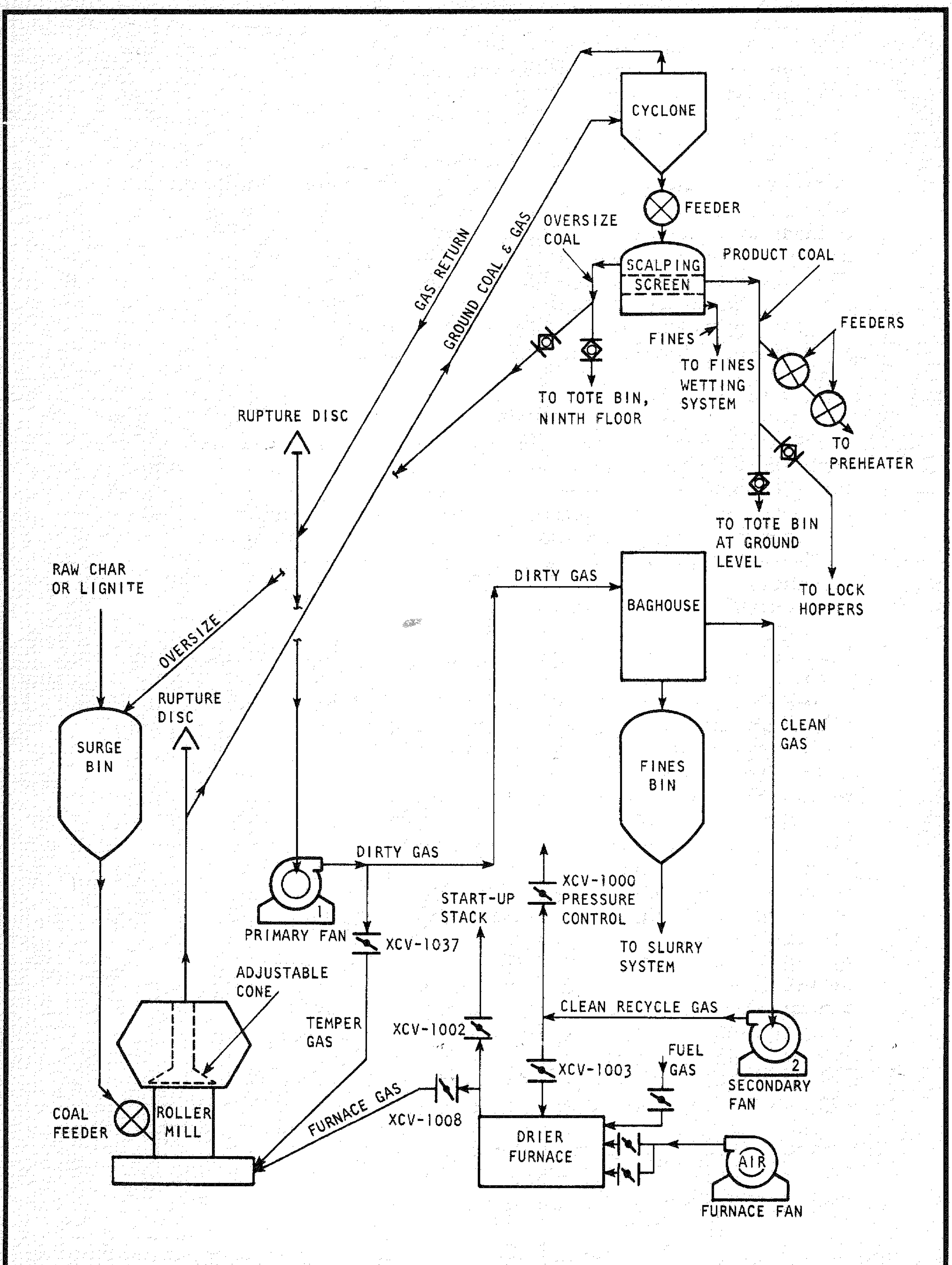


Figure 2-1. FEEDSTOCK PREPARATION AREA (100 AREA) FLOW DIAGRAM

Dickinson, North Dakota. It was then conveyed to the char storage tank and processed in the same manner as the other coal feedstock.

2.1.3 LIGNITE AND SUBBITUMINOUS COALS

The lignite and subbituminous coals were hauled to a local storage location by truck and rail car, depending on freight methods available between mine and plant site. At the local storage location, the coal was unloaded, piled, and compacted. The coal was then hauled to the plant site as needed.

The onsite storage for the coal consists of one 300-ton capacity coal storage tank.

2.1.4 ROLLER MILL

The coal is conveyed from the coal storage tank to a feed surge tank and through a feeder into the roller mill (see Figure 2-1). The feed rate to the mill can be controlled manually or by automatic control. The automatic control receives a signal from a differential pressure transmitter across the mill proper. This differential indicates how loaded the mill is and, in turn, activates a switch in feeder speed to maintain a given loading rate on the mill.

Heat is supplied to the mill from a drier furnace. The drier furnace burner and fuel/air ratio is controlled so as to provide a low-oxygen-content flue gas to the roller mill. The maximum allowable oxygen content of the grinding system is 10 percent, with the oxygen level normally running 4 to 5 percent. An inert gas system (refer to Subsection 2.5.2) supplies a low-oxygen (0.5 percent) gas to the system when needed.

The dry ground coal is conveyed pneumatically by recycle and furnace flue gases to a cyclone on the main structure. The solids are separated in the cyclone and flow down to the scalping screen while the recycle gas returns to the coal preparation area. The coal is separated into three streams by two screens. The fines are slurried and sent to the settling pond. The oversize material is returned to the surge bin and fed back through the roller mill. The desired size product stream (8 x 10 mesh with less than 5 percent below 100 mesh) is fed into a fludized bed preheater. The preheater bed is fludized with superheated steam. The lignite feed lock-hoppers are filled from the preheater as needed.

Typical yields from the grinding system for Velva lignite, the most often used feedstock, were the following:

(1) Product 62 Wt %
(2) Fines 38 Wt %

Optimization of the pilot plant feedstock grinding operation was attempted to the extent that an adequate rate of specification product was reliably produced. Extensive efforts to further optimize the operation were not attempted, since the study of coal grinding was not of particular concern.

2.1.5 GAS CIRCULATION

The recycle gas is circulated by a primary fan and a secondary fan. The drier furnace flue gas is moved from the drier furnace to the roller mill by pressure from the combustion air intake fan. The direction and quantity of gas flow is controlled by large butterfly valves, as shown on Figure 2-1. The returning gas from the cyclone is moved by the primary fan back to the mill or through the baghouse. The secondary fan receives the cleaned gas from the baghouse and discharges it to atmosphere through a pressure control valve or through the drier furnace as secondary tempering gas for use in drying. The furnace flue gas, with the secondary tempering gas, returns to the mill, combines with some of the primary fan discharge gas, and completes the drying and conveying gas cycle. Pressure control of the system is maintained by the pressure control valve receiving a signal from the sensor-transmitter located on the furnace to roller mill line.

2.1.6 BAGHOUSE

All gas vented to atmosphere or recycled back through the drier furnace is first passed through the baghouse for cleaning. The baghouse was a part of the original Williams package unit. It contains sixteen Nomex bags. Wool and dacron bags were tried, but the Nomex type material was used due to its higher temperature tolerance. The fines collected in the baghouse are blown down into a bin, isolated from the system, and then slurried for disposal.

2.1.7 SLURRY SYSTEM

The slurry system was designed and installed in the coal preparation area primarily to handle the finely ground coal and char. Disposal of the fine material had proven difficult in windy conditions and also was a major fire hazard. The slurrying water is pumped from the settling pond, downstream of a straw dam, to the slurrying tank and through an eductor under the slurrying tank. The fines are well wetted and slurried and flow back to the settling pond upstream of the straw dam. The strained slurrying water is also used as the flow to the lignite preheater vent scrubber and is handled in the same manner as the fines slurry system. Between runs, the settling pond water is siphoned off and the wetted solids are dredged out and hauled to the dump for sanitary land fill. A flow diagram of the lignite fines slurry system is included as Figure 2-2.

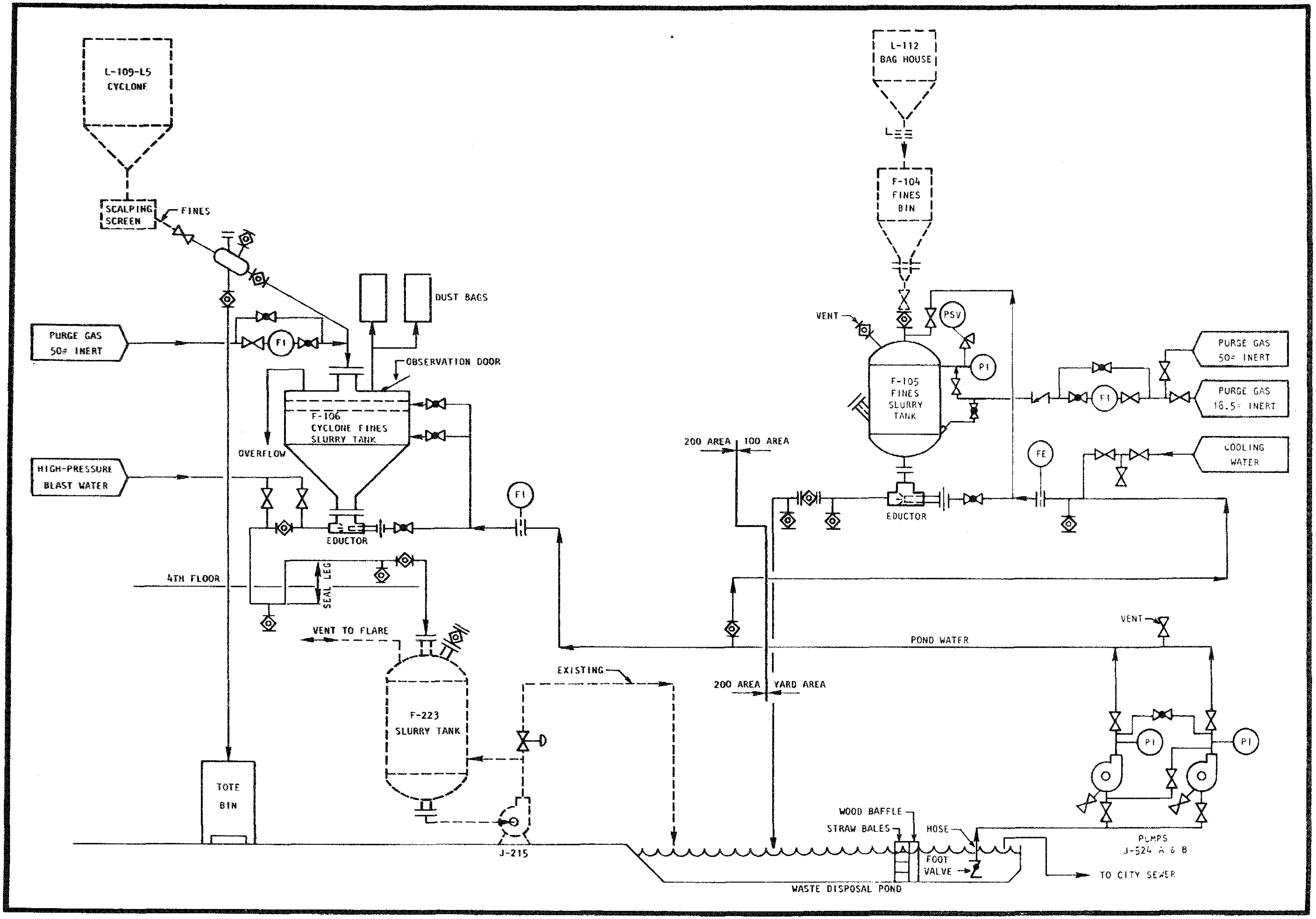


Figure 2-2. LIGNITE FINES DISPOSAL SYSTEM

2.2 SYNTHESIS SYSTEM (200 AREA)

The Synthesis System or gasification section (200 area) of the CO_2 Acceptor Process consists of two major reaction vessels, the gasifier, D-202, and regenerator, D-203, and associated equipment. (See Figure 2-3.) The gasifier and regenerator are both water-jacketed, refractory-lined, fluidized-bed reactors. Acceptor and char are both circulated between both reactors; their movement through the system is traced in this subsection. The overhead gases (flue gases from the regenerator and product gas from the gasifier) are traced from the vessels through the Gas Cleanup Systems (300 Area) in Subsection 2.3.

2.2.1 FEED LOCKHOPPERS

After the coal has been ground and sized in the Feedstock Preparation Area (100 Area), it is stored in the fluidized preheater, D-101, where it is kept hot. The sized coal leaves the preheater through a rotary feeder and gravity flows to one of the two feed lockhoppers, F-204A or F-204B. When a lockhopper becomes full, the feed from the preheater is stopped and the lockhopper is automatically isolated and pressurized to system pressure on signal from an operator's control panel. Since there are two F-204 lockhoppers, continuous feed to the system is possible from one while the other is filling.

2.2.2 COAL FEED SYSTEM

The feeders, L-207 A/B, which are located below the F-204 lockhoppers, are rotating pocket-type feeders. These feeders are on/off and RPM-controlled from the 200 Area Control Room. Both feeders are also interlocked to a differential pressure (dP) controller which allows them to run only when the lockhoppers are at approximately 2 psig above system pressure.

From the L-207 feeders the coal flows by gravity to the gasifier, entering slightly above the gasifier sideflow ring. After the lignite or coal feedstock enters the gasifier it is rapidly heated to about 1500°F by the fluidized bed of char. The feedstock is immediately devolatilized and a char is produced. Gasification of the remaining fixed carbon in the char then takes place through reaction of the char carbon with steam.

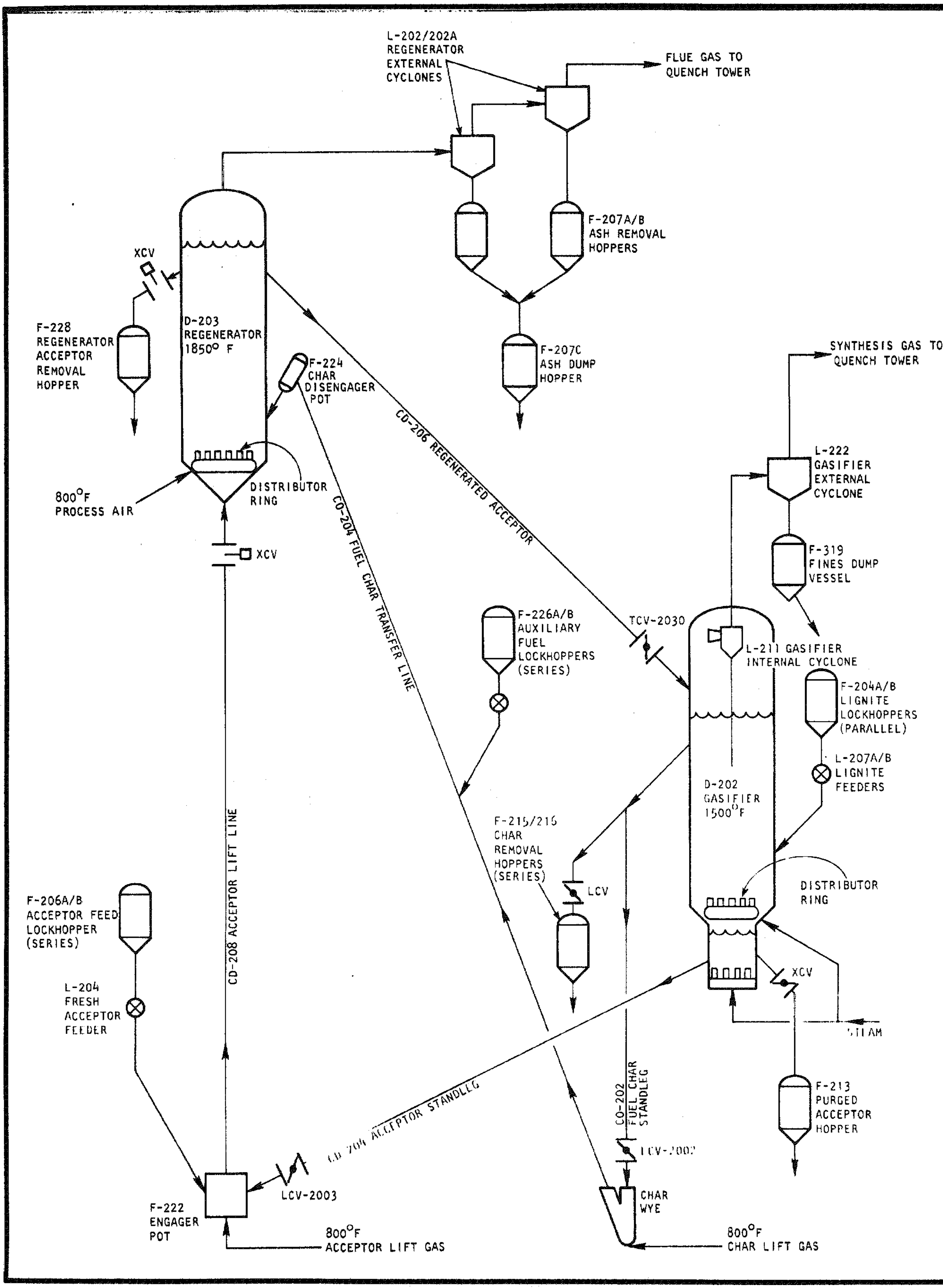


Figure 2-3. SYNTHESIS SYSTEM (200 AREA) FLOW DIAGRAM

2.2.3 CHAR TRANSFER SYSTEM

A portion of the gasifier char is drawn off through the char transfer line, CO-202, and transferred to the regenerator to be burned. The amount of char transferred from the gasifier to the regenerator is controlled by level control valve, LCV-2002, which is a 3-inch stainless steel butterfly valve in the CO-202 pipeline. The line above LCV-2002 completely fills with char from the gasifier, resulting in plug flow through a standleg above the butterfly valve. In this application, the term "standleg" indicates the condition whereby the line is effectively sealed by solids which continually fall from the bottom, through the control valve at a controlled rate, and are replaced at the top of the standleg by solids entering the line.

When the LCV-2002 control valve is operating, a pneumatic pressure controlling device ("kicker") periodically sends a 1 psi impulse signal to the valve actuator. The impulse momentarily "kicks" or opens the valve slightly. This action helps to avoid the bridging of solids in the valve. The char which passes LCV-2002 falls into a V-pot (generally called the "char wye") where it is picked up by gases moving up the char trasfer line, CO-204, to the regenerator. The differential pressure in the top section of CO-204 is then measured by differential pressure controller, dP-2028, and fed back to the level control valve, LCV-2002, which increases or decreases total flow to the regenerator accordingly. The char transfer system is shown in Figure 2-4.

2.2.4 CHAR REMOVAL AND AUXILIARY FUEL SYSTEMS

Two major modifications to the char transfer system from the gasifier to regenerator, which were installed after initial construction, are a char removal system (char removal hoppers, F-215 and F-216) and the auxiliary fuel system (regenerator auxiliary fuel lockhoppers F-226 A/B). Both systems are shown in Figure 2-3. The auxiliary fuel system was still in use at final shutdown, although char removal had been abandoned.

The char removal system, F-215 and F-216, was installed in 1974 and put into service during Run 22B. The purpose of the char removal system was to remove some of the intermediate fines particles (known as "junk") which accumulated in the gasifier char bed causing excessively high bed densities.

The char removal hoppers were connected to the char standleg, CO-202, above control valve, LCV-2002, enabling char to be removed from the gasifier through the same line furnishing fuel char to the regenerator. The char removal system was modified several times during its service life to improve the reliability of fuel char transfer, but was finally removed from the system before Run 40A. Alternative procedures had been devised to "de-junk" the gasifier, which made char removal unnecessary.

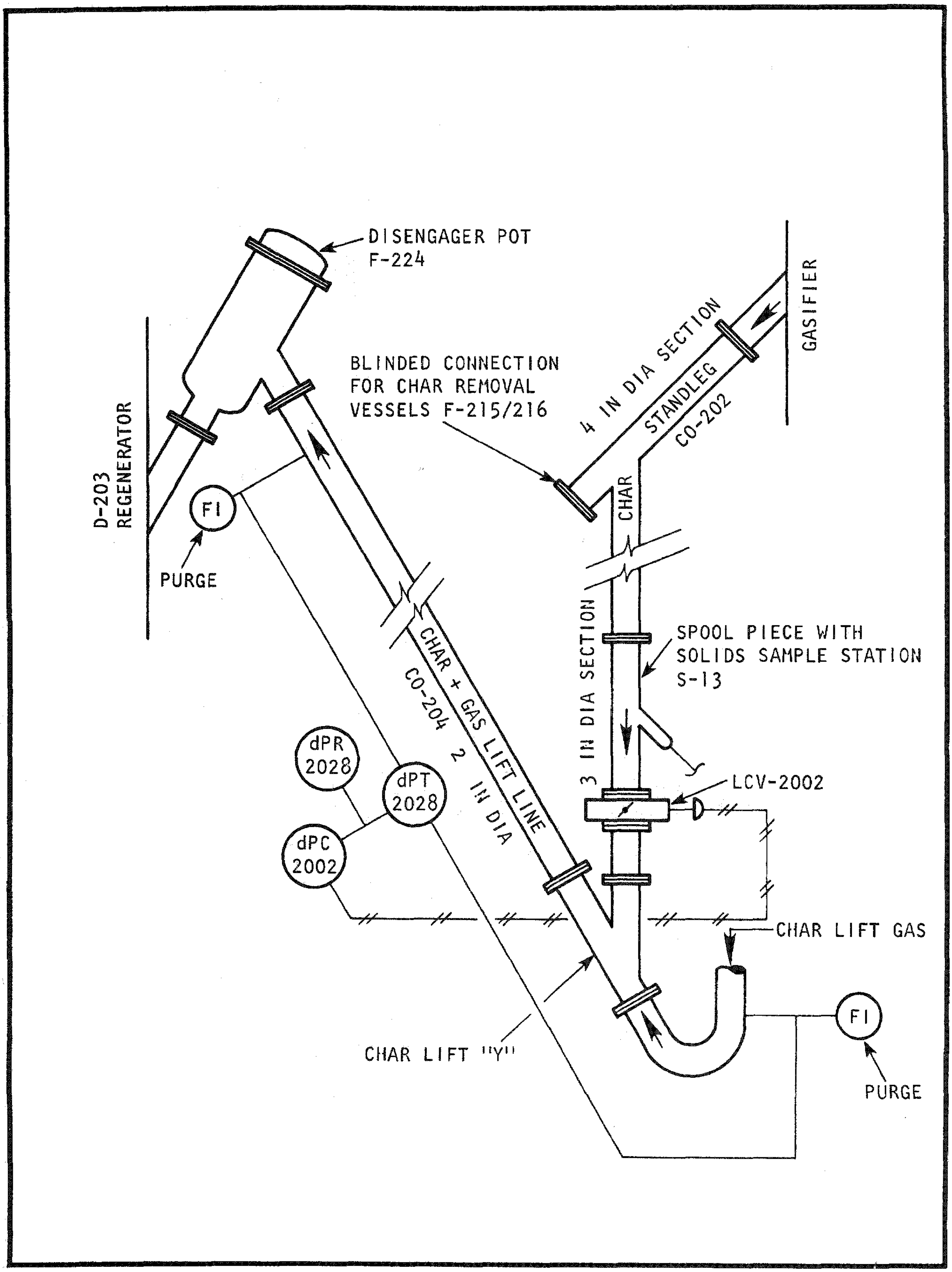


Figure 2-4. CHAR TRANSFER SYSTEM

The auxiliary fuel system, F-226, was first put into service during Run 26A. The system feeds auxiliary solid fuel directly into char transfer line, CO-204, from the F-226 A/B lockhoppers. Natural gas (CH_4) had initally been fed to the spent char line, CO-204, as an auxiliary fuel to keep the regenerator hot during char transfer upsets. However, since extended use of the CH_4 caused undesirable conditions in the regenerator, a different auxiliary fuel feed was necessary.

The use of methane as fuel when the bed temperature was low, caused high CO concentrations in the flue gas. This, in turn, caused localized hot spots at the bottom of the regenerator where the recycled CO-containing flue gas was mixed with incoming air. Solid fuel reacts with air to produce CO_2 as a primary product. The rate of subsequent CO production via the CO_2-C reaction is too slow to result in high CO concentrations in the flue gas.

The use of auxiliary fuel in the regenerator instead of gasifier char also permitted the gasifier heat duty to be reduced considerably when necessary. Char was used as auxiliary fuel for the first several runs. For the first time in Run 40A dried lignite, and in Run 41A subbituminous coal, were used as auxiliary fuels. Lignite or subbituminous coal was used in all subsequent runs.

The auxiliary fuel system provided the process with another degree of freedom and made plant operation more flexible. The use of auxiliary fuel served serveral purposes, which included:

(1) Auxiliary fuel aids in the startup of both the regenerator and gasifier.
(2) Auxiliary fuel provides a reliable source of fuel for the regenerator during char transfer upsets.
(3) The fixed carbon burnoff level in the gasifier can be adjusted without any effect on the regenerator operation.
(4) The commercial practice of injecting gasifier overhead char fines back into the system can be simulated.

Auxiliary fuel fed to the regenerator during startup provides some of the regenerator's fuel requirements that would otherwise be met by char from the gasifier. Char can then be withdrawn from the gasifier at a lower rate. The lower char withdrawal rates allow more gasification of the char particles, thereby lowering the char particle density. Acceptor showering through the gasifier char bed is facilitated by a less dense char bed, so the acceptor circulation rate can be maintained at a reasonable level. This aids gasifier startup, since the circulating acceptor can now take on more of the gasifier heat duty.

Auxiliary fuel also allows char transfer upsets to be handled without affecting regenerator operation. When char transfer from the gasifier is lost, the auxiliary fuel feed rate is increased to supply all of the regenerator fuel requirements. This keeps the regenerator at operating temperature and allows acceptor calcination to continue.

Fixed carbon burnoff in the gasifier can be adjusted independently of the regenerator with the use of auxiliary fuel. The fixed carbon burnoff is primarily controlled by the residence time of the char particles in the gasifier. Decreasing the char transfer rate to the regenerator while keeping coal feed to the gasifier constant increases the average residence time of the char in the gasifier, and, hence, the fixed carbon burnoff. The total fuel feed to the regenerator can be maintained by increasing the auxiliary fuel feed rate as char transfer from the gasifier is lowered. Thus, regenerator operation is not affected by adjustments made in gasifier operation.

The use of auxiliary fuel simulates the commercial operating practice of returning gasifier overhead char fines back to the system. In the pilot plant the gasifier overhead fines (and the quench tower solids) are discarded. In a commercial coal gasification plant, however, all of the coal feed is utilized completely in order to maximize thermal efficiency. Unreacted char fines that are entrained out of the gasifier would be separated from the gas stream through the use of cyclones and fed back to the regenerator to be used for fuel. The feed system for the fines in the commercial plant would be similar to the pilot plant auxiliary fuel feed system.

The F-226 lockhoppers are set up in series, allowing continuous feed to the regenerator from the lower hopper while the top hopper is depressurized and filled. A differential pressure recorder control, dPRC, is used to hold higher-than-system pressure in the lower lockhopper, to ensure positive flow from the hopper, through a rotary feeder, to the system. The rotary feeder is RPM-controlled from the control room.

The presence of auxiliary fuel entering the char transfer line, CO-204, is sensed by differential pressure transmitter, dPT-2028, which, in conjunction with differential pressure controller, dPC-2002, controls the total flow of fuel to the regenerator. Therefore, when auxiliary fuel is increased, controller, dPC-2002, will automatically cut back on the amount of char LCV-2002 withdraws from the gasifier. Thus, the total amount of fuel to the regenerator is kept constant.

2.2.5 REGENERATOR

The fuel (gasifier char and/or auxiliary fuel) is pneumatically conveyed by inert gas through the CO-204 transfer line to the stop pot, F-224 (also called the char disengager pot). Within the stop pot, the flow direction of the fuel and conveying gas is changed so that the fuel is injected downwardly into the regenerator. The stop pot also serves as a check valve to help prevent the contents of the regenerator from backflowing and plugging the transfer line, CO-204, in the event that lift gas flow is lost or the regenerator acceptor bed becomes defluidized due to circumstances such as power failures.

Solids from F-224 enter the regenerator just above the distributor ring. In the regenerator, air is used to burn the fuel in the fluidized acceptor bed. The fluidized acceptor bed is maintained at about 1850°F and at a depth of approximately 20 feet.

Fluidizing gases are supplied to the regenerator through the acceptor lift line, CD-208, and through the distributor ring located at the bottom of the regenerator. Prior to Run 42, all air was supplied to the regenerator through the ring distributor and recycled regenerator flue gas, which contains no free oxygen, was used for acceptor lift gas. This was done to prevent combustion in the acceptor lift line should gasifier char be transferred with recarbonated acceptor during a process upset. Under such conditions, the acceptor line design which featured the use of a metal liner would be inadequate.

Prior to Run 41, the acceptor lift line was modified to eliminate the metal liner. An all-refractory design was used. Beginning with Run 42, the normal operating mode was to use air alone for acceptor lift gas.

The air admitted to the regenerator supplies the oxygen for combustion of the char. Normally 75,000 to 85,000 SCFH of air is needed. The total gas flow in the acceptor lift line is usually about 60,000 SCFH; thus, when air is used for lift gas, most of the combustion air enters the regenerator from the lift line. The air flow to the distributor ring is about 20,000 SCFH.

The temperature in the regenerator fluid bed is controlled at about 1850°F. This high temperature is needed to provide the driving force for calcination of the acceptor. The fluidized bed in the regenerator provides good mixing of the char and acceptor. As a result, the heat released by char burning is dispersed throughout the bed so that the temperature gradient between the top and bottom of the bed is usually only about 30°F. The heat released from char burning is about 7 to 8 million Btu/hr.

Because the regenerator is operated under reducing conditions, the regenerator temperature is controlled by adjusting the air flow rather than the fuel rate. Since the total gas flow in the lift line is normally set at 60,000 SCFH in order to insure steady transport of the acceptor solids up the lift line, air flow changes are made to the distributor ring air flow. High temperatures are brought down by decreasing ring air flow so that less char combustion takes place. Likewise, when regenerator temperatures are low, air flow is increased to the ring so that more combustion takes place and the bed temperature increases.

High carbon burnout levels are achieved in the regenerator. Nearly all the char feed to the regenerator is burned to a fine ash which is elutriated out of the regenerator bed. The small amount of carbon present in the ash normally represents less than 1 percent of the total carbon fed to the gasifier.

The regenerator is operated with a reducing atmosphere in order to prevent transient liquid derived deposits. The carbon monoxide concentration is held in the range of 1 to 5 percent. This small percentage of CO has been shown in bench-scale work to prevent deposit formation. In the pilot plant no problems with regenerator deposits were incurred while the regenerator was operating under reducing conditions.

While a small percentage of CO in the regenerator exit gas is desirable for the purpose of preventing deposits, high CO levels are undesirable in both the pilot plant and in a commercial plant. During startup and throughout the early pilot plant runs, regenerator overhead gas was frequently recycled back to the regenerator. If the gas contains a large percentage of CO, then rapid burning of the CO will take place at the regenerator inlet. High temperatures could result at the inlet and cause the char ash to fuse. In a commercial plant the CO contained in the regenerator overhead gas must be utilized in order to maximize the plant's thermal efficiency. Since it is preferable to burn carbon in the regenerator as fuel rather than in downstream waste heat boilers, the CO level in a commercial plant would be kept at the minimum level required to prevent deposit formation. It should be noted that operation of the regenerator under oxidizing conditions was successfully demonstrated in Run 45. Detailed operating data are contained in the Run 45 section of Volume 8, Book 6 of this report. The results and process performance are discussed in Section 5.4.4, Volume 12, Book 2, of this report.

The CO concentration in the regenerator is controlled by regulating the rate of char withdrawal from the gasifier. High CO concentrations are lowered by reducing char transfer from the gasifier, and low CO concentrations are brought up by increasing char transfer.

During startup the regenerator is initially heated by recycling hot gas through the vessel. The recycle gas is heated by natural gas fired furnaces. When the regenerator reaches 1000°F, char transfer from the gasifier is established. Char combustion continues the heating of the regenerator to the process temperature of about 1850°F.

A minimum of 800°F is required in the regenerator before char combustion is attempted. This requirement ensures that the char fed to the regenerator will burn immediately.

The regenerator has two acceptor outlets slightly below the top of the fluidized bed. The smaller of the two outlets is the purged acceptor dump, UAD-205, which leads to the regenerator acceptor removal lockhopper, F-228. The acceptor flows by gravity from the regenerator through a butterfly valve, XCV-2187, to the F-228 lockhopper.

Acceptor is withdrawn from the regenerator via F-228 to make room for continuous makeup of fresh acceptor and to help remove accumulated junk (intermediate fines) such as acceptor fines and silica particles from the bed. The regenerator bed level controls the amount of acceptor dumped to F-228.

The larger calcined acceptor outlet from the regenerator is the overhead standleg line, CD-206. Hot acceptor flows from the regenerator down CD-206 and into the gasifier where it provides the necessary heat to sustain the gasification reactions. The flow of acceptor down CD-206 is controlled by temperature control valve, TCV-2030, which is also a butterfly valve similar to LCV-2002. Like LCV-2002, TCV-2030 is equipped with a kicker to keep solids from bridging in the valve.

Since the gasifier is always operated at slightly higher pressure than the regenerator, balance gas purges above TCV-2030 are necessary to create a positive differential across the valve in the direction of solids flow.

2.2.6 GASIFIER

Hot calcined acceptor from the regenerator enters the gasifier above the char bed and showers into it. As the acceptor works its way down through the fluidized bed, the calcined particles react exothermicly with carbon dioxide. By the time the acceptor has reached the boot in the bottom of the gasifier, the active CaO in the acceptor has been completely recarbonated. The acceptor is ready to be sent back to the regenerator to be recalcined.

The boot in the bottom of the gasifier has a smaller diameter than the rest of the vessel. The fluidizing gas velocity in the boot is kept high enough to completely strip char particles from the showering acceptor, but the velocity is not high enough to back-mix the acceptor into the gasifier bed. Consequently, when the acceptor particles reach the boot they continue to settle. Once in the boot, the recarbonated acceptor can be either transferred back to the regenerator or withdrawn to the purged acceptor lockhopper, F-213.

Prior to the installation of the regenerator acceptor removal lockhopper, F-228, F-213 had been used exclusively to withdraw spent acceptor from the system. After the installation of F-228, F-213 remained in service to be used in emergencies and to dump the gasifier after plant shutdown.

The recarbonated acceptor which enters the gasifier boot leaves through acceptor standleg, CD-204, en route to the engager pot, F-222, and back to the regenerator. The amount of acceptor passing through CD-204 is controlled by an automatic level controller. As the acceptor level in the gasifier boot increases, level control valve, LCV-2003, opens and acceptor flows to the engager pot. LCV-2003 is also a butterfly valve equipped with a kicker and purges similar to those on control valve, TCV-2030.

The acceptor which enters the engager pot is picked up by fast-moving carrier gas (air or recycle gas) which lifts it via line (CD-208) to the regenerator.

The engager pot is also the addition point for fresh acceptor. Fresh acceptor from feed hopper, F-206B, is continually fed to the engager pot through a rotary feeder. F-206B is kept full by F-206A, which can be isolated, depressurized, and filled without stopping acceptor makeup to the system.

Acceptor that returns to the regenerator is recalcined and is then ready for another cycle through the gasifier.

2.2.7 DEVOLATILIZER

Included in the original plant design was a devolatilizer, D-201, and its supporting equipment (quench towers, knockout pots, water jackets, etc.). The design concept of the devolatilizer was to remove the volatile constituents of the coal prior to exposure to gasification conditions. However, bench-scale studies during pilot plant construction, together with economic analyses, indicated that simultaneous devolatilization and gasification in a single vessel was preferable. Accordingly, the pilot plant devolatilizer was never put in service.

Although it was unnecessary to put the devolatilizer into service, its similarity to the gasifier system made it an invaluable source of spare parts. Without materials from the devolatilizer, certain repairs to the gasifier would have been considerably more costly and time consuming.

2.3 GAS CLEAN-UP SYSTEMS (300 AREA)

The 300 Area comprises two separate gas clean-up systems; one to scrub, cool, and dry the regenerator flue gas, and one to scrub, cool, and dry the gasifier product gas. Flow diagrams of these systems are shown in Figures 2-5 and 2-6.

2.3.1 REGENERATOR FLUE GAS

As shown in Figure 2-3, the flue gas which exits the regenerator overhead is a combination of nitrogen, CO_2, CO, acceptor fines, and coal ash. The overhead temperature is normally 1850°F. Since power recovery equipment was not included in the pilot plant, the overhead flue gas line is water jacketed. Acceptor fines and coal ash which are carried by the flue gas are removed by two cyclones. L-202A and B, which are piped in series for maximum effect. The ash is collected in lockhoppers which are then dumped to tote bins for disposal.

After the majority of solids have been removed from the regenerator overhead gases, the flue gas is routed to the regenerator quench tower, E-302, for additional scrubbing. The flue gas initially is contacted with potable water in two parallel venturi scrubbers which are just upstream of the quench tower. This treatment quickly wets the remaining solids in the flue gas and helps to prevent quench tower deposit scale from forming.

The regenerator quench tower is a standard plate-and-donut type tower with counter-current gas and quench water flow. In the original plant design, all the quench towers in the structure were equipped with horizontal quench water separators to remove oils from the foul quench water before it was sent to the holding pond. However, since oils were never produced and the separator had a tendency to fill with fines, all separators were eventually removed from the system. The lower sections of the quench towers then served as separators, and the foul quench water went directly to the pond.

The scrubbed and cooled flue gas which leaves the quench tower overhead goes through the SO_2 scrubber, E-309, and to water knock-out pots (K.O. pots) where the entrained water is removed. The quenched flue gas is then either vented or compressed to be used as recycle or purge gas.

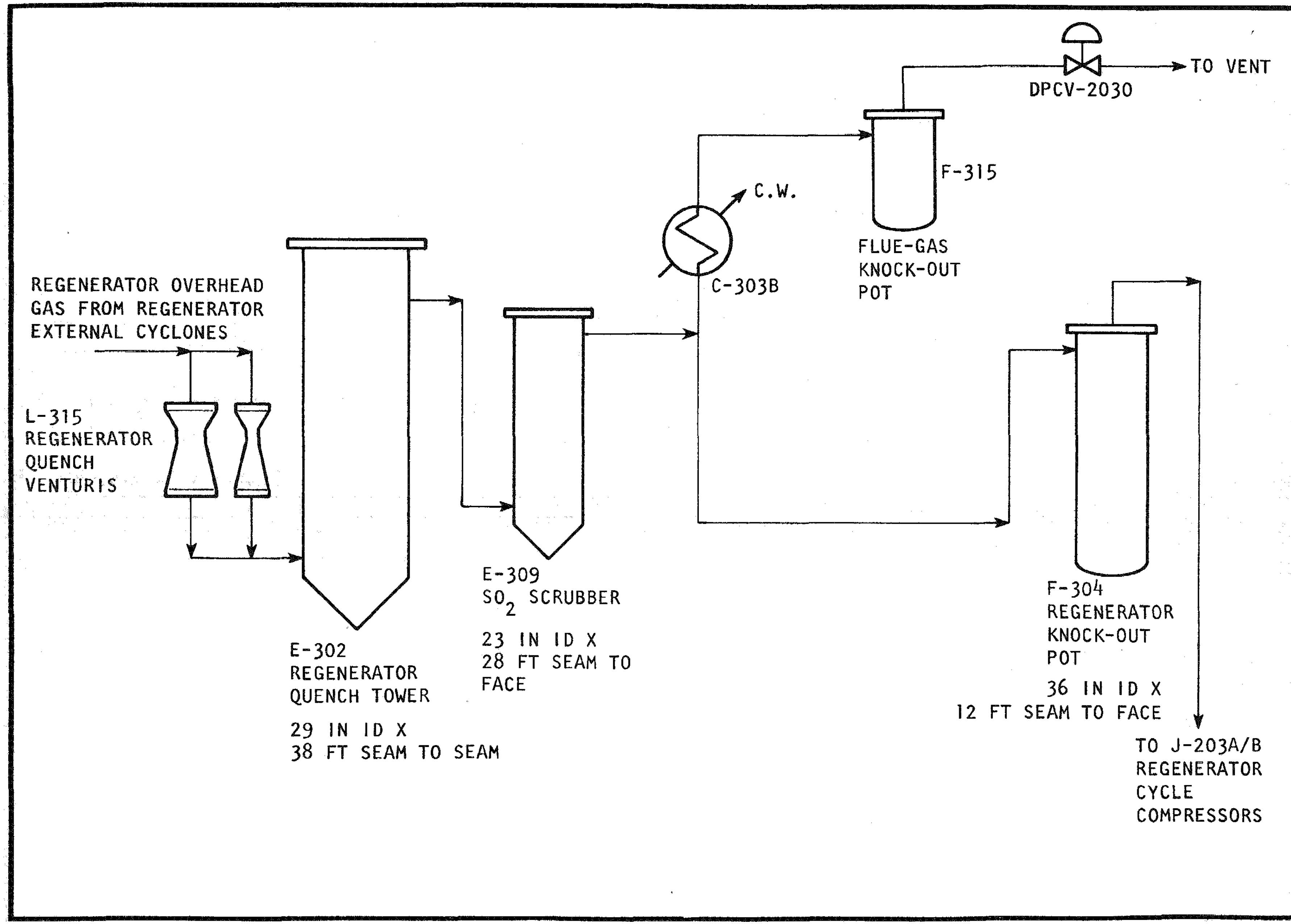


Figure 2-5. REGENERATOR FLUE GAS CLEANUP SYSTEM (300 AREA) FLOW DIAGRAM

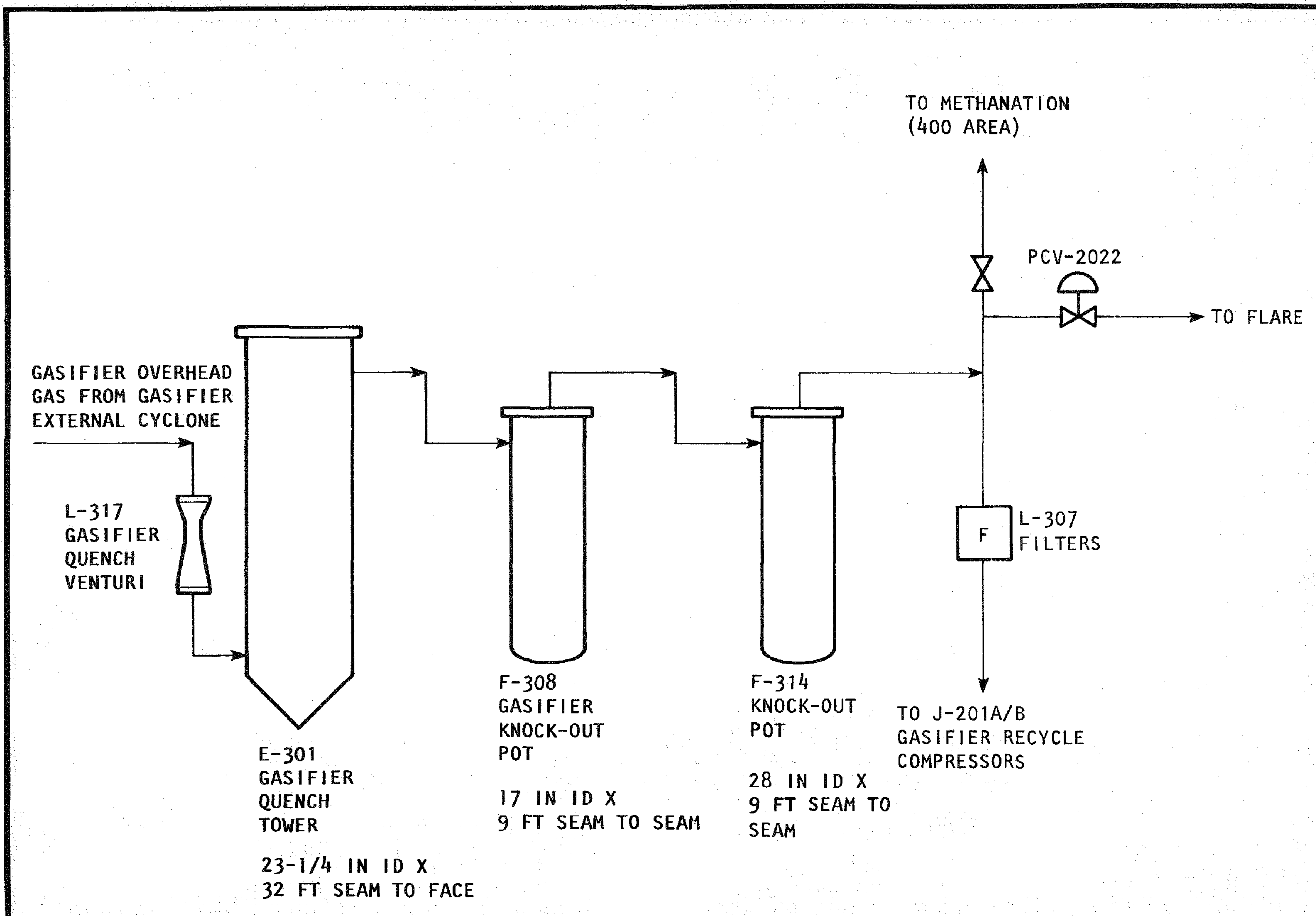


Figure 2-6. GASIFIER PRODUCT GAS CLEANUP SYSTEM (300 AREA) FLOW DIAGRAM

2.3.2 HOT POTASSIUM CARBONATE SYSTEM

A hot potassium carbonate system, designed to remove CO_2 from the flue gas after it passed through the regenerator quench tower, was part of the original plant. The CO_2 which was removed from the flue gas was then to be compressed for further use in the process as inert gas or as a means of adjusting the gasifier CO_2 partial pressure when desired. However, the short duration of early runs made CO_2 removal from the regenerator flue gas unfeasible; therefore, high-pressure inert gas was substituted for CO_2 in the process. The hot potassium carbonate system was nonetheless kept serviceable in order to provide potassium carbonate to the SO_2 scrubber to control the pH of the scrubber water. Later, the hot potassium carbonate system equipment was used in the methanation area as part of the synthesis gas purification facilities.

