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BLACK LIQUOR GASIFICATION PHASE 2D FINAL REPORT

PREPARED FOR:

CHAMPION INTERNATIONAL STAMFORD, CONNECTICUT

Work performed under Subcontract STR/DOE-12 of Contract DE-AC05-80CS40341

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By

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I. SUMMARY

This report covers work conducted for Champion International by the Rocketdyne Division of Rockwell International under a subcontract to a Cooperative Agreement between Champion and the Department of Energy (DOE) on the subject of black liquor gasification. The work represents Phase 2D of a longrange program aimed at developing the Rockwell process for converting concentrated Kraft black liquor into low-Btu fuel gas and reduced melt.

Prior phases have included bench-scale and low-pressure, pilot-scale tests and the conceptual design of a pilot plant capable of operating at pressures up to 45 lb/in^2 (absolute). Concurrent with the earlier Rockwell studies, Champion International evaluated the process for application in pulp mills as a possible alternative to the conventional Tomlinson recovery furnace, and concluded that gasification offers many advantages. It was also concluded that pilot plant operation at a pressure higher than 45 lb/in^2 (absolute) would be desirable to reduce risks in the next step of scaling the process up to a full-scale demonstration unit operating at about 150 lb/in² (absolute).

A principal objective of the work covered by this report is to upgrade the pilot plant conceptual design to increase its operating pressure and capacity by a factor of approximately 2 (to 90 lb/in² (absolute) and 2068 lb/h black liquor feed, respectively). A second objective is to evaluate three types of nozzles previously selected as candidates for feeding black liquor into the gasifier.

The gasifier design was modified to make it capable of $90-1b/in^2$ (absolute) operation, and performance calculations were made for several highpressure cases. At the same time, the computer program for modeling gasifier performance was expanded to include the effect of venting a portion of the hot gas from the gasification zone into the quench tank with the product melt.

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The calculations showed that venting 10% of the gas has little effect on product gas quality, but does decrease the temperature at the top of the gasifier by about 68°F since less gas is cooled by the black liquor spray. Increasing the operating pressure from 45 to 90 lb/in² (absolute) while holding the gas velocity constant at 2 ft/s in the gasification zone doubles the gasifier capacity and causes the predicted heating value of the product gas to increase from 101 to 114 Btu/scf (dry basis). Increasing the solids concentration of the black liquor from 63 to 75% at 90-lb/in² (absolute) operating pressure further increases the predicted heating value of the product gas to 118 Btu/scf, but has little effect on black liquor solids gasification capacity.

Material balance calculations were completed showing flow rates and conditions in all major process lines for three reference conditions representing operation at 13.7, 45, and 90 lb/in^2 (absolute) in the gasifier. Operating envelopes were developed for the gasifier and quench tanks.

New engineering drawings were made to define the gasifier, gasifier refractory installation, quench tank, product gas separator, vent gas separator, and the equipment arrangement. The equipment list was revised in accordance with design changes and a pipeline list developed giving pipe sizes, schedules, and materials for all major lines.

Design guidelines were developed for the instrumentation and control system and a complete list of instrumentation requirements for the pilot plant was compiled.

A test program was conducted to evaluate three types of nozzles proposed for use in the pilot plant to spray black liquor through the drying zone of the gasifier. The three selected types were a narrow-angle, full-cone pressure nozzle, a two-fluid (pneumatic) nozzle, and a specially designed splashtube nozzle. The tests were conducted by spraying hot molten wax¹ through the nozzles at an elevation of about 11 ft above a catch basin flooded with a horizontal spray of cold water. The wax droplets solidify and are collected

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and characterized by sieve analysis and microscopic examination. The tests indicated that all three types of nozzles could be operated to provide black liquor droplets in the desired size range. The splash-tube and full-cone pressure nozzles are recommended for testing with black liquor in the pilot plant facility. The splash-tube concept is the preferred design primarily because it is less subject to plugging than the full-cone pressure nozzle (which contains internal vanes), and uses much less steam (or air) than the two-fluid nozzle.

It is concluded that the black liquor gasification pilot plant as now configured represents a well-integrated, cost-effective facility for demonstrating all critical operations of the process and providing data for the design of much larger units. Several available spray nozzle designs can be expected to provide effective spraying of black liquor feed in the pilot plant gasifier. One design has been selected that appears to offer both reliable pilot plant performance and scalability to much larger sizes.

