

**PULSED COMBUSTION PROCESS FOR BLACK LIQUOR GASIFICATION**

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Conservation and Renewable Energy  
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## **EXECUTIVE SUMMARY**

This first annual report summarizes the work accomplished over the period October 1989 through October 1990 for DOE Cooperative Agreement No. DE-FC05-90CE40893. The overall project objective is to field test an energy-efficient, innovative black liquor recovery system at a significant industrial scale. This is intended to demonstrate the maturity of the technology in an industrial environment and serve as an example to the industry of the safer and more energy-efficient processing technique.

The project structure is comprised of three primary activities: process characterization testing, scale-up hardware development, and field testing. The objective of the process characterization testing is to resolve key technical issues regarding the black liquor recovery process that were identified during earlier laboratory verification tests. This will provide a sound engineering data base for the design, construction and testing of a nominal 1.0-TPH mill integrated, black liquor recovery gasifier.

The objective of the scale-up hardware development effort is to ensure that key hardware components, in particular the pulse heater module, will perform reliably and safely in the field.

Finally, the objective of the field test is to develop an industrial data base sufficient to demonstrate the capabilities and performance of the operating system with respect to thermal efficiency, product quality, fuel handling, system control, reliability and cost. These tests are to provide long-term and continuous operating data at a capacity unattainable in the bench-scale apparatus.

The results of the process characterization work, completed this year, established definitive performance data for the thermochemical reactor including product gas quality, carbon conversion efficiency, and theoretical system thermal efficiency. Modifications and enhancements of the laboratory



verification unit were made under the current program to include the critical downstream equipment components needed to evaluate integrated system performance which is vital to establish the engineering design criteria for construction of a field test unit.

Key additions to the test rig included:

- Incorporation of a recirculation cyclone to increase solids residence time and enhance carbon conversion efficiency.
- Improved catch cyclone design for enhanced particulate removal efficiency.
- An isokinetic probe to allow characterization of solids carry-over.
- A quench scrubber and heat removal system for production of dry, particulate-free product gases.
- Cartridge filters for total recovery of unconverted carbon in the quench loop.
- A sulfur recovery column to allow characterization of green liquor regeneration potential.
- Improved combustor tube design allowing higher throughput.
- A gas chromatograph with enhanced sulfur characterization capability.
- Direct and indirect (tracer methods) measurements of dry gas product flow rates enabling more complete material balance closure.
- A recirculation compressor for investigating use of recycle gas as a fluidization media.

Two separate Kraft black liquor feedstocks and one Neutral Sulfite Semichemical (NSSC) liquor feedstock were utilized during the current test program. The first Kraft feedstock comprises a liquor supplied from Weyerhaeuser's Everett, Washington mill. The second Kraft feedstock comprises a liquor supplied from Weyerhaeuser's Longview, Washington cross recovery mill. The NSSC feedstock comprises a liquor supplied from Weyerhaeuser's North Bend, Oregon mill.

In the integrated commercial recovery system, a portion of the gasifier product gas is returned to the pulse combustors to provide the energy requirements of the reactor. In the earlier work, natural gas and simulated product gas were tested in a pulse combustor to ensure that the unit is capable of operating on a gas that was characteristic of that generated from the gasifier. Under normal laboratory conditions, the product gases were combusted in an incinerator using a significant pilot support flame. In order to ensure the flammability of the product gases during unsupported combustion and prior to testing in a pulse combustor, the incinerator support flame was shut off and the continued combustion intensity within the incinerator indicated that the fuel gas was suitable for testing in the pulse unit. The fuel gas was then ignited in a pulse combustor firing at a rate of approximately 80,000 Btu/hr. The pulse combustor lighted off without difficulty and operated with excellent turndown performance. This test confirmed the ability to operate the pulse combustor in a self-sustaining manner using fuel gas generated from the gasifier reactor.

In the MTCI recovery process, black liquor is sprayed directly onto a bed of sodium carbonate solids which is fluidized by steam. Direct contact of the black liquor with hot bed solids promotes high rates of heating and pyrolysis. Residual carbon, which forms as a deposit on the particle surface, is then gasified by reaction with steam. Heat for the endothermic pyrolysis and steam-gasification reactions is supplied from pulse combustor resonance tubes which are immersed within the fluid bed.

The gasifier operating conditions are maintained at temperatures below that at which smelt can form. In this dry recovery process, oxidized sulfur species are partially reduced by reaction with the gasifier products, principally carbon monoxide and hydrogen. The reduced sulfur form is unstable in the gasifier environment, decomposing to solid sodium carbonate and gaseous hydrogen sulfide ( $H_2S$ ). Sulfur values are recovered by discharging a dry sodium carbonate product from the gasifier.

MTCI's indirectly heated gasification technology for black liquor recovery also relies on the scrubbing of  $H_2S$  from product gases to regenerate green liquor for reuse in the mill circuit. Due to concerns relative to the efficiency of sulfur recovery in the MTCI integrated process, an experimental investigation was undertaken to establish performance and design data for this portion of the system.

Operating data for tests conducted at the different scrubber column heights are summarized in Table ES-1. Test data is shown only for test runs where both gas and liquid analyses were available. For each case, the column height, liquid and gas flow rate,  $H_2S$  concentration at column exit as measured by gas chromatograph, and the calculated sulfur recovery efficiency are shown. All tests were conducted utilizing a single batch of premixed sodium carbonate solution with a concentration of 145 g/L as  $Na_2CO_3$  (84.5 g/l as  $Na_2O$ ). While the gas and liquid flow rates were varied in the test matrix, the sulfur-to-sodium ratios entering the column in the gas and liquid phases were held approximately constant, and reflected values typical of a mill circuit operating at a sulfidity of approximately 30 percent (with sulfidity defined as  $Na_2S/TTA$  as  $Na_2O$ ).

Liquid samples of the rich solution were collected at the column exit for each of the test points shown in Table ES-1. These liquid samples were sent to Weyerhaeuser's laboratories for analysis. The results of these analyses are shown in Table ES-2 and include sodium, total sulfur and total carbonate. The calculated sulfidity is also shown. Note that it is assumed that all of the sulfur is present in reduced form since oxidation of the sulfur species can only occur due to incidental air exposure during sampling or analysis.

**TABLE ES-1: OPERATING DATA FOR SCRUBBING TESTS**

<u>SAMPLE DESIGNATION</u>	<u>PACKING HEIGHT (FT)</u>	<u>LIQUID* FLOW RATE (LPM)</u>	<u>GAS** FLOW RATE (SCFM)</u>	<u>H<sub>2</sub>S @ COLUMN EXIT (PPMV)</u>	<u>SULFUR RECOVERY EFFICIENCY (%)</u>
MTCI-7-11-1000	10.000	0.210	3.667	50.000	99.750
MTCI-7-11-1015	10.000	0.330	5.040	271.000	98.645
MTCI-7-12-1145	5.000	0.296	5.040	763.000	96.185
MTCI-7-12-1152	5.000	0.296	5.040	713.000	96.435
MTCI-7-13-1022	15.000	0.296	5.040	140.000	99.300
MTCI-7-13-1030	15.000	0.137	2.330	447.000	97.765

NOTE: Column was operated at 1 atm exit pressure and ambient temperature.

\*Scrubbing liquid consisted of aqueous mixture of sodium carbonate at concentration of 145 g/L as Na<sub>2</sub>CO<sub>3</sub> (84.8 g/L as Na<sub>2</sub>O).

\*\*Simulated gas contained 20% CO<sub>2</sub>, 2.0% H<sub>2</sub>S, and balance nitrogen.

**TABLE ES-2: ANALYSIS OF LIQUID SAMPLES FROM SCRUBBER BOTTOMS**

<u>SAMPLE DESIGNATION</u>	<u>SODIUM Na (%)</u>	<u>SULFUR S (%)</u>	<u>CARBONATE CO<sub>3</sub> (%)</u>	<u>SULFIDITY* (%)</u>
MTCI-7-11-1000	5.64	1.20	8.43	30.59
MTCI-7-11-1015	5.45	1.13	7.75	29.81
MTCI-7-12-1145	5.66	1.06	7.96	26.92
MTCI-7-12-1152	5.53	1.07	7.56	27.81
MTCI-7-13-1022	5.52	1.12	7.73	29.17
MTCI-7-13-1030	5.51	1.03	7.92	26.87

\*Sulfidity is calculated as total sulfur as Na<sub>2</sub>O divided by total sodium as Na<sub>2</sub>O.

As seen in Table ES-2, the desired rich solution (green liquor) sulfidity range (25-35%) is achievable while maintaining low sulfur breakthrough in the gas phase. These results provided confirming evidence that green liquor can be regenerated at a high efficiency using the simple alkaline scrubbing technique developed for the MTCI integrated recovery process.

Ten black liquor tests were done during this year. The seven for which the data analysis were completed are reported here. Samples from the last test were not yet analyzed by Weyerhaeuser Research Center. However, the results for one Kraft liquor, one cross recovery liquor, and one neutral sulfite semichemical (NSSC) liquor were analyzed and are summarized in Table ES-3.

**TABLE ES-3:  
SUMMARY OF BLACK LIQUOR TESTS**

TEST DATE	LIQUOR FEED TYPE	GASIFIER TEMP. (°F)	TEST DURATION (hours)	WET FEED RATE (lbs/hr)	DRY FEED RATE (lbs/hr)	STEAM RATE (lbs/hr)	SULFUR GASIF. EFFIC. (%)	CARBON GASIF. EFFIC. (%)
1/24/90	Kraft	1138	2	18	10	35	--	--
1/24/90	Kraft	1130	5	23	13	35	--	--
1/24/90	Kraft	1050	2.5	40	23	35	--	--
2/23/90 <sup>1</sup>	Cross-Recovery	1145	8	19.3	12.7	38.2	91.2	88.2
3/07/90 <sup>2</sup>	Kraft	1150	7.5	27.1	15.3	33.7	90.5	83.3
3/21/90 <sup>2</sup>	Kraft	1143	7.5	29.3	16.6	37.8	88.3	84.9
3/27/90 <sup>3</sup>	Kraft	1160	6	28.8	16.3	24.10	81.4	79.5
9/18/90	NSSC	1130	8	21.5	8	36	84.5	83.1
9/24/90	NSSC	1150	5.5	26.8	10	38	85.2	84.9

<sup>1</sup>Scrubbed product gas fired in pulse combustor.

<sup>2</sup>Gas scrubbing.

<sup>3</sup>Gas recycle for fluidization.

These tests demonstrated that the MTCI indirect gasification process works well for any type of black liquor, separating sulfur as H<sub>2</sub>S from the sodium which forms mainly Na<sub>2</sub>CO<sub>3</sub> and producing a consistent high-quality

hydrogen-rich, medium-Btu gas. This process enables pulp and paper mills to recover chemicals and energy without smelt formation from Kraft, sulfite (NSSC or BCTMP) or combination (cross recovery) liquors. This process offers great flexibility in selecting pulping method to meet market demand.

The tests completed to date have demonstrated that in the temperature range of approximately 1150°F (625°C), carbon gasification efficiencies of up to 88 percent and sulfur gasification efficiencies of up to 89 percent are achievable. Significant sulfate reduction is also achieved, 60 percent for NSSC liquor and 88 percent for Kraft liquor, without employing carbon or char recycle. It is possible that the sulfate reduction may be improved by char recycle.

The present investigation has confirmed that high sulfur recovery efficiencies exceeding 99 percent, can be achieved under the anticipated operating conditions of MTCI's integrated recovery process using a simple, single column, alkaline scrubbing technique. The formation of sulfur gases other than H<sub>2</sub>S has been found to be negligible, usually less than 10 ppm. Overall, sulfur recovery is expected to be over 99 percent and sodium recovery, over 99.9 percent. Further process optimization to improve the net gas yield and reduce the gas fired as fuel is being planned for testing in early next year. These results suggest that there exists no major technical barriers to regenerating a high sulfidity green liquor in the MTCI process. Furthermore, the results indicate that the MTCI process has a potential for reducing emissions of SO<sub>2</sub> from energy recovery equipment compared to the conventional Kraft recovery boiler.

In addition to the process characterization activities, the design and development of the scaled-up system was partially completed. The Engineering Design for the Black Liquor Gasification Field Test Unit has been consolidated into a single Bid Package separated into three volumes: 1) Commercial, 2) Technical, and 3) Plans and Drawings. The purpose of the Bid Package is to form the Commercial and Technical basis for quotation on a turnkey Black Liquor Gasification Field Test Pilot Plant to be located at a Host Paper Mill. The Bid Package covers specifications and requirements for detailed engineer-

ing, procurement, fabrication, assembly, transportation, field erection, precommissioning and permitting as pertaining to the recovery plant.

Computer simulations of material and energy balances have also been prepared for black liquor solid feedstocks. The field test unit is being designed to handle two extreme operating conditions, as follows:

- 1 ton/hr Black Liquor Solids at 33% solids by weight, and
- 2 ton/hr Black Liquor Solids at 65% solids by weight.

At the 65 percent solids level, the products from gasification of black liquor solids include chemical recovery of pulping chemicals in the form of green liquor (11323 lb/hr), a clean medium Btu fuel gas (293 Btu/SCF) rich in hydrogen, export steam from waste heat recovery (1134 lb/hr), and insoluble carbon filter dregs (53.5 lb/hr).

The "Break-even" point for product gas production occurs at approximately the 33 percent solids level. At this minimum concentration there is just enough process gas produced to fire the combustors at a rate to support the gasification reaction. The largest utility is cooling water (276,000 lb/hr) for removing steam and condensable hydrocarbons from the product gas. The cooling water load is reduced and the overall thermal efficiency is increased substantially if the fluidizing gas is a mixture of recycled product gas and steam instead of steam alone. Use of recycle gas will also be investigated during the field tests.

## **ACKNOWLEDGMENT**

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**SECTION 1.0**  
**INTRODUCTION**

This report summarizes technical achievements accomplished over the period from October 1989 through October 1990, relevant to the development of a Pulsed Combustion Process for Black Liquor Gasification. Extensive bench-scale experimental testing was conducted to firmly establish critical design data needed for scaling the technology to a nominal 1-TPH field test unit. This data was employed in the preparation of process and mechanical designs for an integrated black liquor recovery facility to be installed and field tested at Weyerhaeuser's New Bern, North Carolina Kraft pulp mill. Fabrication of the field test unit is scheduled to begin in early 1991. The objective of the field tests is to verify gasifier performance at a significant industrial scale thereby providing a fast-track introduction of this innovative energy recovery technology in the pulp and paper industry.

The Pulsed Combustion Process for Black Liquor Recovery comprises a pulse-enhanced, indirectly heated, fluidized bed reactor using the principle of steam gasification which effects the recovery of inorganic sodium chemicals and energy from the organic matter contained within the black liquor. The integrated system results in the production of a green liquor product stream that is recycled to the pulping process as well as a high-quality medium-Btu gas that is used to supplement and/or reduce purchased fuel requirements within the mill.

The MTCI thermochemical recovery technology is differentiated from existing combustion-based recovery methods by an avoidance of a sodium smelt formation that routinely results in fouling of heat exchange surfaces and corrosion of boiler components. The molten smelt also poses serious safety problems due to smelt-water explosions. Also, the gasification technology converts organic matter to fuel gas, as opposed to combustion-based processes which generate steam as the sole energy product. This is advantageous since the fuel gas can be combusted in a gas turbine combined cycle system offering higher electric conversion efficiencies compared to simple steam cycles.

Finally, the Pulsed Combustion Process for Black Liquor Gasification offers the singularly unique ability to separate sulfur from sodium regardless of the chemical nature of the starting liquor. This is significant since it offers an economic and efficient means of recovering a broad range of feedstocks ranging from Kraft through sulfite liquors. A more detailed description of the integrated gasification process and its benefits can be found in "Testing of an Advanced Thermochemical Conversion Reactor System," January 1990, DOE Contract No. DE-AC06-76RLO1830.

In Section 2.0 of this report, key program objectives are highlighted. A detailed listing of project work tasks are found in Section 3.0. Section 4.0 provides a description of experimental hardware utilized in bench-scale testing. Section 5.0 discusses the analytical methods for treatment of experimental data. Section 6.0 presents results from extensive bench-scale testing and relates this data to process design criteria for the field test unit. Scale-up hardware and control development testing is described in Section 7.0. Details of the field test system engineering design are presented in Section 8.0. And, finally, conclusions and plans for the next period are discussed in Section 9.0.

**SECTION 2.0**  
**PROJECT OBJECTIVES**

The overall project objective is to field test an energy-efficient, innovative black liquor recovery system at a significant industrial scale. This is intended to demonstrate the maturity of the technology at a scale that will prompt the industry to take advantage of the more energy-efficient processing technique.

The project structure is comprised of three primary activities: process characterization testing, scale-up hardware development, and field testing. The objective of the process characterization testing is to resolve key technical issues regarding the black liquor recovery process that were identified during prior feasibility tests conducted DOE Contract No. DE-AC06-76RL01830. This will provide a sound engineering data base for the design, construction and testing of a nominal 1.0-TPH integrated, black liquor recovery gasifier.

The objective of the scale-up hardware development effort is to ensure that key hardware components, in particular the pulse heater module, will perform reliably and safely in the field.

Finally, the objective of the field test is to develop an operational data base sufficient to assess the capabilities and performance of the system with respect to thermal efficiency, product quality, fuel handling, system control, reliability and cost. These tests are to provide long-term and continuous operating data at a capacity scale unattainable in bench-scale apparatus.

**SECTION 3.0**  
**WORK TASK DESCRIPTION**

The following section summarizes the work task description for Phase I and Phase II activities under DOE Contract DE-FC05-90CE40893. Phase I tasks have focused on the collection of process design verification data from bench-scale test equipment, and the translation of this data to the engineering design of a nominal 1.0-TPH field test unit, Phase I tasks have been completed in full at the time of this report. A summary of Phase I results and accomplishments are found in Section 6.0, "Experimental Test Results," and Section 8.0, "Engineering Design of a Field Test Unit."

Phase II tasks comprise a set of activities leading to operational testing of a nominal 1.0-TPH field test gasification system. These activities include key hardware and control development, detailed plant engineering, test plan preparation, equipment procurement, construction and installation, and finally shakedown and field testing. A portion of the Phase II hardware and control development testing has been completed at the time of this report. The results of these Phase II accomplishments are discussed in Section 7.0, "Development Testing."

A detailed description of Phase I and Phase II work tasks follows here.

**3.1 PHASE I - PROCESS DESIGN VERIFICATION**

**3.1.1 TASK I.1: GASIFIER RECOMMISSIONING**

The 8-inch process design verification gasification system, laboratory feasibility unit, will be recommissioned. The effort will include modification of the black liquor feed subsystem, fabrication and installation of a gasifier slip-stream scrubber facility, modification of the gas analysis sampling train, and gas chromatograph to extend the range of detectable gas components to include sulfur species and upgrading of the systems controls.

### **3.1.2 TASK I.2: PROCESS DESIGN VERIFICATION TESTS**

The unit will be operated to collect critical design data that will be needed for scaling to the field-test unit.

#### **A. SUBTASK I.2.1: GAS PHASE SULFUR DISTRIBUTION**

Steady-state gasification test will be conducted to establish the identity and quantity of sulfur-containing constituents in the product gas stream. A specialized gas chromatograph capable of differentiating a broad spectrum of sulfur species will be employed to accomplish this work. Analysis will be made to quantify the sulfur species contained in the solid products from the gasifier. An overall sulfur elemental balance will then be calculated from both solid phase and gas phase analysis.

#### **B. SUBTASK I.2.2: SCRUBBER VERIFICATION TESTS**

A sample gas scrubber subsystem will be constructed and installed. The scrubber will be employed to verify the recovery of green liquor by absorption of  $H_2S$  from a product gas slip-stream. The scrubbing solution will consist of an aqueous sodium carbonate solution that simulates the solids dissolution product from the gasifier. The  $H_2S$  concentration at the exit of the scrubber will be analyzed to obtain estimates of achievable scrubbing efficiencies. In addition, the scrubbing solution will be analyzed for relevant sulfur compounds. This subtask will provide essential information regarding the basic chemistry of the scrubbing operation, and in particular, the nature of trace sulfur species that may be absorbed in the green liquor solution. In addition, solid samples from the gasifier will be dissolved in a tank and its properties will be characterized. This solution, being representative of the scrubbing solution to be used in the integrated field-test unit, will allow meaningful analysis of the process requirements for handling this stream. Particular attention will be given to the treatment and filtering of carbon from the solution.

#### **C. SUBTASK I.2.3: COMBUSTION OF SIMULATED PRODUCT GAS**

Commercial gasification systems may utilize part of the product gas as the pulse combustor fuel source. In contrast, prior laboratory tests have

employed natural gas fuels. Since the combustion properties of these fuels differ, tests are necessary to verify pulse combustor operation on fuel gases with chemical compositions and energy densities similar to that of the product gas.

#### **D. SUBTASK I.2.4: EVALUATION OF EFFECT OF RECYCLE GAS**

Commercial gasification systems will utilize recycle gas to assist in bed fluidization and enhance system thermal efficiency by reducing the amount of steam to the minimum required by the gasification process. In contrast, prior laboratory tests have been conducted without recycle gas by using steam as the sole fluidization medium. The primary difference in these cases relates to the steam partial pressure within the gasifier that impacts the gasification rate and the product gas yield distribution. Thus, a set of tests will be specifically designed to evaluate the effect of steam partial pressure on the gasifier performance.

### **3.1.3 TASK I.3: DATA REDUCTION AND ANALYSIS**

The data collected during Phase I testing will be reduced and summarized. This work will include development of correlations, graphs, plots, and design equations as necessary for scaling to the field test, complete recovery train unit.

### **3.1.4 TASK I.4: ENGINEERING DESIGN**

At the conclusion of the experimental portion of Phase I, an engineering design will be prepared for the 1.0-TPH field-test unit. The engineering design will be guided by this data obtained from the experimental program and will include the following preliminary engineering documents:

- Process Flow Diagram
- Heat and Material Balance
- Plot Plan
- Piping Diagram
- Process Equipment Drawings and Specifications



In addition, a budget estimate for the proposed field-test plant will be prepared.

### **3.2 PHASE II - FIELD UNIT DESIGN, CONSTRUCTION (SHAKEDOWN AND TEST)**

#### **3.2.1 TASK II.1: ESTABLISH PERFORMANCE AND CONTROLS DEVELOPMENT**

Objectives for system design, performance and operation will be established. Performance and design objectives will be specified for each system and feed to be tested and will include:

- Air/fuel ratio,
- Fuel feed rate,
- Steam injection rates,
- Energy production rate (in millions of Btu/hr) to be achieved at a specific air/fuel ratio, fuel feed rate, and steam injection rate,
- Producer gas production rate and energy content (Btu per standard cubic foot),
- The concentration (ppm<sub>v</sub>) and distribution of low to high molecular weight hydrocarbons in the producer gas,
- Producer gas particulate matter concentration and size distribution, and
- Char or ash production rate.

A single module heater bundle of the size that will be used in the field-test unit will be constructed and evaluated. This shall consist of multiple resonance tubes of about sixty. The mechanical design criteria with respect to the differential thermal expansion of the tubes, relationship between tube packing pattern, fluidizing velocity and heat transfer from tubes to the bed, and pulse burner safety controls will be established.

An overall control scheme for the process will be established and a control system will be constructed and tested at the laboratory scale. In the single module heater test rig, sand of different size ranges shall be used for achieving different bed conditions; air shall be employed as fluidizing medium and water shall be used (in place of black liquor) for heat load. Hot operating conditions that are expected in the field-test units between 1100°F to 1200°F shall be established and controlled using the firing rate of the pulse combustor and the feed liquor (water) rate. The control protocol shall be established from these tests for the field-test unit.

### **3.2.2 TASK II.2: SYSTEM DESIGN**

During Phase II, designs will be prepared for the 1.0-TPH black liquor recovery gasifier. The design will include mass and energy balances for the system. In addition, chemical kinetic calculations, subsystem performance sensitivity analysis, environmental impact analysis, safety analysis, and critical materials analysis will be conducted for the overall system and each of the following subsystems:

- System instrumentation and control,
- Fuel feed system,
- Air and stream injection systems,
- Pulse combustor,
- Ash and/or char handling system, and
- Air pollution control system.

### **3.2.3 TASK II.3: TEST PLAN**

A test plan for system testing and evaluation will be evaluated. The plan will describe overall objectives of each major phase of testing, and for each of the subsystems. It will include performance testing over the full operational range of the system. The plan will include a description of each fuel to be tested, each test performed, a protocol for conducting the test, equipment necessary, and data to be obtained.

### **3.2.4 TASK II.4: FINAL DESIGN**

Based on the work completed in Phase I and Phase II, a set of final designs will be developed including materials selection, for the combustor and other support subsystems. A set of working final designs of the system will be prepared that will include the following:

- Blueprints or detailed drawings of the overall system,
- Individual blueprints and materials specifications for each of the major subsystems, and
- Estimated bills of materials for all major subsystems.

### **3.2.5 TASK II.5: EQUIPMENT PROCUREMENT, CONSTRUCTION AND INSTALLATION**

During Phase II, a modular 1.0-TPH integrated black liquor recovery plant will be constructed. The unit will be skid-mounted in transportable pallets for shipment to the mill test site. It will be interfaced in the mill as required. Documentation showing all required local, state, and federal permits, if any, will be obtained for the project.

The gasifier hot section with necessary cyclones and venturi scrubber shall be in one group of skids and the balance of plant consisting of scrubber equipment for sulfur gases shall be in another group of skids.

### **3.2.6 TASK II.6: SHAKEDOWN TESTING - 1.0-TPH BLACK LIQUOR RECOVERY UNITS**

Shakedown testing of the gasifier will be performed in order to establish mechanical integrity and operational reliability of the gasifier on start-ups and shutdowns.

### **3.2.7 TASK II.7: MANAGEMENT AND REPORTING**

This task is devoted to the program's technical direction, in order to maintain the program hardware oriented, and to support reporting functions required by the DOE Uniform Contract Reporting System. Reports will include monthly, quarterly, and annual reports as well as Management Plans and the annual update of such plans, including manpower and other resource

requirements. All projects in the program will be included in the reporting function in addition to the specific requirements of the DOE-sponsored projects.

### **3.2.8 TASK II.8: MOVE AND INSTALL GASIFIER IN PULP MILL SITE**

The test unit will be transported to the host mill site and installed including interconnection of all required utility piping.

### **3.2.9 TASK II.9: FIELD TESTS**

Gasification tests with black liquor will be performed in the 1.0-TPH unit. This effort includes the performance and documentation of all tests necessary to ensure that all components of the facility can and will be operated in a safe and responsible manner prior to achievement of full operational status. An operational test of 24 hours per day, 7 days a week for a mutually agreed upon term (not less than 1 week) will be required in this phase.

This effort includes a planned approach to fully assessing the performance capabilities of the system in accordance with the stated objectives, including (but not limited to) efficiency assessments, process chemistry analysis, system fuel handling, overall system operational flexibility, cost performance, system reliability, and all other information necessary to demonstrate the potential for commercial gasification of black liquor. The participants and DOE will negotiate a mutually agreeable operational schedule for this phase.

### **3.2.10 TASK II.10: REMOVE FIELD TEST UNIT AND RESTORE HOST SITE**

At the successful conclusion of the field-test operation, the Participant shall remove the gasifier facility and restore the host site to as close to the original condition as possible to meet with the host mill manager's satisfaction.

## **SECTION 4.0**

### **EXPERIMENTAL TEST RIG DESIGN**

A bench-scale gasification test rig was previously constructed and tested (1987-1989) under funding from the United States Department of Energy (Contract No. DE-AC06-76RLO1830) and the California Energy Commission ETAP program (Contract No. 500-86-012). This prior work was undertaken to develop process design verification data for an indirectly heated, thermochemical, fluid-bed reactor capable of processing renewable sources of energy (such as biomass) and industrial by-products (such as sludge waste and black liquor) into useful product gas.

The results of this work established definitive performance data for the thermochemical reactor including product gas quality, carbon conversion efficiency, and theoretical system thermal efficiency. However, the prior experimental test rig was constructed primarily to collect data on gasifier hot section and did not include critical downstream equipment components needed to evaluate integrated system performance. Since integrated system testing is vital to establishing engineering design criteria for construction of a field test unit, modifications and enhancements of the existing test rig were made under the current contract.

Key additions to the test rig included:

- Incorporation of a recirculation cyclone to increase solids residence time and enhance carbon conversion efficiency.
- Improved catch cyclone design for enhanced particulate removal efficiency.
- An isokinetic probe to allow characterization of solids carry-over.
- A quench scrubber and heat removal system for production of dry, particulate-free product gases.
- Cartridge filters for total recovery of unconverted carbon in the quench loop.

- A sulfur recovery column to allow characterization of green liquor regeneration potential.
- Improved combustor tube design allowing higher throughput.
- A gas chromatograph with enhanced sulfur characterization capability.
- Direct and indirect (tracer methods) measurements of dry gas product flow rates enabling more complete material balance closure.
- A recirculation compressor for investigating use of recycle gas as a fluidization media.

The primary objective of these equipment modifications was to allow bench-scale system operation in a fashion closely representative of the anticipated field test configuration and to enhance system performance and data collection capability. In this way, a significant reduction in risk associated with scale-up of the bench-scale equipment could be realized.

A detailed description of the integrated bench-scale test rig as constructed under the current contract is provided in the following section.

#### **4.1 PROCESS FLOW DIAGRAM**

A flow diagram for the bench-scale plant is shown in Figure 4-1. 100 psig saturated steam generated in boiler H-1 is reduced in pressure to 60 psig and conveyed in an electrically heated line prior to flow measurement in an orifice meter. The steam is then directed to an electric preheater (X-1) where it is superheated to approximately 800°F. The steam is introduced into the base of the gasifier (R-1) through a series of sparge tubes. This steam serves as the primary fluidization media for the reactor bed.

Black liquor is stored in a steam-jacketed vessel (V-1) where it is heated to approximately 180°F. Black liquor is intermittently withdrawn from V-1 to a weigh tank (not shown) which rests on a platform scale. A diaphragm metering pump (P-1) is used to feed a controlled rate of black liquor to the gasifier. The metering pump is manually calibrated; however, precise measurement of cumulative flow rate is determined by weight loss from the weigh tank/platform-scale system. The black liquor is injected approximately

P-1 BLACK LIQUOR PUMP  
 H-1 STEAM BOILER  
 X-1 ELECTRIC STEAM SUPERHEATER  
 R-1 GASIFIER  
 V-2 RECIRCULATION CYCLONE  
 V-3 FINES CYCLONE  
 P-1 COMBUSTOR AIR BLOWER  
 V-4 FINES COLLECTION DRUM  
 X-2 VENTURI SCRUBBER  
 V-5 BLACK LIQUOR FEED TANK  
 V-6 A/B FILTERS  
 V-7 GREEN LIQUOR DRUM  
 V-8 ALKALINE FEED TANK  
 P-4 ALKALINE FEED PUMP

V-1 SCRUBBER COLLECTION DRUM  
 P-2 FILTER PUMP  
 P-3 QUENCH PUMP  
 E-1 QUENCH COOLER  
 C-1 GREEN LIQUOR REGENERATION COLUMN  
 V-7 GREEN LIQUOR DRUM  
 V-8 ALKALINE FEED TANK  
 P-4 ALKALINE FEED PUMP

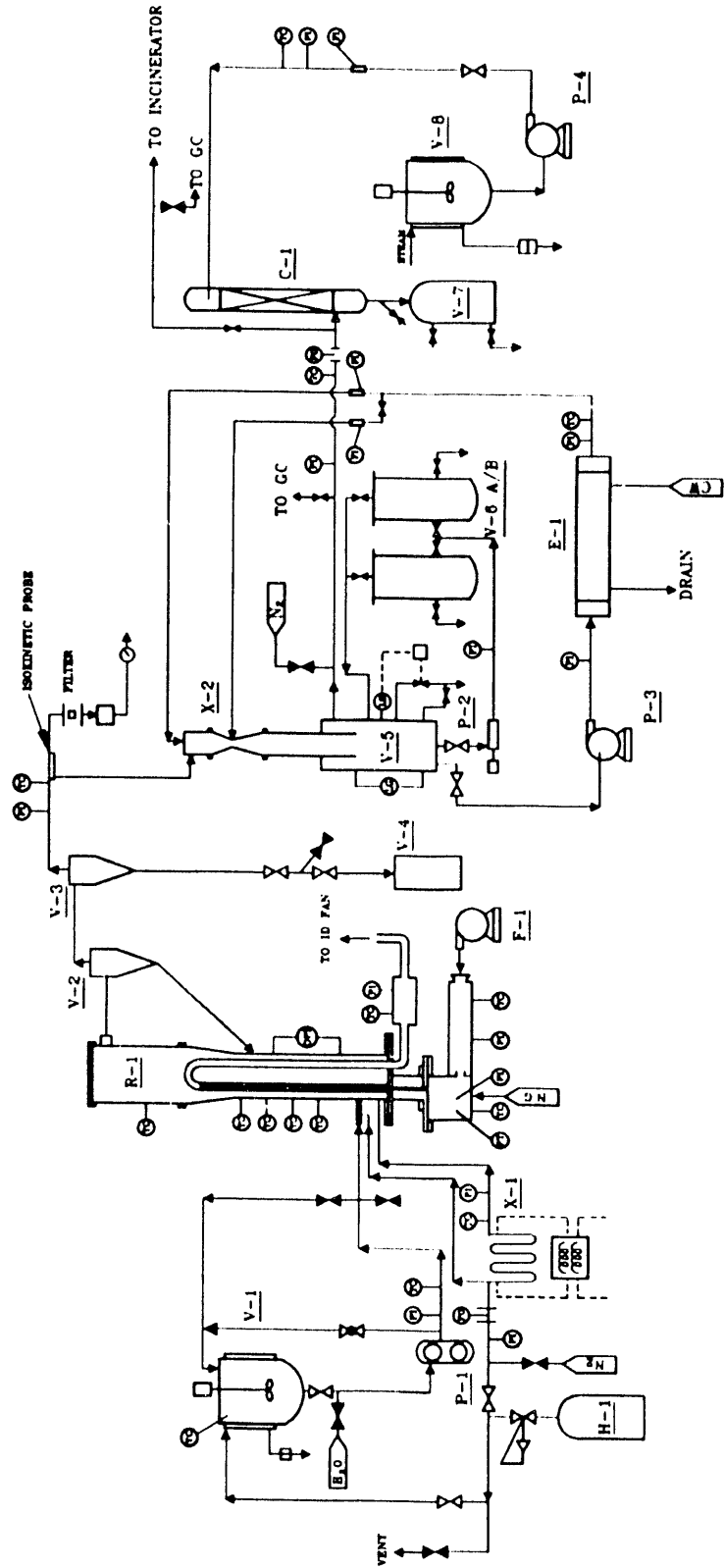


FIGURE 4-1: PROCESS FLOW DIAGRAM FOR BENCH-SCALE TEST RIG

2 feet above the gasifier base utilizing a single coaxial injector containing steam assist in the outer annulus.

The gasifier is heated by means of indirect fire tubes which are immersed within the fluid bed and which are in direct communication with a pulsating combustor. The pulsating combustor is fired using natural gas fuel and a forced draft fan (F-1) is utilized to deliver combustion air to the combustor air plenum.

Product gases emanating from the fluid-bed reactor are directed to two cyclones in series. The first cyclone (V-2) functions in a complete recycle mode. Here, coarse particles which are recovered enter the cyclone dip leg and are redirected to the top of the bed via a steam eductor nozzle (not shown). The recirculation cyclone (V-2) serves primarily to increase residence time of particulate within the hot reactor environment to enhance conversion efficiency.

The product gases then enter a second fines catch cyclone (V-3). Here, fine particulate is captured and collected in a drum (V-4). The catch drum (V-4) is intermittently emptied and weighed to determine the fines collection rate.

The product gases, containing small fractions of fines carry-over material, enter a venturi scrubber system. The venturi scrubber (X-2) serves to provide high momentum contact between a circulating water stream and the product gas. This promotes the removal of fine particulate while simultaneously achieving cooling of the gas and condensation of steam contained therein. The circulation water is separated in scrubber collection drum (V-5) and the product gases exit in a saturated state at approximately 120°F.

The heat of cooling and condensation is rejected in scrubber cooler (E-1). This is accomplished by circulating the 120°F scrubber tank water using pump (P-3) through cooler (E-1). The scrubber water is circulated on the tube side of E-1 where it is cooled to approximately 100°F or less. City water flows countercurrently on the exchanger shell side.



Also, scrubber tank water is circulated in an independent loop through parallel filters (V-6 A/B) using pump (P-2). The filters serve to remove accumulated solids within the tank and to provide a quantitative measure of insolubles (primarily unconverted carbon) collected during the test run. The filter flow rate is selected to reduce steady-state solids levels in the scrubber to typically less than 1.0 wt.%. This assists in preventing fouling in the heat rejection exchanger and plugging in the venturi quench loop.

The product gases then enter the base of a packed scrubbing column where they are countercurrently contacted against an alkaline solution composed of sodium carbonate and water. The alkaline scrubbing solution is batch mixed in tank (V-8) and conveyed to the top of the packed column (C-1) via pump (P-4).

The packed column (C-1) serves to remove H<sub>2</sub>S contained within the product gases. The alkaline solution formed from this contacting process comprises a solution of sodium carbonate and sodium sulfide, referred to as "green liquor." The green liquor is collected in drum (V-7).

The clean product gas exits the top of column C-1 and is combusted in a continuously piloted incinerator (not shown). The incinerated gases are quenched and scrubbed further in a packed column (not shown) containing a circulating sodium carbonate solution. The scrubbed flue gases are then vented to the atmosphere.

## **4.2 BLACK LIQUOR FEED SYSTEM**

The black liquor feed system consists of an 80-gallon storage tank, a 21-gallon metering tank, a recirculation pump, a black liquor feed pump, a temperature control system, a weigh scale, and an agitator.

The stainless steel storage tank is steam jacketed and insulated. Heat input is thermostatically controlled by regulation of a solenoid valve on the steam inlet line. Condensate discharge is controlled by a steam trap at the exit of the steam jacket. A 3/4 HP Teel pump with 10 feet head capacity at 3450 rpm recycles the black liquor to ensure uniform liquor temperature and

consistency. In addition, the storage tank is agitated by an air-driven mixer.

Black liquor from the storage tank is intermittently discharged to the 21-gallon metering tank. The transfer line is heat traced to keep the black liquor from solidifying during intermittent use. The rate of black liquor feed to the gasifier is controlled by a 1½-HP Yorkway Cyclo/Phram positive displacement metering pump model #0712-26-3431. This pump has a maximum capacity of 21.7 gph. The black liquor feed rate is measured with an Arlyn digital scale with a capacity of 200 lbs. The weight of the tank and contents are measured with the Arlyn scale and recorded periodically (every 10 to 20 minutes). The tank is insulated and has a steam coil and thermocouple to maintain a constant temperature. During initial testing, the black liquor metering pump was operated by manual adjustment of the diaphragm stroke volume (varidrive). The pump was later retrofitted with a Honeywell Actionator Motor #M940A1067 and a Honeywell Motor Positioner #R7195A1031. This allowed the gasifier temperature to be controlled at a fixed firing rate by automatic adjustment of the black liquor feed rate. An Omega model #6071A auto-tuning controller with 2 alarm relays and a 4-20mA controlled output was used to supervise control the Honeywell Motor Positioner in conjunction with a thermocouple in the gasifier bed.

### **4.3 GASIFIER HOT SECTION**

The gasifier hot section consists of the gasifier shell, steam distributor nozzles, liquor injectors, pulse combustor module with integral immersed fire tubes, recirculation cyclone and polishing cyclone. The gasifier shell comprises an 8-inch lower portion and a 12-inch upper (freeboard) section. The expanded fluid bed height is approximately 6 feet.

Steam distribution to the base of the gasifier is accomplished by means of six sparge tubes. Liquor is injected directly into the fluid bed using a single coaxial steam-assisted nozzle.

The hot product gases exit the gasifier to a recycle cyclone. This functions to capture entrained particulate for return to the bed. A second

cyclone is utilized in series to remove the bulk of the remaining particulate contained in the product gases.

The pulse combustor module is mounted at the base of the gasifier. The pulse combustor has a nominal firing rate of approximately 200,000 Btu/hr. The pulse combustor is connected to two independent fire tubes which are immersed within the fluid bed. The fire tubes serve to indirectly transfer heat to the bed to support the endothermic gasification reactions. The fire tubes are fashioned in a U-tube arrangement.

#### **4.4 SCRUBBER AND HEAT REJECTION HEAT EXCHANGER**

The product gases are scrubbed of most remaining particulates and cooled to remove sensible and latent heat of the steam and hydrocarbons. Condensation of steam is required to facilitate data collection via gas chromatography and to allow efficient H<sub>2</sub>S removal in the green liquor recovery column. The heat rejection is accomplished by circulating 6 gpm of water using an Ingersol-Rand 2-HP, 3450-rpm pump through a single-pass heat exchanger, 14 feet long and containing approximately 75 ft<sup>2</sup> of heat transfer surface area. The scrubber water exits the heat exchanger at 100°F and is sprayed into a venturi scrubber at 4 points using 3/8-inch diameter stainless steel tubing. The water impinges upon the venturi wall cooling the gas stream to approximately 120°F and condensing the water vapor into the scrubber tank.

#### **4.5 CARTRIDGE FILTERS**

The solids scrubbed from the product gases are collected in the scrubber tank. A cumulative calculation of the solids in the product gases is determined by recycling the scrubber tank contents continuously through two Harmsco Industrial Cartridge pressure filters with five micron screens. A material balance is then calculated based on knowledge of the total mass accumulated on the filters.

The Harmsco filters each include seven cartridges and are enclosed in a stainless steel vessel. The recycle of the scrubber tank through the filters is accomplished by a 3/4-HP Teel pump rated at approximately 30 psi differential pressure.

## 4.6 HYDROGEN SULFIDE SCRUBBING SYSTEM

The hydrogen sulfide scrubbing system was employed to reclaim green liquor by absorption of sulfur species contained in the gasifier product gases. The system comprised an alkaline feed tank, a packed scrubbing column, and a receiving drum.

An alkaline scrubbing solution (14.5 wt.%  $\text{Na}_2\text{CO}_3$ ) was batch mixed in the alkaline feed tank. This solution was then pumped to the top of the scrubbing column at a controlled rate. Here, the upward flowing product gases were counter-currently contacted with the downward flowing solution.

The scrubbing solution was fed to the column by a 1/4-inch SS tubing. The solution was distributed evenly throughout the column cross-sectional area by a distribution plate. The liquid distribution plate consisted of a 3" OD x 1/4" thick SS plate with 25" x 1/4" drill-thru holes. Also, baffles were incorporated inside the column to redirect the gas and liquid flow. The baffles were made from 3-inch SS washers with a 2-inch concentric hole. The scrubbing solution flowed down through the packed section and was collected in a drum where it was neutralized by adding a metered quantity of sodium hydroxide. A sample valve was incorporated on the drum line where liquid samples were collected after the scrubber.

The scrubbing column consisted of a 3" x 21" SS 304 tube. The column was fabricated in four sections (5, 7, 6, and 3 feet sections), enabling ease of assembly and disassembly and allowing flexibility to vary packed column height. Sections were attached to one another by SS 304 lap joints and carbon steel slip-on flanges. A U-shaped standing pipe was incorporated to maintain a 6-inch liquid pool at the bottom of the column and to prevent column siphoning. Thermocouple and pressure gauge ports were incorporated at various sections of the column. The column was insulated with a 1-inch thick fiberglass insulating material.

The column was packed with 10 mm long, ceramic berl-saddle packing rings. the column packings were obtained from Jaeger Products, Inc., Spring, Texas. The height of the packing could be increased or decreased by adding or discharging packing rings.

#### **4.7 RECYCLE COMPRESSOR**

A recycle compressor was provided to allow recirculation of product gases to the fluid bed. Recirculation of product gases allows a reduction of steam flow to the gasifier, particularly during turndown, and improves overall system thermal efficiency. The compressor is rated for 3 SCFM at 30 psig discharge pressure.

#### **4.8 INCINERATOR/SCRUBBER**

After passing through the sulfur recovery column, the product gases are incinerated and vented through a polishing scrubber. The incinerator is supported by a continuous natural gas pilot flame. The scrubber serves to simultaneously cool and scrub gases of residual sulfur dioxide before release into the atmosphere.

#### **4.9 GAS ANALYSIS EQUIPMENT**

The instruments used for gas analysis included two MUI M-200 gas chromatographs, one Tracor 540 gas chromatograph, one Teledyne MAX-5 efficiency monitor, and two Horiba PIR-2000 gas analyzers.

The M-200 gas chromatographs were used for detailed quantitative analyses of the gasifier product gases. Currently, they are configured to measure the concentrations of  $H_2$ ,  $N_2$ ,  $O_2$ ,  $CO$ , and  $CO_2$ , methane, ethane, ethylene, propane, propylene, butane, iso-butane,  $H_2S$ , and methyl mercaptan.

To complement the data provided by the gas chromatograph, three continuous monitors were employed. The Teledyne MAX-5 efficiency monitor measures the concentrations of oxygen, carbon dioxide, carbon monoxide, and total combustible gases. It also calculates the combustion efficiency. The Horiba PIR-2000 gas analyzers are used for continuous monitoring of a single specified gas component each. One of the units is configured to monitor

nitric oxide (NO), another is configured to monitor sulfur dioxide (SO<sub>2</sub>) only. The continuous analyzers were used primarily to monitor combustion products from the pulse combustor and incinerator units.

A third gas chromatograph was purchased during the course of testing. The Model 540 GC from Tracor Instruments was equipped with a capillary column and a FPD with FID output. The purchase of this GC was necessary to meet the requirement of separating and detecting different sulfur compounds to an extent unattainable in the M-200 model.

Two IBM-AT compatible computers were equipped for data collection and control of the GCs. The M-200 GC is controlled using EZ Chrom200 software from MCI. A Spectra Physics SP4270 integrator was used to record chromatograms from the M-200s. A Linseis L-6012 dual channel chart recorder was also used for real-time recording of chromatograms on a hard copy for M-200.

#### 4.9.1 M-200 GAS CHROMATOGRAPHS

Each M-200 has two channels which have separate chromatographic columns and detectors. By combining different types of column, carrier gas and operation conditions (temperature and column pressure) the range of detectable compounds can be optimized. Table 4-1 summarizes the configuration of two of the M-200s and the compounds they can detect.

**TABLE 4-1: CONFIGURATION SUMMARY OF TWO MCI M-200s**

GC CHANNEL	CARRIER GAS	COLUMN	DETECTOR	TEMP. (°C)	PRESSURE (psi)	COMPOUNDS	
#1	A	Ar	Molecular Sieve 5A	TC	60	10.5	H <sub>2</sub> , O <sub>2</sub> , N <sub>2</sub> , CH <sub>4</sub> , CO
	B	Ar	HayeSep A	TC	40	15.5	CH <sub>4</sub> , CO <sub>2</sub> , ethylene, ethane
#2	A	He	DB-5	TC	35	16.5	propane, SO <sub>2</sub> and H <sub>2</sub> O (co-elute) iso-butane, n-butane, methyl mercaptan and HC higher than C <sub>4</sub>
	B	He	HayeSep A	TC	70	18.0	CO <sub>2</sub> , ethylene, ethane, H <sub>2</sub> S, COS, propylene, SO <sub>2</sub> , and H <sub>2</sub> O

Channel A of GC #1 is equipped with a Molecular Sieve 5A column. This channel is configured to detect the so-called permanent gases:  $H_2$ ,  $O_2$ ,  $N_2$ , CO, and  $CH_4$ . In some instances moisture and carbon dioxide in the sample gas is retained on the column after repeated sample injections, resulting in a deterioration of column separation efficiency. When this situation occurs, the column needs to be heated to an elevated temperature to re-condition it. As re-conditioning may interrupt data acquisition, two parallel molecular sieve columns were later installed. With two molecular sieve columns installed in the #1 GC, gas analysis could be conducted on one column while re-conditioning another column.

Channel B of GC #1 is equipped with a HayeSep A column and uses argon as the carrier gas. The compounds that can be detected in this channel include methane, carbon dioxide, ethylene and ethane. Because these components can also be detected on other columns, it was later replaced with another molecular sieve column, as previously discussed.

Channel A of GC #2 is equipped with a DB-5 column and uses helium as the carrier gas. This channel detects heavier hydrocarbons such as propane, isobutane and normal-butane. Hydrocarbons heavier than  $C_4$  can also be detected on this column at varying levels of accuracy.

Two sulfur compounds,  $SO_2$  and  $CH_3SH$ , can also be detected on this column. Unfortunately, both of them are interfered by other compounds.  $SO_2$  co-elutes with  $H_2O$  while  $CH_3SH$  has retention times quite similar to that of normal butane. This co-elution effect caused difficulties in detecting these sulfur compounds during black liquor gasification tests. However, this problem was resolved using the sulfur specific detector (FPD) provided with the Tracor gas chromatograph.

Channel B of GC #2 has a HayeSep A column and uses helium as the carrier gas. This channel detects  $CO_2$ , ethylene, ethane, hydrogen sulfide, carbonyl sulfide, propylene and sulfur dioxide. Again, the sulfur compounds are interfered by other components co-eluting on the column. The most serious interference is between water and  $SO_2$ . Again, the Tracor gas chromatograph was useful in overcoming these sulfur detection limitations.

#### **4.9.2 TELEDYNE MAX-5 EFFICIENCY MONITOR**

The MAX-5 efficiency monitor measures the oxygen, carbon monoxide and total combustible compounds directly. It calculates the carbon dioxide and combustion efficiency based on pre-programmed fuel properties. This instrument provides accurate measurement of oxygen and low concentration carbon monoxide (<1000 ppm) in the test gas.

#### **4.9.3 HORIBA PIR-2000 GAS ANALYZER**

The PIR-2000 gas analyzer determines the concentrations of a given component by measuring the infrared absorption specific to the component of interest. The two PIR-2000 gas analyzers are specifically configured to measure NO and SO<sub>2</sub> concentrations in the sample gas stream, respectively. The gas analyzer uses solid filters to eliminate possible interference from other components which have absorption spectra partially overlapping the absorption peak of the monitored component.



## **SECTION 5.0**

### **ANALYTICAL METHODS**

The gasification test rig was furnished with sufficient sampling means to provide rigorous material balances for both carbon and sulfur. The primary sample collection points for the gasification test rig are illustrated in **Figure 5-1**. Key test data collected for each run as needed for preparation of material balances is summarized below.

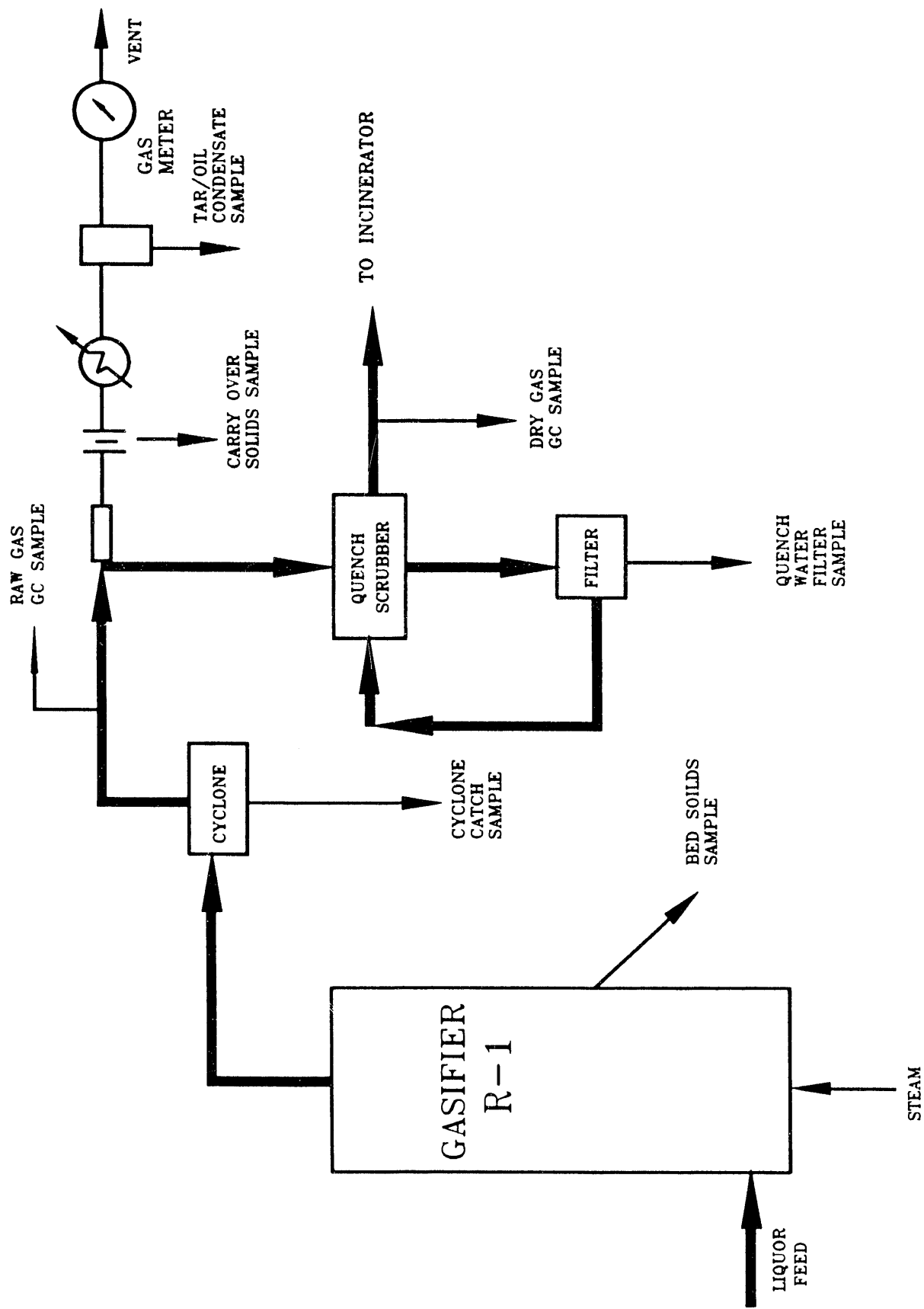
#### **5.1 BLACK LIQUOR FEED RATE**

Black liquor feed rate was recorded utilizing a weight loss method employing a feed tank and a digital platform scale. Cumulative feed was recorded approximately each 20 minutes and translated to an average mass rate over the period. During the course of the run, a feedstock sample was withdrawn from the feed tank and analyzed by an outside laboratory for elemental analysis. The mass feed rate and elemental analysis allowed calculation of the influx of each relevant element to the gasifier.