2.3.3 GASIFIER PRODUCT GAS

The gasifier overhead product gas consists primarily of H_2, CH_4, N_2, and CO_2.

At 1500°F, gas which leaves the gasifier overhead is handled very much like the regenerator flue gases. An internal cyclone in the gasifier removes most of the entrained solids before the gas leaves through a water-jacketed line. The product gas cools in the jacketed line and then passes through another cyclone for further removal of solids. After passing through the external cyclone, the gas flows through a venturi and into the gasifier quench tower, E-301. After the gas leaves the gasifier quench tower, it is routed through two water knock-out pots and eventually compressed as recycle gas, vented to the flare, or sent to the methanator.

2.4 METHANATION UNIT (400 AREA)

The Methanation Unit of the CO_2 Acceptor Process Gasification Pilot Plant, which was completed in 1975, is covered in detail in Book 3, Section 8 of this report.

2.5 UTILITIES (500 AREA)

2.5.1 BOILER

The Boiler, U-501, is a 20,000 lb/hr boiler providing steam to two systems called the 350 psig and the 50 psig systems. (See Figure 2-7.) The 350 psig system provides steam for the following.

(1) Gasifier steam superheater, B-207B, for gasification.
(2) Jacket steam drum, F-218, and regenerator steam drum, F-219, for warm-up.
(3) Cooling tower heaters, L-509A and L-509B, for winter use.
(4) Process steam to the methanation feed to adjust gas composition as required.
(5) Preheater furnace, B-102, for fluidization and drying of ground lignite and coal feedstocks.

The 50 psig steam system provides steam to the following:

(1) The 50 psig utility hose station which has steam available on each floor of 200 structure, plus the Methanation and 100 Areas.
(2) Steam tracing throughout the plant.
(3) Steam heaters in the 100 and 200 Areas.
(4) Inert gas heat exchanger, C-508.
(5) Compressor jacket water heater, C-210, to heat water going to the jackets on all the compressors in the plant.
(6) Potassium carbonate storage tank, F-302; potassium carbonate stripper reboiler, C-307; boiler feed water heater, U-503; Dowtherm expansion tank, F-407; and propane storage tank, F-509.

The pilot plant initially also had a third steam system which was a 25 psig system. This was used for steam tracing, but was found to be inadequate because of the cold weather in Rapid City. The steam reducing valve was taken out, and the 25 psig system then became part of the 50 psig system.

2.5.2 INERT GAS GENERATOR

The inert gas generator, U-519, burns methane gas with air under approximately 1 psig pressure. The oxygen in the air is consumed until the oxygen content in the outlet gas is 0.5 percent. The maximum output of the inert gas generator is 20,000 SCFH (wet). After being cooled, the inert gas is split into two 10,000 SCFH streams. (See Figure 2-8.) One stream goes to an inert gas low pressure compressor, J-516, which boosts the pressure to 18 psig. This gas is used in the following systems:

(1) Recycle CO_2 compressor, J-309, that compresses gas to 385 psig to be used for char lift gas.
(2) Auxiliary purge system and emergency inert to lignite preheater, D-101, in the coal grinding area.

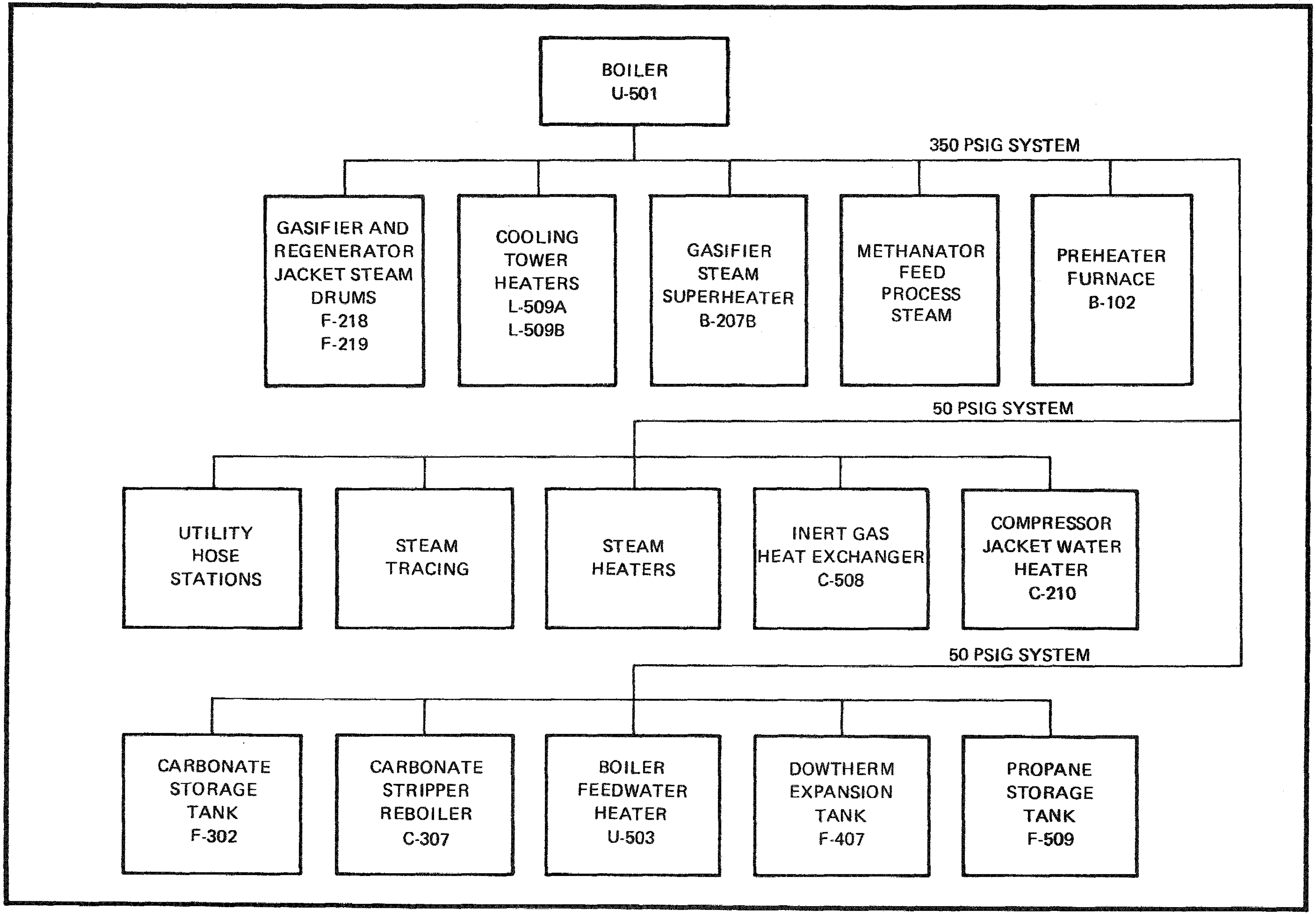


Figure 2-7. STEAM SYSTEM

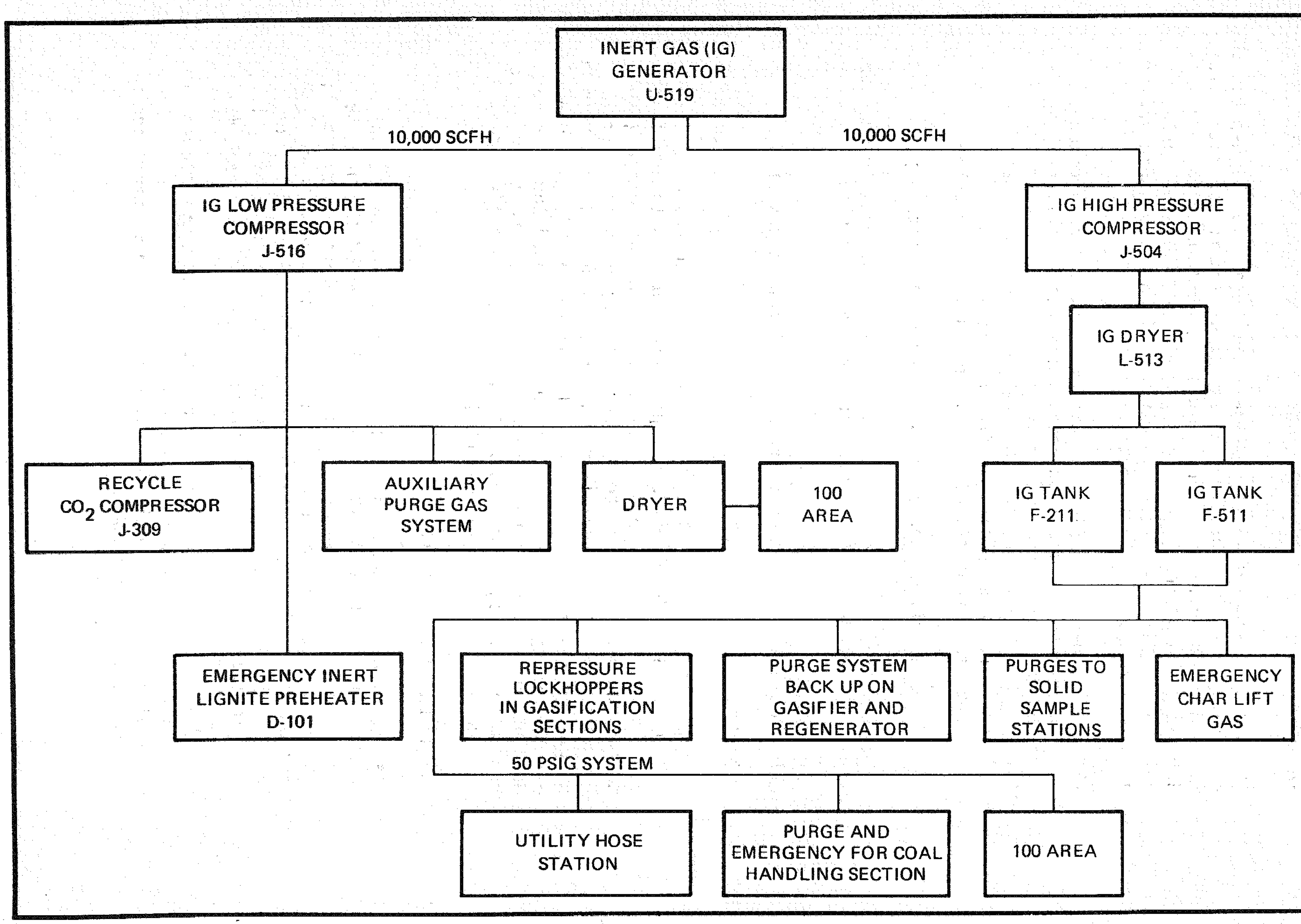


Figure 2-8. INERT GAS SYSTEM

There is a drier on this system to dry the gas going to the 100 Area.

The second stream of inert gas goes to an inert gas high-pressure compressor, J-504, where the gas is compressed to 485 psig. It is then dried by the IG drier, L-513, reduced in pressure to 350 psig, and placed into two large holding inert gas tanks, F-211 and F-511, for surge capacity. This high-pressure inert gas provides gas to the following:

(1) To repressure all lockhoppers in gasification section when vessels are periodically taken out of service for dumping and filling.
(2) As a backup system for the two purge systems on all pressure taps in the gasifier and regenerator.
(3) Provides purges to solids sample stations throughout the plant.
(4) Provides extra char lift gas in cases of emergency.

The high-pressure inert gas is reduced from 350 psig to 50 psig in the 200 structure to provide the following services:

(1) Utility hose station gas.
(2) Purges and emergency inert for coal handling section.

2.5.3 PROCESS AIR

Atmospheric air is compressed in the main three-stage process air compressor, J-202, over any pressure range up to 350 psig. (See Figure 2-9.) There is an offtake on the last stage going to the instrument air system in case of compressor failures. The main flow goes to an air surge tank, F-225, and then through an air heater, B-203, that heats the air up to 800°F. The heated air then goes to the regenerator where it is used for char combustion in the regenerator. Part of the air can be transferred to another heater, B-205, where it is heated to 800°F and used for acceptor lift gas in line CD-208.

2.5.4 INSTRUMENT AND PLANT AIR

Instrument air is compressed from ambient to 100 psig in air compressors, J-502 and J-503. It is then cooled through the instrument air aftercooler, C-501, and goes into a surge tank, F-520, to knock out moisture. The air is then sent through an instrument air drier, L-516, where the rest of the moisture is eliminated. The air then goes into the air receiver, F-503, and from there divides into instrument and plant air systems.

(1) The instrument air provides air to all instrument control valves and controllers throughout the plant.
(2) The plant air provides air at utility hose stations and the fire water standlegs.

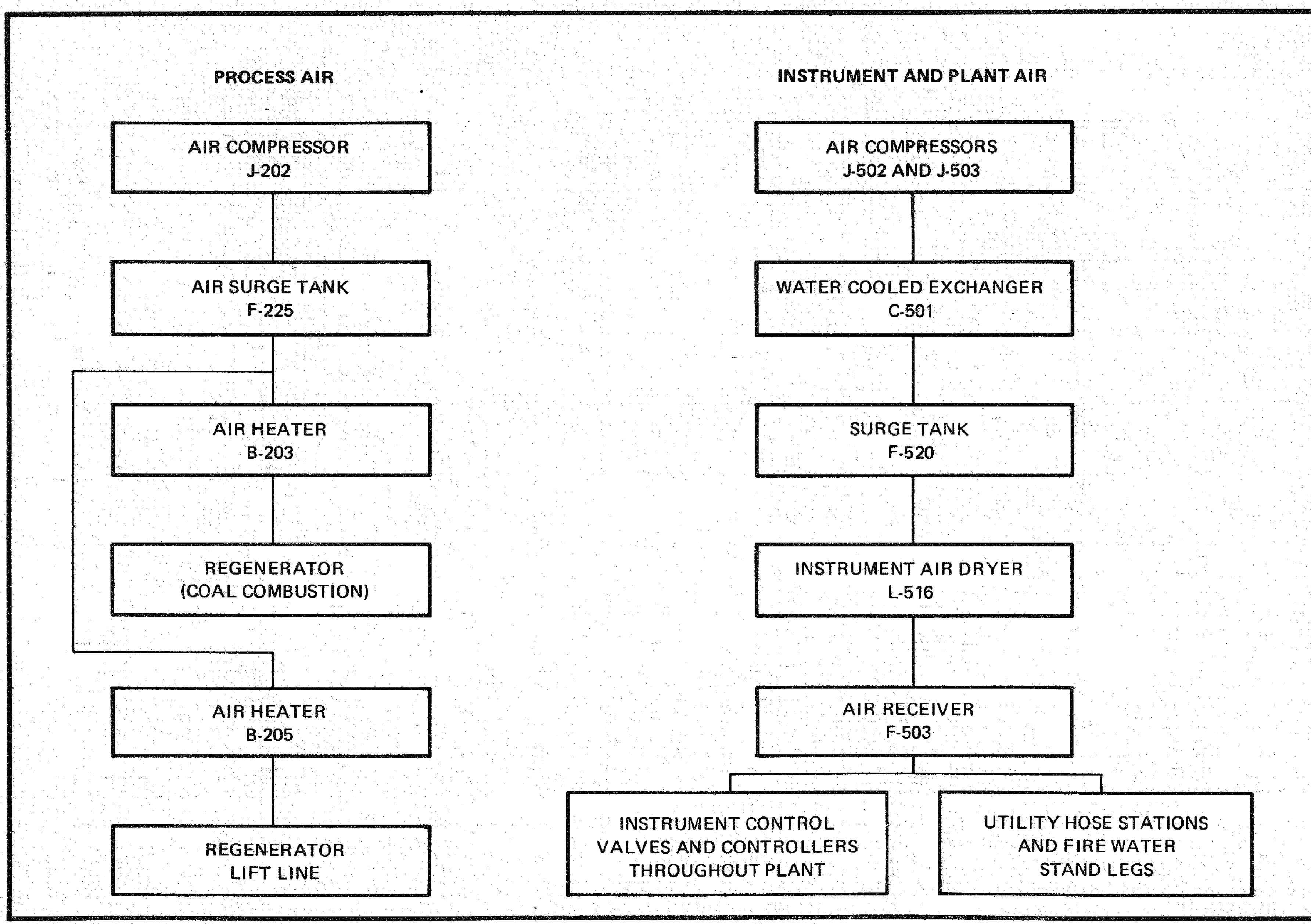


Figure 2-9. PROCESS AIR AND INSTRUMENT/PLANT AIR SYSTEMS

2.5.5 PURGE GAS SYSTEMS

Flue gas from the regenerator is used as purge gas on regenerator pressure taps, and a similar system is employed on the gasifier. In each purge gas system, the gas is compressed from 120 psig to approximately 230 psig and subsequently dried in desiccant dryers.

2.5.6 NITROGEN SYSTEM

A high-pressure nitrogen gas system is provided in the pilot plant as a back-up for both the high- and low-pressure inert gas systems and to the instrument air system. Purges to the calcined acceptor standleg and to the line which connects the regenerator with the spent acceptor dump hopper, F-228, are also supplied with pure nitrogen.

The nitrogen system forces liquid nitrogen through heat exchangers which use natural convection of ambient air to vaporize the liquid. The system has a capacity of 40,000 SCFH N_2 gas at 450 psig. Because of cost, the use of the nitrogen system is minimized.

2.5.7 COOLING AND FIRE WATER SYSTEM

The cooling water is taken from the city water and put in the plant cooling water basin, U-504. (See Figure 2-10.) The water level is controlled by a level control valve. The water is circulated by two cooling water pumps, J-505A and J-505B, with capcities of 1500 GPM each. The water is circulated to all coolers, compressors, and pumps throughout the 100, 200 and Methanation Areas. Cooling tower water is also provided to each floor at the utility hose station. The pressure is held at approximately 90 psig by holding back-pressure on system to get water to the eighth floor of the structure. The cooling water returns to the cooling tower. A fan is provided to draw air up through the tower when necessary for cooling. The cooling tower is also equipped with steam heating for winter use.

The fire water system is also located in the cooling tower pump room and gets its source from the same basin as the cooling water pumps. (See Figure 2-9.) The fire water system in the cooling tower room consists of a fire water pump, J-501, emergency fire water pump, J-508, and a fire water jockey pump, J-509. This system performs the following functions:

(1) The jockey pump, J-509, is a small volume pump that works off a pressure switch to keep the underground fire water line full and to make up water to compensate for small leaks in the system.

(2) If the jockey pump cannot keep the pressure up, the main electric fire water pump, J-501, starts providing 1500 GPM of fire water.

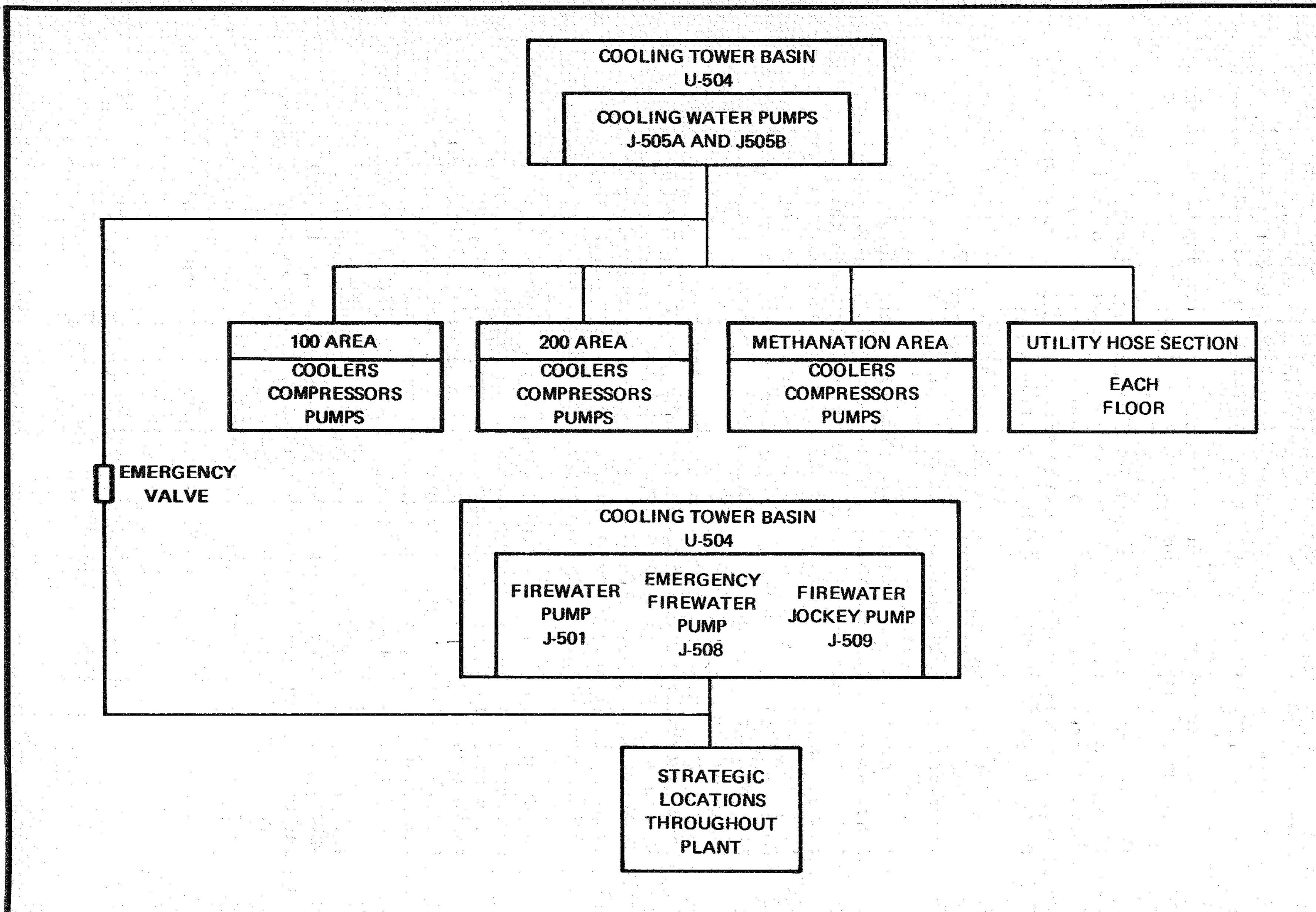


Figure 2-10. COOLING AND FIRE WATER SYSTEM

(3) In case a large volume of water is used or the electric fire water pump fails as the pressure drops on the system, the propane engine emergency fire water pump, J-508, starts to provide fire water.

(4) In cases where more water is needed, the cooling water pumps can be valved into the fire water system.

The fire water lines run underground to strategic locations throughout the plant. Where the system is exposed to cold weather, the standlegs above ground are filled with plant air using a special trip valve between the air and fire water to prevent the fire water line from freezing.

2.5.8 EFFLUENT HANDLING

The flare system is provided primarily to burn the gas that is produced from the gasifier. All the relief valves that are on flammable gas systems vent into the flare header. The flare header goes to a large drum where moisture is removed; then the flammable gas flows to the flare stack. As the gas travels up the flare header, it goes through a flame arrestor to prevent flashback. Moisture in the gas is removed and sent back to the knock-out pot. The gas continues to travel upward in the flare stack until it reaches the top and is ignited by three natural-gas pilot burners.

There are two settling ponds in the plant. The small settling pond was provided to receive light oils from gasification. The oil was to be pumped along with any water into the thermal oxidizer to be burned. The gasification system never produced any oil, so the small pond was bypassed and its use discontinued.

The fines from coal grinding, the effluents from the gasifier and regenerator quench systems, and the plant chemical drains are discharged to the large settling pond. Solids settle to the bottom of the pond, while the effluent water, after passing through a straw dam and a surface float, is discharged to the city sewer. The pond is periodically drained, and the solids are dredged and trucked to the city sanitary land fill.

The thermal oxidizer was to be used in conjunction with the small pond for disposal of light oils. The light oils never materialized in the process; therefore, its use was very limited.

2.6 INSTRUMENTATION

The CO_2 Acceptor Process Gasification Pilot Plant presents unique process control problems. Because the large amounts of solids transferred pneumatically throughout the high-temperature, high-pressure system and the large fluidized beds maintained in the reactor vessels, delicate pressure balances were a necessity. Instrumentation is thus critical in the control of the solids movement and the pressure balance of the system. The following subsections summarize the instrumentation used to measure temperature, pressure, flow rates, and gas composition. Details of plant instrumentation are presented in the Appendices, Subsection 13.3 of Book 4.

2.6.1 PRESSURE AND TEMPERATURE MEASUREMENTS

2.6.1.1 Gasifier

Figure 2-11 shows the location of pressure and temperature sensing elements in the gasifier. As described in Book 4, Subsection 13.3, this configuration was used after Run 18 and evolved as a result of a series of revisions to overcome problems of corrosion, erosion, and plugging of probes within the vessel. Figure 2-12 illustrates a typical gasifier tap which is inserted through the vessel wall.

2.6.1.2 Regenerator

Figure 2-13 shows the location of pressure and temperature sensing elements in the regenerator; this configuration was adopted following Run 5.

Figure 2-14 presents the details of the tap construction. As in the case of the gasifier taps, this construction permitted safe replacement of thermowells during a run as well as the use of rod-outs if the tap became plugged with solids.

2.6.1.3 Temperature-Pressure Taps in Jacketed Transfer Lines

Pressure and differential pressure measurements on the jacketed solids transfer lines were originally made through 1/4-inch tubes that entered through the jacket, made several spiral turns in the annular space, and entered the side of the inner pipe. The spiral turns within the jacket allowed for differential movement between the inner pipe and the jacket when the system was being heated up or cooled down. Unfortunately, when the 1/4-inch tubes plugged and could not be blown clear with a jet of high-pressure gas, the spirals inside the jacket prohibited other means of clearing the tap. A sketch of the original 1/4-inch, spiral coiled-pipe tap is shown in Figure 2-15.

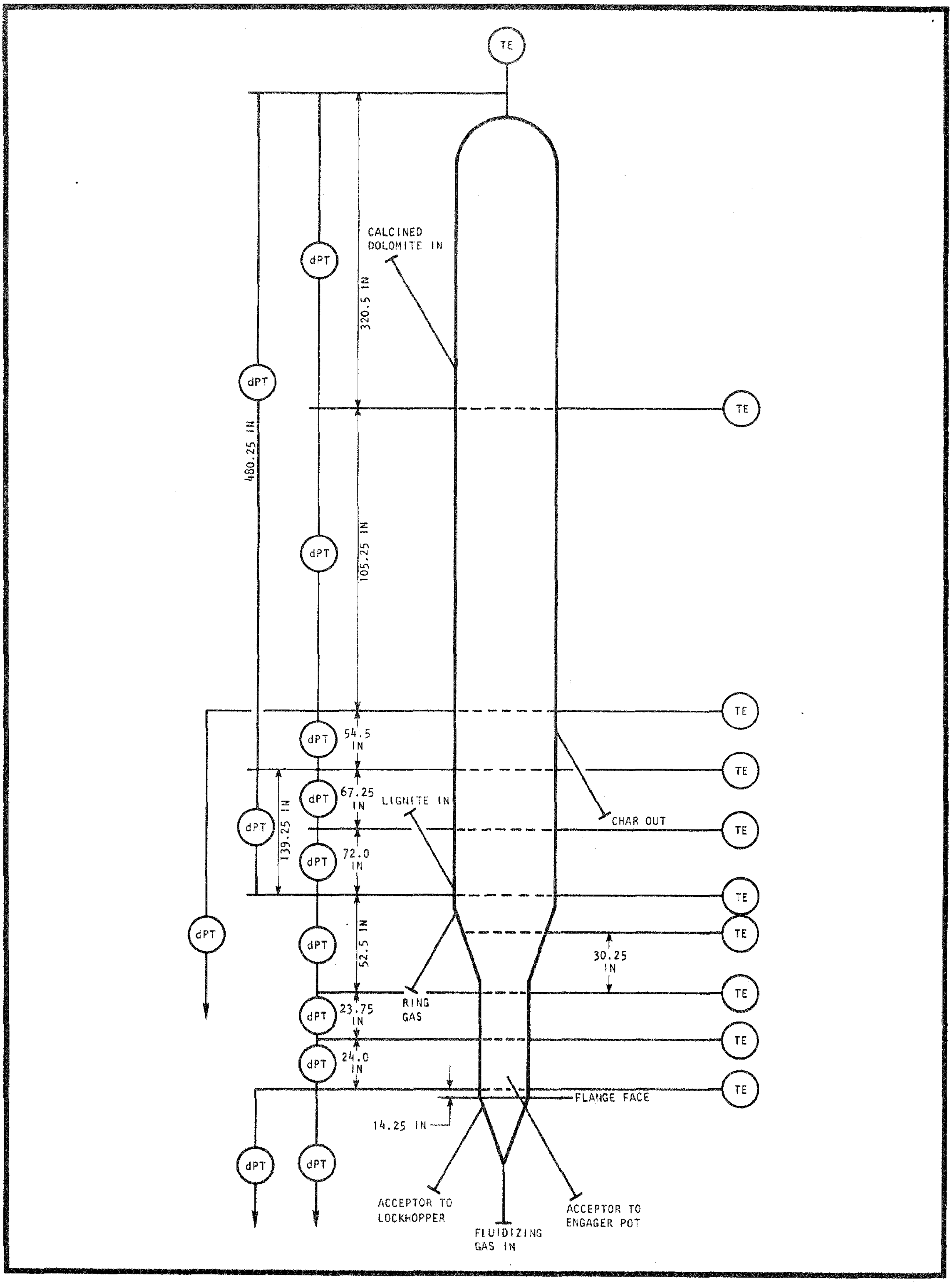


Figure 2-11. GASIFIER DIFFERENTIAL PRESSURE TRANSMITTER (dPT) AND TEMPERATURE ELEMENT (TE) LOCATIONS

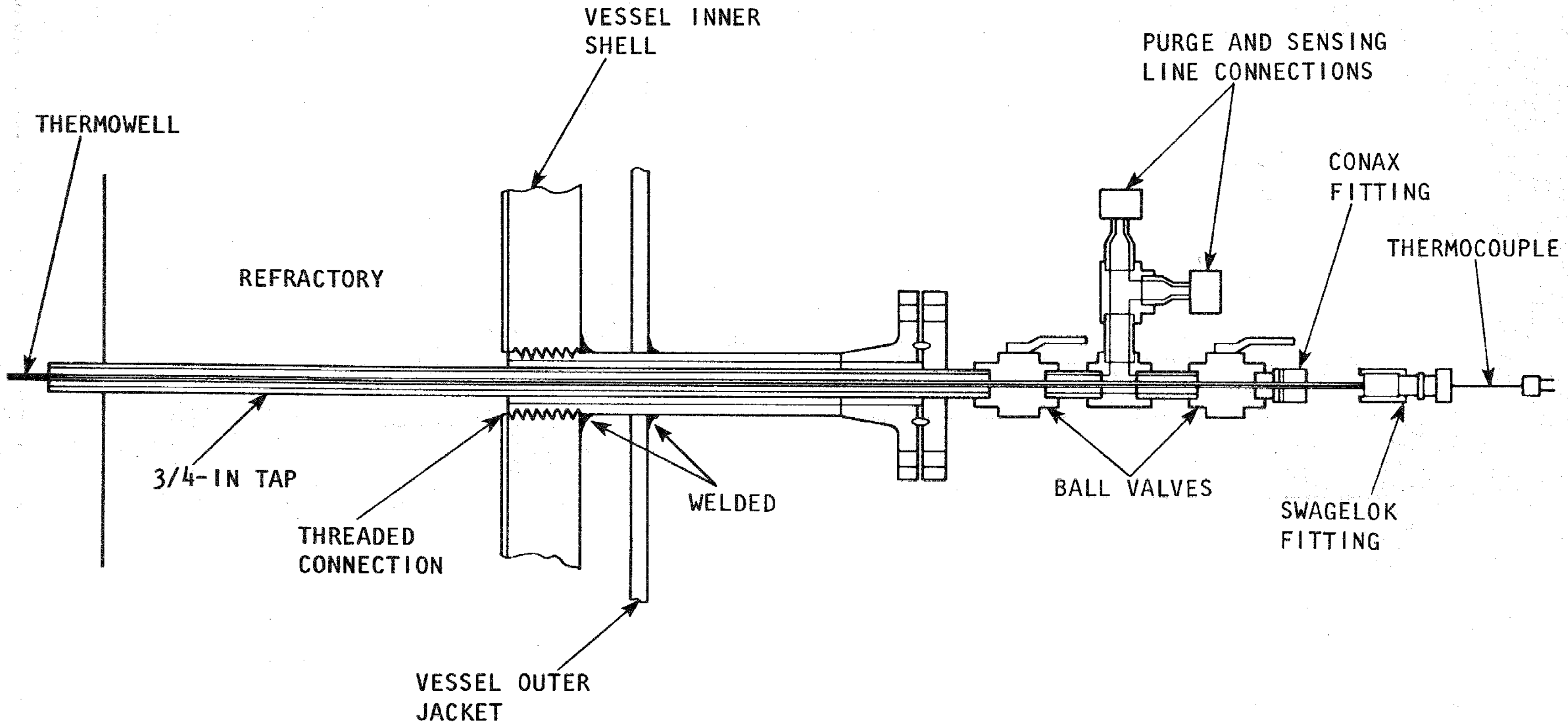


Figure 2-12. TYPICAL GASIFIER SIDE TAP

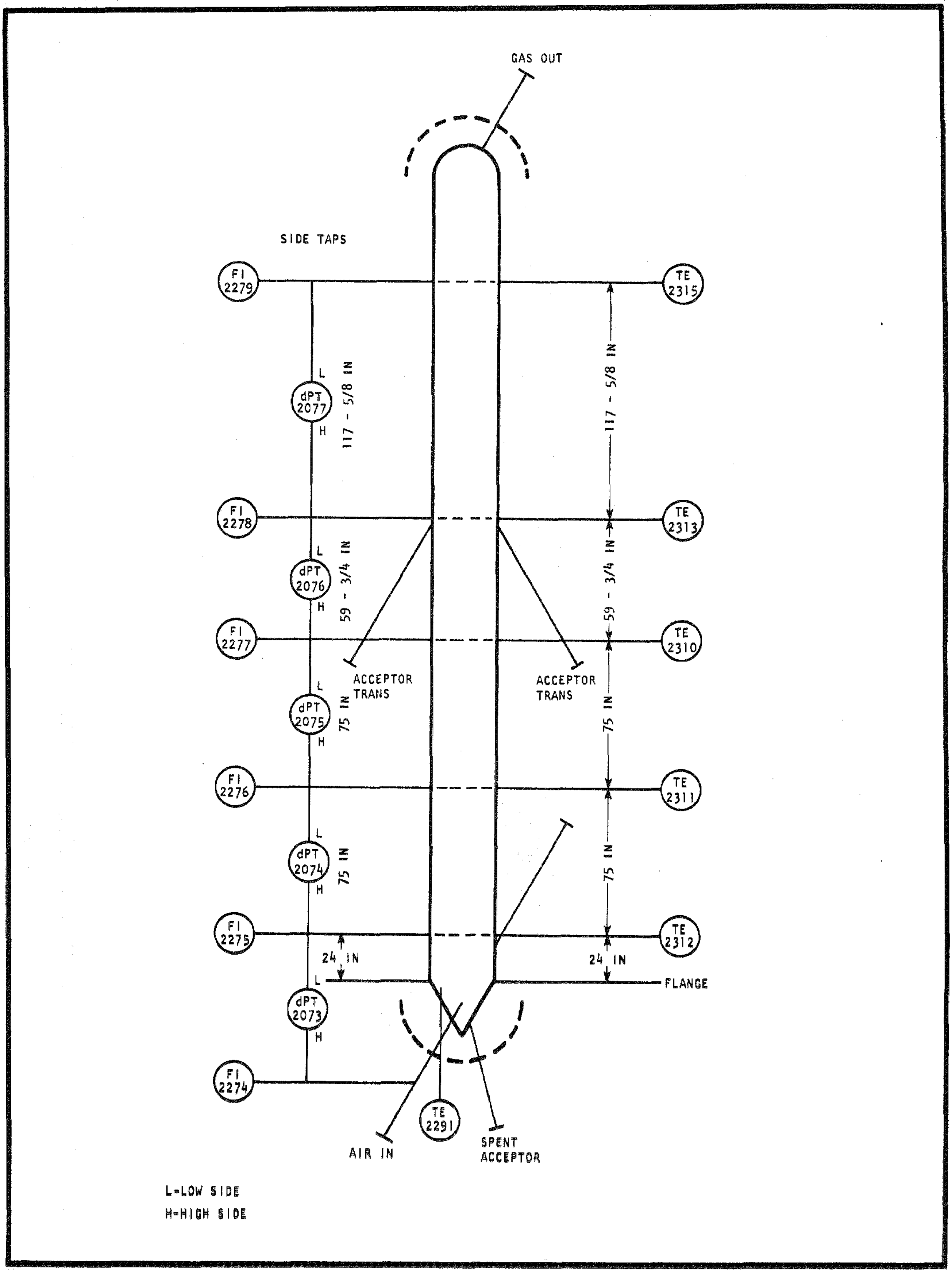


Figure 2-13. REGENERATOR dPT AND TE LOCATIONS

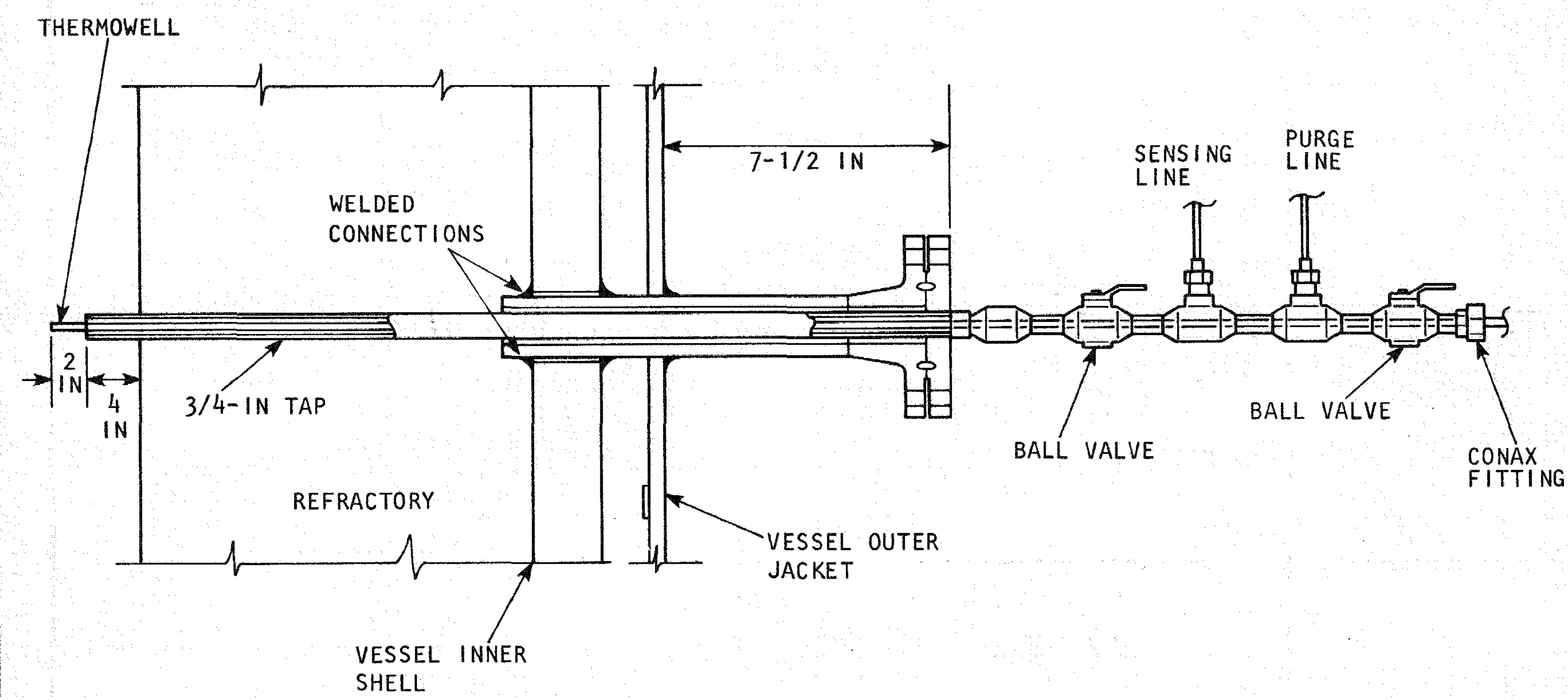


Figure 2-14. TYPICAL REGENERATOR SIDE TAP

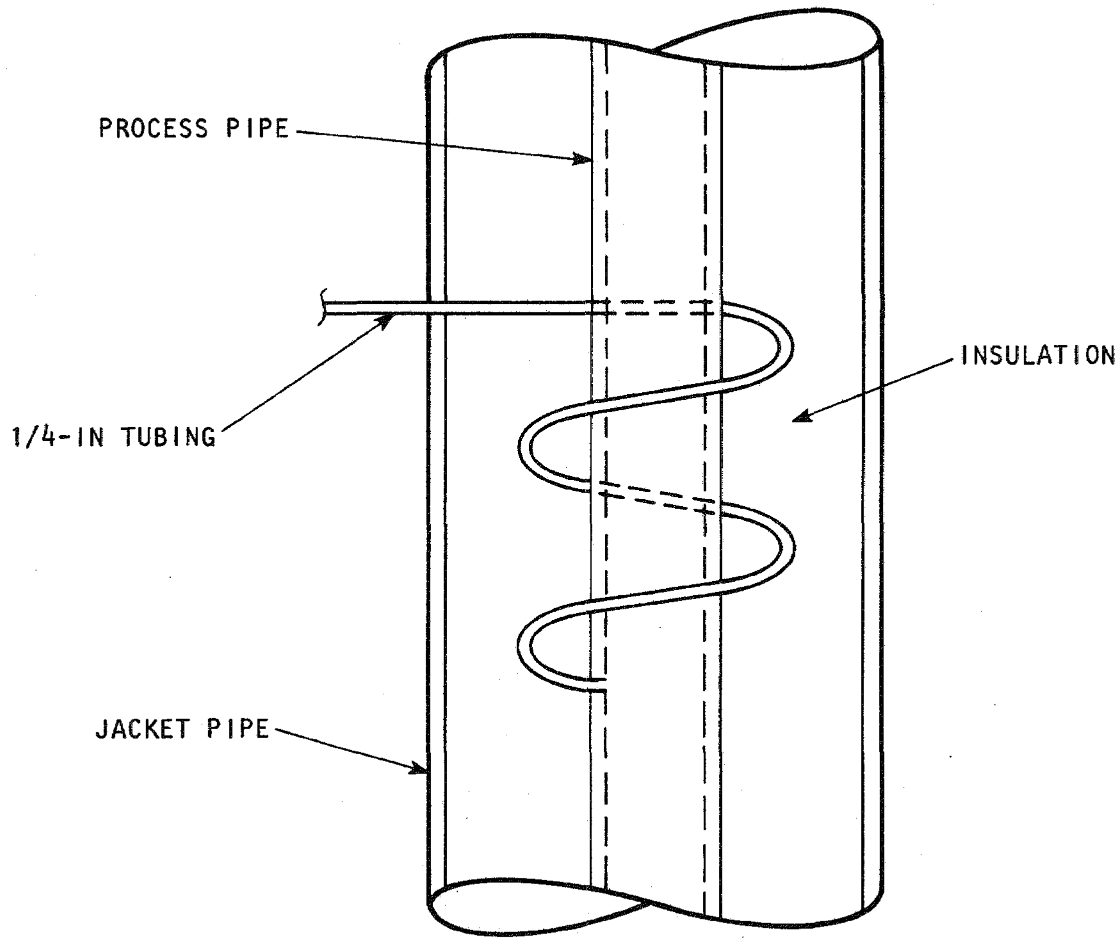


Figure 2-15. ORIGINAL COILED-PIPE TAP ON JACKETED LINES

To eliminate the problem of not being able to clear a plug from the spiral tap during a run, the pressure tap was redesigned. The pressure-sensing tube (see Figure 2-16) entered directly through the exterior jacket, through the insulation, and through an opening in the interior pipe. A section of flexible tubing in the sensing tube, at a distance from the high temperature of the internal pipe, allowed movement of the tube to accommodate differential expansion between the jacket and the inner pipe. External valving attached to the pressure tap assembly allowed a rod-out device to be attached in order to remove solids that could not be blown free.