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II. INTRODUCTION

This report covers work conducted by Rockwell International under Amendment 5 to Subcontract STR/DOE-12 of Cooperative Agreement DE-AC-05-80CS40341 between St. Regis Corporation (now Champion International) and the Department of Energy (DOE). The work has been designated Phase 2D of the overall program to differentiate it from prior work under the same subcontract.

The overall program is aimed at demonstrating the feasibility of and providing design data for the Rockwell process for gasifying Kraft black liquor. In this process, concentrated black liquor is converted into low-Btu fuel gas and reduced melt by reaction with air in a specially designed gasification reactor.

Phase 1 of the program was conducted under Subcontract STR/DOE-10 of the St. Regis/DOE cooperative agreement and consisted of nine bench-scale screening tests. This work is described in the Phase 1 final report prepared by St. Regis Corporation.¹ Phase 2 of the Rockwell activities initially included 23 bench-scale tests in a 6-in.-diameter gasifier and two runs in a 33-in.-ID pilot-scale unit. This work was extended to include Phase 2A, which consisted of modifying the pilot-scale unit and performing a third extended run.

The bench-scale tests and the three pilot-plant runs are described in detail in Rockwell reports on the subcontract activities^{2,3} and in the Champion International Phase 2 final report.⁴

In parallel with the Phase 2 experimental programs conducted by Rockwell, Champion International performed analytical studies to evaluate the black liquor gasification process as a possible alternative to the conventional Tomlinson recovery furnace. They developed detailed computer simulations of overall electric power and steam supply systems for pulp and paper mill applications. The study indicated that gasification has high potential and offers many advantages including:

- Higher energy efficiency than state-of-the-art conventional technology
- Higher output ratio of electrical power-to-steam, better matching mill demands
- Modularity, making it feasible to add incremental capacity to mills economically
- Significantly improved safety, eliminating the possibility of smeltwater explosions
- Significantly reduced air emissions
- Reduced capital and operating costs.

The Phase 2 bench-scale tests adequately demonstrated the basic process chemistry and correlated well with theoretical predictions. Product gas higher heating values (HHVs) (dry basis) ranged from about 120 to 140 Btu/scf depending on black liquor composition and other variables, and sulfur in the melt was generally over 90% in the form of sodium sulfide.

The three pilot-scale tests demonstrated the operability of the process in engineering-scale equipment and the predictability of gasifier performance. However, the results were adversely affected by a very high heat loss from the pilot-scale gasifier, an existing test unit originally designed for applications requiring a high rate of heat dissipation. The high heat loss resulted in the requirement to operate with a high air/black liquor feed ratio to maintain a constant temperature, and this caused both a low heating value gas and a poorly reduced melt to be produced.

It was concluded that additional pilot-plant tests would be desirable to provide an engineering-scale demonstration of the process, including the production of gas with a reasonably high heating value (over about 90 Btu/scf), the production of highly reduced melt, operation at elevated pressure, and melt discharge and quenching in a closed system. The next two phases of the program (Phases 2B and 2C) were aimed at developing the conceptual design of a pilot-scale gasification plant that would overcome the limitations of the existing facility and accomplish the above objectives. They also included specific tasks to evaluate refractory materials for use in the gasifier; develop the conceptual design of a melt discharge and quenching system for use in a pressurized environment; and study the requirements and potential techniques for spraying black liquor into the gasifier. The results of these activities are described in the Rockwell Phase 2B and 2C final reports^{5,6} and in a comprehensive report by Dr. E. Kelleher of Champion International.⁷

The pilot plant design resulting from the above studies was based on an operating pressure of 45 lb/in^2 (absolute), representing a factor of 3 increase over the previous atmospheric pressure tests. Subsequent to this design work, it was concluded that higher pressure pilot plant operation would be desirable to further reduce risks in scaling the process up to a demonstration plant operating at about 150 lb/in² (absolute).