#### **5.2 DRY GAS PRODUCTION RATE**

The dry gas production rate was monitored utilizing two separate methods. In the first method, an orifice meter was utilized to provide a direct measurement of the dry gas flow rate at the exit of the venturi scrubber. However, due to pressure fluctuations emanating from the bubbling fluid bed, precise determination of the orifice pressure drop was difficult. Therefore, the orifice-based reading was used primarily only as an approximate real-time indicator of the dry gas flow rate.

A second dry gas flow rate measurement technique was used based on the tracer gas method. Here, a carefully controlled quantity of nitrogen was metered to the gasifier using a mass flow meter. A dry gas sample was withdrawn for analysis in the gas chromatograph which provided a direct ratio of nitrogen to the balance of the dry gas species. With this knowledge, and



**FIGURE 5-1: PRIMARY SAMPLE COLLECTION POINTS FOR THE GASIFICATION TEST RIG**

information on the inlet nitrogen flow rate to the gasifier, the actual dry gas flow rate could be calculated as follows:

$$F_D = F_N \frac{(\sum X_i - X_N - X_O)}{X_N - 79/21 X_O}$$

where  $F_D$  = Dry Gas Flow Rate (SCFM),  
 $F_N$  = Inlet Nitrogen Tracer Flow Rate (SCFM),  
 $X_i$  = Dry Gas Composition (vol.%),  
 $X_N$  = Nitrogen Concentration (vol.%), and  
 $X_O$  = Oxygen Concentration (vol.%).

Note that the value  $79/21 X_O$  represents a correction factor to account for any air which might leak into the vacuum dry gas sample line. In general, this air leakage was negligible.

### 5.3 GAS CHROMATOGRAPH DATA

Gas chromatograph sample ports were furnished in three separate locations: the cyclone exit, the venturi scrubber exit, and the sulfur recovery column exit. During the course of a typical test run, samples were intermittently withdrawn and analyzed from each location. The sample at the cyclone exit provided direct measure of gases emanating from the gasifier and was utilized for calculating elemental material balances. The sample at the venturi scrubber exit provided evidence for determining absorption of specific components during the scrubbing stage. Finally, the sample at the sulfur recovery column exit provided information on the sulfur recovery efficiency of the packed column.

In each case, the gas samples were withdrawn utilizing a vacuum pump. The gas sample was cooled in a refrigerated coil to condense any steam contained therein prior to delivery to the gas chromatograph. In the case of the cyclone exit sample, a hot filter (5 micron) was employed to remove

particulate from the gas stream. All sample lines were purged for approximately 10 minutes prior to injection in the gas chromatograph.

The gas chromatograph was capable of separating and identifying fixed gases, hydrocarbons (through butanes), and several sulfur species ( $H_2S$ ,  $COS$ ,  $CH_3SH$ ,  $CH_3SCH_3$ ,  $SO_2$ ). A thermal conductivity detector-based (TCD) gas chromatograph was employed for several test runs. However, it was found that definitive separation of certain sulfur species from the hydrocarbon matrix was difficult in this system leading to potentially significant errors in the sulfur balance. Later, a flame photometric detector-based (FPD) gas chromatograph was procured to allow more precise and reliable characterization of sulfur components. A more detailed discussion of the gas chromatograph equipment and difficulties is found in other sections of this report.

In conjunction with the measured dry gas flow rate, the compositional data from the gas chromatograph was utilized to calculate quantitative carbon and sulfur mass rates emanating from the gasifier.

#### 5.4 TAR/OIL SAMPLE

A metered quantity of gas was withdrawn from the cyclone exit and condensed and the condensate was collected in a knock-out pot. The dry gas sample rate was measured using a wet test meter or rotameter. The knock-out pot was evacuated and the condensate was weighed and sent to an outside laboratory for analysis of total petroleum hydrocarbon (TPH). Based on knowledge of the total gasifier dry gas flow rate, the sample gas flow rate, the total condensate collected over the sample period and the condensate TPH, the total yield of condensible hydrocarbon could be calculated as follows:

$$M_{TPH} = X_{TPH} M_C \frac{F_D}{F_S}$$

where  $M_{TPH}$  = Mass Flow Rate of Condensate TPH (lb/hr),  
 $M_C$  = Condensate Flow Rate (lb/hr),  
 $X_{TPH}$  = TPH in Condensate (mass fraction),  
 $F_D$  = Dry Gas Flow Rate (SCFM), and  
 $F_S$  = Sample Gas Rate (SCFM).

## 5.5 CYCLONE CATCH SOLIDS

Cyclone catch solids were collected in a drum which was emptied and weighed at regular intervals to calculate the average catch rate. Solid samples were withdrawn and sent to an outside laboratory for elemental analysis. This information was then used to calculate the efflux of specific elements in the solid product.

## 5.6 ISOKINETIC PROBE SOLIDS (ELUTRIATE)

An isokinetic probe was fabricated in-house to calculate the entrained solids rate at the exit of the cyclone. A metered quantity of gas was withdrawn through a metal filter which collected the entrained solids. Knowledge of the entrained solids collection rate, the sample gas rate, and the total dry gas rate allowed calculation of the total entrained solids as shown here:

$$M_E = M_S \frac{F_D}{F_S}$$

where  $M_E$  = Total Entrained Solids (lb/hr),  
 $M_S$  = Entrained Solids Sample (lb/hr),  
 $F_D$  = Total Gas Flow Rate (SCFM), and  
 $F_S$  = Sample Gas Flow Rate (SCFM).

The entrained solids sample was sent to an outside laboratory for analysis. This information was used to calculate the efflux of specific elements in the entrained solids.

## 5.7 BED SOLIDS

Bed solids were withdrawn from the reactor at regular intervals using a screw valve. The solids were sent to an outside laboratory for elemental analysis. This information was used to determine the steady-state concentration of specific elements within the gasifier and to confirm achievement of steady-state operation. In addition, the bed composition was utilized in closing the sodium material balance as described in later sections.

## 5.8 SAMPLE ANALYSIS SUMMARY

Solid, gaseous, and liquid flow streams were sampled and analyzed to provide sufficient data for calculation of elemental material balances. Primary attention was focused on determination of carbon and sulfur balances. Table 5-1 summarizes the typical sample analyses performed for each sample stream.

## 5.9 MATERIAL BALANCE

Material balances were performed using measured data on input and output stream mass flow rates and compositions. Material balances were calculated for total carbon, organic carbon, total sulfur and sulfate as the available data allowed. Organic carbon was defined as total carbon minus carbonate carbon and was calculated as follows:

$$C_{\text{organic}} = C_{\text{total}} - 12/60 (\text{CO}_3)$$

As previously mentioned, product solids are represented in the material collected from both the cyclone catch and the isokinetic probe filter (elutriate). However, in general, some product solids tend also to accumulate in the fluid bed itself. This is due to the fact that the experimental gasifier is operated in a batch mode with respect to bed solids. It should be noted that a commercial reactor would be furnished with a continuous overflow port which, when operating over long periods, would prevent the net accumulation of bed material and avoid the inventory effects described above.

Determination of the quantity of product solids which have accumulated in the bed over the entire test run could be accomplished by measuring the difference between the initial and final bed mass. However, since discharge of the bed for each test run is cumbersome and inevitably involves some material losses, this method was not considered for material balance purposes.

Rather, the determination of accumulated solids was based on an indirect calculation method which assumed a 100 percent mass balance for sodium. In this method, the influx of sodium in the black liquor was compared with the efflux of sodium in the cyclone catch and isokinetic solids stream. Any

**TABLE 5-1: SAMPLE ANALYSIS SUMMARY**

<u>SAMPLE NAME</u>	<u>MASS FLOW RATE</u>	<u>VOLUMETRIC FLOW RATE</u>	<u>GAS COMPOSITION</u>	<u>MOISTURE CONTENT</u>	<u>TOTAL CARBON</u>	<u>ORGANIC CARBON<sup>(1)</sup></u>	<u>SODIUM</u>	<u>TOTAL SULFUR</u>	<u>SULFUR AS SO<sub>4</sub></u>	<u>SULFIDE</u>	<u>TPH</u>
<b>INPUT:</b>											
Black Liquor Feed	X			X	X	X	X	X	X	X	
<b>OUTPUT:</b>											
Dry Product Gas		X	X								
Cyclone Catch	X			X	X	X	X	X	X	X	
Entrained Solids	X			X	X	X	X	X	X	X	
Condensate	X										X
<b>INVENTORY:</b>											
Bed Solids					X	X	X	X	X	X	

deficit in effluent sodium was assumed to be accounted for by accumulation in the bed. Also, since the bed composition was measured, the associated accumulation of all other relevant elements could be calculated based on their ratio to sodium in the bed.

The calculation method is described below.

$$M_{Na}^i = M_{BLS} X_{Na}^{BLS}$$

$$M_{Na}^o = M_{CC} X_{Na}^{CC} + M_{IKS} X_{Na}^{IKS}$$

$$M_{Na}^A = M_{Na}^i - M_{Na}^o$$

$$M_i^A = M_{Na}^A X_i^B / X_{Na}^B$$

where  $M_{Na}^i$  = Mass Influx of Sodium (lb/hr),  
 $M_{Na}^o$  = Mass Efflux of Sodium (lb/hr),  
 $M_{Na}^A$  = Mass Accumulation of Sodium (lb/hr),  
 $M_{BLS}$  = Total Mass Influx of Black Liquor Solids (lb/hr),  
 $M_{CC}$  = Total Mass Efflux of Cyclone Catch Solids (lb/hr),  
 $M_{IKS}$  = Total Mass Efflux of Isokinetic Filter Solids (lb/hr),  
 $X_{Na}^{BLS}$  = Mass Fraction of Sodium in BLS,  
 $X_{Na}^{CC}$  = Mass Fraction of Sodium in Cyclone Catch,  
 $X_{Na}^{IKS}$  = Mass Fraction of Sodium in Isokinetic Filter Solids,  
 $M_i^A$  = Mass Accumulation of  $i^{th}$  Element (lb/hr),  
 $X_{Na}^B$  = Mass Fraction of Sodium in Bed, and  
 $X_i^B$  = Mass Fraction of  $i^{th}$  Element in Bed.



## 5.10 MATERIAL BALANCE ACCURACY

Calculation of material balances for the gasification system depends on accurate measurement of numerous input and output stream flow rates and compositions. The combination of errors associated with each independent measurement dictates the accuracy of the material balance. In most instances, the carbon material balances for the test runs conducted under this program ranged between 100 to 120 percent (defined as output divided by input). This level of accuracy is considered to be quite satisfactory given the complex nature of the effluent stream analyses.

Initial testing relied on use of thermal conductivity detector-based gas chromatography for analysis of gas product streams. During the course of testing, it was recognized that complete separation of certain sulfur compounds was difficult using the existing equipment. Since the gas phase sulfur compounds are present in concentrations of less than a few percent, interference from even small quantities of higher hydrocarbon isomers may have a significant impact on measurement accuracy. For this reason, sulfur balances during early testing deviated substantially from 100 percent closure and ranged from 82 percent to as high as 173 percent. In later tests, improved gas chromatograph procedures and the addition of a flame photometric detector-based gas chromatographic unit significantly resolved inaccuracies in the sulfur material balances.

While difficulties were encountered in closing sulfur balances for some test runs, solid phase sulfur analyses are considered to be reliable. Therefore, the calculation of sulfur gasification efficiency based on measurement of residual sulfur concentrations in the product solids is anticipated to yield accurate results, despite uncertainties which may exist in the measurement of gas phase sulfur concentrations for some runs.

## 5.11 PERFORMANCE PARAMETERS

Key performance parameters for the black liquor gasifier are organic carbon gasification efficiency and sulfur gasification efficiency. Organic carbon gasification efficiency (OCGE) is defined as follows:

$$\text{OCGE} = 100 \times \left( 1 - \frac{\sum M_{\text{OC}}^i}{M_{\text{BLS}}^{\text{OC}}} \right)$$

where  $M_{\text{OC}}$  = Mass Organic Carbon in BLS, and  
 $M_{\text{OC}}^i$  = Mass Organic Carbon in  $i^{\text{th}}$  Solid Effluent.

It should be mentioned that the theoretical gasification limit (100%) will still result in carbon in the product solids due to the formation of sodium carbonate ( $\text{Na}_2\text{CO}_3$ ). Therefore, it is not appropriate to define gasification efficiency by calculating the ratio of carbon in the product gases to carbon in the liquor feed. Instead, measurement of organic carbon remaining in all solid products should be used as the basis for calculating gasification efficiency, such as in the method described above.

In addition, a normalized organic carbon gasification efficiency (NOCGE) is calculated by normalizing the organic carbon material balance to 100 percent as follows:

$$\text{NOCGE} = 100 \times \left( 1 - \frac{\sum M_{\text{OC}}^i / F_{\text{OC}}}{M_{\text{BLS}}^{\text{OC}}} \right)$$

where  $F_{\text{OC}}$  = Ratio Of Organic Carbon In All Effluent Streams To That In Feed Stream.

Sulfur gasification efficiencies were calculated, where applicable, along similar lines as shown:

$$SGE = 100 \times \left(1 - \frac{\sum M_S^i}{M_S^{BLS}}\right)$$

and

$$NSGE = 100 \times \left(1 - \frac{\sum M_S^i}{M_S^{BLS}}\right)$$

where SGE = Sulfur Gasification Efficiency, and  
NSGE = Normalized Sulfur Gasification Efficiency.

Note that when material balance closure is in the range of 80 to 120 percent, the difference between the normalized and unnormalized efficiency parameters is typically less than 2 to 3 percent.

**SECTION 6.0**  
**EXPERIMENTAL TEST RESULTS**

The following section provides a comprehensive summary of experimental test runs conducted during the current program. In Section 6.1, the spent liquor feedstocks employed in the test runs are described. In Section 6.2, reduced data for each sequential test run is presented. In Section 6.3, results from scrubber verification tests are discussed in detail. And, in Section 6.4, gasifier and system performance is summarized with respect to the intended objectives of the overall test program.

**6.1 FEEDSTOCK**

Two separate Kraft black liquor feedstocks and one Neutral Sulfite Semichemical (NSSC) liquor feedstock were utilized during the current test program. The first Kraft feedstock comprises a liquor supplied from Weyerhaeuser's Everett, Washington mill. The second Kraft feedstock comprises a liquor supplied from Weyerhaeuser's Longview, Washington cross recovery mill. The NSSC feedstock comprises a liquor supplied from Weyerhaeuser's North Bend, Oregon mill. Elemental analysis for each liquor is provided in Table 6-1.

**TABLE 6-1: SPENT LIQUOR ANALYSES**

SOLIDS, Wt.% HHV, Btu/lb SOLIDS	56.6 6374	65.4 6647	36.6 5450
<u>ELEMENT (Wt.% O.D Basis)</u>	<u>EVERETT</u> (Kraft)	<u>LONGVIEW</u> (Cross Recovery)	<u>NORTH BEND</u> (NSSC)
Sodium	18.89	17.40	17.60
Potassium	0.62	1.90	0.52
Carbon	38.08	38.40	32.15
Hydrogen	4.02	3.99	4.10
Nitrogen	0.07	0.11	0.40
Sulfur	3.60	4.50	8.17
Chloride	0.35	0.58	0.11
Oxygen (By Diff.)	34.38	33.10	36.95
Sulfate, SO <sub>4</sub>	1.74	0.54	7.58
Sulfide, S	0.49	1.00	0.00
Carbonate, CO <sub>3</sub>	4.66	3.18	1.64

For each separate liquor type, samples were withdrawn directly from the liquor feed tank during the course of a test run. Note that the solids content of the Everett Kraft liquor (56.6%) and the North Bend NSSC liquor (36.6%) appear to be low compared to typical mill run specifications (67.4% and 42.5%, respectively). Since liquor samples are collected from the surface of the liquor storage tank, it is possible that segregation of solids to the lower portions of the tank might explain the discrepancies in measured solids content, compared to mill run liquor. Also, in the case of the Kraft liquors, heated dilution water is added to the metering tank during start-up in order to preheat feed lines and prevent plugging. This dilution water may have contributed to the lower solids content for the sampled liquor.

It should also be noted that the material balances presented in the following sections typically reflect output values in excess of 100 percent of input. This tends to corroborate the hypothesis that the sampled liquor solids content is artificially low compared to the true mean solids content of the bulk liquor as fed to the gasifier. In fact, in most cases, the discrepancy in solids content accounts quite closely for deviations in the total carbon elemental balance. For this reason, normalization of the experimental results according to the methods described in Section 5.11 appears to be justified for accurate and meaningful reduction of the data.

## **6.2 TEST DATA**

Data from sequential gasification tests performed to date are described below.

### **6.2.1 KRAFT TEST RUN (1/24/90)**

Gasification tests were conducted using Everett Kraft liquor on January 24, 1990. The gasifier was operated for a period of approximately 9 hours during which time a total of 264 pounds of liquor (wet basis) was fed. The liquor feed rate was approximately 18 lb/hr from 12:00 to 14:00, increased to 23 lb/hr from 14:00 to 19:00, and further increased to over 40 lb/hr from 19:00 to 21:30, after which the run was terminated. The fluidization steam rate was maintained at an approximately constant level of 35 lb/hr throughout the run. The average gasifier temperature was 1130°F for the first two feed

rates over the first seven hours and 1050°F for the remaining two hours. Table 6-2 summarizes the average feed and effluent flow rates during the period from 14:00 to 19:00.

**TABLE 6-2: MATERIAL FLOW SUMMARY FOR 1/24/90 TEST RUN**

INPUT:

Black Liquor Feed (lb/hr Wet)	23.0
Black Liquor Feed (lb/hr Dry)	13.0
Steam Feed (lb/hr)	35.0

OUTPUT:

Cyclone Catch (lb/hr)	3.0
Elutriate (lb/hr)	0.40
Product Gas (SCFM)	4.29

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Elemental analyses for solid samples collected during the course of the test run are summarized in Table 5-3. As seen in the table, carbon and sulfur concentrations in the bed and cyclone catch material increased monotonically during the test run suggesting that steady-state operation was not fully established. The dynamic behavior of the fluid bed was consistent with the fact that the feed rate was incrementally increased throughout the test period.

While the lack of steady-state data prevents meaningful evaluation of gasifier material balances, the test run provided verification of gasifier reliability over a wide range of capacity.

Typical gas analyses for the test run are summarized in Table 6-4. The product gas exhibits a high concentration of hydrogen (64-68%) and only modest quantities of methane, carbon monoxide and higher hydrocarbons. Gas analyses indicate that sulfur is released primarily as hydrogen sulfide (1.2-1.8%). Only trace quantities of carbonyl sulfide and methyl mercaptan were observed. Note that initial analysis suggested the potential presence of unexpectedly high levels of SO<sub>2</sub>. However, later investigations revealed that separation of SO<sub>2</sub> from C<sub>4</sub><sup>+</sup> isomers and water vapor was imperfect, leading to misrepresentation of the SO<sub>2</sub> peak. Based on more precise measurement of sulfur gas

**TABLE 6-3: ELEMENTAL ANALYSIS OF SOLIDS FOR 1/24/90 TEST RUN**

<u>SAMPLE DESIGNATION</u>	BL FEED RATE (lb/hr)	(AS REC'D →)		ORGANIC CARBON C%	SODIUM Na%	TOTAL SULFUR %
		TOTAL CARBON C%	CARBONATE CO <sub>3</sub> %			
<u>BED SOLIDS:</u>						
012490-W-EV-1-1355-BS	18	12.00	55.53	0.89	41.40	0.12
012490-W-EV-1-1500-BS	23	12.40	55.38	1.32	41.40	0.16
012490-W-EV-1-1730-BS	23	13.70	54.96	2.71	39.90	0.23
012490-W-EV-1-1830-BS	23	14.70	54.89	3.72	38.95	0.27
012490-W-EV-1-2130-BS	40	19.90	48.57	10.19	37.10	0.54
<u>CYCLONE SOLIDS:</u>						
012490-W-EV-1-1304-CL	18	12.90	57.52	1.40	38.70	0.12
012490-W-EV-1-1700-CL	23	13.90	56.49	2.60	38.70	0.22
012490-W-EV-1-1820-CL	23	18.10	50.74	7.95	36.60	0.68
012490-W-EV-1-2030-CL	40	23.00	48.39	13.32	35.30	0.86

**TABLE 6-4: GAS ANALYSES FOR 1/24/90 TEST RUN**

Time	16:15	16:42	17:18	17:25	18:11	18:44	19:05
Gasifier Temp. (°F)	1116	1127	1135	1134	1132	1130	1129
<u>Components (Vol.%)</u>							
H <sub>2</sub>	64.618	65.598	66.546	66.879	66.212	67.529	67.542
CH <sub>4</sub>	2.116	1.849	1.498	1.507	1.500	1.595	1.450
CO	2.099	2.086	1.820	1.756	1.690	1.766	1.684
CO <sub>2</sub>	27.820	26.367	26.962	26.565	26.444	25.704	26.081
Ethane	0.175	0.000	0.000	0.000	0.425	0.000	0.000
Ethylene	0.108	1.007	0.077	0.000	0.368	0.355	0.165
Propane	0.119	0.132	0.144	0.120	0.230	0.248	0.256
Propylene	0.177	0.190	0.175	0.234	0.229	0.253	0.274
I-Butane	0.148	0.145	0.142	0.154	0.185	0.188	0.193
H <sub>2</sub> S	1.726	1.669	1.690	1.798	1.655	1.215	1.200
COS	0.000	0.013	0.000	0.000	0.013	0.000	0.000
CH <sub>3</sub> SH	0.010	0.013	0.012	0.014	0.000	0.015	0.014
C <sub>4</sub> <sup>+</sup> /H <sub>2</sub> O	0.885	0.933	0.934	0.973	1.051	1.132	1.141
TOTAL	100.000	100.000	100.000	100.000	100.000	100.000	100.000

release from similar liquors in subsequent runs using a FPD-based gas chromatograph, it was quite conclusively determined that SO<sub>2</sub> is not present in gas products from Kraft liquors in any appreciable quantity. For this reason, SO<sub>2</sub> was assumed to be absent in the co-eluted peak and therefore did not contribute to the sulfur material balance.

### **6.2.2 KRAFT TEST RUN (2/23/90)**

Gasification tests were conducted using Longview spent liquor on February 23, 1990. The gasifier was operated for a period of approximately 8 hours during which time a total of 167.8 pounds of spent liquor (wet basis) was fed. The average liquor feed rate during the steady-state period was 19.3 lb/hr (wet) and the fluidization steam rate was approximately constant at 38.2 lb/hr. The average gasifier temperature was 1145°F.

A material flow summary during steady-state conditions is shown in Table 6-5.

**TABLE 6-5: MATERIAL FLOW SUMMARY FOR 2/23/90 TEST RUN**

INPUT:

Black Liquor Feed (lb/hr Wet)	19.30
Black Liquor Feed (lb/hr Dry)	12.66
Steam Feed (lb/hr)	38.20

OUTPUT:

Cyclone Catch (lb/hr)	2.60
Elutriate (lb/hr)	0.19
Product Gas (SCFM)	4.90

---

Elemental analyses for solid samples collected during the course of the test run are summarized in Table 6-6. As seen in the table, carbon, total sulfur, and sulfate concentrations in the bed solids and cyclone catch exhibit constant or asymptoting values indicative of steady-state operation. This is further illustrated in Figures 6-1 and 6-2. Note that the elutriate sample was insufficient in size to allow a complete solids analysis.



**TABLE 6-6: ELEMENTAL ANALYSIS OF SOLIDS FOR 2/23/90 TEST RUN**

(AS REC'D →)

<u>SAMPLE DESIGNATION</u>	<u>SOLIDS %</u>	<u>TOTAL CARBON C%</u>	<u>CARBONATE CO<sub>3</sub>%</u>	<u>ORGANIC CARBON C%</u>	<u>SODIUM Na%</u>	<u>TOTAL SULFUR %</u>	<u>SULFATE SO<sub>4</sub>%</u>	<u>SULFIDE S%</u>
<u>BED SOLIDS:</u>								
0222390-W-LV-1-1340-BS	99.59	13.77	55.14	2.74	37.30	0.15	0.11	N/A
0222390-W-LV-1-1700-BS	99.64	15.87	53.85	5.10	35.20	0.17	0.11	N/A
0222390-W-LV-1-1800-BS	99.49	16.32	54.46	5.43	35.10	0.27	0.11	N/A
0222390-W-LV-1-1855-BS	99.60	16.31	52.02	5.91	36.30	0.29	0.11	N/A
0222390-W-LV-1-2000-BS	99.64	17.04	52.28	6.58	37.40	0.29	0.11	N/A
<u>CYCLONE SOLIDS:</u>								
0222390-W-LV-1-1433-CL	89.84	20.25	40.03	12.24	31.00	1.07	2.12	N/A
0222390-W-LV-1-1603-CL	88.99	20.94	41.06	12.73	31.40	1.38	2.48	N/A
0222390-W-LV-1-1800-CL	85.13	21.54	36.87	14.17	30.30	1.70	3.25	N/A
0222390-W-LV-1-1900-CL	81.21	21.83	36.78	14.47	27.70	1.52	2.76	N/A
0222390-W-LV-1-2000-CL	83.30	21.59	37.95	14.00	28.90	1.53	2.82	N/A

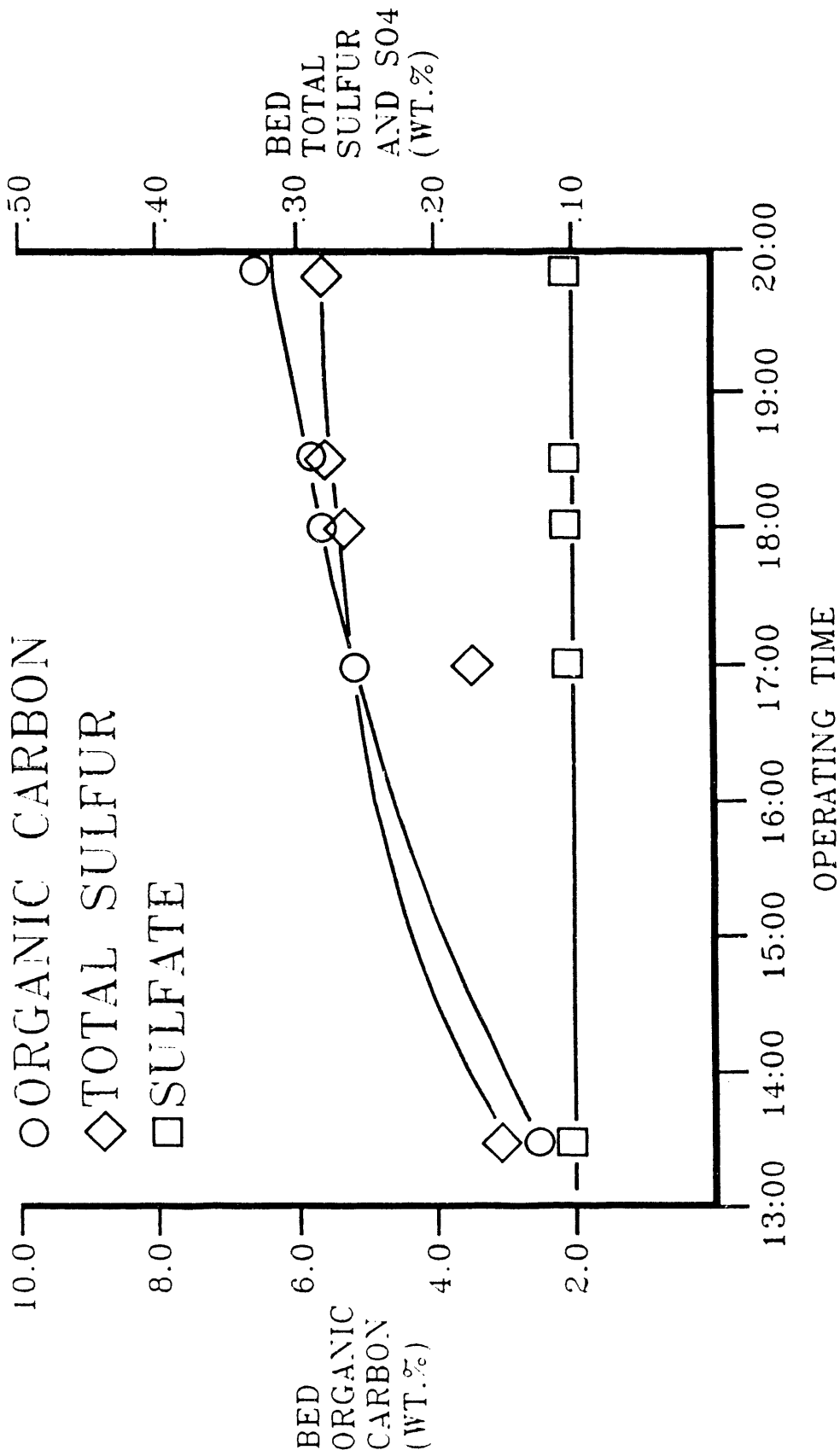


FIGURE 6-1: COMPOSITION OF BED SOLIDS DURING TEST RUN

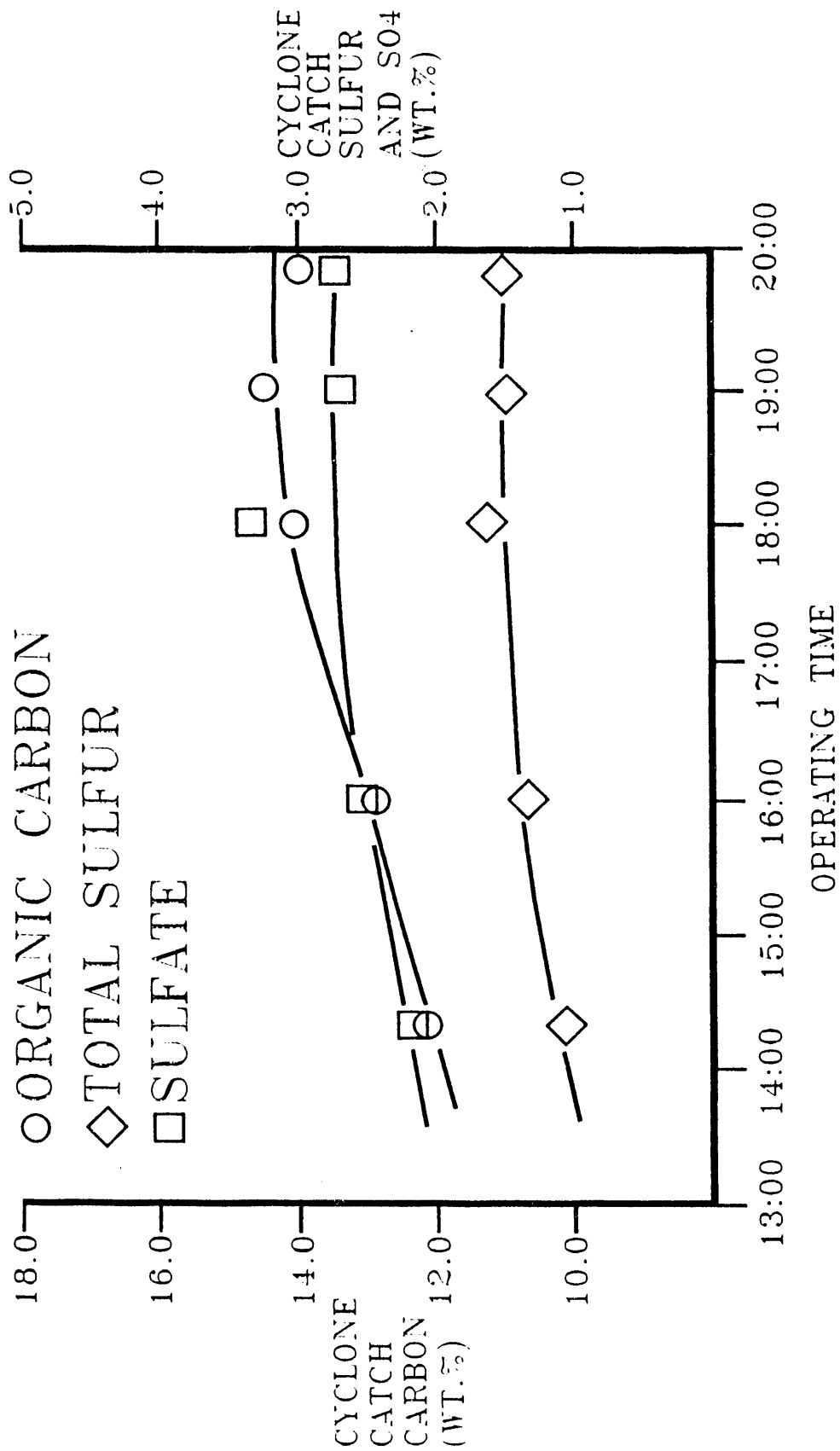


FIGURE 6-2: COMPOSITION OF CYCLONE CATCH SOLIDS DURING TEST RUN

**Table 6-7** summarizes the average elemental concentration for solids taken during the steady-state period. The steady-state period was defined as the operating time between 17:00 and 20:00. These values were utilized in calculation of gasifier material balances.

**TABLE 6-7: AVERAGE SOLIDS CONCENTRATION DURING STEADY-STATE PERIOD FOR 2/23/90 TEST RUN**

(AS REC'D →)

	TOTAL CARBON % C	CARBONATE % CO <sub>3</sub>	ORGANIC CARBON % C	SODIUM % Na	TOTAL SULFUR %	SULFATE % SO <sub>4</sub>	SULFIDE % S
BED SOLIDS	16.56	52.92	5.97	36.27	0.28	0.11	N/A
CYCLONE	21.65	37.20	14.21	28.97	1.58	2.94	N/A
ELUTRIATE	N/A	N/A	N/A	N/A	N/A	N/A	N/A

**Table 6-8** summarizes gas analyses collected during the steady-state period. These gas analyses were collected at the hot cyclone exit prior to product gas quenching and scrubbing. The gas analyses again confirm the presence of high concentrations of hydrogen (55-60 vol.%) and modest quantities of methane (approximately 2.0 wt.%), carbon monoxide (approximately 3.0 vol.%), and higher hydrocarbons (less than 2.0 vol.% total). The average gas yield during steady-state was 4.90 SCFM.

The gas analyses further support the conclusion that reduced sulfur species other than H<sub>2</sub>S are present in negligible quantities. Hydrogen sulfide concentrations ranged from 1.2 to 1.6 vol.% and represented the primary gaseous sulfur product. As previously discussed, the TCD-based gas chromatograph utilized during this test run was incapable of differentiating individual components of the co-eluted SO<sub>2</sub>/C<sub>4</sub><sup>+</sup>/H<sub>2</sub>O peak. Also as discussed, subsequent test runs confirmed that SO<sub>2</sub> is not present in the co-eluted peak in any appreciable quantity and therefore can be ignored in calculating sulfur material balances.

**TABLE 6-8: GAS ANALYSES FOR 2/23/90 TEST RUN (CYCLONE EXIT)**

Time	17:11	17:24	17:34	17:55	18:04	19:18	19:26
Gasifier Temp. (°F)	1142.0	1141.0	1144.0	1145.0	1146.0	1146.0	1148.0
Gas Rate (SCFM)	4.730	4.943	4.569	4.731	4.927	4.934	5.300
<u>Components (Vol.%)</u>							
H <sub>2</sub>	56.191	55.662	59.746	58.785	57.622	58.735	59.343
CH <sub>4</sub>	1.945	2.080	2.159	2.006	1.850	1.860	1.746
CO	2.815	2.880	3.060	2.928	2.832	2.950	2.873
CO <sub>2</sub>	33.733	34.278	29.331	30.309	31.692	29.931	29.923
Ethane	0.488	0.657	0.533	0.541	0.507	0.546	0.609
Ethylene	0.276	0.291	0.323	0.324	0.273	0.375	0.305
Propane	1.558	1.375	1.666	1.748	1.789	1.974	1.850
Propylene	0.752	0.602	0.782	0.802	0.828	0.892	0.731
I-Butane	0.693	0.644	0.737	0.845	0.847	0.753	0.713
H <sub>2</sub> S	1.149	1.165	1.309	1.362	1.430	1.639	1.607
COS	0.025	0.000	0.034	0.046	0.000	0.043	0.000
CH <sub>3</sub> SH	0.000	0.000	0.000	0.000	0.000	0.000	0.000
C <sub>4</sub> <sup>+</sup> /H <sub>2</sub> O	0.377	0.366	0.321	0.304	0.332	0.302	0.300
TOTAL	100.000	100.000	100.000	100.000	100.000	100.000	100.000

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The absence of higher hydrocarbons in any significant quantity in the gas product suggest that the production of heavier tars and oils is negligible. To confirm this, samples of the gasifier product condensate were analyzed for total petroleum hydrocarbon. The results verified that extremely low levels of hydrocarbons (8 mg/L) were present in the condensate stream. This result is somewhat striking considering that gasification of biomass materials, such as wood chips, commonly yield TPH levels in excess of several thousand parts per million (mg/L). The low tar/oil yield from spent liquor is believed to be due, in part, to the highly catalytic nature of the sodium salts contained within the spent liquor which promotes steam cracking and gasification. Due to the extremely low tar/oil yield resulting from gasification of spent liquor, unconverted carbon in the tar/oil product can be ignored during preparation of material balances without impact on material balance accuracy.

Based on the available data, a detailed material balance was calculated as shown in Table 6-9 using methods described in Section 5.0. Recall that the material balance methodology includes bed accumulation/depletion effects based on the criterion of 100 percent sodium recovery. As seen in Table 6-9, the organic carbon material balance is quite excellent at 109 percent. The total sulfur material balance is calculated to be 82 percent.

Also note that due to the absence of elemental analysis for the elutriate, the contribution of this effluent is not included in the material balance. However, based on typical elemental analyses for elutriate from other test runs, the net contribution of this stream to the organic carbon material balance is anticipated to be less than 2 to 3 percent.

Also shown in Table 6-9 are the performance parameters for the gasifier. The unnormalized gasification efficiency is shown to be 87.2 percent. The normalized organic carbon efficiency is 88.2 percent. The unnormalized sulfur gasification efficiency is calculated to be 91.2 percent. The normalized sulfur gasification efficiency is 89.3 percent.

**TABLE 6-9: MATERIAL BALANCE SUMMARY FOR 2/23/90 TEST RUN**

	<u>TOTAL CARBON (lb/hr)</u>	<u>ORGANIC CARBON (lb/hr)</u>	<u>SODIUM (lb/hr)</u>	<u>TOTAL SULFUR (lb/hr)</u>	<u>SULFATE (SO<sub>4</sub>) (lb/hr)</u>
Liquor Feed	4.86	4.78	2.20	0.57	0.07
Cyclone Catch	0.56	0.37	0.75	0.04	0.08
Elutriate	N/A	N/A	N/A	N/A	N/A
Bed ACC/DEP	0.66	0.24	1.45	0.01	0.00
TOTAL SOLIDS	1.22	0.61	2.20	0.05	0.08
Gas Product	4.58	4.58	0.00	0.42	0.00
TOTAL GAS PLUS SOLID	5.80	5.19	2.20	0.47	0.08
Out/Input	1.20	1.09	1.00	0.82	1.14

PERFORMANCE PARAMETERS (SOLIDS BASIS):

	<u>UNNORMALIZED</u>	<u>NORMALIZED</u>
Organic Carbon Gasification Efficiency (%)	87.2	88.2
Sulfur Gasification Efficiency (%)	91.2	89.3

---

Note that the material balance for the Longview liquor test run suggests that no appreciable direct reduction of sulfate contained in the initial feedstock occurred. However, it should be recognized that due to the very low concentration of sulfate in the liquor feed (0.54% accounting for only 4% of total sulfur), accurate evaluation of the sulfate reduction potential in the gasifier is impossible. Later tests conducted using liquor feedstocks containing higher initial sulfate levels, particularly the NSSC liquors, do support the occurrence of direct sulfate reduction.

### GAS RECYCLE TO PULSE COMBUSTOR

In the integrated commercial recovery system, a portion of the gasifier product gas is returned to the pulse combustors to self-satisfy the heat demand of the reactor. However, until the current test, the bench-scale pulse combustor module has been fired only on natural gas.

Under the initial scope of work for this contract, simulated gas was to be tested in a pulse combustor to ensure that the unit is capable of operating on gas characteristic of that generated from the gasifier. However, since it was felt that a more convincing demonstration would involve combustion of actual product gas emanating from the experimental reactor, this approach was selected.

Under normal circumstances, the product gases are combusted in an incinerator using a significant pilot support flame. In order to ensure the flammability of the product gases during unsupported combustion prior to testing in a pulse combustor, the incinerator support flame was shut off. Continued combustion intensity within the incinerator was observed indicating that the fuel gas was suitable for testing in the pulse unit.

The fuel gas was then ignited in a pulse combustor firing at a rate of approximately 80,000 Btu/hr. The pulse combustor lighted off without difficulty and operated with excellent turndown performance. This test confirmed the ability to operate the pulse combustor in a self-sustaining manner using fuel gas generated from the gasifier reactor.



### 6.2.3 KRAFT TEST RUN (3/7/90)

Gasification tests were conducted using Everett Kraft liquor on March 7, 1990. The gasifier was operated for a period of approximately 7.5 hours during which time a total of 188.4 pounds of liquor (wet) was fed. The liquor feed and steam rate were 27.1 lb/hr and 33.7 lb/hr, respectively, during the steady-state period. The average gasifier temperature was 1150°F.

A material flow summary for the steady-state period is shown in Table 6-10.

**TABLE 6-10: MATERIAL FLOW SUMMARY FOR 3/7/90 TEST RUN**

INPUT:

Black Liquor Feed (lb/hr Wet)	27.1
Black Liquor Feed (lb/hr Dry)	15.3
Steam Feed (lb/hr)	33.7

OUTPUT:

Cyclone Catch (lb/hr)	2.27
Elutriate (lb/hr)	0.46
Product Gas (SCFM)	5.59

---

Elemental analyses for solid samples collected during the test run are summarized in Table 6-11. As seen in the table, in most instances, the elemental compositions indicate definitive attainment of a steady-state condition, with organic carbon in the bed solids appearing to be the only exception. Figures 6-3 and 6-4 illustrate the product solid composition as a function of run time. Based on these figures, the operating period between 15:00 and 17:30 gives an excellent characterization of the steady-state performance under the selected gasifier conditions. Average product solid compositions used for calculation of material balances during the steady-state period are shown in Table 6-12. It is interesting to note that virtually no sulfides were observed in any of the solid products. This result is consistent with thermodynamic predictions as well as with prior data collected on similar liquors (MTCI, "Testing of An Advanced Thermochemical Reactor").

**TABLE 6-11: ELEMENTAL ANALYSIS OF SOLIDS FOR 3/7/90 TEST RUN**

(AS REC'D →)

<u>SAMPLE DESIGNATION</u>	<u>SOLIDS %</u>	<u>TOTAL CARBON C%</u>	<u>CARBONATE CO<sub>3</sub>%</u>	<u>ORGANIC CARBON C%</u>	<u>SODIUM Na%</u>	<u>TOTAL SULFUR %</u>	<u>SULFATE SO<sub>4</sub>%</u>	<u>SULFIDE S%</u>
<u>BED SOLIDS:</u>								
030790-W-EV-1-1300-BS		13.18	53.73	2.43	41.20	0.31	0.16	0.06
030790-W-EV-1-1500-BS		14.44	53.25	3.79	39.60	0.41	0.18	0.06
030790-W-EV-1-1700-BS		15.93	52.34	5.46	39.40	0.31	0.19	0.07
<u>CYCLONE SOLIDS:</u>								
030790-W-EV-1-1530-CL	88.49	24.67	35.19	17.64	27.10	2.50	7.16	0.01
030790-W-EV-1-1630-CL	89.41	25.21	35.25	18.16	28.40	2.43	6.68	0.01
030790-W-EV-1-1730-CL	86.97	23.95	35.58	16.84	26.40	2.54	6.83	0.00
<u>ISO PROBE FILTER SOLIDS:</u>								
030790-W-EV-1-1730-IKS		71.70	5.99	70.50	6.81	2.83	1.53	0.00
<u>H<sub>2</sub>S SCRUBBER LIQUID:</u>								
030790-W-EV-1-1745-SCL			8.83		6.04	0.62	0.33	0.53

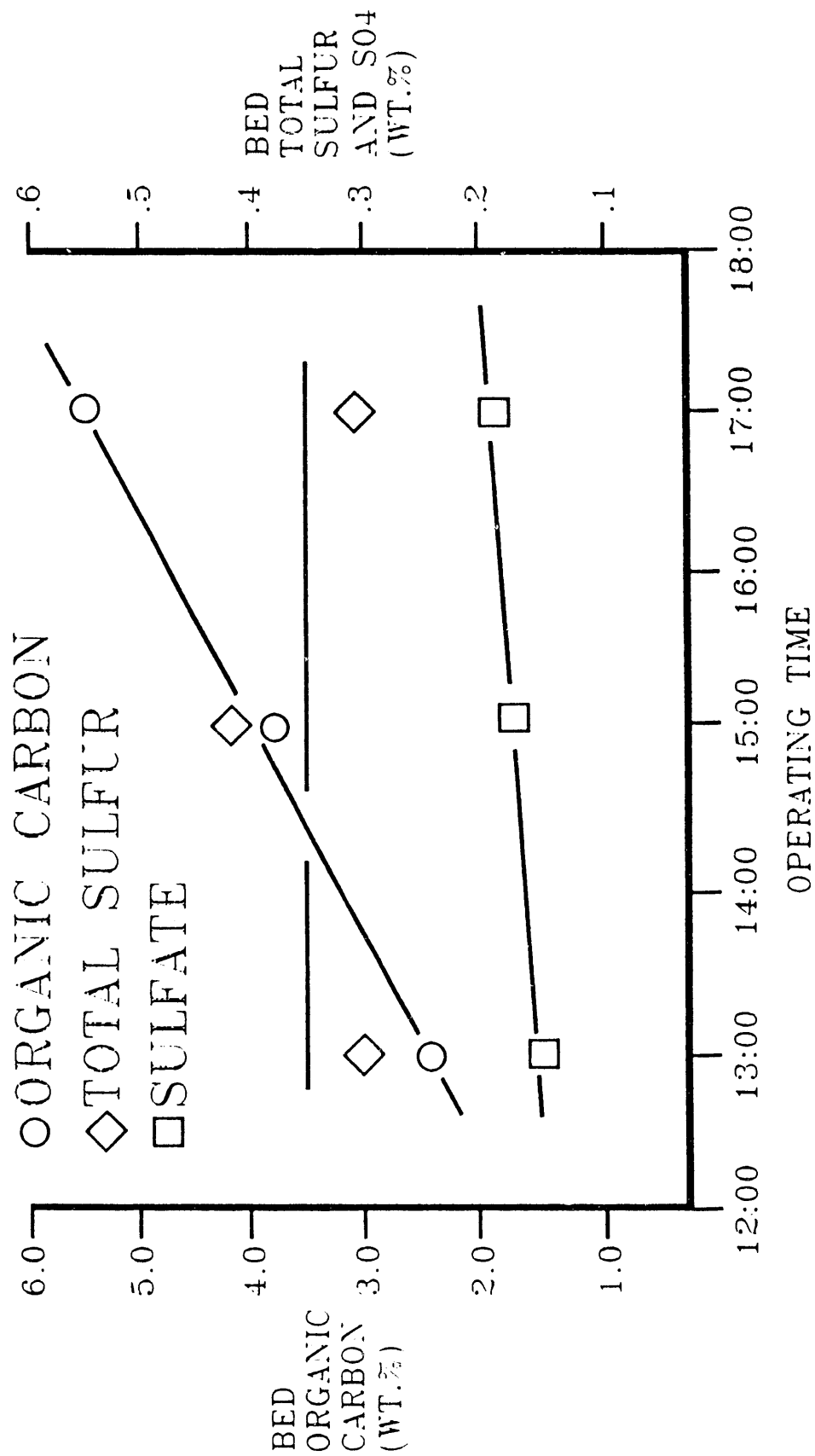
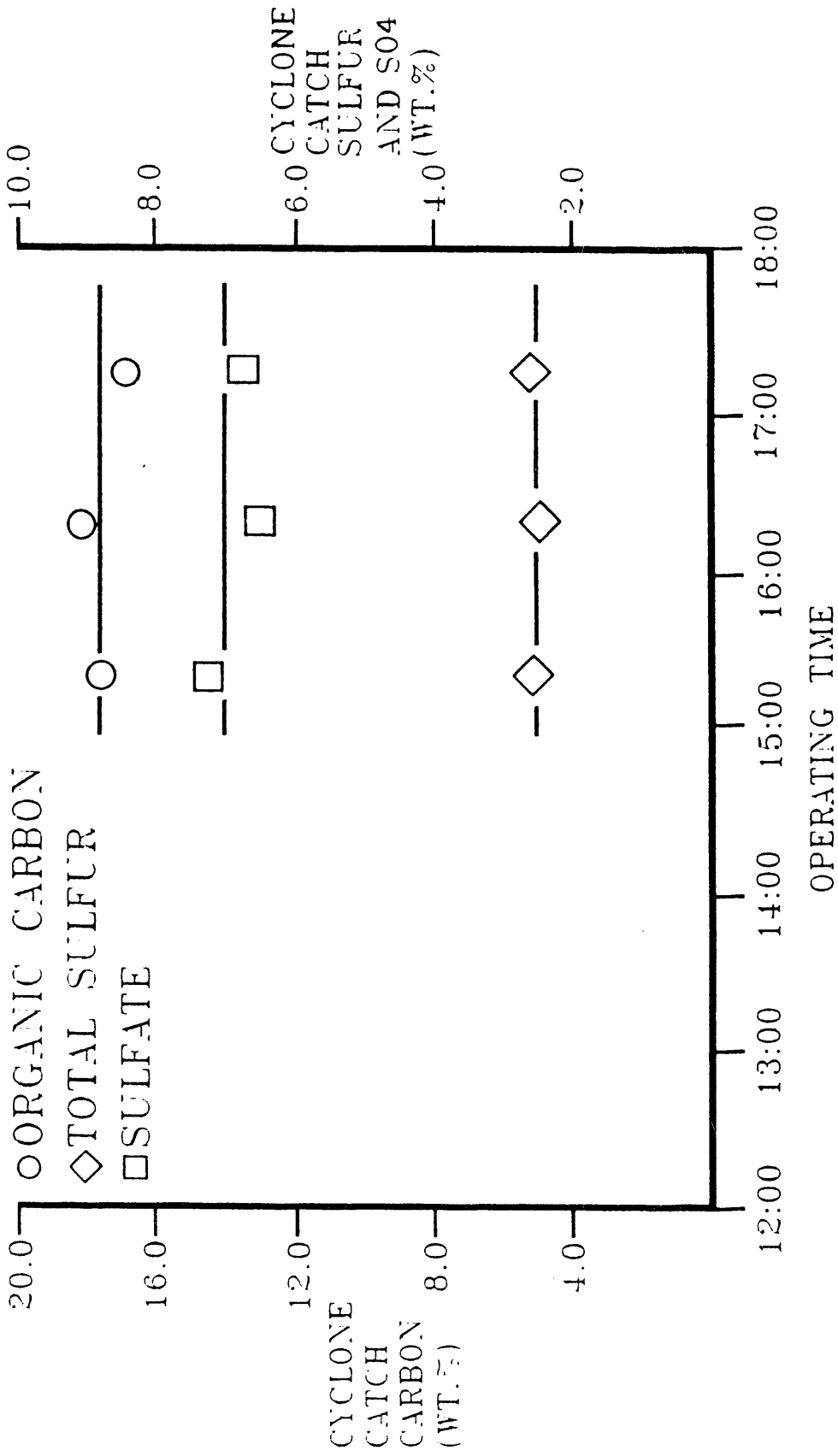


FIGURE 6-3: BED SOLIDS COMPOSITION DURING TEST RUN



**FIGURE 6-4: CYCLONE CATCH SOLIDS COMPOSITION DURING TEST RUN**

**TABLE 6-12: AVERAGE SOLIDS CONCENTRATION DURING STEADY-STATE PERIOD FOR 3/7/90 TEST RUN**

(AS REC'D →)

	TOTAL CARBON % C	CARBONATE % CO <sub>3</sub>	ORGANIC CARBON % C	SODIUM % Na	TOTAL SULFUR %	SULFATE % SO <sub>4</sub>	SULFIDE % S
BED SOLIDS	15.18	52.80	4.63	39.50	0.36	0.19	0.07
CYCLONE	24.61	35.34	17.55	27.30	2.49	6.89	0.01
ELUTRIATE	71.70	5.99	70.50	6.81	2.83	1.53	0

It is interesting to note that the elutriate material has a very high organic carbon content (70.5%). In fact, the ratio of organic carbon to sodium is almost five times higher than the original black liquor feed material. This trend is reproduced in several of the test runs as reported in the following sections.