2.6.2 FLOW MEASUREMENTS

2.6.2.1 Orifice-Type Flow Meters

Fourteen orifice-type meters are employed in various parts of the gasification system; their accuracy is essential for operating control and collection of valid data for heat and material balances. During 1975, these meters (and their runs) were removed and calibrated at Colorado Engineering Experiment Station, Incorporated at Nunn, Colorado. Calibration permitted mass flows to be measured within an error of ±1.0 percent.

2.6.2.2 Magnetic Flow Meters

In March, 1976, magnetic flow meters were installed at the following locations:

(1) Preheater venturi, L-114.
(2) Regenerator quench tower, E-302.
(3) Gasifier quench flash tank, F-324.

Service with these instruments was satisfactory on liquids containing from 1-3 percent solids.

2.6.2.3 Swirl Meters

A swirl meter was employed in the gasifier recycle gas loop. Its operation was unsatisfactory and it was replaced by an orifice.

2.6.3 GAS COMPOSITION MEASUREMENTS

2.6.3.1 Process Chromatographs

A Honeywell H-1000 chromatograph was employed on the regenerator and the gasifier offgases to determine H_2, N_2, O_2, CO, CO_2, and CH_4.

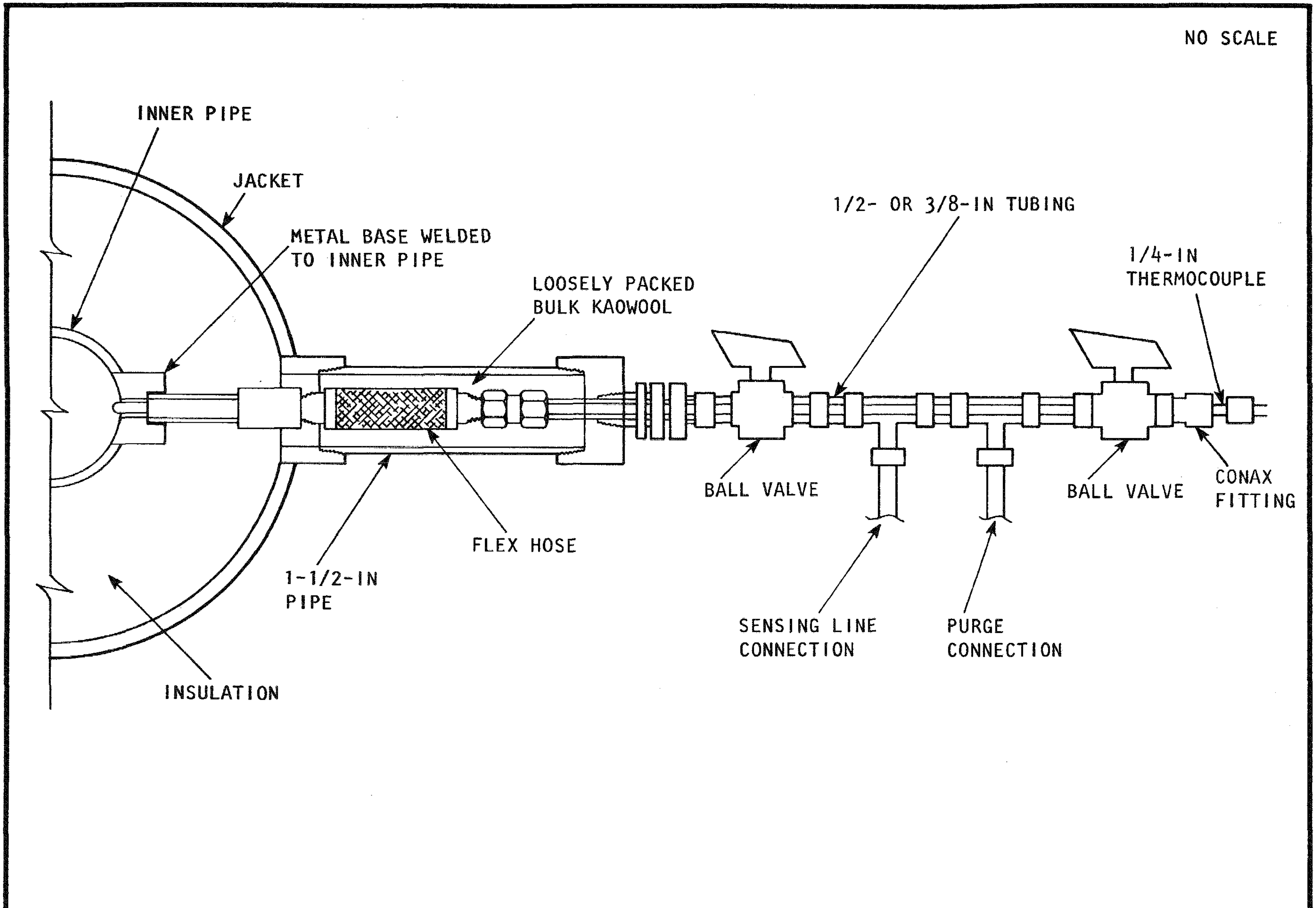


Figure 2-16. TYPICAL TEMPERATURE PRESSURE TAP USED ON JACKETED LINES

2.6.3.2 Analyzers

The following analyzers were employed for individual constituents:

(1) Oxygen: Beckman Models F-3 and G2X, and Bailey (in utilities area).
(2) Carbon monoxide: Mine Safety Appliance Lira M 200X.
(3) Carbon dioxide: Mine Safety Appliance Lira M 200X.

2.6.3.3 Safety Analyzers

Safety analyzers were used to monitor oxygen content, carbon monoxide content, and explosive gas content in various atmospheres. Instruments used in various areas were:

(1) Oxygen: Boiler room, coal preparation area-Beckman Model 741.
(2) Carbon monoxide: General plant areas-elevator shaft-Beckman Model 315B, Mine Safety Appliance 701.
(3) Explosive gases: General plant areas-Mine Safety Appliance I-501.

SECTION 3

PILOT PLANT PRACTICES

3.1 PLANT OPERATION

This section of the report presents a condensation of the operating procedures and the means of communicating them which were used in the operation of the CO_2 Acceptor Process Gasification Pilot Plant. More extensive details and examples of the procedures are appended in Book 4, Subsection 13.2 of this report.

3.1.1 START-UP PROCEDURE

Prior to the original plant startup, a proposed plant startup and operating procedure was drafted. This original draft has since been greatly modified and has evolved into a basic startup and operating procedure, with certain changes made for each run to satisfy individual run requirements.

The Exhibit in Book 4, Subsection 13.2.1.1, contains a typical set of the preliminary conditions and a step-by-step procedure as issued to operations personnel.

In general, the sequence of events on a run startup has remained the same from run to run, although varying in timing and type of feedstock. Prior to every run, a static pressure test is made with the system in a cold condition. This is accomplished by bringing the system up to pressure (nominally 150 psig), manually blocking all vents, and measuring the amount of inert gas required to maintain system pressure.

After a successful pressure test, gas circulation was started and the process heater pilots are lit. When the oxygen level in the system has been reduced to a satisfactory level (2%), the process heater main burners are lit. The oxygen level is important, in order to prevent the burning of any residual carbon left in the system from a previous run. The required time for heatup of the system is dictated by the heatup rate limits on the furnace tubes or by refractory cure requirements made necessary if repair work has been done to the refractory prior to the run. A typical set of heatup conditions for a refractory cure appears in the Exhibit, Book 4, Subsection 13.2.1.2.

Gas circulation through the process heaters and the system brings the temperatures in the vessels up to 1100^{o}F range. The first charge of dead-burned dolomite is then fed into the gasifier. Dead-burned dolomite has been previously fired to 3400^{o}F and is inactive. This is advantageous because this acceptor will not go through the calcination/recarbonation cycle while the system is being brought up to temperature. Some of the acceptor is transferred to the purged acceptor hopper and across to the regenerator via the lower acceptor standleg, engager pot, and lift line. This procedure is used to indicate whether or not the lines and valves are cleared for solids flow and to establish a seal in the acceptor standleg, CD-204. The acceptor bed is then established in the regenerator by feeding the dead-burned dolomite directly to the vessel from the acceptor make-up lockhopper system.

After the regenerator is filled with dead-burned dolomite, steam flow to the gasifier transition section is initiated. Char is then fed and an interface is established between the acceptor in the lower gasifier boot and the char bed above it. During the setting of this interface, the gasifier gas flows become critical. As the char bed is built up, the interface becomes more stable. During the filling of the gasifier with char, a low rate of dolomite circulation is maintained from the regenerator to the gasifier through the upper acceptor standleg and back to the regenerator again.

After the char bed is established in the gasifier, air injection is begun to burn some of the char and raise system temperature. A temperature of at least 800°F is required in the gasifier (as in the regenerator) to ensure combustion of the char when air is admitted to the vessel. As the temperature is increased, more steam is added to the gasifier; steam/air/recycle flows are continuously adjusted to maintain the fluid char bed, the interface, and fluidization of dead-burned dolomite in the boot.

Steam is always fed along with air to the gasifier. The steam prevents local hot spots caused by char combustion from developing. The steam will absorb some of the heat generated by burning char. Additionally, heat released by char combustion will promote the endothermic steam-carbon reaction:

$$C + H_2O \longrightarrow H_2 + CO \qquad +56{,}500/\text{Btu/lb mole of CO}$$

This reaction absorbs heat released by char burning. Both of these effects will limit the temperature rise in the char bed.

Steam flow to the gasifier char bed is intially set at 30,000 SCFH and introduced to the gasifier before air is injected. The air rate is usually about 20,000 SCFH. This steam-to-air ratio of 1.5 is the minimum ratio that is maintained whenever air is used in the gasifier.

When the char bed rises above the inlet to the fuel char standleg, CD-202, char transfer from the gasifier to the regenerator is initiated. Using regenerator air flow and char transfer control, the regenerator bed temperature of 1850°F is reached, with a desired CO concentration of 1 to 3 percent in the overhead gas. Auxiliary fuel feed is then started to the regenerator. The lignite coal feedstock to be used in the run is used for this auxiliary fuel. The purpose of the auxiliary fuel is to provide a backup source of heat in the regenerator and to provide a control in the event that the main char transfer becomes temporarily unstable.

During this time, char is being fed to the gasifier and acceptor circulation is increased. The gasifier is brought up to a temperature of 1500°F to 1550°F, depending on run target conditions.

When the gasifier temperature has reached the desired level, coal feed is started to the gasifier replacing the char feed. The minimum temperature at which raw lignite is fed to the gasifier in order to reduce the possiblity of tar formation is 1460°F.

A lineout period is required during the transition from char feed to raw lignite, because of the higher gas flow from the gasifier on lignite feed. After conditions are lined out, the active 6 x 8 mesh acceptor is fed at a rate of 600 lb/hr to displace the dead-burned dolomite. The reject acceptor withdrawal is set up on level control (regenerator level) in order to automatically withdraw inactive material as it is replaced by the active acceptor. About 3.5 days of fresh acceptor addition are required to change out 95 to 98 percent of the dead-burned dolomite (5 days were required prior to Run 42, when the regenerator was larger).

The air flow to the gasifier is gradually removed and replaced with steam as the active acceptor circulation and recarbonization reaction provides heat to the gasifier. The recycle flow is also removed from both vessels as conditions permit. The air/recycle removal procedure is shown in the Exhibit, Book 4, Subsection 13.2.1.3. The plant is considered in commercial mode when the gasifier flows are 100 percent steam and the regenerator flows are all air except for some inert gas and some recycle gas used as the char transfer lift gas.

3.1.2 PROGRESS REVIEW MEETINGS

After a run was in progress, the day-by-day operation was discussed in a daily process meeting. Attending this meeting were the Conoco Coal Development Company Project Manager, the Stearns-Roger Plant Manager, the Operations Superintendent, Process Engineering Superintendent, and Day Superintendent, and those engineers available while not monitoring the back shifts. During the meeting, a copy of the computer printout of current run conditions was distributed to each participant (Book 4, Figure 13-1). Notes of the meeting were made and run plans for the next 24 hours were typed up for distribution (Exhibit, Book 4, Subsection 13.2.1.4).

3.1.3 SPECIAL TESTS

Periodically, special tests were carried out during a run to obtain specific information on a selected subject. Written procedures were prepared and issued detailing each step required.

A typical example was the study of chemical additives to prevent solid deposits and plugging in the quench systems, which was carried out in cooperation with Betz Laboratories. See Book 3, Section 7 of this report.) The procedure of application of these chemicals is shown in the Exhibit, Book 4, Subsection 13.2.1.5.

Similar written instructions for the operation of the Elliott turbine erosion tests are shown in the Exhibit, Book 3, Subsection 13.2.1.6. Elliott test results are presented in Book 3, Section 10 of this report.

3.2 ANALYTICAL PROCEDURES

3.2.1 OPERATIONS

Analytical analyses for data collection and process control at the CO_2 Acceptor Process Gasification Pilot Plant were provided by a large on-site laboratory staffed with ten professional chemists. The laboratory was directed by the chief chemist who reported to the process superintendent. The chief chemist was assisted by an analytical chemist. Four shifts with two chemists per shift provided 24-hour-per-day, 7-day-per-week analytical support for the process and operations groups. Analytical results were collected daily and published.

3.2.2 GAS SAMPLE ANALYSIS

Gas samples were analyzed on a routine basis. Gasifier overhead and zinc oxide tower effluent were analyzed six times per day. The regenerator overhead was analyzed three times per day and the SO_2 scrubber effluent once per day. Other special gas samples were done on request. The gasifier overhead, regenerator overhead, and SO_2 scrubber were analyzed for hydrogen, nitrogen, oxygen, carbon monoxide, carbon dioxide, methane, hydrogen sulfide, carbonyl sulfide, sulfur dioxide, and carbon disulfide. The effluent from the zinc oxide towers was analyzed for the latter four compounds.

All gases except for the sulfur species were determined with a Varian Model 90P gas chromatograph coupled to a Hewlett-Packard Model 3380A integrator. A molecular sieve, 13A, or a silica gel column was used for gas separation. These chromatographs were calibrated with standard gas mixtures obtained from Air Products and Chemicals, Inc. and from the Linde Division of Union Carbide Corporation.

The sulfur gas species were determined using a Varian Model 1400 gas chromatograph equipped with a flame photometric detector specific for sulfur. This instrument was coupled to the Hewlett Packard 3380A integrator. The column was Deactigel. The chromatograph was calibrated with a permeation tube dilution instrument and a dilution flask.

3.2.3 PROCESS SOLIDS ANALYSIS

Samples of process solids were obtained on a routine basis. These samples included ground lignite to the preheater, preheated lignite to the gasifier, calcined acceptor from the regenerator, recarbonated acceptor from the gasifier, and purged acceptor. A solids sample containing char and acceptor was obtained from the gasifier bed. The regenerator cyclone, gasifier cyclone, and gasifier quench tower solids were sampled routinely.

Ultimate analyses on coal samples were performed according to ASTM Method D271-68, Laboratory Sampling and Analysis of Coal and Coke, for moisture, carbon, hydrogen, nitrogen, and calorific value. Sulfur was determined by the bomb washing method and a turbidimetric sulfate test on the washings. The coal ash was brought into solution using hydrochloric and nitric acid digestion. The undissolved silica was filtered from the digested solution and dried and weighed. Aluminum, iron, titanium, and phosphorous analyses were done on this solution using the procedure in ASTM Method D2795-69, Analysis of Coal and Coke Ash. Sodium, calcium, magnesium, and potassium analyses of this solution were determined with a Perkin Elmer Model 290 atomic absorption spectrophotometer. Sulfur in the ash sample was determined by a turbidimetric sulfate test. The particle density of coal and char was measured by mercury displacement in a porosimeter. Sieve analyses of coal samples were performed using ASTM-approved sieves and a mechanical rotap.

Acceptor samples, either limestone or dolomite, were analyzed by the same methods as the coal ash. Particle density and sieve analysis were also obtained on these samples.

On the recarbonated acceptor sample, an acceptor activity is defined as the ratio of the capacity of the sample to recarbonate, relative to the amount of carbon dioxide lost upon initial calcination. To determine activity, the sample was calcined while being swept with nitrogen in a steel tube heated by a fluidized sand bath furnace at 1600°F. The weight loss of carbon dioxide while calcining divided by the calcined sample weight times the ratio of CO_2 to CaO for the raw limestone is equal to the acceptor activity of the particular sample:

$$R = \text{Activity} = \frac{\text{Weight loss of } CO_2}{\text{Calcined sample Wt. X } F_2}$$

$$\text{Where } F_2 = \frac{\text{Wt. of } CO_2 \text{ equivalent to the useful } CaCO_3 \text{ in raw stone}}{\text{Calcined Wt. of raw stone}}$$

The procedure for determining F_2 is described in Section 5.7, Book 2.

A sample containing both char and acceptor was withdrawn from the gasifier bed every 4 hours to determine percent acceptor, percent ash in the char, and the amount of "junk" in the sample. To accomplish this, a portion of the sample was ashed at 1600°F until constant weight was achieved. The ash was then screened into +80 mesh and -80 mesh fractions. The +80 mesh fraction divided by the weight of the sample gave the percent acceptor plus "junk" in the gasifier bed sample. The difference between the +80 mesh fraction and the sample weight is the char fraction. The weight of the -80 mesh fraction divided by the weight of the char fraction gives the percent ash in the char fraction of the gasifier bed sample. The -20 mesh fraction of the +80 mesh acceptor fraction is the amount of "junk" in the gasifier bed.

The above analytical procedures were followed by the pilot plant laboratory. The results of these analyses are presented in the individual run reports presented in Volume 8.(1) After each pilot plant run for which significant stable operating periods were obtained, sets of solids samples were forwarded to the Conoco Coal Development Company (CCDC), Library, PA laboratory for analyses. Detailed work-ups were then obtained. Although the analytical methods between the pilot plant and the Library labs varied somewhat, the results were generally in agreement. Because more thorough analysis was generally obtained for the samples sent to the CCDC lab, particularly with respect to analyzing individual size fractions of a given sample, only the CCDC lab results were used for the detailed heat and material balances presented in Section 5 of this report.

Table 3-1 lists the analytical techniques used by the CCDC laboratory for heating value, ultimate and elemental analyses. Interested readers may contact the CCDC, Library, PA laboratory for procedural details of any analysis which is not considered to be proprietary. For the details concerning the determination of the recarbonated acceptor activity, acceptor particle density, and the gasifier char bed composition, refer to Book 2, Subsections 5.7.1.1, 5.7.1.3, and 5.7.2.3.

3.2.4 WATER SAMPLE ANALYSIS

Water samples from the gasifier quench tower, foul water stripper, flue gas quench tower, the inlet to the organic pond, the inlet to the settling pond, and the preheater venturi scrubber were filtered and the percent undissolved solids determined. Water samples for control of chemical additives to the boiler, cooling water, quench water systems, and jacket water systems were analyzed according to Betz Chemical Company procedures for water analysis. Laboratory assistance and space were given to Betz Chemical Company research engineers while tests were conducted for the gasifier and regenerator quench venturi deposit study. Samples from the SO_2 scrubber and CO_2 absorber were routinely analyzed to determine the percent of potassium carbonate in the systems.

Work space and assistance from laboratory personnel were given to Carnegie-Mellon University and Radian Corporation during their research on solid, liquid, and gas samples.

Laboratory personnel obtained effluent samples that were studied by the South Dakota School of Mines and Technology for trace element analysis.

---

(1) Run Reports-CO_2 Acceptor Process Gasification Pilot Plant, DOE Final Report No. FE-1734-41: Volume 8, Books 1-6.

Analysis	Procedure	Determination Method
Ultimate:		
C, H	Macrocombustion[2]	Gravimetric
N	CCDC Method - 186 (Kjeldahl)	Specific Ion Electrode
S (Total)	ASTM D3177-75 (Method A)	Gravimetric
Ash	ASTM D1374-73	Gravimetric
Heating Value:	ASTM D7015-66	Bomb Calorimeter
Elemental:		
Na_2O	CCDC Method - 117	Atomic Absorption
K_2O	" " "	" "
CaO	" " "	" "
MgO	" " "	" "
Mn_3O_4	" " "	" "
SrO	" " "	" "
Fe_2O_3	CCDC Method - 39	Colorimetric
TiO_2	" " "	"
P_2O_5	CCDC Method - 38	"
SiO_2	CCDC Method - 42	"
Al_2O_3	CCDC Method - 43	"
SO_3	Modified British Method	Gravimetric

Table 3-1. ANALYTICAL METHODS USED BY THE CCDC LABORATORY

---

(2) T. T. White, et al., Analytical Chemistry, Vol. 30, No. 3, 409-414.

3.3 SAMPLING PROCEDURES

The nature of pilot plant operation necessitates numerous samples to be taken during a run for data acquisition. Many more samples are taken than would be necessary in a commercial plant. Each process stream is sampled on a regular scheduled basis. In addition, many process samples are taken on a nonscheduled basis dictated by the type of operation--such as an ash lockhopper dump. Other samples are taken routinely in support of operations as in any chemical plant. These samples include such materials as boiler water, cooling water, and plant effluents.

Because of the difficulty of obtaining representative samples under frequently adverse conditions, elaborate sampling procedures were developed. A description of these procedures and the usual sampling frequency is appended in Book 4, Subsection 13.2.2.

3.4 DATA ACQUISITION PROCEDURES

The "Data Logger" at the Rapid City coal gasification facility is a PDP 11/34 minicomputer and associated peripherals. The raw data are collected through several devices, changed to a form the computer can accept, and sent to the computer for processing. After the data have been processed, the information is available for display or recording if the situation warrants. Once information has been permanently recorded on a magnetic disk, it can be selectively removed at a later date for display or additional processing.

With the exception of the Scani-Valve, all hardware was purchased from and serviced by the Digital Equipment Corporation. The Scani-Valve set-up came from Scani-Valve, Inc., San Diego, California. The computer software was written by and purchased from Biles & Association of Houston, Texas.

A detailed description of the PDP 11/34 and associated peripherals is presented in Book 4, Subsection 13.2.3.1 of this report.

3.5 SAFETY PROCEDURES

The Rapid City Gasification Pilot Plant is an experimental facility to test the feasibility of the CO_2 Acceptor Process. As a test facility, this plant presents a multitude of new safety considerations unique to coal gasification facilities. The facility incorporates many of the special problems found in related industries (such as coal handling, petrochemical, and natural gas transmission) into a single plant; because of this, special safety problems were created for which adequate consideration had to be given. The problems involving safety considerations and the procedures developed to insure adequate safeguard of all plant personnel are presented in Book 4, Subsection 13.2.4 of this report.

The overall safety consideration of the CO_2 acceptor plant enabled it to earn the corporate Stearns-Roger safety award for seven straight years. As many as 500,000 man-hours of work were completed without a lost-time injury. The lost-time injuries which occurred were not of a very serious nature and generally resulted in only 3 to 4 days away from work.

SECTION 4

MECHANICAL PERFORMANCE

The purpose of this section is to review the history of mechanical integrity of critical items of equipment in the CO_2 Acceptor Gasification Pilot Plant.

The combination of operating conditions (pressure, temperature, and erosive and corrosive process streams) encountered in the system is extremely severe and has not been employed previously on a major scale. Accordingly, it is essential that the materials required for satisfactory commercial mechanical performance be evident from the pilot plant performance.

4.1 REFRACTORIES

When examining the life of vessel refractories, it should be recognized that the pilot plant was subjected to many temperature and pressure cycles during the 5-1/2 years of operation. An installation which has demonstrated a reasonable mechanical integrity in the pilot plant would, in all probability, have an extended life in a commercial application.

4.1.1 GASIFIER

The original gasifier vessel refractory lining is shown in Figure 4-1. The lining in the boot section of the vessel consisted of 17 inches of Johns-Manville Superex block insulation next to the vessel shell, 3 inches of A. P. Green VSL-50 insulating castable as an intermediate layer, and 3 inches of A. P. Green KS4V hard-face castable as an inner layer. The lining in the upper (40-inch ID) section of the vessel consisted of 7 inches of Johns-Manville Superex block insulation, 3 inches of A. P. Green VSL-50, and 3 inches of A. P. Green KS4V hard-face inner layer.

After Run 4 in 1972, the gasifier bottom head was removed to investigate the vessel internals in an attempt to explain the unusual flow behavior which occurred during the run. There were wide cracks in the bottom one-third of the boot and in the bottom head. These cracks were through both layers of hard refractory down to the Superex block insulation. Gas had been flowing into the cracks behind the hard inner layers. The refractory had clearly failed after less than 900 hours of hot service.

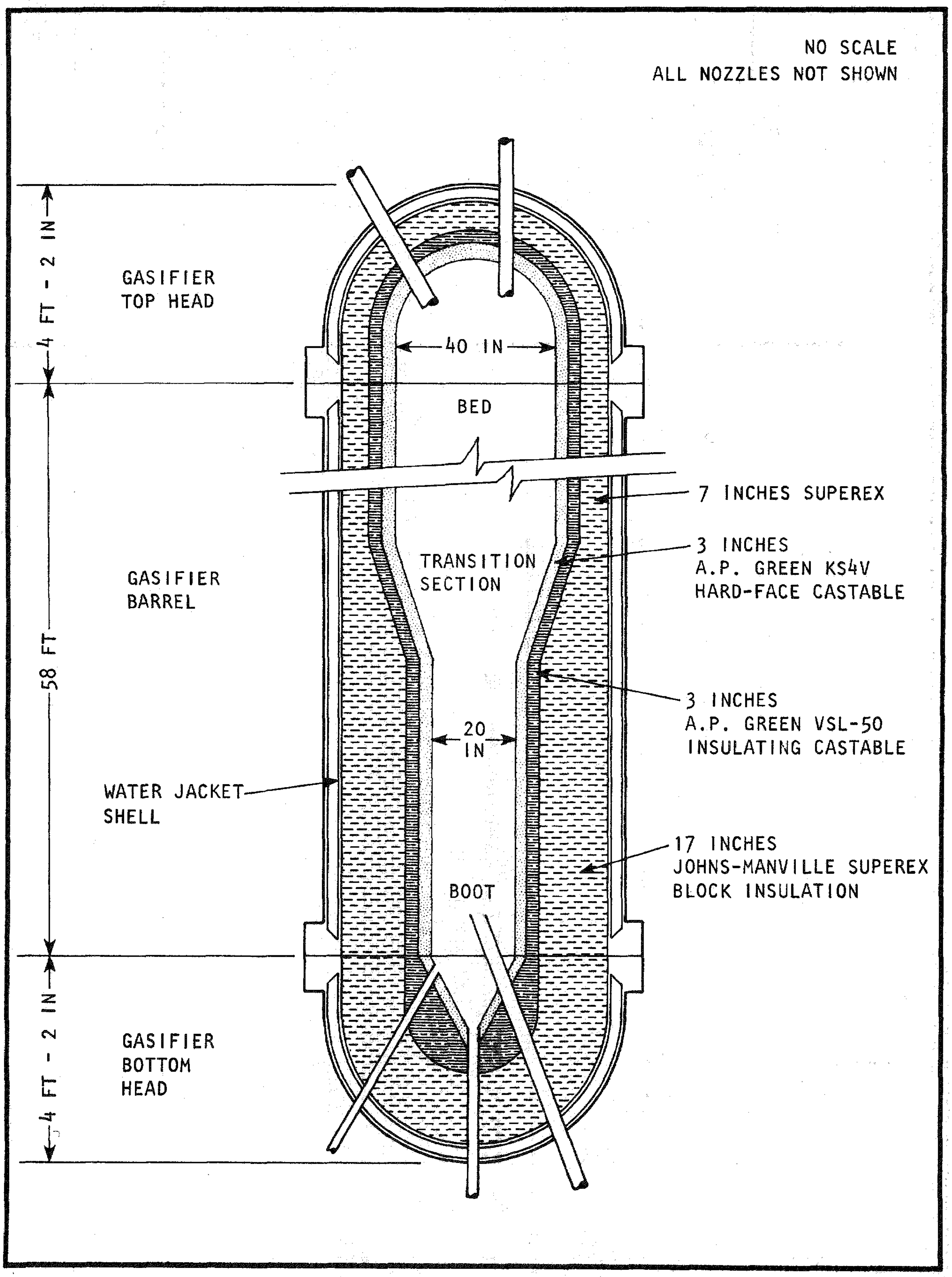


Figure 4-1. ORIGINAL GASIFIER REFRACTORY

Refractory and insulation were removed from the bottom head, boot, and transition sections of the vessel. Channeling in the Superex block insulation extended above the transition section. Upon removal of the top head, some channeling was observed in the Superex at the top of the vessel and entering the Superex in the top head. The bypassing gases reentered the main vessel volume around the outlet pipes coming through the refractory at the top.

It was concluded that pressure surges may have exerted forces on the refractory wall, causing it to crack and compress the Superex block insulation. Further, differential thermal expansion between the relatively cold vessel wall and the hot refractory may have contributed to the compression of the relatively soft Superex. The refractory internal diameter had increased, and the metal anchors holding the inner layers of refractory were bowed from compression.

A rugged castable refractory was substituted for the Superex in the new installation in the boot section, transition section, and bottom head. The materials used do not compress and have crushing strengths sufficient to withstand a force equivalent to a pressure differential of 150 psi.

The refractory installed in the boot and the bottom head was 18 inches of A. P. Green VSL-50 insulating castable next to the shell and 6 inches of A. P. Green Lo-Abrade hard-faced castable as an inner liner. In the transition section, the VSL-50 next to the shell was 18 inches thick at the bottom and tapered to 7 inches thick at the top of the transition. The Lo-Abrade inner liner was applied 6 inches thick throughout the transition section. The new installation is shown in Figure 4-2.

Refractory vapor stops were installed in the upper section (40-inch-ID section) of the vessel to eliminate any future gas bypassing in the Superex block insulation remaining in this section. The vapor stops were made using a pneumatically applied Lo-Abrade. They were installed at the top of the transition section, at elevation 18 feet-4 inches, at elevation 28 feet-4 inches, at elevation 36 feet-4 inches, and at the vessel top flange.

In June, 1976, just prior to Run 38A, extensive repair work was required on the gasifier top outlet nozzle. This work necessitated the removal of the top head refractory. The refractory was replaced with a single pour of Kaiser Purotab with the same shape and dimensions as the original combination of the three refractories. (See Figure 4-3.)

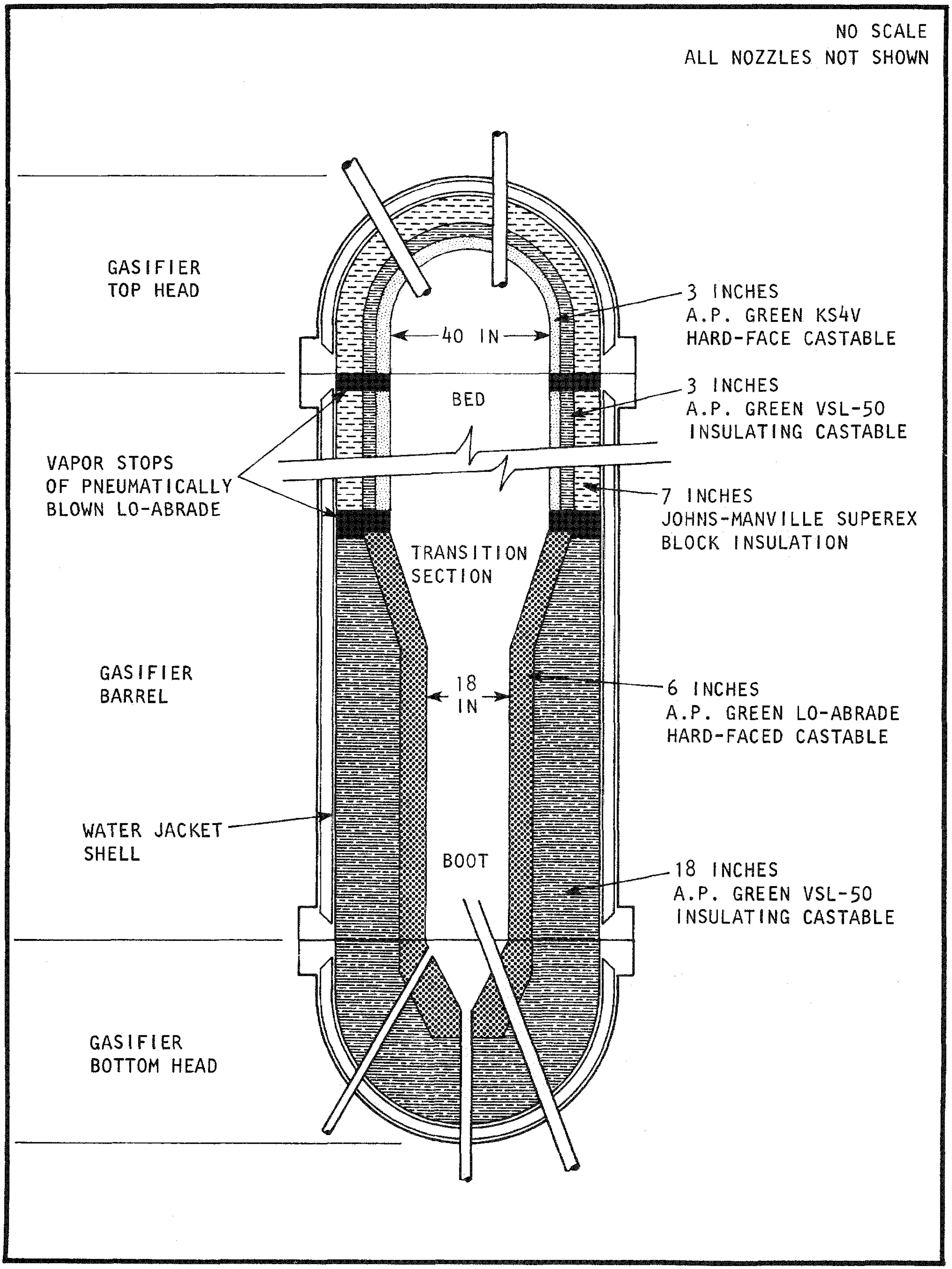


Figure 4-2. GASIFIER REFRACTORY AFTER 1972 REPAIR

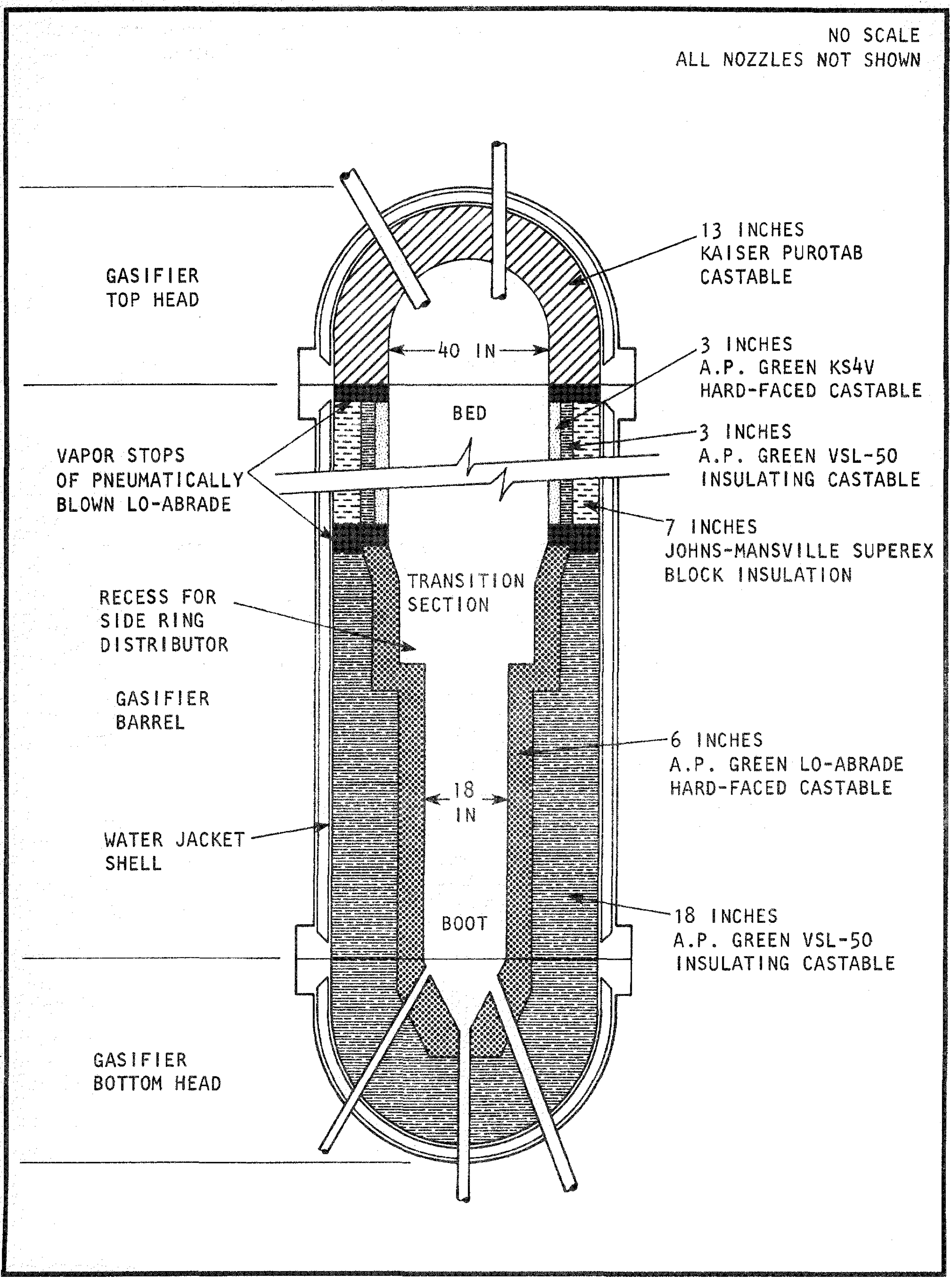


Figure 4-3. GASIFIER REFRACTORY AT END OF OPERATION, 1977, SHOWING 1976 TOP HEAD REPLACEMENT

During the time after the aforementioned series of events, the gasifier refractory was inspected many times between runs. Several patches, some large and some small, were applied to the internal portion of the gasifier. The material used for these patches was Resco AA-22, due to its ease of application, simple cure, and long life. There were also minor refractory configuration changes made in the bottom head and around the boot and side distributors. The materials used to modify the bottom boot gas distribution and solids withdrawal were Resco AA-22 and Kaiser Purotab. These changes were not due to failure of the vessel refractory but rather to physical modifications to the vessel.