A preliminary review of the pilot plant design and the capabilities of existing facilities was made to determine the maximum operating pressure at which meaningful tests could be conducted without a major increase in the cost and scope of the pilot plant program. Since the gasifier throughput is limited by gas velocity to avoid excessive entrainment, the design black liquor feed rate for a given size gasifier increases directly with pressure. The quantity of black liquor to be handled was, therefore, one factor in determining the optimum design pressure. Other factors were the flow and pressure capabilities of the existing air compressors and air heaters and the capacity of the existing product gas combustor. It was concluded that valuable high-pressure performance data could be obtained by operating the pilot plant for relatively short periods at a pressure of about 90 lb/in² (absolute) in conjunction with longer runs at lower pressure and feed rate conditions, and that this change would not require a major increase in scope of the pilot plant program.

A principal objective of this phase of the program (2D) is to upgrade the pilot plant conceptual design to increase both its operating pressure and throughput by a factor of approximately 2. A second objective is to evaluate spray nozzles proposed for feeding black liquor into the gasifier by conducting spray tests using molten wax to determine spray patterns and drop sizes.

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III. PILOT PLANT DESIGN

A. DESIGN OBJECTIVES AND GUIDELINES

The specific objective of the pilot plant design is to provide a facility that will demonstrate satisfactory operation and performance of the black liquor gasification process at pressures up to 90 lb/in² (absolute) and black liquor feed rates up to about 2000 lb/h, including the production of gas with a higher heating value (HHV) of about 100 Btu/scf (dry basis) and the production of melt in which over 90% of the sulfate is reduced to sulfide.

Additional objectives are to provide the capabilities for: (1) determining the effects of gasifier temperature, pressure, feed rate, air distribution pattern, melt pool depth, and black liquor composition; (2) evaluating the performance of components and materials; and (3) procuring data for the design of larger plants.

A key requirement of the design is that the new gasifier be compatible with existing auxiliary facilities but have the low heat loss and highpressure capabilities necessary for it to meet the specific objectives given above. Existing test facilities that will be incorporated into the overall pilot-plant system include the feed air compressors, feed air heater, product gas baghouse, product gas combustor, data acquisition and control center (DAC), and gas analysis instrumentation.

The plant must meet all safety and environmental requirements at the site; however, it may be designed for a relatively short operating life (~2 years). It is expected that this criterion will permit the use of low-cost construction materials such as carbon steel in the green liquor circuit and in other moderately corrosive environments of the pilot-plant system.

The following specific design criteria have been established for the pilot plant:

•	Internal gasifier dimensions:	~30-in. internal diameter by ~17-ft active height
•	Operating pressure capability:	90 lb/in ² (absolute) (~6 atm)
•	Gas velocity at maximum pressure:	~2 ft/s
•	Feed air temperature:	~700°F
•	Black liquor feed rate capability:	2068 lb/h
•	Black liquor solids concentration:	~63%
•	Black liquor feed temperature:	220°F
•	Pool depth (maximum):	24 in.
•	Air injection levels:	10 and 32 in.
•	Black liquor spray nozzles:	four feed points
٠	Heat loss from gasification zone:	<600 Btu/1b of feed
•	Melt quench medium:	green liquor
•	Melt shatter jets:	steam and recycled green liquor
•	Product gas heating value:	~100-Btu/scf HHV, dry basis, at maximum throughput.

B. PROCESS DESIGN

1. <u>Process Description</u>

A flowsheet of the overall pilot-plant gasification system is given in Figure 1. Concentrated black liquor will be delivered to the site by tank truck and pumped into black liquor tank V-5 for storage. This tank is equipped with a heater to maintain the temperature of the black liquor above approximately 200°F, an agitator to minimize settling or stratification, and a charcoal vent filter to prevent the release of odorous gases to the atmosphere. During operation of the system, a high volume of black liquor is

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continuously pumped out of the black liquor tank by pump P-1 through a full stream strainer and the black liquor recirculation heater H-5 and returned to the tank. This recycle system serves to provide a clean, well-mixed, and heated source of black liquor close to the gasifier vessel to supply the smallvolume gasifier feed stream.

The gasifier feed stream passes through a fine-screen strainer and a highpressure pump (P-4) before being injected into the top of the gasifier (V-1) through one to four spray nozzles. In the gasifier, the droplets of black liquor fall downward through a drying zone countercurrent to a rising stream of hot product gas. Gasification occurs at the bottom of the gasifier, where heated air reacts with the carbonaceous material in the black liquor under substoichiometric (partial oxidation) conditions. The partial oxidation reactions are exothermic and maintain the temperature in the gasification zone at about 1750°F. Inorganic salts in the black liquor melt and form a pool at the bottom of the gasifier, the depth of which is determined by the location of an overflow port.