The elutriate material represents the finest sized solids which are not easily captured in the cyclone. It was originally thought that these fine solids resulted from natural attrition processes occurring between carbonate particles contained within the fluid bed. If this were the case, then the elutriate particles should tend to have lower carbon to sodium ratios than the liquor feed, since attrition would randomly occur after some finite level of gasification.

However, the enrichment of carbon in these solids suggest some potential mechanism of hydrocarbon condensation or soot formation. Under this hypothesis, small nucleation centers entrained within the product gases might serve as catalytic substrates for hydrocarbon cracking, thereby explaining the enrichment phenomena.

A second explanation might simply be due to the elutriate sampling method. Recall that the elutriate is collected using an isokinetic probe attached to a sintered metal filter. As gas is drawn through the filter, collected particles, having significant residence time on the filter surface,

may accumulate condensible tars and oils. In contrast, under normal operating conditions, these particles would pass through the hot reactor system (including cyclones and transfer lines) in a matter of only several seconds before being quenched in the venturi scrubber. In this case, tars and oils would primarily be collected in the condensate. However, from a material balance perspective, it is irrelevant to distinguish tars and oils collected in the condensate from tars and oils collected on the elutriates material.

Gas analyses for samples drawn from the cyclone exit are summarized in **Table 6-13**. The gas analyses are generally similar in nature to those recorded for the Everett liquor gasification run on January 24, 1990. Minor differences in gas composition for the March 7, 1990 test run include a somewhat reduced hydrogen yield and slightly increased higher hydrocarbon yield.

In addition, hydrogen sulfide concentrations during the steady-state period ranged from 2.5 to 3.5 vol.%. This compares with values of only 1.2 to 1.8 vol.% for the prior test run on January 24, 1990. Theoretical calculations of gasifier material balances indicate that the total sulfur concentration in the dry gas should be approximately 2 vol.%. Therefore, the unusually high concentrations of  $H_2S$  measured during this test appears to be due to a calibration error, interference with other hydrocarbon gases, or some other currently unexplained reason. For this reason, as seen in the following section, material balance closure for sulfur based on gas phase data is quite poor for this test run.

**Table 6-14** summarizes the material balances for the March 7, 1990 test run. Material balance closure for organic carbon is excellent at 104 percent. However, as previously discussed, significant discrepancies occur for sulfur material balances (173%). Unnormalized and normalized organic carbon gasification efficiencies are 82.6 percent and 83.3 percent, respectively. Sulfur gasification efficiency is found to be 83.6 percent and 90.5 percent, respectively. Recall that this value is independent from the suspect gas phase analyses.

**TABLE 6-13: GAS ANALYSIS FROM CYCLONE EXIT FOR 3/7/90 TEST RUN**

	13:48	13:53	14:42	14:47	15:07	15:12	15:17	16:29	16:34	16:39
TIME	1140	1142	1144	1146	1151	1151	1150	1150	1151	1152
GASIFIER TEMP (°F)	4.988	4.712	4.839	4.579	5.137	5.186	5.155	6.187	6.152	5.755
GAS RATE (SCFM)										
<u>COMPONENTS (Vol.%)</u>										
H <sub>2</sub>	58.855	58.997	58.541	58.827	59.019	59.433	60.551	55.348	56.127	57.915
CH <sub>4</sub>	2.359	2.462	2.509	2.524	2.400	2.360	2.408	2.039	2.071	2.126
CO	2.925	3.040	2.864	2.917	3.192	3.129	3.227	2.753	2.805	2.957
CO <sub>2</sub>	31.451	31.242	30.853	30.428	30.012	30.609	29.107	34.097	34.142	32.683
Ethane	0.321	0.291	0.344	0.322	0.310	0.266	0.271	0.252	0.278	0.389
Ethylene	0.430	0.440	0.413	0.379	0.417	0.276	0.387	0.431	0.440	0.410
Propane	0.631	0.670	0.672	0.637	0.577	0.590	0.632	0.652	0.725	0.708
Propylene	0.462	0.429	0.442	0.397	0.361	0.372	0.443	0.394	0.422	0.483
I-Butane	0.233	0.220	0.203	0.205	0.208	0.215	0.201	0.172	0.197	0.153
H <sub>2</sub> S	1.403	1.273	2.300	2.563	3.779	2.749	2.773	3.091	2.786	2.167
COS	0.009	0.009	0.000	0.000	0.000	0.000	0.000	0.000	0.006	0.009
CH <sub>3</sub> SH	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
SO <sub>2</sub> /C <sub>4</sub> <sup>+</sup> /H <sub>2</sub> O	0.921	0.927	0.859	0.800	0.724	0.000	0.000	0.770	0.000	0.000
Total	100.000	100.000	100.000	100.000	100.000	100.000	100.000	100.000	100.000	100.000

**TABLE 6-14: MATERIAL BALANCE SUMMARY FOR 3/7/90 TEST RUN**

	<u>TOTAL CARBON (lb/hr)</u>	<u>ORGANIC CARBON (lb/hr)</u>	<u>SODIUM (lb/hr)</u>	<u>TOTAL SULFUR (lb/hr)</u>	<u>SULFATE (SO<sub>4</sub>) (lb/hr)</u>
Liquor Feed	5.78	5.64	2.87	0.55	0.26
Cyclone Catch	0.56	0.40	0.62	0.06	0.16
Elutriate	0.33	0.32	0.03	0.01	0.01
Bed ACC/DEP	0.85	0.26	2.22	0.02	0.01
TOTAL SOLIDS	1.74	0.98	2.87	0.09	0.18
Gas Product	4.89	4.89	0.00	0.86	0.00
TOTAL GAS PLUS SOLID	6.63	5.87	2.87	0.95	0.18
Out/Input	1.15	1.04	1.00	1.73	0.69

PERFORMANCE PARAMETERS (SOLIDS BASIS):

	<u>UNNORMALIZED</u>	<u>NORMALIZED</u>
Organic Carbon Gasification Efficiency (%)	82.6	83.3
Sulfur Gasification Efficiency (%)	83.6	90.5



Note that only 69 percent of the feed liquor sulfate could be accounted for by the gasifier solid effluents. This result suggests that 31 percent of the feed sulfate was directly reduced to sulfur containing product gas species.

### H<sub>2</sub>S RECLAIMING IN GREEN LIQUOR

The gas analyses exhibited in Table 6-14 represent samples withdrawn directly from the hot cyclone exit. Table 6-15 summarizes gas samples collected at the exit of the static mixer first and H<sub>2</sub>S reclamation column later. Recall that the H<sub>2</sub>S reclamation column is an integral part of the overall recovery process. This column serves to contact product gas with an alkaline scrubbing liquid to remove H<sub>2</sub>S from the gas phase and to recover sulfur in the form of green liquor. Efficient scrubbing is vital to the overall sulfur recovery efficiency of the process.

As seen in Table 6-15, H<sub>2</sub>S at the column exit is negligible, typically ranging from 30 to 70 ppm corresponding to scrubbing efficiency of about 99.8 to 99.9 percent. No other gas phase sulfur compounds were detected. This result is consistent with simulated scrubber testing which indicated that effluent H<sub>2</sub>S concentrations as low as 50 ppm were achievable. Note that this "clean" fuel gas product would ultimately be combusted in a commercial system to yield a flue gas containing less than 10 ppm SO<sub>2</sub>. This low level of SO<sub>2</sub> emissions exceeds performance of most modern Tomlinson recovery boilers currently in use.

A static mixer made by Koch (1/2" dia. x 9" long) was used for 4 hours to scrub the H<sub>2</sub>S. Typical results from the single-stage static mixer is also given in Table 6-15. The H<sub>2</sub>S scrubbing efficiency for the static mixer was about 80 to 84 percent in only about 10 milliseconds contact time. By using multi-stage static mixers, we can achieve nearly the same H<sub>2</sub>S-scrubbing efficiency without CO<sub>2</sub> absorption. CO<sub>2</sub> absorption is detrimental to the economics. CO<sub>2</sub> absorption forms bicarbonate which causes plugging of the column. This will be eliminated if we use static mixers.

**TABLE 6-15: GAS ANALYSES AFTER H<sub>2</sub>S  
SCRUBBING, 3/7/90 TEST RUN**

Time	STATIC MIXER			10 ft., 3" DIA. PACKED COLUMN		
	12:57	13:02	13:06	17:44	17:49	17:54
Gasifier Temp. (°F)	1132	1132	1132	1150	1150	1150
<u>Normalized Conc. (%)</u>						
H <sub>2</sub>	62.091	62.334	62.231	63.190	62.540	62.627
CH <sub>4</sub>	3.258	2.702	2.626	2.127	2.093	2.092
CO	3.685	3.264	3.344	3.216	3.195	3.203
CO <sub>2</sub>	27.786	28.478	28.550	29.522	30.134	30.000
Ethane	0.427	0.423	0.444	0.372	0.406	0.441
Ethylene	0.333	0.320	0.312	0.357	0.351	0.473
Propane	0.668	0.685	0.663	0.690	0.672	0.635
Propylene	0.450	0.446	0.437	0.376	0.450	0.383
i-Butane	0.125	0.160	0.216	0.145	0.153	0.142
H <sub>2</sub> S	0.487	0.461	0.404	0.006	0.006	0.002
COS	0.000	0.000	0.000	0.000	0.000	0.000
CH <sub>3</sub> SH	0.000	0.000	0.000	0.000	0.000	0.000
C <sub>4</sub> <sup>+</sup> /H <sub>2</sub> O	0.689	0.726	0.773	0.000	0.000	0.000
TOTAL	100.000	100.000	100.000	100.00	100.000	100.000

#### 6.2.4 KRAFT TEST RUN (3/21/90)

Gasification tests were conducted using an Everett Kraft liquor on March 21, 1990. The gasifier was operated for a period of approximately 7.5 hours during which time a total of 221 pounds of liquor (wet) was fed. The average liquor feed rate was 29.3 lb/hr and the average fluidization steam rate was 37.8 lb/hr. Average gasifier temperature was 1143°F.

A material flow summary is provided in Table 6-16.

**TABLE 6-16: MATERIAL FLOW SUMMARY FOR 3/21/90 TEST RUN**

INPUT:

Black Liquor Feed (lb/hr Wet)	29.3
Black Liquor Feed (lb/hr Dry)	16.6
Steam Feed (lb/hr)	37.8

OUTPUT:

Cyclone Catch (lb/hr)	4.61
Elutriate (lb/hr)	0.11
Product Gas (SCFM)	N/A

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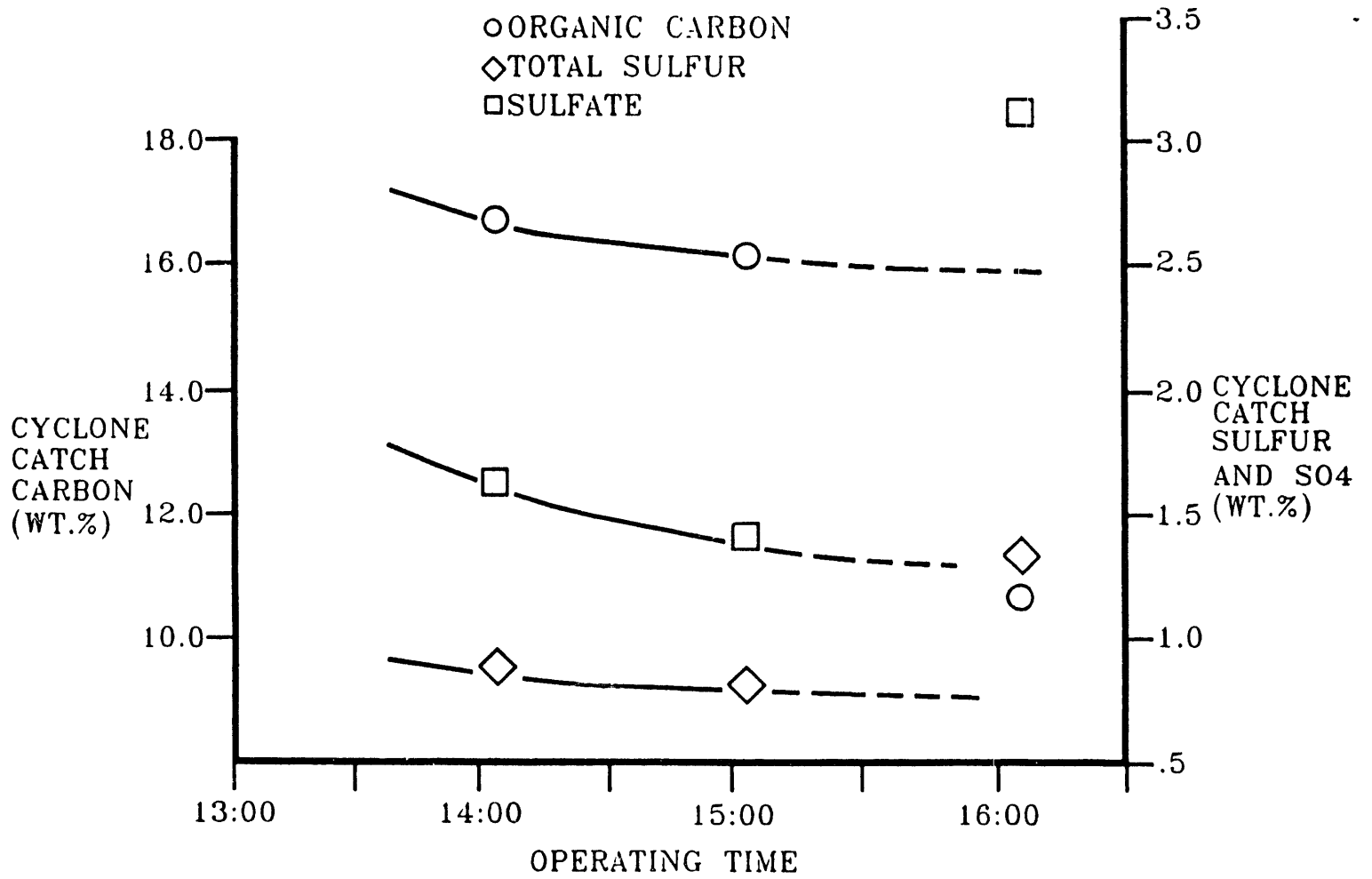
Note that the measurement of product gas flow rate was not available for this run due to mechanical difficulties with the nitrogen tracer gas mass flow meter.

Elemental analyses for product solids collected during the test run are shown in Table 6-17. As illustrated in Figure 6-5, solids collected at 14:03 and 15:07 exhibit a typical asymptotic steady-state behavior which would be anticipated to project along the depicted dotted line. However, the final sample collected at 16:08 appears to deviate significantly from this trend for all of the measured species. The reason for this aberration may simply be due to statistical sampling error, or possibly due to a gasifier upset condition. For the purposes of calculating material balances, the composite average of all three sample points was used. This would tend to increase the carbon gasification efficiency and reduce the sulfur gasification efficiency compared

**TABLE 6-17: ELEMENTAL ANALYSIS OF SOLIDS FOR 3/21/90 TEST RUN**

(AS REC'D →)

<u>SAMPLE DESIGNATION</u>	<u>SOLIDS %</u>	<u>TOTAL CARBON C%</u>	<u>CARBONATE CO<sub>3</sub>%</u>	<u>ORGANIC CARBON C%</u>	<u>SODIUM Na%</u>	<u>TOTAL SULFUR %</u>	<u>SULFATE SO<sub>4</sub>%</u>	<u>SULFIDE S%</u>
<u>BED SOLIDS:</u>								
032190-W-EV-1-1345-BS		15.43	52.35	4.96	39.40	0.35	0.25	0.04
032190-W-EV-1-1445-BS		15.88	51.43	5.59	40.60	0.40	0.51	0.06
<u>CYCLONE SOLIDS:</u>								
032190-W-EV-1-1403-CL	97.56	25.12	41.37	16.85	36.10	0.92	1.63	0.00
032190-W-EV-1-1507-CL	92.70	24.42	41.89	16.04	35.00	0.75	1.42	0.02
032190-W-EV-1-1608-CL	92.30	18.87	40.86	10.70	33.70	1.35	3.15	0.01
<u>ISO PROBE FILTER SOLIDS:</u>								
032190-W-EV-1-1315-IKS		57.05	9.85	55.08	9.06	4.07	3.04	0.01
<u>H<sub>2</sub>S SCRUBBER LIQUID:</u>								
032190-W-EV-1-1205-SCL			7.76		5.24	0.79	0.18	0.57
032190-W-EV-1-1502-SCL			7.41		4.71	0.69	0.38	0.39



**FIGURE 6-5: COMPOSITION OF CATCH SOLIDS DURING TEST RUN**

to data consistent with the projected steady-state trend. The average product solid compositions are summarized in Table 6-18.

Product gas analyses collected at the hot cyclone exit are shown in Table 6-19. Hydrogen sulfide concentration typically ranges between 1.7 to 1.8 percent as expected. Only traces of other sulfur-containing gases were observed. Yield patterns for major gas species are similar to prior test runs.

In order to verify accuracy of in-house gas chromatographic analyses, a gas bottle sample was withdrawn at 14:20 and sent to an outside laboratory for analysis of fixed gases and hydrocarbons. Table 6-20 compares the results of this gas analysis with in-house data measured by gas chromatography. As seen in Table 6-20, the accuracy of the gas chromatographic analysis is well corroborated for most species. The only significant deviation observed for major species is for CO<sub>2</sub> (22.7% vs. 29.36%). Hydrogen was used as a carrier gas for the outside analysis; therefore, hydrogen could not be detected directly, but was calculated by difference.

The material balance for the March 21, 1990 test run is shown in Table 6-21. Note that a nitrogen shut-off valve connected to the gasifier was found to leak during post-test inspection. Due to this problem, accurate gas flow calculations using the nitrogen tracer method could not be performed and a material balance closure is not available.

As seen in the table, based on solid analysis, the organic carbon gasification efficiency is 84.9 percent and the sulfur gasification efficiency is 88.3 percent. Also note that only 38 percent of the feed liquor sulfate could be accounted for by solid effluents. This result suggests a direct sulfate reduction of 62 percent.

#### H<sub>2</sub>S GAS SCRUBBING AND SULFATE REDUCTION EFFICIENCY

Gas analyses were also collected from the green liquor reclamation column (H<sub>2</sub>S scrubber) and are presented in Table 6-22. As seen in the table, the H<sub>2</sub>S concentration at the exit ranged from 40 to 70 ppm. This represents a gas

**TABLE 6-18: AVERAGE SOLIDS COMPOSITION FOR 3/21/90 TEST RUN**

(AS REC'D →)

	TOTAL CARBON % C	CARBONATE % CO <sub>3</sub>	ORGANIC CARBON % C	SODIUM % Na	TOTAL SULFUR %	SULFATE % SO <sub>4</sub>	SULFIDE % S
BED SOLIDS	15.66	51.89	5.27	40.00	0.37	0.38	0.05
CYCLONE	22.80	41.37	14.53	34.93	1.01	2.07	0.01
ELUTRIATE	57.05	9.85	55.08	9.06	4.07	3.04	0.01

**TABLE 6-19: GAS ANALYSES FOR SAMPLES COLLECTED AT CYCLONE EXIT DURING 3/21/90 TEST RUN**

Time	10:34	10:44	12:55	13:05	14:00	16:28
Gasifier Temp. (°F)	1136.0	1136.0	1135.0	1135.0	1146.0	1142.0
<u>Components (Vol.%)</u>						
H <sub>2</sub>	67.373	66.973	68.393	68.326	69.199	70.867
CH <sub>4</sub>	2.413	2.493	2.476	2.498	2.517	2.317
CO	2.974	2.955	3.219	3.296	3.331	2.934
CO <sub>2</sub>	23.926	24.371	22.871	22.911	22.701	21.069
Ethane	0.063	0.074	0.051	0.051	0.050	0.072
Ethylene	0.170	0.159	0.049	0.049	0.048	0.053
Propane	0.600	0.513	0.494	0.559	0.507	0.482
Propylene	0.334	0.325	0.312	0.285	0.310	0.365
I-Butane	0.104	0.142	0.197	0.107	0.114	0.132
H <sub>2</sub> S	1.801	1.745	1.701	1.682	1.216	1.698
COS	0.000	0.008	0.011	0.012	0.007	0.010
CH <sub>3</sub> SH	0.000	0.000	0.000	0.000	0.000	0.000
C <sub>4</sub> <sup>+</sup> /H <sub>2</sub> O	0.243	0.243	0.226	0.223	-	-
TOTAL	100.000	100.000	100.000	100.000	100.000	100.000

**TABLE 6-20: COMPARISON OF IN-HOUSE GAS CHROMATOGRAPHIC ANALYSIS WITH RESULTS FOR OUTSIDE LABORATORY**

<u>COMPONENT</u>	<u>(Vol. %)</u>	
	<u>IN-HOUSE ANALYSIS</u>	<u>OUTSIDE LABORATORY</u>
H <sub>2</sub>	69.20	63.85
CH <sub>4</sub>	2.52	2.65
CO	3.33	3.70
CO <sub>2</sub>	22.70	29.36
Ethane	0.05	0.13
Propane	0.51	0.20
Butanes	0.11	0.11

**TABLE 6-21: MATERIAL BALANCE SUMMARY FOR 3/21/90 TEST RUN**

	<u>TOTAL CARBON (lb/hr)</u>	<u>ORGANIC CARBON (lb/hr)</u>	<u>SODIUM (lb/hr)</u>	<u>TOTAL SULFUR (lb/hr)</u>	<u>SULFATE (SO<sub>4</sub>) (lb/hr)</u>
Liquor Feed	6.31	6.17	3.13	0.60	0.29
Cyclone Catch	1.05	0.67	1.61	0.05	0.10
Elutriate	0.06	0.06	0.01	0.01	0.00
Bed ACC/DEP	0.59	0.20	1.51	0.01	0.01
TOTAL SOLIDS	1.70	0.93	3.13	0.07	0.11
Gas Product	N/A	N/A	N/A	N/A	N/A
TOTAL GAS PLUS SOLID	N/A	N/A	N/A	N/A	N/A
Out/Input	N/A	N/A	N/A	N/A	N/A

PERFORMANCE PARAMETERS (SOLIDS BASIS):

	<u>UNNORMALIZED</u>	<u>NORMALIZED</u>
Organic Carbon Gasification Efficiency (%)	84.9	N/A
Sulfur Gasification Efficiency (%)	88.3	N/A



**TABLE 6-22: GAS ANALYSES FOR SAMPLES COLLECTED AT H<sub>2</sub>S SCRUBBER EXIT DURING 3/21/90 TEST RUN**

Time	12:36	12:37	13:37	13:47	15:10	15:20
Gasifier Temp. (°F)	1138	1138	1143	1143	1142	1142
<u>Normalized Conc.</u>						
H <sub>2</sub>	68.789	69.123	68.473	68.527	71.143	71.239
CH <sub>4</sub>	2.601	2.593	2.639	2.553	2.421	2.302
CO	3.499	3.499	3.508	3.465	3.457	3.042
CO <sub>2</sub>	23.769	23.413	24.055	23.921	21.556	21.971
Ethane	0.053	0.053	0.054	0.053	0.047	0.061
Ethylene	0.051	0.051	0.052	0.051	0.044	0.054
Propane	0.573	0.556	0.515	0.741	0.591	0.610
Propylene	0.201	0.287	0.299	0.283	0.343	0.314
I-Butane	0.195	0.155	0.144	0.132	0.126	0.138
H <sub>2</sub> S	0.004	0.006	0.004	0.005	0.007	0.007
COS	0.000	0.000	0.000	0.005	0.000	0.000
CH <sub>3</sub> SH	0.000	0.000	0.000	0.000	0.000	0.000
C <sub>4</sub> <sup>+</sup> /H <sub>2</sub> O	0.265	0.264	0.259	0.264	0.281	0.264
TOTAL	100.000	100.000	100.000	100.000	100.000	100.000

phase sulfur recovery of 99.7 percent, again confirming the excellent sulfur recovery potential of the integrated system.

It should be mentioned at this point that the reduction efficiency for the integrated gasification process can be calculated by multiplying the sulfur gasification efficiency by the scrubber H<sub>2</sub>S removal efficiency. This assumes that all gas products are in reduced form. It also assumes negligible oxidation of the scrubber liquid product (green liquor). For instance, for the test run on March 21, 1990, the sulfur reduction efficiency would be calculated as follows (i.e., amount of total sulfur reporting as sulfide in green liquor):

$$\text{Sulfur Reduction Efficiency} = (.883 \times .997) \times 100 = 88.0\%$$

This reduction efficiency is comparable to commercial recovery boilers which typically operate in the range from 85 to 95 percent.

Referring back to Table 6-17, analyses were also made of green liquor product from the scrubber bottoms. The molar ratio of sulfur to sodium is 0.11. This translates to a sulfidity (molar S to molar Na<sub>2</sub>) of 22 percent. Note that the sulfidity of the liquor depends not only on the H<sub>2</sub>S scrubbing efficiency but also the metered flow rate of alkaline solution sent to the scrubbing column. For the experimental tests, the alkaline flow rate was independently controlled. In this way, virtually any sulfidity could be achieved within practical equilibrium constraints. In contrast, for the integrated commercial plant, the total alkaline solution available for scrubbing is directly tied to the operating black liquor capacity. This is due to the fact that the alkaline solution is formed in situ by dissolution of product salts. Under these conditions, the theoretical sulfidity of the green liquor product will by and large reflect the typical operating sulfidity of the mill circuit (25-35%). Note that controlled scrubber simulation tests (described in later sections) verified that sulfidities of 30 percent were achievable even while maintaining scrubbing efficiency at 99.7 percent.

### **6.2.5 KRAFT TEST RUN (3/27/90)**

Gasification tests were conducted using Everett Kraft liquor on March 27, 1990. The gasifier was operated for a period of approximately 6 hours during which time a total of 175.5 lb/hr of liquor was fed. The average liquor feed rate was 28.8 lb/hr (wet). The average gasifier temperature was approximately 1160°F.

### **GAS RECYCLE FOR FLUIDIZATION**

During the initial several hours of testing, the gasifier was operated utilizing only steam as a fluidizing media (33.9 lb/hr). During the latter stages of the test run, a recycle gas compressor was utilized to supplement the fluidization steam. The recycle gas was withdrawn from the venturi scrubber exit. Under this condition, approximately 30 percent of the fluidization flow rate was attributed to recycle gas.

The use of recycle gas was considered for two reasons. First, it provides additional flexibility for control of fluidization velocity during turndown. And second, by reducing the required steam load, overall thermal efficiency of the gasifier plant is improved. No operating problems were encountered during the recycle portion of the test run.

A material flow summary is provided in Table 6-23.

**TABLE 6-23: MATERIAL FLOW SUMMARY FOR 3/27/90 TEST RUN**

INPUT:

Black Liquor Feed (lb/hr Wet)	28.8
Black Liquor Feed (lb/hr Dry)	16.3
Steam/Recycle Gas (lb/hr)	24.7/10.0

OUTPUT:

Cyclone Catch (lb/hr)	2.95
Elutriate (lb/hr)	0.59
Product Gas (SCFM)	N/A

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Elemental analyses of product solids are summarized in Table 6-24. As seen in the table, bed organic carbon, total sulfur, and sulfate are relatively constant. Cyclone catch total sulfur and sulfate are also relatively constant; however, the organic carbon value shows some significant variation.

Average elemental compositions for the solid samples used in material balance calculations are shown in Table 6-25.

During the test run, a circuit board in the gas chromatograph malfunctioned preventing collection of gas analysis data. Based on solids analysis only, a material balance for the test run is summarized in Table 6-26.

**TABLE 6-24: ELEMENTAL ANALYSIS OF SOLIDS FOR 3/27/90 TEST RUN**

(AS REC'D →)

<u>SAMPLE DESIGNATION</u>	<u>SOLIDS %</u>	<u>TOTAL CARBON C%</u>	<u>CARBONATE CO<sub>3</sub>%</u>	<u>ORGANIC CARBON C%</u>	<u>SODIUM Na%</u>	<u>TOTAL SULFUR %</u>	<u>SULFATE SO<sub>4</sub>%</u>	<u>SULFIDE S%</u>
<u>BED SOLIDS:</u>								
032790-W-EV-1-1500-BS		17.89	50.19	7.85	40.40	0.43	0.34	0.06
032790-W-EV-1-1600-BS		17.29	49.56	7.38	37.10	0.50	0.28	0.05
<u>CYCLONE SOLIDS:</u>								
032790-W-EV-1-1330-CL	95.52	22.48	43.10	13.86	33.90	2.02	3.63	0.01
032790-W-EV-1-1415-CL	94.58	27.06	38.53	19.35	30.30	2.01	4.20	0.01
<u>ISO PROBE FILTER SOLIDS:</u>								
032790-W-EV-1-1515-IKS		65.65	11.78	63.29	9.34	4.03	2.54	0.01
<u>H<sub>2</sub>S SCRUBBER LIQUID:</u>								
032790-W-EV-1-1316-SCL			8.16		5.24	0.82	0.16	0.53

**TABLE 6-25: AVERAGE SOLIDS COMPOSITION FOR 3/27/90 TEST RUN**

(AS REC'D →)

	<u>TOTAL CARBON % C</u>	<u>CARBONATE % CO<sub>3</sub></u>	<u>ORGANIC CARBON % C</u>	<u>SODIUM % Na</u>	<u>TOTAL SULFUR %</u>	<u>SULFATE % SO<sub>4</sub></u>	<u>SULFIDE % S</u>
BED SOLIDS	17.59	49.88	7.62	38.75	0.47	0.31	0.05
CYCLONE	24.77	40.82	16.61	32.10	2.01	3.92	0.01
ELUTRIATE	65.65	11.78	63.29	9.34	4.03	2.54	0.01

**TABLE 6-26: MATERIAL BALANCE SUMMARY FOR 3/27/90 TEST RUN**

	<u>TOTAL CARBON (lb/hr)</u>	<u>ORGANIC CARBON (lb/hr)</u>	<u>SODIUM (lb/hr)</u>	<u>TOTAL SULFUR (lb/hr)</u>	<u>SULFATE (SO<sub>4</sub>) (lb/hr)</u>
Liquor Feed	6.21	6.06	3.08	0.59	0.28
Cyclone Catch	0.73	0.49	0.95	0.06	0.12
Elutriate	0.36	0.34	0.05	0.02	0.01
Bed ACC/DEP	0.95	0.41	2.08	0.03	0.02
TOTAL SOLIDS	2.04	1.24	3.08	0.11	0.15
Gas Product	N/A	N/A	N/A	N/A	N/A
TOTAL GAS PLUS SOLID	N/A	N/A	N/A	N/A	N/A
Out/Input	N/A	N/A	N/A	N/A	N/A

PERFORMANCE PARAMETERS (SOLIDS BASIS):

	<u>UNNORMALIZED</u>	<u>NORMALIZED</u>
Organic Carbon Gasification Efficiency (%)	79.5	N/A
Sulfur Gasification Efficiency (%)	81.4	N/A

As seen in Table 6-26, organic carbon gasification efficiency is 79.5 percent while sulfur gasification efficiency is 81.4 percent. The somewhat lower gasification efficiency for this test run compared to several prior tests may be due, in part, to the reduced steam partial pressure resulting from use of recycle gas. The results also suggest a direct sulfate reduction efficiency of 46 percent.

### **6.2.6 SULFITE TEST RUN (9/18/90)**

Gasification tests were conducted using North Bend NSSC spent liquor on September 18, 1990. The gasifier was operated for a period of approximately 8 hours during which time a total of 170.4 pounds of spent liquor (wet) was fed. The average liquor feed rate during the steady-state period was 21.5 lb/hr (wet) and the fluidization steam rate was approximately 36.0 lb/hr. The average gasifier temperature was 1130°F.

A material flow summary during steady-state conditions is shown in Table 6-27.

**TABLE 6-27: MATERIAL FLOW SUMMARY FOR 9/18/90 TEST RUN**

INPUT:

Black Liquor Feed (lb/hr Wet)	21.46
Black Liquor Feed (lb/hr Dry)	7.85
Steam Feed (lb/hr)	36.00

OUTPUT:

Cyclone Catch (lb/hr)	2.23
Elutriate (lb/hr)	0.36
Product Gas (SCFM)	3.64

---

Elemental analyses for solid samples collected during the course of the test run are summarized in Table 6-28. As seen in Table 6-28 and Figures 6-6 and 6-7, carbon, sulfur, and sulfate concentrations in both the bed and cyclone catch solids are relatively constant throughout the test period from 14:00 to 18:00, suggesting that the results are representative of steady-state operation.

Table 6-29 summarizes the average elemental concentrations for solids collected during the steady-state period. These values were utilized in calculation of gasifier material balances.

Gas analyses for the test run are summarized in Table 6-30. It is interesting to note that the gas composition using NSSC feedstock is quite similar to that generated during the gasification of Kraft liquors; the only significant difference being the higher concentration of H<sub>2</sub>S for NSSC liquors (2.8%-3.3% vs. 1.5%-1.8%). It is also significant to note that the NSSC liquors do not release any measurable quantity of SO<sub>2</sub> into the gas phase. Since these measurements were made using the Tracor 540 flame photometric detector (FPD), the accuracy of these results are considered to be quite good. A comparison of the H<sub>2</sub>S concentrations measured using the Tracor 540 and the MTCI M-200 gas chromatographs is shown in Table 6-31. As seen in the comparison, the independent gas chromatographs provide excellent corroboration of the data accuracy.

The material balance for the September 18, 1990 NSSC test run is summarized in Table 6-32. Material balance closure for organic carbon and sulfur are 123 percent and 111 percent, respectively. As previously discussed, material balance closure in excess of 100 percent appears to be related to the use of a low value for the solids content of the feed liquor.

The unnormalized and normalized organic carbon gasification efficiencies are calculated as 79.2 percent and 83.1 percent, respectively. The unnormalized and normalized sulfur gasification efficiencies are 32.8 percent and 84.5 percent, respectively. In Kraft liquor tests it was noted that the

**TABLE 6-28: ELEMENTAL ANALYSIS OF SOLIDS FOR 9/18/90 TEST RUN**

(AS REC'D →)

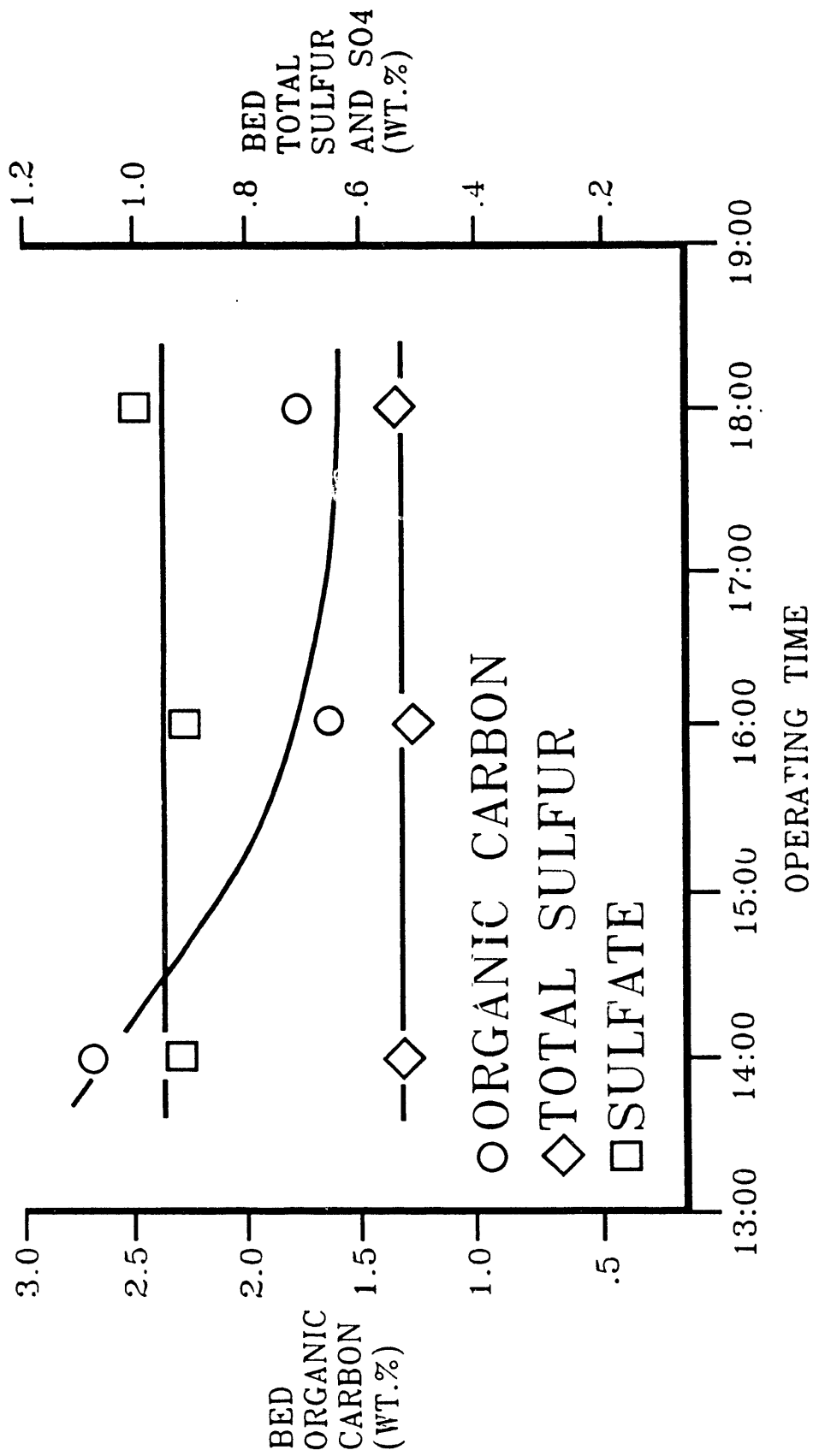
<u>SAMPLE DESIGNATION</u>	<u>SOLIDS %</u>	<u>TOTAL CARBON C%</u>	<u>CARBONATE CO<sub>3</sub>%</u>	<u>ORGANIC CARBON C%</u>	<u>SODIUM Na%</u>	<u>TOTAL SULFUR %</u>	<u>SULFATE SO<sub>4</sub>%</u>	<u>SULFIDE S%</u>
<u>BED SOLIDS:</u>								
091890-W-NSSC-1400-BS		13.20	52.65	2.67	41.90	0.56	0.93	N/A
091890-W-NSSC-1600-BS		12.37	53.80	1.61	44.60	0.53	0.89	N/A
091890-W-NSSC-1800-BS		12.07	51.50	1.77	45.20	0.57	1.00	N/A
<u>CYCLONE SOLIDS:</u>								
091890-W-NSSC-1400-CL		27.41	31.20	21.17	31.50	4.51	9.88	N/A
091890-W-NSSC-1600-CL		26.36	33.70	19.62	33.60	3.99	8.33	N/A
091890-W-NSSC-1800-CL		24.38	34.70	17.44	33.50	3.77	8.51	N/A
<u>PROBE FILTER SOLIDS:</u>								
091890-W-NSSC-1800-PF		27.59	32.10	21.35	31.20	3.77	8.11	N/A

**TABLE 6-29: AVERAGE SOLIDS CONCENTRATION DURING STEADY-STATE PERIOD FOR 9/18/90 TEST RUN**

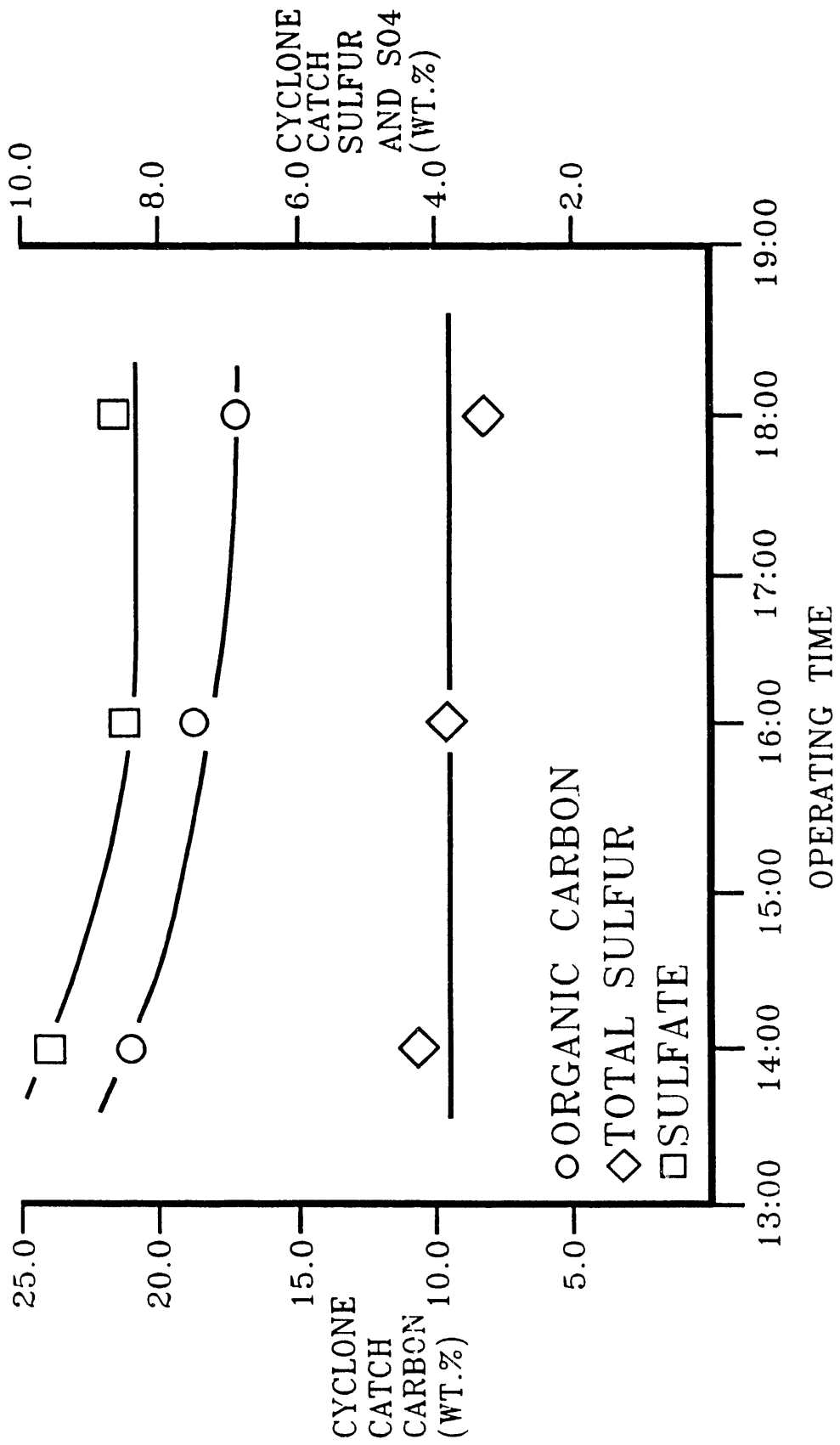
(AS REC'D →)

	<u>TOTAL CARBON % C</u>	<u>CARBONATE % CO<sub>3</sub></u>	<u>ORGANIC CARBON % C</u>	<u>SODIUM % Na</u>	<u>TOTAL SULFUR %</u>	<u>SULFATE % SO<sub>4</sub></u>
BED SOLIDS	12.55	52.65	2.02	43.90	0.56	0.94
CYCLONE	26.04	33.20	19.41	32.90	4.09	8.91
ELUTRIATE	27.59	32.10	21.17	31.20	3.77	8.11





**FIGURE 6-6: COMPOSITION OF BED SOLIDS DURING 9/18/90 NSSC TEST RUN**



**FIGURE 6-7: COMPOSITION OF CYCLONE SOLIDS DURING 9/18/90 NSSC TEST RUN**

**TABLE 6-30: GAS ANALYSES FOR 9/18/90 TEST RUN**  
**(CYCLONE EXIT)**

Time	13:14	14:14	15:15	16:14	17:16	18:15
Gasifier Temp. (°F)	1120	1120	1126	1133	1129	1125
Gas Rate (SCFM)	2.486	3.604	3.890	3.773	4.422	3.646
<u>Components (Vol.%)</u>						
H <sub>2</sub>	60.120	59.865	61.668	60.263	60.328	61.431
CO <sub>2</sub>	34.422	33.902	32.158	33.253	32.882	31.865
CO	1.406	1.383	1.456	1.525	1.693	1.507
CH <sub>4</sub>	1.485	1.509	1.419	1.491	1.438	1.591
C <sub>2</sub> H <sub>4</sub>	0.000	0.000	0.000	0.000	0.000	0.000
C <sub>2</sub> H <sub>6</sub>	0.193	0.200	0.188	0.195	0.187	0.216
C <sub>3</sub> H <sub>6</sub>	0.230	0.247	0.221	0.246	0.253	0.086
C <sub>3</sub> H <sub>8</sub>	0.000	0.000	0.000	0.000	0.000	0.000
I-Butane	0.001	0.026	0.001	0.008	0.000	0.002
n-Butane	0.001	0.017	0.015	0.020	0.015	0.019
Pentane	0.001	0.011	0.007	0.012	0.010	0.005
Hexane	0.000	0.000	0.000	0.000	0.000	0.000
H <sub>2</sub> S	2.107	2.848	2.853	2.971	3.179	3.253
COS	0.000	0.000	0.000	0.000	0.000	0.000
CH <sub>3</sub> SH	0.011	0.017	0.013	0.015	0.013	0.022
SO <sub>2</sub>	0.000	0.000	0.000	0.000	0.000	0.000
TOTAL	100.000	100.000	100.000	100.000	100.000	100.000

**TABLE 6-31: COMPARISON OF H<sub>2</sub>S CONCENTRATION MEASURED**  
**IN TWO INDEPENDENT GAS CHROMATOGRAPHS**

TIME	(H <sub>2</sub> S Vol. %)	
	TRACOR 540	MTI M-200
15:03	2.97	2.72
16:07	2.97	2.71
17:08	3.08	3.09
18:13	3.24	3.24
18:48	3.02	2.94

sulfur gasification efficiency was typically 2-4 percentage points higher than the carbon gasification efficiency. This relation also appears to be exhibited by NSSC liquors.

Based on total sulfate in the solid products, it is estimated that 60 percent of the feed liquor sulfate is directly reduced within the gasifier.

**TABLE 6-32: MATERIAL BALANCE SUMMARY FOR 9/18/90 TEST RUN**

	<u>TOTAL CARBON (lb/hr)</u>	<u>ORGANIC CARBON (lb/hr)</u>	<u>SODIUM (lb/hr)</u>	<u>TOTAL SULFUR (lb/hr)</u>	<u>SULFATE (SO<sub>4</sub>) (lb/hr)</u>
Liquor Feed	2.53	2.50	1.38	0.64	0.60
Cyclone Catch	0.58	0.43	0.73	0.09	0.20
Elutriate	0.09	0.06	0.10	0.01	0.03
Bed ACC/DEP	0.16	0.03	0.55	0.01	0.01
TOTAL SOLIDS	2.83	0.52	1.38	0.11	0.24
Gas Product	2.56	2.56	0.00	0.60	0.00
TOTAL GAS PLUS SOLID	3.39	3.08	1.38	0.71	0.24
Out/Input	1.34	1.23	1.00	1.11	0.40

PERFORMANCE PARAMETERS (SOLIDS BASIS):

	<u>UNNORMALIZED</u>	<u>NORMALIZED</u>
Organic Carbon Gasification Efficiency (%)	79.2	83.1
Sulfur Gasification Efficiency (%)	82.8	84.5

### **6.2.7 SULFITE TEST RUN (9/24/90)**

A second gasification trial was conducted using North Bend NSSC spent liquor on September 24, 1990. The gasifier was operated for a period of approximately 5.5 hours during which time a total of 147.6 pounds of spent liquor (wet) was fed. The average liquor feed rate during the trial was 26.8 lb/hr (wet) and the fluidization steam rate was approximately 38 lb/hr. The average gasifier temperature was 1150°F.

A material flow summary for the trial is shown in Table 6-33.

**TABLE 6-33: MATERIAL FLOW SUMMARY FOR 9/24/90 TEST RUN**

INPUT:

Black Liquor Feed (lb/hr Wet)	26.80
Black Liquor Feed (lb/hr Dry)	9.81
Steam Feed (lb/hr)	38.00

OUTPUT:

Cyclone Catch (lb/hr)	3.20
Elutriate (lb/hr)	0.26
Product Gas (SCFM)	4.38

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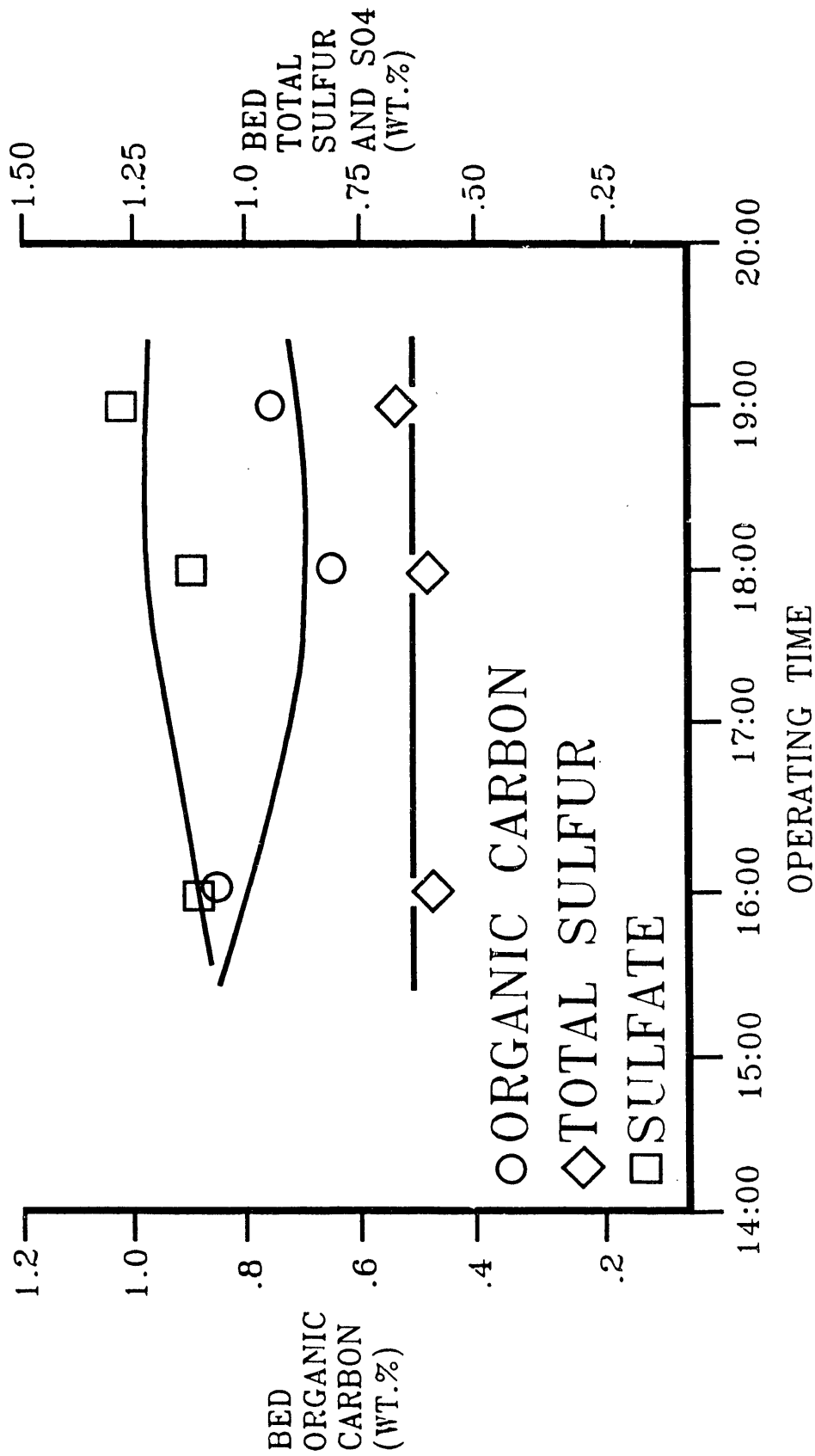
Elemental analyses for solid samples collected during the course of the test run are summarized in Table 6-34. As seen in Figures 6-8 and 6-9, carbon, sulfur and sulfate concentrations in both the bed solids and cyclone solids exhibit behavior indicative of approach to steady-state.