In general, there were no major gasifier refractory changes from 1972 to plant shutdown in 1977. Thus, a reliable basis for the design of commercial gasifier refractories has been demonstrated.

4.1.2 REGENERATOR

The regenerator vessel refractory was replaced completely twice after the original installation. There were also major and minor repairs and patching done. Additionally, some modifications were made to facilitate repairs and design modifications of nozzles and flow distributors. The last major change was the installation of a brick lining on the inside of the existing refractory to decrease the internal diameter of the regenerator. This was done during the latter term of plant operation to study the use of 100-percent air and a lower total flow, while still maintaining the regenerator fluidizing-gas velocity.

The original refractory lining consisted of 7 inches of Johns-Manville Superex block insulation next to the shell, 3 inches of A. P. Green VSL-50 insulating castable intermediate layer, and 3 inches of A. P. Green KS4V hard castable as an inner layer. Figure 4-4 shows the original refractory installation.

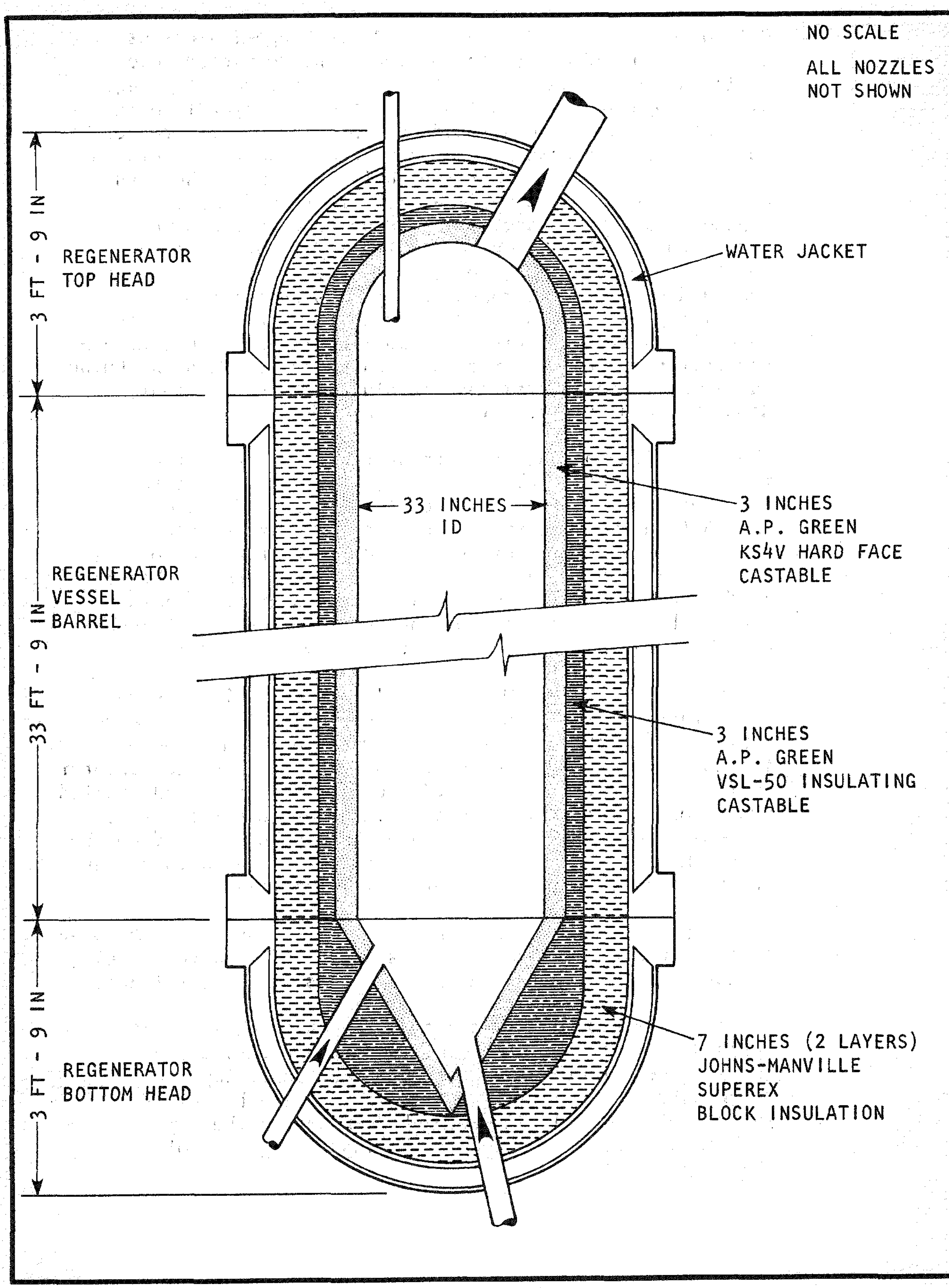


Figure 4-4. ORIGINAL REGENERATOR REFRACTORY

Following Run 5, in November, 1972, the regenerator was opened for a refractory inspection. The refractory had failed at the 4-foot-6-inch level above the bottom flange directly opposite and in line with the air inlet line in the bottom head where air and makeup gas enter the vessel. The hot air, hot makeup gas, char, and dolomite had eroded a hole through the refractory and insulation to the vessel wall. The hole was approximately 1 foot in diameter; it angled toward the vessel wall and up through the Superex for approximately 1 foot behind the refractory. At the point where the hole stopped, the hot air, makeup gas, char and dolomite had channeled up through the Superex, depositing char and dolomite in the eroded channels. At some time during the previous run (Run 5) prior to shutdown, the char had burned in the hole and channel. This had burned out some of the Superex and, in places, fused the char, dolomite, Superex and refractory together.

The old refractory and insulating block were completely removed from both heads and the vessel barrel. The replacement refractory consisted of 10 inches of Resco RS-3-35AA high-strength, low-density insulating castable next to the shell in both heads and the main barrel of the vessel. The inner layer of the bottom head and the bottom 10 feet of the vessel barrel was 3 inches of Resco AA-22. The top head and the rest of the vessel barrel had 3 inches of Resco RS-17A as an inner liner. (See Figure 4-5.)

In the latter part of May and early June of 1974, pieces of refractory were discovered in the solids transfer valves during Run 19 startup. The regenerator head was removed for inspection of the vessel refractory. The inside diameter surface had cracks and spalled areas. The cracks and spalled areas were cleaned and patched. One area (about 18 by 36 inches) around the fuel char inlet had to be replaced to the vessel metal wall. New anchors, insulating castable refractory, and Resco AA-22 face refractory were installed.

The refractory was patched between each run until mid-October, 1974, when new refractory was installed in the main barrel of the vessel. The top and bottom heads were not relined.

The new refractory consisted of a 9-inch-thick layer of Resco RS-3-35AA high-strength, low-density insulating castable next to the shell and a 4-inch inner layer of Resco AA-22. Around the nozzles in the main barrel of the vessel, Resco AA-22 was packed in around the pipe back to the shell in place of the insulating castable. (See Figure 4-6.)

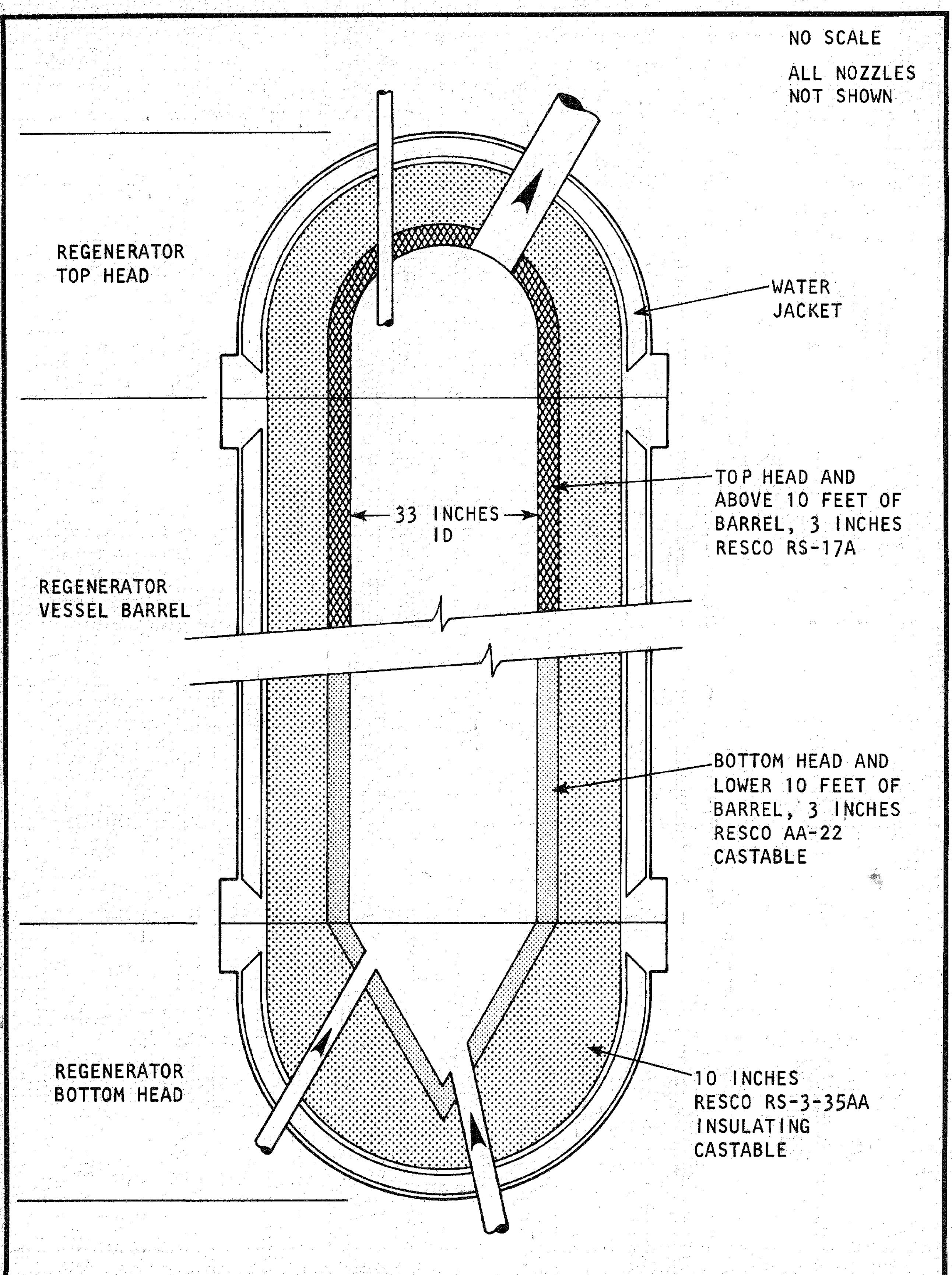


Figure 4-5. REGENERATOR REFRACTORY AS INSTALLED AFTER RUN 5

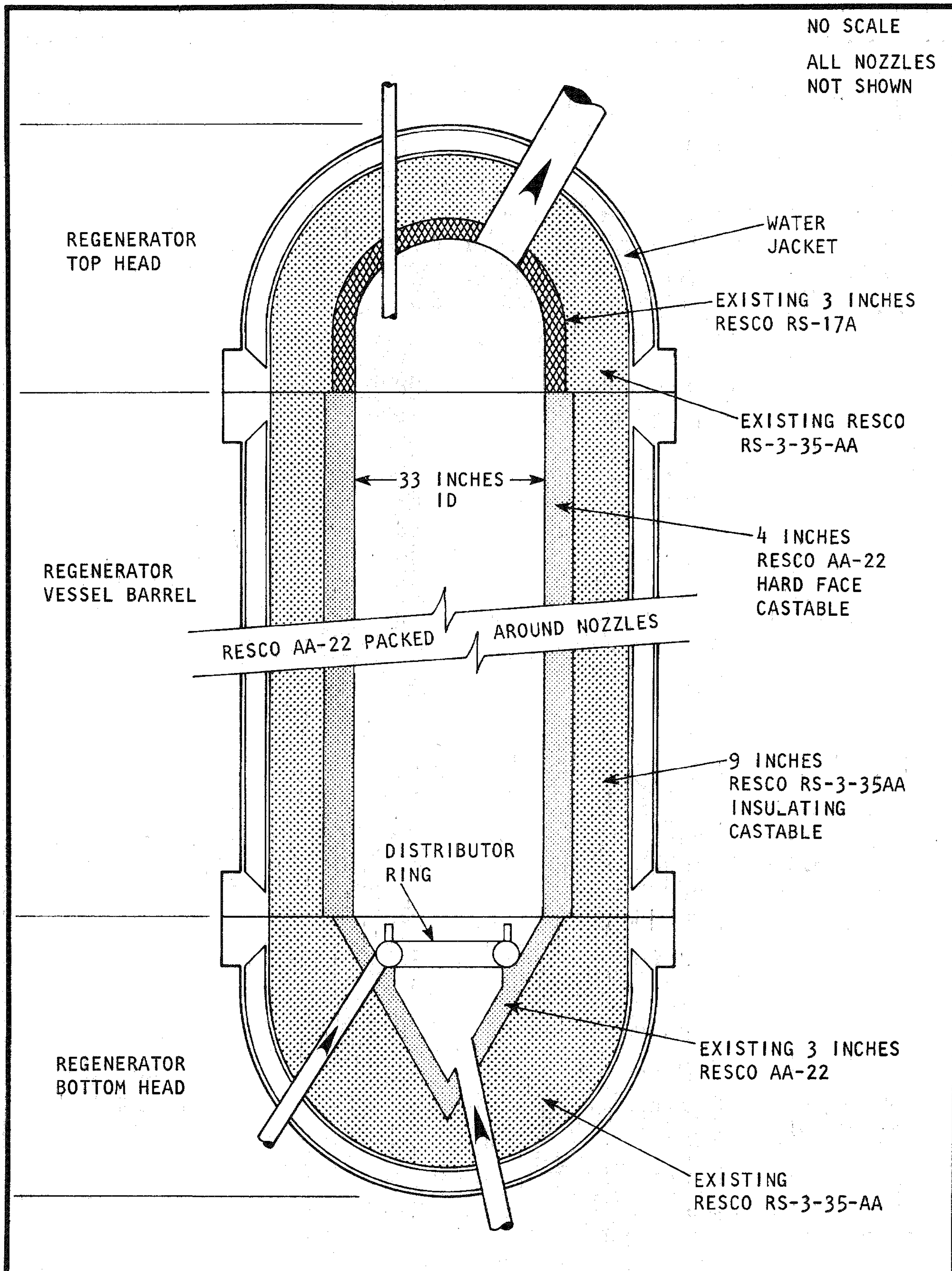


Figure 4-6. REGENERATOR REFRACTORY AS INSTALLED AFTER RUN 22A IN MID-OCTOBER 1974

As the project progressed and modifications were made to the ring distributor, the refractory in the bottom head was also modified. These refractory changes and additions did not basically alter the vessel refractory but filled voids or trimmed the existing refractory to make room for distributor design changes. (See Figure 4-7.)

The regenerator refractory remained as described with only minor repairs until after Run 41B. In January, 1977, a layer of refractory brick was installed inside the existing refractory to decrease the internal diameter from 33 inches to 24 inches, the brick layer being 4-1/2 inches thick.

The brick used was C-E Refractories' Chemel 85 High Alumina Arch Brick. The refractory mortar was C-E Refractories Chemel 111. The existing refractory surface was sand-blasted and the mortar used to fill cracks and voids of up to 1/2 inch. For larger voids, Kaiser Purotab was used. Figure 4-8 shows the brick in the main barrel of the vessel, with the existing refractory exposed on the top and bottom heads.

Eleven runs using brick lining were completed with good results. A few loose bricks were re-cemented and the bottom two rows slumped a small amount several times. The slumping bricks were mortared and pushed back in place. Otherwise, the brick lining proved successful in the abrasive environment of the regenerator.

Satisfactory performance was achieved in the regenerator in operations after Run 22 using either castable refractory or high alumina brick. Thus, a reliable basis for refractory design for a commercial regenerator has been demonstrated.

The various types of refractory and insulation materials used in the regenerator are shown in Table 4-1 with their thermal conductivity ratings.

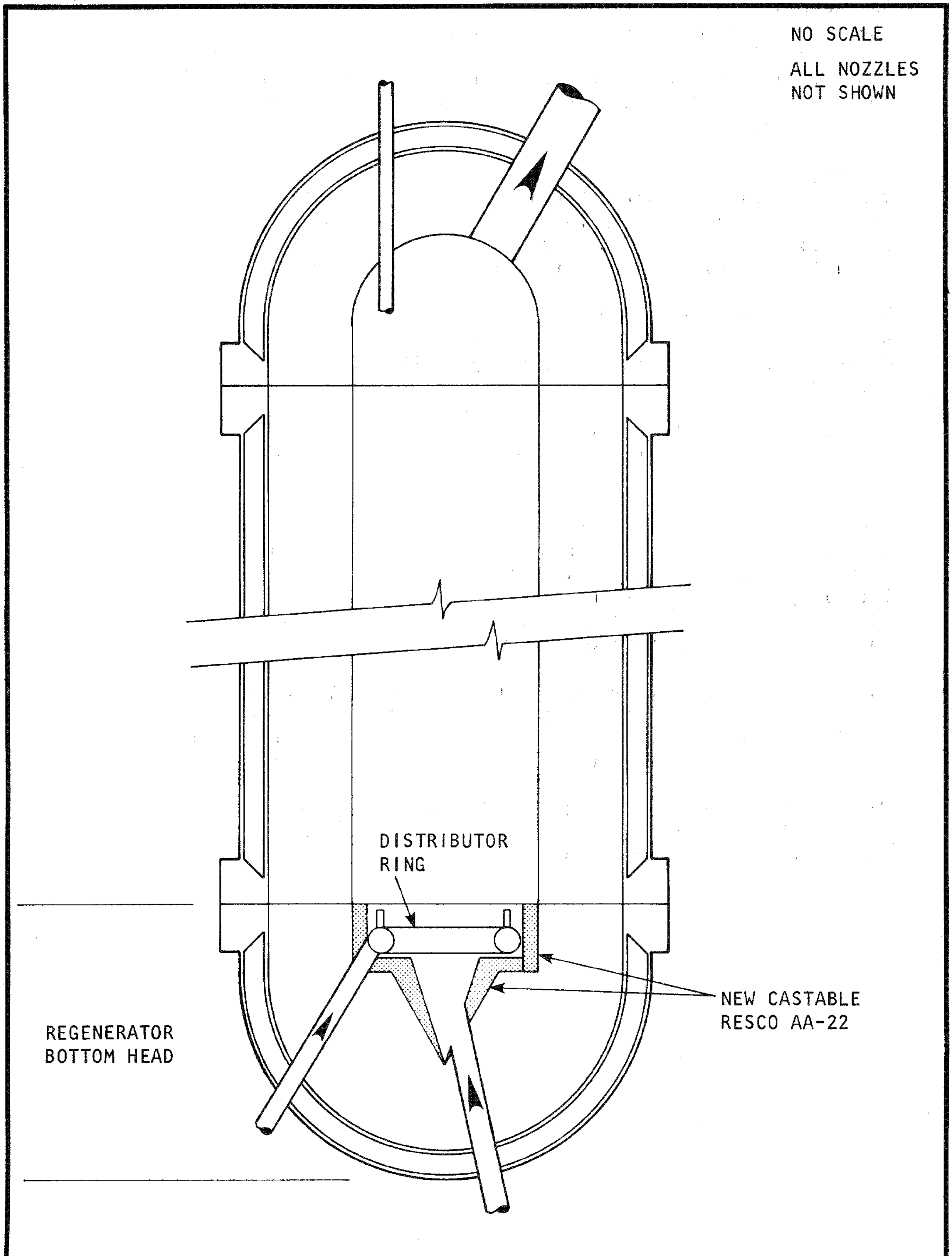


Figure 4-7. EXAMPLE OF REGENERATOR REFRACTORY MODIFICATIONS IN BOTTOM CONE AROUND RING DISTRIBUTOR

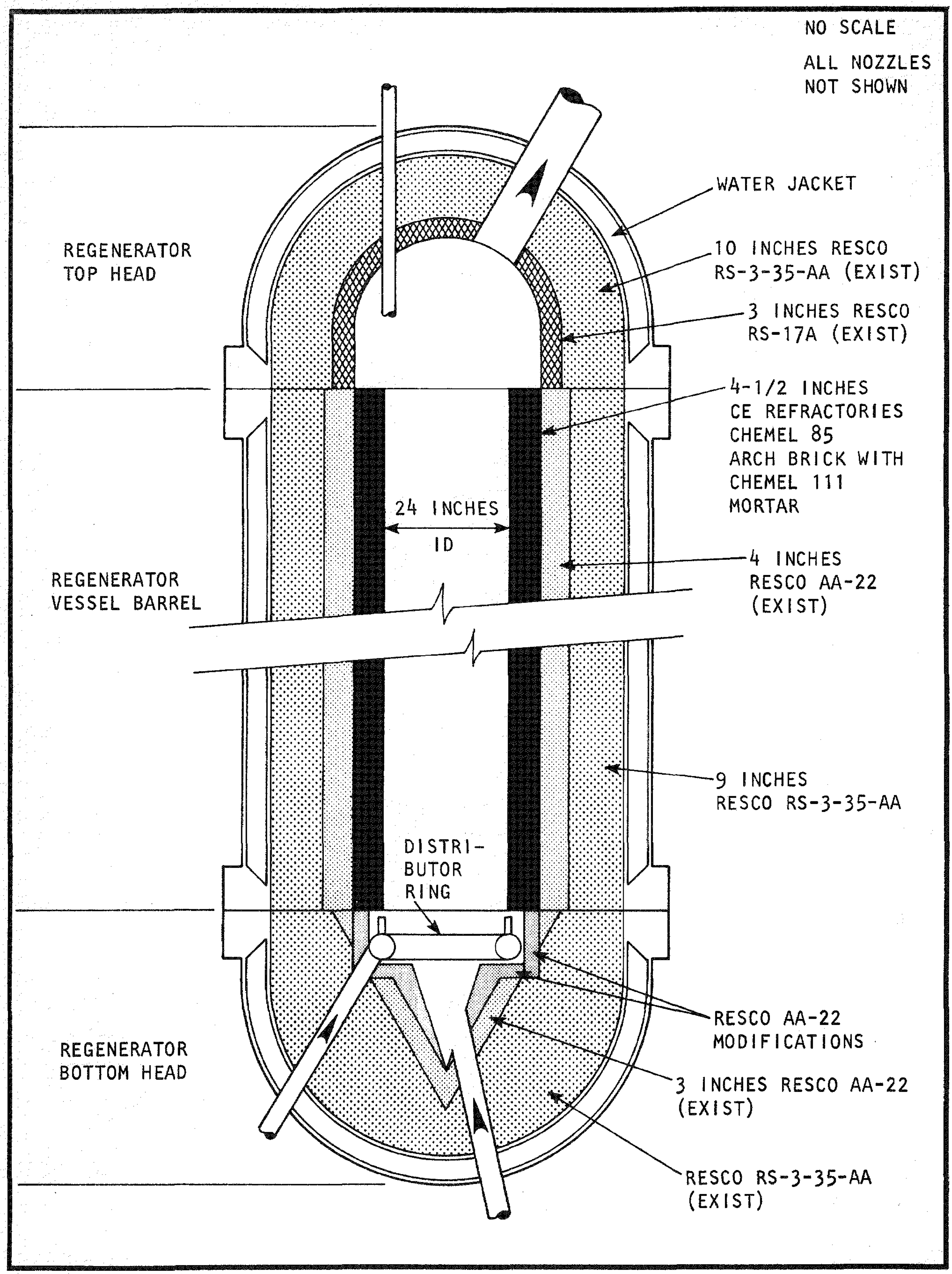


Figure 4-8. REGENERATOR REFRACTORY BRICK AS INSTALLED AFTER RUN 41B IN JANUARY 1977

Material	Thermal Conductivity, K, At Mean Temp (Btu-in/sq-ft/hr/$^{\circ}$F)	Description
Johns-Manville Superex	0.82 at 1000°F	Block insulation composed of diatomaceous silica blended with other insulating materials and bonded with a fiber.
A. P. Green VSL-50	2.09 at 1000°F 2.11 at 1500°F	Insulating castable.
A. P. Green KS4V	9.75 at 1000°F 9.9 at 1500°F 10.1 at 2000°F	Hard-face castable.
A. P. Green Lo-Abrade	4.16 at 1000°F 4.97 at 2000°F	Hard-face castable.
Resco AA-22	9.30 at 500°F 9.15 at 1000°F 9.10 at 1500°F	Air-setting, phosphate bonded, erosion-resistant, castable refractory.
Resco RS-3-35AA	1.75 at 1000°F 1.80 at 1500°F	Combination refractory seal and insulating barrier composed of the oxides of Al, Si, and Ca.
Resco RS-17A	4.31 at 1000°F	High-alumina, low-iron castable for high-temperature operation. Resistant to sulfur reducing atmospheres, carbon deposition, and hydrogen atmospheres.
Kaiser Purotab	9.57 at 2000°F	High-purity tabular alumina castable with high-temperature limits.
C-E Refractories Chemel 85	--	Phosphate-bonded, high-alumina brick with volume stability, strength, and resistance to abrasion, impact, and thermal shock.
C-E Refractories Chemel 111	--	85% alumina, phosphate-bonded mortar used with Chemel 85 brick.

Table 4-1. REFRACTORIES AND INSULATIONS USED

4.2 HOT LINES

It is inherent in the design of the CO_2 Acceptor Process that the lines transferring solids and gases between vessels must absorb major differential expansion as the system is brought from ambient conditions to full process temperatures and pressures. A major development has been the evolution of a line design which could reliabily withstand the adverse conditions through the many temperature and pressure cycles associated with pilot plant operation.

The original design intended for the containment of moving solids at high temperatures used a relatively thin alloy inner pipe. (See Figure 4-9.) The outer pipe was to contain the pressure. The annular space between the inner and outer pipe contained an insulating material, and a gas purge into this space prevented solids and process gas from leaking out through the inner pipe slip joint. An external, relatively cold expansion joint provided for expansion and contraction of the outer pipe.

Modifications made to this original construction include the redesign of the line end plates, change in material of construction of the external expansion joints, change in design and material of construction of the inner slip joints, replacement of the inner slip joints with a refractory lift line, and standleg design and materials of construction changes.

4.2.1 END PLATES

The original end plates were welded to the ends of the alloy inner liner and the carbon steel outer pipe of the hot line, as shown in Figure 4-9. In early operation, the weld to the inner pipe failed frequently. At first observation, it was thought that the inner slip joint had bound and caused the expansion or contraction of the inner pipe longitudinally through the end plate, causing the weld to fail. Upon inspection, the inner slip joints were found to be bound up and full of solids. After consultation with experts in the field of hot line construction, it was decided that expansion breaking the plate and weld was circumferential rather than longitudinal. The purge and process gases then flowed through the break, in the wrong direction, allowing solids to fill the slip joint and cause binding.

A new end support was designed in the form of a cone. The cone design was installed as shown in Figure 4-10. The cone support takes up the stresses by flexing.

The cone is filled with hand-packed insulation, and a flat plate is tack-welded on the end to retain the insulation only.

These cones have functioned satisfactorily through operation since installation in mid-1972.

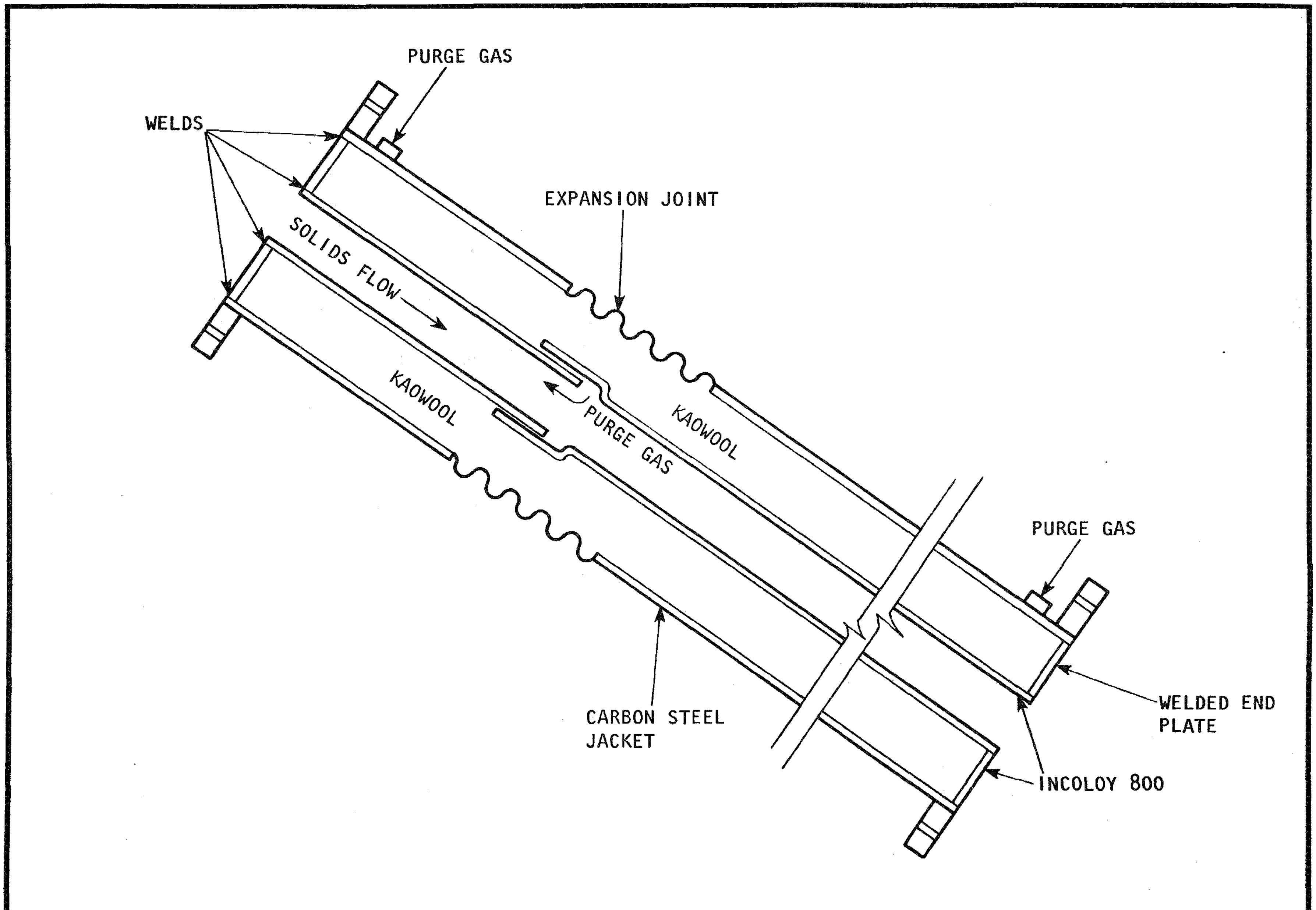


Figure 4-9. TYPICAL HOT PIPE ORIGINAL DESIGN

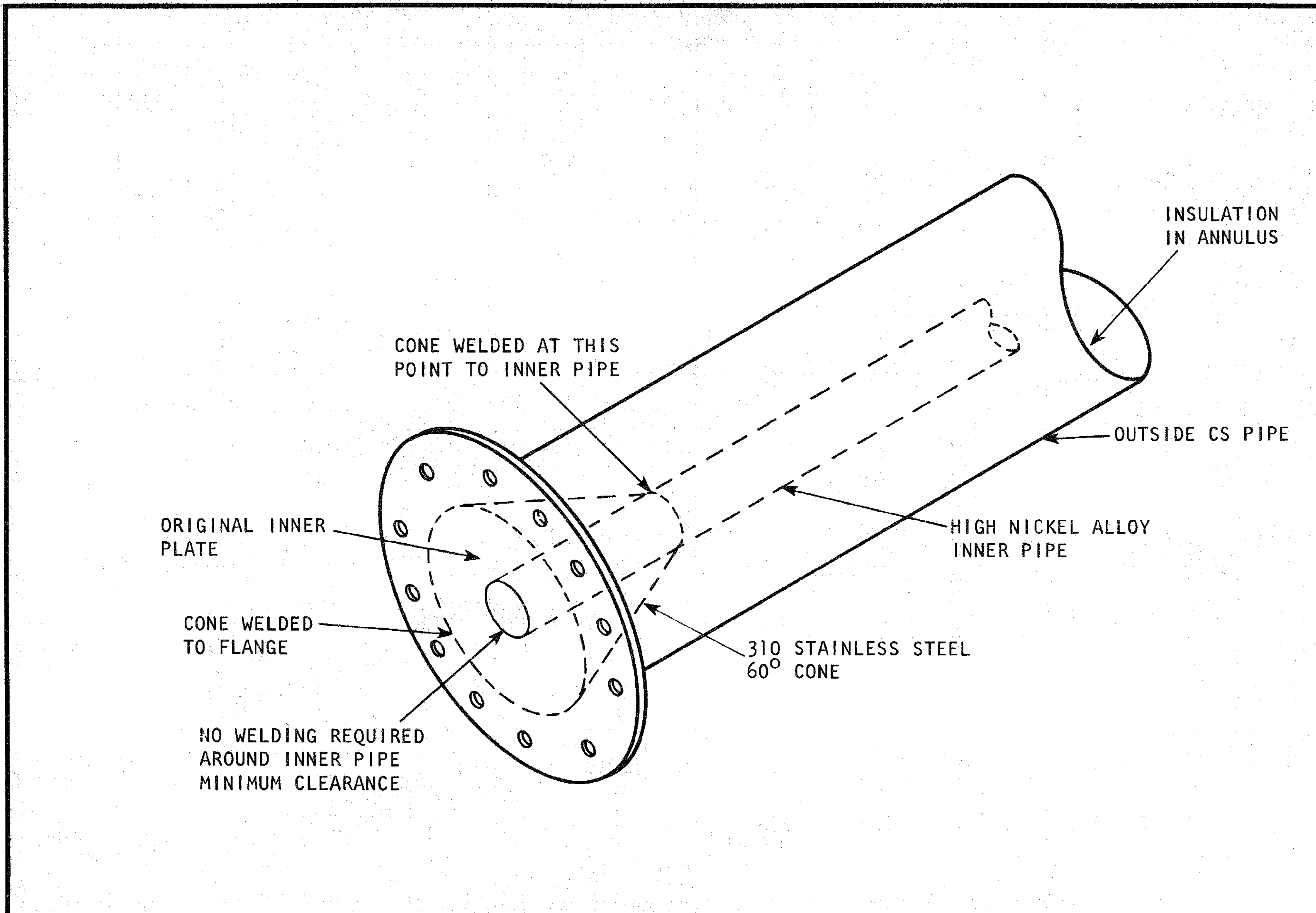


Figure 4-10. TYPICAL CONE SUPPORT

4.2.2 EXTERNAL EXPANSION JOINTS

The original external expansion joints on the hot lines contained bellows of Type 304 stainless steel. The first indication of bellows failure occurred as a leak during Run 19 startup. Some 29 runs (including run subdivisions) and numerous additional temperature and pressure cycles had been experienced in the previous two years of plant operation. The bellows was replaced with a like type and the run continued. When Run 19 was terminated due to a second leaking bellows, a metallographic examination of the bellows was made. It revealed that the failure resulted from stress corrosion cracking which was probably induced by the presence of chlorides, to which the Type 304 stainless steel metal is susceptible.

The leaking bellows was repaired for continuing operation while bellows of a different material of construction were ordered.

The Type 304 stainless bellows was replaced with a solution-annealed Incoloy 825 bellows which resists both stress cracking and chloride attack. The original Type 304 stainless bellows had not been solution annealed.

There were no failures of the Incoloy 825 bellows after their installation in December, 1974.

4.2.3 INTERNAL SLIP JOINTS

The metal liner of the hot solids transfer lines was originally designed and fabricated with slip joints to allow for longitudinal expansion and contraction. The joints performed satisfactorily, with the exception of the two in the acceptor lift line, CD-208. The acceptor lift line service was the most severe of all transfer lines and was the major source of slip joint problems. Consequently, all slip joint modifications were made on this line.

The fuel char lift line slip joints, CD-204, were replaced twice with original design units. Because the original design performed well in this application, no attempt was made to modify these slip joints.

Since commercial transfer lines will be refractory lined without metallic liners, no slip joints will be required.

4.2.3.1 Bell-Shaped Slip Joints

In the original design, all inner pipe slip joints incorporated a bell-shaped design (Figure 4-11). In the acceptor lift line, two were installed. They swedged abruptly, in a 4-inch run, from a 4.5-inch internal diameter to a 4-inch internal diameter. This discontinuity resulted in impingement of solids on the bell. In early runs, the gas velocities were 70 to 100 feet per second with solid loadings of 0.1 to 0.2 pounds solids per standard cubic foot of gas. After the first failure, the gas velocity was lowered to a maximum of 50 feet per second. When the bell-shaped slip joints failed, the hot lift gas cut through the insulation and impinged on the outer carbon steel pipe, creating a hazardous condition. Figure 4-12 is a photograph of the hole caused by such an action.

The two bell-shaped slip joints in the acceptor lift line, CD-208, were replaced with metal cones after Run 16 (January, 1974).

4.2.3.2 Metal Cones

To eliminate the bell-shaped discontinuity in the original design, two truncated cones were fabricated and installed in the lift line. The cones were 6 inches in length with a 4.5-inch major internal diameter and a 4.0-inch minor internal diameter. Before installation, the cones and downstream throat were plasma sprayed with a 0.030-inch-thick hard surfacing of 25 percent nichrome matrix and 75 percent chromium carbide. Figure 4-13 shows the cone-type inner slip joint.

After the duration of one run (Run 17A), the cone joints were inspected. The inspection revealed that most of the chromium carbide hard facing had eroded away during the 110 hours of acceptor circulation up the lift line.

A second set of metal cones was then coated with a 0.050-inch-thick layer of nickel alumide. After 184 hours of circulation in Run 17B, the cones were inspected and the coating found to be badly eroded.

A new set of cones of Type 310 stainless steel, hard faced with Stellite 12, was installed. The Stellite 12 hard facing was applied with stick electrode circumferentially around the inside of the cones. During the following runs, the Stellite 12 hard-faced overlay was eroded somewhat, but after 835 total hours of acceptor operation the base metal was not affected. The Stellite 12 hard facing was replaced and another 630 hours of circulation were logged before one cone was eroded through.

Both cones were replaced with Type 310 stainless steel cones hard faced with longitudinal beads of Stellite 12.

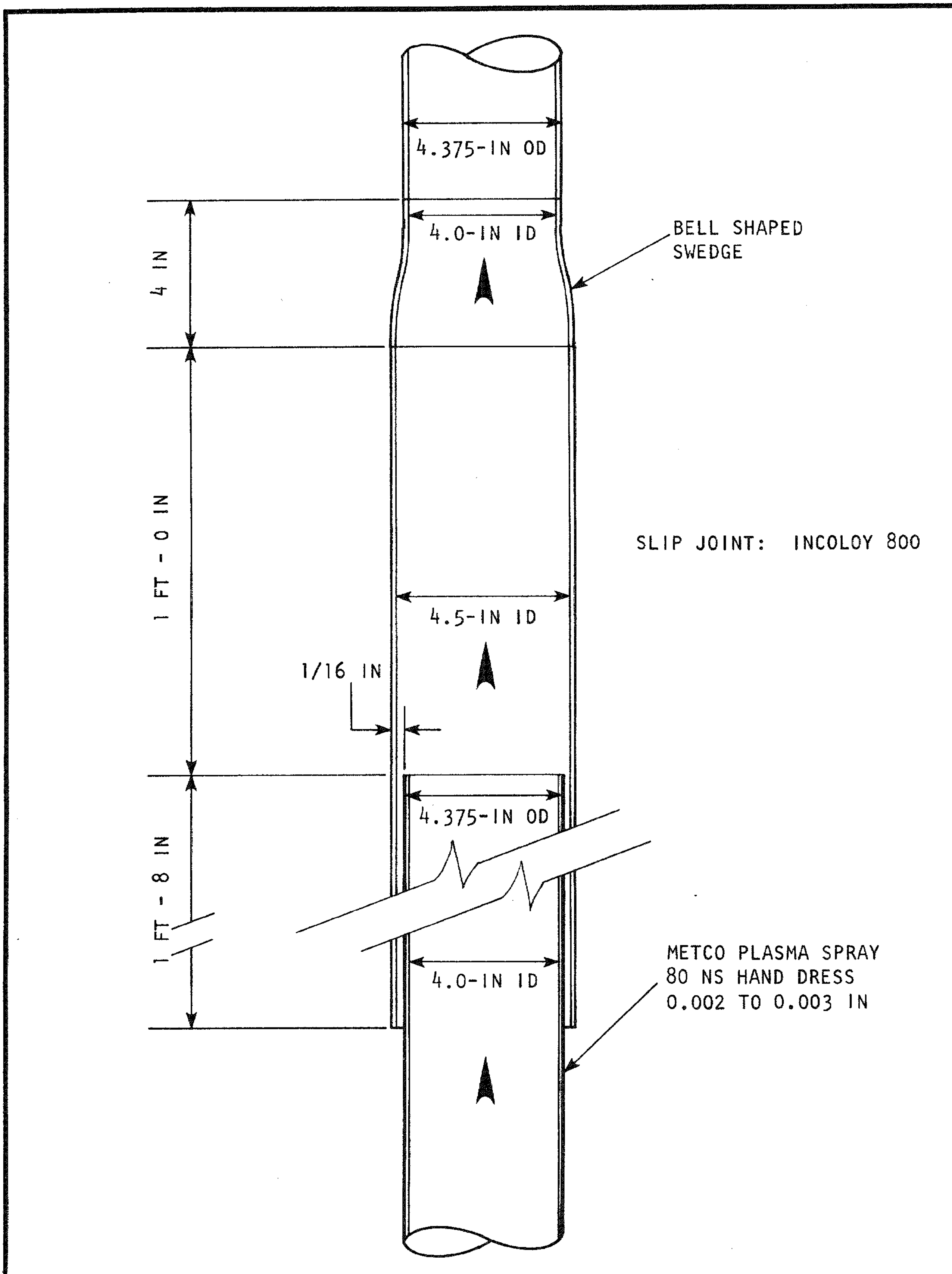
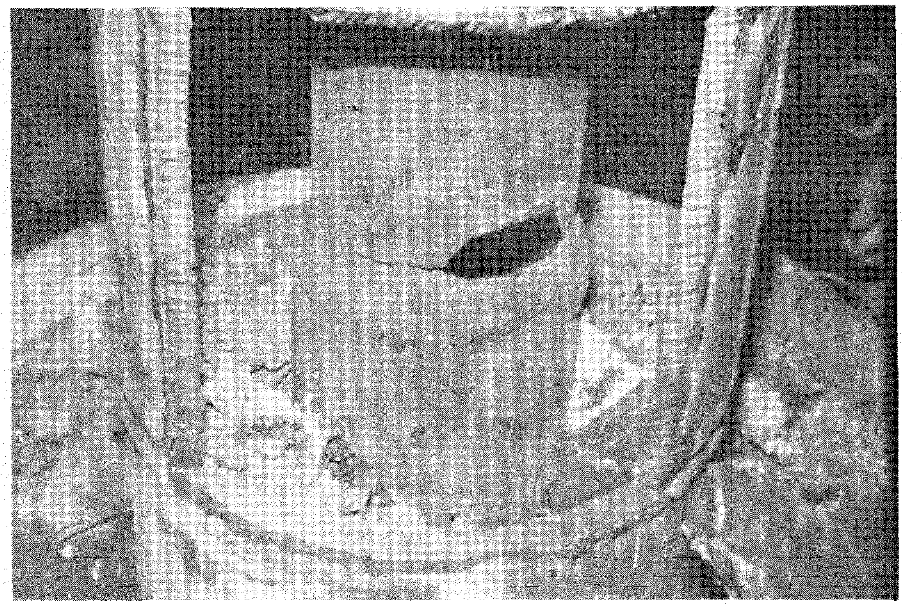


Figure 4-11. ORIGINAL REDUCING BELL DESIGN FOR CD-208 SLIP JOINT



ERODED INNER BELL, SHOWING CUT OUT INNER PIPE
WITH INSULATION REMOVED.