Feed air for the gasifier is compressed in compressors C-1 and C-2 and heated to about 700°F in feed air heater H-2 before it enters the gasifier. A portion of the air is fed above the surface of the pool of melt to promote gasification reactions in this region, and the balance is fed directly into the melt pool to ensure that char entering the melt is oxidized.

After steady-state operation is attained, product melt continuously overflows from the pool and, together with a portion of the hot gas, flows to the quench tank (V-2). The melt is shattered by jets of steam and recycled green liquor as it falls into the pool of green liquor in the quench tank. The green liquor is maintained at a temperature of about 220°F when the system is at elevated pressure (or 180°F at atmospheric pressure) by the use of a heater (H-6) or cooler (H-8) in the green liquor recirculation line. Green liquor is removed from the quench tank through a strainer, which retains large undissolved particles of solidified melt, and a high volume pump (P-2). Most of the pump product is recirculated through the shatter jet nozzle, but a small amount, representing the net product, flows through a finer strainer to the green liquor storage tanks (V-6A and B).

A small fraction of the gas generated in the gasification zone is allowed to flow out of the gasifier with the melt to aid in maintaining a sufficiently high temperature in the melt discharge line. A gas burner, H-3, is also provided in this line as an auxiliary heat source. Gas which enters the quench tank with the melt stream is vented through a separator and joins the product gas stream after pressure reduction. The main stream of product gas, which is cooled from approximately 1750°F to less than 1000°F in the drying zone of the gasifier, leaves the top of the gasifier and passes through a cyclone separator to remove relatively large solid particles then through a pressure control valve, which reduces its pressure to near atmospheric.

After pressure reduction the product gas flows through a cooler, H-9, which reduces its temperature to less than 500°F. It then enters the baghouse, S-1, which removes very fine solid particles, primarily sodium carbonate fume. The cleaned product gas is finally burned with excess air in combustor, H-1, and the resulting flue gas is vented to the atmosphere.

2. <u>Performance Analysis</u>

The basic computer program used for the gasifier performance calculations was described in a previous report.² During the current phase of the program, the calculation procedure has been upgraded to incorporate the effects of venting about 10% of the high-temperature gas stream through the melt discharge line. The purpose of the vent gas is to aid in maintaining an elevated temperature over the flowing stream of melt.

Key assumptions used in the performance calculations are listed in Table 1.

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••	Black Liquor Com	position (wt.%)
	Reference Liquor	High-Solids Liquor
С	24.60	29.27
Н	2.37	2.82
0	20.32	24.18
S	2.63	3.13
Na	11.88	14.13
К	1.13	1.34
C1	0.06	0.07
Inerts	0.05	0.06
н ₂ 0	<u>36.96</u>	_25.00
	100.00	- 100.00
Solids, %	63.04	75.00
HHV, Btu/1b	4168	4959
Temperature i	n gasification zone, °F	1750
Temperature of	f feed air, °F	700
Temperature of	f feed liquor, °F	. 220
Drying efficie	ency in drying zone, %	90
Reduction effi	iciency of sulfur in mel	t,% 95
Temperature of gasification z	f dried solids entering zone, °F	220
Gasifier ID in	n gasification zone, ft	2.50
Heat loss from	n gasification zone, Btu	/h 294,500
Heat loss from	n drying zone, Btu/h	82,000
Total gasifier	wall-heat loss, Btu/h	376,500

Table 1. Assumptions for Gasifier Performance Calculations

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Several additional assumptions are required to calculate the cases with partial gas venting. It is assumed that 10% of the total gas volume leaving the gasification zone of the gasifier is allowed to flow through the melt discharge line and the remaining 90% flows upward through the drying zone. The gas leaving the gasification zone is assumed to be in equilibrium with regard to the water gas shift reaction at 1750°F, and the 10% vented gas, therefore, has a composition corresponding to this equilibrium. The 90% stream picks up additional water that is evaporated from the black liquor in the drying zone of the gasifier. For calculation purposes, this stream is assumed to re-equilibrate with the evaporated water at a temperature of 1750°F. Gas compositions measured during previous tests agree better with published equilibrium data when the gasification zone temperature rather than a lower drying zone temperature is assumed.