**TABLE 6-34: ELEMENTAL ANALYSIS OF SOLIDS FOR 9/24/90 TEST RUN**

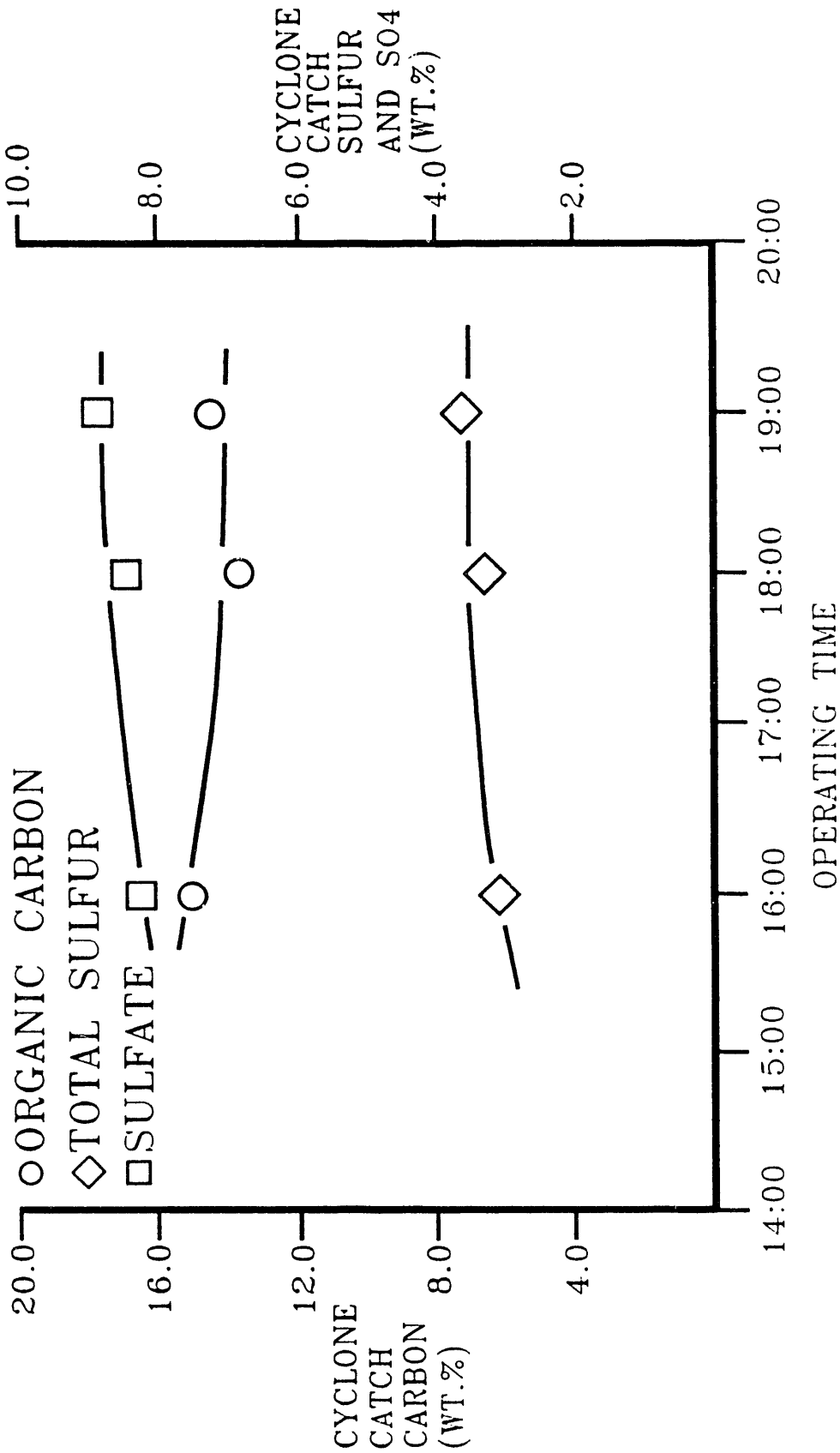
(AS REC'D →)

<u>SAMPLE DESIGNATION</u>	<u>SOLIDS</u> %	<u>TOTAL</u> <u>CARBON</u> C%	<u>CARBONATE</u> CO <sub>3</sub> %	<u>ORGANIC</u> <u>CARBON</u> C%	<u>SODIUM</u> Na%	<u>TOTAL</u> <u>SULFUR</u> %	<u>SULFATE</u> SO <sub>4</sub> %
<u>BED SOLIDS:</u>							
092490-W-NSSC-1600		11.67	54.10	0.85	45.60	0.61	1.12
092490-W-NSSC-1800		11.50	54.30	0.64	45.50	0.62	1.13
092490-W-NSSC-1900		11.58	54.00	0.78	45.20	0.69	1.26
<u>CYCLONE SOLIDS:</u>							
092490-W-NSSC-1600		22.75	36.60	15.43	35.70	3.32	8.16
092490-W-NSSC-1800		22.10	36.90	14.72	36.20	3.43	8.42
092490-W-NSSC-1900		22.32	36.80	14.96	35.10	3.88	9.26
<u>PROBE FILTER SOLIDS:</u>							
092490-W-NSSC-1800		33.67	23.70	28.93	30.40	4.66	9.98

---



**FIGURE 6-8: COMPOSITION OF BED SOLIDS DURING 9/24/90 NSSC TEST RUN**



**FIGURE 6-9: COMPOSITION OF CYCLONE CATCH SOLIDS DURING 9/24/90 NSSC TEST RUN**

Table 6-35 summarizes the average elemental concentrations for solids collected during the test period. These values were utilized in subsequent material balance calculations.

**TABLE 6-35: AVERAGE SOLIDS CONCENTRATION DURING STEADY-STATE PERIOD FOR 9/24/90 TEST RUN**

(AS REC'D →)

	TOTAL CARBON % C	CARBONATE % CO <sub>3</sub>	ORGANIC CARBON % C	SODIUM % Na	TOTAL SULFUR %	SULFATE % SO <sub>4</sub>
BED SOLIDS	11.59	54.13	0.76	45.46	0.64	1.17
CYCLONE	22.39	36.77	15.03	35.66	3.54	8.61
ELUTRIATE	33.67	23.70	28.93	30.40	4.66	9.98

Gas analyses for the test run are listed in Table 3-36. As seen in Table 6-36, gas yield was quite constant for most of the test run period (4.2-4.7 SCFM). The hydrogen concentration ranged from 61 - 63 volume percent. Carbon dioxide ranged from 30 - 31 volume percent with methane and carbon monoxide levels both at approximately 1.7 volume percent.

The hydrogen sulfide concentration was typically 2.9 - 3.4 volume percent. It was noted that the H<sub>2</sub>S concentration appears to be increasing slowly during the course of the test run. This trend was also observed for the 9/18/90 NSSC trial. Although the increased H<sub>2</sub>S release rate does not appear to be correlated with any significant build-up of total sulfur or sulfate (SO<sub>4</sub>) within the bed, it may be related to accumulation of partially reduced sulfur species which are more prone to decomposing to H<sub>2</sub>S. It is also significant to note that no appreciable quantity of SO<sub>2</sub> could be detected in the product gases. This appears to further verify that both NSSC and Kraft liquors release sulfur only in reduced form under the gasifier conditions.



**TABLE 6-36: GAS ANALYSES FOR 9/24/90 TEST RUN**  
**(CYCLONE EXIT)**

Time	15:18	16:18	17:19	18:19	18:55
Gasifier Temp. (°F)	1150	1150	1150	1145	1150
Gas Rate (SCFM)	4.29	4.28	4.38	4.24	4.24
<u>Components (Vol.%)</u>					
H <sub>2</sub>	63.134	61.899	62.468	61.361	61.073
CO <sub>2</sub>	30.435	31.185	30.477	31.125	30.877
CO	1.732	1.665	1.730	1.702	1.840
CH <sub>4</sub>	1.726	1.670	1.709	1.779	2.039
C <sub>2</sub> H <sub>4</sub>	0.085	0.074	0.084	0.082	0.051
C <sub>2</sub> H <sub>6</sub>	0.245	0.227	0.243	0.250	0.301
C <sub>3</sub> H <sub>6</sub>	0.288	0.293	0.294	0.305	0.343
C <sub>3</sub> H <sub>8</sub>	0.000	0.000	0.000	0.000	0.000
I-Butane	0.006	0.001	0.001	0.000	0.001
n-Butane	0.028	0.022	0.027	0.028	0.030
Pentane	0.013	0.016	0.015	0.013	0.013
Hexane	0.000	0.000	0.000	0.000	0.000
H <sub>2</sub> S	2.287	2.922	2.928	3.325	3.400
COS	0.000	0.000	0.000	0.000	0.000
CH <sub>3</sub> SH	0.020	0.022	0.024	0.030	0.032
SO <sub>2</sub>	0.000	0.000	0.000	0.000	0.000
TOTAL	100.000	100.000	100.000	100.000	100.000

Material balances for the 9/24/90 test run are summarized in Table 6-37. Material balance closure for organic carbon and sulfur are 121 percent and 110 percent, respectively.

The unnormalized organic carbon and sulfur gasification efficiencies are calculated as 81.8 percent and 83.8 percent. Normalized gasification efficiencies are 84.9 percent and 85.2 percent. These latter results are quite similar to that of the 9/18/90 test which exhibited normalized gasification efficiencies of 82.8 percent and 84.5 percent. These results confirm the

reproducibility of performance data for gasification tests conducted under relatively similar conditions.

**TABLE 6-37: MATERIAL BALANCE SUMMARY FOR 9/24/90 TEST RUN**

	<u>TOTAL CARBON (lb/hr)</u>	<u>ORGANIC CARBON (lb/hr)</u>	<u>SODIUM (lb/hr)</u>	<u>TOTAL SULFUR (lb/hr)</u>	<u>SULFATE (SO<sub>4</sub>) (lb/hr)</u>
Liquor Feed	3.16	3.13	1.73	0.80	0.74
Cyclone Catch	0.72	0.48	1.14	0.11	0.28
Elutriate	0.09	0.08	0.08	0.01	0.02
Bed ACC/DEP	0.13	0.01	0.51	0.01	0.01
TOTAL SOLIDS	0.94	0.57	1.73	0.13	0.31
Gas Product	3.21	3.21	0.00	0.75	0.00
TOTAL GAS PLUS SOLID	4.15	3.78	1.73	0.88	0.31
Out/Input	1.31	1.21	1.00	1.10	0.42

PERFORMANCE PARAMETERS (SOLIDS BASIS):

	<u>UNNORMALIZED</u>	<u>NORMALIZED</u>
Organic Carbon Gasification Efficiency (%)	81.8	84.9
Sulfur Gasification Efficiency (%)	83.8	85.2

### 6.3 SCRUBBER VERIFICATION TESTS

MTCI's indirectly heated gasification technology for black liquor recovery relies on the scrubbing of H<sub>2</sub>S from product gases to regenerate green liquor for reuse in the mill circuit. Due to concerns relative to the efficiency of sulfur recovery in the MTCI integrated process, an experimental investigation was undertaken to establish performance and design data for this system.

A 3-inch diameter by 20-foot high scrubbing column was constructed for the experimental test program. Scrubbing liquor and gas mixtures were formulated to simulate the conditions of the MTCI integrated recovery process.

Test runs were conducted at three different column packing heights, and absorption rate coefficients and selectivities were calculated. The results of the simulated scrubber tests are reported here.

### **6.3.1 INTRODUCTION**

In the MTCI recovery process, black liquor is sprayed directly onto a bed of sodium carbonate solids which is fluidized by steam. Direct contact of the black liquor with hot bed solids promotes high rates of heating and pyrolysis. Residual carbon, which forms as a deposit on the particle surface, is then gasified by reaction with steam. Heat for the endothermic pyrolysis and steam-gasification reactions is supplied from pulse combustor resonance tubes which are immersed with the fluid bed.

The gasifier operating conditions are maintained at temperatures below which smelt can form. In this dry recovery process, oxidized sulfur species are partially reduced by reaction with the gasifier products, principally carbon monoxide and hydrogen. The reduced sulfur form is unstable in the gasifier environment, decomposing to solid sodium carbonate and gaseous hydrogen sulfide ( $H_2S$ ). Sulfur values are recovered by discharging a dry sodium carbonate product from the gasifier.

Since the sulfur constituents contained within the black liquor feed are released as  $H_2S$  and are thereby mixed with the gasifier products, it is necessary to recover these sulfur values in order to regenerate the desired green liquor product stream. This is envisioned to involve scrubbing of the product gas with an alkaline solution formed by dissolution of the sodium carbonate product salts.

While the recovery process described above is quite straightforward, it was determined that investigations were needed to fully verify the potential for high sulfur recovery efficiency, as this may significantly impact operating economics and environmental emissions for the process. Furthermore, additional scrubber design support data was needed for scale-up for the integrated recovery process.

Therefore, it was the objective of this present program to experimentally characterize the sulfur recovery performance for the alkaline scrubber under conditions simulating those of the integrated process and to develop design data for scale-up purposes.

### **6.3.2 BACKGROUND**

Aqueous sodium carbonate has been employed in some of the earliest acid gas scrubbing systems for the removal of  $H_2S$  and  $CO_2$ . In most cases of industrial significance, these systems utilized two separate columns: an absorber and a stripper. Gases containing acid constituents were fed to the bottom of a counter-current absorption column which may be operated under pressure. Purified product gases were then collected at the top of the absorption column, and the acid gas-enriched solution was pumped to the top of a stripper column. Here, acid gas vapors were flashed off by a combination of heating (reboiling/steam stripping) and pressure reduction (or vacuum stripping).

Since the objective of these systems was to remove acid gases to low levels, the sodium carbonate solution circulation rates, and thus sulfur loading of the rich solution, could be independently adjusted as needed to achieve the desired performance. However, in the MTCI recovery process, the objective is to produce a rich solution, i.e., green liquor product, possessing a specific alkalinity and sulfidity level.

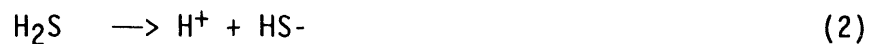
Therefore, the ratio of  $H_2S$  in the product gas phase to sodium contained in the circulating sodium carbonate solution phase cannot be independently varied. Because of this lack of flexibility, special consideration must be given to the design of the absorber column to simultaneously achieve high sulfur loading (sulfidity) in the rich green liquor product stream and low sulfur breakthrough in the gas product stream.

Although the absorption rates of  $CO_2$  and  $H_2S$  in alkaline solutions have been well studied in the industrial sector, much of the data remains proprietary, and fundamental rate data which does exist in the public domain must be extrapolated to the specific conditions of the MTCI recovery process. As

extrapolation of absorption data can lead to significant errors, the present investigation was devised to provide valuable process data collected under conditions specific to the MTCI recovery environment.

A primary interest in this investigation involved characterizing the selective absorption behavior of H<sub>2</sub>S relative to CO<sub>2</sub>. Since CO<sub>2</sub> is a stronger acid gas than H<sub>2</sub>S, equilibrium predicts that CO<sub>2</sub> will be absorbed to a greater extent than H<sub>2</sub>S. Also, since the ratio of CO<sub>2</sub> to H<sub>2</sub>S in the gasifier product stream is approximately 10:1, a strong equilibrium driving force exists for the preferential absorption of CO<sub>2</sub> relative to H<sub>2</sub>S. However, the absorption of CO<sub>2</sub> is highly undesirable since it limits the ability of the solution to achieve a high sulfidity and it places an increased burden on the causticizing circuit.

Fortunately, the kinetic rate of CO<sub>2</sub> absorption is quite slow compared to that of H<sub>2</sub>S. This is due to the fact that CO<sub>2</sub> absorption requires a chemical hydration reaction, while H<sub>2</sub>S is absorbed by a simple ionic dissociation step as shown below:



Also, since CO<sub>2</sub> absorption involves an activated chemical reaction, its rate is highly sensitive to temperature. This is in contrast to H<sub>2</sub>S absorption which is only modestly affected by temperature. Therefore, increased selectivity for H<sub>2</sub>S absorption can be achieved at lower temperatures.

The ability to achieve the sulfur recovery and energy efficiency goals for the MTCI process requires that the H<sub>2</sub>S absorption selectivity be maximized. The absorption rate for each species is characterized by a global absorption rate parameter, K<sub>g a</sub>, which is defined as:

$$(\text{K}_g \text{ a})_i = n_i / (L_C A_C \text{ LMPD}_i) \quad (3)$$

Where,  $n_i$  is the rate of absorption of species ( $i$ ) in gmole/min;  $L_c$  and  $A_c$  are the column height and cross sectional area; LMPD is the log mean pressure driving force, and  $K_g a$  is the rate constant in gmole/min/ft<sup>3</sup>/atm.

The LMPD is defined as:

$$\text{LMPD} = (P_{g,in} - P_{1,out}^* - P_{g,out} - P_{1,in}^*) / \quad (4)$$

$$\text{LOG}_e ((P_{g,in} - P_{1,out}^*) / (P_{g,out} - P_{1,in}^*))$$

where  $P_{g,in}$  and  $P_{g,out}$  are the gas phase partial pressures at the column inlet and outlet, and  $P_{1,out}^*$  and  $P_{1,in}^*$  are the equilibrium partial pressures above the liquid at the outlet and inlet.

The equilibrium partial pressure of CO<sub>2</sub> and H<sub>2</sub>S in the liquid phase are calculated based on liquid species concentration as follows:

$$P_{\text{CO}_2} \text{ (atm)} = 1.65 \times 10^2 f^2 / (1-f) C^{1.362} \text{EXP}(-2729/T) \quad (5)$$

$$P_{\text{H}_2\text{S}} \text{ (atm)} = 9.746 \times 10^{-4} P_{\text{CO}_2} C^{1.25} [\text{NaHS}] / [\text{NaCO}_3] \text{EXP}(2275/T) \quad (6)$$

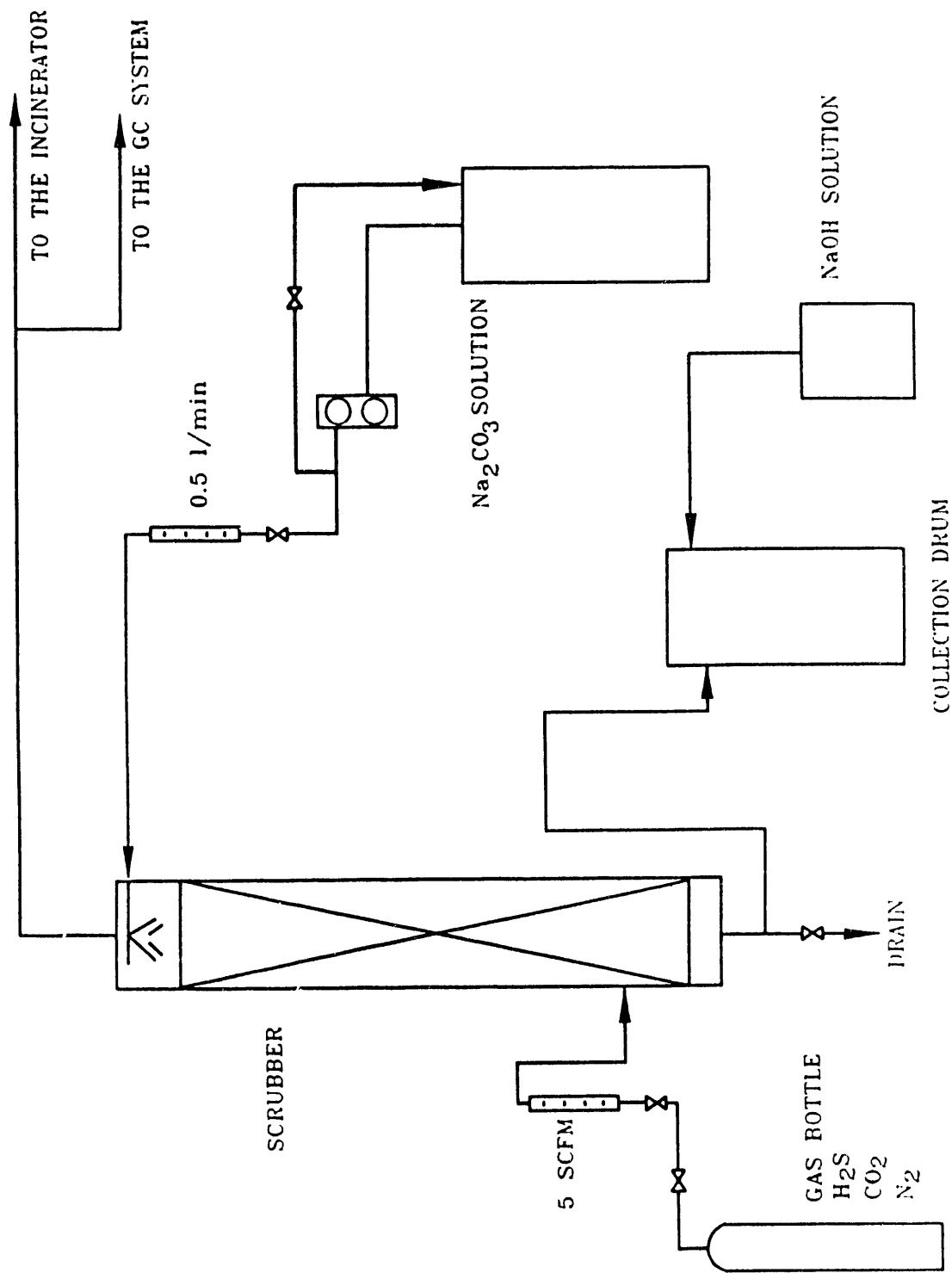
where  $f = [\text{NaHCO}_3] / ([\text{NaHCO}_3] + 2 [\text{Na}_2\text{CO}_3])$ ,  $C$  is the sodium normality, and  $T$  is the solution temperature (°K).

Calculation of the absorption rate constants for the experiments conducted in this investigation followed the guidelines and definition described above.

### **6.3.3 EXPERIMENTAL SYSTEM**

#### **A. TEST RIG**

Schematic diagram of the experimental scrubber test rig is shown in **Figure 6-10**. A 14.5 wt.% solution of sodium carbonate is pumped from the feed drum to the scrubber column via a rotary gear pump. A bypass line is



**FIGURE 6-10: SCHEMATIC OF EXPERIMENTAL SCRUBBER TEST RIG**

installed on the feed system to allow excess flow to return to the sodium carbonate feed drum. The solutions feed rate to the column is metered by a high accuracy rotameter. The sodium carbonate solution is continuously agitated inside the feed drum by a stirrer.

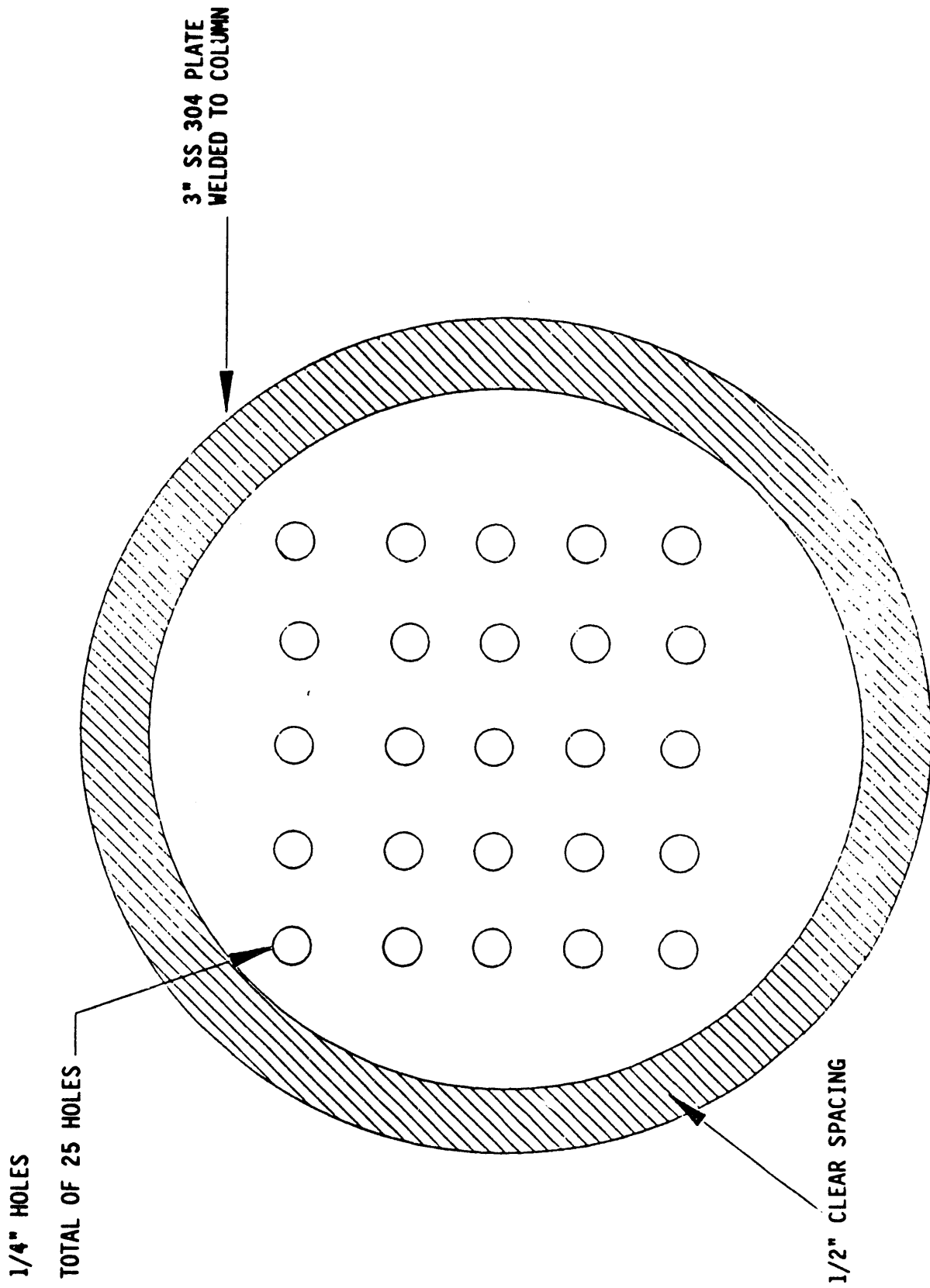
The scrubbing solution is fed to the column by a 1/4-inch SS tubing. The solution is distributed evenly throughout the column cross-sectional area by a distribution plate, shown in Figure 6-11. The liquid distribution plate consists of a 3" OD x 1/4" thick SS plate with 25" x 1/4" drill-thru holes. Also, baffles were incorporated inside the column to redirect the gas and liquid flow. The baffles were made from 3-inch SS washers with a 2-inch concentric hole. The scrubbing solution flows down through the packed section and is collected in a drum where it is neutralized by adding a metered quantity of sodium hydroxide. A sample valve is incorporated on the drum line, where liquid samples are collected after the scrubber.

A premixed gas mixture (containing 2% H<sub>2</sub>S, 20% CO<sub>2</sub>, and 78% N<sub>2</sub>) is injected at the bottom of the column. The gas flow rate is metered using a high accuracy rotameter. The gas mixture exiting the scrubber is sent to the incinerator. Gas samples are pumped to the GC room for continuous gas analysis.

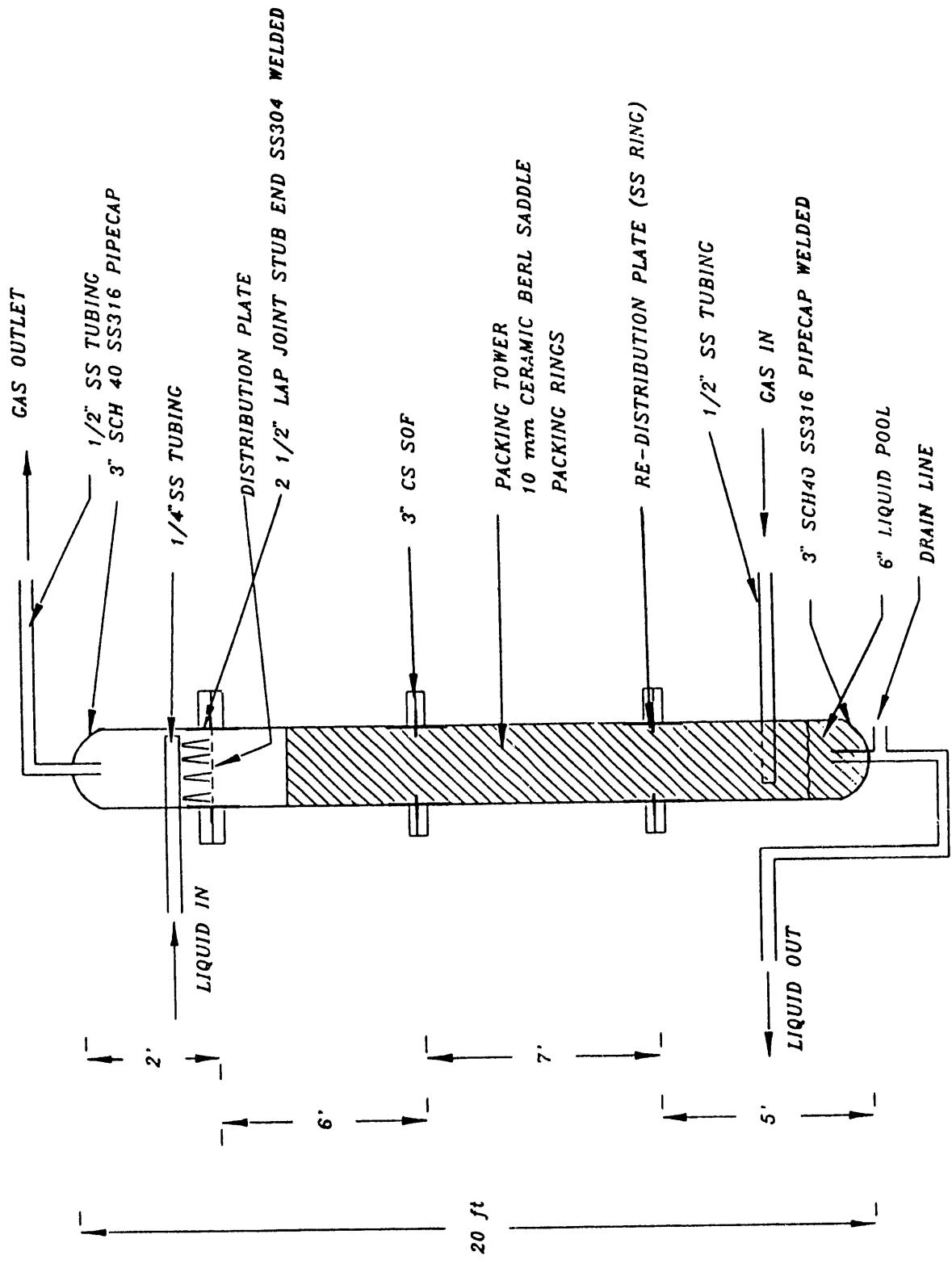
## **B. ABSORPTION COLUMN**

Figure 6-12 shows a schematic diagram of the scrubber. The column consists of a 3" x 21' SS 304 tubing. The column is fabricated in four sections (5, 7, 6, and 3 feet sections), enabling ease of assembly and disassembly. Sections are attached to one another by SS 304 lap joints, and carbon steel slip-on-flanges. A U-shaped standing pipe is incorporated to maintain a 6-inch liquid pool at the bottom of the column, and to prevent column siphoning. Thermocouple, and pressure gauge ports are incorporated at various sections of the column. The column is insulated with a 1-inch thick fiberglass insulating material. The column is clamped to two angle-iron beams, which are bolted to the floor and roof.





**FIGURE 6-11: COLUMN DISTRIBUTION PLATE**



**FIGURE 6-12: SCRUBBER COLUMN**

The column was packed with 10 mm long, ceramic berl-saddle packing rings. The column packings were obtained from Jaeger Products, Inc., Spring, Texas. The height of the packing could be increased or decreased by adding or discharging packing rings.

#### **6.3.4 DESIGN BASIS**

Prior tests using the indirectly heated black liquor gasifier indicated that the CO<sub>2</sub> concentration in the dry gas product was typically in the range of 25 - 30 volume percent. As these tests were conducted at high steam-to-black liquor feed ratios, increased concentrations of CO<sub>2</sub> and hydrogen relative to CO are expected to be found in the product gas due to the influence of the water-gas shift reaction as shown below:



In the commercial gasifier, however, lower steam-to-black liquor ratios will be employed. Based on equilibrium considerations, CO<sub>2</sub> concentrations from the commercial unit are anticipated to be in the range of 15 - 20 volume percent. Furthermore, material balance calculations indicate the expected H<sub>2</sub>S concentration is approximately 2.0 volume percent, assuming a high level of sulfur release.

Based on this data, the simulated gas mixture selected for testing in the scrubbing column was formulated as shown below:

Nitrogen	78%
CO <sub>2</sub>	20%
H <sub>2</sub> S	2%

Note that the substitution of nitrogen for other species expected to appear in the actual gasifier products, i.e., H<sub>2</sub>, CO, CH<sub>4</sub>, etc., is not anticipated to substantially influence the scrubbing results.

### 6.3.5 EXPERIMENTAL RESULTS

Scrubbing tests were conducted for packed column heights of 5, 10, and 15 feet. Each of the tests were conducted at 1 atm pressure (column exit) and ambient temperature, utilizing a simulated product gas containing 2.0 volume percent H<sub>2</sub>S (20,000 ppmv).

Operating data for tests conducted at the different column heights are summarized in Table 6-38. Test data is shown only for test runs where both gas and liquid analyses were available. For each case, the column height, liquid and gas flow rate, H<sub>2</sub>S concentration at column exit as measured by gas chromatograph, and the calculated sulfur recovery efficiency are shown. All tests were conducted utilizing a single batch of premixed sodium carbonate solution with a concentration of 145 g/L as Na<sub>2</sub>CO<sub>3</sub> (84.5 g/l as Na<sub>2</sub>O). While the gas and liquid flow rates were varied in the test matrix, the sulfur-to-sodium ratios entering the column in the gas and liquid phases were held approximately constant, and reflected values typical of a mill circuit operating at a sulfidity of approximately 30 percent (with sulfidity defined as Na<sub>2</sub>S/TTA as Na<sub>2</sub>O).

**TABLE 6-38: OPERATING DATA FOR SCRUBBING TESTS**

<u>SAMPLE DESIGNATION</u>	<u>PACKING HEIGHT (FT)</u>	<u>LIQUID* FLOW RATE (LPM)</u>	<u>GAS** FLOW RATE (SCFM)</u>	<u>H<sub>2</sub>S @ COLUMN EXIT (PPMV)</u>	<u>SULFUR RECOVERY EFFICIENCY (%)</u>
MTCI-7-11-1000	10.000	0.210	3.667	50.000	99.750
MTCI-7-11-1015	10.000	0.330	5.040	271.000	98.645
MTCI-7-12-1145	5.000	0.296	5.040	763.000	96.185
MTCI-7-12-1152	5.000	0.296	5.040	713.000	96.435
MTCI-7-13-1022	15.000	0.296	5.040	140.000	99.300
MTCI-7-13-1030	15.000	0.137	2.330	447.000	97.765

NOTE: Column was operated at 1 atm exit pressure and ambient temperature.

\*Scrubbing liquid consisted of aqueous mixture of sodium carbonate at concentration of 145 g/L as Na<sub>2</sub>CO<sub>3</sub> (84.8 g/L as Na<sub>2</sub>O).

\*\*Simulated gas contained 20% CO<sub>2</sub>, 2.0% H<sub>2</sub>S, and balance nitrogen.

As seen in Table 6-38, high sulfur recovery was achieved at all test conditions, varying from 96.2 to 99.75 percent. H<sub>2</sub>S concentrations in the simulated gas at the column exit ranged from 50 to 763 ppm. In a commercial integrated system, where the fuel gas is combusted to generate steam, this would translate to stack SO<sub>2</sub> emissions in the range of 12 to 190 ppmv. These low emission values represent a significant improvement compared to typical Kraft recovery boilers.

Liquid samples of the rich solution were collected at the column exit for each of the test points shown in Table 6-38. These liquid samples were sent to Weyerhaeuser's laboratories for analysis. The results of these analyses are shown in Table 6-39 and include sodium, total sulfur and total carbonate. The calculated sulfidity is also shown. Note that it is assumed that all of the sulfur is present in reduced form since oxidation of the sulfur species can only occur due to incidental air exposure during sampling or analysis.

**TABLE 6-39: ANALYSIS OF LIQUID SAMPLES FROM SCRUBBER BOTTOMS**

<u>SAMPLE DESIGNATION</u>	<u>SODIUM Na (%)</u>	<u>SULFUR S (%)</u>	<u>CARBONATE CO<sub>3</sub> (%)</u>	<u>SULFIDITY* (%)</u>
MTCI-7-11-1000	5.64	1.20	8.43	30.59
MTCI-7-11-1015	5.45	1.13	7.75	29.81
MTCI-7-12-1145	5.66	1.06	7.96	26.92
MTCI-7-12-1152	5.53	1.07	7.56	27.81
MTCI-7-13-1022	5.52	1.12	7.73	29.17
MTCI-7-13-1030	5.51	1.03	7.92	26.87

\*Sulfidity is calculated as total sulfur as Na<sub>2</sub>O divided by total sodium as Na<sub>2</sub>O.

As seen in Table 6-39, the desired rich solution (green liquor) sulfidity range (25-35%) is achievable while maintaining low sulfur breakthrough in the gas phase. These results provide confirming evidence that green liquor can be

regenerated at high efficiency using the simple alkaline scrubbing technique proposed in the MTCI integrated recovery process.

**Table 6-40** shows the results of material balances for the gas and liquid phase samples. The H<sub>2</sub>S scrubbing rate is determined for the liquid phase based on the ratio of sulfur to sodium at the column inlet and outlet, and the known liquid flow rate and sodium concentration of the inlet as shown below:

$$N_{1,H_2S} \text{ (gmole/h)} = L \text{ (LPM)} \times 145 \text{ (g/l as Na}_2\text{CO}_3) \times 2/106 \times S(\%)/Na(\%) \times 23/32 \quad (8)$$

where S and Na are the sulfur and sodium concentrations of the sample liquid (rich solution) in weight percent, and L is the liquid flow rate in liters per minute.

**TABLE 6-40: MATERIAL BALANCES FOR SCRUBBER TESTS**

SAMPLE DESIGNATION	H <sub>2</sub> S SCRUBBING RATE (gmole/min)		GAS/LIQUID DEVIATION	CO <sub>2</sub> ** SCRUBBING RATE (gmole/min)	CALCULATED CO <sub>2</sub> AT EXIT (Vol.%)	MEASURED CO <sub>2</sub> AT EXIT (Vol.%)
	BASED ON* LIQUID SAMPLE	BASED ON** GAS SAMPLE				
MTCI-7-11-1000	0.088	0.088	0.000	0.042	19.040	19.020
MTCI-7-11-1015	0.135	0.119	-11.900	0.041	19.320	19.910
MTCI-7-12-1145	0.109	0.116	6.400	0.032	19.470	19.620
MTCI-7-12-1152	0.113	0.116	2.600	0.020	19.670	19.610
MTCI-7-13-1022	0.118	0.120	1.700	0.030	19.510	19.770
MTCI-7-13-1030	0.050	0.054	8.000	0.020	19.280	18.700

\*Calculated based on difference in total sulfur in liquid from inlet to outlet of column.

\*\*Calculated based on difference in total sulfur in gas from inlet to outlet of column.

\*\*\*Calculated based on difference in total carbonate in liquid from inlet to outlet of column.

Similarly, the H<sub>2</sub>S scrubbing rate is independently determined based on the gas flow rates, known H<sub>2</sub>S concentration at the inlet, and measured H<sub>2</sub>S concentration at the exit. By comparison of the independent values calculated from liquid and gas samples (columns two and three of Table 6-40), it is seen that two measurements are relatively consistent, with material balance deviations of typically less than 10 percent. These deviations are of the order anticipated for rotameter readings of the liquid and gas flow rates.

The CO<sub>2</sub> scrubbing rate is shown in column five of Table 6-40. The CO<sub>2</sub> scrubbing rate is calculated using only the liquid data. While the H<sub>2</sub>S concentration in the gas phase changes from inlet to outlet by a factor of almost 99 percent, the CO<sub>2</sub> concentration varies by only 2.5 - 5.0 relative percent. Since this is of the order of accuracy for the gas chromatograph, reliable CO<sub>2</sub> scrubbing rates cannot be obtained from gas data. However, absorption of CO<sub>2</sub> in the liquid phase results in a significant change in the ratio of total carbonate to sodium which can be used as an accurate measure of the CO<sub>2</sub> scrubbing rate as follows:

$$N_{1,CO_2} = L(LMP) \times 145(\text{g/l as } N_2CO_3)/106 \times (2 - R)/R \quad (9)$$

Where R is given by:

$$R = \text{Na}(\%)/CO_3 (\%) \times 60/23 \quad (10)$$

It should be noted that most of the liquid samples collected from the scrubber bottom contained a precipitated material. Analysis of this precipitate identified it as primarily sodium bicarbonate. The precipitate, which apparently forms as a result of supersaturating the solution due to the absorption of CO<sub>2</sub>, was typically found to be of the order of 5 g/l. Since the calculation method discussed above, which considers only liquid data, does not account for precipitate losses, the calculated CO<sub>2</sub> absorption rate is anticipated to error on the low side. However, magnitude of this error is estimated to be only about 20 percent for most cases.

Based on the CO<sub>2</sub> scrubbing rate as determined using the above equations, the CO<sub>2</sub> concentration at the column exit can be calculated as shown in column

six of table 6-40. These are compared with the actual measured values in column seven of Table 6-40.

Table 6-41 presents a summary of results pertaining to the calculation of absorption rate constants for H<sub>2</sub>S and CO<sub>2</sub> in the alkaline solution. The basis for these calculations were described in the prior section; however, some additional notes are made here.

**TABLE 6-41: ABSORPTION RATE CONSTANTS AND SELECTIVITY**

<u>SAMPLE DESIGNATION</u>	<u>H<sub>2</sub>S* ABSORPTION RATE (g/mole/min)</u>	<u>CO<sub>2</sub>* ABSORPTION RATE (g/mole/min)</u>	<u>H<sub>2</sub>S** LMPD ATM</u>	<u>CO<sub>2</sub>** LMPD ATM</u>	<u>H<sub>2</sub>S*** Kg a</u>	<u>CO<sub>2</sub>*** Kg a</u>	<u>H<sub>2</sub>S/CO<sub>2</sub> SELECTIVITY</u>
MTCI-7-11-1000	0.088	0.042	0.0018	0.1911	99.61	0.448	222
MTCI-7-11-1015	0.127	0.041	0.0027	0.1935	94.71	0.432	219
MTCI-7-12-1145	0.113	0.032	0.0041	0.1947	119.11	0.671	178
MTCI-7-12-1152	0.114	0.02	0.0039	0.1961	119.81	0.416	288
MTCI-7-13-1022	0.119	0.03	0.0024	0.1948	66.85	0.209	319
MTCI-7-13-1030	0.052	0.02	0.0034	0.1934	20.96	0.141	149

\*H<sub>2</sub>S absorption rate based on average of values calculated from both gas and liquid sample data. CO<sub>2</sub> absorption rate based on liquid sample only.

\*\*LMPD is the log mean pressure driving force.

\*\*\*Kg a has units of gmole/min/ft<sup>3</sup>/atm and is defined as the absorption rate divided by product of LMPD and column volume.

First, in order to calculate the equilibrium partial pressures of CO<sub>2</sub> and H<sub>2</sub>S above the scrubber bottoms liquid, the concentrations of Na<sub>2</sub>CO<sub>3</sub>, NaHCO<sub>3</sub>, and NaHS are needed. However, since these constituents were not measured directly, they must be calculated based on an elemental balance approach.



That is, the three species must satisfy the measured constraints with respect to sodium, total sulfur, and total carbonate as follows:

Sulfur balance:

$$\text{NaHS(gmole/l)} = \frac{\text{S(\%)}{\text{Na(\%)}} \times \frac{23}{32} \times 145(\text{g/l as Na}_2\text{CO}_3) \times \frac{2}{106} \quad (11)$$

Total carbonate balance:

$$\begin{aligned} \text{Na}_2\text{CO}_3(\text{gmole/l}) + \text{NaHCO}_3(\text{gmole/l}) \\ = \frac{\text{CO}_3(\%)}{\text{Na(\%)}} \times \frac{23}{60} \times 145(\text{g/l as Na}_2\text{CO}_3) \times \frac{2}{106} \end{aligned} \quad (12)$$

and, Total sodium balance:

$$\begin{aligned} 2 \times \text{Na}_2\text{CO}_3(\text{gmole/l}) + \text{NaHCO}_3(\text{gmole/l}) + \text{NaHS(gmole/l)} \\ = 145(\text{g/l as Na}_2\text{CO}_3) \times \frac{2}{106} \end{aligned} \quad (13)$$

Solution of the above three simultaneous equations yield values for  $\text{Na}_2\text{CO}_3$ ,  $\text{NaHCO}_3$  and  $\text{NaHS}$  which can then be substituted into the appropriate expressions for calculating liquid equilibrium partial pressures. Note that these calculations assume that the concentration of  $\text{Na}_2\text{S}$  is negligible. It can be shown, for the measured pH range of the scrubber bottoms, that this approximation is quite accurate.

As seen in Table 6-41, the pressure driving force for  $\text{CO}_2$  is relatively constant for all runs. This is due to the fact that the gas phase  $\text{CO}_2$  concentration change is small over the entire column and the typical equilibrium concentration of  $\text{CO}_2$  above the liquid at the scrubber bottom is only 0.5 - 0.8 volume percent (at 1 atm).

In contrast, the driving force for  $\text{H}_2\text{S}$  is largely dictated by the partial pressure at the column exit, i.e., the pinch point is at the column exit. However, examination of the  $\text{H}_2\text{S}$  equilibrium concentration above the scrubber bottom liquid shows that this value begins to approach the gas phase  $\text{H}_2\text{S}$  inlet concentration in some cases. Note that the equilibrium partial pressure of

H<sub>2</sub>S is a strong function of the bicarbonate concentration, which is influenced by the quantity of CO<sub>2</sub> absorbed over the column. This point is significant since an improperly sized absorption column can result in excessive levels of CO<sub>2</sub> absorption leading to an equilibrium pinch point at the column bottom, thus limiting the achievable sulfur scrubbing efficiency.

The seventh and eight columns of Table 6-41 summarizes the calculated Kg a values for H<sub>2</sub>S and CO<sub>2</sub> for each run. Note that the values of Kg a are relatively constant, with the exception of run No. MTCI-7-13-1030. The consistency of these values indicates that the simplified column model is valid over the limited test conditions. The low Kg a values for the last run in Table 6-30 is believed to be due to insufficient column wetting resulting from the low liquid and gas rates used in this test.

The absorption selectivity of H<sub>2</sub>S relative to CO<sub>2</sub> (on gmole/ft<sup>3</sup>/atm) is seen to be of the order of 200:1. Note that these values can be used for design purposes only when similar column packings and operating conditions are employed.

An in-line static mixer, 1/2" diameter by 9" in length, was tested as an alternative to the packed column. The scrubbing efficiency was approximately 80 to 84 percent in less than 10 milliseconds. A two-stage in-line static mixer or a static mixer followed by a short packed column is predicted to absorb 99.9 percent of the H<sub>2</sub>S with very little or no absorption of CO<sub>2</sub>. This will be experimentally verified during the next phase. There is evidence in the literature that CO<sub>2</sub> absorption was slower than the H<sub>2</sub>S absorption rate and that CO<sub>2</sub> absorption has an induction time of over 20 milliseconds. Thus, short contact time static mixers avoid CO<sub>2</sub> absorption (Ref.: R.W. Hohfeld, Dow Chemical, "Selective Absorption of H<sub>2</sub>S from Syngas," SPE 7972, April 18-20, 1979, Ventura, CA).

It is important to note that all tests were run at relatively low temperatures (70°F). As previously mentioned, low temperatures favor high H<sub>2</sub>S selectively. In actual commercial systems, where the process gas must be

cooled prior to scrubbing, operation at such low temperatures may be impractical. Thus, additional tests are needed to characterize the scrubbing performance at higher temperatures in the range of 100 - 140°F.

#### 6.4 SUMMARY AND CONCLUSIONS

Ten black liquor tests were done during this year - seven of which are reported here for which the data analysis were completed. Samples from the last test were not yet analyzed by Weyerhaeuser Research Center. Two other tests were sponsored by private clients on their liquors and data are reported because of confidentiality. One of the latter cases used liquor from a soda pulp mill having no sulfur and the other from a bleached chemi thermo-mechanical (BCTMP) pulp mill. The results from one Kraft liquor, one cross recovery liquor, and one neutral sulfite semichemical (NSSC) liquor were analyzed and are reported here. The results are summarized in Table 6-42.

**TABLE 6-42:**  
**SUMMARY OF BLACK LIQUOR TESTS**

<u>TEST DATE</u>	<u>LIQUOR FEED TYPE</u>	<u>GASIFIER TEMP. (°F)</u>	<u>TEST DURATION (hours)</u>	<u>WET FEED RATE (lbs/hr)</u>	<u>DRY FEED RATE (lbs/hr)</u>	<u>STEAM RATE (lbs/hr)</u>	<u>SULFUR GASIF. EFFIC. (%)</u>	<u>CARBON GASIF. EFFIC. (%)</u>
1/24/90	Kraft	1138	2	18	10	35	--	--
1/24/90	Kraft	1130	5	23	13	35	--	--
1/24/90	Kraft	1050	2.5	40	23	35	--	--
2/23/90 <sup>1</sup>	Cross-Recovery	1145	8	19.3	12.7	38.2	91.2	88.2
3/07/90 <sup>2</sup>	Kraft	1150	7.5	27.1	15.3	33.7	90.5	83.3
3/21/90 <sup>2</sup>	Kraft	1143	7.5	29.3	16.6	37.8	88.3	84.9
3/27/90 <sup>3</sup>	Kraft	1160	6	28.8	16.3	24.10	81.4	79.5
9/18/90	NSSC	1130	8	21.5	8	36	84.5	83.1
9/24/90	NSSC	1150	5.5	26.8	10	38	85.2	84.9

<sup>1</sup>Scrubbed product gas fired in pulse combustor.

<sup>2</sup>Gas scrubbing.

<sup>3</sup>Gas recycle for fluidization.

These tests proved that the MTCI indirect gasification process works well for any type of black liquor, separating sulfur as H<sub>2</sub>S from the sodium, which reports mainly as Na<sub>2</sub>CO<sub>3</sub>, producing consistent quality hydrogen-rich, medium-Btu gas. This process enables pulp and paper mills to recover chemicals and energy without smelt formation from Kraft, sulfite (NSSC or BCTMP) or combination (cross recovery) liquors. This process offers great flexibility in selecting pulping method to meet market demand.

The tests done so far have shown that in the temperature range of approximately 1150°F (625°C), carbon gasification efficiencies of up to 88 percent and sulfur gasification efficiencies of up to 89 percent were achievable. Significant sulfate reduction is achieved 60 percent for NSSC liquor and 88 percent for Kraft liquor, without employing carbon or char recycle. It is possible that the sulfate reduction may be improved by char recycle.

Gas recycle for fluidization has been shown to work well. Using product gas in the pulse combustor firing has been demonstrated here. For Kraft application, the H<sub>2</sub>S gas is scrubbed with sodium carbonate solution producing green liquor. This has been demonstrated very convincingly. The formation of sulfur gases other than H<sub>2</sub>S has been found to be negligible, usually less than 10 ppm. Overall, sulfur recovery is expected to be over 99 percent and sodium recovery, over 99.9 percent. Further process optimization to improve the net gas yield and reduce the gas fired as fuel is being planned for testing in early next year.

The present investigation has confirmed that high sulfur recovery efficiencies exceeding 99 percent, can be achieved under the anticipated operating conditions of MTCI's integrated recovery process using a simple, single column, alkaline scrubbing technique. These results suggest that there exists no major technical barriers to regenerating a high sulfidity green liquor in the MTCI process. Furthermore, the results indicate that the MTCI process has a potential for reducing emissions of SO<sub>2</sub> from energy recovery equipment compared to the conventional Kraft recovery boiler.

## **SECTION 7.0**

### **DEVELOPMENT TESTING**

The bench-scale testing described in Section 6.0 provided a significant data base regarding the process chemistry and performance parameters for the indirect black liquor gasifier. This information provided essential input to the preparation of a rational process design for the scale-up field test unit as discussed in Section 8.0.

In a parallel effort, development work was undertaken to construct and test a full-scale pulse heater module. The objective of this work was to ensure that key hardware components would perform reliably in field service.

In the following section, this hardware development work is highlighted. This includes discussion of cold flow fluidization tests, combustor development, and hot fluidization tests.

#### **7.1 COLD FLOW FLUIDIZATION TESTS**

The promotion of good solid circulation and effective heat transfer is key to the successful operation of the indirect fluid-bed gasifier. Sufficient solid circulation is needed to ensure the uniform distribution of heat and reactant throughout the bed. Effective heat transfer is vital in order to protect overheating of the fire-tube wall surfaces and prevent melting of the bed material.