Figure 4-12. ERODED BELL-SHAPED SLIP JOINT

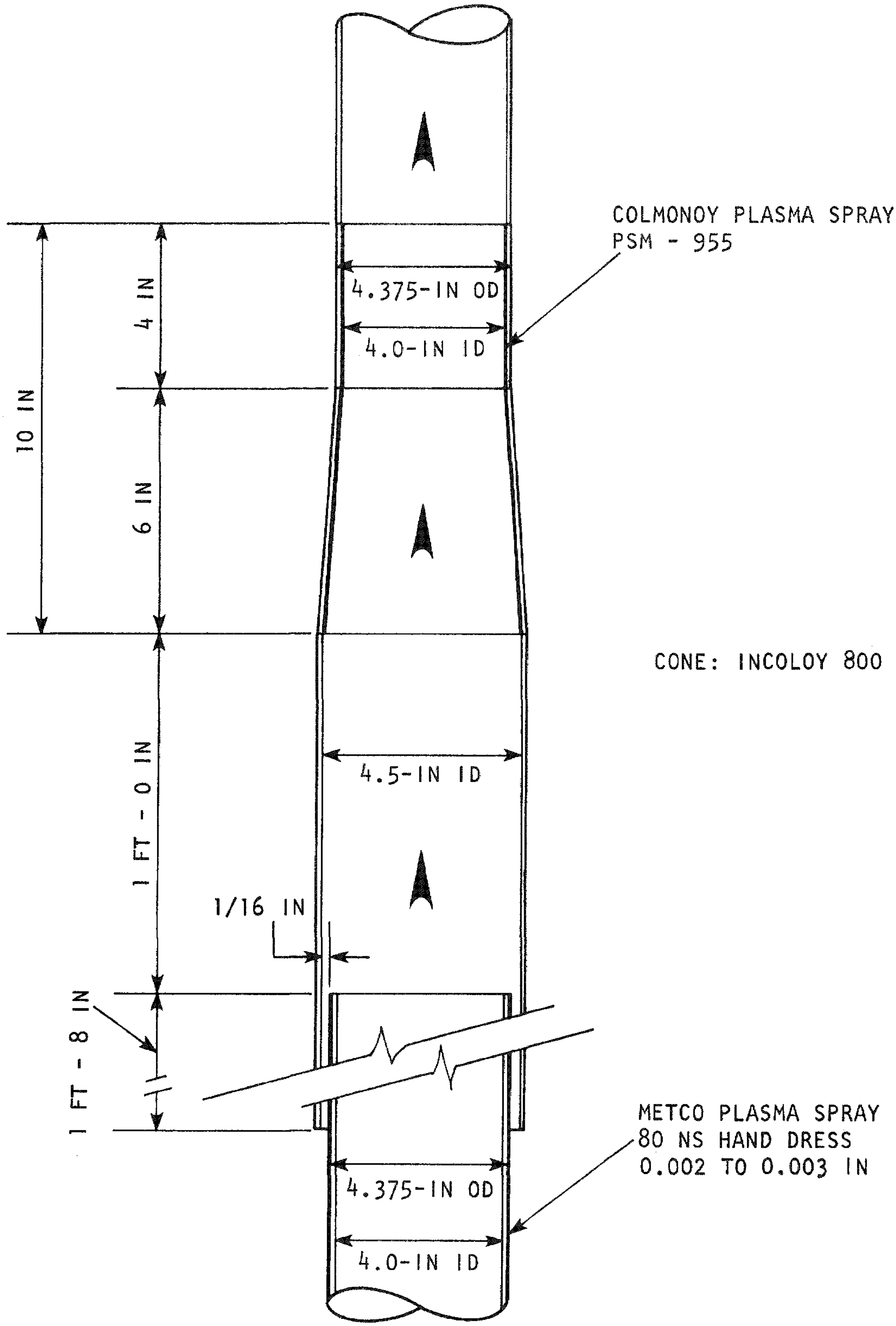


Figure 4-13. FIRST MODIFICATION-CONE DESIGN FOR CD-208 SLIP JOINT

While the truncated cone with the Stellite 12 hard-faced overlay did increase the life of the lift line, the design did not completely solve the problem. Maintenance procedures were initiated whereby the cones were inspected and overlayed or replaced between runs. As runs became longer in duration, the erosion of the cones became more severe. An example of the severe erosion is shown in Figure 4-14. Efforts were directed toward finding a better material or design for the lift line service.

During this period some erosion had also occurred on the lift line at locations other than at the cone. As the inner line heated and cooled, it buckled slightly and threw the metal cone out of alignment. A metal support alignment cone was designed and fabricated to hold the inner pipe straight in the middle of the outer pipe (Figure 4-15.) It was welded to the inside of the outer pipe and beveled to allow the inner pipe to slide while still maintaining the centering of the pipe.

4.2.3.3 Ceramic Cones

The next development in the lift line internal slip joint was the ceramic cone. Coors Porcelain Company was approached as to the possibility of coating or fabricating the two internal slip joints with some type of ceramic. Coors AD-85, an alumina ceramic, was chosen. The ceramic was formed and encased in metal as shown in Figure 4-16. The cone has good compressive strength characteristics but poor tensile strength, so the metal outside shield was designed to contain the ceramic cone.

The two ceramic cones were installed after Run 30, but the line was not in use until Run 33A. After a total of 419 hours of service in Runs 33A and 33B, the cones were found to have no discernible wear. The joints were inspected between runs using a fibers optics viewing device. After Run 38B the upper cone was found to have a crack near the bottom and a chip at the top. The cone was replaced after being subjected to 1233 hours of acceptor circulation. This replacement cone also was found to be cracked, and was replaced, after another 672.7 hours of acceptor circulation.

The two cones and the lift line were replaced by a refractory lift line following Run 40B in the latter part of November, 1976. The lower cone has never been replaced due to wear in service, but had been replaced after sustaining damage during an inspection. The replacement cone had been in service for 1803.7 hours when replaced by the refractory lift line. When removed, the cone was still in excellent condition.

The use of the material in this service was a success, the only drawback to the ceramic being its inability to take tension stress and extreme temperature variations.

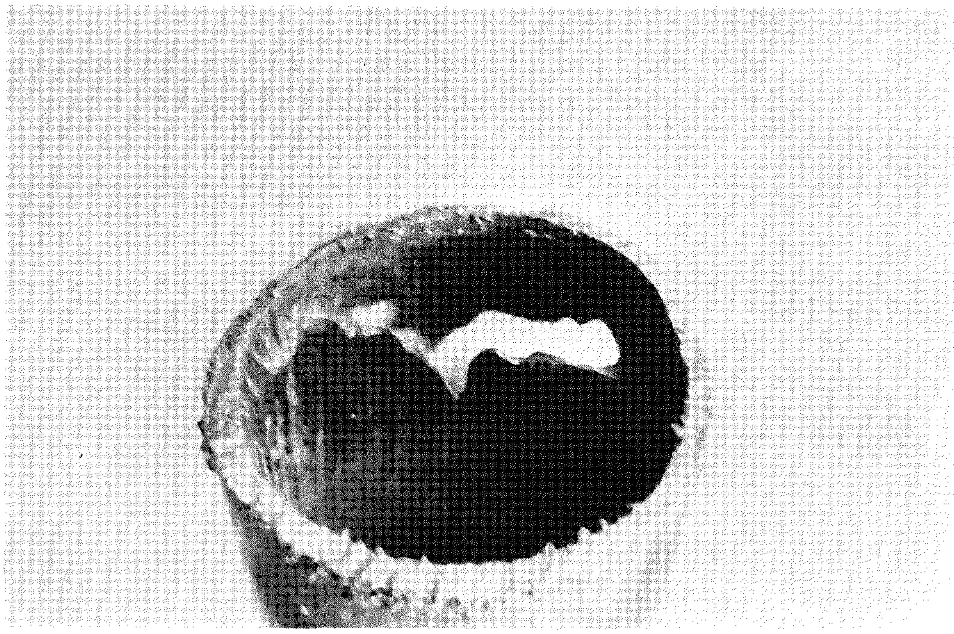


PHOTO SHOWS THE EROSION OF THE INLET OF THE CONE AFTER CONE WAS CUT OUT OF LINE. ALSO NOTE THE LONGITUDINAL STELLITE 12 HARD FACE OVERLAY ON INSIDE OF CONE.

Figure 4-14. ERODED METAL CONE

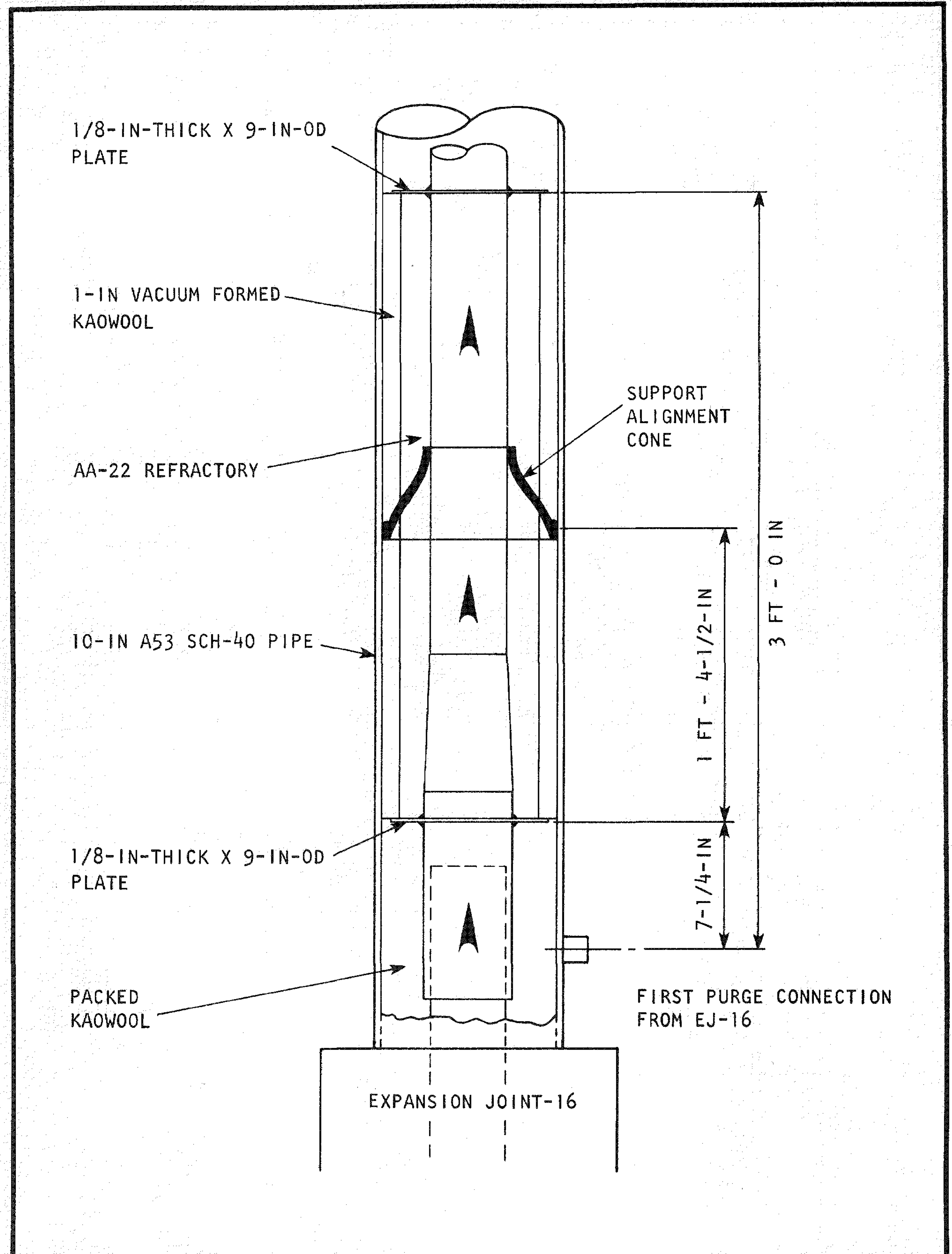


Figure 4-15. AS-BUILT OF SLIP JOINT IN ACCEPTOR LIFT LINE (CD-208) SHOWING SUPPORT ALIGNMENT CONE

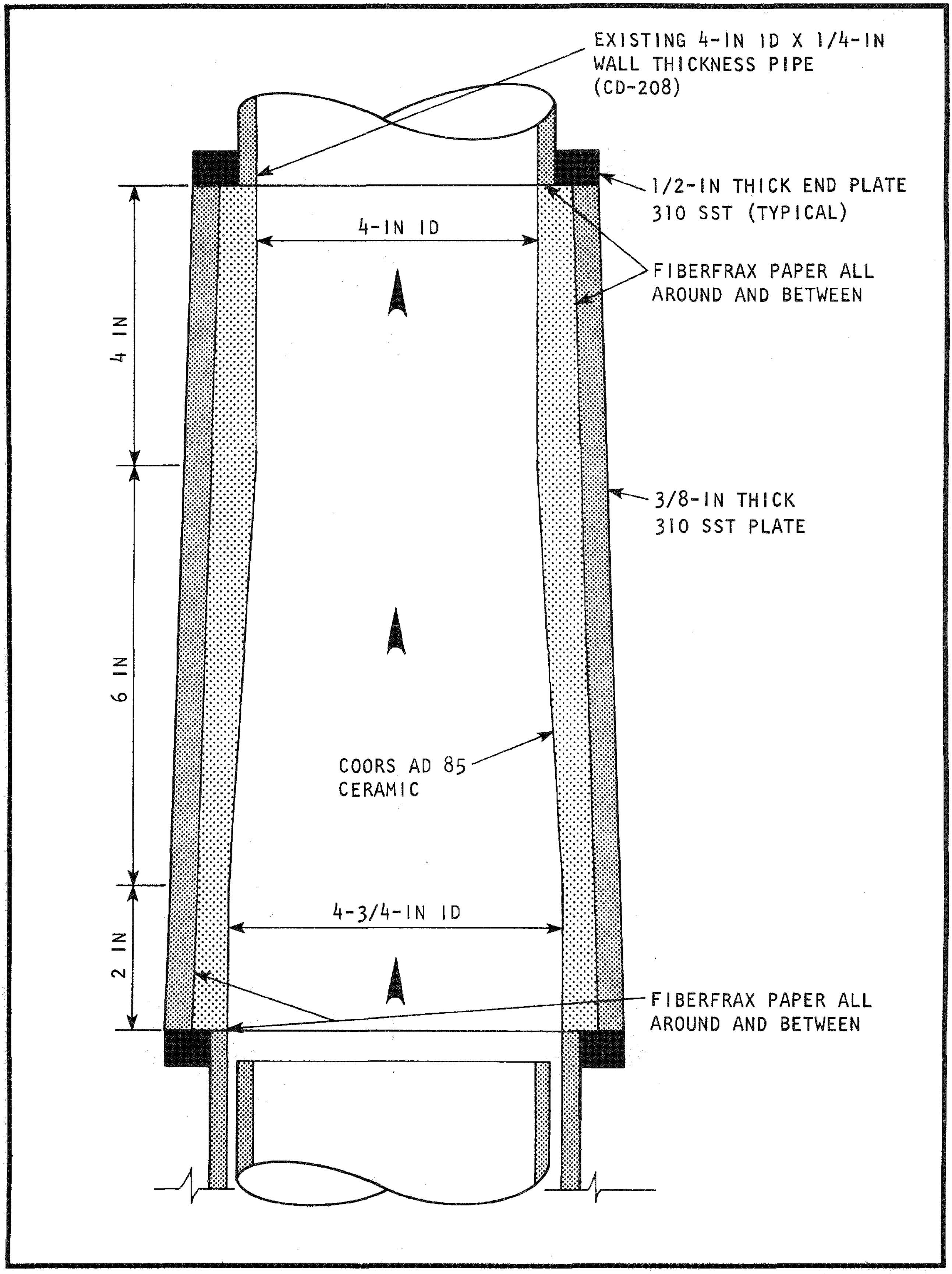


Figure 4-16. CERAMIC TRANSITION PIECE FOR LINE CD-208 (ASSEMBLY)

4.2.4 REFRACTORY LIFT LINE

During the latter part of November and first part of December, 1976, a refractory-lined acceptor lift line was installed. It replaced the lift line containing the Incoloy 800 inner pipe and the ceramic cones.

Figure 4-17 shows a typical cross-section of the installation. The refractory line is contained in a 10-inch ID carbon steel pipe shell. The insulation at the wall is composed of 1-inch thick cylinders of Carborundum Company's Vacuum Cast Fiberfrax ceramic fiber.

To provide compressive support for the inner Mullfrax cylinders, a castable refractory backup material was preferred over the Fiberfrax insulation. However, because the pilot plant configuration required that the original outside diameter of the lift line be maintained, the use of Fiberfrax was necessary.

The Fiberfrax cylinders as fitted into the outer pipe vary in length as per the design requirements, with the longest pieces being 12 inches long. The refractory forming the inside wall is composed of formed cylinders of Carborundum Company's Mullfrax 202 Fused Mullite/High Alumina Refractory. The refractory forms a 4-inch internal diameter. The gasket material is Carborundum Company's Fiberfrax Paper.

The line extends from the engager pot on the first floor up to the upper sixth floor and flanges onto the slide valve, XCV-2010, under the regenerator. Two solution-annealed, Incoloy 825 bellows-type expansion joints provide for the expansion and contraction of the 10-inch outer carbon steel pipe. Flange-face to flange-face, the line measures 86-feet-9-1/4 inches long. The line is composed of six flanged sections, varying in length from 12 feet to 16 feet. The design length of each section was dictated by the existing structure.

Temperature and pressure measurement of the process side of the line posed a design problem. The circular block construction did not lend itself to side-of-pipe taps due to the shift when heating. Holes were drilled through the flanges, insulation, and refractory. At this point there is no movement of the refractory during operation. The design served as a rough temperature indication, but experience showed the temperature measured to be slightly lower than the actual temperature. This is probably due to the location of the thermowell tip at the refractory edge, the conduction of heat through the thermowell piping, and the effect of the larger mass of metal at this location. Figure 4-18 shows the schematic of the temperature and pressure probes.

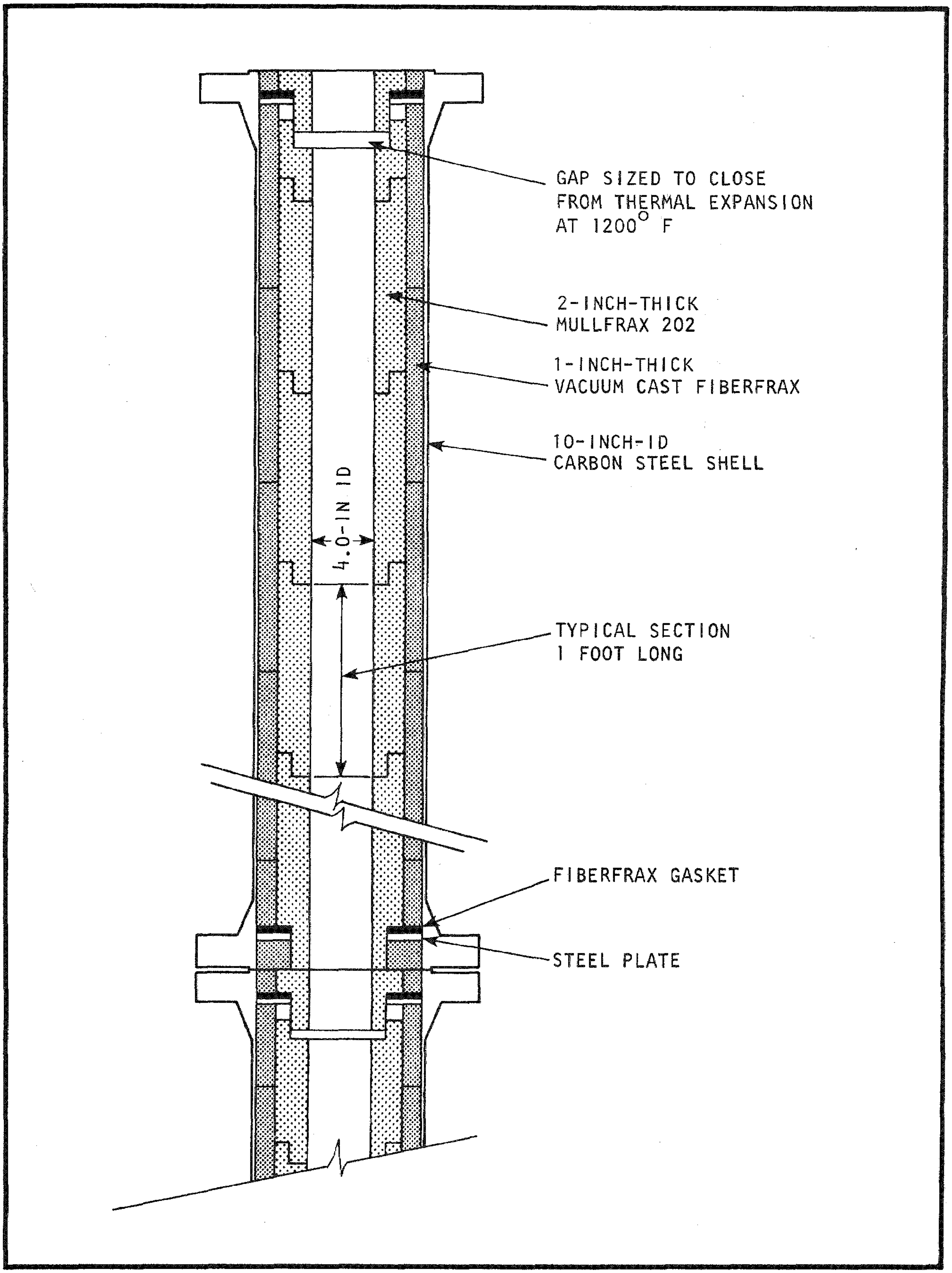


Figure 4-17. TYPICAL SKETCH SHOWING REFRACTORY LIFT LINE CD-208

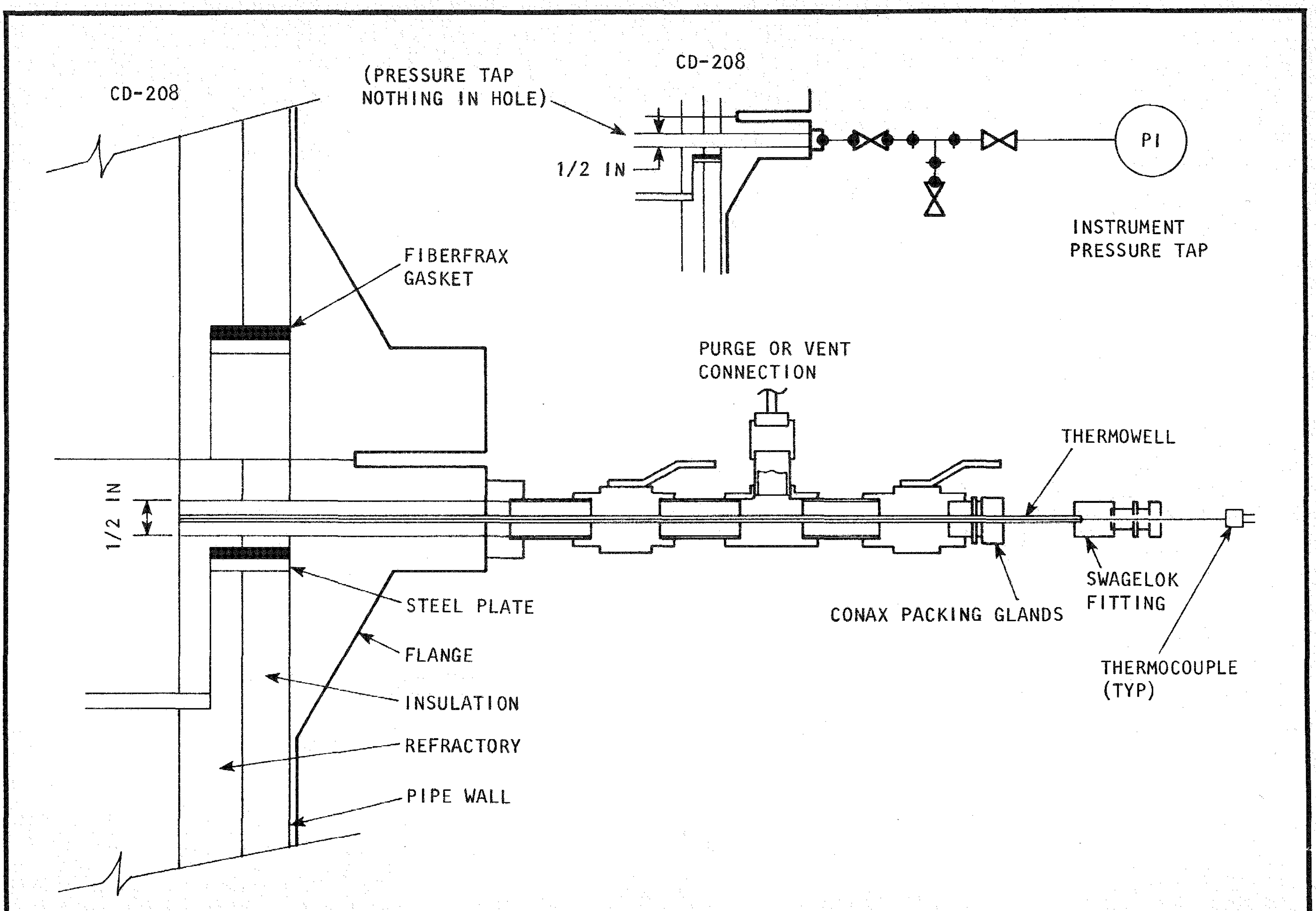


Figure 4-18. TEMPERATURE AND PRESSURE PROBE CONSTRUCTION FOR CD-208 REFRACTORY LINE

Through the end of Run 43A, the lift line had been subjected to a total of 516.5 hours of acceptor circulation. Inspection of a hot spot, which had developed during the run, revealed cracked refractory liners. The majority of the refractory liners in the bottom three sections were cracked longitudinally. The lift line had been overpressured on previous occasions and this was thought to have caused the cracking. The end liners of each section were replaced and the remaining layers received a brush coat of Resco AA-22 hard-face castable refractory.

During the next five runs an additional 850.7 hours of acceptor circulation were logged, bringing the total hours of acceptor circulation service to 1367.2. Repair work to the line between runs included the replacement of liners with three or more cracks, patching cracks, and the filling of insulation voids.

After Run 46C and a total of 1565.2 hours of acceptor circulation, the majority of the refractory liners in the top two sections were replaced with new liners. Then, after 1693.2 hours, the liners in the bottom pipe section of the line were replaced.

At the termination of pilot plant operation, the refractory lift line had been in acceptor circulation service for a total of 1845.2 hours. It is believed that the rapid over-pressuring and pressure surges encountered in the plant operation were the cause of the cracking. The successful use of a refractory-lined acceptor lift line forms an adequate basis for commercial design. The problem of refractory cracking will be reduced in a commercial line, which will be large enough in diameter to tolerate the use of castable refractory backup for the inner hard-face refractory or a totally monolithic construction.

4.2.5 STANDLEG MODIFICATIONS

The solids transfer standlegs were modified during the pilot plant operation to correct both process and mechanical problems. Experience showed that each line must be treated differently according to the individual problems encountered.

4.2.5.1 Slip Joints

The major standleg slip joint modification was the removal of the original internal slip joint in the calcined acceptor overhead transfer line, CD-206. This joint frequently filled with solids and bound up, pulling the ends loose upon cooling.

The 26-foot-9-3/4-inch section of the calcined acceptor standleg which contained the slip joint was replaced with two spool sections prior to Run 43B. (See Figure 4-19.) As shown in the figure, the line expansion provided by the single tapered internal slip joint was accommodated by the use of sleeves at the bottom end of each spool piece. The sleeves provide a loose fit which supports the internal lines and allows the liner to grow freely in the axial direction. This design worked extremely well.

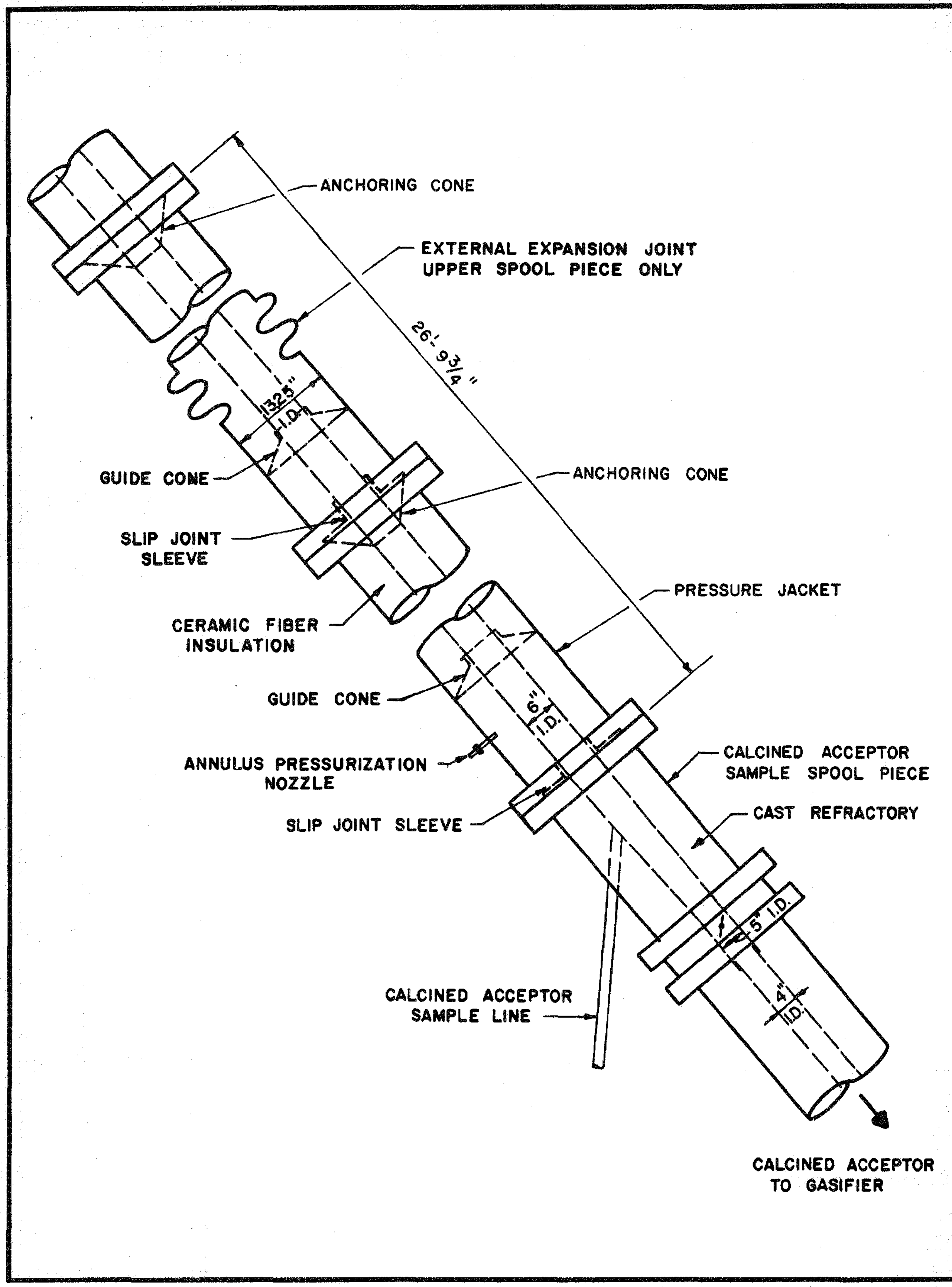


Figure 4-19. CALCINED ACCEPTOR STANDLEG (AFTER RUN 43A)

4.2.5.2 Refractory-Lined Spool Pieces

The fabrication and installation of several refractory-lined spool pieces solved two main problems in the solids standleg lines.

As pilot plant operation progressed, the opening, rolling out, and inspection of the inside of these lines became more frequent. Since the lines were large and ran through floors, this job was very cumbersome. Spool pieces were installed in advantageous locations. Removal of the spool pieces left the inside of the remaining line accessible for inspection and the line could then also be moved longitudinally if necessary.

Another problem encountered with the original standleg design was the failure of the sample piping where it connects to the inner standleg pipe. The sample outlet pipe would shear off where welded to the inner pipe. There are extreme thermal stresses as the inner pipe expands lengthwise when heating. Also, the alternate withdrawal of a hot solids sample and injection of the cool purge gas puts the sample outlet pipe through numerous temperature cycles each day of operation.

The spool piece design which was decided upon used the existing-diameter outer line pipe and inserted flanges for the spool and mating line. Kaowool insulation was used next to the outer wall and a layer of Resco AA-22 Refractory Castable formed the inner wall. The location and design of the spool pieces were changed during plant operation to improve their use and to accommodate other modifications. The final location of the spool pieces is shown on Figure 4-20.

The original spool piece at the sample Station, S-9, on the overhead acceptor transfer line, CD-206, is shown in Figure 4-21. The Kaowool insulation was used as shown and the inner lining was formed with castable refractory. The sample outlet was a stainless pipe inserted through a hole formed in the refractory, with clearance to expand or contract. This design was successful but the spool piece had to be cut apart several times to replace the sample pipe.

The design was modified as shown in Figure 4-22 which shows the spool piece used at sample point S-13 in the char transfer line, CD-204. Insulating refractory is used in place of the Kaowool so that the formed sample outlet is hard surfaced on the inside. The sample outlet nozzle is flanged, and refractory forms the sample outlet rather than a metal pipe. The figure shows a typical thermowell assembly for a spool piece. In this case, two were used.

Figure 4-23 shows a typical inline spool piece with no sample point location. Kaowool insulation is used. This design of spool piece was used at the upper ends of the standlegs. No spool piece was required on the fuel char line, CO-204, as the pickup point at the bottom and the disengager pot at the top were both flanged and could be rolled out of the line.