The results of gasifier performance calculations for seven typical cases are given in Table 2.

Cases A, B, and C represent the reference design conditions for operation at nominal pressures of 1, 3, and 6 atmospheres, respectively. The reference design cases are based on a superficial gas velocity in the gasifier of 3.0 ft/s when the gasifier is operated at one atm $(13.7 \text{ lb/in}^2 \text{ (absolute)})$ at the test site) and 2.0 ft/s when the nominal pressure is 3 or 6 atm. These gas velocities are considered conservative and are based primarily on consideration of entrainment of black liquor and melt droplets in the up-flowing gas stream. Case D is included to show the effect of increasing the allowable gas velocity from 2.0 to 2.5 ft/s at 90 lb/in² (absolute). This results in a 26% increase in feed capacity and a slight increase in product gas heating value.

Case E provides an indication of the effect of black liquor concentration on performance. For this case it is assumed that the black liquor has the very high solids concentration of 75% (versus 63% for the other cases). Comparing Case E with reference Case C shows that the increase in solids concentration reduces the black liquor feed capacity by 17% (but increases the

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		Pi	lot Pl	ant Ca	ses		Commercial Plant
	A	8	С	D	E	F	Design
Pressure, lb/in ² (absolute)	13.7	45.0	90.0	90.0	90.0	90.0	233
Solids. %	63.0	63.0	63.0	63.0	75.0	63.0	63.0
Black liquor feed rate. 1b/h	391	982	2068	2610	1776	2068	65,540
Gas velocity. ft/s*	3.00	2.00	2.00	2.50	2.00	2.00	2.00
Air. % of theoretical	56.4	45.2	41.4	40.7	40.8	41.4	38.9
Gas vented to guench tank, %	10	10	10	10	10	0	0
Material balance 1b/1b of black liquor							
Air	1.76	1.41	1.29	1.27	1.52	1.29	1.22
Gasifier outlet gas	2.27	1.95	1.85	1.83	1.99	2.01	1.94
Vent gas	0.21	0.18	0.17	0.17	0.20	-	-
Melt	0.28	0.28	0.28	0.28	0.33	0.28	0.28
Gasifier outlet gas							
Volume, scf/lb of black liquor	33.8	30.3	29.2	29.0	30.5	31.6	30.8
Temperature, °F	796	884	921	928	1200	989	1024
Water content, %	25.4	24.9	24.7	24.6	16.8	23.2	22.9
HHV, Btu/scf (dry)	70	101	113	115	117	113	121
Composition, % (dry basis)				-			
C0 ₂	16.4	15.4	15.0	14.9	12.9	14.7	14.4
ເວົ	7.2	10.3	11.5	11.8	14.4	12.0	12.9
CH ₄	1.1	1.7	1.8	1.9	1.9	1.8	1.9
C ₂ H ₄	0.15	0.17	0.18	0.18	0.18	0.18	0.18
H ₂	9.8	14.5	16.4	16.8	14.8	16.1	17.4
H ₂ S	0.14	0.16	0.17	0.17	0.17	0.17	0.17
N2	64.2	57.8	54.9	54.3	55.7	55.1	53.1
Vent gas							
Volume, scf/lb of black liquor	3.0	2.6	2.5	2.4	2.9		
Temperature, °F	1750	1750	1750	1750	1750		
Water content, %	9.5	7.9	7.2	7.1	6.3		
HHV, Btu/scf (dry)	72	106	120	123	123		
Composition, % (dry basis)							
^{C0} 2	12.0	10.3	9.2	9.0	8.5		
CO	11.6	17.0	19.2	19.6	20.2		
CH ₄	1.2	1.8	2.0	2.0	2.0		
с ₂ н ₄	0.16	0.18	0.19	0.19	0.19		
H ₂	6.1	9.3	10.6	10.9	10.5		•
H ₂ S	0.15	0.17	0.18	0.18	0.18		
N ₂	67.8	61.3	58.6	58.1	58.5		
Total product gas							
Volume, (dry) scf/lb of black liquor	27.9	25.2	24.3	24.1	28.1	24.3	23.8
HHV, Btu/scf (dry)	70	101	114	116	118	113	121

Table 2. Calculated Gasifier Performance

*Based on cross-sectional area of the gasification zone.