While conventional fluid beds containing little or no immersed tube surfaces are known for excellent circulation and heat transfer characteristics, few studies have been conducted to establish the properties of fluid beds containing closely spaced tube bundles. For this reason, cold flow fluidization tests were performed to assess the fluidization characteristics of a bed containing an array of closely spaced tubes.

The full-scale prototype pulse heater module is constructed of 61 triangular-pitch fire tubes (1½" nominal pipe) spaced on 3-inch centers. The fire tubes are approximately 9 feet in length. The cold flow model was constructed at a one-third scale of the prototype pulse heater module. The model tube bundle was constructed from 61 ½-inch O.D. acrylic tubes spaced on 1-inch centers. The 3 feet long tube bundle was inserted into a 3' x 1' fluid bed. The upper portion of the fluid bed was constructed from acrylic plate allowing visual inspection of the bed. Photographs of the tube bundle and test bed are shown in Figures 7-1 and 7-2.

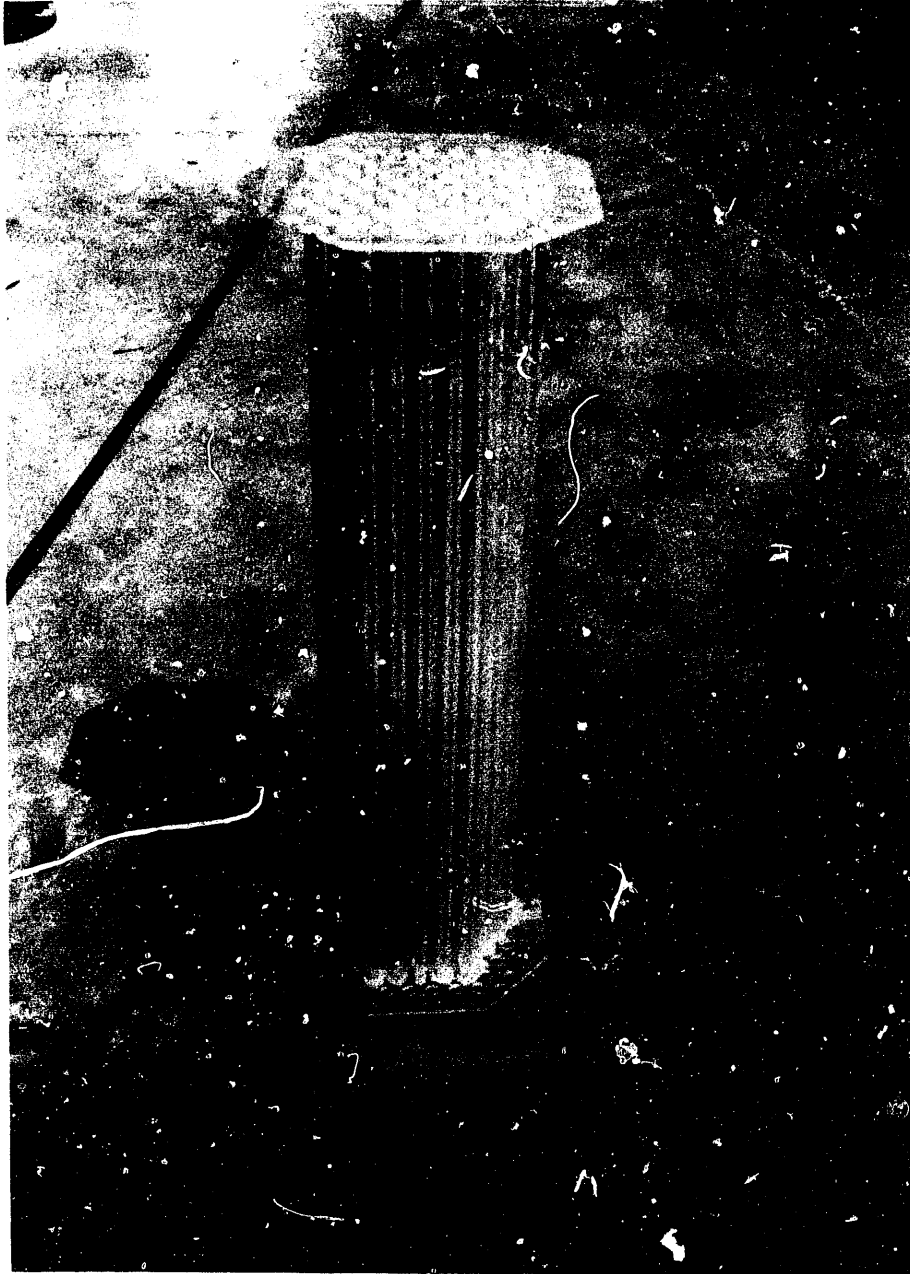
A primary objective of the cold flow model tests was to determine if fluid bed-side heat transfer coefficients were diminished by the presence of the tube bundle. In order to accomplish this, a heat transfer probe was constructed using a standard electric cartridge heater. The cartridge heater (OMEGA model CAR-3025) consisted of a 1/2-inch O.D. by 2 3/8-inch long metal cartridge which was internally heated by an electric resistance coil. An integral thermocouple was welded to the inside wall of the cartridge.

The cartridge heater supplied a constant heat flux of 26 watt/in<sup>2</sup> (12,767 Btu/ft<sup>2</sup>/hr) to the metal surface. This heat flux is similar to the actual heat flux for the prototype pulse heater module (8,000 - 10,000 Btu/ft<sup>2</sup>/hr). Since the cartridge heat flux is fixed, the relative fluid bed heat transfer coefficient is directly related to the cartridge wall surface temperature as described below:

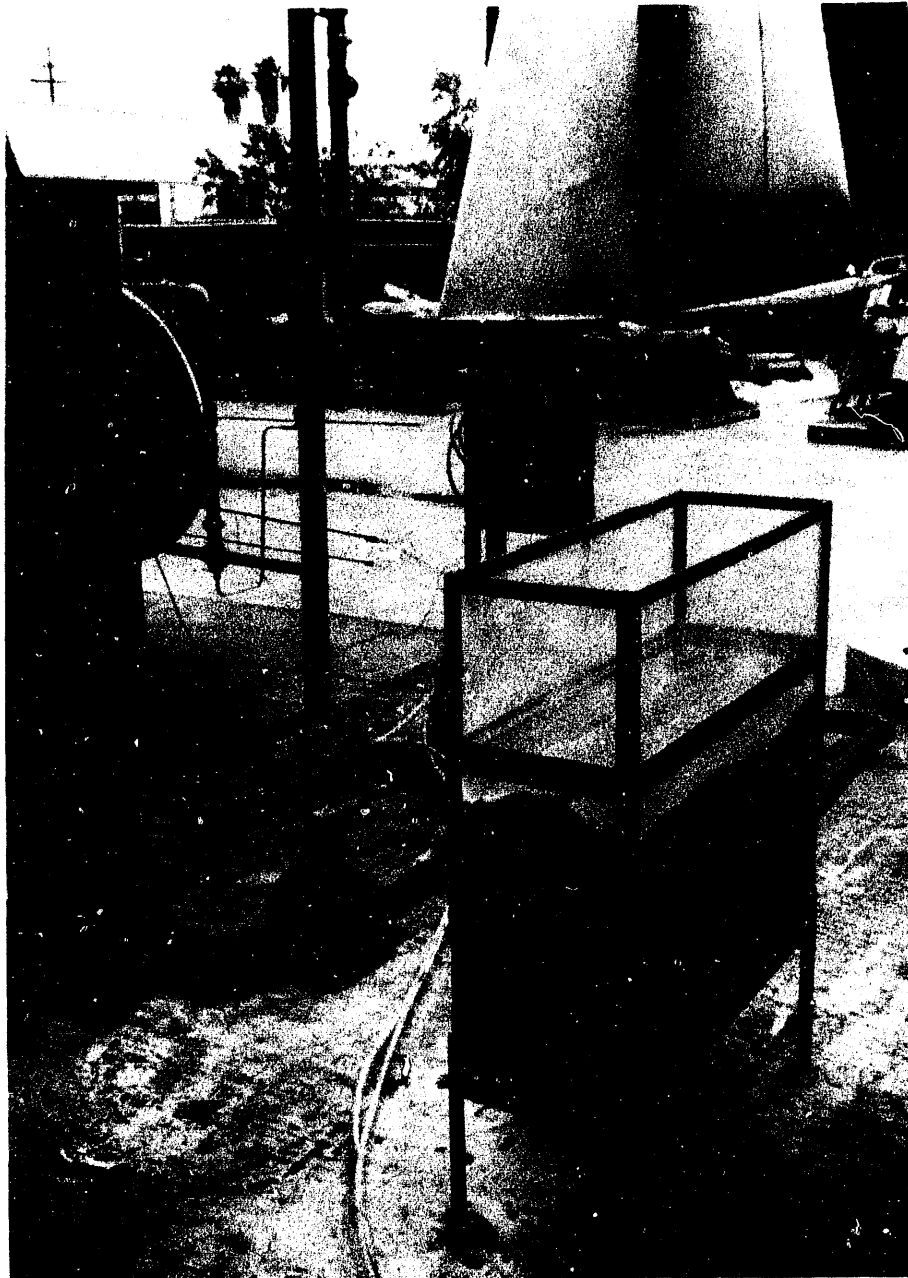
$$q = h_B (T_W - T_B)$$

where  $q$  is the cartridge heat flux,  $h_B$  is the bed side heat transfer coefficient,  $T_W$  is the measured cartridge wall temperature, and  $T_B$  is the bed temperature (ambient).

Using the cartridge heater as a heat transfer probe, relative bed heat transfer coefficients were measured in the fluid bed both with and without the tube bundle inserted. When the tube bundle was removed, the cartridge wall temperature was approximately 206°F and 178°F for fluidization velocities of 1.5 ft/sec and 2.0 ft/sec, respectively. The tube bundle was then placed in



**FIGURE 7-1: TUBE BUNDLE**



**FIGURE 7-2: TEST BED**



the bed and the heat flux probe was inserted approximately mid-way into the bundle. The steady-state cartridge wall temperatures were observed to be virtually unchanged from the previous test at similar fluidization velocities. This indicated that the presence of the tube bundle had little or no effect on the average bulk fluid-bed heat transfer coefficient.

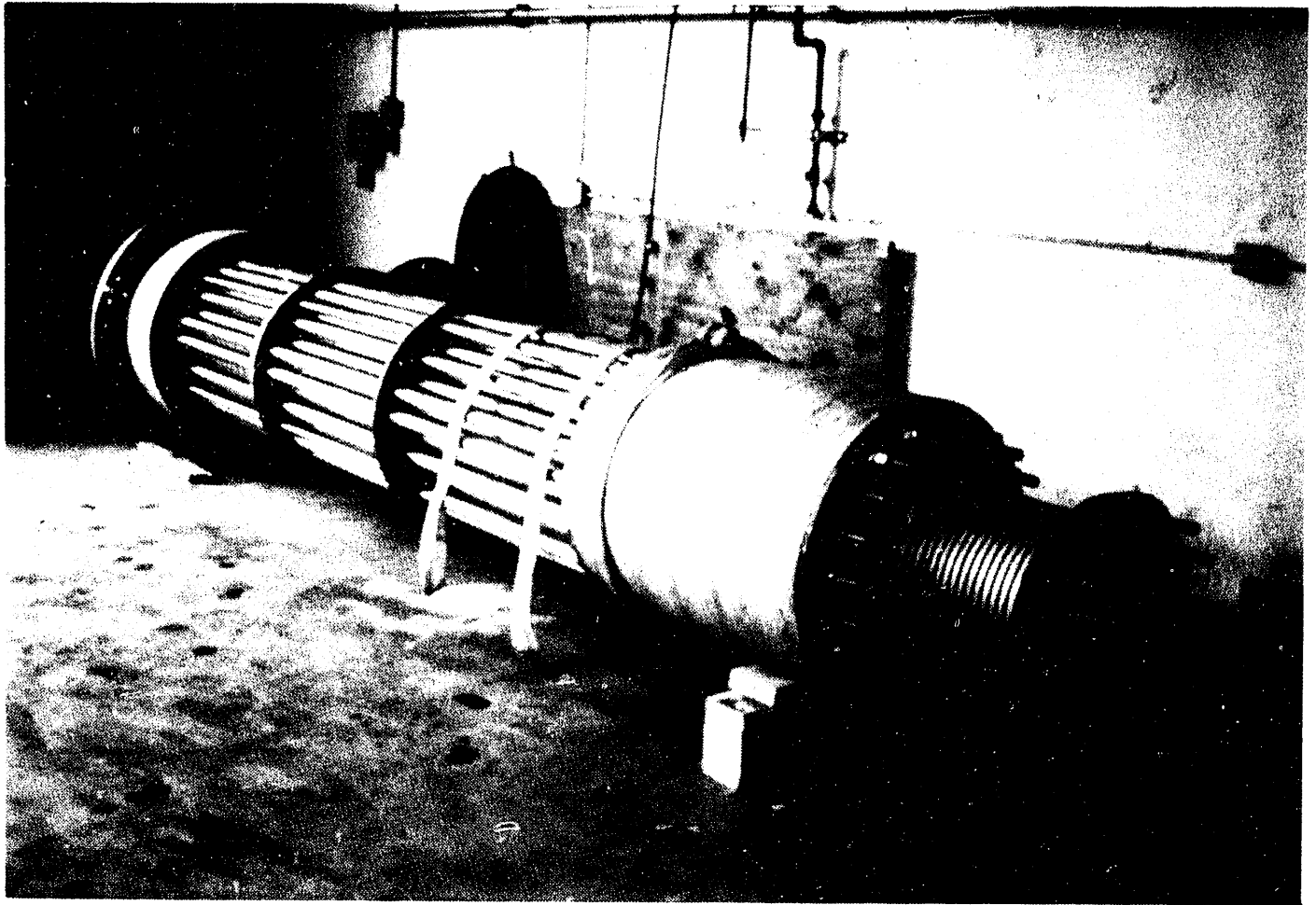
It is important to note that while the average bulk heat transfer coefficient may be similar, significant local variations in the solids to wall heat transfer coefficient may occur around the circumference of the tubes. Studies have shown that a relative dead zone can occur on the leeward side of a horizontal heat transfer tube. In some cases, measured differences in heat transfer coefficients between the sides of the tube, where interstitial velocities are high, and the leeward side of the tube, where relative dead zones may exist, of several fold have been reported.

For this reason, it is vital that full-scale hot testing be performed to assess the performance of the pulse heater module under simulated gasifier conditions. Progress in this area is discussed in the following section.

## **7.2 COMBUSTOR DEVELOPMENT**

A program was initiated to construct a prototype pulsating heater module with a nominal firing rate of  $5 \times 10^6$  Btu/hr and capable of processing 1/2 TPH of black liquor solids. The 1 TPH field test unit would be furnished with two of these combustor modules to allow increased flexibility and turndown.

Based on heat transfer data collected at the bench-scale, a tube bundle was designed to incorporate 61, 1½-inch diameter, partially shielded tubes arranged in a hexagonal staggered array and spaced on 3-inch centers. The tube bundle included several vertical baffles and longitudinal tie rod supports. The tube bundle was fabricated by REPCO Engineering and is shown in Figure 7-3. This tube bundle forms the resonant member for the pulsating heat module and is fully immersed in the fluid bed under operating conditions.



**FIGURE 7-3: FULL-SCALE PULSE HEATER MODULE**

An exploded view of the pulse heater module is shown in Figure 7-4. The pulse heater module comprises four main elements: an air inlet plenum, pulse combustor, tube bundle, and flue gas exit plenum. Air is supplied to the air inlet plenum from a forced draft fan. The air is then aspirated into the pulse combustor through four separate aerovalves. Fuel is injected into the pulse combustor through a series of distributed nozzles surrounding the aerovalve. The combustion products then enter the tube bundle where heat is released to the fluid bed. The cooled combustion gases are collected in a flue gas plenum and vented through a muffled stack.

A schematic of the pulse combustor and the tube sheet acting as an interface with the tube bundle is shown in Figure 7-5.

The combustion chamber is insulated with Shamrock 881 plastic refractory 2-2.5 inches thick and hammered onto approximately 1000 studs on 1-inch centers with a rubber-tipped bench rammer. The studs are 3/8" x 1.5" 304 SS bolts welded into place. The refractory is cured in an oven to a final temperature of 1000°F after air drying for 8 hours. This takes approximately 24 hours. The shell of the combustor is water jacketed and requires a cooling water flow of 2 gpm. The water jacket is equipped with a pressure relief valve set to open at 240°F. The combustion chamber is 27.5 inches diameter and 12 inches deep. Figure 7-6 shows the pulse combustor under different stages of construction. In order to promote uniform fuel/air mixing within the pulsating combustion chamber the unit has been furnished with four aerovalves. Note that the use of multiple aerovalves represents a new advance in MTCI's pulse combustor technology. A photograph of the installed pulse combustor with the air plenum removed is shown in Figure 7-7. The four aerovalves are readily visible.

The pulse combustor is also furnished with a pre-mixed pilot and flame supervision system. A Fire Eye UV flame sensor is used to monitor flame status and de-energize fuel solenoid valves in the case of a flame failure.

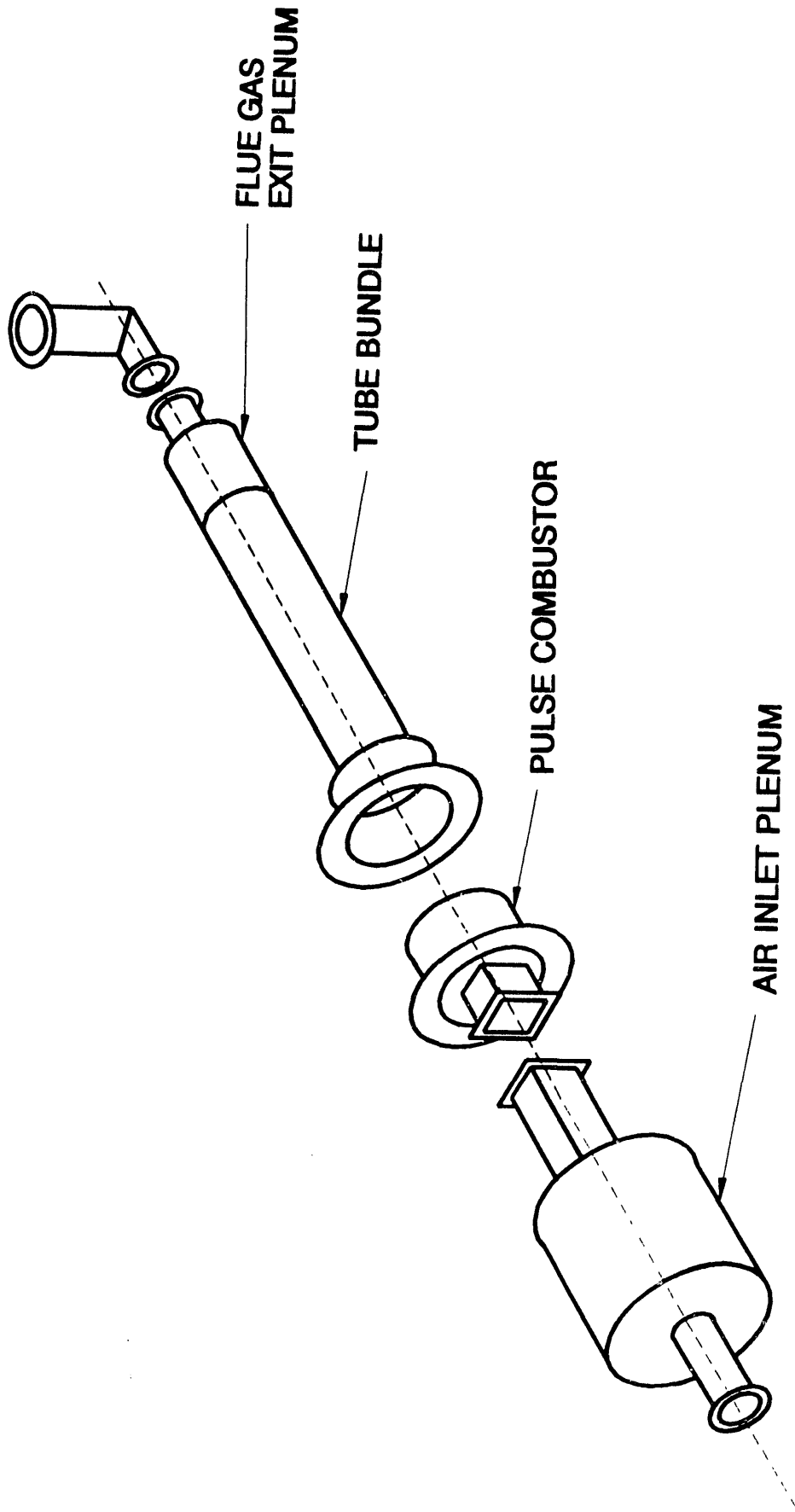
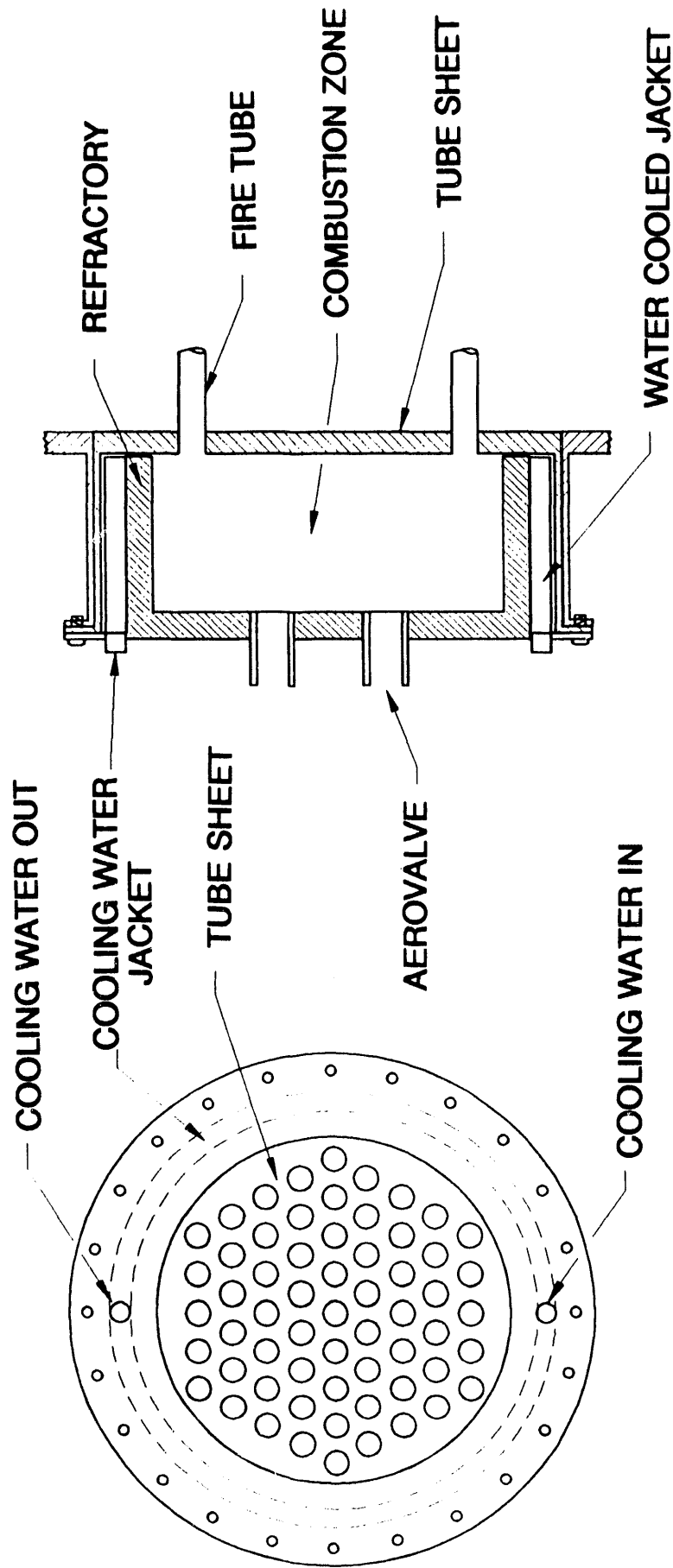
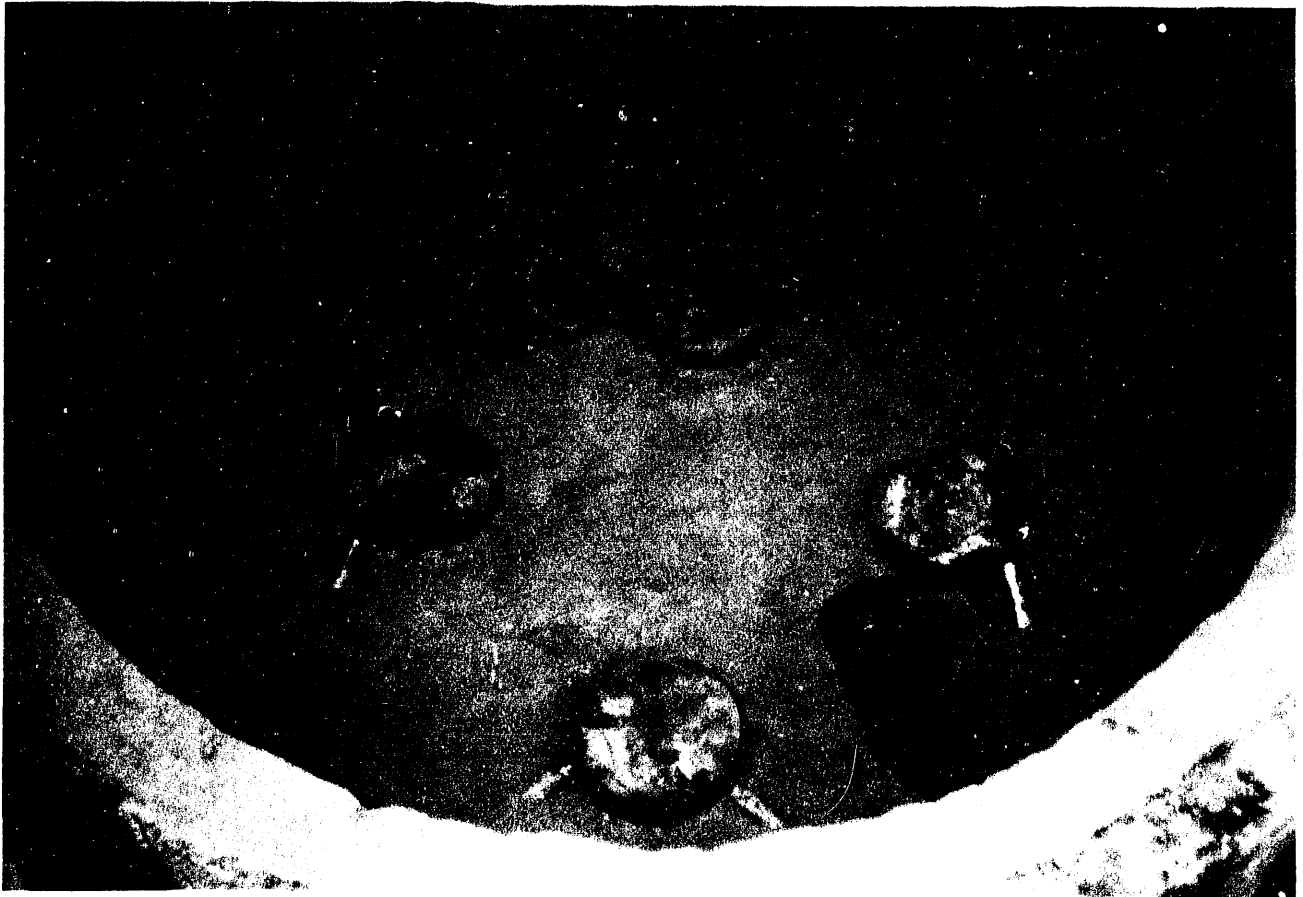
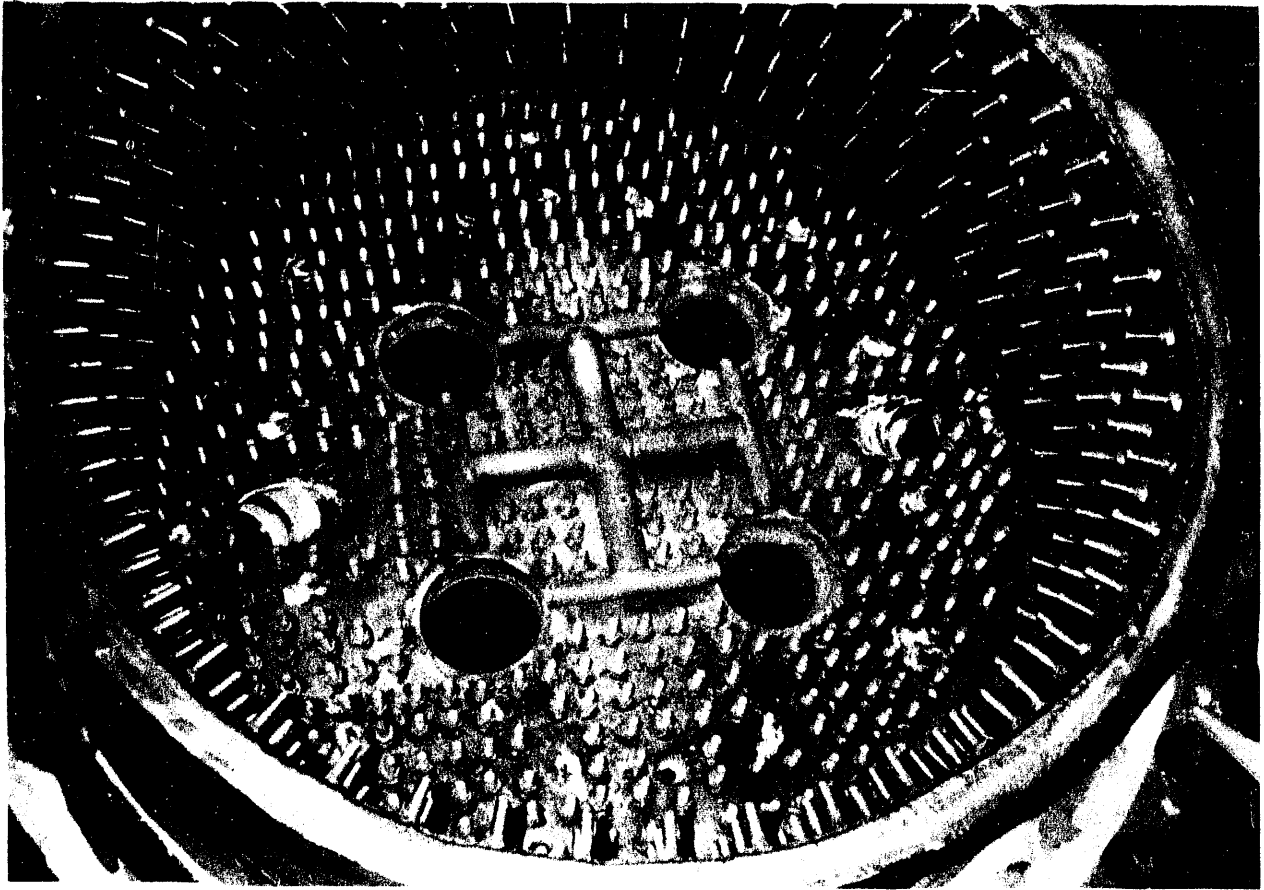


FIGURE 7-4: EXPLODED VIEW OF PULSE HEATER MODULE



**FIGURE 7-5: COMBUSTOR TUBE BUNDLE INTERFACE**



**FIGURE 7-6: PULSE COMBUSTOR DURING CONSTRUCTION**



**FIGURE 7-7: FULL-SCALE PULSE COMBUSTOR MODULE**

A significant effort was expended to develop a reliable flame shield for the tube sheet which is directly exposed to the high-temperature environment of the pulsating combustor. The flame shield serves to protect the metal tube sheet from flame impingement and to limit the heat flux through the tube sheet so as to avoid over temperature conditions which would lead to bed agglomeration.

A removable thin gauge metal radiation shield was initially tested (Figure 7-8); however, severe thermal warping of the metal shield resulted in a misalignment with the tube sheet holes thereby restricting the outflow of combustion products. In a second design, a 1/2-inch metal flame shield was constructed with a refractory lining attached to the inner surface (Figure 7-9). Metal sleeves were employed inside the refractory portion of the shield to assist in alignment of the flame shield holes with the main tube sheet. However, the metal sleeves, being thermally isolated, rapidly corroded by high temperature oxidation. In addition, thermal warpage of the metal shield plate resulted in refractory damage.

In a final configuration (Figure 7-10), plastic refractory was air hammered directly onto studs covering the tube sheet itself. PVC tubes were inserted into the fire tubes to provide a template for the flame shield holes. These tubes were later removed after air drying the refractory. Initial tests have indicated reliable performance for this design approach.

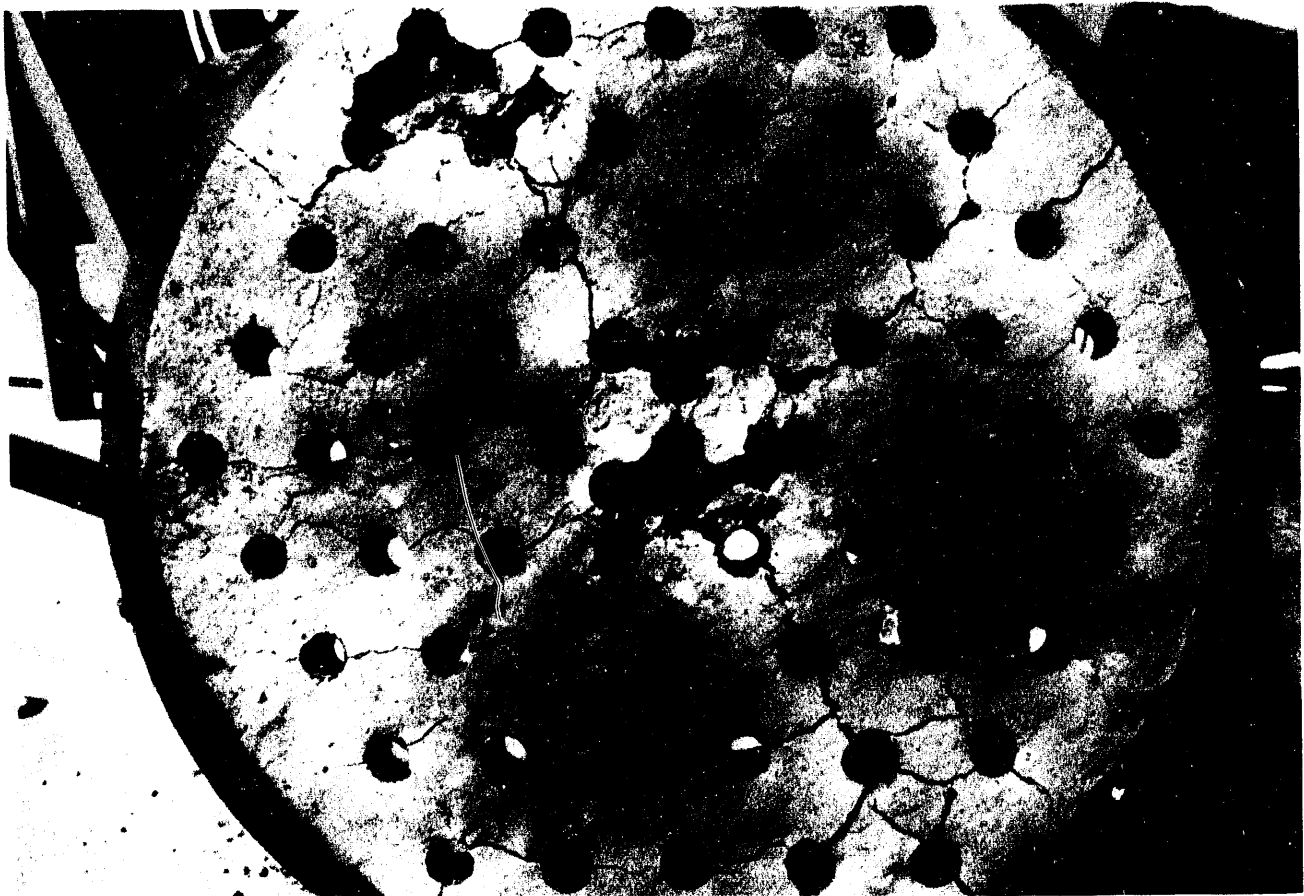
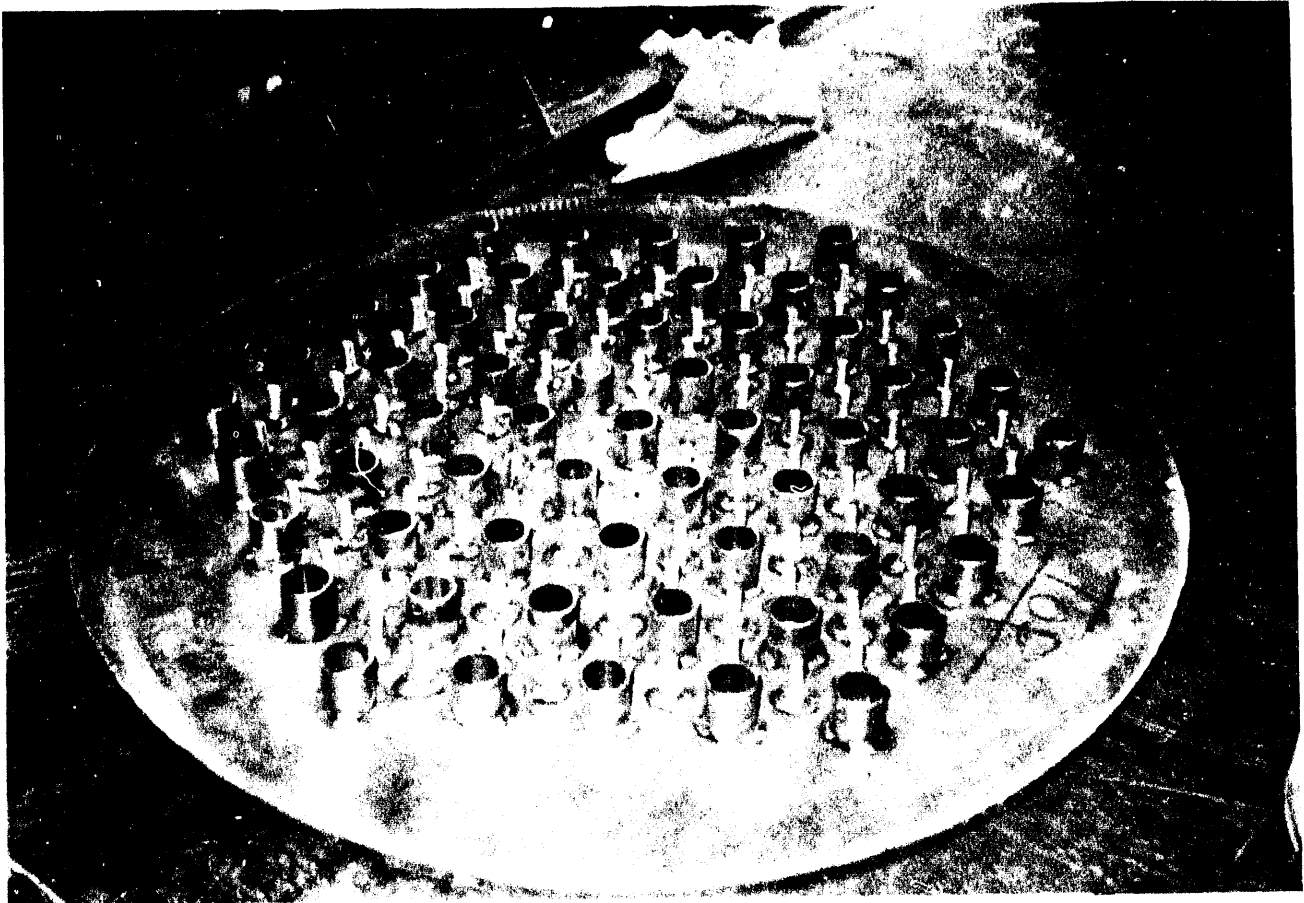
### **7.3 HOT FLUIDIZATION TESTS**

A fluidized bed test rig was constructed in order to assess the performance of the prototype pulse heater module. The fluid bed test rig comprises several main components including the fluid-bed reactor with contained pulse heater module, an air blower for supplying fluidization air, a bucket elevator for discharging bed solids, and a storage hopper to receive bed solids. A photograph of the hot fluidization test rig is shown in Figure 7-11. A schematic of the system with each component identified is shown in Figure 7-12.

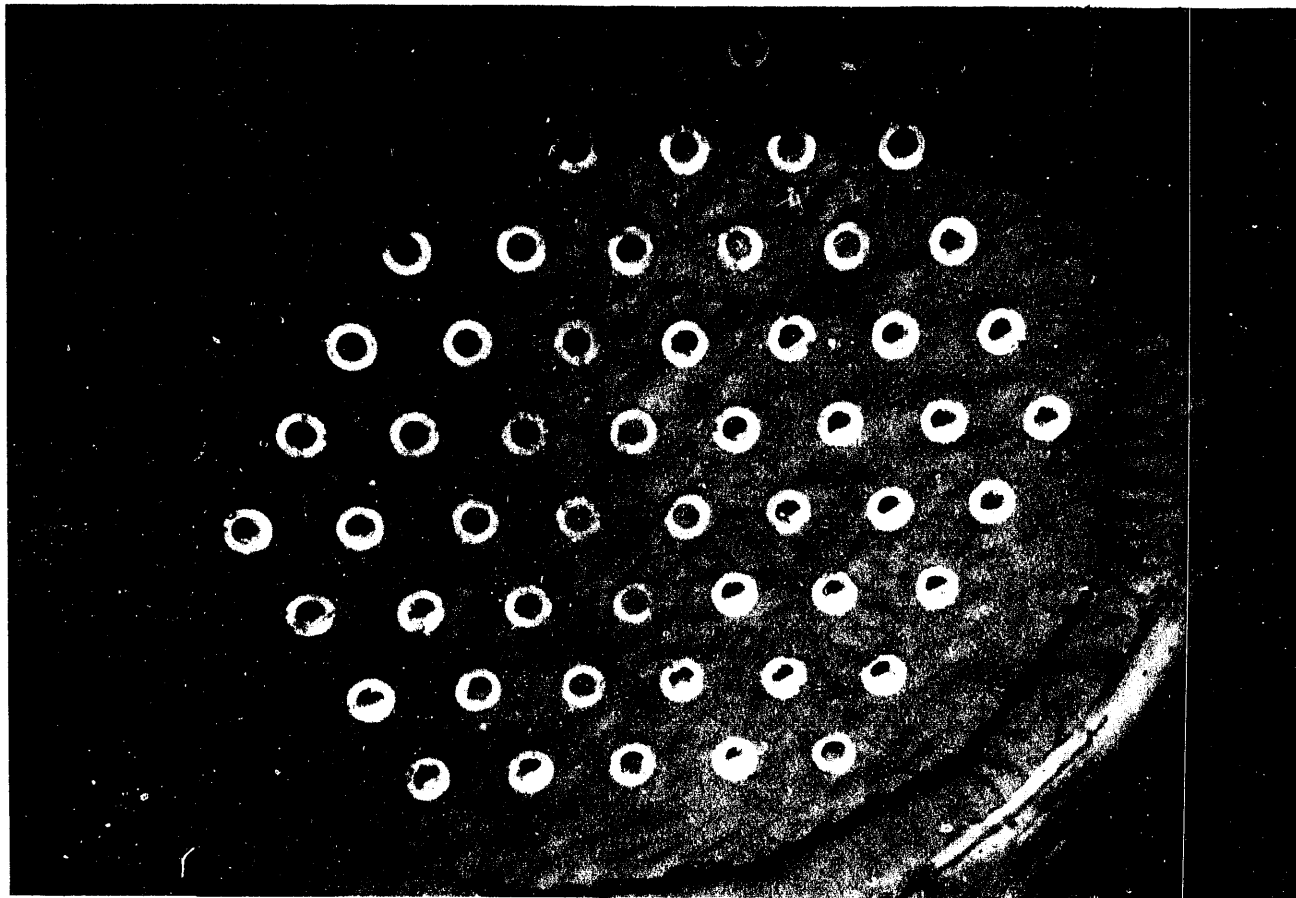
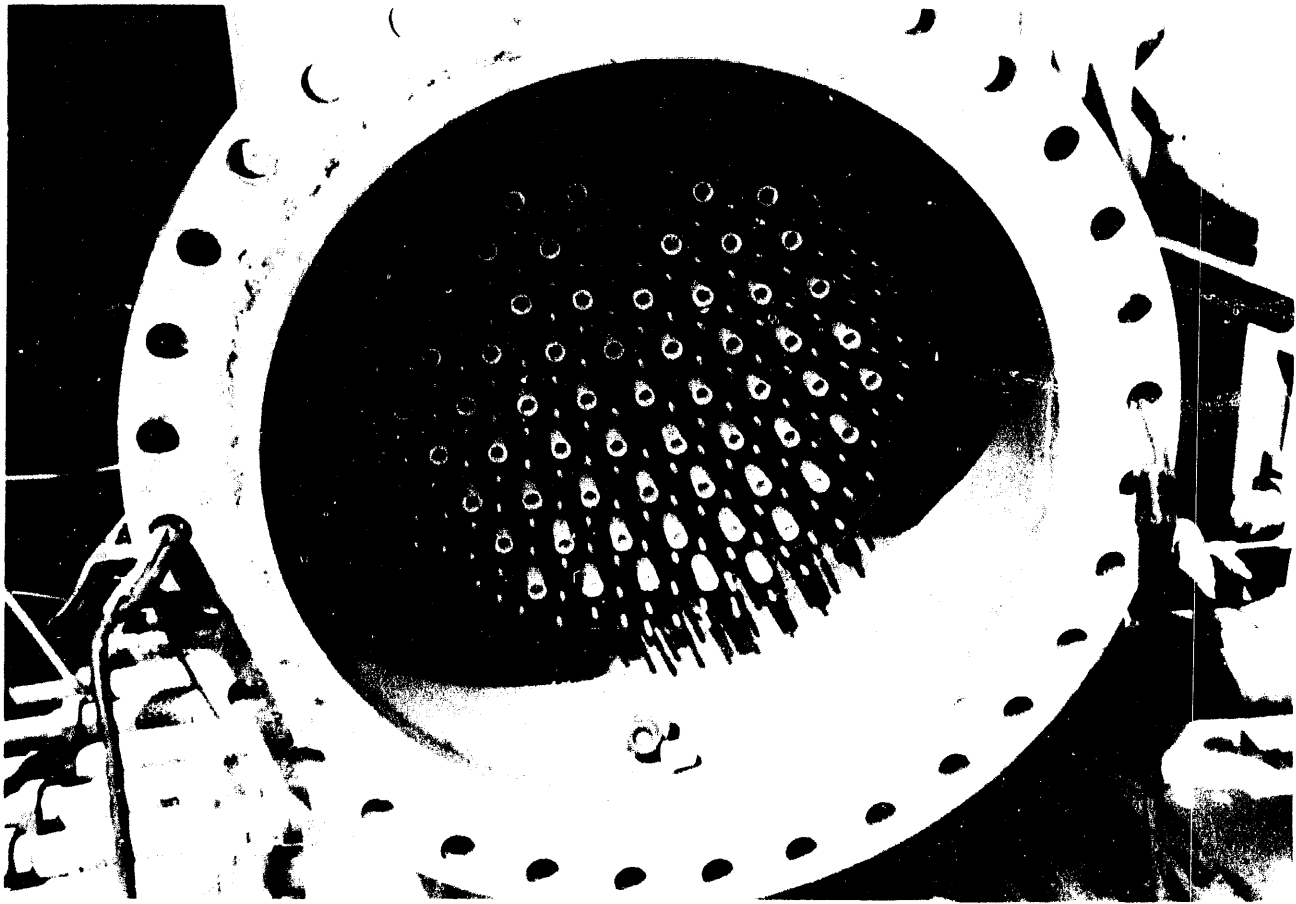




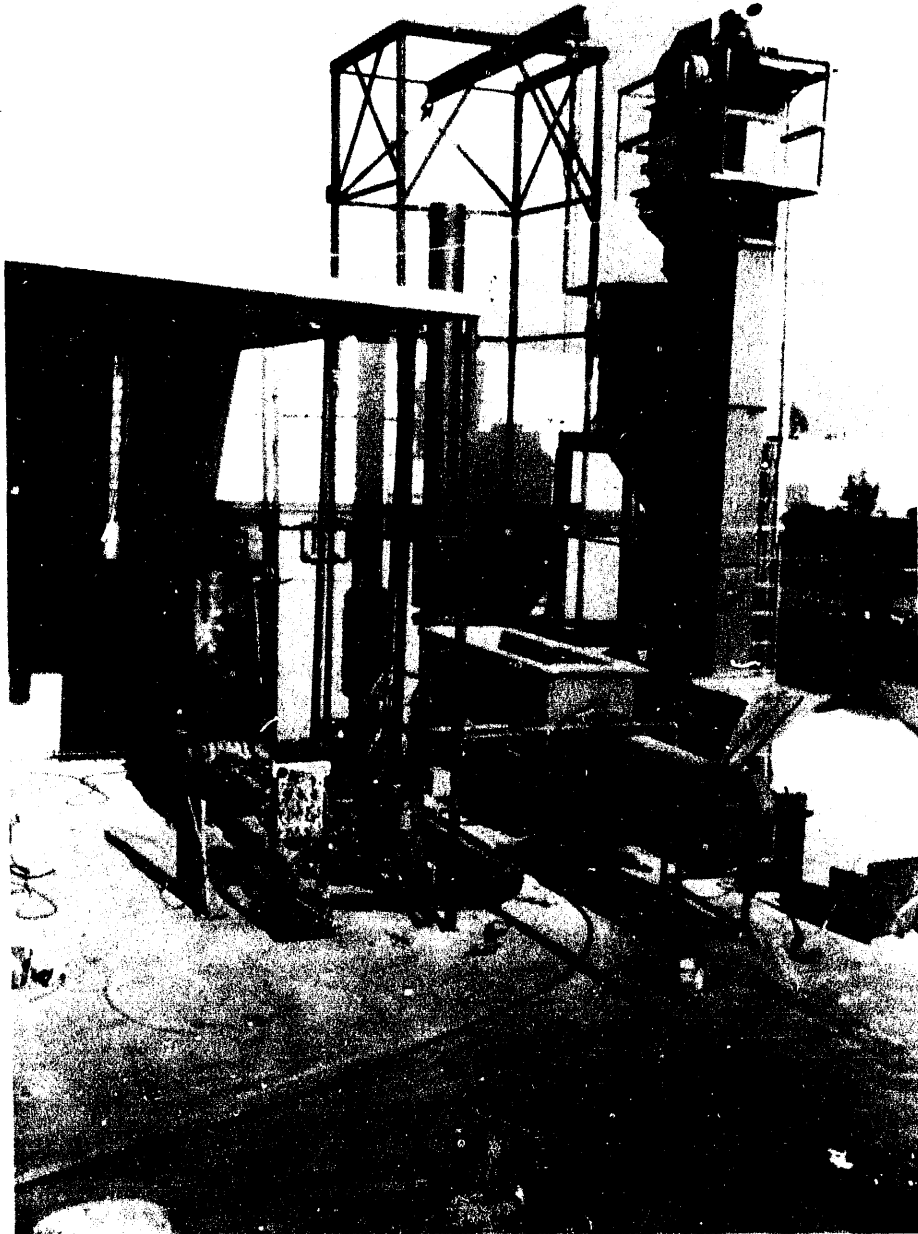
**FIGURE 7-8: METAL RADIATION SHIELD**



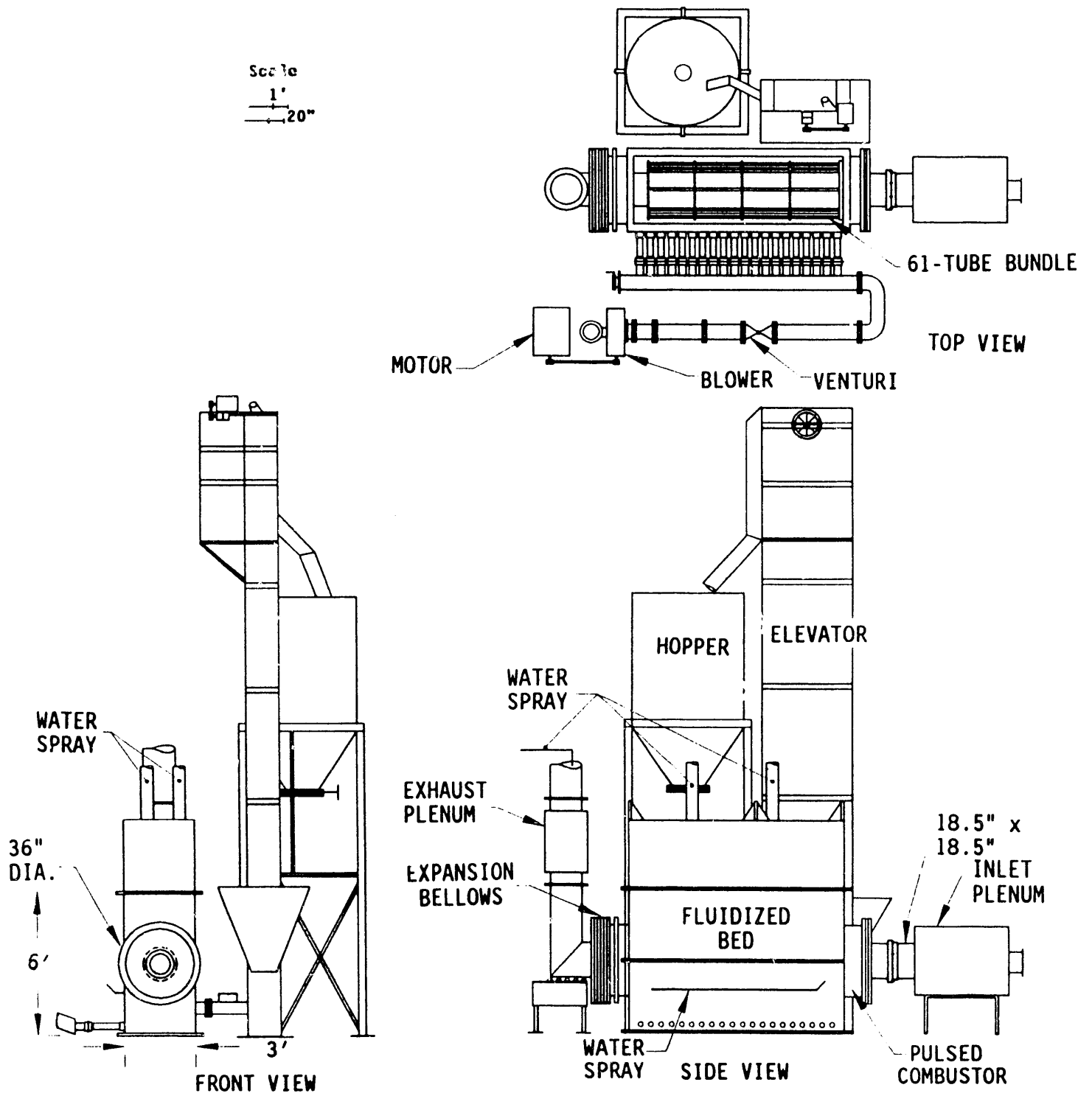
**FIGURE 7-9: REFRACTORY-LINED RADIATION SHIELD**



**FIGURE 7-10: INTEGRAL RADIATION SHIELD**



**FIGURE 7-11: HOT FLUIDIZATION TEST RIG**



**FIGURE 7-12: SCHEMATIC OF HOT TEST RIG**

### **7.3.1 FLUID BED**

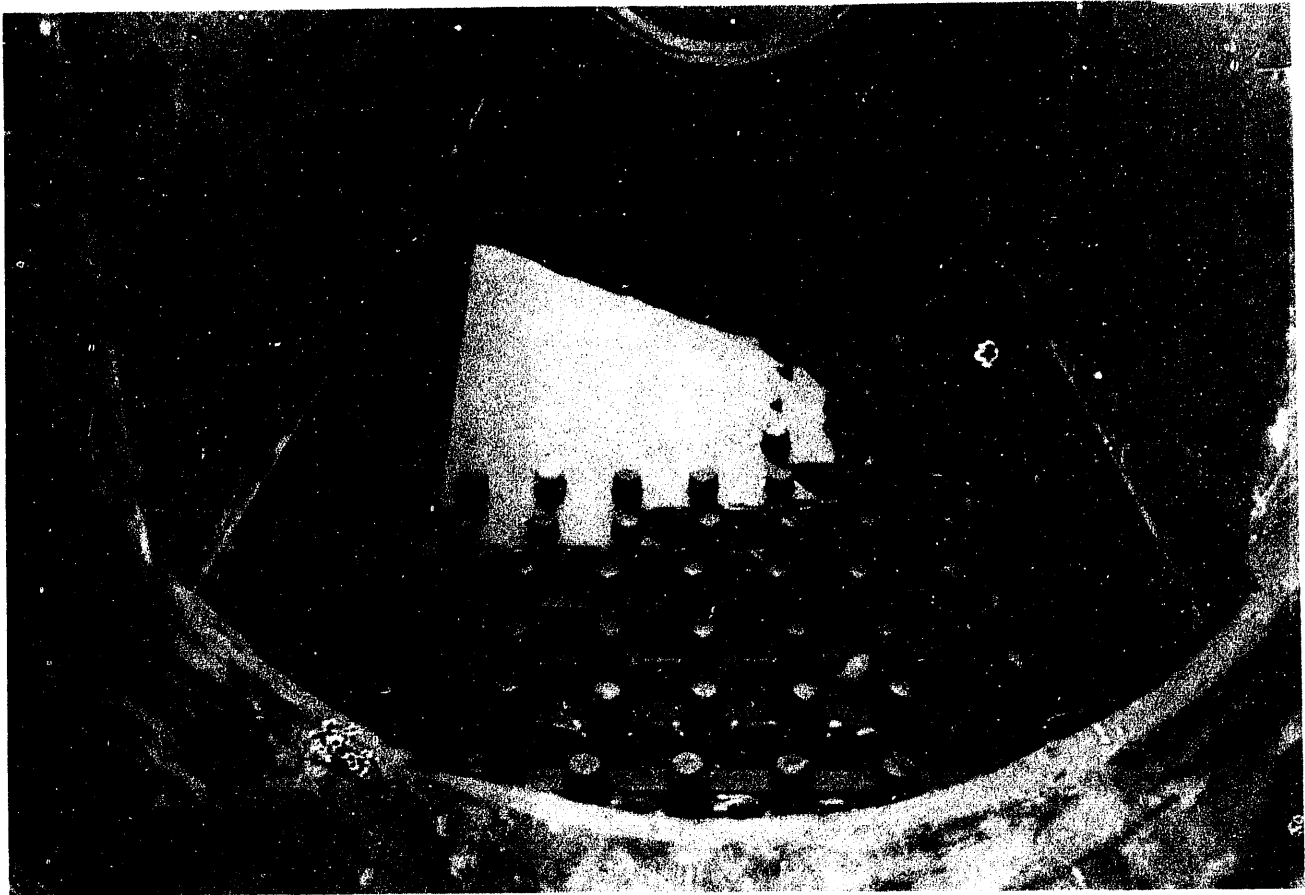
The fluid bed is constructed from a lower portion consisting of a carbon steel tank lined with Pyro Engineering, Inc. ThermoTek block insulation. The upper freeboard region of the fluid bed is formed from an uninsulated stainless steel tank.