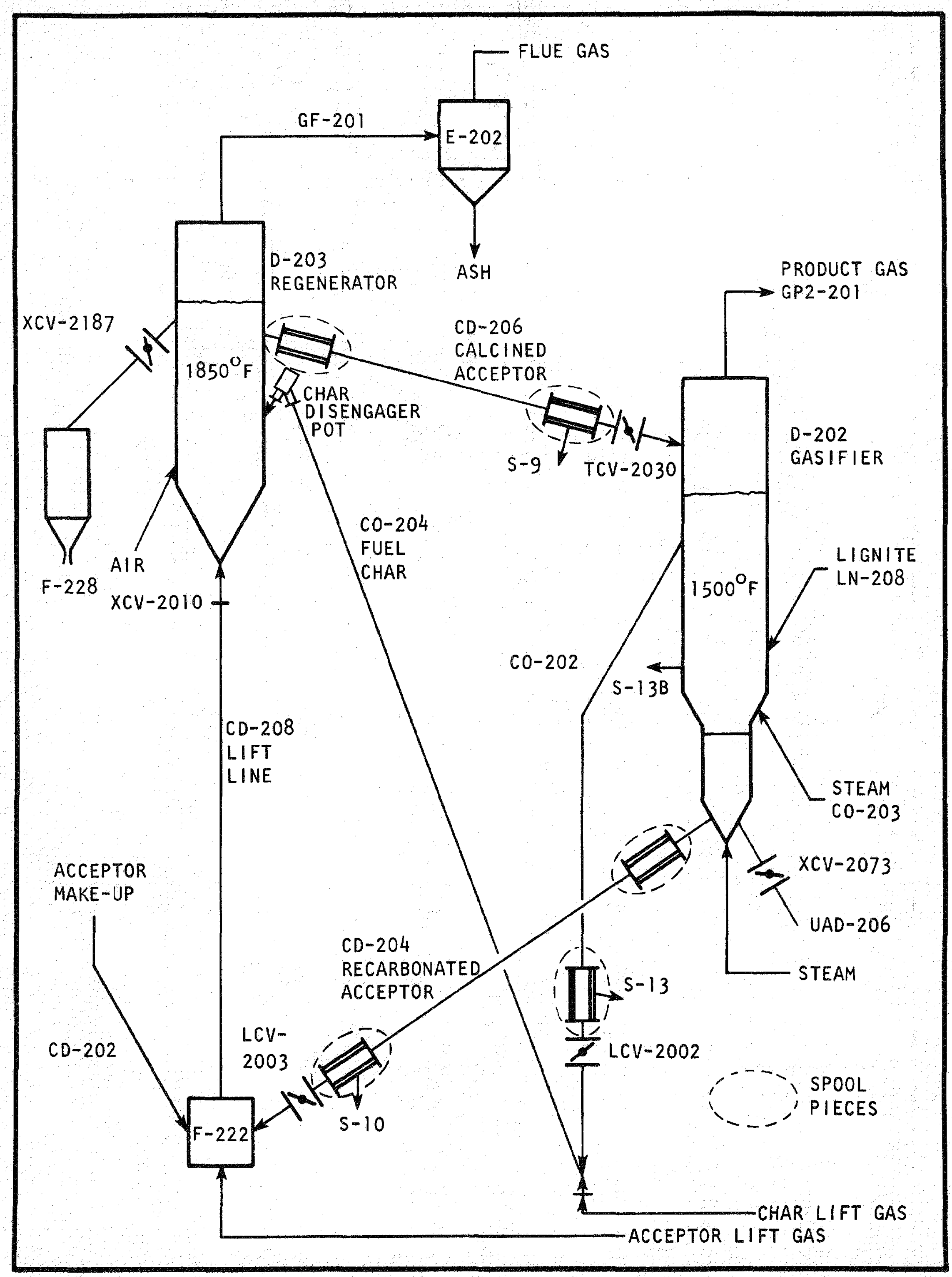


Figure 4-20. SAMPLE CONNECTIONS AND REFRACTORY-LINED SPOOL PIECES

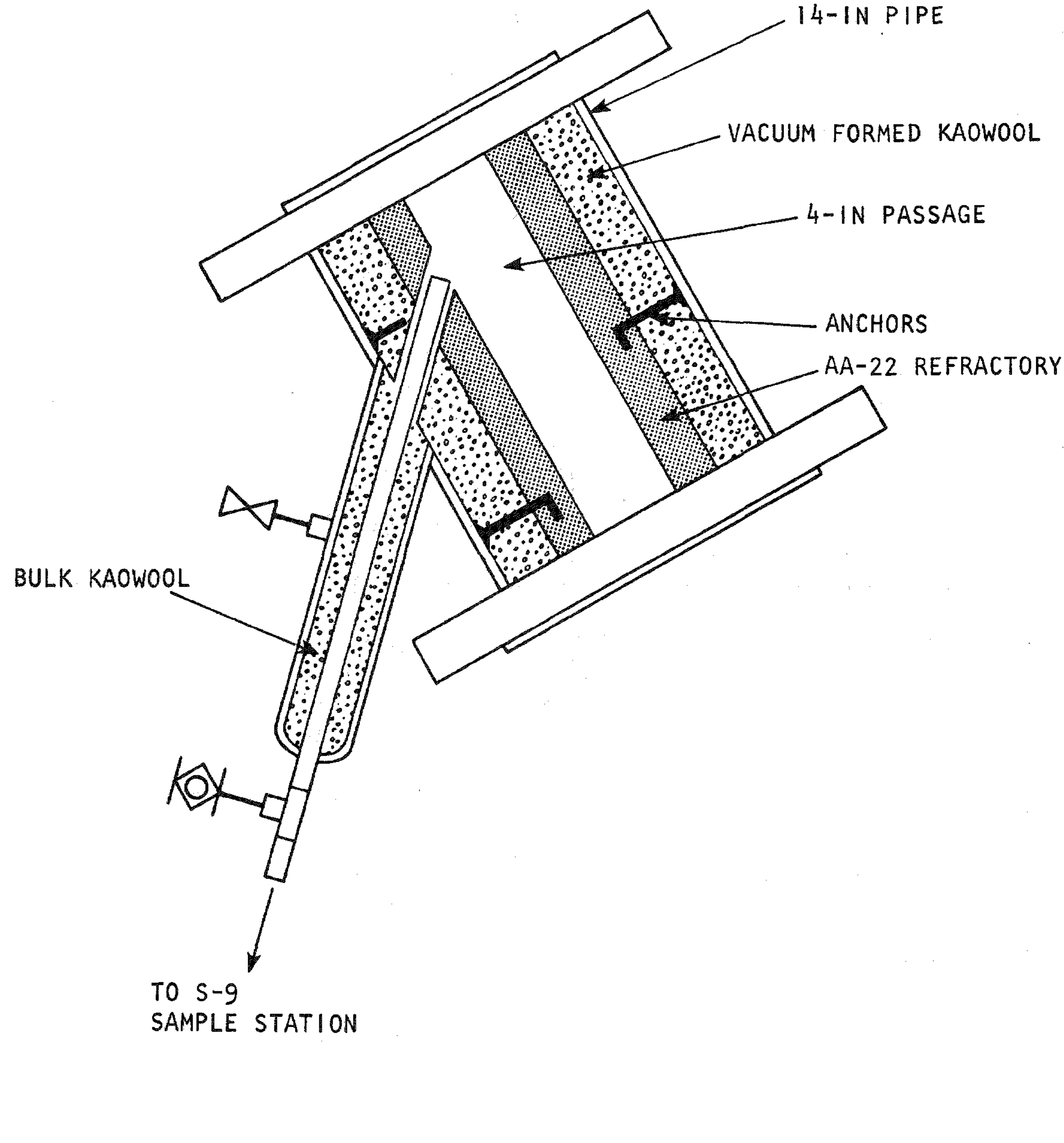


Figure 4-21. ORIGINAL SPOOL PIECE ON OVERHEAD ACCEPTOR TRANSFER LINE (CD-206) WITH CONNECTION FOR S-9 SAMPLE POINT

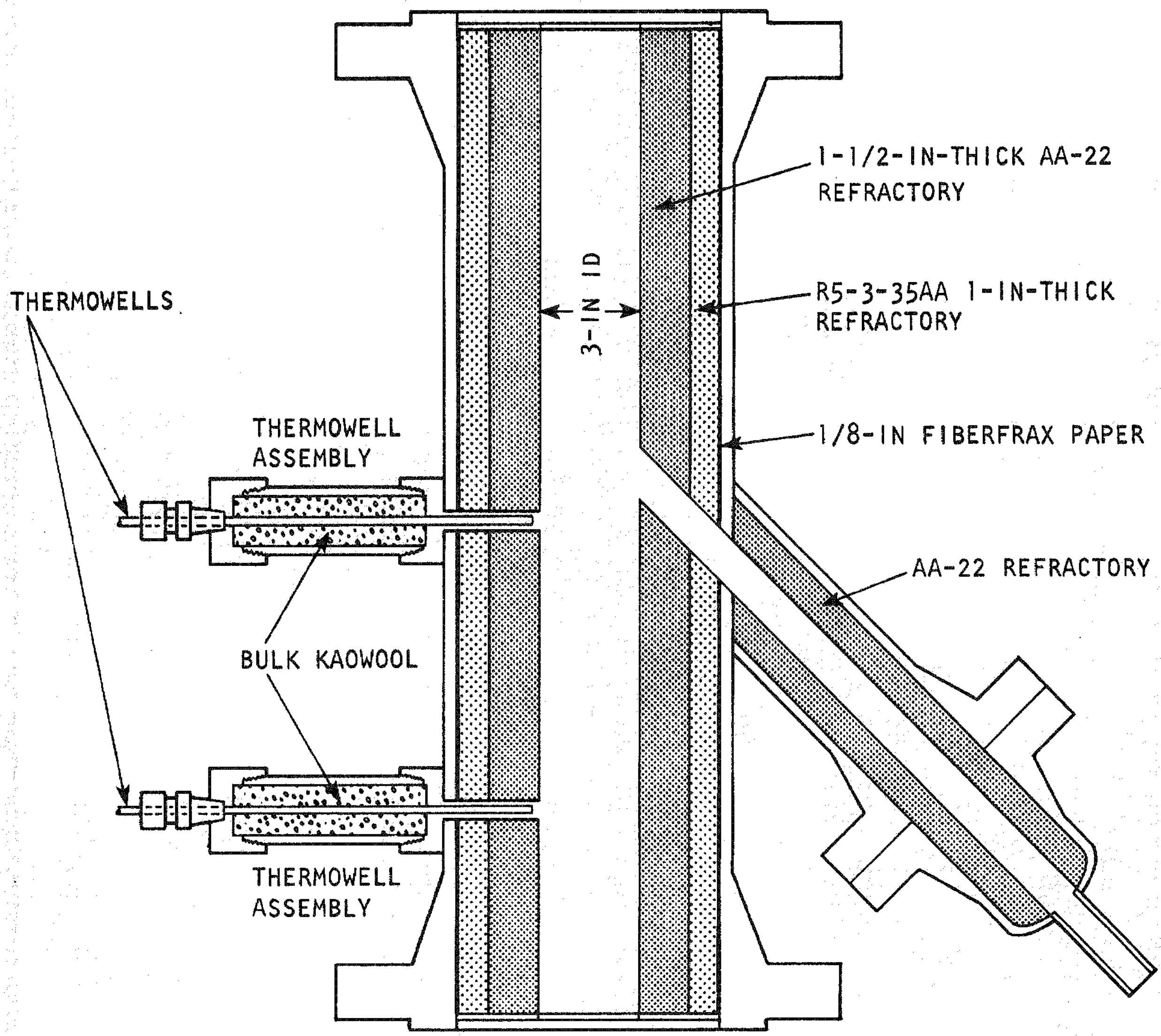


Figure 4-22. SPOOL PIECE ABOVE LCV-2002 IN LINE WITH CONNECTION FOR S-13 CHAR TRANSFER SAMPLE STATION

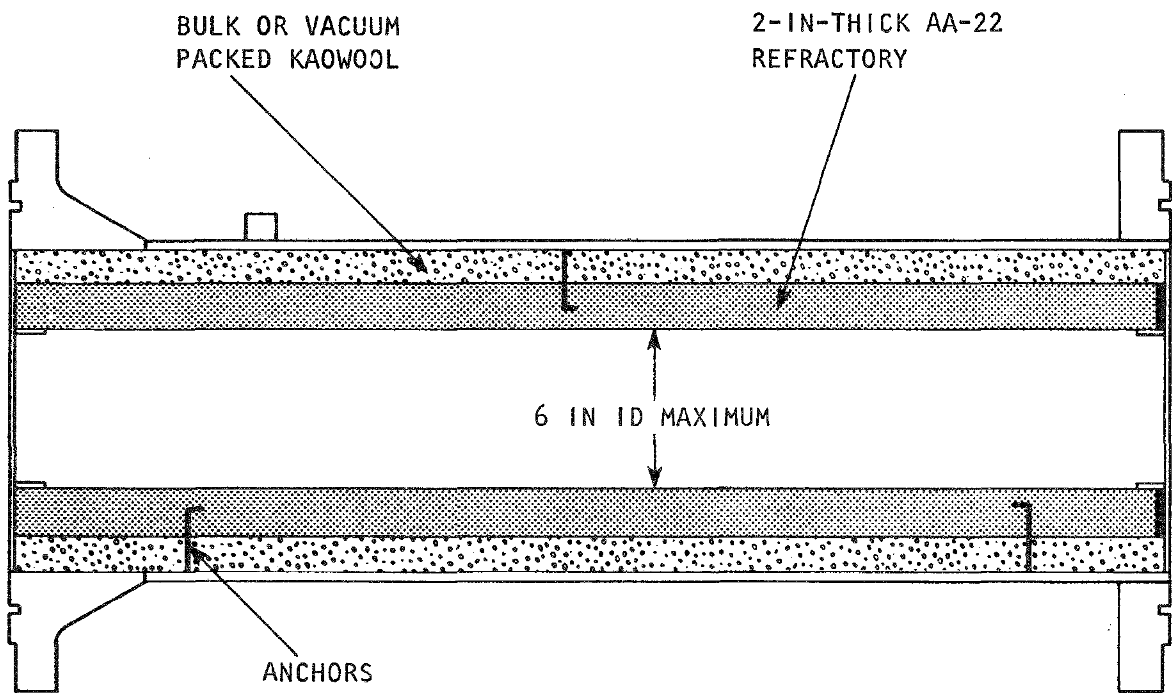


Figure 4-23. TYPICAL INLINE SPOOL PIECE WITH NO SAMPLE POINT

4.3 FIRED HEATERS

The original plant design required a total of 15 process heaters. Several were radiant sections; several were convection sections located in the stack breeching to preheat the process gases. Table 4-2 is a listing of all process heaters, including a history of heater coils replacements, the original materials of construction, and the sequence of replacement.

Since the devolatilizer system was not utilized, consideration is limited to the heaters in gasifier or regenerator service.

4.3.1 GASIFIER FIRED HEATERS

The gasifier heaters were a major source of problems and concern during the first three years of operation. Several runs had to be aborted due to tube failures in the heaters or plugging of tubes and downstream piping with iron-nickel-sulfide deposits.

Figure 4-24 is a schematic of the process flows for the gasifier heaters. As indicated, the heaters were used to heat both the gasifier boot flow and the side flow to the transition section. The recycle gas passes through heater, B-201-C, then splits into two streams, one the boot flow and the other the side flow. The boot flow passes sequentially through heaters, B-201-IIIA, B-201-IIA, and B-201-IA, while the side flow passes sequentially through heaters B-201-IIIB, B-201-IIB, and B-201-IB. During early runs, recycle gas (gasifier product gas) was split between the boot flow and side flow. In later runs, the boot flow was a mixture of steam and recycle gas after the gasifier was at 1500°F and lignite or subbituminous coal was being fed. As the run progressed, recycle gas was replaced by steam until the boot flow contained no recycle gas. No recycle gas was admitted to the side flow, except during startup when the recycle was inert gas (88 percent N_2, 12 percent CO_2). The side flow was either steam or a mixture of steam and air.

High-temperature corrosion, resulting from H_2S in the recycled gasifier product gas, was severe during the initial operation. Corrosion rates were greater than 1 inch per year. The Incoloy 800 tube material has excellent properties for strength at 1500°F, but it is subject to sulfur attack above 800°F.

The worst problem originally was in the gasifier boot and side flow heaters, B-201-1A and IB. The coil in B-201-IA was replaced in April, 1973, because of severe thinning throughout the coil, particularly in the outlet elbows. The replacement coil was the original B-207-IA devolatilizer boot heater, which was identical to the B-201-IA coil. (See Figures 4-25 and 4-26.)

The initial attempt to reduce sulfur corrosion was a reduction of the outlet temperature of the boot flow heater. The original operating temperature was 1600°F; this was systematically lowered over several runs to 600°F. Continued deposits of an iron-nickel-sulfide material in the outlet piping gave evidence of continued corrosion.

HEATER	ORIGINAL MATERIAL	ALPHA	BETA	GAMMA	DELTA	EPSILON	ZETA	ETA
B-201-IA Gasifier Boot Heater-I	Incoloy 800	Failed, removed, cleaned, then repaired.	Installed April, 1973. Failed but was not repaired. At plant site. Original B-207-IA coil.	Installed Dec. 1973. Inlet ells replaced with Alonized mat'l. Now in service	Fabrication complete, at plant site.			
B-201-IB Gasifier Side Heater-I	Incoloy 800	Failed, removed, not repaired. At plant site.	Installed Dec. 1973. Now in service. Incoloy 800.	Fabricated of Alonized RA-330; at plant site.				
B-201-IIA Gasifier Boot Heater-II	Incoloy 800	Failed, removed, not repaired. At plant site.	Installed Mar. 1976. Now in service. Incoloy 800.	Deleted.	Fabrication complete, at plant site.			
B-201-IIB Gasifier Side Heater-II	Incoloy 800	Now in service.	Fabrication complete, at plant site.					
B-201-IIIA Gasifier Boot Convection Heater	321 SS GrH	Failed, removed, not repaired. At plant site.	Installed Mar. 1974. B-207-IIIA was used, has been removed due to thinning of outlet ells. At plant site.	Now in service. Fabricated of Alonized 304 SS 1.625" O.D. X 0.312" W.T. Replaced "D" pass outlet ell because of sample.				
B-201-IIIB Gasifier Side Flow Convection Heater	321 SS GrH	Failed, removed, not repaired. At plant site.	Installed Dec. 1976. Now in service. Incoloy 800.					
B-201-C Gasifier Upper Convection Heater	321 SS GrH	Now in service.	Fabrication complete at plant site.					
B-203 Air Heater	Inlet-321 SS GrH. Outlet-Incoloy 800.	321 SS section was replaced with B-207-IIA coil in Jan. 1974. Furnace in poor condition due to coil support.	Incoloy 800 coil was fabricated but used to make B-205 Epsilon coil.	Fabrication complete, at plant site. Alonized RA-330.				
B-204 Char Lift Gas Heater	Inlet-321 SS GrH. Outlet-Incoloy 800.	Now in service.	Fabrication complete, at plant site. RA-330.					
B-205 Acceptor Lift Gas Heater	Inlet-321 SS GrH. Outlet-Incoloy 800.	Failed & was rebuilt using Alonized RA-330 & Incoloy 800 on outlets. Failed second time & removed Oct. 1975. At plant site.	Installed Oct. 1975. Failed, removed, not repaired. At plant site.	Installed Oct. 1975. Fabricated of Inconel 702 with vertical header design. Removed after failure & at plant site.	Fabrication was started using Inconel 702 & vertical header design. Stopped after Gamma coil failure.	Fabricated from B-203 Beta coil. Failed due to polythionic stress corrosion.	Fabricated using Alonized RA-330 & installed in February 1977.	Fabrication complete at plant site.
B-207-IA Devolatilizer Boot Heater-I	Incoloy 800	Was removed & used as Beta coil in B-201-IA.						
B-207-IIA Devolatilizer Boot Heater-II	Incoloy 800	Was removed & used to repair B-203 Alpha coil.						
B-207-IIIA Devolatilizer Boot Convection Heater	320 SS GrH	Was used as B-201-IIIA Beta coil.						
B-207-B Devolatilizer Side Heater	Inlet-321 SS GrH. Outlet-Incoloy 800	Converted to a steam superheater for steam to gasifier.						
B-207-C Devolatilizer Upper Convection Heater	321 SS GrH	Original use was as steam superheater. Not adequate & is not in service.						

Table 4-2. PROCESS HEATERS HEATER COIL REPLACEMENT STATUS

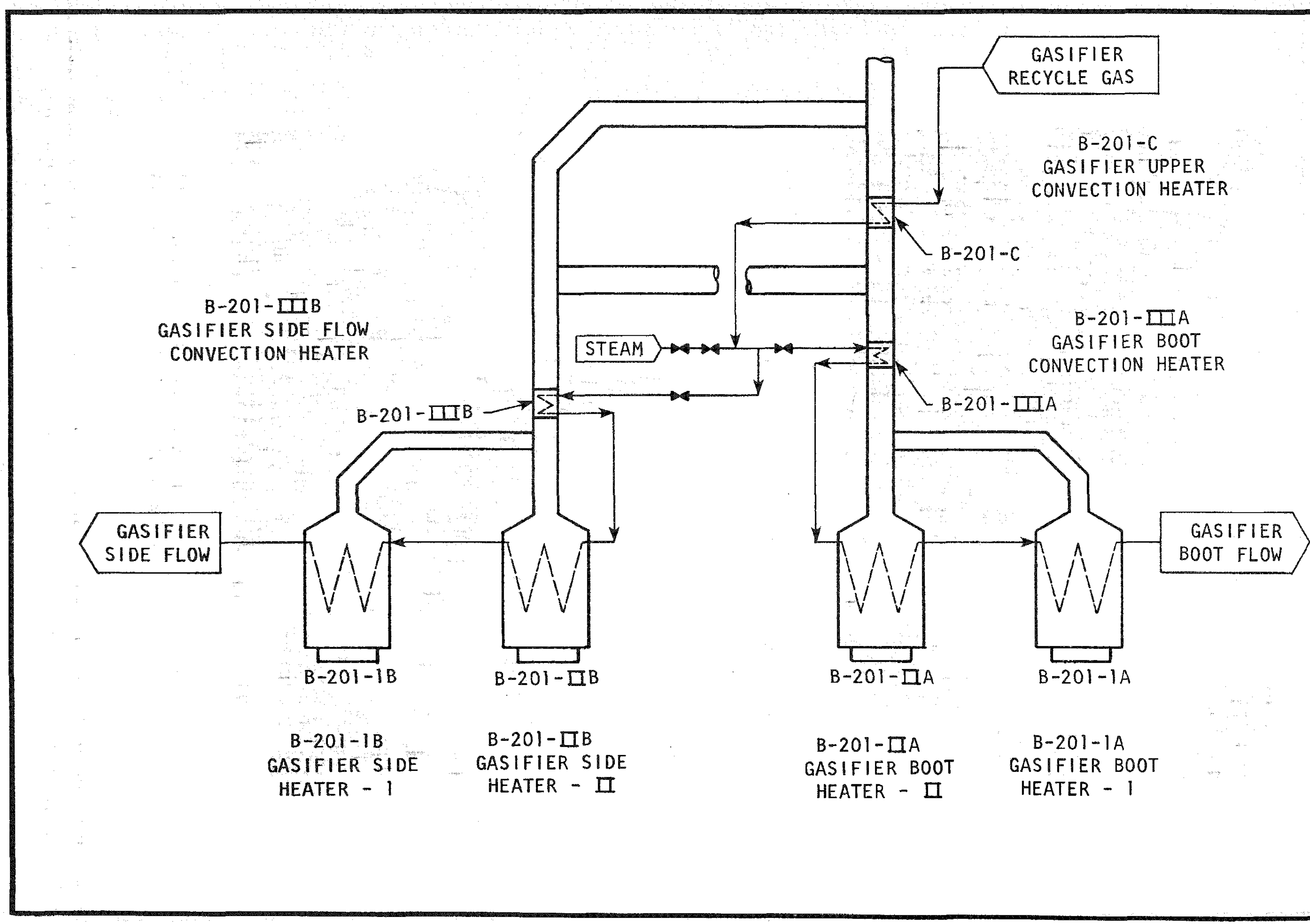
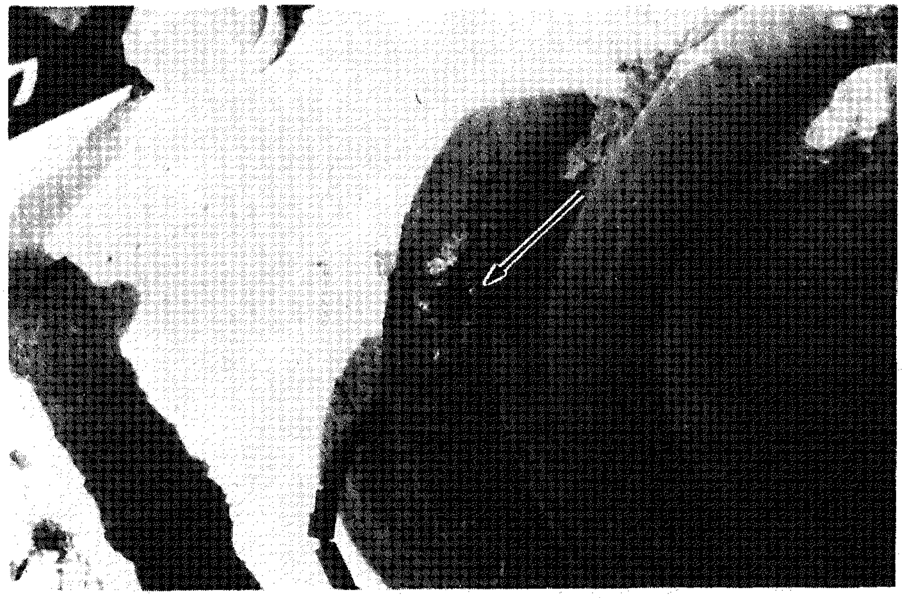
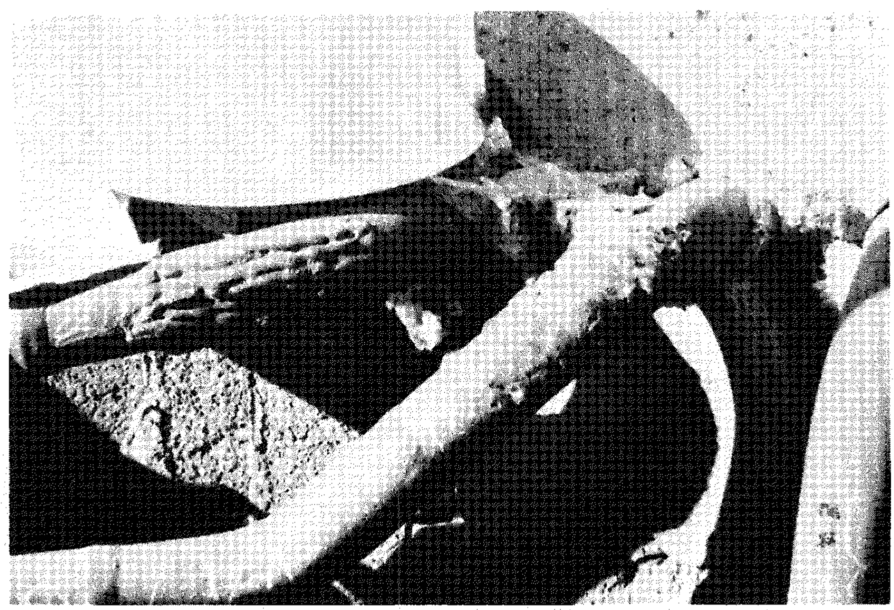


Figure 4-24. GASIFIER RECYCLE GAS HEATERS

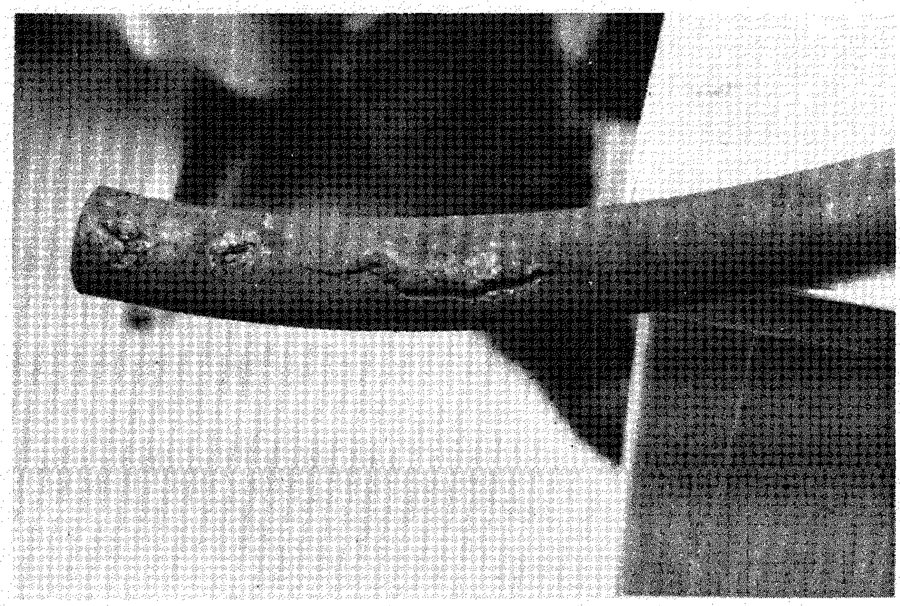


B-201-IB, GASIFIER SIDE FLOW HEATER-I, OCTOBER 20, 1973, SHOWING LEAK AT WELD OVERLAY ON OUTLET ELBOW.

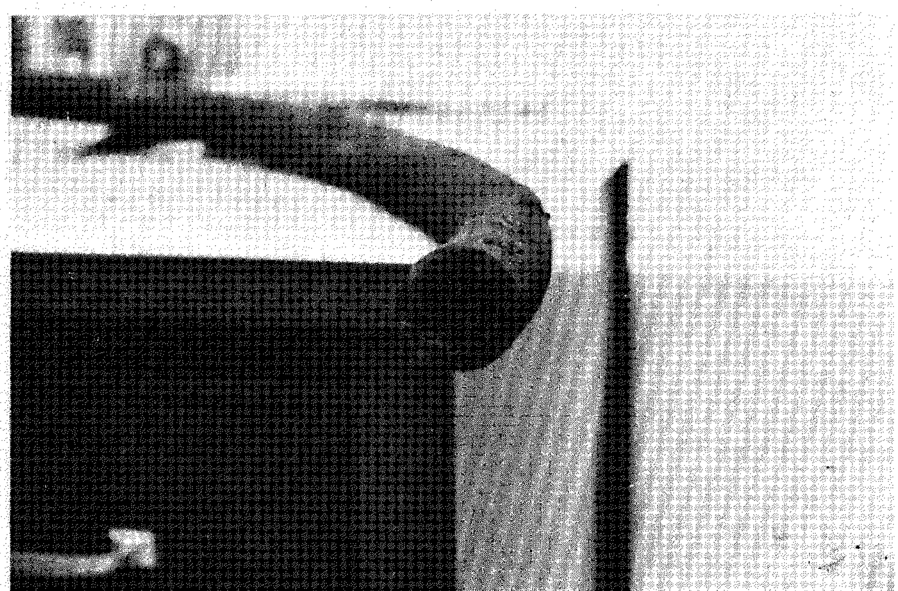


B-201-IB, GASIFIER SIDE FLOW HEATER-I, OCTOBER 20, 1973, SHOWING MORE WELD OVERLAYS ON OUTLET ELBOWS.

Figure 4-25. SULFUR CORROSION IN GASIFIER SIDE FLOW HEATER COIL



B-201-IB, GASIFIER SIDE FLOW HEATER-I, OCTOBER 20, 1973, SHOWING CRACK IN OUTSIDE RADIUS OF TUBE.



B-201-IB, GASIFIER SIDE FLOW HEATER-I, OCTOBER 20, 1973; END VIEW OF TUBE IN TOP PICTURE SHOWING DEPOSIT OF IRON-NICKEL-SULFIDE.

Figure 4-26. SULFUR CORROSION IN GASIFIER SIDE FLOW HEATER COIL

The literature [1] indicated that increasing the hydrogen-to-hydrogen sulfide ratio in the recycle gas would reduce the rate of sulfur attack. However, the addition of steam to the system, which because of process considerations required elevated outlet temperatures, seemed to have little, if any, effect upon corrosion rates. Elimination of sulfur in the recycle gas appeared to be the only solution to the problem.

After comparison of alternative systems, a zinc oxide H_2S removal system was selected and installed in December, 1973. This system, shown in Figure 4-27, reduced the normal H_2S concentration to less than 5 parts per million. Also in December, 1973, new coils were installed in B-201-IA, gasifier boot heater-I, and in B-201-IB, gasifier side heater-I. No more deposits of iron-nickel-sulfide or corrosion due to sulfur attack were found after the installation of the zinc oxide unit.

After the sulfur corrosion problem was solved, a second problem became evident. This metal attack is referred to as "metal dusting" or carburization, and occurs in high-nickel-chrome alloys that operate in a reducing atmosphere at 750°F to 1100°F with gases containing carbon dioxide, carbon monoxide, and lower molecular weight hydrocarbons, such as methane and ethane. Thus, the coils affected were the lower temperature coils B-201-IIIA and B-201-IIA. (See Figures 4-28 and 4-29.) When the B-201-IIA heater was bypassed in March, 1974, the critical temperature region existed at the inlet of B-201-IA heater, which began to undergo metal pitting.

In October, 1973, a corroded section of an Incoloy 800 tube From B-201-IA was sent to Lockheed Palo Alto Research Laboratory for metallurgical study.

The conclusions reached by Lockheed were as follows:

(1) The Incoloy 800 pipe had been degraded by a combination of carburization, sulfidation, and oxidation.
(2) Carburization in depth preceded the other reactions and established a chromium-rich phase at the grain boundaries.
(3) Carbon had been preferentially oxidized from the carbide in the grain boundaries and twin bands, leaving a chromium-rich iron-nickel-chromium alpha phase in these regions. This phase, in turn, is readily oxidized and sulfidized, leading to total destruction of the alloy.
(4) The unusual history of exposure to a cyclic combination of carburizing, oxidizing, and sulfidizing environments is believed to be responsible for this behavior. A low oxidizing potential limits the formation of protective oxide films on the surface which are needed to retard diffusion of carbon and sulfur.

---

(1) Draynieds and Samans "Kinetics of Reaction of Steel with Hydrogen Sulfide-Hydrogen Mixtures", Journal of the Electrochemical Society, Vol. 105, No. 4 (April, 1958), p. 183.

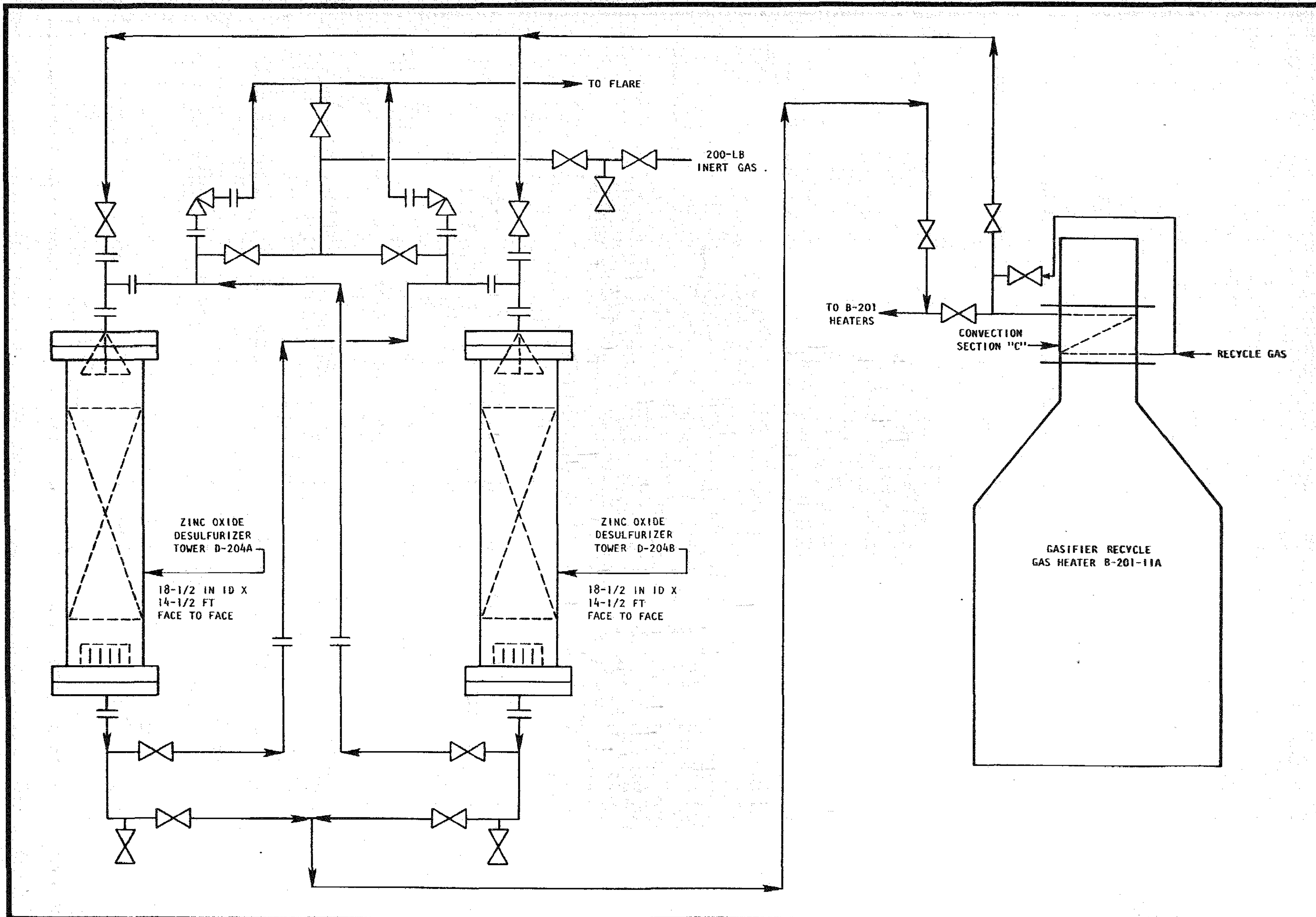


Figure 4-27. ZINC OXIDE DESULFURIZER SYSTEM

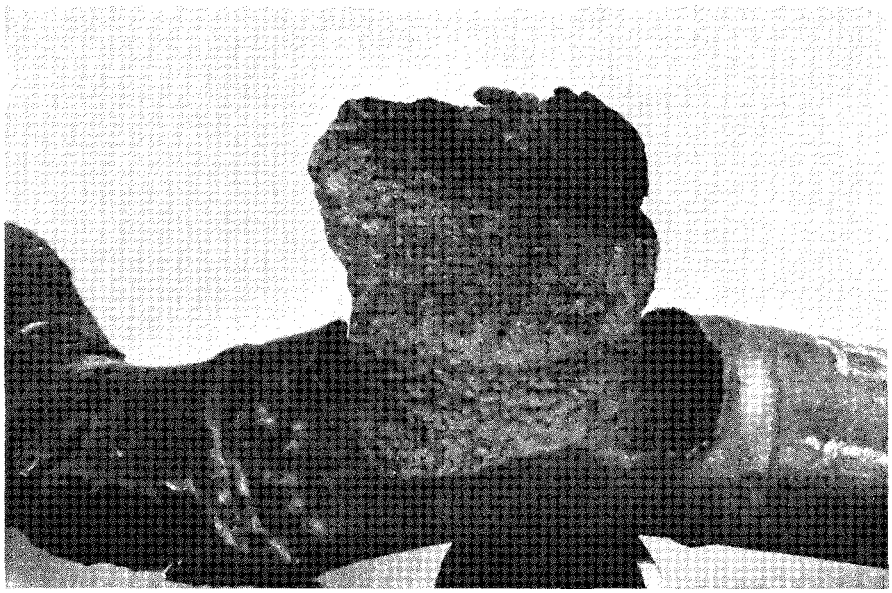


Figure 4-28. B-201-IIIA, GASIFIER BOOT CONVECTION HEATER, MARCH 7, 1974, SHOWING THE THINNING AND PITTING FROM CARBURIZATION

Figure 4-29. HEATER COILS FROM B-201-IIIA
DAMAGED BY CARBURIZATION

(5) Incoloy 800 does not appear to have adequate resistance to oxidation in the environment for this application. A higher chromium alloy which can form a more protective oxide film to retard the diffusion of carbon and sulfur to the underlying metal may give better performance. Additions of aluminum, titanium, and silicon also would be helpful.

(6) Oxidation-resistant coatings could be applied to prevent or slow down these reactions if an alloy having adequate resistance to the environment could not be found.

In mid-1974, a decision was made that the most expedient solution to the carburization problem was the formation and maintenance of an impervious film on the tube surface. Two methods were suggested: either sulfiding (by re-adding up to 50 PPM of sulfur), or by oxidizing. A decision was made to carry out oxidation by steam addition, since steam is required for the gasification reaction.

The coil in B-201-IA, gasifier boot Heater-I, was sampled three more times for analysis by Lockheed. A new coil was installed in December, 1973. One sample was removed in May, 1974, the second in July, 1974, and the third in October, 1974. The coil had been subjected to a carburizing atmosphere from January, 1974, through May, 1974, at which time the first sample was removed. The second and third samples were removed after oxidizing procedures with steam were established.

Conclusions reached by Lockheed based on these samples were as follows:

(1) Preexisting pits did not grow in size, and new pits did not form during a 510-hour exposure of Incoloy 800 pipe to desulfurized recycle gas containing 30- to 50-percent steam.
(2) There was no significant increase in the amount of internal carburization and sulfidation during this exposure. Protective oxide films formed on both pitted and nonpitted surfaces.
(3) The addition of greater than 30-percent steam to the recycle gas, coupled with little exposure to alternate reducing/oxidizing conditions, appears to be directly responsible for the good resistance to high-temperature pitting (metal dusting).
(4) Incoloy 800 is a suitable material for construction of recycle gas heaters, provided that the gas is sufficiently oxidizing (high H_2O and/or CO_2) to prevent the deposition of free carbon on exposed surfaces at all times.

Only one coil failed after the startup procedure was modified to have an oxidizing atmosphere. This was B-201-IIIB, gasifier side flow convection heater. The reason for failure was not sulfur corrosion or carburization but was thermal fatigue and creep of the Type 321 stainless steel. This occurred after about 12,500 hours of service.

4.3.2 REGENERATOR FIRED HEATERS

There are three fired heaters in the regenerator flue gas circuit. These are shown in Figure 4-30 and are as follows:

(1) B-203 air heater.
(2) B-204 char lift gas heater.
(3) B-205 acceptor lift gas heater.

4.3.2.1 B-203, Air Heater

The first heater in the plant to present any problem was B-203, air heater. In early April, 1973, a small hole developed in the Type 321 stainless steel portion of the coil. This failure was reported as severe oxidation pitting. Inspection also revealed that the Type 321 stainless steel tube outside diameter had increased from 1.66 inches to 1.76 inches due to creep from heat and pressure.

Although this unit was designed as an air heater, frequently various concentrations of recycle flue gas containing CO, CO_2, N_2, and SO_2 were included in the heater feed.

Two kinds of corrosive attack occurred in this heater. At the outlet bends of B-203, where temperatures approach 1500 to 1600°F, the attack was due to SO_2 corrosion. This attack was not nearly as severe as the H_2S attack in the gasifier recycle gas heaters. However, a very severe attack occurred in the inlet sections of B-203 from oxidative pitting of the Type 321 stainless steel that comprised approximately two-thirds of the original tubing. The Type 321 stainless steel sections failed in early November, 1973. (See Figure 4-31.) When attempts were made to repair the tube rupture, the Type 321 stainless steel section was extremely brittle and broke easily. The embrittlement was caused by overheating of the furnace coil due to the combustion of CO in the recycle gas inside the tubes. Severe oxidation with some carburization occurred, resulting in a brittle metal which could not be repaired.

Replacement of the Type 321 stainless steel section with the B-207-IIA Incoloy 800 coil was undertaken at the same time new coils were being installed in the B-201-IA and B-201-IB furnaces. After installation of the B-207-IIA coil, the heater operated satisfactorily except for an initial plug which was due to acceptor that had been left in the tubes. The rupture of the Type 321 stainless steel section had caused a backflow of acceptor into the heater from the regenerator.

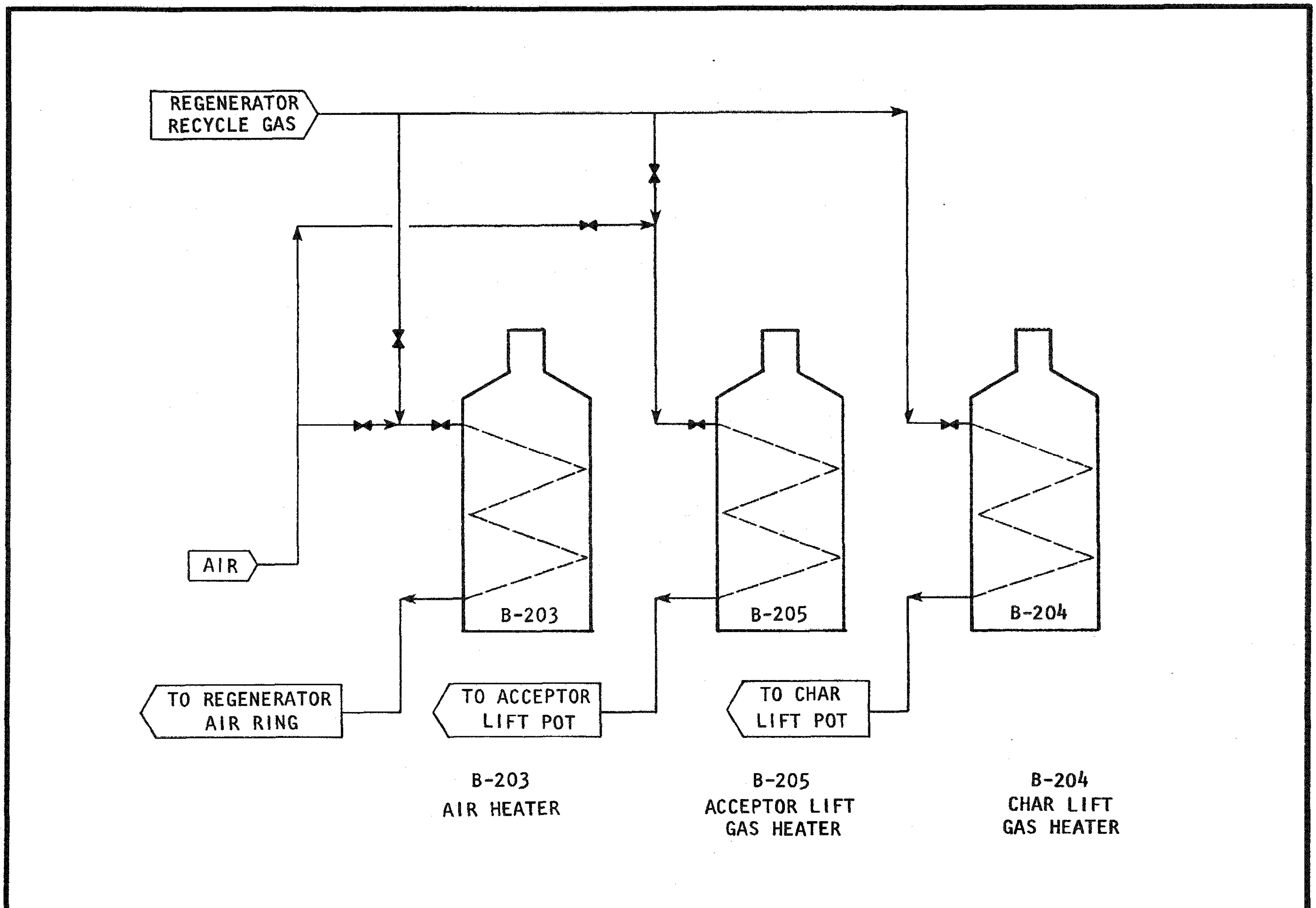


Figure 4-30. REGENERATOR RECYCLE GAS HEATERS

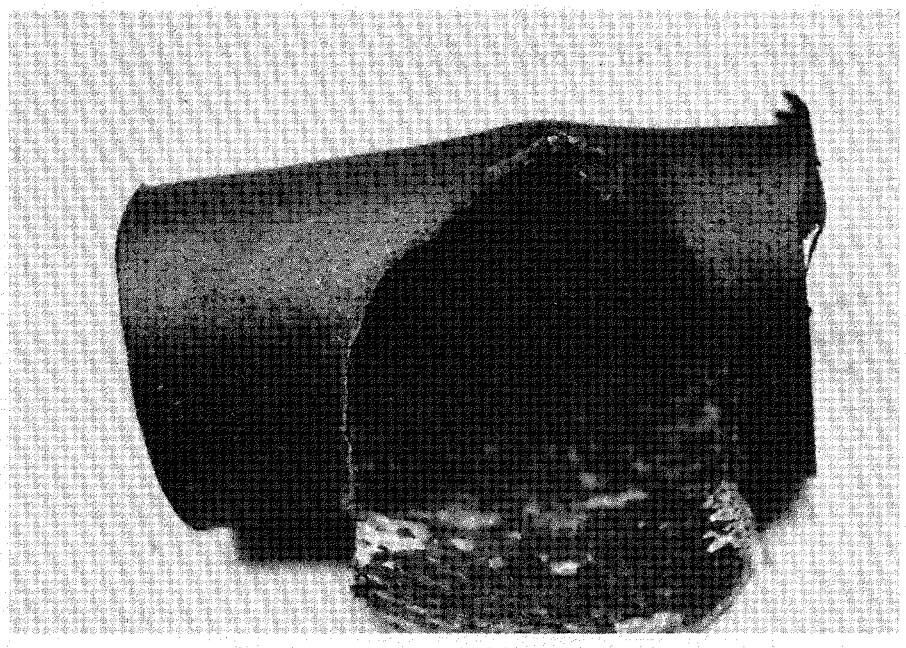


Figure 4-31. 321 STAINLESS STEEL TUBE FROM B-203, AIR HEATER, NOVEMBER 11, 1973, AFTER RUPTURE WHICH TERMINATED RUN 14B

In the original plant design, a hot potassium carbonate system was installed to remove and recover carbon dioxide from the regenerator flue for use as purge gas. This operation was not instituted, because of a process requirement for all the available recycle gas. However, once the sulfur corrosion was discovered in the B-203 heater, the SO_2 scrubber was activated using a dilute potassium carbonate solution to remove sulfur from the regenerator flue gas.