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black liquor <u>solids</u> feed capacity by 2%) and increases the product gas heating value by about 3.5%. Its main effect is on the temperature of the product gas which increases from 921 to 1200 because of the much smaller amount of water evaporated in the drying zone.

Case F is identical to reference Case C except that no gas is vented through the quench tank. The product gas quantity and heating value are not changed significantly; however, the gasifier outlet gas discharged from the top of the gasifier is about 68°F hotter because there is about 10% more to be cooled by the black liquor spray.

A commercial plant design case is included for comparison with the projected pilot plant results. This case does not include the venting of part of the product gas through the quench tank and, therefore, can best be compared with pilot plant Case F. The commercial plant is projected to produce slightly less gas/lb of black liquor feed, but the gas will have a higher heating value (121 Btu/scf versus 113 Btu/scf). This effect is due entirely to the lower relative heat loss of the commercial size plant.

Flow rates, stream characteristics, temperatures, and pressures in key process lines are given in Table 3 for Cases A, B, and C. These cases represent: A - operation at 13.7 lb/in^2 (absolute) and 3 ft/s superficial gas velocity, B - operation at 45 lb/in² (absolute) and 2 ft/s, and C - operation at 90.0 lb/in² (absolute) and 2 ft/s. Case C represents the reference design conditions for the gasifier; however, the other cases are included as they may impose special requirements on the pilot plant systems.

3. <u>Operating Envelope</u>

Figures 2, 3, and 4 define the normal operating envelopes and predicted performance for the pilot plant. The figures are based on the assumptions for gasifier performance calculations given in Table 2. The points marked A, B, and C on the charts represent the three cases described above.

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Line number Line designation Case*	A	101 Air Feed B	C	Gasif A	102 Tier Top Ou B	tlet C	Bagh A	103 iouse Out B
Temperature, °F Pressure, 1b/in ² (absolute)	700 15.7	700 47.0	700 92.0	796 13.7	884 45.0	921 90.0	300 13.5	300 14.0
Liquid phase Nag S, 1b/h Nag CO3, 1b/h Other salts, 1b/h Total salts, 1b/h Dissolved solids, 1b/h Liquid water, 1b/h Total liquid, 1b/h Density, 1b/ft ³ GPM					`			, , , , , , , , , , , , , , , , , , ,
Gas phase Dry gas, 1b/h	689	1387	2674	728	1567	3114	885	1812
Water vapor, 1b/h Total gas, 1b/h	- 689	-		159 887	353 1920	707 3822	254 1139	439
SCFM (dry)	150.1	302.2	582.5	164.0	372.0	818.2	197.2	426.6
ACFM	313.5	210.9	207.8	609.3	495.6	435.9	455.5	891.0
Density (STP), 1b/ft ³ Density, actual, 1b/ft ³	0.0765	0.0765 0.1096	0.0765 0.2145	0.0673	0.0646 0.0765	0.0633 0.1459	0.0663	0.0646
Mol. wt (wet) HHV (dry), Btu/scf	29.0	29.0	29.0	25.5	24.5	24.0	25.1	24.5
Line number		205			205			207
Line designation Case*	•	Wall Wash B	C	Green A	n Liquor Pr B	oduct C	Green A	n Liquor (B
Temperature, °F Pressure, 1b/in ² (absolute)	180 75	220 105	220 150	180 ~20	220 ~20	220 ~20	180 13.7	220 45.0
Liquid phase								
Na ₂ S, 1b/h Na ₂ CO ₂ , 1b/h	286 1065			20.7	52.1 193.4	109.6 407.4	1643 6112	1674 6228
Other salts, 1b/h	147	A 2	s A	10.6	26.5	55.8	842	858
Dissolved solids, 1b/h	1498	a and	sme a	108.3	272.0	572.8	8596	8760
Liquid water, 1b/h Total liquid, 1b/h	8488 9986	ŭ	ιö	613.7	1541.3 1813.3	3245.9 3818.7	48/12 57308	49639 58399
Density, 1b/ft ³ GPM	69.3 18.0			69.3 1.30	69.3 3.27	69.3 6.88	69.3 103.3	69.3 105.3
Gas phase Dry gas, lb/h Water vapor, lb/h								
Total gas, lb/h SCFM (dry) SCFM (wet) ACFM								
Density, actual, 1b/ft ³ Mol. wt (wet) HHV (dry), Btu/scf								