The base of the fluid bed contains a series of sparge tubes to uniformly distribute the fluidization air. Each sparge tube contains seven risers which are terminated with a cap containing four nozzle holes. Air is supplied to the sparge tube distribution manifold using a Sutorbilt Model 721 rotary lobe blower capable of 3046 CFM at a maximum blower RPM of 2440. The blower prime mover consists of an eight-cylinder automotive engine capable of 125 HP at 3600 engine RPM. Figure 7-13 shows a photograph of the inside of the empty fluid bed as seen through the pulse combustor attachment nozzle. Figure 7-14 shows a view of the sparge distribution manifold with the bed being fluidized in a partially filled condition. A close-up of the fluidized bed is shown in Figure 7-15.

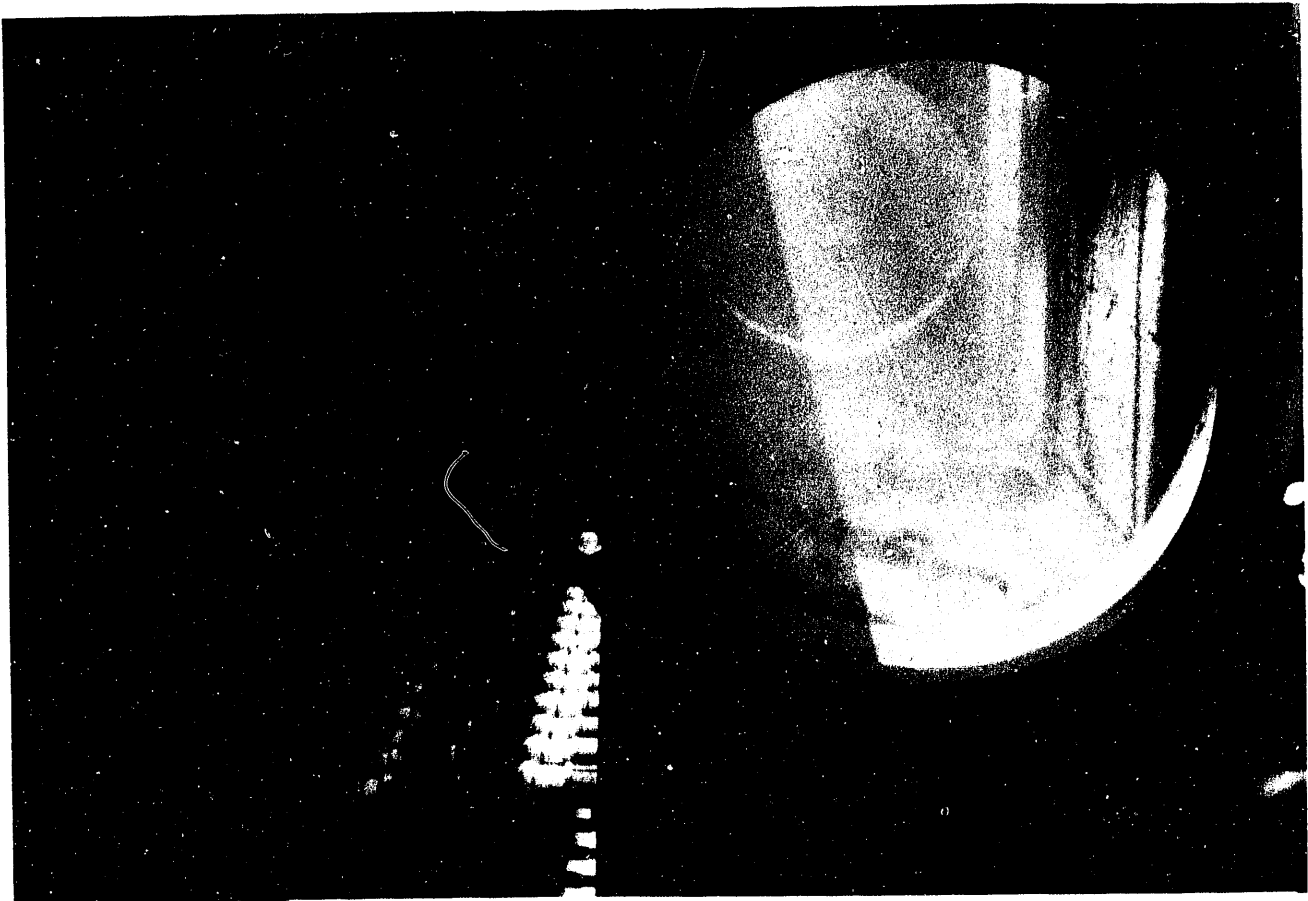
### **7.3.2 PULSE MODULE**

The pulse heater module is inserted through a 36-inch nozzle port on one side of the fluid bed. The tube sheet and pulse combustor is bolted to the fluid bed by means of a 150# flange. A side view of the inserted pulse module at the combustor end is shown in Figure 7-16. The exit plenum of the pulse heater module is attached to the opposite end of the fluid bed by means of a flanged expansion bellows shown in Figure 7-17. The expansion bellows is required to accommodate thermal expansion during hot operation. The travel of the bellows from cold start to full operating temperature is approximately 2 inches. The bellows are precompressed during installation so that the stress is relieved during expansion at full temperature. The bellows include an internal sleeve with a purge port to prevent interference of movement due to intruding bed particles.

The flue gases exiting the pulse module are vented through a muffled stack. The stack is supported on a roller assembly to allow free movement during bellows expansion.



**FIGURE 7-13: FLUID BED INTERNALS**

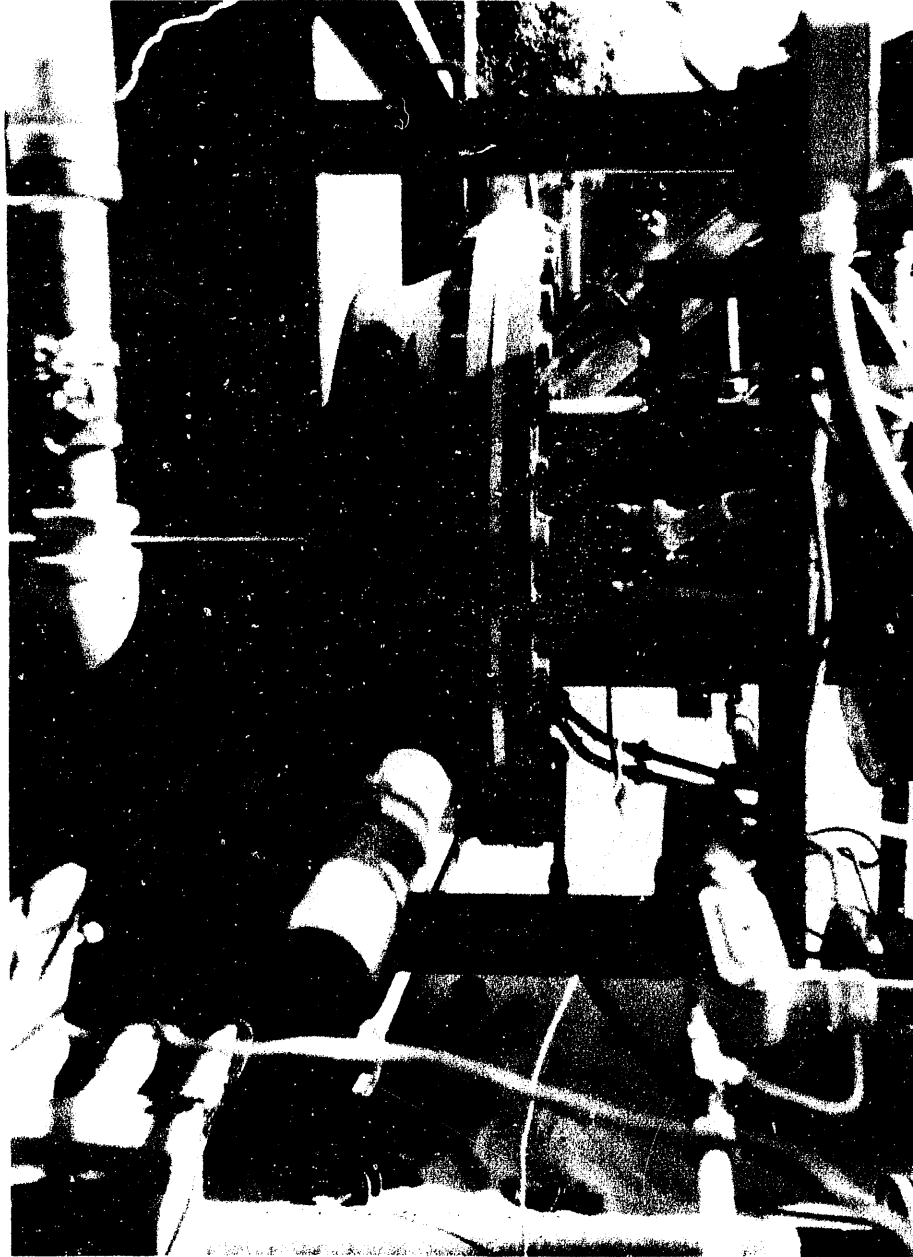


**FIGURE 7-14: SPARGE DISTRIBUTION MANIFOLD SYSTEM**

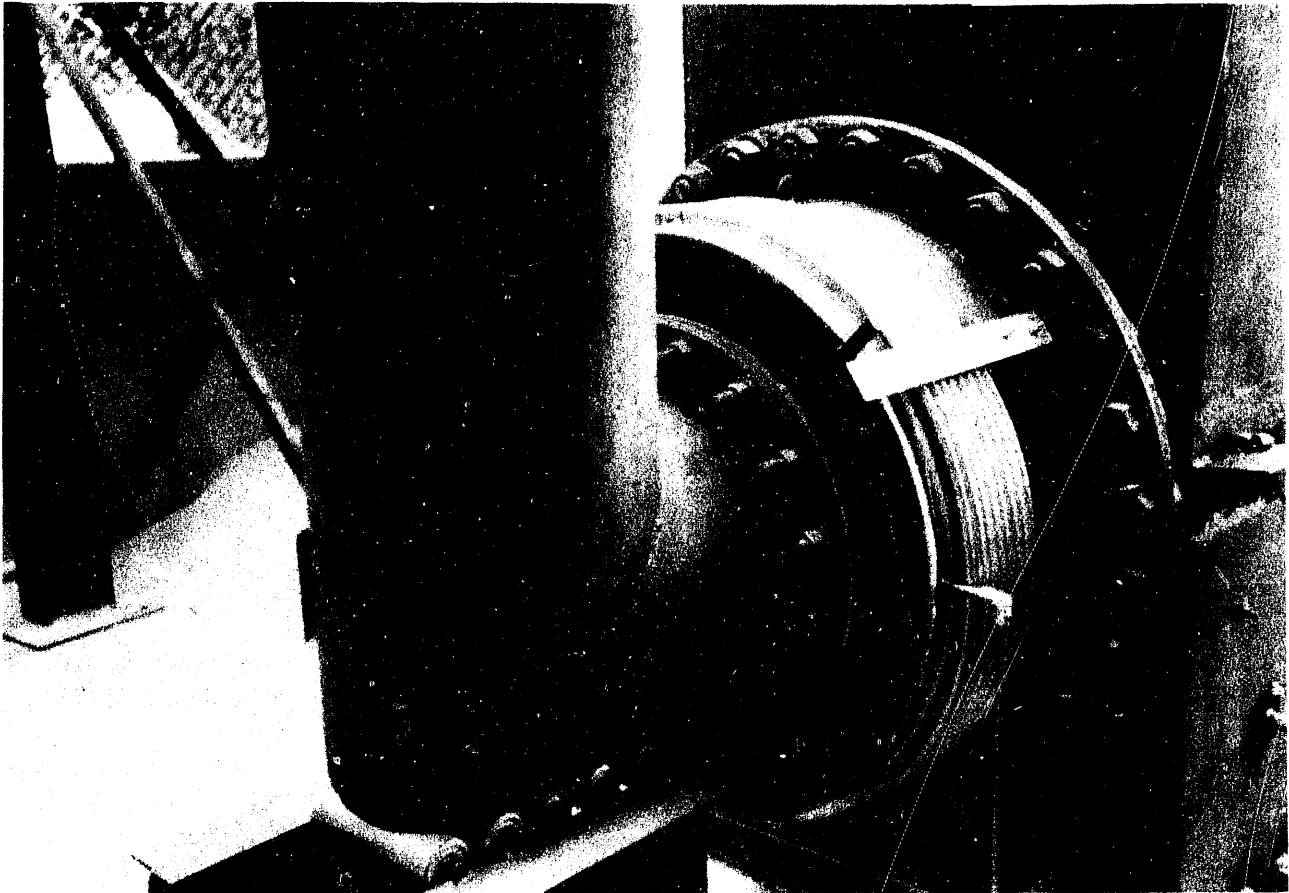




**FIGURE 7-15: BED UNDER FLUIDIZATION CONDITIONS**



**FIGURE 7-16: SIDE VIEW OF FULL-SCALE PULSE COMBUSTOR**



**FIGURE 7-17: PULSE MODULE EXPANSION JOINT**

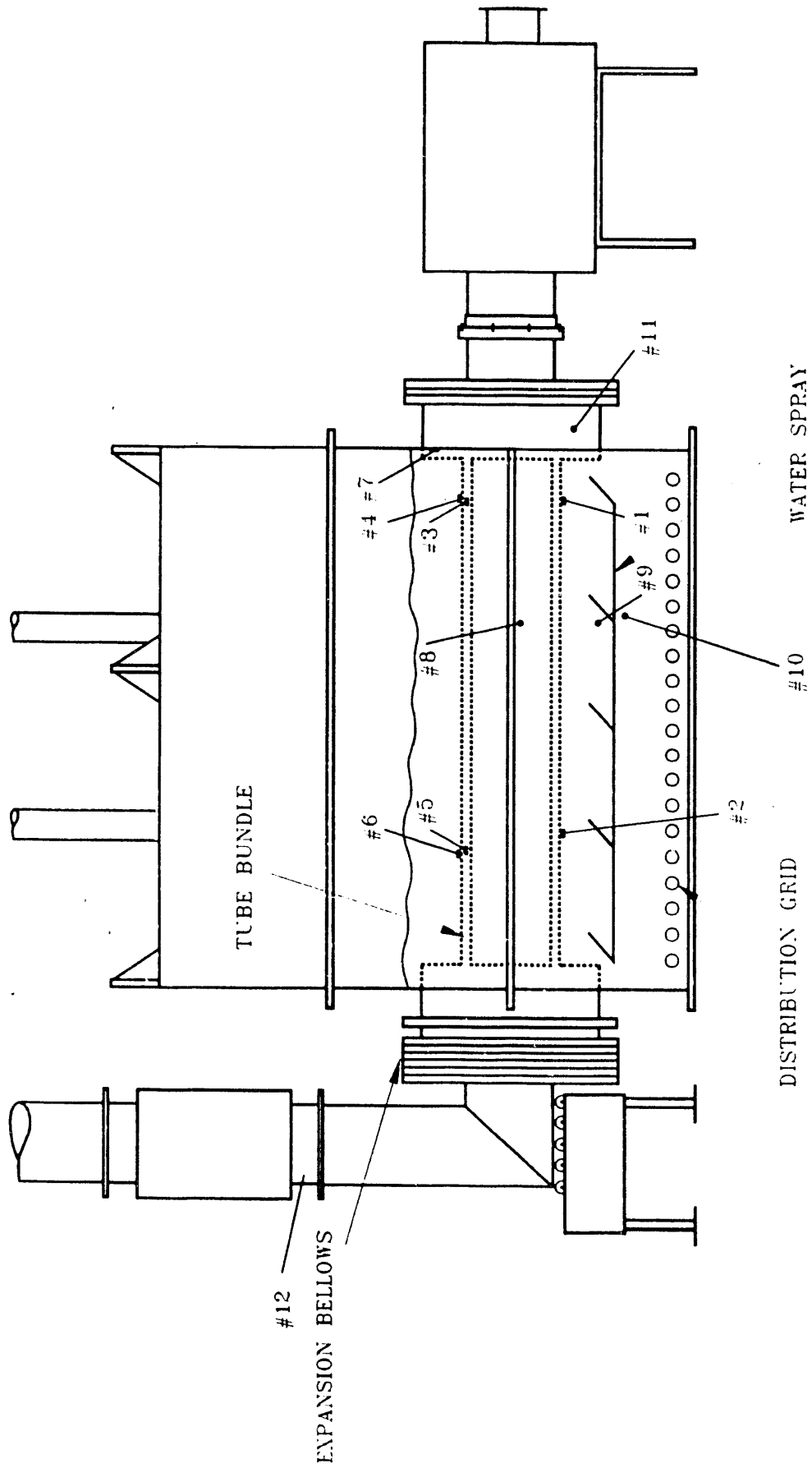
### **7.3.3 MONITORING INSTRUMENTATION**

A primary objective of the hot fluidization tests was to monitor and characterize the combustion and heat transfer performance of the module including measurement of temperature profiles along the pulse heater tube bundle. Type K ungrounded thermocouples were used to measure temperatures at 12 separate locations. A schematic of the temperature measurement locations are shown in Figure 7-18.

The pulse combustor chamber temperature was monitored to characterize combustor performance. Thermocouples were placed at several locations to monitor the fluid bed temperature. A uniform bed temperature is highly desirable since local variations in bed temperature could be indicative of insufficient solids circulation due to the presence of the tube bundle. Finally, tube skin thermocouples were welded to tube surfaces at several locations. This included thermocouple pads mounted at different circumferential orientations (leeward or top and side-mounted). This was done to monitor the potential for developing tube-skin hot spots due to the existence of de-fluidized dead zones.

Also, a Teledyne MAX 5 stack gas analyzer was furnished to monitor combustion efficiency. Parameters recorded included % O<sub>2</sub>, ppm CO, and % combustibles. A Horiba analyzer was used to measure NO<sub>x</sub> emissions. Combustor pulsation performance was monitored by means of a pressure transducer and oscilloscope to characterize pulsation frequency and intensity.

In the following section, results of hot fluidization tests on the prototype pulse heater module are discussed.



**FIGURE 7-18: THERMOCOUPLE LOCATION (61-TUBE TEST UNIT)**

#### **7.3.4 TEST RESULTS**

The prototype pulse heater module was operated over a range of firing conditions to assess its thermal and combustion performance. A summary of these tests is provided in Table 7-1.

As seen in Table 7-1, test firing rates ranged from  $2.8 \times 10^6$  Btu/hr up to  $5.1 \times 10^6$  Btu/hr. An orifice plate was placed in the flue gas stack to simulate the back pressure anticipated for actual operation with waste heat recovery equipment. The orifice plate maintained a back pressure of 4.0 inches water column at the lower firing range and about 7.5 inches water column at the higher firing range.

The air stoichiometry (excess air) was varied by adjusting air plenum pressure via a fan damper. Air plenum pressures were varied from 8 - 12 inches water column.

The test was operated at a nominal bed temperature of 850°F. This was the maximum safe long-term operating temperature of the hot test vessel. This temperature is sufficiently close to the gasifier design operating temperature (1150°F) to yield combustion and heat transfer data which can be accurately projected.

The tubeskin thermocouples, designated 1-6, are as depicted in Figure 7-18. As seen in Table 7-1, tubeskin temperatures never exceeded 200°F above the bed temperature. Since heat fluxes will diminish somewhat at higher bed temperatures, this maximum temperature difference will also be reduced slightly during actual gasifier operation. Note that MTCI's simulation models predict a maximum tubeskin temperature of about 150°F above the bed temperature. This should be sufficient to avoid bed agglomeration.

However, it was noted that the tubesheet temperature exceeded the bed temperature by over 400°F in some cases. The tubesheet was insulated with 2 inches of refractory. It is apparent that increased refractory thickness will be required to prevent overheating near the tubesheet surface.

**TABLE 7-1: COMBUSTION PERFORMANCE FOR PROTOTYPE PULSE HEATER**

TOTAL FIRING RATE (MMBtu/hr)	XSA (%)	PLENUM PRESSURE (INCHES WAT.COL.)	BACK PRESSURE (INCHES WAT.COL.)	BED (°F)	TUBESKIN (°F)						TUBE- SHEET TEMP. (°F)	FLUE TEMP. (°F)	O <sub>2</sub> (%)	COMB.'s (%)	(ppm @ 3% O <sub>2</sub> ) NO <sub>x</sub>	ppm CO
					1	2	3	4	5	6						
2.8	83	12.0	6.0	850	1115	925	986	994	940	930	1190	--	9.5	0.4	19	OFF
3.7	42	12.0	6.0	825	991	925	975	995	935	915	1290	--	6.2	0.2	21	OFF
4.6	19	12.0	7.5	870	1053	945	1024	1055	960	936	1300	1100	3.4	0.1	31	209
5.1	7	12.5	7.5	875	1065	954	1010	1030	963	940	1300	1138	1.3	0.4	NA	763
2.8	30	8.0	4.0	810	1003	910	951	958	955	942	1234	--	4.9	0.2	28	OFF
2.8	74	10.0	5.5	775	923	807	979	973	874	850	975	950	8.9	0.2	22	OFF
3.7	27	10.0	6.0	870	1070	990	1060	1055	1062	1010	1060	1170	4.5	0.1	16	OFF
4.6	2.4	10.0	7.5	960	1135	1020	1015	955	1035	1017	1272	1391	0.5	0.5	17	OFF
5.6	2.0	10.0	8.0	980	1150	1040	1110	1122	1055	1040	1304	1264	0.4	ND	35	202

The combustion performance is summarized in Figures 7-19 and 7-20. In Figure 7-19,  $\text{NO}_x$  (at 3%  $\text{O}_2$ ) is plotted versus excess air. As seen in the figure,  $\text{NO}_x$  emissions increase as excess air approaches zero as expected. This is due to the increased flame temperature and thermal  $\text{NO}_x$  production which results as excess air is reduced. Note that even the highest  $\text{NO}_x$  value of 31 ppm is considered quite good compared to conventional burners. Furthermore,  $\text{NO}_x$  values of 20 ppm appear achievable at reasonable excess air levels (20-40%). It should be noted that these tests were performed using natural gas fuel. When synthesis gas containing hydrogen is combusted, such as in actual gasifier operation, significantly lower  $\text{NO}_x$  emissions are expected.

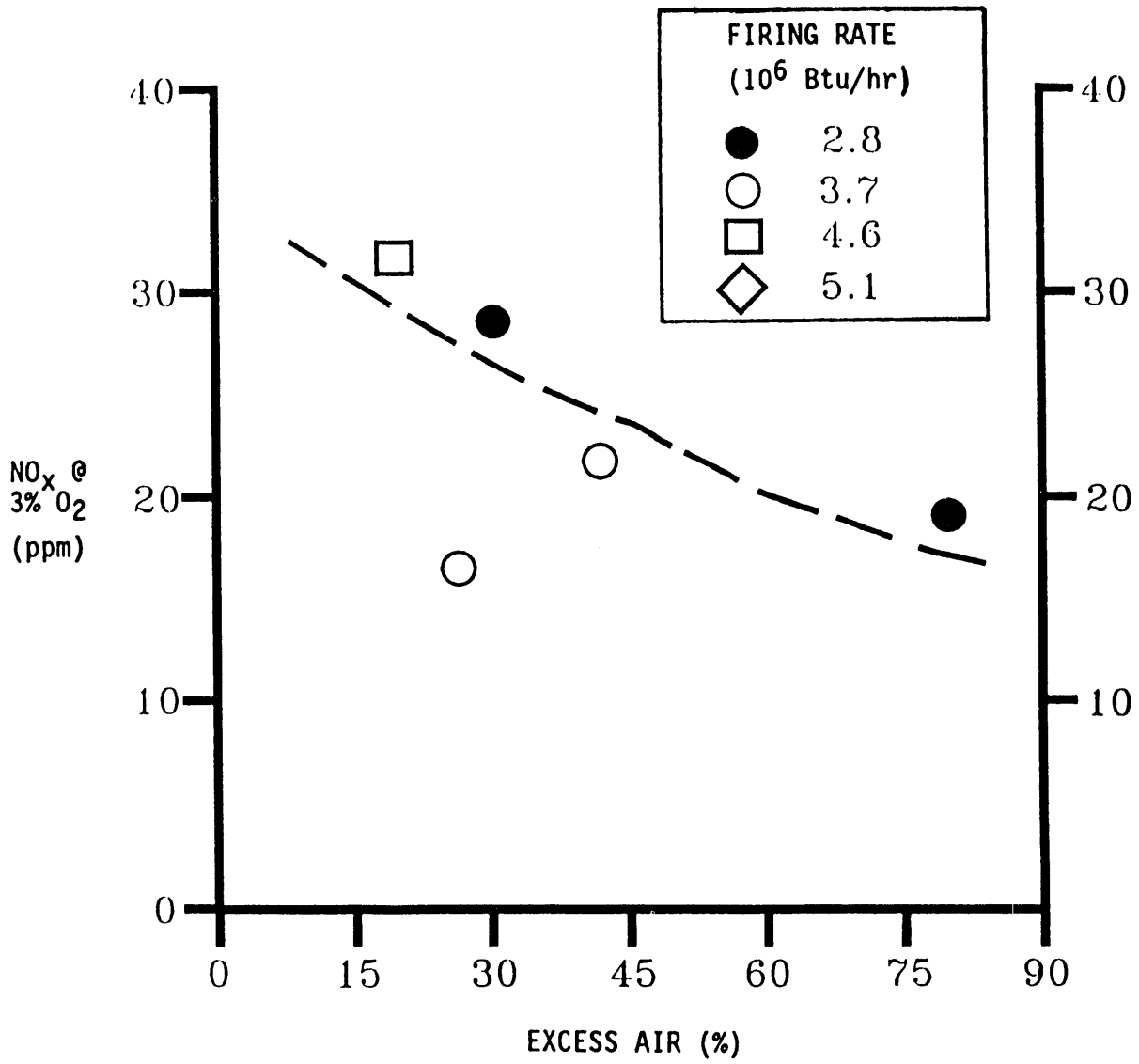
Furthermore,  $\text{NO}_x$  values of 20 ppm appear achievable at reasonable excess air levels (20-40%). It should be noted that these tests were performed using natural gas fuel. When synthesis gas containing hydrogen is combusted, such as in actual gasifier operation, significantly lower  $\text{NO}_x$  emissions are expected.

Figure 7-20 shows combustible emissions as a function of excess air. Note that the MAX-5 gas analyzer only resolves combustibles in increments of 0.1 percent. As seen in the figure, combustibles reach a minimum of excess air levels in the range of 20 to 45 percent. As excess air reduces below 10 percent, a significant increase in combustibles arises due to insufficient oxygen and reduced mixing intensity. As combustion air exceeds 60 percent, reduced flame temperature and combustor residence time probably contribute to the increased combustible emissions.

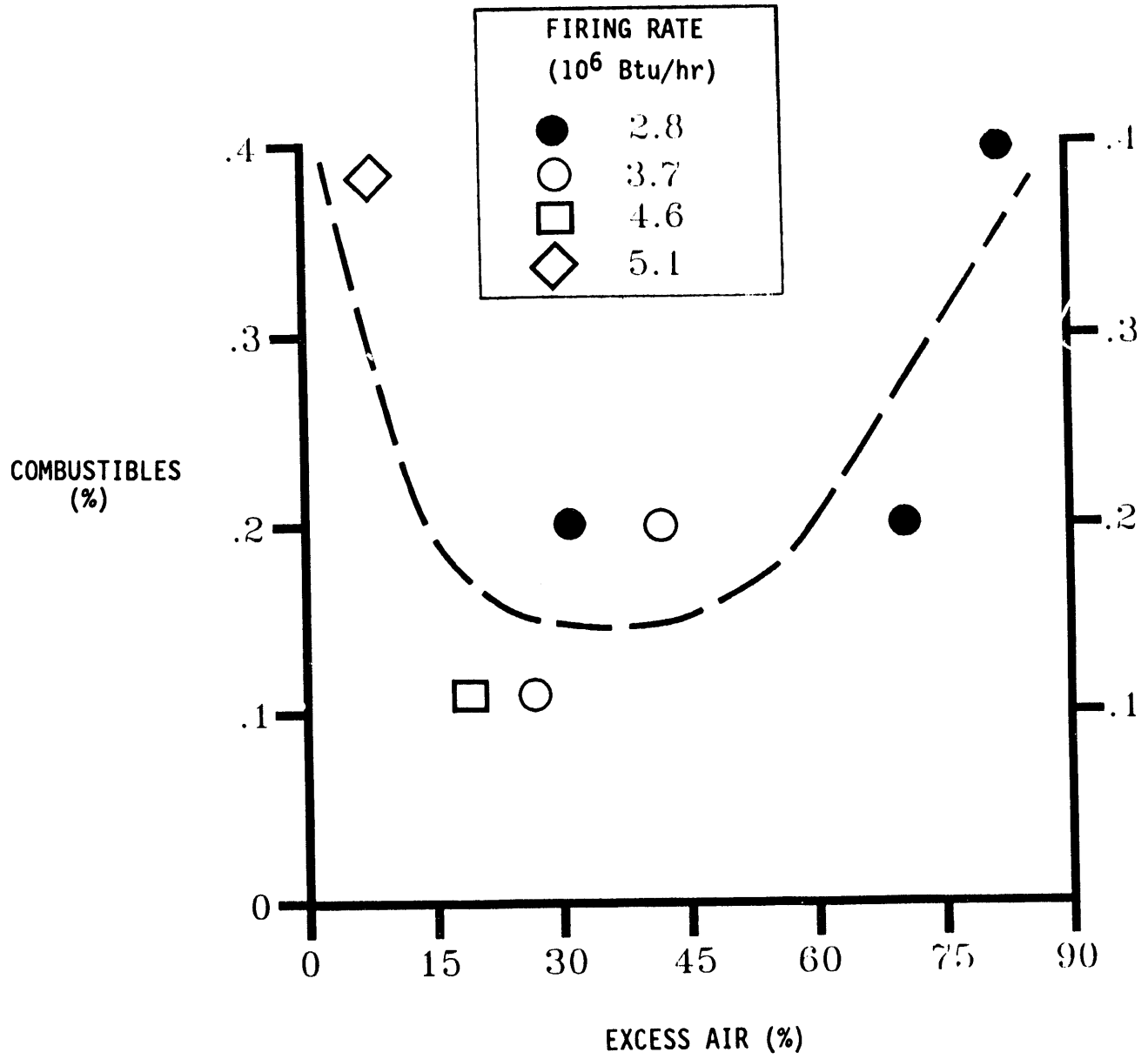
For the demonstration plant, a design excess air level of 20 percent is selected. This appears to offer conditions allowing good combustion performance and low  $\text{NO}_x$ .

Carbon monoxide emissions were recorded from 200 ppm to "off-scale." The maximum CO range for the MAX-5 was 1000 ppm. It should be noted that gas chromatograph analysis of the flue gas showed zero CO concentration and did not corroborate the relatively high CO readings by MAX-5. In any case, when combusting synthesis gas in the pulse heater operating at higher bed temperatures, CO emissions are not expected to pose a problem.





**FIGURE 7-19: NO<sub>x</sub> (AT 3% O<sub>2</sub>) vs. EXCESS AIR FOR PROTOTYPE PULSE HEATER**



**FIGURE 7-20: COMBUSTIBLES vs. EXCESS AIR FOR PROTOTYPE PULSE HEATER**

A long-term steady-state test was conducted to confirm the thermal balance for the pulse heater at a defined operating conditions and to compare these results with computer simulation model predictions.

The pulse heater was fired at approximately  $4.65 \times 10^6$  Btu/hr. The combustor was operated at 10 percent excess air and the fluid bed was maintained at 875°F. The fluid bed temperature was controlled by injecting water. The bed heat load includes the heat to vaporize the injected water and the sensible heat required to heat the fluidization air and the water vapor to the bed temperature.

The flue gas exit temperature was recorded as 1498°F. Based on this exit temperature, a calculated heat release to the bed of 2,613,000 Btu/hr was calculated. A heat balance for the test is shown in Table 7-2. As seen in the table, the heat load for heating the fluidization air and vaporizing the injected water (2,696,305 Btu/hr) closely matches the calculated absorbed duty (2,613,085 Btu/hr). The calculated heater thermal efficiency is 56.2 percent (HHV basis).

**TABLE 7-2: HOT FLUIDIZATION TEST HEAT BALANCE**

CONDITIONS:

FUEL FIRED:	4,650,000 Btu/hr
EXCESS AIR:	10%
FLUE TEMPERATURE:	1498
ABSORBED DUTY:	2,613,085 Btu/hr

HEAT LOAD:

FLUIDIZATION AIR:	807,681
VAPORIZED WATER:	<u>1,888,624</u>
TOTAL	<u>2,696,305 Btu/hr</u>

MTCI's pulse heater computer simulation model was run to compare predictions of heater efficiency with those calculated from experimental data. The model predicted a pulse heater efficiency of approximately 60 percent, which is reasonably close to the measured value of 56.2 percent. These tests

confirm the validity of the simulation model for design and performance predictions of the pulse heater.

## **7.4 CONTROL SYSTEMS DEVELOPMENT**

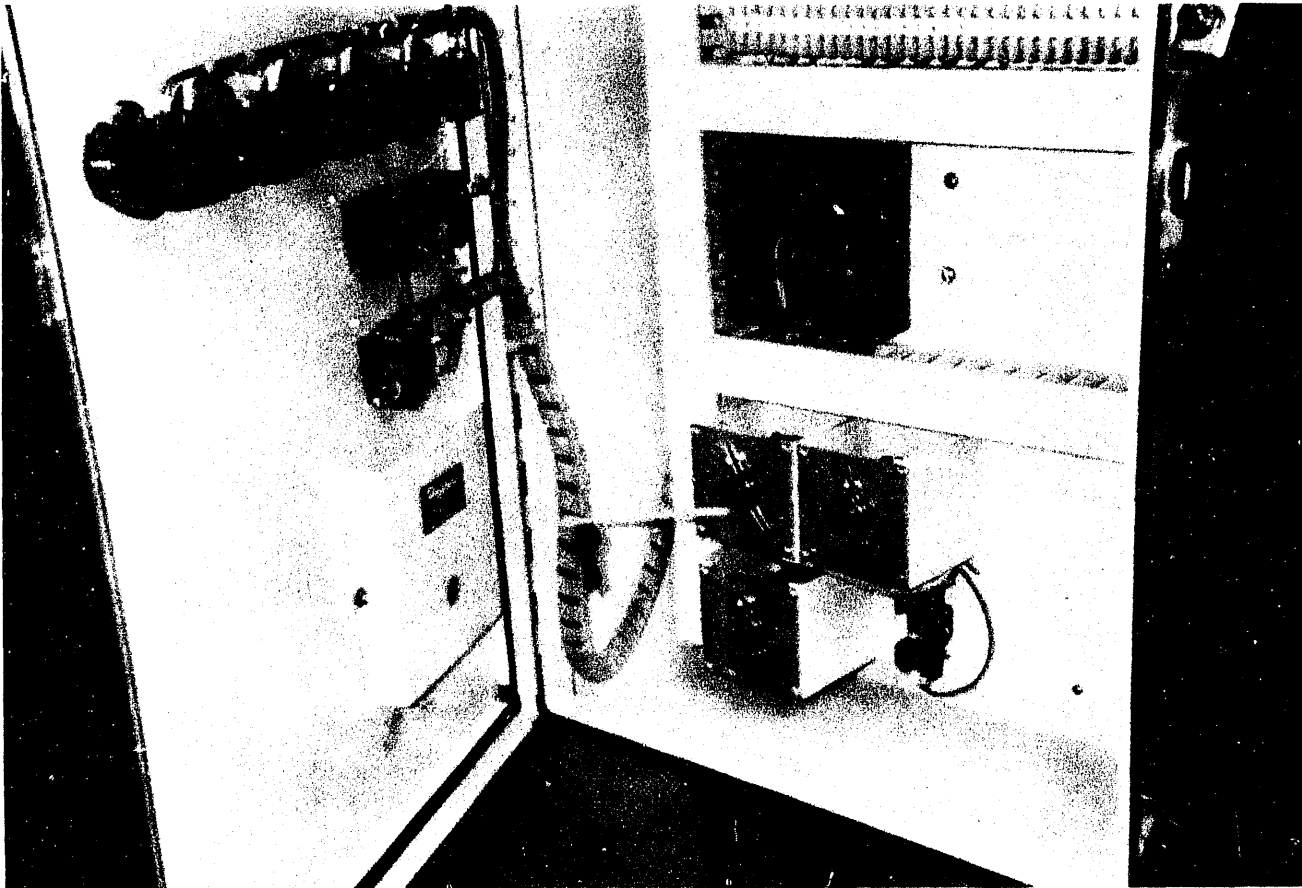
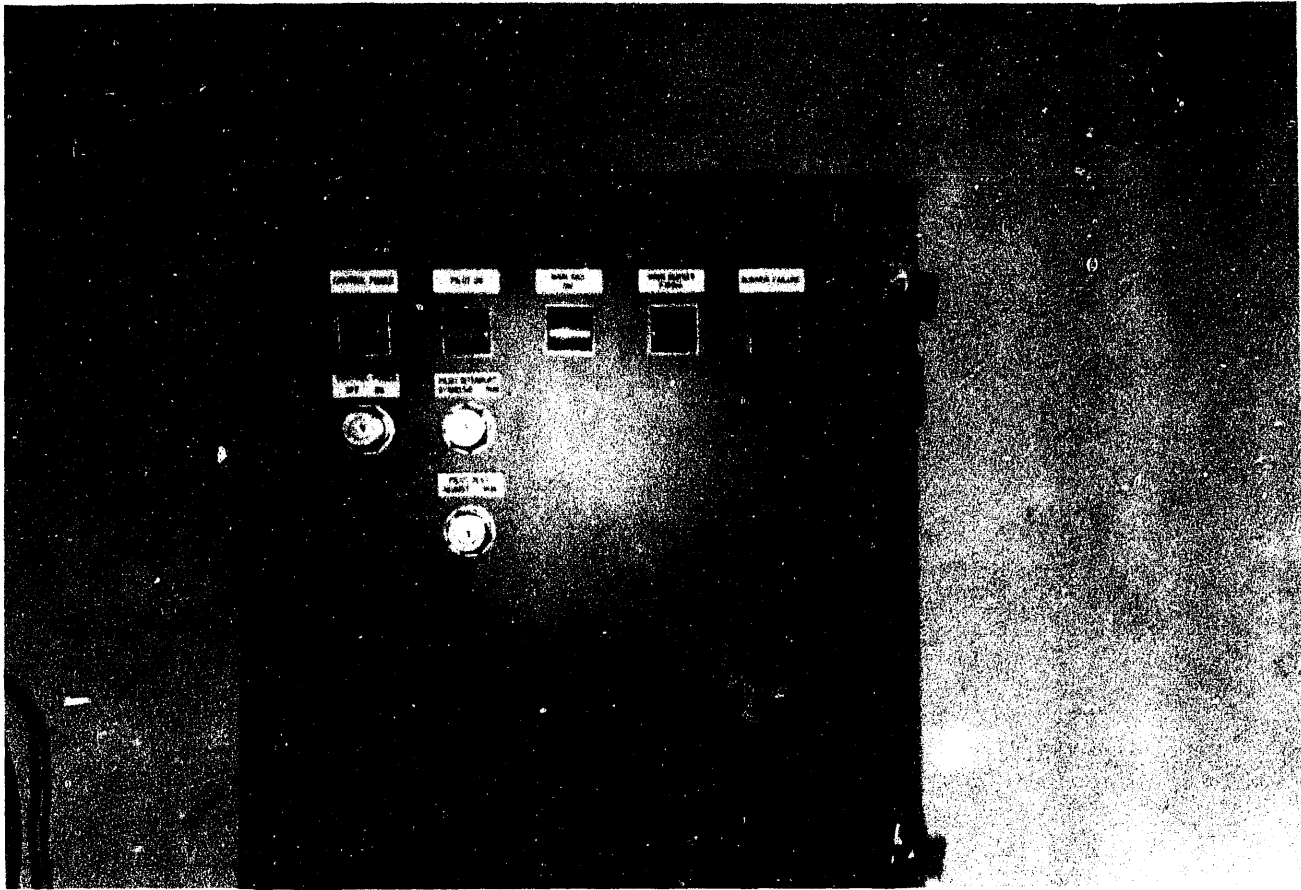
Bench-scale testing was performed using primarily manual control instrumentation. Manual control is essential during early stages of development since system response parameters are either unknown or ill-defined. However, manual control of the gasification plant requires the special attention of highly trained operators who are well-versed with the necessary control actions for each condition.

The MTCI indirect gasification plant represents a new technology for the commercial mill industry. While modern mills employ staff skilled in the operation of conventional recovery systems, they will be faced with a host of new operating and control protocol for the gasification plant. Therefore, to enhance acceptance of the gasification technology and to reduce the burden on mill operators, it was determined that the modular gasification plant will be furnished with a highly automated control system. The following section highlights some of the control systems development work undertaken toward this objective.

### **7.4.1 COMBUSTOR CONTROL AND SUPERVISION**

While supervision and control of combustor systems does not itself constitute a new art, special consideration must be given to the control of pulsating combustors. Therefore, in order to establish the reliable operation of a pulsating combustor in a manner compatible with the commercial environment, a burner supervision system was designed, fabricated and tested using the prototype combustor described in the prior sections.

The burner supervision system was housed in a weatherproof control panel as shown in Figure 7-21. The system was designed for maximum flexibility and included manually adjustable timer elements to allow variation in the time constants for the interlock permissives.



**FIGURE 7-21: PULSE COMBUSTOR BURNER CONTROL SYSTEM**

The supervisor control system incorporates the following logic capability:

- An air plenum pressure switch is monitored to verify operation of combustor fan. A minimum purge time is required before light-off sequence can be initiated.
- During initial light-off, a spark ignitor is energized along with the pilot air and fuel solenoid valves.
- An ultra-violet flame sensor verifies presence of pilot flame. If pilot flame is not verified after a predetermined time, the spark ignitor and pilot fuel and air solenoid valves are de-energized.
- If the pilot flame is proven, the main gas solenoid valve is energized. The pilot is then de-energized after a defined time delay. Flame failure actuates an immediate de-energization of the main fuel valve.
- A pulsation intensity sensor is incorporated as a secondary permissive. Loss of pulsation, constituting failure of the pulse module, also actuates a shut-down response.

Initial testing of the combustor supervision system revealed that the forced draft fan deflected the pilot flame out of the viewing range of the UV-flame sensor, thereby preventing proper light-off. This was overcome by modifying the respective orientations of the pilot flame and UV sensor. Final system adjustments are underway to ensure reliable and repeatable light-offs. Also, testing of the pulsation detection circuit is being initiated.

#### **7.4.2 TEMPERATURE CONTROL**

Automatic control of the gasifier operating temperature can be accomplished through adjustment of the pulse combustor firing rate or the black liquor feed rate. Adjustments in the combustor firing rate are reflected by a changing heat load to the gasifier. Conversely, adjustments in the black liquor feed rate are reflected by a changing cooling load to the gasifier. The gasifier temperature stabilizes when the heat load equals the cooling load at the specified condition.

Initial bench-scale testing relied on close operator supervision of the combustor firing rate and black liquor feed rate to maintain the desired operating temperature. However, to reduce the need for operator intervention, an automatic control logic is required.

For this reason, the black liquor feed pump was outfitted with an electric actuator supervised by an Omega temperature controller. Direct control of the cooling load was selected for several reasons. First, the commercial gasifier is anticipated to employ several independent pulse combustor modules. Therefore, if control was dependent on adjustment of the firing rate, then each pulse combustor fuel train must be integrated with a master controller. This would increase system complexity and reduce the flexibility to independently determine the combustor module firing pattern. Second, since the pulse combustor represents a new technology, supervised control of the combustor operating range is considered to be more essential than that of the liquor feed pump. Thus, by manual setting of the combustor firing range, only a single control loop is needed for automatic adjustment of the liquor feed pump output.

Tests on the bench-scale gasifier have indicated that stable operation can be achieved by suitable PID control of the cooling load via adjustment of the liquor pump output. However, it was noted that careful selection of the controller parameters is needed to prevent excessive cycling of the feed pump output during the "settling" period. These large variations can be detrimental to the maintenance of proper fluidization characteristics and can negatively impact the stable operation of downstream control elements.

### **7.4.3 AUTOMATED START-UP**

Strict control of bed parameters during start-up is essential to avoid potential operating difficulties including bed agglomeration, excessive elutriation, etc. In order to prevent mishaps during start-up through operator error, it was determined that the start-up control sequence should be fully automated.

The anticipated start-up control logic is highlighted here:

- If gasifier temperature is less than 400°F, then the start-up will be initiated by fluidization with preheated air.
- When gasifier temperature is greater than 400°F but less than 1000°F, then the preheated fluidization air will be replaced with steam, followed by automatic ignition of pulse heaters. The steam flow rate will be metered in a manner conforming to a control schedule based on gasifier temperature. As the gasifier temperature increases, the steam flow rate will be reduced to maintain a constant fluidization velocity of 1.5 ft/sec at all times.
- When gasifier temperature exceeds 1000°F, the black liquor feed pump will be actuated; its output being dictated by a PID temperature controller.

An assessment is being made to determine the feasibility of testing start-up control logic using the bench-scale gasifier.



**SECTION 8.0**  
**ENGINEERING DESIGN OF FIELD TEST UNIT**

**8.1 INTRODUCTION**

The following paragraphs summarize the current status of the Engineering Design for the Black Liquor Gasification Field Test Unit. The engineering design has been consolidated into a single Bid Package separated into three volumes: 1) Commercial, 2) Technical, and 3) Plans and Drawings. The purpose of the Bid Package is to form the Commercial and Technical basis for quotation on a turnkey Black Liquor Gasification Field Test Pilot Plant to be located at a Host Paper Mill. The Bid Package covers specifications and requirements for detailed engineering, procurement, fabrication, assembly, transportation, field erection, precommissioning and permitting as pertaining to the recovery plant. A summary of key elements of the Engineering Design is provided here.

**8.2 MATERIAL BALANCES**

Computer simulations of material and energy balances have been prepared for black liquor solid feedstocks. The field test unit is being designed to handle two extreme operating conditions, as follows:

- 1 ton/hr Black Liquor Solids at 33% solids by weight, and
- 2 ton/hr Black Liquor Solids at 65% solids by weight.

A summary of material balances for each case is given in Tables 8-1 and 8-2. The black liquor solids feedstock analysis for the material balance contains by dry weight percent (C=38.3%, H=4.1%, O=32.6%, N=1.0%, S=4.4%, Na=19.1%, Cl=0.5%).

At the 65 percent solids level, the products from gasification of black liquor solids include chemical recovery of pulping chemicals in the form of green liquor (11323 lb/hr), a clean medium Btu fuel gas (293 Btu/SCF) rich in hydrogen, export steam from waste heat recovery (1134 lb/hr), and insoluble carbon filter dregs (53.5 lb/hr).