From January, 1974, until the end of scheduled operations, no further significant problems were encountered with this heater.

4.3.2.2 B-204, Char Lift Gas Heater

The original coil was still in service at the termination of plant operations. No repair was ever necessary to the tubes in this heater.

4.3.2.3 B-205, Acceptor Lift Gas Heater

This heater coil was a continuing problem throughout the program. A total of six different coils were used from initial operation until the final run.

As in the case of the B-203 air heater, sulfidation and carburization were both responsible for corrosion in the B-205 heater. The sulfur attack resulted from sulfur components in the recycle flue gas during runs when the SO_2 scrubber was not in operation.

The original coil was removed in May, 1974, following a tube rupture (see Figure 4-32) which terminated Run 18. Figures 4-33 and 4-34 are photographs of two different tubes that were analyzed. The metallurgical analysis reported severe pitting with sulfidation and oxidation.

Figures 4-35 and 4-36 are photographs of restricted tubes removed from the Beta coil used in the B-205 heater. This coil was removed because of three tubes being restricted, causing an extremely high pressure drop across the heater. The scale on the tube inside diameter is a mixture of iron oxides, chromium oxides, nickel sulfide, and chromium sulfide. The scale is similar in composition to that on the tubes from the Alpha coil in May, 1974. The metal below the scale was carburized, with 50- to 80-percent of the original wall thickness left. The method of attack on this coil was sulfidation followed by oxidation.

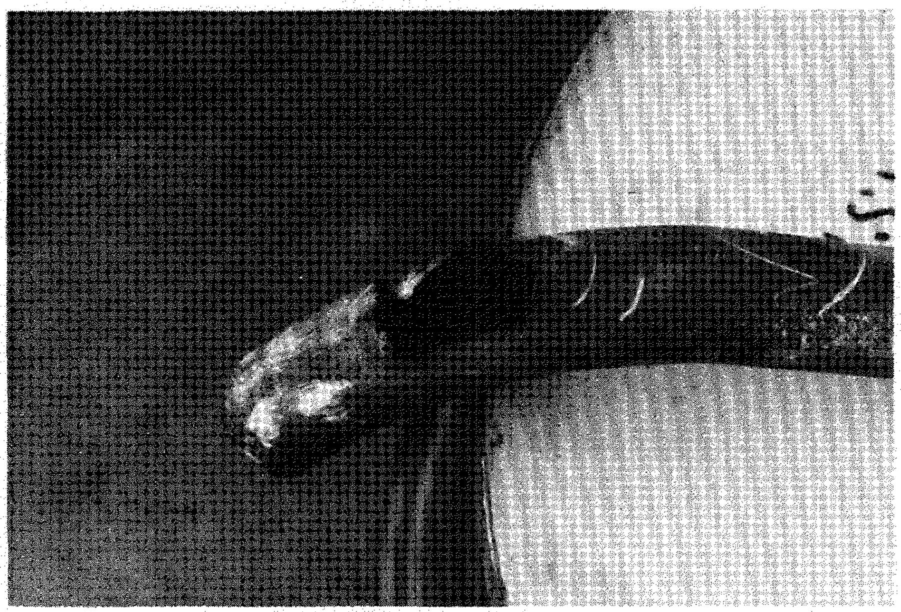
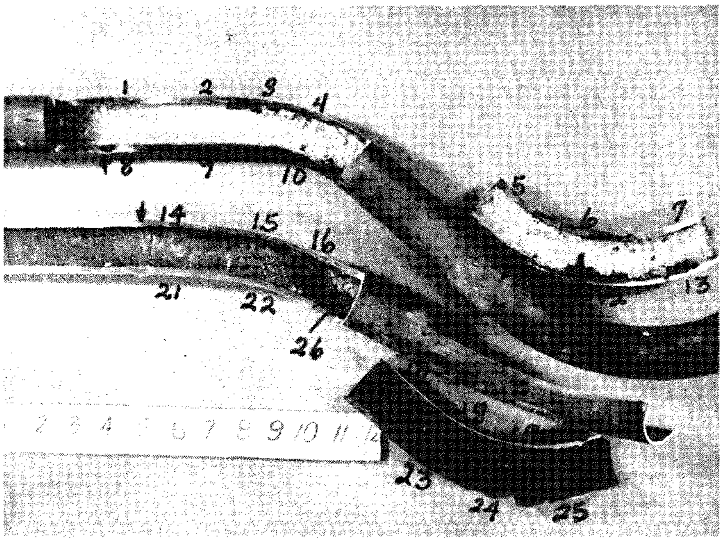


Figure 4-32. FAILURE OF ORIGINAL ALPHA COIL IN B-205, ACCEPTOR LIFT HEATER, APRIL 30, 1974



WALL THICKNESS			
Location	Thickness (inches)	Location	Thickness (inches)
1	0.200	14	0.200
2	0.200	15	0.150
3	0.105	16	0.150
4	0.105	17	0.135
5	0.210	18	0.090
6	0.130	19	0.075
7	0.185	20	0.070
8	0.215	21	0.210
9	0.215	22	0.180
10	0.160	23	0.080
11	0.065	24	0.040
12	0.070	25	0.020
13	0.080	26	0.050

Figure 4-33. TWO TUBES FROM B-205, ACCEPTOR LIFT GAS HEATER, ORIGINAL COIL

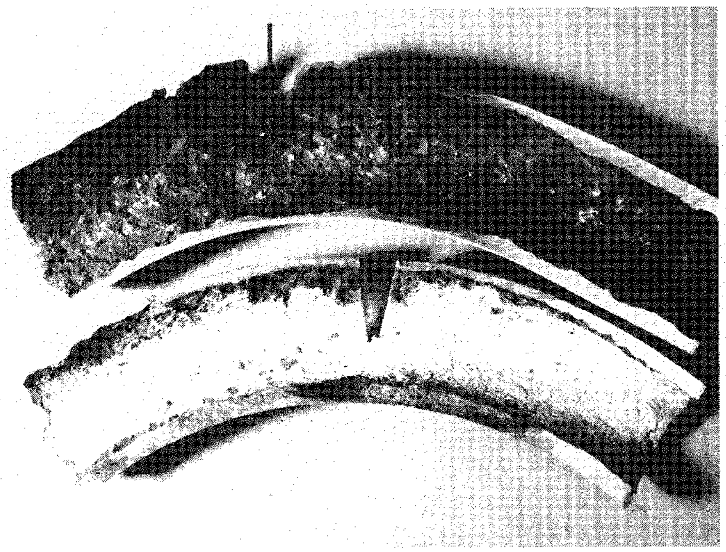
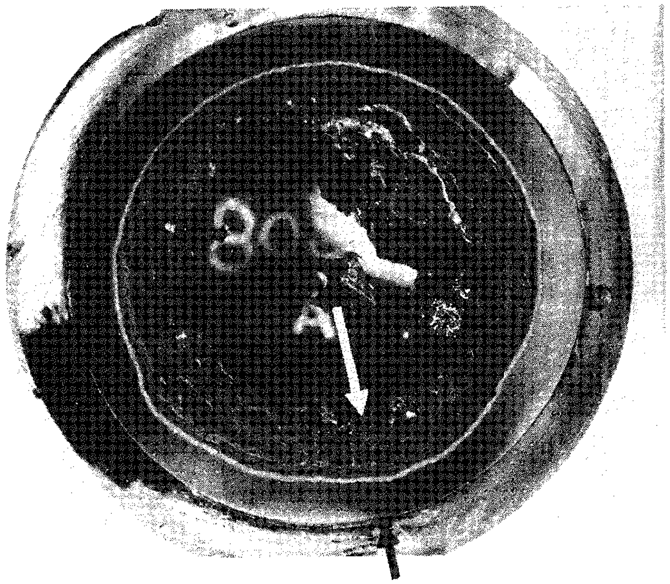
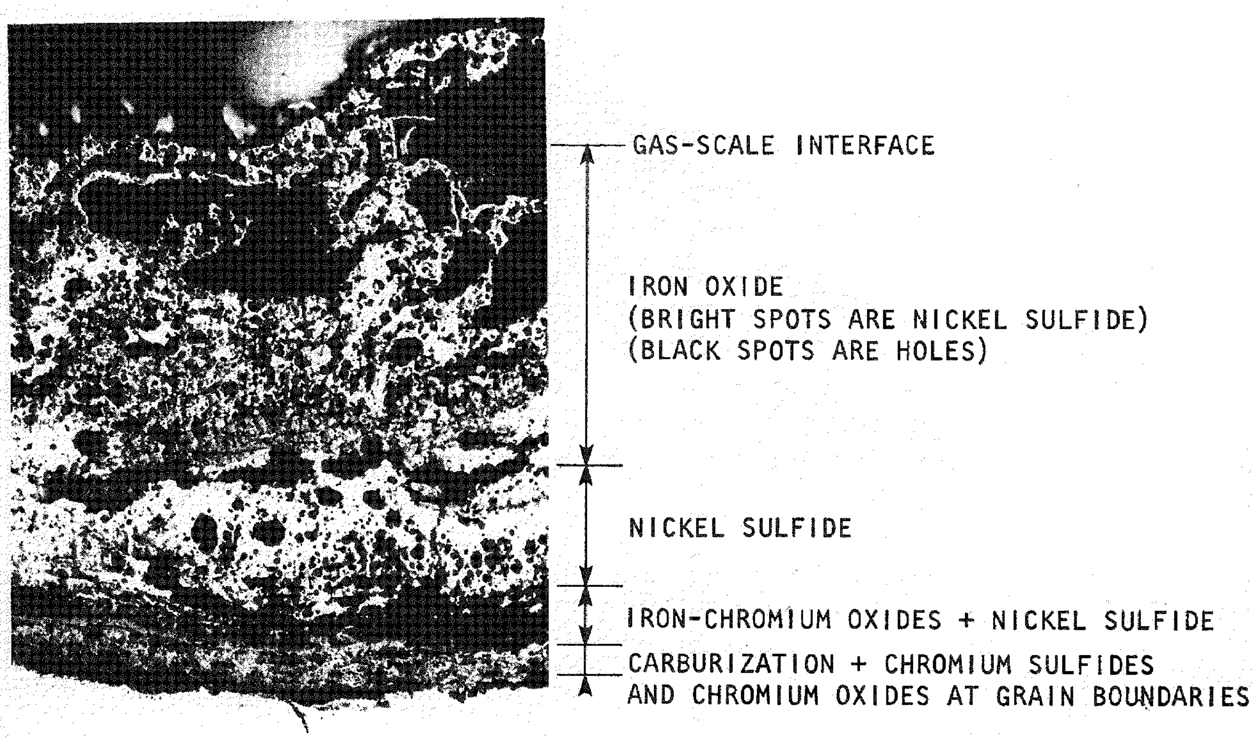


Figure 4-34. TWO TUBES FROM B-205, ACCEPTOR LIFT GAS HEATER, ORIGINAL COIL



THE ARROWS SHOW THE APPROXIMATE LOCATION OF THE MICROPROBE ANALYSES PICTURED IN FIGURE 4-36.

Figure 4-35. TYPICAL CROSS SECTION OF RESTRICTED TUBE FROM B-205 HEATER BETA COIL



BECAUSE OF ITS HIGHER REFLECTIVITY, THE PYRITE-COLORED PART OF THE SAMPLE LOOKS WHITE IN THIS PICTURE.

Figure 4-36. PHOTOGRAPH OF SCALE IN TUBE FROM B-205 HEATER BETA COIL (MAGNIFICATION 14.2X)

In October, 1975, a new B-205 coil fabricated of Inconel 702 alloy was installed. The Inconel 702 was preoxidized for about 4 hours using steam and for 16 hours with air at 1400°F to 1450°F to form a protective oxide scale on the interior of the tubes. When the coil was placed in service, three tubes failed because of holes near the outlets after 48 hours of service heating inert gas (87 percent N_2, 12 percent CO_2, 0.2 percent O_2) and 13-1/2 hours of service heating recycle flue gas from char combustion (80 percent N_2, 17 percent CO_2, 3 percent CO and COS in the PPM range). (See Figure 4-37.)

Metallurgical examination and X-ray microprobe studies of selected areas show the attack to be primarily from sulfur, as many large chromium sulfide precipitates were present at the inside surface. (See Figure 4-38.) In some cases, smaller nickel sufides were also detected.

Inconel 702 was selected for this service because of its aluminum content (3 percent). Inconel 702 and other high-nickel alloys are not very resistant to sulfur attack in high-temperature sulfur environments, so the fact that the tubes were attacked by sulfur is not particularly surprising. However, it is surprising for this amount of attack to occur in the relatively low-sulfur environment.

The last failure of B-205 coil occurred in February, 1977. Examination of the failure determined the cause as polythionic acid attack of sensitized tube material near the heater inlet.

Scanning electron microscope observation confirmed that the fracture was a grain boundary failure. Polythionic acid cracking usually occurs during downtime when moisture or moist air comes in contact with a sulfide scale. The cracking occurs only in sensitized materials and is always a grain boundary type of cracking.

Sensitization means that chromium has combined with something, usually carbon from the metal, to form grain boundary precipitates and has left a thin chromium-depleted (sensitized) zone adjacent to the grain boundaries. Because of the loss of chromium, these thin zones are much less corrosion-resistant than the parent metal. Sensitization occurs when the metal temperature is held at 800°F to 1400°F. Sensitization can usually be prevented by adding titanium, tantalum, or niobium to the alloy, or by restricting the carbon availability.

Incoloy 801, which is a higher titanium version of Incoloy 800, is more resistant to sensitization grain boundary attack and ploythionic acid cracking. Presumably, improved heater life could be obtained using this material.

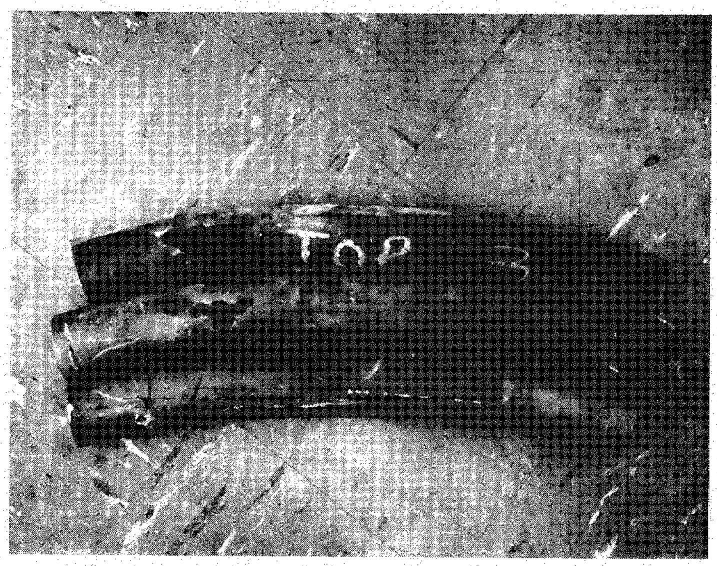


Figure 4-37. FAILED TUBES FROM B-205 HEATER, GAMMA COIL, FABRICATED OF INCONEL 702

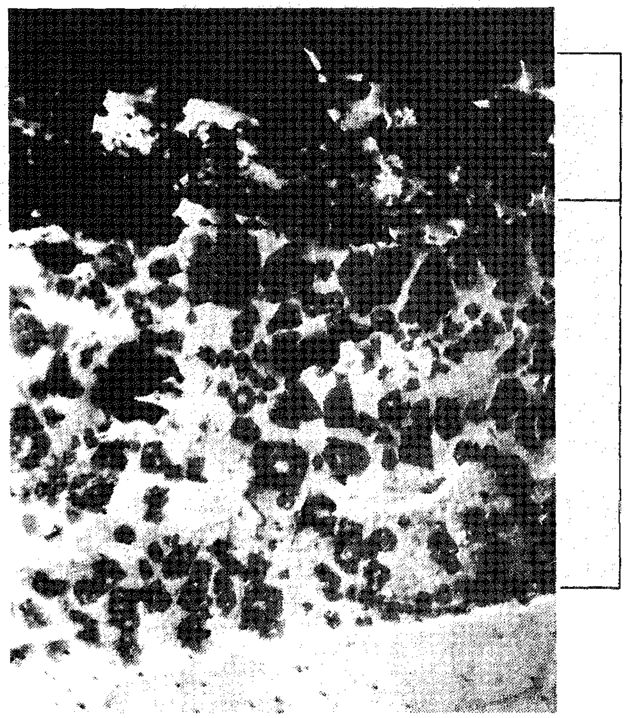


Figure 4-38. PHOTOMICROGRAPH OF INSIDE SURFACE OF FAILED TUBE FROM B-205 HEATER, GAMMA COIL, INCONEL 702 (MAGNIFICATION 75X)

After the February failure, the B-205 coil was purged with dry inert gas, upon shutdown, to remove the moist recycle flue gas when idle to prevent polythionic acid cracking in the new coil which was fabricated from alonized RA-330.

Since steam would be a detriment to the regenerator operation, it was never tried as an oxidizing agent in this coil. Air or oxygen could not be utilized for fear of burning residual char in the acceptor lift line, which had a metal interliner. However, two major changes were made in the pilot plant. The acceptor lift line was changed to a hard refractory line in the fall of 1976, and the regenerator was reduced in diameter by fire brick addition. These changes allowed the unit to be operated with air in the lift line and, therefore, air was present in the B-205 coil from January, 1977, until operation ceased. At the time of termination of plant operation, no problems had been encountered with the latest heater coil.

4.4 VALVES

Numerous problems with valve installations arose during the 5-1/2 years of pilot plant operation. The problems were generated by the severe combination of process conditions involving the handling of solids and corrosive gases at high temperatures and pressures. The more serious problems and their solutions are reviewed in this section. Primary emphasis is placed upon problems which will arise in commercial operations; problems which are unique to the Rapid City Pilot operation have been deemphasized.

Valve problems are grouped into two categories: areas of cyclic operation, such as pressurizing and depressuring of lockhoppers, sample containers, etc., and areas of continuous controls, such as pressure control, flow control, and level control.

4.4.1 REGENERATOR PRESSURE CONTROL SYSTEM

In the regenerator pressure control system (see Figure 4-39), the pressure on the regenerator is maintained at a few psi less than the gasifier under normal operation. The range is from 2 to 8 psi, depending upon the mode of operation and the point in time of startup.

The gasifier pressure is maintained by direct pressure control, with the pressure of the regenerator controlled by a dPRC. The control valve is located several vessels downstream of the regenerator to permit operation of the gas cleanup system at elevated pressure. The original valve used in the system was a Fisher 1-inch 657-ED, equal percentage, single port, cage stem and plug. Even though the gases passed through a cyclone and two scrubbers, the stream contained enough solids that the stem would "freeze" in one position.

A V-ball valve was substituted for the plug valve; no further problems were encountered with the valve or the control system. The model used was a 1-1/2-inch 657-8V ball constructed with a Type 316 stainless steel body, ball, and bushing.

4.4.2 REGENERATOR AND GASIFIER SEAL LOOPS

There are four locations (see Figure 4-40) which require handling of hot solids: (1) the calcined acceptor flowing from the regenerator to the gasifier, (2) the recarbonated acceptor flowing from the gasifier to pickup pot, (3) the char flowing from the gasifier to its pickup point, and (4) the purged and rejected acceptor from the regenerator.

The solids flow by gravity (see Figure 4-41) from their respective vessels and the pressure balance around the reactor system is maintained by purge gas.

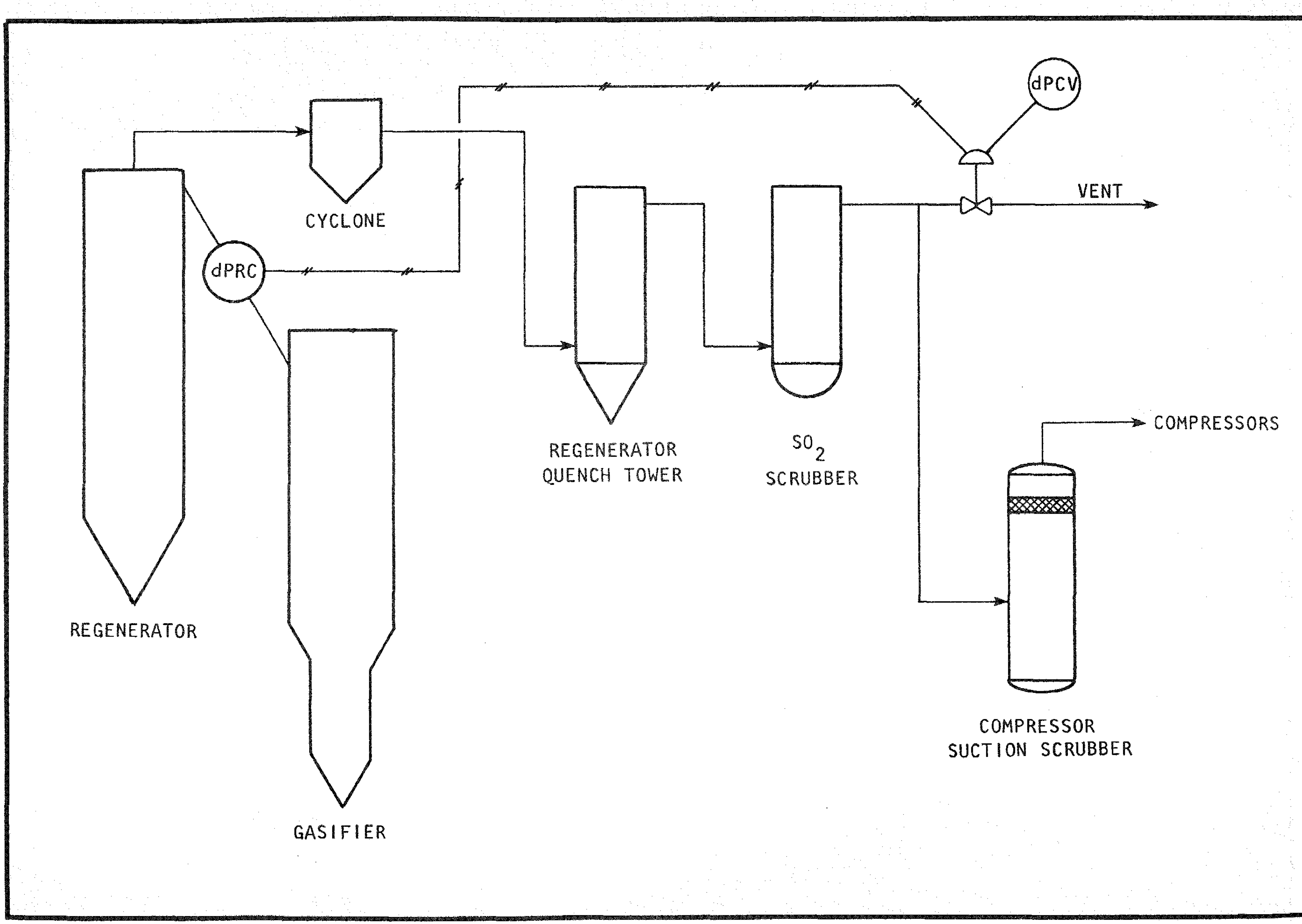


Figure 4-39. REGENERATOR PRESSURE CONTROL SYSTEM

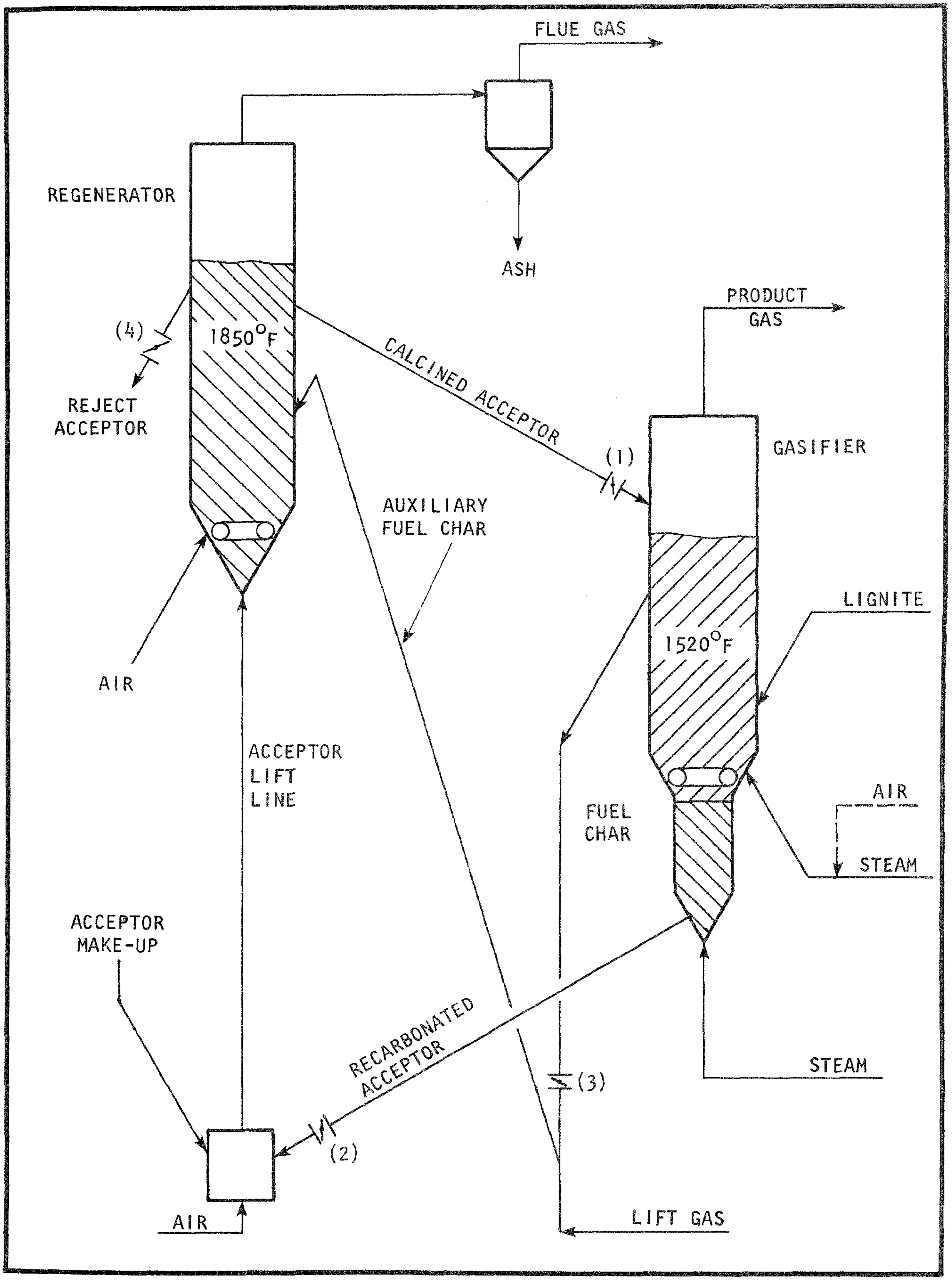


Figure 4-40. CO_2 ACCEPTOR PROCESS DIAGRAM

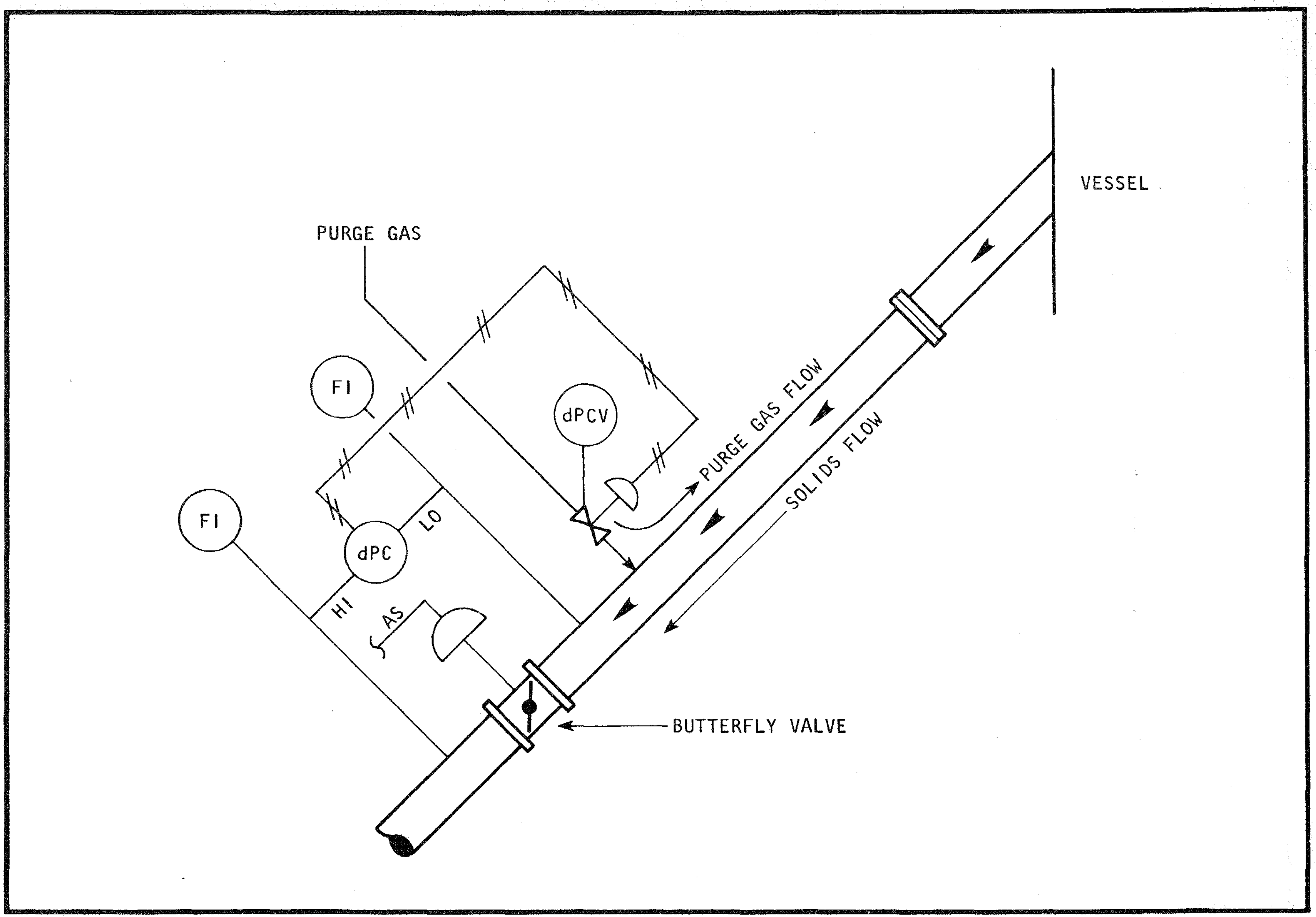


Figure 4-41. SOLIDS TRANSFER LINE

The flow of solids is controlled by a butterfly valve (see Figure 4-42) which was designed by Dally Engineering Company of Pittsburgh. The positions of these valves are controlled by either a level controller or flow controller. Several sizes are used in the Rapid City plant; for example:

(1) 14-inch nominal pipe size with a 5-inch butterfly.
(2) 10-inch nominal pipe size with a 5-inch butterfly.
(3) 8-inch nominal pipe size with a 3-inch butterfly.
(4) 12-inch nominal pipe size with a 3-inch butterfly.

The valve bodies are of carbon steel. An Incoloy 310-SST sleeve is used to contain the process solids. Castable refractory is used to insulate the inner sleeve from the valve body. The shaft is Alloy 25 in all cases.

Some of the original Alloy 25 butterfly disks were replaced with disks fabricated from 310-SST plate when the size was increased for more solids flow. The valves are operated with a double-acting air-operated cylinder with a Moore H/FR positioner.

In general, these valves functioned well. Occasional problems which were resolved are:

(1) Shafts breaking: Originally one valve was 2-inch. The shaft was counter-sunk to hold the disk and twisted into the notch. This was corrected using a non-sunk shaft and notching the disk to fit the shaft.
(2) Disks breaking off: The original design was to pin the disk to the shaft. Repair was to weld the disk to the shaft in addition to using the pins.
(3) Sleeve becoming egg-shaped which caused the disk to bind: The original sleeves were replaced with heavy-walled material. Also the disk was ground to a 30- to 40-thousandth clearance between disk and sleeve.
(4) Shaft and stuffing box would become plugged with solids: The stuffing box was modified to allow better purging along the shaft to keep solids out.

This valve is a satisfactory item of equipment for commercial use.

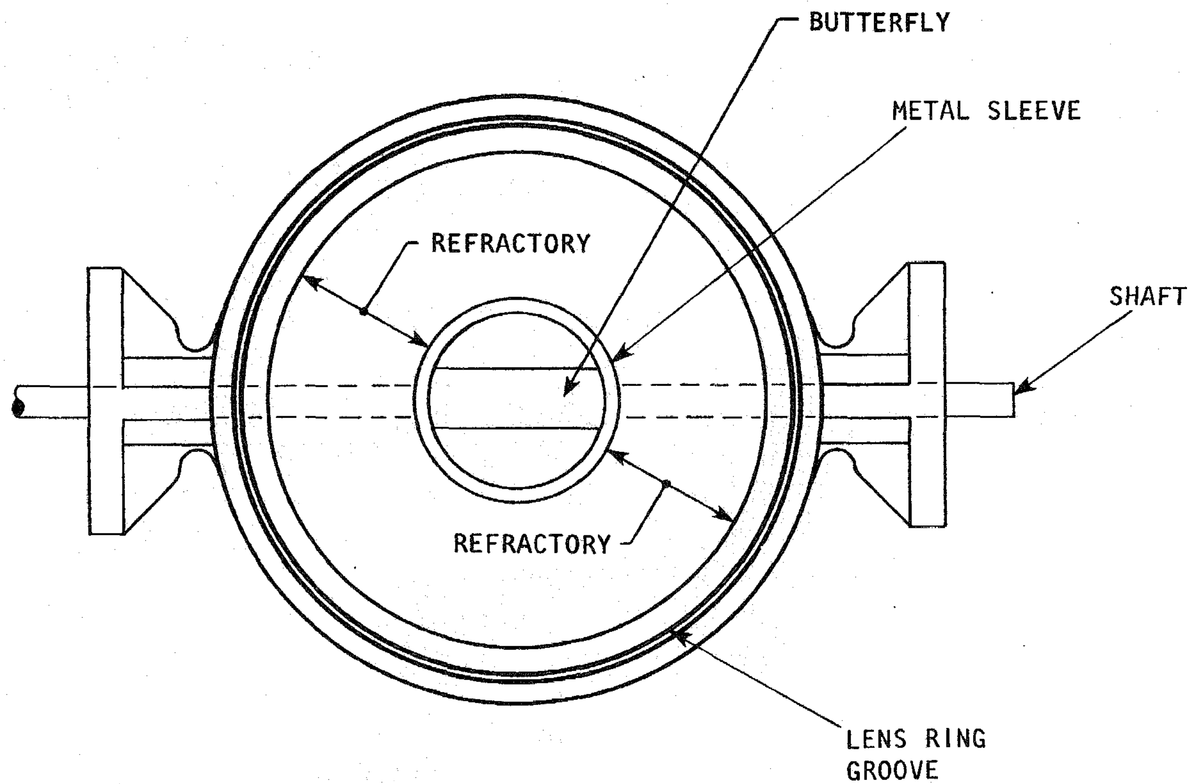


Figure 4-42. SOLID CONTROL BUTTERFLY VALVE

4.4.3 LIGNITE LOCKHOPPER VALVE SYSTEM

The lignite lockhopper valve system (see Figure 4-43) is used to feed lignite to the gasifier. Lignite enters through the upper ball valve at atmospheric pressure and 500°F. When the weigh cells (WR) indicate the required weight, the upper valve closes along with the ball valve in the vent line. Next, the vessel is pressured to system pressure (150 psig) using dried inert gas. Once the vessel is at pressure, the XCV ball valve below the feeder opens, placing the lockhopper "on line." After the hopper weigh cells indicate a low level, the feeder is stopped. A time delay of about 30 seconds is completed before the lower ball closes. This allows lignite to clear the valve prior to closing. Originally, the valve closed when the feeder stopped. This resulted in the ball turning through solids, forcing lignite between the ball and seat.

The original valves employed a stainless ball and seal ring with ratchets to rotate the seal ring. The valve presented two problems: The ratchet seized and sheared the dog which turned the ring, and the lignite scratched the ball or seal ring which started a small leak. Further cycles increased the leak and erosion deepened the grooves until repair was necessary.

These valves were replaced with ones utilizing stainless balls and soft seats. The soft seats (reinforced Teflon) are capable of absorbing the coal particles rather than being scratched. The normal life is three to four months before replacement. A manual valve is located below the XCV, in case the XCV develops a severe leak during a run. The seats and balls can be replaced in 10 minutes, thus preventing a run shutdown.

A technique developed at the pilot plant was to fill the cavity behind the ball with a silicone rubber compound to prevent lignite from working into this area. This has been utilized on all ball valves in solids services.

4.4.4 ASH LOCKHOPPER SYSTEM

The ash lockhopper system (see Figure 4-44) is quite similar to the lignite system. Here the solids are elutriated ash and acceptor fines from the regenerator at 800°F. Several valves were tried in this system before a successful combination was designed.

The original system was parallel lockhoppers below one cyclone. A flapper diverter valve switched the solids from one side to the other, with a conventional ball valve above each hopper for isolation. Erosion and leakage occurred in the valves, which prevented depressuring of the hoppers.