*Key operating conditions: A – Atmospheric pressure operation, superficial velocity 3.0 ft/s in gasifier, 391 lb/h B.L. feed B – 45 lb/in² (absolute) operation, superficial velocity 2.0 ft/s in gasifier, 982 lb/h B.L. feed C – 90 lb/in² (absolute) operation, superficial velocity 2.0 ft/s in gasifier, 2,068 lb/h B.L. feed

Table 3. Black Liquor Gasification Pilot Plant Flow Rates for Three Operating Cases

let Gas C	Gasifi A	104 er Bottom B	Outlet C	A	201 Burner Gas B	c	Quench A	202 Tank Melt B	Inlet • C	A	203 Makeup Wat B	er C	Recyc	204 le Green L B	iquor C
300 14.0	1750 13.7	1750 45.0	1750 90.0	2850 13.7	2850 45.0	case.	1750 13.7	1750 45.0	1750 90.0	60 ~150	60 ~150	60 ~150	180 75	220 105	220 150
	20.7 77.0 10.6 108.3 - - 108.3 120.4 0.112	52.1 193.4 26.5 272.0 - 272.0 120.4 0.282	109.6 407.4 55.8 572.8 572.8 572.8 120.4 0.593			Burner not used for this	20.7 77.0 10.6 108.3 - 108.3 120.4 0.112	52.1 193.4 26.5 272.0 272.0 120.4 0.282	109.6 407.4 55.8 572.8 572.8 120.4 0.593	- - - 645.2 646.2 62.4 1.29	- - - 1560.3 1560.3 62.4 3.12	- - 3227.9 3227.9 62.4 6.46	1336 4970 684 6990 39610 46600 69.3 84.0	Same as A	Same as A
3445 756 4201 896.9 1100.7 1689.2 0.0636 0.0415 24.1 114	79.0 5.1 84.1 17.5 19.3 88.0 0.0725 0.0159 27.5 72	166.9 9.8 176.7 38.9 42.3 58.7 0.0696 0.0502 26.4 106	330.9 16.5 347.4 78.7 84.8 58.9 0.0685 0.0987 26.0 120	78.0 7.0 85.0 15.7 18.1 123.6 0.0780 0.0114 29.6 -	78.0 7.0 85.0 15.7 18.1 37.6 0.0780 0.0375 29.6 -		157.0 12.1 169.1 33.2 37.4 211.6 0.0753 0.0133 28.5 36	244.9 16.8 261.7 54.6 60.4 96.3 0.0722 0.0452 27.4 72	330.9 16.5 347.4 78.7 84.8 58.9 0.0685 0.0987 26.0 120						
								<u> </u>							
Pump Feed	Que A	208 ench Tank V B	/ent C	A	209 Shatter Ste B	eam C	Black Li A	301 quor Tank B	Discharge C	B1	302 ack Liquor B	Return C	Bla A	303 Ick Liquor B	Feed C
Pump Feed C 220 90.0	A 180 14_0	208 ench Tank V B 220 14.0	/ent C 220 14.0	A 366 165	209 Shatter Sta B 366 165	eam C 366 165	Black Li A 220 25	301 quor Tank B 220 25	Discharge C 220 25	B1 A 220 20	302 ack Liquor B 220 20	Return C 220 20	B1a A 220 55	303 ick Liquor B 220 85	Feed C 220 130
Pump Feed C 220 90.0 1732 6442 887 9061 9061 51344 60405 69.3 108.8	Que A 180 14.0	208 ench Tank V B 220 14.0	Vent C 220 14.0	A 366 165	209 Shatter Sto B 366 165	2am C 366 165	Black Li A 220 25 - 40967 24018 64985 81.1 100	301 quor Tank B 220 25	Discharge C 220 25	B1 A 220 20 - - - - - - - - - - - - - - - -	302 ack Liquor B 220 20 - - - 40348 23655 64003 81.1 98.5	Return C 220 20 39663 23254 62917 81.1 96.8	Bla A 220 55 - - - 246 145 391 81.1 0.602	303 ick Liquor B 220 85 - - - 619 363 982 81.1 1.51	Feed C 220 130 - - - 1304 764 2058 81.1 3.18