**TABLE 8-1: MATERIAL BALANCE  
LIQUOR SOLIDS**

COMPONENT	1	2	3	4	5	6	FU : P:
	BLACK LIQUOR FEEDSTOCK	SUPERHEATED STEAM &/or GAS RECYCLE	PRODUCT GAS TO E-3	PRODUCT SALTS TO V-3	PRODUCT GAS TO X-1	PRODUCT GAS TO C-1	
lb/Hr----->							
O2	0.00	0.00	0.00	0.00	0.00	0.00	0.00
N2	0.00	0.00	0.00	0.00	0.00	0.00	0.00
CH4	0.00	0.00	54.12	0.00	54.12	54.12	54.12
C2H6	0.00	0.00	5.41	0.00	5.41	5.41	5.41
C2H4	0.00	0.00	9.47	0.00	9.47	9.47	9.47
C3H8	0.00	0.00	3.97	0.00	3.97	3.97	3.97
C3H6	0.00	0.00	5.68	0.00	5.68	5.68	5.68
C4	0.00	0.00	5.23	0.00	5.23	5.23	5.23
C5	0.00	0.00	4.87	0.00	4.87	4.87	4.87
C6	0.00	0.00	3.88	0.00	3.88	3.88	3.88
H2	0.00	0.00	181.42	0.00	181.42	181.42	181.42
H2S	0.00	0.00	92.86	0.00	92.86	92.86	92.86
NH3	0.00	0.00	24.71	0.00	24.71	24.71	24.71
CO	0.00	0.00	378.85	0.00	378.85	378.85	378.85
CO2	0.00	0.00	1488.33	0.00	1488.33	1488.33	1488.33
H2O(V)	0.00	2000.00	4892.49	0.00	4892.49	272.13	272.13
H2O(L)	0.00	0.00	0.00	0.00	0.00	0.00	0.00
CARBON	0.00	0.00	0.00	26.73	0.00	0.00	0.00
TOTAL SODIUM	0.00	0.00	0.00	381.99	0.00	0.00	0.00
TOTAL SULFUR	0.00	0.00	0.00	0.00	0.00	0.00	0.00
TOTAL CARBONATE	0.00	0.00	0.00	489.93	0.00	0.00	0.00
TOTAL CHLORIDE	0.00	0.00	0.00	10.16	0.00	0.00	0.00
BLACK LIQUOR	6060.61	0.00	0.00	0.00	0.00	0.00	0.00
<b>TOTAL (lb/Hr)</b>	<b>6060.61</b>	<b>2000.00</b>	<b>7151.30</b>	<b>908.81</b>	<b>7151.30</b>	<b>2530.94</b>	
TEMPERATURE (F)	200.00	800.00	1150.00	1150.00	500.00	125.00	
PRESSURE (PSIG)	30.00	30.00	10.00	10.00	8.00	6.00	
HHV (MMBTU/Hr)	13.00	0.00	15.75	0.38	15.75	15.75	
ENTHALPY (MMBTU/Hr)	13.52	2.79	24.75	0.65	21.61	16.07	

**SUMMARY FOR 1 TON/HR BLACK  
AT 33% SOLIDS BY WEIGHT**

7	8	9	10	11	12	13	14
FLUE GAS EXPORT PRODUCT	FUEL GAS TO H-1	COMBUSTION AIR TO H-1	FLUE GAS TO E-1	FLUE GAS TO E-2	FLUE GAS TO STACK	MAKE-UP WATER TO V-3	SCRUBBER WATER TO V-3
0.00	0.00	2410.01	401.67	401.67	401.67	0.00	0.00
0.00	0.00	7935.78	7935.78	7935.78	7935.78	0.00	0.00
0.00	54.12	0.00	0.00	0.00	0.00	0.00	0.00
0.00	5.41	0.00	0.00	0.00	0.00	0.00	0.00
0.00	9.47	0.00	0.00	0.00	0.00	0.00	0.00
0.00	3.97	0.00	0.00	0.00	0.00	0.00	0.00
0.00	5.68	0.00	0.00	0.00	0.00	0.00	0.00
0.00	5.23	0.00	0.00	0.00	0.00	0.00	0.00
0.00	4.87	0.00	0.00	0.00	0.00	0.00	0.00
0.00	3.88	0.00	0.00	0.00	0.00	0.00	0.00
0.00	181.42	0.00	0.00	0.00	0.00	0.00	0.00
0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
0.00	378.85	0.00	0.00	0.00	0.00	0.00	0.00
0.00	1488.33	0.00	2347.92	2347.92	2347.92	0.00	0.00
0.00	178.39	0.00	1978.43	1978.43	1978.43	0.00	0.00
0.00	0.00	0.00	0.00	0.00	0.00	99.10	4719.46
0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
0.00	2319.63	10345.79	12663.80	12663.80	12663.80	99.10	4719.46
110.00	110.00	77.00	1350.00	1229.66	300.00	77.00	119.10
5.00	5.00	0.40	0.20	0.10	0.00	40.00	40.00
0.00	14.85	0.00	0.00	0.00	0.00	0.00	0.00
0.00	15.05	0.00	6.87	6.39	2.86	0.00	0.20

**TABLE 8-1: MATERIAL BALANCE  
LIQUOR**

	15	16	17	18	19	20
COMPONENT	ALK. SOLUTION TO C-1	FILTER DREGS	GREEN LIQUOR PRODUCT	BOILER FEED WATER	BFW TO E-2	BFW TO E-3
lb/Hr----->						
O2	0.00	0.00	0.00	0.00	0.00	0.00
N2	0.00	0.00	0.00	0.00	0.00	0.00
CH4	0.00	0.00	0.00	0.00	0.00	0.00
C2H6	0.00	0.00	0.00	0.00	0.00	0.00
C2H4	0.00	0.00	0.00	0.00	0.00	0.00
C3H8	0.00	0.00	0.00	0.00	0.00	0.00
C3H6	0.00	0.00	0.00	0.00	0.00	0.00
C4	0.00	0.00	0.00	0.00	0.00	0.00
C5	0.00	0.00	0.00	0.00	0.00	0.00
C6	0.00	0.00	0.00	0.00	0.00	0.00
H2	0.00	0.00	0.00	0.00	0.00	0.00
H2S	0.00	0.00	0.00	0.00	0.00	0.00
NH3	0.00	0.00	24.71	0.00	0.00	0.00
CO	0.00	0.00	0.00	0.00	0.00	0.00
CO2	0.00	0.00	0.00	0.00	0.00	0.00
H2O(V)	0.00	0.00	0.00	0.00	0.00	0.00
H2O(L)	4719.46	0.00	4813.20	5780.73	3061.39	2719.2
CARBON	0.00	26.73	0.00	0.00	0.00	0.00
TOTAL SODIUM	381.99	0.00	381.99	0.00	0.00	0.00
TOTAL SULFUR	0.00	0.00	87.38	0.00	0.00	0.00
TOTAL CARBONATE	489.93	0.00	489.93	0.00	0.00	0.00
TOTAL CHLORIDE	10.16	0.00	10.16	0.00	0.00	0.00
BLACK LIQUOR	0.00	0.00	0.00	0.00	0.00	0.00
TOTAL (lb/Hr)	5601.54	26.73	5807.37	5780.73	3061.39	2719.2
TEMPERATURE (F)	100.00	100.00	120.00	77.00	77.00	77.00
PRESSURE (PSIG)	20.00	0.00	5.00	60.00	60.00	60.00
HHV (MMBTU/Hr)	0.00	0.38	0.24	0.00	0.00	0.00
ENTHALPY (MMBTU/Hr)	0.12	0.38	0.46	0.00	0.00	0.00



**TABLE 8-2: MATERIAL BALANCE  
LIQUOR SOLIDS**

COMPONENT	1	2	3	4	5	6	FI P
	BLACK LIQUOR FEEDSTOCK	SUPERHEATED STEAM &/or GAS RECYCLE	PRODUCT GAS TO E-3	PRODUCT SALTS TO V-3	PRODUCT GAS TO X-1	PRODUCT GAS TO C-1	
lb/Hr---->							
O2	0.00	0.00	0.00	0.00	0.00	0.00	
N2	0.00	0.00	0.00	0.00	0.00	0.00	
CH4	0.00	0.00	108.24	0.00	108.24	108.24	
C2H6	0.00	0.00	10.82	0.00	10.82	10.82	
C2H4	0.00	0.00	18.94	0.00	18.94	18.94	
C3H8	0.00	0.00	7.94	0.00	7.94	7.94	
C3H6	0.00	0.00	11.37	0.00	11.37	11.37	
C4	0.00	0.00	10.46	0.00	10.46	10.46	
C5	0.00	0.00	9.74	0.00	9.74	9.74	
C6	0.00	0.00	7.76	0.00	7.76	7.76	
H2	0.00	0.00	362.85	0.00	362.85	362.85	
H2S	0.00	0.00	185.72	0.00	185.72	185.72	
NH3	0.00	0.00	49.41	0.00	49.41	49.41	
CO	0.00	0.00	757.70	0.00	757.70	757.70	
CO2	0.00	0.00	2976.66	0.00	2976.66	2976.66	
H2O(V)	0.00	4000.00	3817.62	0.00	3817.62	544.26	
H2O(L)	0.00	0.00	0.00	0.00	0.00	0.00	
CARBON	0.00	0.00	0.00	53.46	0.00	0.00	
TOTAL SODIUM	0.00	0.00	0.00	763.98	0.00	0.00	
TOTAL SULFUR	0.00	0.00	0.00	0.00	0.00	0.00	
TOTAL CARBONATE	0.00	0.00	0.00	979.87	0.00	0.00	
TOTAL CHLORIDE	0.00	0.00	0.00	20.32	0.00	0.00	
BLACK LIQUOR	6153.85	0.00	0.00	0.00	0.00	0.00	
TOTAL (lb/Hr)	6153.85	4000.00	8335.23	1817.63	8335.23	5061.87	
TEMPERATURE (F)	200.00	800.00	1150.00	1150.00	500.00	125.00	
PRESSURE (PSIG)	30.00	30.00	10.00	10.00	8.00	6.00	
HHV (MMBTU/Hr)	26.00	0.00	31.50	0.75	31.50	31.50	
ENTHALPY (MMBTU/Hr)	26.53	5.57	40.11	1.31	36.35	32.14	

**SUMMARY FOR 2 TON/HR BLACK  
AT 65% SOLIDS BY WEIGHT**

7	8	9	10	11	12	13	14
EL GAS EXPORT PRODUCT	FUEL GAS TO H-1	COMBUSTION AIR TO H-1	FLUE GAS TO E-1	FLUE GAS TO E-2	FLUE GAS TO STACK	MAKE-UP WATER TO V-3	SCRUBBER WATER TO V-3
0.00	0.00	2736.19	456.03	456.03	456.03	0.00	0.00
0.00	0.00	9009.85	9009.85	9009.85	9009.85	0.00	0.00
46.80	61.45	0.00	0.00	0.00	0.00	0.00	0.00
4.68	6.14	0.00	0.00	0.00	0.00	0.00	0.00
8.19	10.75	0.00	0.00	0.00	0.00	0.00	0.00
3.43	4.51	0.00	0.00	0.00	0.00	0.00	0.00
4.91	6.45	0.00	0.00	0.00	0.00	0.00	0.00
4.52	5.94	0.00	0.00	0.00	0.00	0.00	0.00
4.21	5.53	0.00	0.00	0.00	0.00	0.00	0.00
3.35	4.40	0.00	0.00	0.00	0.00	0.00	0.00
156.87	205.98	0.00	0.00	0.00	0.00	0.00	0.00
0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
327.57	430.12	0.00	0.00	0.00	0.00	0.00	0.00
1286.89	1689.77	0.00	2665.70	2665.70	2665.70	0.00	0.00
154.25	202.54	0.00	2246.20	2246.20	2246.20	0.00	0.00
0.00	0.00	0.00	0.00	0.00	0.00	6165.56	9438.92
0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
2005.68	2633.58	11746.04	14377.78	14377.78	14377.78	6165.56	9438.92
110.00	110.00	77.00	1350.00	1137.16	300.00	77.00	91.91
5.00	5.00	0.40	0.20	0.10	0.00	40.00	40.00
12.84	16.86	0.00	0.00	0.00	0.00	0.00	0.00
13.01	17.09	0.00	7.80	6.84	3.25	0.00	0.14

TABLE 8-2: MATERIAL BALANCE  
LIQUOR SCHEMATIC  
(Continued)

COMPONENT	15 ALK. SOLUTION TO C-1	16 FILTER DREGS	17 GREEN LIQUOR PRODUCT	18 BOILER FEED WATER	19 BFW TO E-2	20 BFW TO E-3	PRO
lb/Hr----->							
O2	0.00	0.00	0.00	0.00	0.00	0.00	0.00
N2	0.00	0.00	0.00	0.00	0.00	0.00	0.00
CH4	0.00	0.00	0.00	0.00	0.00	0.00	0.00
C2H6	0.00	0.00	0.00	0.00	0.00	0.00	0.00
C2H4	0.00	0.00	0.00	0.00	0.00	0.00	0.00
C3H8	0.00	0.00	0.00	0.00	0.00	0.00	0.00
C3H6	0.00	0.00	0.00	0.00	0.00	0.00	0.00
C4	0.00	0.00	0.00	0.00	0.00	0.00	0.00
C5	0.00	0.00	0.00	0.00	0.00	0.00	0.00
C6	0.00	0.00	0.00	0.00	0.00	0.00	0.00
H2	0.00	0.00	0.00	0.00	0.00	0.00	0.00
H2S	0.00	0.00	0.00	0.00	0.00	0.00	0.00
NH3	0.00	0.00	49.41	0.00	0.00	0.00	0.00
CO	0.00	0.00	0.00	0.00	0.00	0.00	0.00
CO2	0.00	0.00	0.00	0.00	0.00	0.00	0.00
H2O(V)	0.00	0.00	0.00	0.00	0.00	0.00	0.00
H2O(L)	9438.92	0.00	9626.39	6374.42	3114.36	3260.05	
CARBON	0.00	53.46	0.00	0.00	0.00	0.00	0.00
TOTAL SODIUM	763.98	0.00	763.98	0.00	0.00	0.00	0.00
TOTAL SULFUR	0.00	0.00	174.77	0.00	0.00	0.00	0.00
TOTAL CARBONATE	979.87	0.00	979.87	0.00	0.00	0.00	0.00
TOTAL CHLORIDE	20.32	0.00	20.32	0.00	0.00	0.00	0.00
BLACK LIQUOR	0.00	0.00	0.00	0.00	0.00	0.00	0.00
TOTAL (lb/Hr)	11203.08	53.46	11614.74	6374.42	3114.36	3260.05	
TEMPERATURE (F)	100.00	100.00	120.00	77.00	77.00	77.00	
PRESSURE (PSIG)	20.00	0.00	5.00	60.00	60.00	60.00	
HHV (MMBTU/Hr)	0.00	0.75	0.48	0.00	0.00	0.00	
ENTHALPY (MMBTU/Hr)	0.23	0.75	0.92	0.00	0.00	0.00	



**SUMMARY FOR 2 TON/HR BLACK  
AT 65% SOLIDS BY WEIGHT**

(CONTINUED)

21	22	23	24	25	26	27	28
ESS STEAM TO E-1	EXPORT STEAM	DISS. TANK WTR TO E-4	DISS. TANK WTR TO V-5A/B	SCRUBBER WTR TO E-5	SCRUBBER WTR TO X-1	PRODUCT (#6) Recycle Gas to Blower B-1	PRODUCT Recycle Gas B-1 to E-1
0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
0.00	0.00	113.46	113.46	0.00	0.00	0.00	0.00
0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
4000.00	2374.42	0.00	0.00	0.00	0.00	0.00	0.00
0.00	0.00	22103.09	22103.09	203966.68	203966.68	0.00	0.00
0.00	0.00	122.76	122.76	0.00	0.00	0.00	0.00
0.00	0.00	1754.17	1754.17	0.00	0.00	0.00	0.00
0.00	0.00	401.28	401.28	0.00	0.00	0.00	0.00
0.00	0.00	2249.86	2249.86	0.00	0.00	0.00	0.00
0.00	0.00	46.65	46.65	0.00	0.00	0.00	0.00
0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
4000.00	2374.42	26791.27	26791.27	203966.68	203966.68	0.00	0.00
300.00	300.00	120.00	100.00	120.00	100.00	125.00	125.00
55.00	55.00	60.00	55.00	50.00	45.00	6.00	30.00
0.00	0.00	2.83	2.83	0.00	0.00	0.00	0.00
4.61	2.74	3.84	3.37	8.76	4.68	0.00	0.00

- B-1 START-UP BLOWER
- E-2 WASTE HEAT BOILER
- H-2 START-UP HEATER
- E-1 SUPERHEATER
- H-1 A-C PULSE HEATER
- V-8 BLACK LIQUOR TANK
- R-1 GASIFIER
- V-2 RECOVERY CYCLONE
- F-1 COMB. AIR SAN
- V-1 RECYCLE CYCLONE
- P-2 DISS. TANK PUMP
- V-3 DISSOLVING TANK
- E-3 PROCESS GAS BOILER
- X-1 VENTURI
- V-5A/B FILTERS
- C-1 GREEN LIQUOR COLUMN
- H-3 FLARE
- E-5 SCRUBBER COOLER
- P-3 SCRUBBER PUMP
- V-4 SCRUBBER TANK
- P-5 GREEN LIQUOR COLUMN FEED PUMP

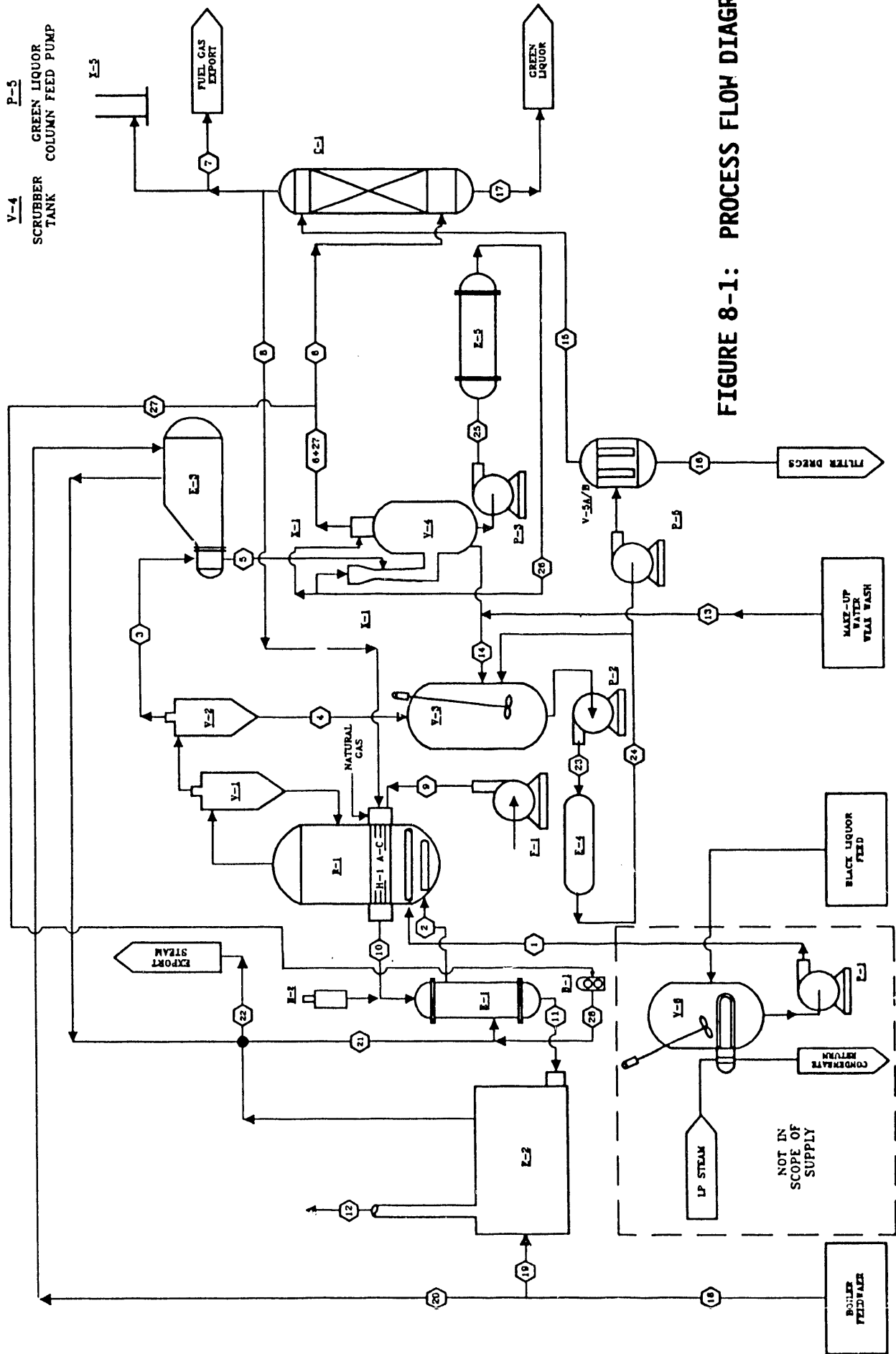


FIGURE 8-1: PROCESS FLOW DIAGRAM

The product gas enters a venturi jet scrubber (X-1) for high efficiency removal of particulate matter contained within the gas product. The venturi jet scrubber additionally cools and condenses steam from the gases. The circulating scrubber water is collected in a scrubber hold-up tank (V-4). The scrubber water is circulated through cooler E-5 to reject the latent and sensible heat of cooling. The product gases exit the hold-up tank and enter a spray cooler column mounted on the top of the scrubber hold-up tank to provide additional direct contact cooling.

The product gases exit the scrubber/cooler loop at approximately 125°F and enter the base of a counter-current packed column (C-1). Here, the gases are contacted against an alkaline scrubbing solution to remove hydrogen sulfide and produce a clean fuel gas product. A portion of the fuel gas product is utilized internally for combustion in the pulse heaters H-1 (A-D). The balance of the fuel gas is available for export to the end-user. Any portion of the fuel gas product which is not utilized is sent to an incinerator/flare (H-3).

Upon gasification of the black liquor, a solid sodium carbonate product is formed. A portion of this product is recovered in cyclone V-2 and discharged to dissolving tank V-3. A second portion of the solid product is recovered from an overflow port located on the reactor (R-1). This overflow port also discharges to the dissolving tank.

Steam condensed in the scrubber loop is discharged from V-4 to dissolving tank V-3 by means of an overflow port. Additional water for dissolution of product solids is provided from battery limits to V-3.

The sensible and dissolution heat of the solid products are removed using cooler E-4 by means of circulation from V-3 using pump P-2. A portion of the alkaline solution formed by dissolving the product solids is withdrawn to a pressure filter. Here, unconverted carbon and other insolubles are removed. The purified alkaline solution is delivered to the top of the counter-current packed column C-1 where hydrogen sulfide is re-adsorbed in solution to form green liquor product.

As previously mentioned, heat for the gasifier is supplied by four pulse heater modules. Clean fuel gases from C-1 are combusted in H-1 (A-D) and enter a pulsating fire-tube bundle that releases heat to the fluid bed. Flue gases exit the combustor tube bundle at 1350°F where they are directed to a steam superheater (E-1). The flue gases then enter waste heat boiler E-2 for final heat recovery.

A portion of the steam generated in process gas boiler E-3 and flue gas waste heat boiler E-2 is delivered to superheater E-1 where it is preheated to 800°F prior to entering the fluid bed gasifier reactor R-1. Any additional steam is exported to the end-user or vented.

During the start-up, blower B-1 supplies air to superheater E-1 in place of steam. An auxiliary burner (H-2) is provided to supply hot combustion products to the flue gas side of E-1 in order to raise the air temperature to approximately 600°F. This preheated air serves to heat the gasifier bed to approximately 400°F. Once the bed temperature is well above the steam saturation temperature, the blower may be turned off and steam can be utilized to fluidize the bed. Once the bed is fluidized, the pulse heaters can be activated.

## 8.4 EQUIPMENT LIST

The major equipment items for the gasification facility are listed below:

### Reactors

R-1 Gasifier

### Vessels

V-1 Recirculation Cyclone (hot section)  
V-2 Recovery Cyclone (hot section)  
V-3 Dissolving Tank  
V-4 Scrubber Tank  
V-5 A/B Green Liquor Filters  
V-6 Black Liquor Tank  
V-7 Flare Accumulator Drum  
V-8 Bed Media Storage Bin (hot section)  
V-9 Bed Media Day Tank (hot section)  
V-10 Dregs Tank

### Heat Exchangers

- E-1 Steam Superheater
- E-2 Flue Gas Waste Heat Boiler
- E-3 Process Gas Boiler
- E-4 Dissolving Tank Cooler
- E-5 Scrubber Cooler

### Columns

- C-1 Green Liquor Recovery Column

### Pumps

- P-1 Black Liquor Feed Pump
- P-2 Dissolving Tank Pump
- P-3 Scrubber Pump
- P-4 Caustic Metering Pump
- P-5 Recovery Column Pump
- P-6 Green Liquor Pump
- P-7 Dregs Pump

### Fans/Blowers

- F-1 Combustion Air Blower
- B-1 Start-Up Blower

### Heaters/Burners

- H-1 (A-D) Pulse Heater Modules (hot section)
- H-2 Start-Up Burner
- H-3 Flare

### Miscellaneous

- X-1 Venturi Scrubber
- X-2 Ash Discharge Screw (hot section)
- X-3 Dissolving Tank Agitator
- X-5 Rotary Lock Hopper (hot section)
- X-7 Storage Conveyor (hot section)
- X-8 Transfer Conveyor (hot section)
- X-9 Bucket Elevator (hot section)
- X-10 Flow Diverter (hot section)
- X-11 Dregs Tank Agitator.

## **8.5 GASIFIER DESIGN**

The Gasifier Hot Section consists of the gasifier (R-1), two cyclones, storage and transfer conveyors, bucket elevator, rotary lock hopper, flow diverter, and ash discharge screw. These items combine to provide solids handling, process reactor, and gas/solids separation.

The bucket elevator, rotary, lock hopper, conveyor, flow diverter, and ash discharge screw exist for two purposes: 1) to fill the gasifier (R-1) with soda ash, and 2) to drain the reactor of all solids.

The gasifier design is critical to the operation of the process. The success of the process depends upon its ability to operate well below the eutectic melting point of an ash/sodium carbonate mixture and the ability to maintain uniform bed temperatures without local hot spots. Heat is supplied indirectly to the fluidized bed by four pulse combustor fire-tube modules stacked two high. The reactor is designed with side walls having a modest radius of curvature to improve pressure capability. The side walls are supported by means of buckstays. The reactor is lined with refractory and the side walls come in close approach to the tube bundle. The bed is composed primarily of soda ash and is fluidized via spargers by either superheated steam, recycled gas, or both. Black liquor is introduced to the bed above the spargers by a series of ports encircling the vessel. The bed height at full fluidization is maintained mechanically by means of an overflow pipe which serves the additional function of defining the freeboard. Entrained solids that escape the gasifier enter cyclone V-1 which collects the coarse particles and returns them to the reactor by means of a fluidized dip leg. Any fines leaving cyclone V-1 are captured by cyclone V-2 and removed from the hot section to the dissolving tank. Solids accumulated during the process are also removed to the dissolving tank via the overflow line.

## **8.6 P&ID**

See attached Piping and Instrumentation Diagrams (5 drawings).

## Drawing List

B100 -	Summary of Standard Symbols
10042-P-001 SH 1, Rev. C -	Tower #1 Reactor R-1
10042-P-001 SH 2, Rev. C -	Tower #2 Chemical and Waste Heat Recovery
10042-P-001 SH 3, Rev. C -	Tower #3 Superheater and Waste Heat Recovery
10042-P-001 SH 4, Rev. C -	Tower #4 Off Skid & Bed Media Storage

## **8.7 LOGIC FLOW DIAGRAM**

### **8.7.1 GASIFIER START-UP LOGIC** (Drawing #10042-L-001, Rev. D)

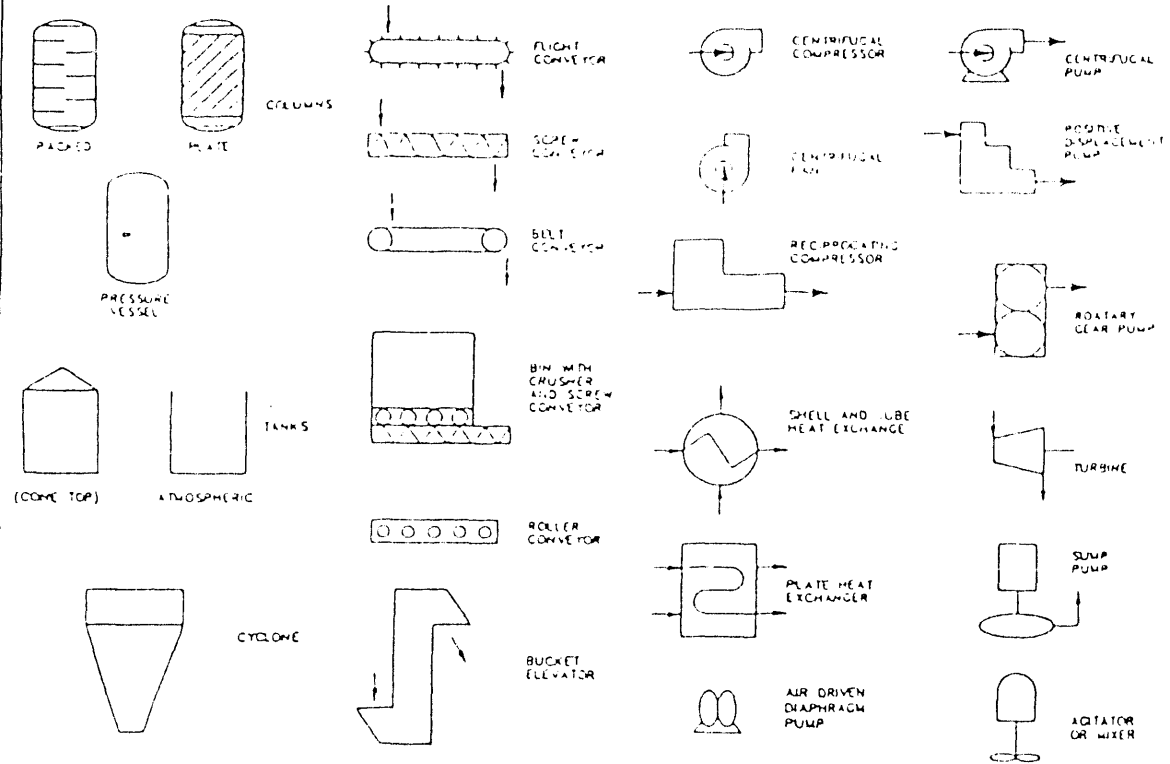
The gasifier process must meet 16 tests of process status correctly before start-up may proceed. These process conditions are further divided into two groups of eight each. The first group includes those process conditions required for start-up, but which will not shut down the pilot plant once start-up is complete. The second group of "critical" conditions monitors fluidization, bed temperatures, pressures, and levels which must be met at all times or a plant shut down will occur. When both group conditions are correct, the "Ready to Start" light will indicate. Initiation of start-up with the "Start-Process" push button initiates the logic checking and action sequences that:

- Start the pumps and mixer for the scrubber and dissolving tanks.
- Fluidize the gasifier bed with air or steam depending upon the gasifier (R-1) temperatures.
- Initiate burner start-up sequences for H-1 (A-D) and H-2.
- Starts the black liquor feed pump at the appropriate temperature.

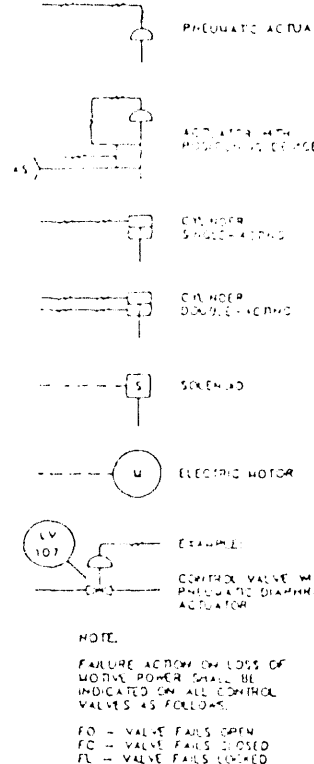
The pumps and mixer for the scrubber and dissolving tank receive the start signal automatically via the process start push button or manually from another push button. Similarly, they may be shut off.

The gasifier bed is fluidized with either air or steam, depending upon the existing gasifier temperature and the set point. If the gasifier temperature is below the temperature switch setting of 400°F, then the start-up blower B-1 is started and logic for the start-up burner H-2 begun. If the temperature of the bed is above 400°F, then the start-up blower B-1 is shut

## MECHANICAL EQUIPMENT SYMBOLS



## ACTUATORS



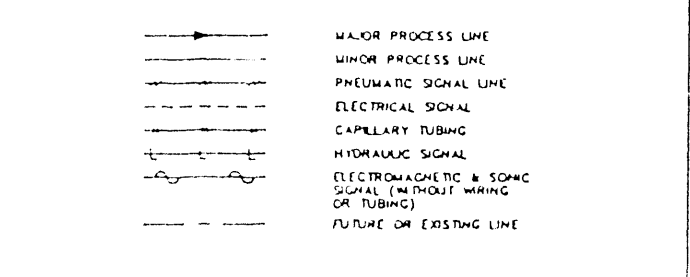
**NOTE:**  
FAILURE ACTION ON LOSS OF MOTIVE POWER SHALL BE INDICATED ON ALL CONTROL VALVES AS FOLLOWS:  
FO - VALVE FAILS OPEN  
FC - VALVE FAILS CLOSED  
FL - VALVE FAILS LOCKED IN LAST POSITION

ALL VALVES FAIL CLOSED UNLESS OTHERWISE DESIGNATED

### PIPE SERVICES LEGEND

PROCESS		UTILITIES	
PROCESS GAS	GP	PLANT AIR	PA
FLUE GAS	GF	INSTRUMENT AIR	IA
RECYCLED-SOLIDS	RS	PROPANE GAS	GPC
O/T SOLIDS	OS	COOLING WATER SUPPLY	CWS
BED MEDIA	BM	COOLING WATER RETURN	CWR
BLACK LIQUOR	BL	PLANT STEAM	PS
GREEN LIQUOR	GL	SUPERHEATED STEAM	SHS
PROCESS AIR	AP	CONDENSING WATER	WML
CAUSTIC	CA	BOILER FEED WATER	BFW
DRESS	DR		
DISSOLVING TANK WATER	DTW		
SCRUBBER WATER	SW		

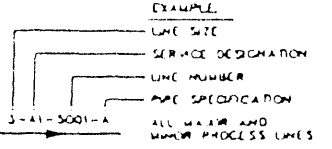
### LINE LEGEND



### LINE NUMBER SERIES

- 100 - FLUE GAS, PROPANE
- 200 - BLACK/GREEN LIQUOR
- 300 - AIR
- 400 - STEAM
- 500 - WATER
- 600 - PROCESS GAS
- 700 - SOLIDS
- 800 - CAUSTIC
- 900 - MISC

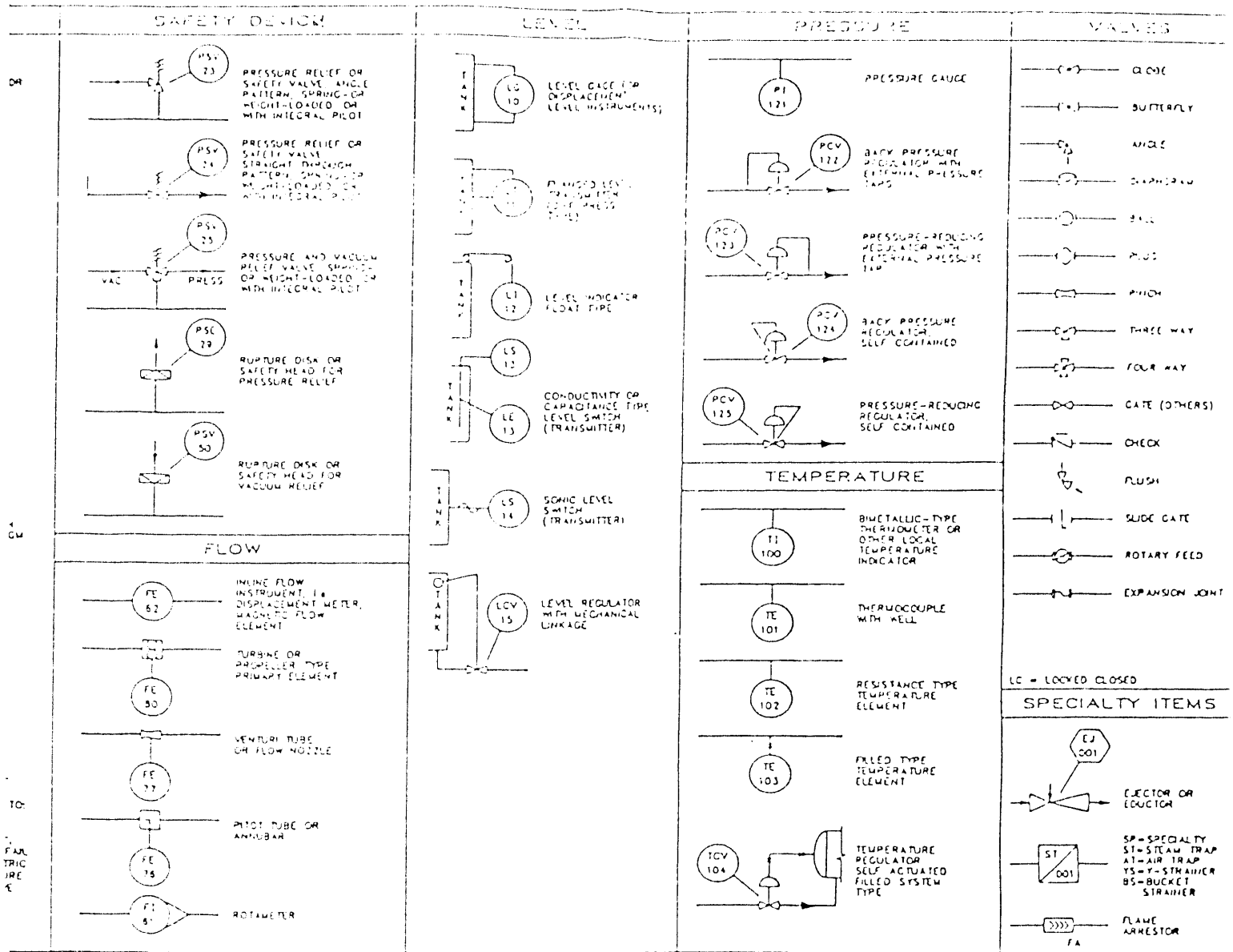
### LINE NUMBERING SYSTEM (REFERENCE STANDARD L-100)



### INSTRUMENT FUNCTIONS AND SYMBOLS

	LOCAL INSTRUMENT FOR SINGLE MEASURED VARIABLE	$\int$	INTEGRATE		CHEMICAL SEAL
	LOCAL INSTRUMENT FOR TWO MEASURED VARIABLES OR MORE THAN ONE FUNCTION	$\Sigma$	ADD OR TOTALIZE		INTERLOCK
	MOUNTED ON THE MAIN PANEL	$\Delta$	SUBTRACT		PURGE OR FLUSHING DEVICE (MEANS OF REGULATION PURGE MAY BE SHOWN IN PLACE OF SYMBOL)
	MOUNTED BEHIND THE PANEL	+	GRADE		MANUAL RESET
	MOUNTED ON LOCAL PANEL	$\sqrt{\quad}$	EXTRACT SQUARE ROOT		
	PILOT LIGHT	x	MULTIPLY		
	SOFTWARE FUNCTION	(x)	CHARACTERIZE		
		$x^{1/n}$	RAISE TO POWER		
		H	HIGH-SELECT (SELECT HIGHEST MEASURED VARIABLE (NOT SIGNAL UNLESS SO NOTED))		
		L	LOW-SELECT (SELECT LOWEST MEASURED VARIABLE)		
		SP	SET POINT		
		PV	PROCESS VARIABLE		
		SR	SPLIT RANGE		
			TRANSMISSION MODE		
		E	VOLTAGE		
		H	HYDRAULIC		
		I	CURRENT		
		O	ELECTROMAGNETIC OR SONIC		
		P	PNEUMATIC		
		R	RESISTANCE		
		C/P/R/P	MODE CONVERSION		





**INSTRUMENT IDENTIFICATION LETTERS**

FIRST LETTER		SUCCEEDING LETTERS		
MEASURED OR INDICATING VARIABLE	MODIFIER	READOUT OR PASSIVE FUNCTION	OUTPUT FUNCTION	MODIFIER
LEVELS		ALARM		
NER FLAME				
QUANTITY (G/G)			CONTROL	
SURVEILLANCE (OR SPEED CHANGE)	DIFFERENTIAL			
TARGE (LOW)		PRIMARY ELEMENT		
TRATE	RAID			
NO DIMENSIONAL		GLASS		
D (TEMPERATURE)				HIGH
MENT		INDICATE		
TR	LEAN			
OR (TEMPERATURE)			CONTROL STATION	
OR (TEMPERATURE)		LOCAL (FIELD)		LOW
OR (TEMPERATURE)				W/DOLE
PS (CHOICE)				
PS (CHOICE)		DIFFERENTIAL		
SSURE OR (PRESSURE)		PROBE (TEST)		
NTITY OR (ELEMENT)	INTEGRAL OR TOTALIZE			
ACCURACY		RECORD OR PRINT		
OR (TEMPERATURE)	SAFETY		SWITCH	
OR (TEMPERATURE)			TRANSMIT	
DYNAMIC		W/ALARM	W/ALARM FUNCTION	
OR (TEMPERATURE)			VALVE DAMPER (OVER)	
OR (TEMPERATURE)				
OR (TEMPERATURE)		RELAY OR (CONTROL)		
OR (TEMPERATURE)		OR (CONTROL)		

WTC (158900) B100 SEN 180690

A	180690	ISSUED FOR DETAIL DESIGN	15/11/80	D/S
REV	DATE	DESCRIPTION	BY	CHK
001	BY DATE	002	BY DATE	PROJ ENGR
001	BY DATE	001	BY DATE	APPROV

PREPARED BY: **ESI** ENGINEERING SERVICES, INC. WASHINGTON, CALIFORNIA

**MAIN** 1988 CHAS. T. WALK, INC. CHARLOTTE, N.C.

PROJECT: **M T C I** SANTA FE SPRINGS, CA. 90670

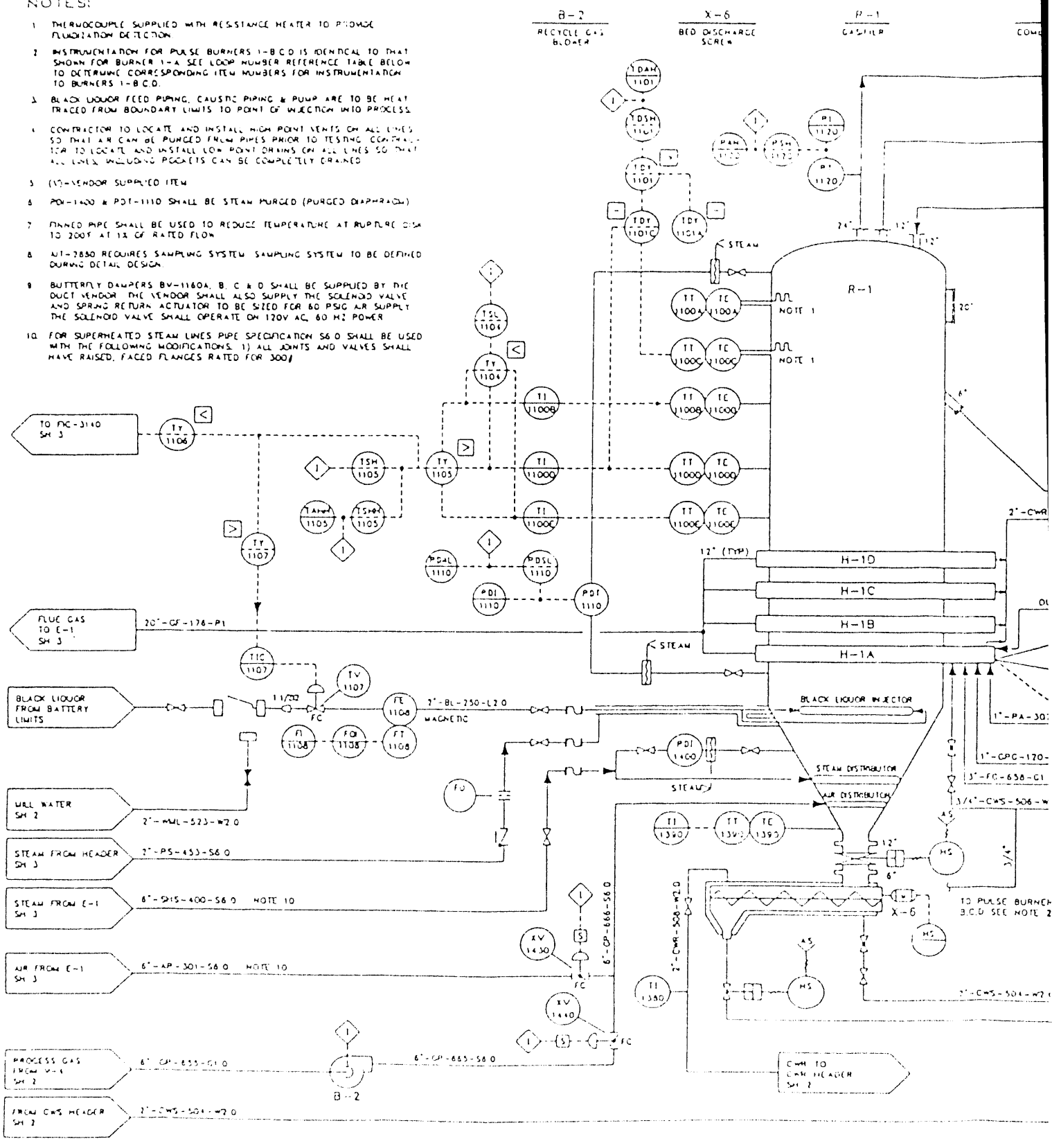
PROJECT: **2 TPH BLACK LIQUOR GASIFIER**

FILE: **PIPING AND INSTRUMENTATION DIAGRAM SUMMARY OF STANDARD SYMBOLS**

SCALE	PROJECT NUMBER	DRAWING NUMBER	REV.
NONE	158900	B100	A

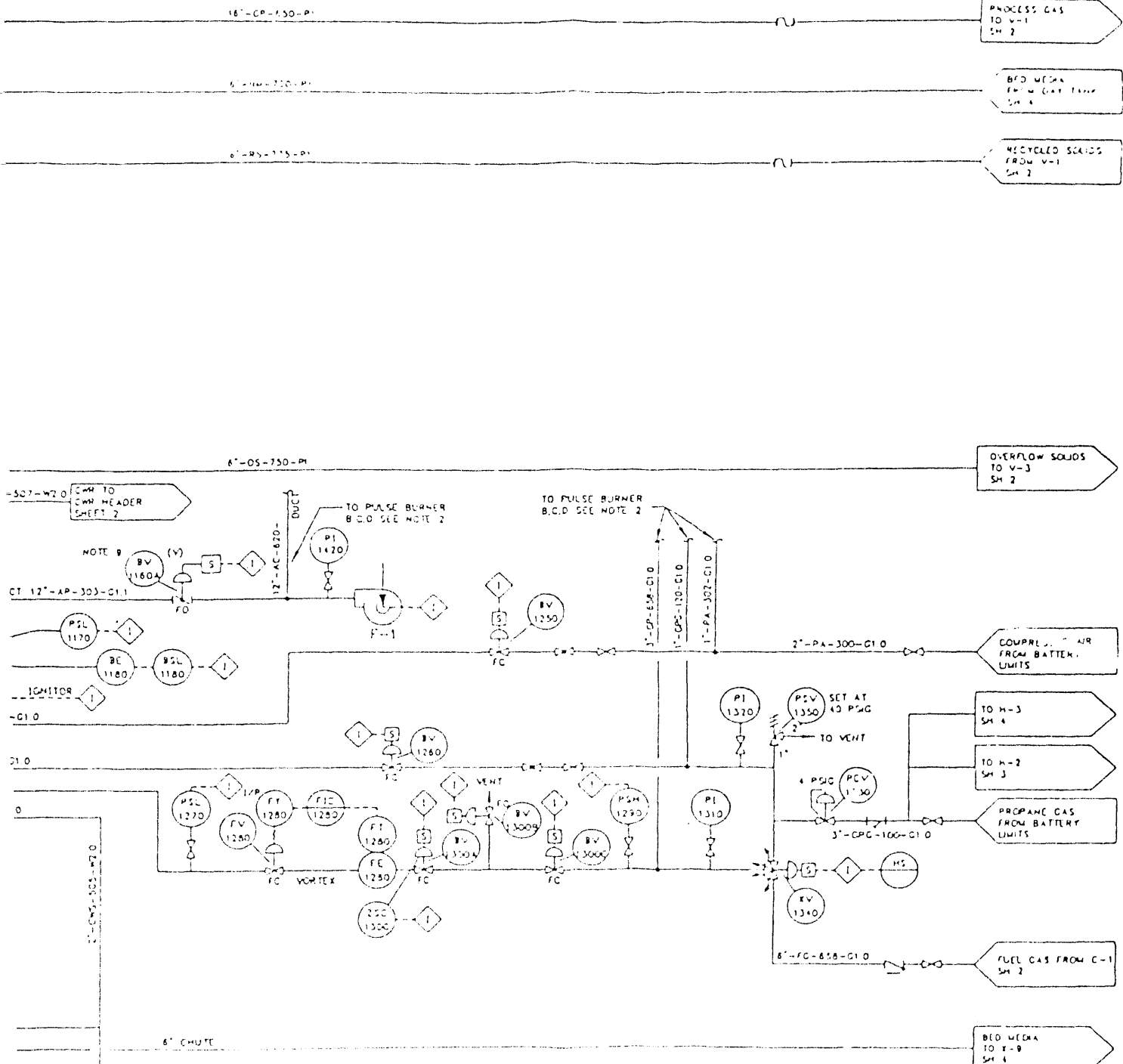
NOTES:

1. THERMOCOUPLE SUPPLIED WITH RESISTANCE HEATER TO PROVIDE FLUORINATION DETECTION
2. INSTRUMENTATION FOR PULSE BURNERS 1-B C D IS IDENTICAL TO THAT SHOWN FOR BURNER 1-A. SEE LOOP NUMBER REFERENCE TABLE BELOW TO DETERMINE CORRESPONDING ITEM NUMBERS FOR INSTRUMENTATION TO BURNERS 1-B C D.
3. BLACK LIQUOR FEED PIPING, CAUSTIC PIPING & PUMP ARE TO BE HEAT TRACED FROM BOUNDARY LIMITS TO POINT OF INJECTION INTO PROCESS.
4. CONTRACTOR TO LOCATE AND INSTALL HIGH POINT VENTS ON ALL LINES SO THAT AIR CAN BE PURGED FROM PIPES PRIOR TO TESTING. CONTRACTOR TO LOCATE AND INSTALL LOW POINT DRAINS ON ALL LINES SO THAT ALL LINES INCLUDING POCKETS CAN BE COMPLETELY DRAINED.
5. (V)-VENDOR SUPPLIED ITEM
6. PDV-1400 & PDT-1110 SHALL BE STEAM PURGED (PURGED DIAPHRAGM)
7. FINNED PIPE SHALL BE USED TO REDUCE TEMPERATURE AT PIPE SURFACE TO 200F AT 1X OF RATED FLOW
8. AT-7850 REQUIRES SAMPLING SYSTEM. SAMPLING SYSTEM TO BE DEFINED DURING DETAIL DESIGN.
9. BUTTERFLY DAMPERS BV-1180A, B, C & D SHALL BE SUPPLIED BY THE OUCT VENDOR. THE VENDOR SHALL ALSO SUPPLY THE SOLENOID VALVE AND SPRING RETURN ACTUATOR TO BE SIZED FOR 60 PSIG AIR SUPPLY. THE SOLENOID VALVE SHALL OPERATE ON 120V AC, 60 HZ POWER.
10. FOR SUPERHEATED STEAM LINES PIPE SPECIFICATION S6.0 SHALL BE USED WITH THE FOLLOWING MODIFICATIONS: 1) ALL JOINTS AND VALVES SHALL HAVE RAISED, FACED FLANGES RATED FOR 300F.