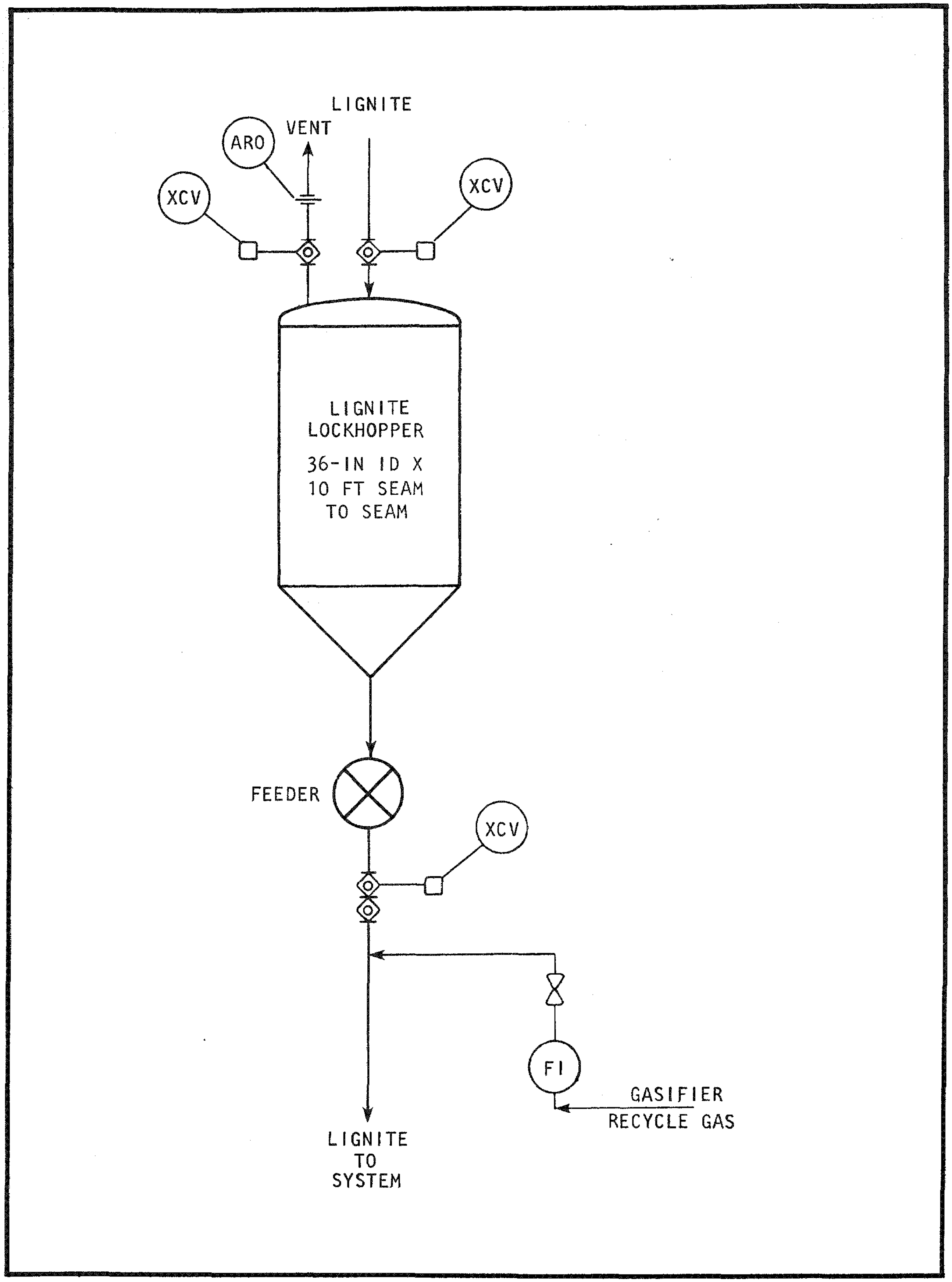


Figure 4-43. LIGNITE LOCKHOPPER VALVE SYSTEM

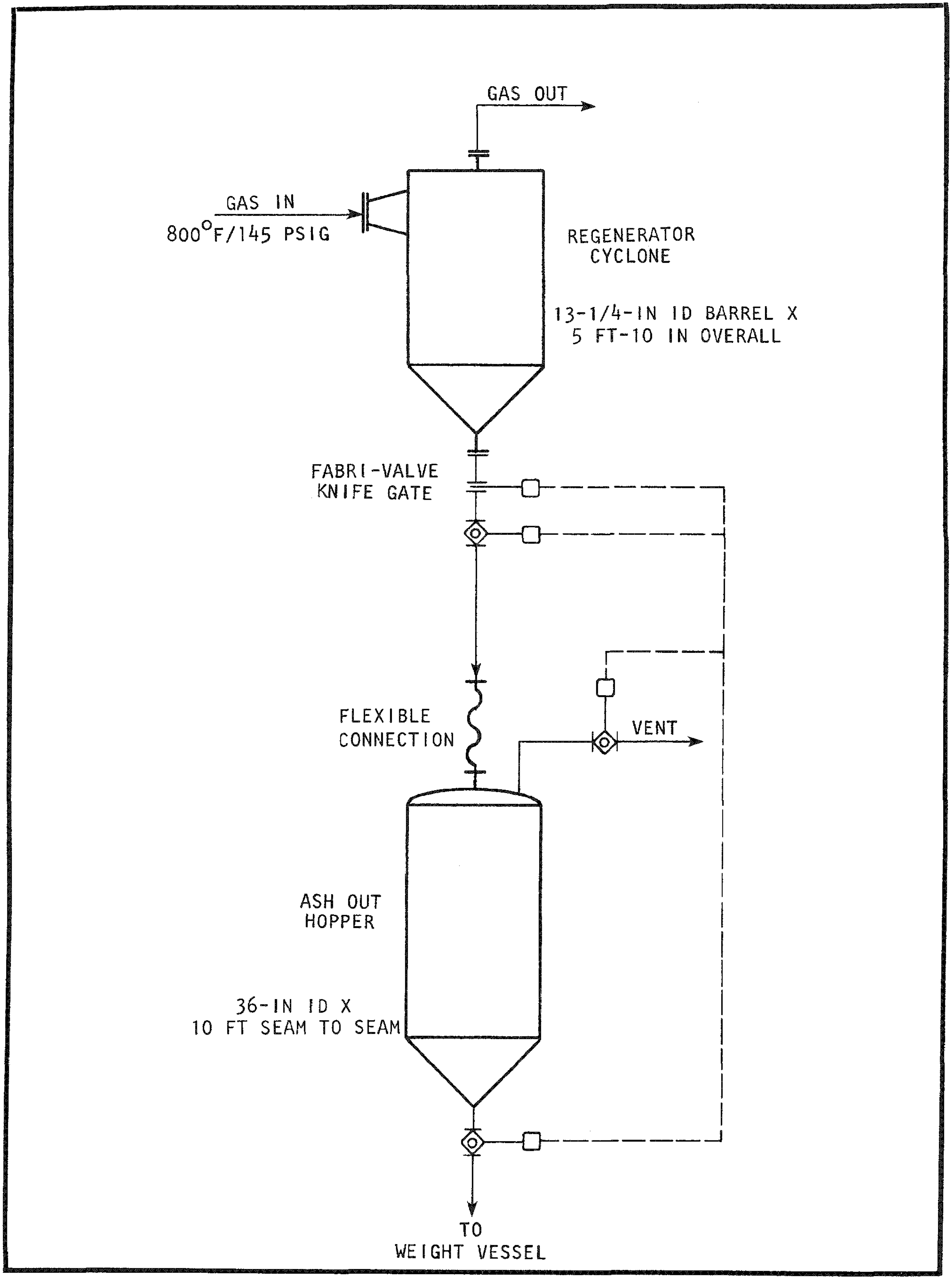


Figure 4-44. ASH LOCKHOPPER SYSTEM

Another system tried was two Posi-seal valves in series below each hopper. A Posi-seal is a form of a butterfly valve with the wafer machined into a mated seat. Again, scratches promoted erosion and leakage.

The successful combination utilized a knife gate to stop the solids flow. After a preset time delay, the ball valve automatically closes. Once the ball valve is closed, the vent ball valve opens to depressure the hopper. After the hopper is low in pressure (1 psig) the lower ball valve opens to dump the contents into a weight hopper. During this operation, a small purge is admitted above the knife gate to prevent the fine ash and acceptor from bridging.

The valve materials in this case are a 316 SST body and ball with a graphitar seat. Reinforced Teflon is not accpetable because of the temperature of the solids.

When the hopper is repressured, the upper ball valve opens before the knife gate is opened. In this way, the ball valve should never open or close on solids.

4.4.5 GASIFIER QUENCH SYSTEM

The gasifier quench tower (see Figure 4-45) was designed to cool gasifier product gas and scrub char particles from the gas stream.

The original level control valve was a Fisher 1-inch 667 BFC equal percentage, three-fluted valve. The control valve was located in a conventional horizontal station with bypass leaving a horizontal separator. This system was totally unoperable. The system was redesigned to convert the vertical tower into a scrubber separator. In addition, the control valve station was located in a 45-degree sloped line and converted to a Fisher 1-inch "U" V-ball 657-8-U valve. The seats and shafts of the valve are Type 316 stainless steel, and the ball surface is Fisher's Alloy 6 (Cobalt-based Stellite) with a V notch. This valve performed well in this service with normal replacement of the sealing ring.

A similar system was employed to quench flue gases from the regenerator; however, flue gas quench will not be utilized in a commercial plant design.

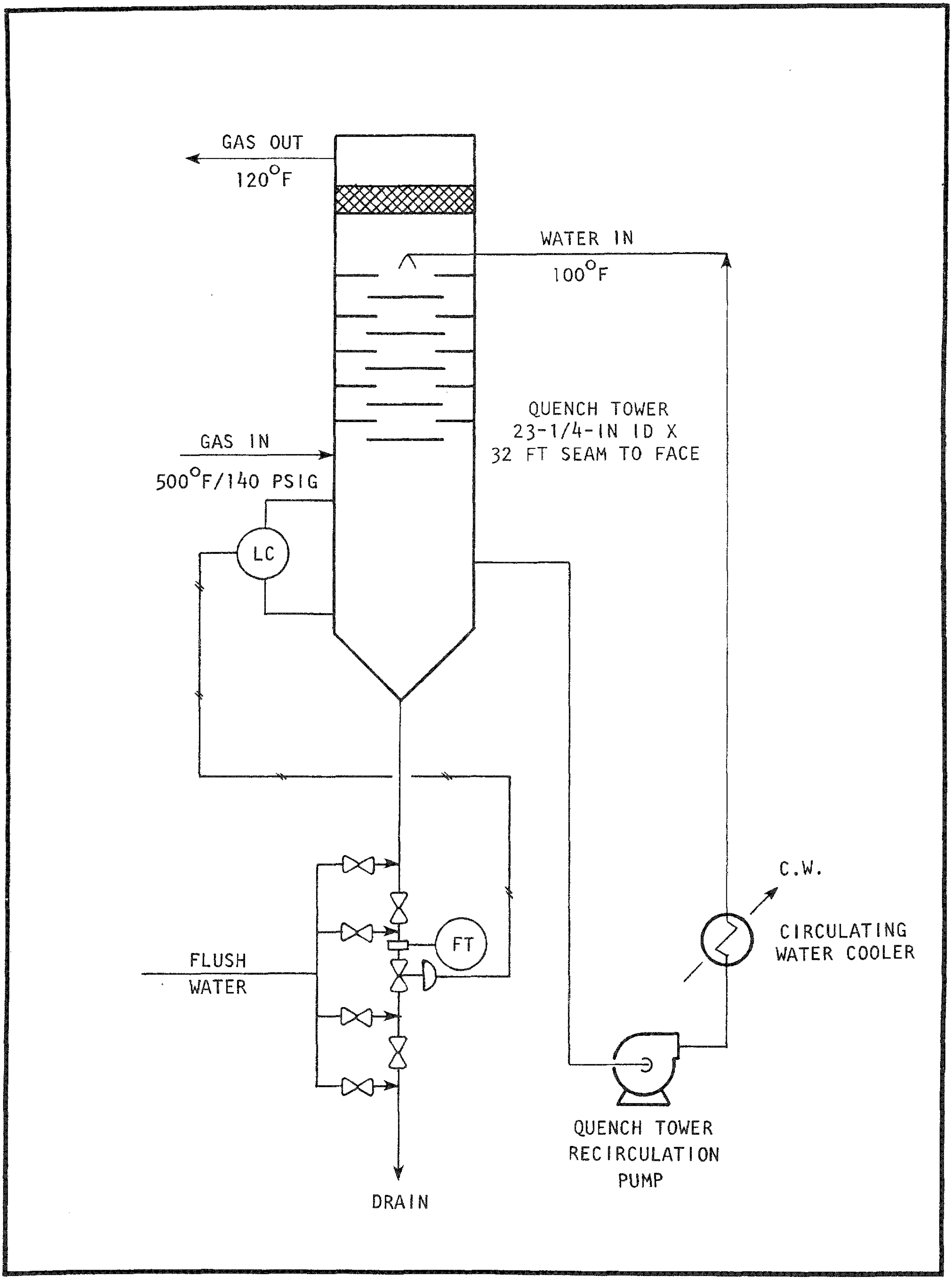


Figure 4-45. GASIFIER QUENCH SYSTEM

4.5 VESSEL INTERNALS

4.5.1 THERMOWELLS AND PRESSURE PROBES

4.5.1.1 Regenerator

The erosion of the thermowells in the regenerator was a serious problem in early plant operation and continued to be a problem throughout the entire life of the unit.

The original vertical temperature and pressure probes did not hold up to the forces of the fluidized bed and were replaced with side taps. The first side-entering taps into the regenerator were Type 316 stainless steel. They were enlarged and changed to Type 310 stainless steel to accommodate the 1/8-inch, Schedule-40, Type 310 stainless thermowells.

The tips of the thermowells continued to fail due to both erosion and corrosion. In an attempt to find a suitable material for this service, a test program was initiated. During the shorter, earlier runs the problem had not been so acute. The thermowells were simply changed out between runs. As the runs become longer in duration, the wells would fail while in service.

The test program included the use of plasma-spray-coated tips. Chrome-carbide, titanium-carbide, and aluminum-oxide tips were all tried. All failed in a short time. Alonized Type 310 stainless steel tips were tested also. They were not an improvement, so the tips were replaced with off-the-shelf Type 310 stainless steel.

In order to maintain the integrity of the temperature measurement, it was decided that the thermowells should be changed out on a regular basis. From experience as to thermowell life in the regenerator, the thermowells are changed out after five days at operating conditions. History had shown that the thermocouple reading became questionable after about five or six days and the well itself would be leaking badly in seven to nine days.

Figure 4-46 is a Type 310 stainless thermowell/pressure tap assembly in which the thermowell tip material had failed. Figure 4-47 shows alonized thermowell tips which had not leaked yet but were being worn away.

4.5.1.2 Lift Line Thermowells

The lift line temperatures were measured on the outside of the metal liner skin and with a thermowell projecting into the process flow. The thermowells projecting into the process flow were quickly eroded and the temperature point rendered useless.

To prevent the rapid erosion, the thermocouples were retracted so that the very tip of the thermowell reached the inside of the liner wall.

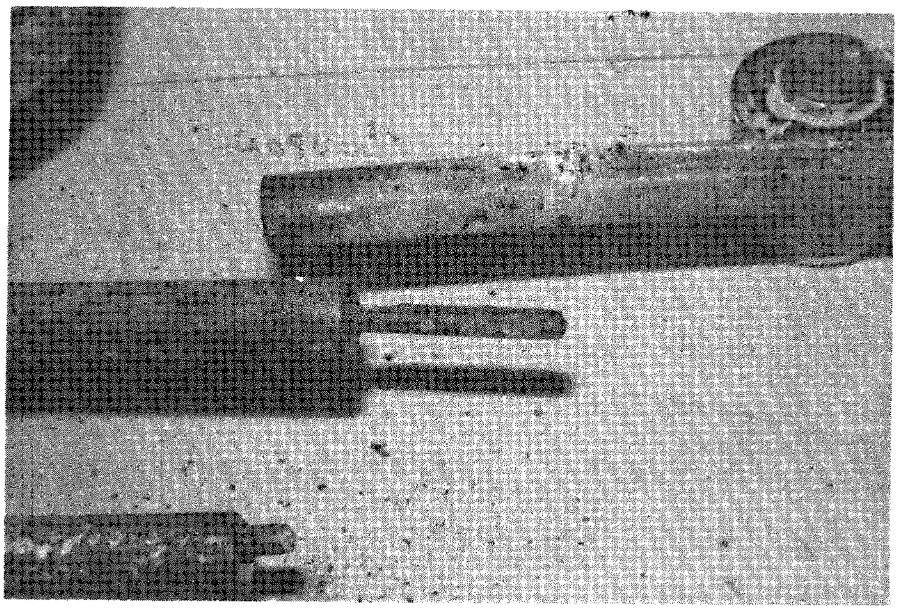


Figure 4-46. FAILED 310 STAINLESS STEEL THERMOWELL TIP

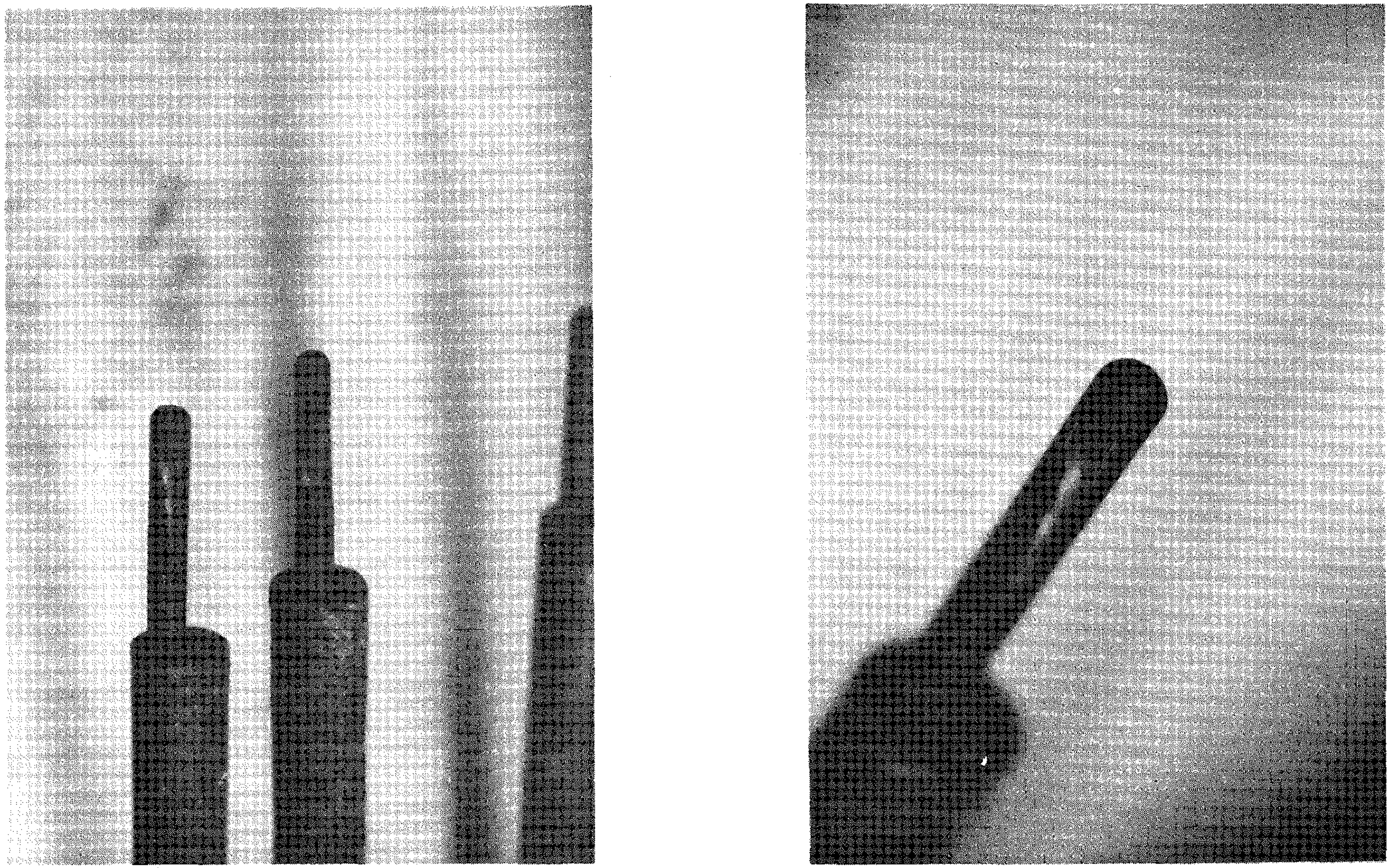


Figure 4-47. WORN ALONIZED THERMOWELL TIPS

This prevented the erosion at the expense of temperature measurement sensitivity.

When the refractory-lined lift line was designed and thermowell assemblies fabricated, the thermowell extended through the insulation and refractory to the inner wall. This also sacrificed temperature measurement sensitivity for the protection of the thermowell itself.

4.5.1.3 Gasifier Thermowells and Pressure Probes

Failure of the vertical gasifier probes, the internal thermowells and pressure sensor tubes (refer to Book 4, Subsection 13.3) was an early problem.

A thorough examination of a pressure probe (Type 310 stainless steel) and sensor tubes (Type 316 stainless steel) revealed that both the probe and tubes had failed as a result of stress-enhanced intergranular sulfur corrosion.[2] Although the alloy types were different, both tubes failed in the same manner.

Since the sulfur corrosion was localized, further tests were performed which verified that sensitization (carbide precipitation and chromium depletion) of the grain boundaries had occurred as a result of extended operation in the 800°-$1600^{\circ}F$ temperature range. This significantly reduced the corrosion resistance at the grain boundaries.

Conversion in the gasifier to the dual-purpose side-entering probes, which had proven to be successful in the regenerator, was initially not possible due to vessel code requirements. After a suitable method was devised for installing new side taps, side-entering Type 310 stainless steel pressure taps and thermowells were used exclusively after pilot plant Run No. 18. During the remaining plant runs, only two of the side-entering pressure taps were found to have cracked. The cracking was highly localized, occurring at about four inches beneath the surface of the refractory through which the probe penetrated or about eight inches from the discharge end of the probe. The probes were not analyzed to determine the cause of failure. However, it is likely that the same mechanisms which caused the failures of the vertical temperature and pressure probes were responsible for the two side-entering probe failures. Unlike in the regenerator, frequent change-out of the thermowells in the gasifier was not necessary as erosion and corrosion were not significant.

---

(2) "Failure of the Gasifier Probe and Sensor Tubes, Consol, Rapid City, South Dakota," Continental Oil Company Technical Service Report No. 116-74-1301, May 13, 1974.

4.5.2 GASIFIER INTERNAL CYCLONE

An internal cyclone is located at the top of the gasifier above the char bed. Entrained fines are separated from the exiting gas stream in the cyclone and return back to the char bed through a dipleg. A flapper valve at the bottom of the dipleg prevented gas flow from the fluidized char bed into the dipleg. The dipleg was supported with metal guides which were anchored into the refractory wall. The original cyclone and dipleg were made of Incoloy 800. Corrosion on the surface of the cyclone was severe and caused considerable pitting.

Prior to Run 6, the original 2-inch diameter dipleg was replaced with a 4-inch diameter dipleg which did not extend as far as the original dipleg into the char bed. The dipleg modifications were made to improve the transfer of captured char fines back into the char bed. Due to the scarcity of available materials, the dipleg was constructed of sections of different metals, including Incoloy 800, RA-330, and Types 310 and 316 stainless steel. This dipleg served for the rest of the pilot plant runs and experienced very little corrosion pitting. Some erosion of the dipleg did occur at the points where the dipleg was held in place by the metal guides. Vibration of the dipleg against the guides accounted for the observed wear.

The cyclone assembly fell from its support into the char bed in Run 20 when a fillet weld on one of the braces failed. The cyclone was re-installed with additional braces employing full penetration welds. Due to the excessive cyclone corrosion, the original cyclone was replaced prior to Run 37B with the cyclone taken from the devolatilizer unit. Though this cyclone was also made of Incoloy 800, surface corrosion was negligible, even though the cyclone was exposed to process conditions for more than 2300 hours.

The digleg broke off at the reducing coupling in Run 40B. The break was due to weld failure and not to dipleg metal corrosion. A full pentration weld was used to repair the fracture and no further problems were encountered.

4.5.3 GAS DISTRIBUTORS

4.5.3.1 Gasifier Boot Distributor

The two significant types of gas distributors that were installed in the gasifier boot did not experience detectable corrosion or erosion. These distributors were the plenum chamber and ring, as discussed in Book 3, Subsection 11.1, and were fabricated from Type 316 stainless steel. A visual inspection of both distributors did not find noticeable evidence of metal attack.

4.5.3.2 Gasifier Side Flow Distributor

Early methods of gas distribution in the gasifier char bed proved to be inadequate. After experimentation, a side flow gas distributor ring was installed. The distributor was fabricated from 4-inch, Schedule 40, Type 316 stainless steel and was in continuous service from February, 1973.

Corrosive attack on the external surface of the ring distributor was not detected visually. Internal corrosion was not expected, since the only gases handled by the ring were inert, a mixture of steam and air, and steam alone.

Repairs were necessary on only one occasion when several of the distributor caps were partially melted. The melting occurred when the first charge of dead-burned dolomite was fed to the gasifier during the early stages of the startup procedure for Run 29A. A small amount of char which had escaped the previous run shutdown clean-out was fed to the gasifier, along with the dead-burned dolomite. Since at the time some air was being circulated through the gasifier, high temperatures were generated at the side flow distributor--causing several caps to be destroyed. The damaged caps were repaired and the distributor was again put into service.

A crack which was apparently due to thermal expansion also occurred at the end-plate which connects the distributor ring to the gasifier nozzle. However, this was not a common occurrence.

4.5.3.3 Regenerator Air Ring

After the success experienced with the gasifier side flow distributor ring, a similar ring was installed in the regenerator to improve gas distribution. The distributor was fabricated from 4-inch, Schedule 40, Type 310 stainless steel.

This distributor was used during the remainder of the project. It was replaced several times, however, as a result of carbon monoxide combustion in the ring. Combustion occurred during plant upsets when air and recycle containing a high level of carbon monoxide were both fed to the ring.

The ring on several occasions also separated from the feed gas inlet pipe weld. This was caused by expansion of the gas pipe which allowed solids to collect between the ring and refractory, followed by contraction during shutdown periods which forced the separation of the ring from the gas pipe at the weld.

No appreciable erosion or corrosion problems were encountered.

4.5.4 CALCINED ACCEPTOR STANDLEG INLET

A metal grate was welded to the inlet of the calcined acceptor standleg, CD-206, to prevent large pieces of spalled refractory from entering the line. This metal grate and approximately the first three feet of the metal inner liner of CD-206 required replacement on several occasions. Both items were Type 310 stainless steel.

Examination revealed that the metal was embrittled in service by carburization and oxidation. Small amounts of sulfide precipitates were also found, but the sulfur attack was not significant.

Although the Type 310 stainless steel performed adequately in the pilot plant service, more corrosion-resistant materials would be desirable.

4.6 EXTERNAL CYCLONES

4.6.1 COAL DRIER CYCLONE

This cyclone separates the ground coal from the circulating gas in the coal preparation area. The ground and dried coal is pneumatically transferred to the top of the main structure and into the cyclone. The coal separates out and the gas is recirculated. The coal handled by the cyclone is a nominal 10 x 65 Tyler mesh with the overall range of 8 x 100 mesh. The cyclone operates at 150 to 200°F and minus 2 to minus 6 inches water column pressure.

Severe erosion occurred on the side of the cyclone, opposite and a third of the way around from the inlet. Less severe erosion occurred in the cone of the cyclone. Steel plating was welded and beveled on the inside lip of the cyclone. This proved to be inadequate as a final solution to the problem.

Anchors were welded on the inside of the cyclone and wire mesh attached to the anchors. Resco AA-22 refractory was then spread over the mesh to form a refractory inner wall surface. It was necessary to cure this refractory with a portable gas-fired heater so it would present an abrasive-resistant surface before being exposed to the circulating gas flow.

The refractory lining resisted further erosion for the duration of plant operation.

4.6.2 GASIFIER EXTERNAL CYCLONE

In December, 1975, prior to Run 31, an external gasifier cyclone was installed. In August, 1977, during Run 47A, a severe leak occurred in the side of the cyclone opposite the inlet. There was thinning of the Type 316 stainless steel metal around the pencil-sized hole. This is shown by the bulging of the metal around the hole in Figure 4-48. Due to the limited further plant operations, a patch was welded around the thinned area and the run continued. Figure 4-49 shows the patch with an arrow indicating the repaired leak location.

Approximate gas flow handled by the cyclone is 90,000 SCFH, with a solids loading of 100 to 200 lb/hr of minus 100-mesh material. The temperature approximates 600°F at a pressure of 150 psig.

4.6.3 REGENERATOR EXTERNAL CYCLONES

The original plant design included one regenerator external cyclone. In November, 1974, a second external cyclone was added in series to the first cyclone.

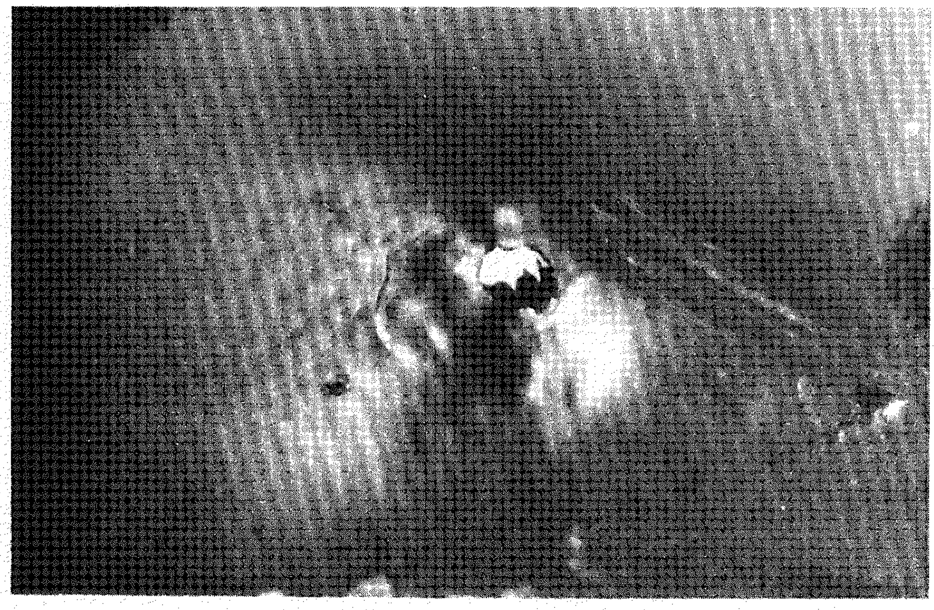


Figure 4-48. THINNED 310 STAINLESS STEEL METAL AROUND HOLE ERODED IN SIDE OF GASIFIER EXTERNAL CYCLONE

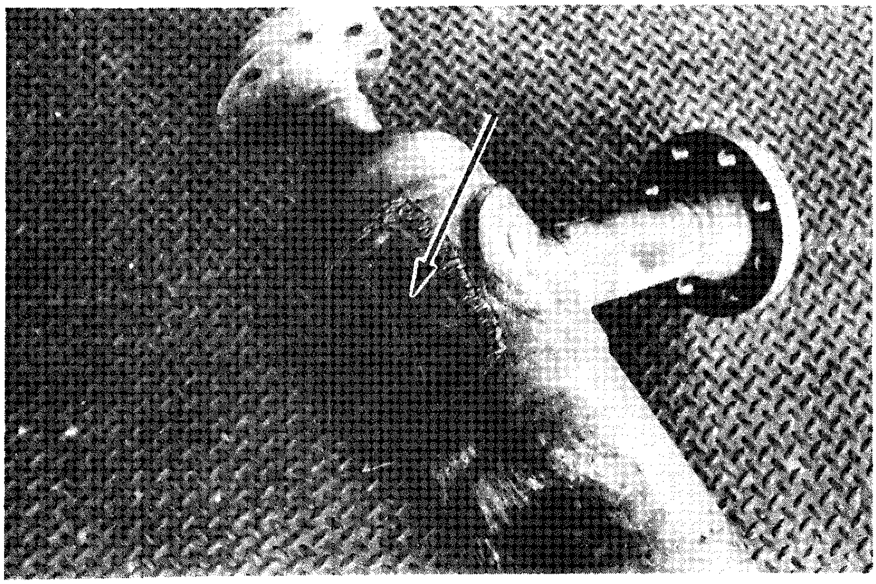


Figure 4-49. PATCHED SIDE OF GASIFIER EXTERNAL CYCLONE (ARROW INDICATES REPAIRED LEAK LOCATION)

Erosion occurred in the barrel of both cyclones, mainly on the wall opposite the inlet. The cones of each cyclone had to be replaced several times due to severe erosion. Stellite 12 hard facing was placed on both the inside and outside of each cyclone when inspection indicated the need.

In October, 1976, the second cyclone developed a leak which could not be repaired and Run 39 was terminated. The cyclones had experienced gas flows of up to 130,000 SCFH and solids loadings of up to 190 lb/hr. The vessels are Type 304 stainless with Type 316 stainless steel flanges. They operate at 800 to 1000^{o}F and 140 to 150 psig.

Following the failure, the damaged parts were replaced and Stellite 12 hard facing applied. Continued maintenance and the addition of the hard facing kept the cyclones in service until the termination of plant operations.

Figure 4-50 shows two views of a replaced cyclone cone.

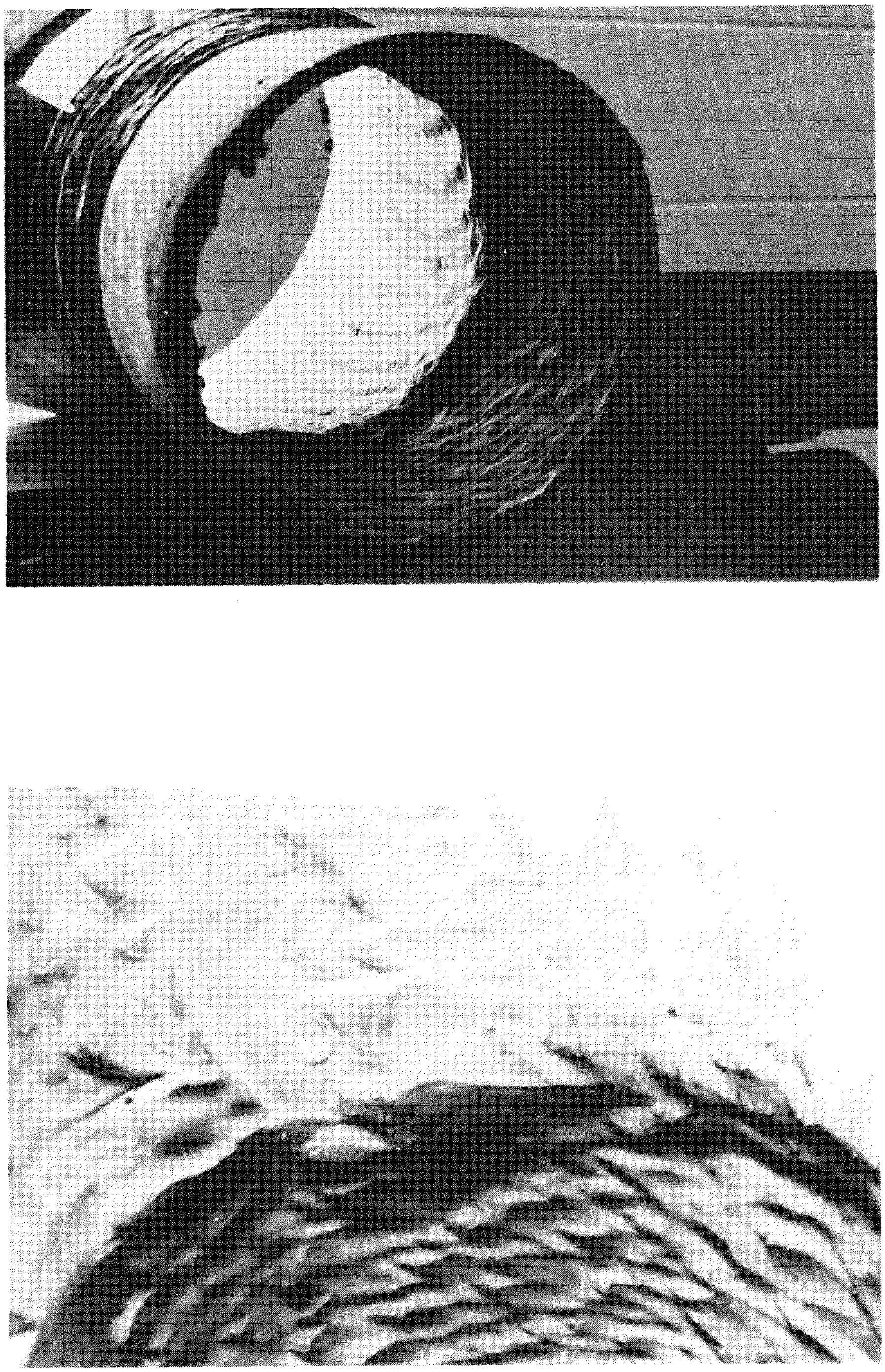


Figure 4-50. TWO VIEWS OF A REPLACED CYCLONE CONE

4.7 ANCILLARY EQUIPMENT

This section summarizes pilot plant problems of a mechanical nature which are not unique to the CO_2 Acceptor Process and for which solutions can be found in industry.

4.7.1 GROUND COAL LIFT LINE

The ground coal lift line extends from ground level in the coal preparation area (100 Area) up to the top of the main structure to the lignite cyclone. The ground and dried coal and char are pneumatically conveyed to the cyclone and the return gas flows from the cyclone back down to ground level.

Both lines are mild steel, 18 inches in diameter with 10-gauge thickness walls. They are insulated and the skin temperatures run 200°F to 240°F.

At the top of the main structure, just upstream of the lignite cyclone inlet, the configuration of the inlet line is as shown in Figure 4-51. The dotted lines on the 90-degree ell show the original construction. This ell eroded away on the top. The ell was repaired several times before the installation of the 18-inch tee.

The tee in this position provides a turbulent area filled with solids which prevents the direct impingement on the metal wall of the solids in the main stream.

The duct below the tee also eroded through the wall at the lesser bend. This was repaired by coating the outside wall with refractory and wrapping for strength.

The line, in general, has eroded and is thin in several places, as well as having holes patched with plate and bands. The tee and refractory repairs were satisfactory in the two most critical areas of erosion. A permanent solution to the problem is the use of an internal refractory lining.

4.7.2 INERT GAS SYSTEM

Corrosion in the high-pressure inert gas system due to nitrogen oxides was quite severe. The nitrogen oxides (NO_x) react with the water of combustion to form nitric acid. The generation of the nitrogen oxides occurs during the methane combustion. Although the highest concentration of nitrogen oxides measured in the product inert gas was about 30 PPM, pH values of less than 4 were found in the condensate. This results from the compressor discharge pressure of 500 psig. Pressure greatly affects the equilibrium of the nitrogen oxide-water system. The corrosive attack was preferentially in the high-pressure system as compared to the low-pressure (15 psig) system.

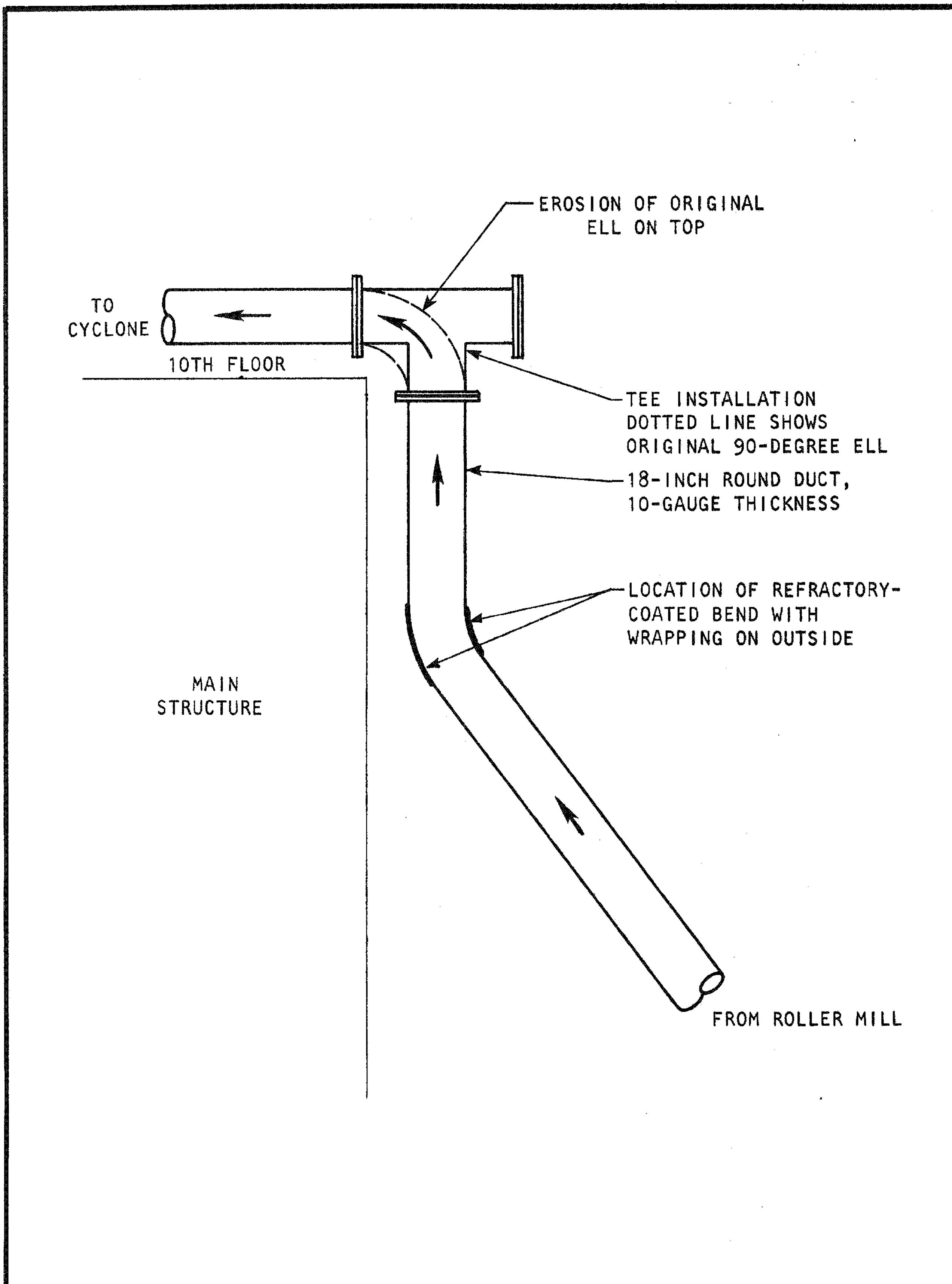


Figure 4-51. TOP PORTION OF GROUND COAL LIFT LINE

Initially, corrosion was observed in the brass parts throughout the system. The brass seats of control valves were corroding very rapidly, and frequent replacement was required. The use of Type 316 stainless steel trim in place of brass eliminated this problem. The high-pressure compressor aftercooler, C-504, with admiralty tubes, had to be retubed with stainless steel.

Starting in October, 1972, leaks developed in the carbon steel piping between the compressor discharge and the driers because of corrosion. Replacement of several sections of piping was required. Initially these were replaced with carbon steel, as it was planned to install a platinum catalytic reduction unit. However, the cost of the catalytic unit could not be justified and the pipe was replaced with stainless steel. The bottom heads on the high-pressure inert gas driers were also changed from carbon steel to stainless steel.

4.7.3 COMPRESSOR PIPING

Gasifier recycle gas compressor suction and discharge piping gave evidence of corrosion after several years of plant operation. The first indication of a problem occurred in June, 1975, following cleaning of the gasifier suction and discharge lines to remove carbon deposits. A layer of tightly adhering scale was noted in the discharge piping. Plant personnel were concerned with the possibility of hydrogen blistering; therefore, a section of the pipe was removed and the scale analyzed. The report stated no evidence of hydrogen blistering or any other type of hydrogen attack. The internal scale consisted of Alpha $Fe_2O_3.H_2O$, Fe_3O_4 (Magnetite), and Fe_2O_3 (Hematite).

In January, 1976, after the plant was at pressure, but before the system was at temperature, the suction line near the gasifier recycle compressors failed because of metal thinning along the bottom of the horizontal sections of pipe. Some 30 to 40 feet of carbon steel pipe were replaced. The cause of failure appeared to be two-fold:

(1) The corrosion was more severe where the steam tracing contacted the pipe.
(2) Horizontal sections were in worse condition in the bottom third of circumference.

Corrosion in vertical sections was more evenly distributed throughout the circumference.

During a startup in June, 1977, several small leaks developed in a carbon steel line near the gasifier compressor water knock-out vessel. Again the corrosion was more severe at the points of contact of the steam tracing.

A major portion of the piping from the gasifier recycle gas water scrubber to the knock-out vessels, compressors, and to the flare header was replaced during the last 18 months of plant operation. The pulsation snubbers of the gasifier recycle compressor suction had to be replaced as well.

This corrosion was greatly accelerated by the frequent shutdowns, typical of a pilot plant, which allow moisture to condense during nonoperating periods. Carbon steel would probably be adequate in a commercial facility in this service; however, in a pilot or demonstration unit, stainless steel should be used.

4.8 CORROSION TEST PROGRAM

The two reactor vessels were the locations for corrosion testing of metals and refractories under an ERDA contract with The Metal Properties Council, Inc. The reference Contract is EX-76-C-01-1784.

The Illinois Institute of Technology Research Institute (IITRI) supplied metal and refractory coupons which were installed inside the gasifier and regenerator through the top Grayloc fitting. The coupon holders were installed during the maintenance shutdowns between runs, and histories of the time, temperatures, and gas composition of the vessel atmospheres were compiled. At IITRI's request, the coupons were removed, pictures taken, and shipped back to the Institute. New coupons were then installed and the test procedure repeated.

Inquiries as to the test results should be referred to:

The Metals Properties Council, Inc.
345 East 47th Street
New York, New York 10017

☆U.S. GOVERNMENT PRINTING OFFICE:1979 -640 -014/ 4606 REGION NO. 4