LOOP NUMBER REFERENCE TABLE

BURNER NO	IILN NO								
H-1A	1160A	1170	1180	1250	1260	1270	1280	1290	1300
H-1B	1160B	1170	1180	1250	1260	1270	1280	1290	1300
H-1C	1160C	1170	1180	1250	1260	1270	1280	1290	1300
H-1D	1160D	1170	1180	1250	1260	1270	1280	1290	1300



REV	DATE	DESCRIPTION	BY	CHK	DATE	PROJ	ENGR	DATE	APP'D	DATE
F	10MCR90	ISSUED FOR DETAIL DESIGN								
E	10SEP90	ISSUED FOR DESIGN								
D	08SEP90	ISSUED FOR DESIGN								
C	13JUN90	ISSUED FOR DESIGN								
B	31MAY90	ISSUED FOR DESIGN								
A	17MAY90	ISSUED FOR APPROVAL								

PREPARED BY: **ESI**  
ENGINEERING SERVICES INC  
WARMER, CALIFORNIA

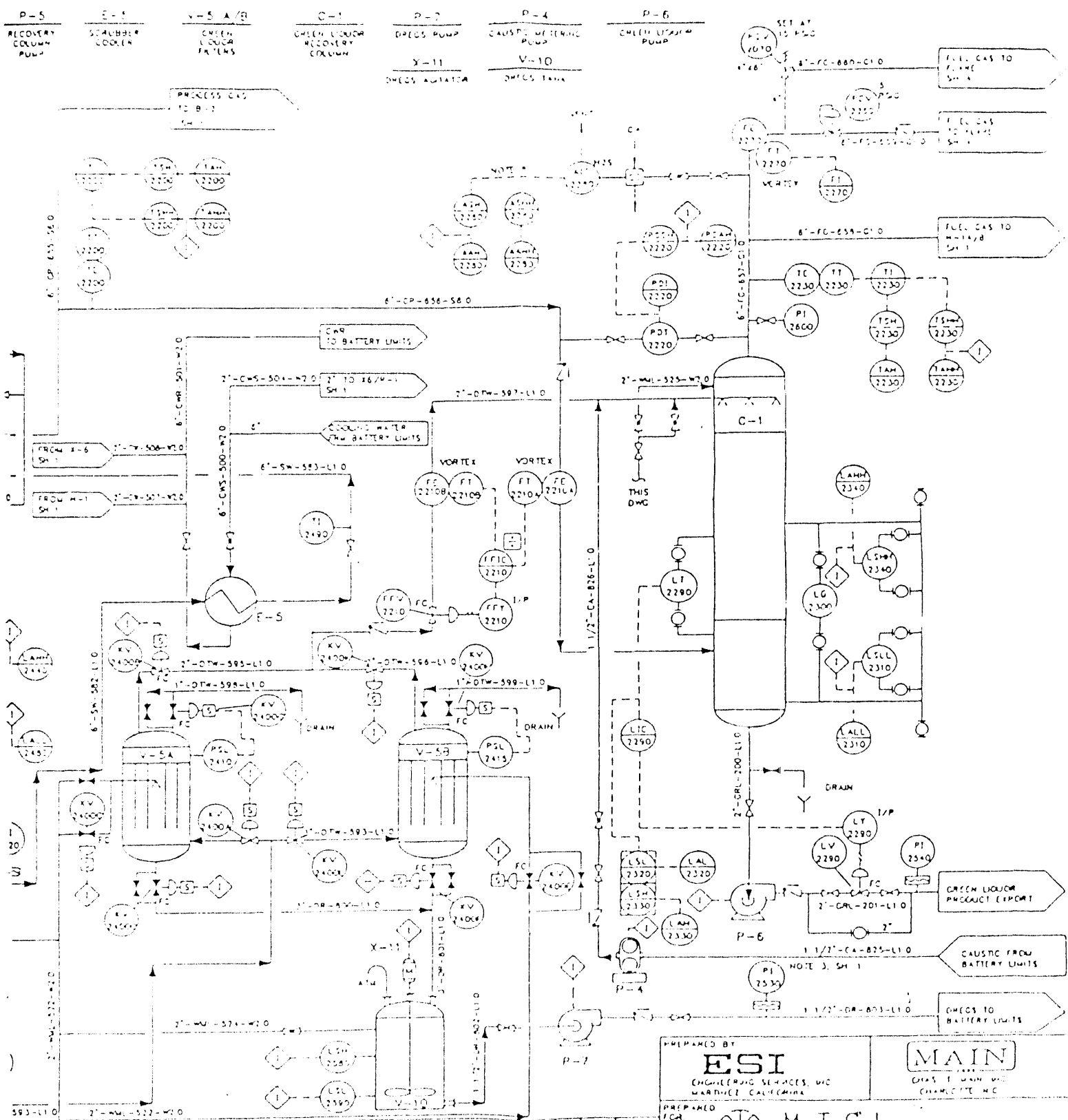
**MAIN**  
CHARLOTTE, NC

PROJECT: **M T C I**  
SANTA FE SPRINGS, CA. 90670

DESCRIPTION: **2, TPH BLACK LIQUOR GASIFIER TOWER #1 PIPING & INSTRUMENT DIAGRAM**

SCALE	PROJECT NUMBER	ENGINEER NUMBER	REV
NONE	458900	10042-P-001 SH 1	F





PREPARED BY  
**ESI**  
 ENGINEERING SERVICES, INC.  
 MARLBOROUGH, CALIFORNIA

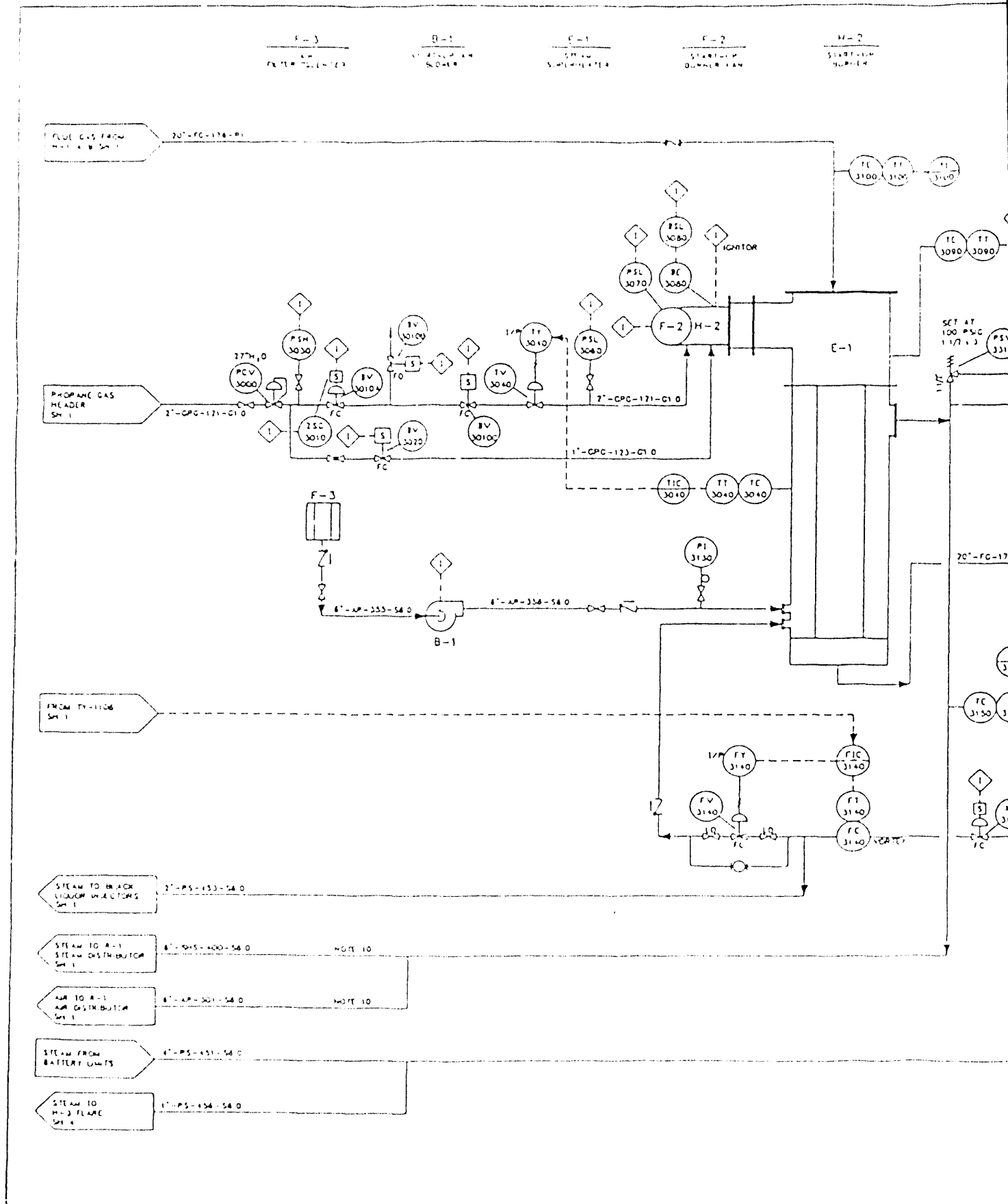
**MAIN**  
 CHAS. F. UHLMANN, INC.  
 CHICAGO, ILL. U.S.A.

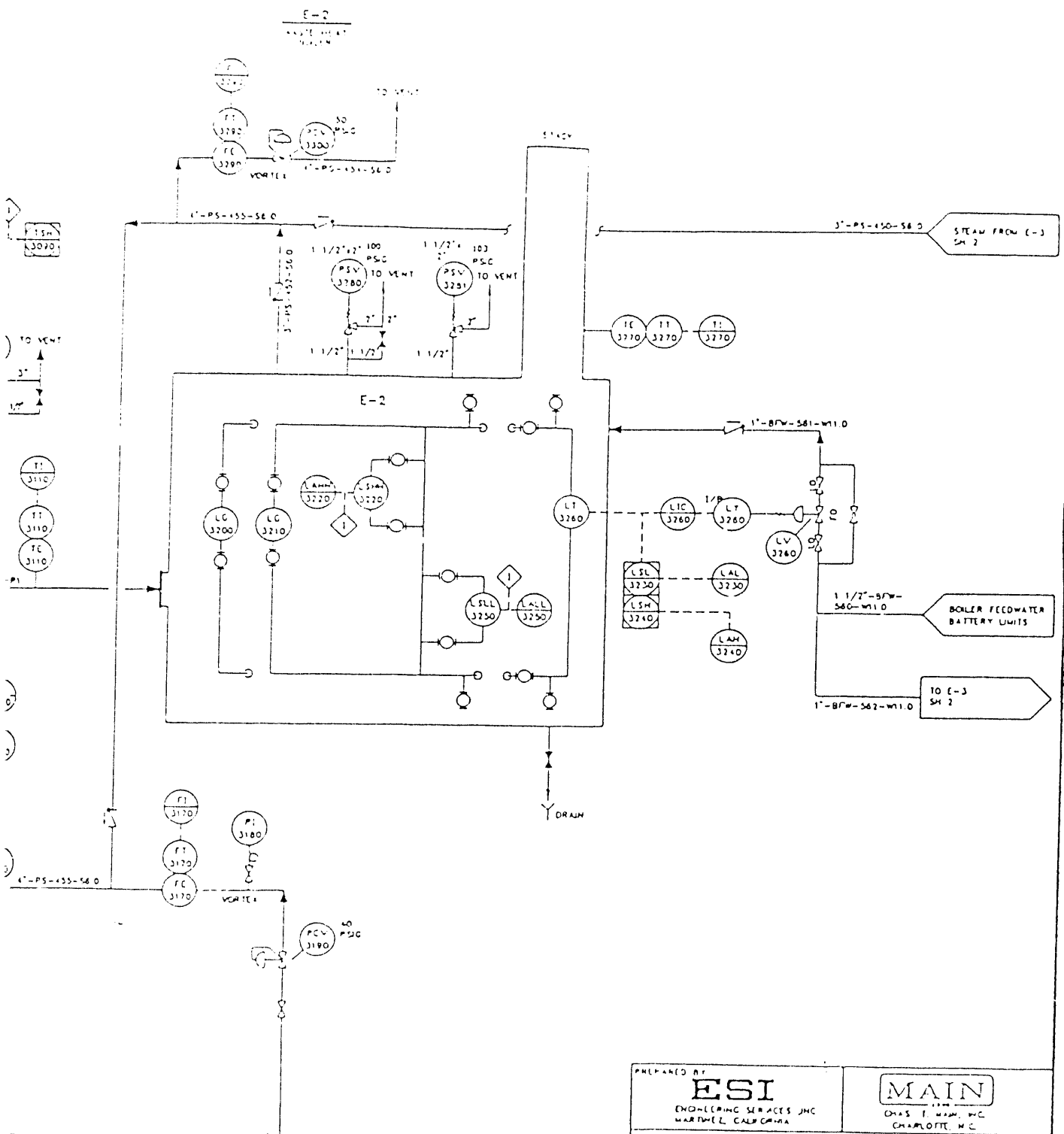
PREPARED FOR  
**MTCI**  
 SANTA FE SPRINGS, CA. 90670

NOTE  
 2 TPH BLACK LIQUOR GASIFIER  
 TOWER #2  
 PIPING & INSTRUMENT DIAGRAM

REV	DATE	DESCRIPTION	DESIGNER	CHECKER	APPROVER
F	10/02/90	ISSUED FOR DETAIL DESIGN	ES	DM	WCB
E	07/26/90	ISSUED FOR DESIGN	ES	DM	WCB
D	04/25/90	ISSUED FOR DESIGN	ES	DM	WCB
C	11/23/89	ISSUED FOR DESIGN	PI	DM	WCB
B	01/14/89	ISSUED FOR DESIGN	PI	DM	WCB
A	11/24/89	ISSUED FOR APPROVAL	PI	DM	WCB

SCALE	PROJECT NUMBER	DRAWING NUMBER	REV
NONE	458900	10042-P-001 SH 2	F





REV	DATE	DESCRIPTION	BY	CHK	PROJ. ENGR	DATE	APP'D	DATE
F	10/26/90	ISSUED FOR DETAIL DESIGN	ES	WJ	SCJ			
E	7/25/90	ISSUED FOR DESIGN	ES	OW	MCB			
D	10/26/90	ISSUED FOR DESIGN	ES	OW				
C	11/22/90	ISSUED FOR DESIGN	J/H		DP			
B	11/22/90	ISSUED FOR DESIGN	PE		WE			
A	11/22/90	ISSUED FOR APPROVAL	PE	WJ	NO			

PREPARED BY  
**ESI**  
 ENGINEERING SERVICES INC  
 MARTINEZ, CALIFORNIA

**MAIN**  
 CHAS. F. HARR, INC.  
 CHARLOTTE, N.C.

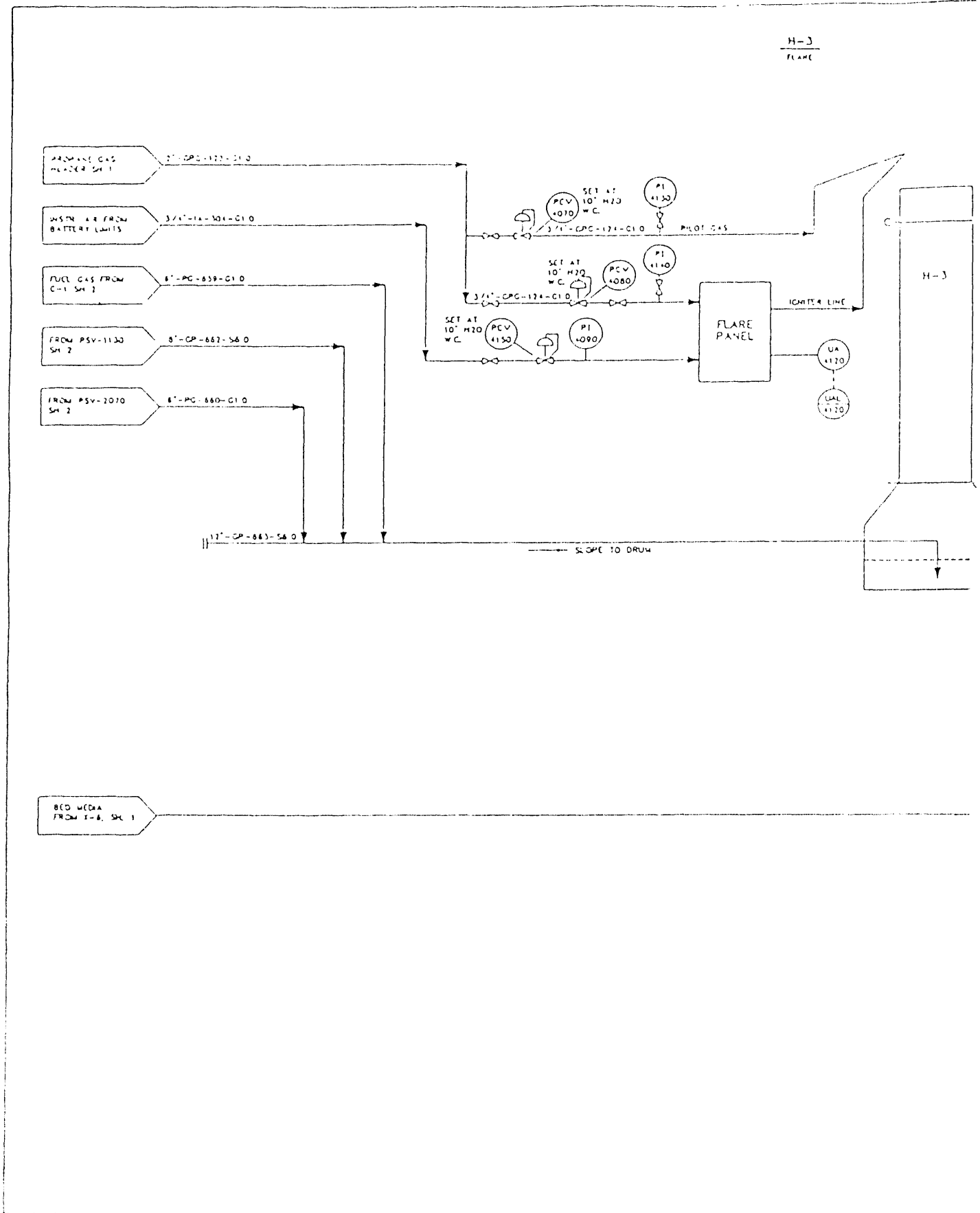
PREPARED FOR  
**MTCI**  
 SANTA FE SPRINGS, CA 90033

PROJECT  
 2TPH BLACK LIQUOR GASIFIER  
 TOWER #3  
 PIPING & INSTRUMENT DIAGRAM

SCALE	PROJECT NUMBER	DRAWING NUMBER	REV
NONE	458900	10042-P-001 SH 3	F

W101458900-001-3 EPS 12/26/90

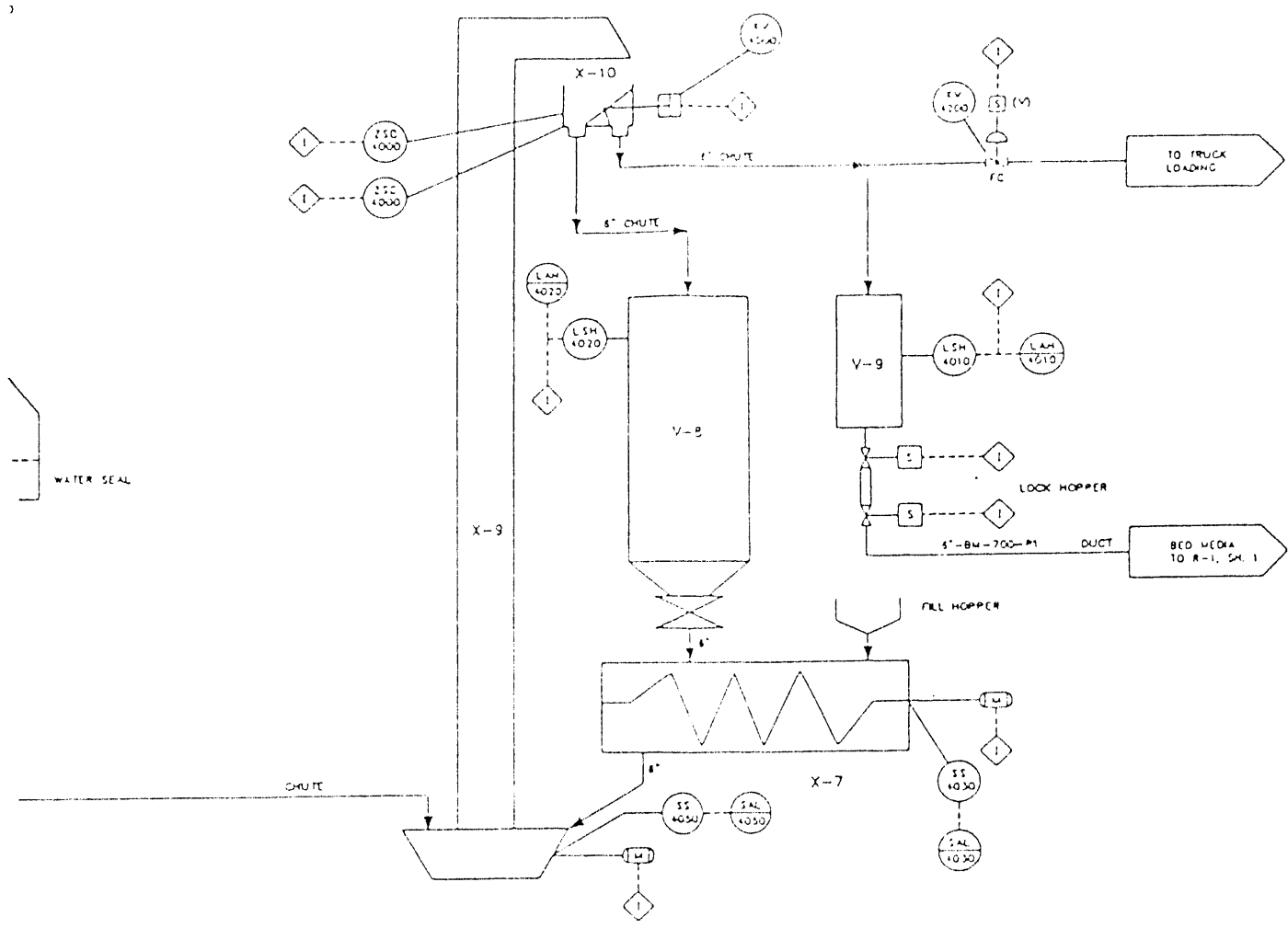
H-3  
FLAME



BCD MEDIA  
FROM I-8, SH 1



X-9 DUCT ELEVATOR	X-10 FLOW DISTRIBUTOR	V-8 MEDIA STORAGE TANK	X-7 LOADING SCHEME CONNECTOR	V-9 MEDIA DUMP TANK
-------------------------	-----------------------------	---------------------------------	---------------------------------------	------------------------------



PREPARED BY <b>ESI</b> ENGINEERING SERVICES, INC. 1000 N. GARDNER AVE. SANTA FE SPRINGS, CA. 90670	<b>MAIN</b> OVER 1000 PIPING COMPLETE
PREPARED FOR <b>M T C I</b> SANTA FE SPRINGS, CA. 90670	TITLE <b>2 TPH BLACK LIQUOR GASIFIER          OFF SKID &amp; BED MEDIA STORAGE          PIPING &amp; INSTRUMENT DIAGRAM</b>
SCALE NONE	PROJECT NUMBER 458900
DRAWING NUMBER 10047-P-001 SH. 4	REV. 1

REV.	DATE	DESCRIPTION	BY	CHKD.	PROJECT NO.	DATE
7	10/01/79	ISSUED FOR DESIGN	ES	WJ		
6	07/24/79	ISSUED FOR DESIGN	ES	WJ		
5	07/24/79	ISSUED FOR DESIGN	ES	WJ		
4	07/24/79	ISSUED FOR DESIGN	ES	WJ		
3	07/24/79	ISSUED FOR DESIGN	ES	WJ		
2	07/24/79	ISSUED FOR APPROVAL	ES	WJ		
1	07/24/79	ISSUED FOR APPROVAL	ES	WJ		

off and the fluidizing steam valve is opened. Confirmation that the main steam is on will be indicated with a panel light. The main steam valve can be operated manually as well.

If the main steam is on, then the logic sequences for firing the pulse combustor modules H-1A through H-1D are begun. If the main steam is on, the bed temperature is at least 1000°F and the gasifier bed is fluidized as measured by differential pressure and differential temperature (heat flux probe), then the black liquor feed pump P-1 will start.

### **8.7.2 H-1 (A-D) BURNER START-UP LOGIC** (Drawing #10042-L-002, Rev. D)

When the gasifier start-up sequence signals to start the pulse combustor burners, the air damper is closed, the combustion air fan is turned on and the combustion air plenum pressure to start-up burner H-2 is checked. This initiates a one-minute timed purge cycle during which the "purging" light comes on. The "purge complete" light comes on after one minute. The fuel gas supply pressure is checked, then the ignitor is turned on. The main fuel valve remains closed until the pilot is verified. After flame has been detected for one minute and the main fuel is on, the pilot gas valves are shut off. Flame failure shuts down the burner requiring a manual reset for a repeated start-up.

### **8.7.3 H-2 START-UP BURNER** (Drawing #10042-L-002, Rev. D)

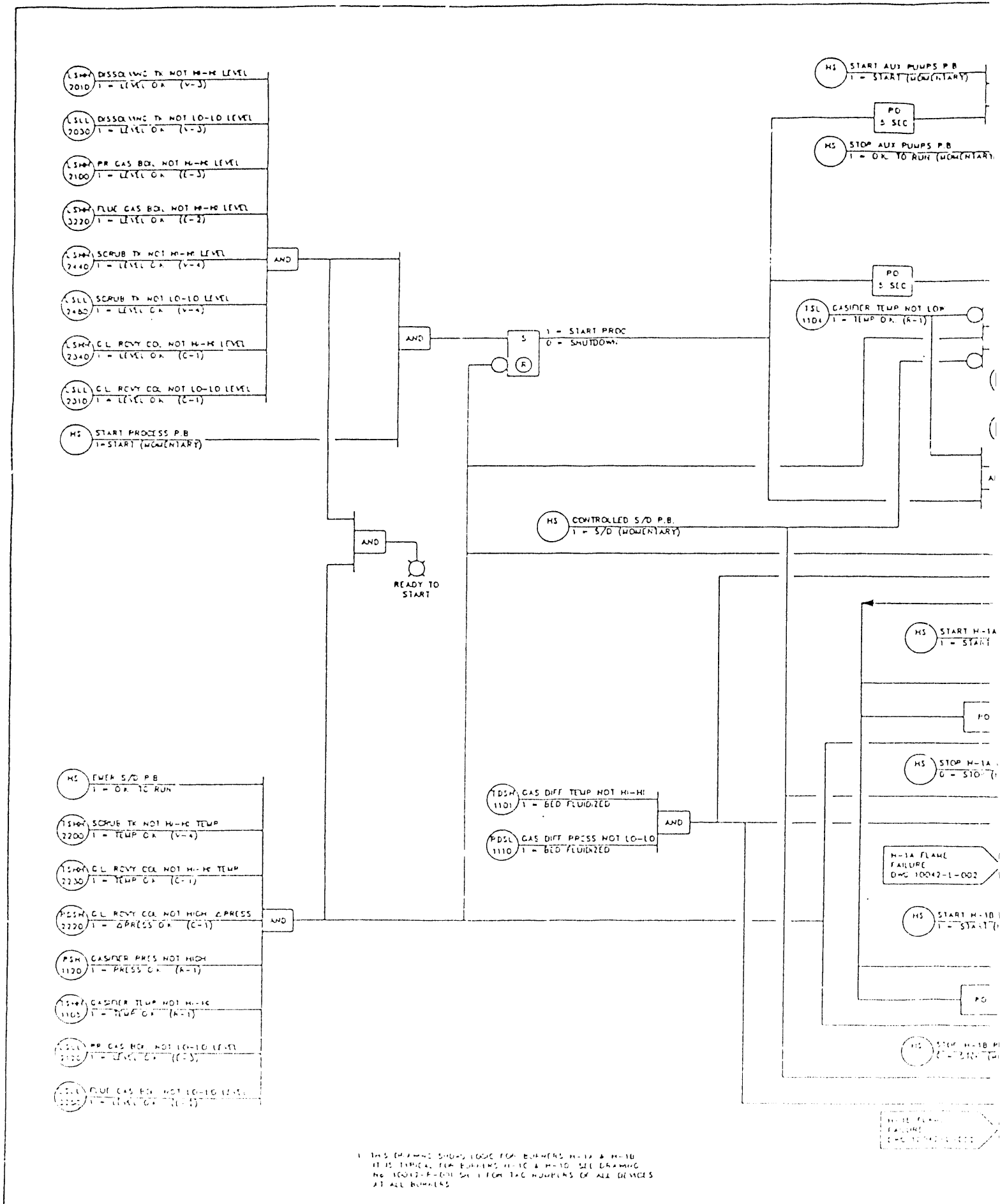
A start signal from either automatic or manual push button first starts the burner start-up fan F-2. When combustion air pressure is sufficient, the purge cycle begins. When the purge cycle is completed (one minute), the ignitor is powered and the pilot gas valves opened. When flame is on, the main fuel gas valves are opened. If flame is lost, the unit shuts down.

### **8.7.4 CONTROL OF H<sub>2</sub>S EMISSIONS** (Drawing #10042-L-003, Rev. D)

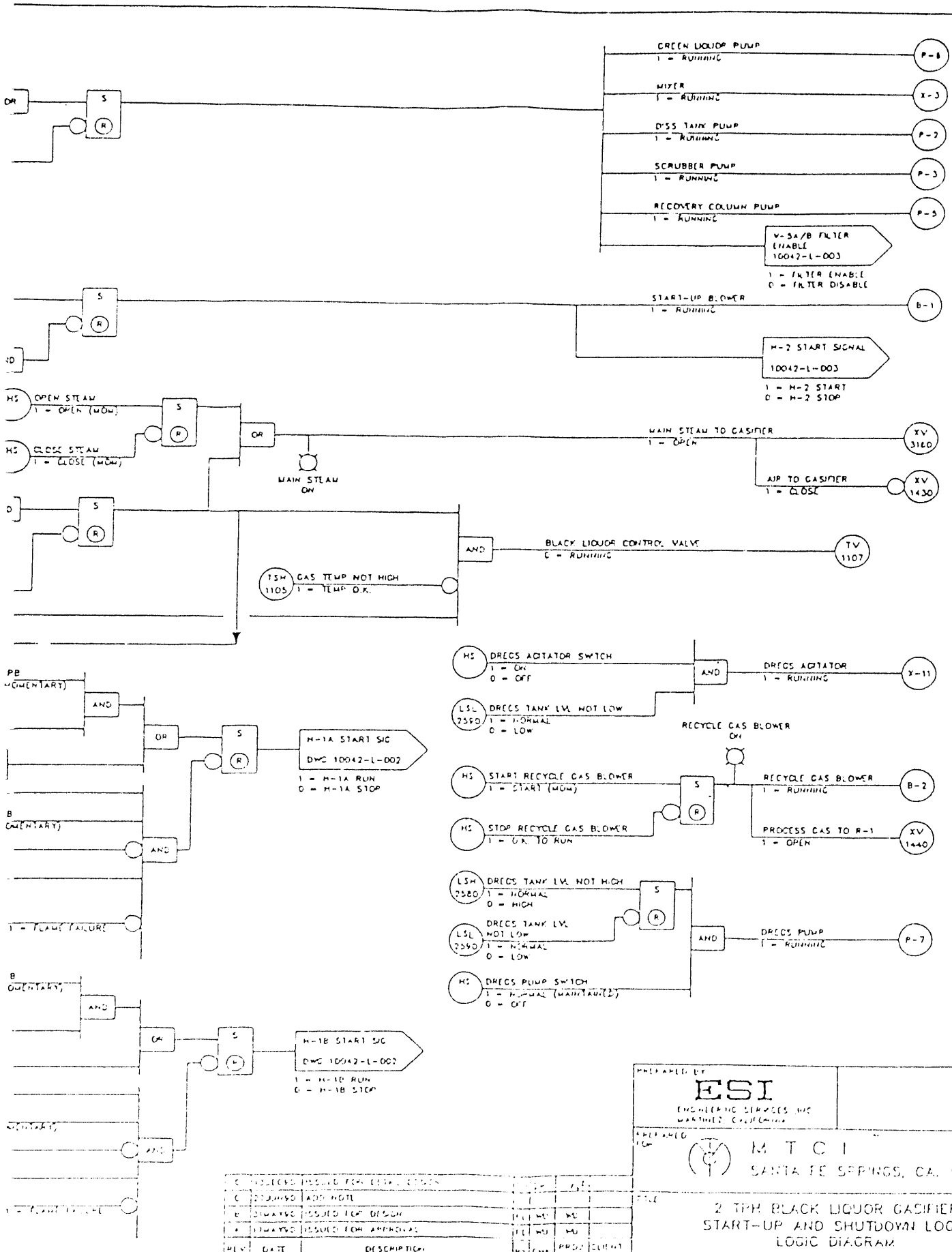
If the H<sub>2</sub>S analyzer switch is tripped, then the caustic metering pump is turned on to add caustic to column C-1. If the H<sub>2</sub>S levels are below the switch setting, then the metering pump to column C-1 is off.

### **8.7.5 FILTER V-5A/B CONTROL LOGIC** (Drawing #10042-L-003, Rev. D)

Two parallel filters (V-5A/B) are operated based on a predetermined logic cycle. When one filter is on-line, the other is isolated by means of three-way valves located at the entrance and exit. When the filter pressure exceeds a set pressure, indicating saturation of the filter elements, then the on-line filter is automatically isolated and drained while the opposing filter is brought on-line. The isolated filter is spray sluiced to discharge the accumulated filter cake. The filter remains isolated until the opposing filter becomes saturated.



1 THIS DRAWING SHOWS LOGIC FOR BURNERS H-1A & H-1B  
 IT IS TYPICAL FOR BURNERS H-1C & H-1D SEE DRAWING  
 NO 10042-P-001 FOR THE TAG NUMBERS OF ALL DEVICES  
 AT ALL BURNERS



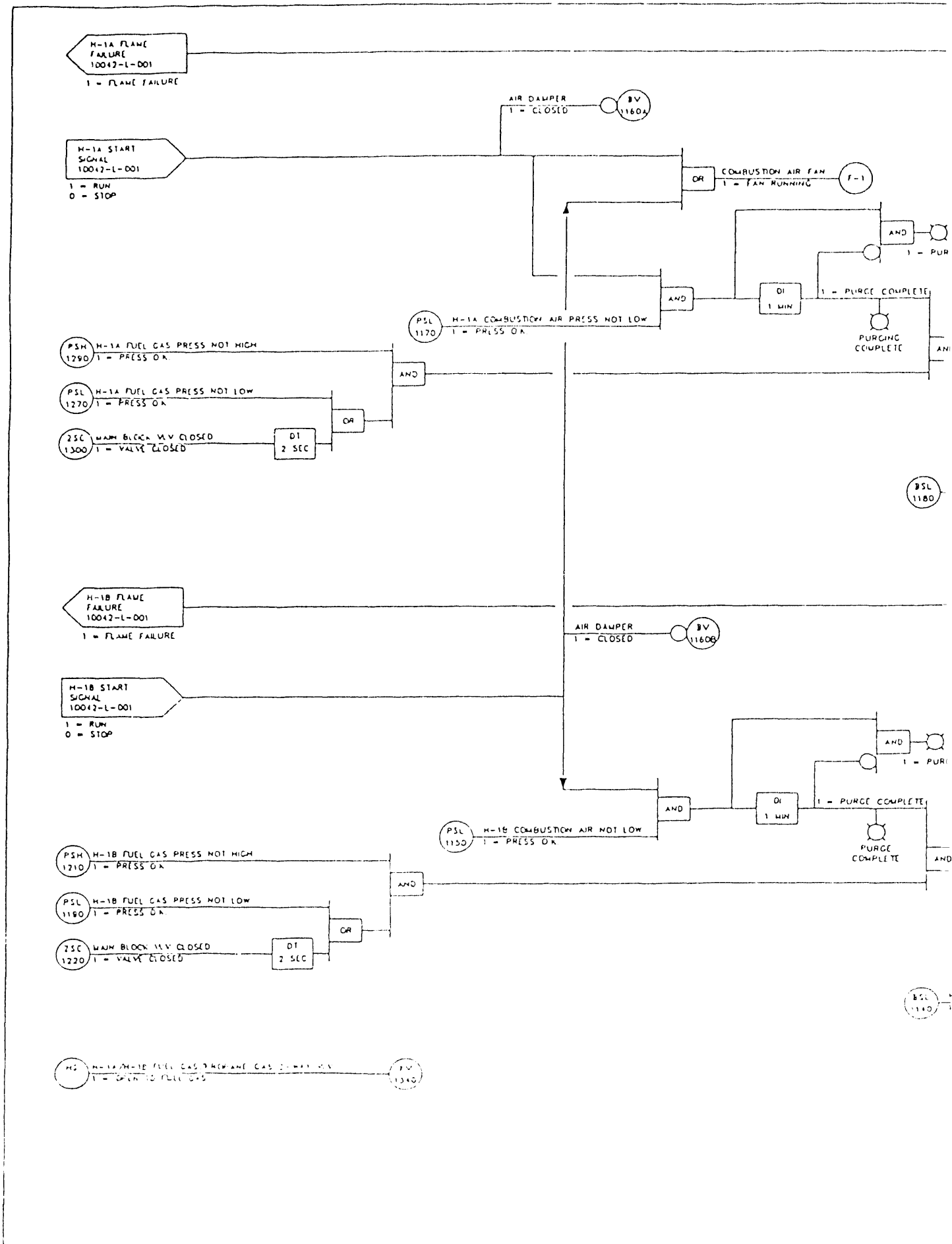
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C		ISSUED FOR MTL						
E		ISSUED FOR DESIGN						
A		ISSUED FOR APPROVAL						
REV	DATE	DESCRIPTION	BY	CHK	PROJ ENGR	CLIENT APPV		
DATE	DES BY	CHK BY	DATE	PROJ ENGR	DATE	APPROV	DATE	
DATE	DATE	DATE	DATE	DATE	DATE	DATE	DATE	

PREPARED BY  
**ESI**  
 ENGINEERING SERVICES, INC.  
 WASHINGTON, CALIFORNIA

PREPARED FOR  
**M T C I**  
 SANTA FE SPRINGS, CA. 90670

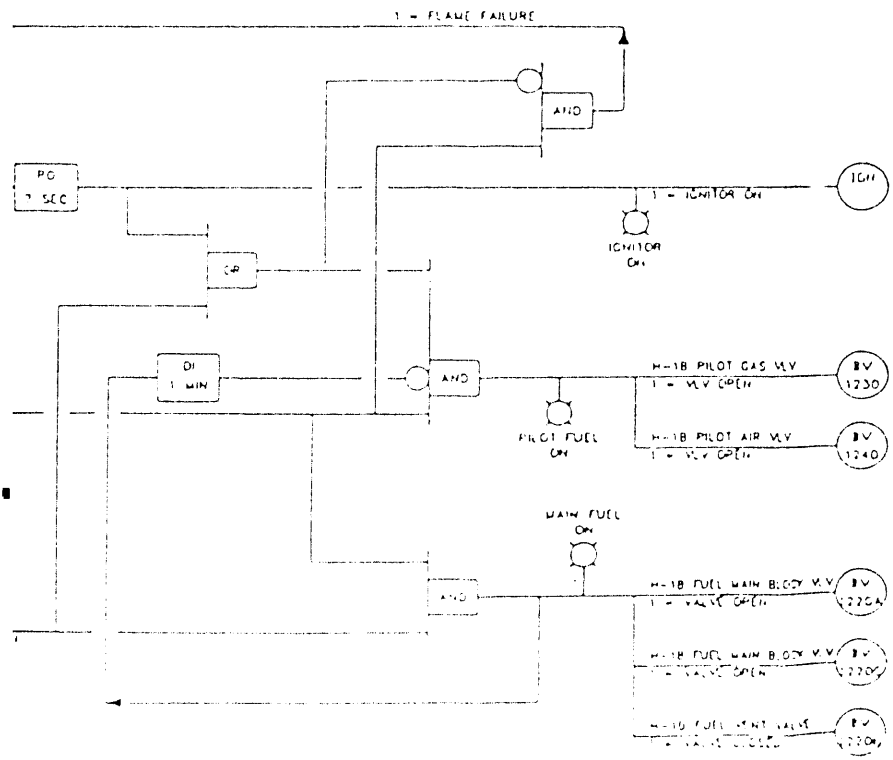
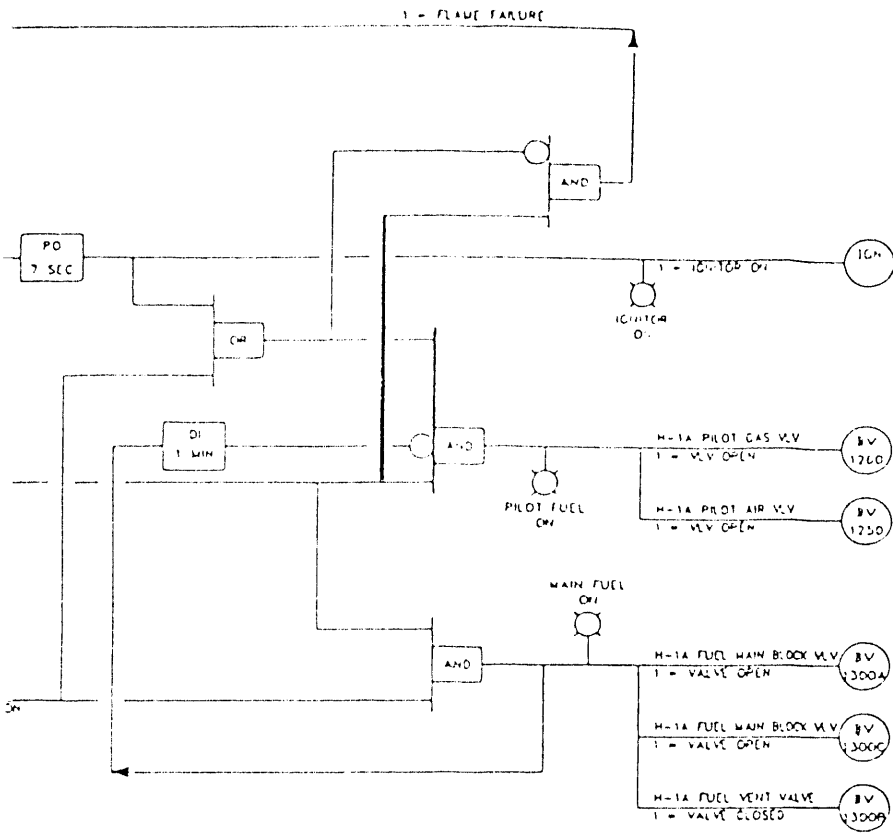
2 TPH BLACK LIQUOR GASIFIER  
 START-UP AND SHUTDOWN LOGIC  
 LOGIC DIAGRAM

SCALE	PROJECT NUMBER	DRAWING NUMBER	REV
NONE	458900	10042-L-001	D



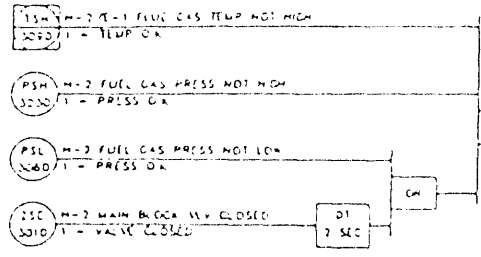
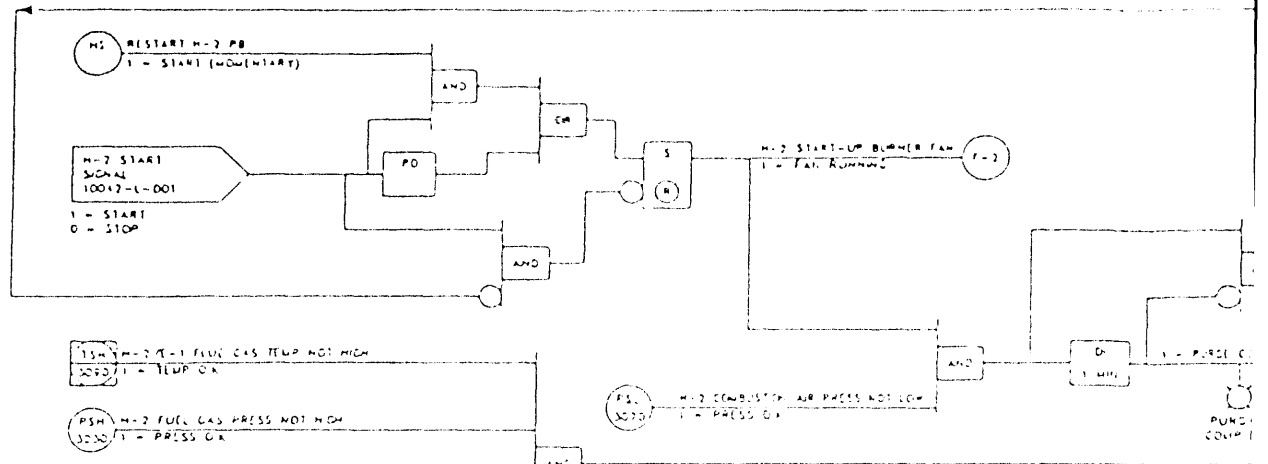
NOTE:

1 THIS DRAWING SHOWS LOGIC FOR BURNERS H-1A & H-1B.  
IT IS TYPICAL FOR BURNERS H-1C & H-1D. SEE DRAWING  
10042-P-001 SH1 FOR TAG NUMBERS OF ALL DEVICES AT  
ALL BURNERS

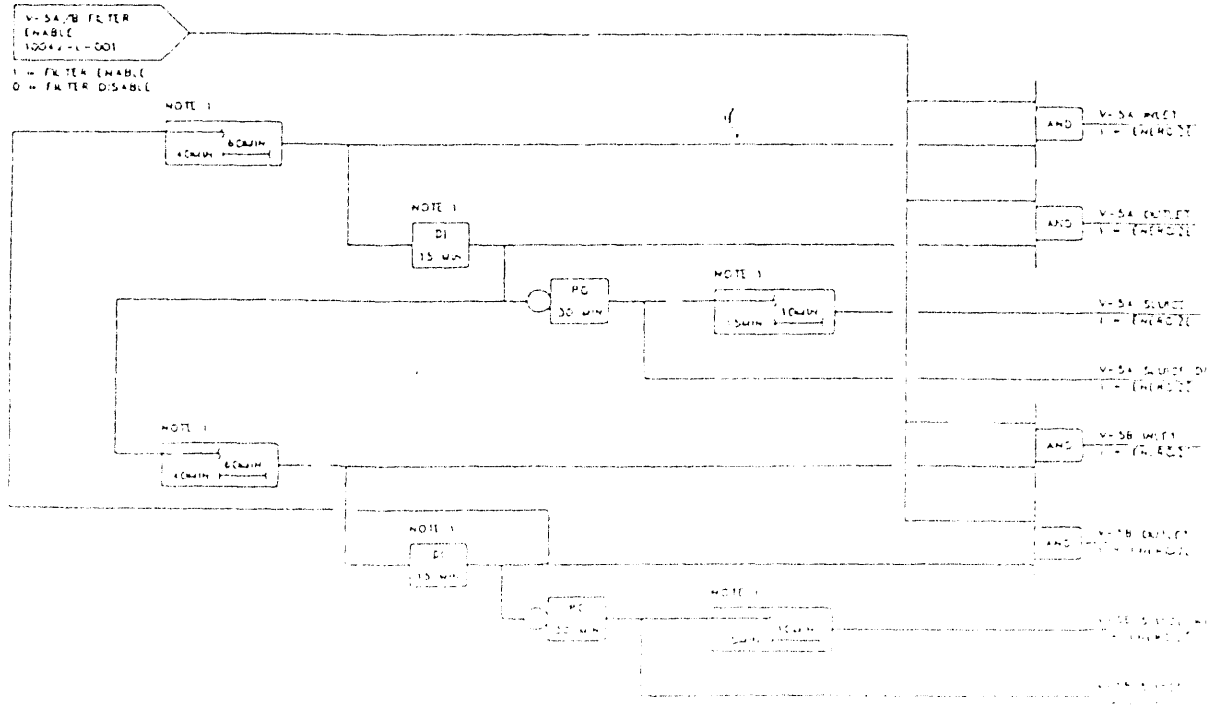
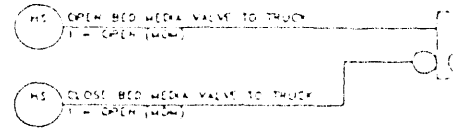
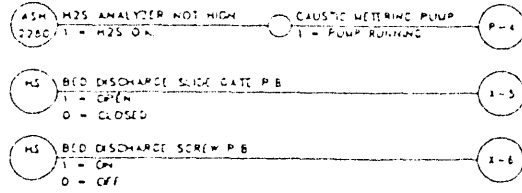


PREPARED BY		ESI	
ENGINEERING SERVICES, INC.		SANTA FE SPRING, CALIF. 90670	
PROJECT NO.		M T C I	
DATE		SANTA FE SPRING, CALIF. 90670	
2 TPH BLACK LIQUOR GASIFIER H-1A/H-1B BURNER LOGIC LOGIC DIAGRAM			
SCALE	PROJECT NUMBER	DRAWING NUMBER	REV.
100%	458900	10042-L-002	D

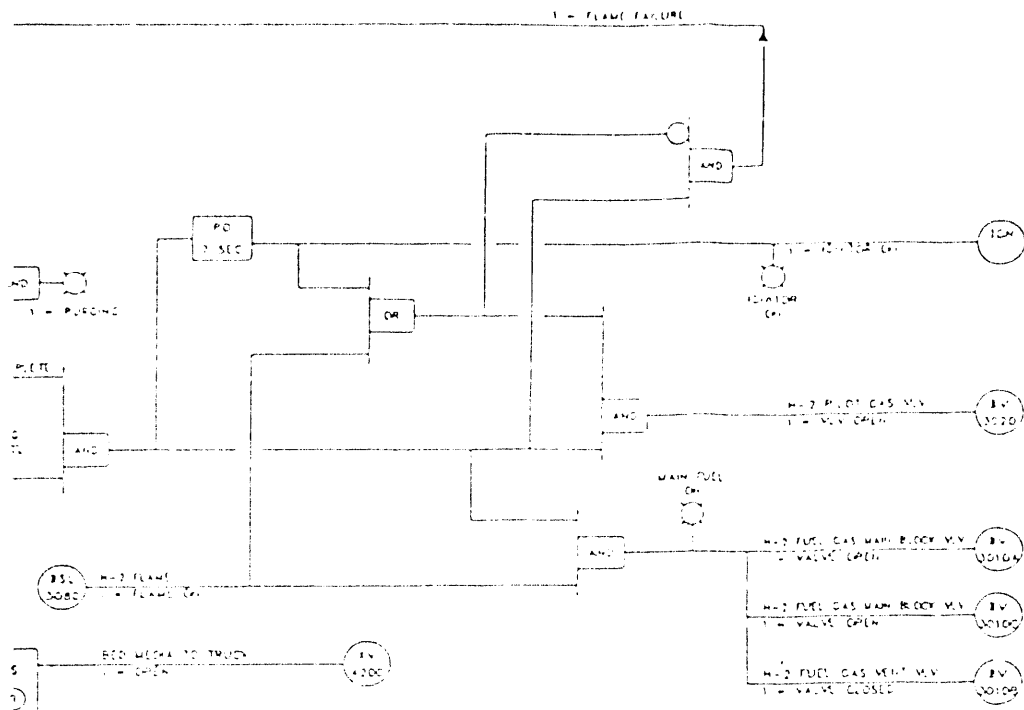
NO.	DESCRIPTION	DATE	BY	CHKD.	APPV.	DATE
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2	ISSUED FOR APPROVAL					
3	ISSUED FOR CONSTRUCTION					



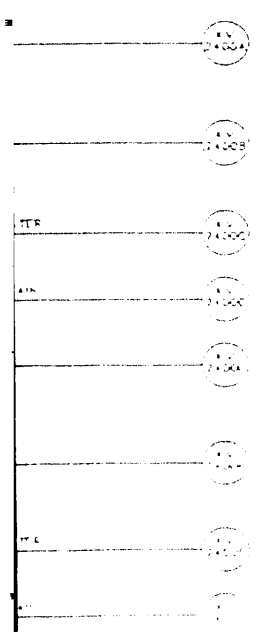
ADD DIFFERENTIAL







- NOTES:
1. TIMERS SHALL STOP TIMING WHEN PUMP P.B IS NOT RUNNING. TIMER OUTPUTS WILL BE HELD IN THEIR LAST STATE.
  2. TIMER SYMBOLOGY FOLLOWS ISA STANDARD 5.2
  3. BED MEDIA LOGIC TO BE DEFINED DURING DETAIL DESIGN TO INCORPORATE VENDOR EQUIPMENT.



**ESI**  
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 FAX: 303.733.1101  
 WWW: www.esi.com

**COMTEC**  
 CONTROL SYSTEMS TECHNOLOGY CENTER  
 1000 WEST 10TH AVENUE  
 DENVER, CO 80202  
 TEL: 303.733.1100  
 FAX: 303.733.1101  
 WWW: www.comtec.com

PROJECT NO. 1000-1000-1000  
 SHEET NO. 1000-1000-1000  
 TITLE: H2 GAS CONTROL LOGIC DIAGRAM

REV.	DATE	DESCRIPTION	DESIGNED BY	CHECKED BY	DATE
1					

**END**

**DATE  
FILMED**

**3 / 11 / 92**