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Figure 4. Quench Tank Operating Envelope

Figures 2 and 3 are intended as guides to gasifier operation. Figure 2 indicates the calculated air rate required for steady-state operation at any given black liquor feed rate and the predicted product gas HHV. The HHV values are for the gas as it leaves the gasifier prior to mixing with vent gas from the quench tank. The air feed rate is also based on the gasifier alone and does not include air required to operate the burner in the melt discharge line. The chart indicates that it should be possible to produce gas with a heating value over 100 Btu/scf by operating with a black liquor feed rate higher than about 1000 lb/h, but the product gas heating value falls off very rapidly if the black liquor feed rate is reduced below about 500 lb/h.

Figure 3 represents the gasifier operating envelope. Operation anywhere within the indicated boundaries is acceptable and can be expected to result in a product gas with the heating value given on the horizontal lines. The boundaries represent normal operating criteria and not physical limitations of the equipment. The gas velocity upper limit of 2 ft/s at 90 lb/in² (absolute) or 3 ft/s at atmospheric pressure is quite conservative and may be exceeded

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for special tests. Similarly, the high-pressure limit of 90 lb/in² (absolute) (~76 lb/in² [gage] at the test site) is well below the pressure rating of the gasifier/quench system (150 lb/in² [gage]) so higher pressure operation is a possibility. However, it is believed that all test objectives can be met by successfully operating the pilot plant within the indicated envelope.

Figure 4 shows the operating envelope for the quench tank. A maximum temperature of 220°F has been selected to minimize the corrosion of carbon steel. Since this temperature is above the boiling point of green liquor at atmospheric pressure (\sim 215°F), the high-temperature limit is reduced to 180°F at atmospheric pressure. It is expected that some boiling will occur where the melt enters the pool of green liquor, but the 35°F subcooling of the bulk of the solution will prevent excessive steam evolution and release with the vent gas.

C. EQUIPMENT

1. Principal Items of Equipment

A listing and brief description of all major process equipment items identified on the Process Flow Diagram (Figure 1) is given in Table 4. The list also indicates the proposed source of the items as follows:

- 1. Special equipment to be designed and fabricated. This category includes the gasifier, quench tank with melt discharge line and other accessories, product gas and vent gas separators, and the product gas cooler, which will be fabricated from standard pipe. Conceptual design drawings have been prepared on these equipment items.
- 2. Standard equipment to be purchased. This category includes relatively standard items such as pumps, strainers, heat exchangers, burners, and agitators, which will require procurement for the pilot plant. This category also includes two low-pressure tanks, the black liquor tank, and spray nozzle test tank, which will require some design work before procurement.
- 3. Existing Rockwell equipment. This category consists primarily of those portions of the existing molten salt test facility which will be incorporated into the pilot plant. The key items AI-DOE-13566

Item	Description	Source*
Vessels and Tan <u>ks</u>		
V-1 Gasifier	Vertical cylindrical tank, 5 ft 7-1/2 in. OD x 24 ft 6 in. total height. Carbon steel shell, refrac- tory lining, design conditions 150 lb/in ² (gage), 650°F (shell)	1
V-2 Quench tank	Horizontal cylindrical tank, 5 ft 6 in. OD x 11 ft 6 in. total length with vertical extension 4 ft OD x ~8 ft height. Carbon steel, design conditions 150 1b/in ² (gage), 650°F	1
V-3 Product gas separator	Cyclonic separator, 24 in. OD x ~86 in. total height, 304 stain- less steel, design conditions 150 lb/in ² (gage), 650°F	1
V-4 Vent gas separator	Cyclonic separator, 12 in. Sched- ule 40 pipe body. ~48 in. total length. Carbon steel, design conditions 150 lb/in ² (gage), 650°F	1
V-5 Black liquor tank	9000 gal, 10 ft diam x 10 ft high, carbon steel, design conditions: 50 lb/in ² (gage), 250°F	2
V-6 Green liquor storage tanks A&B	2 each, 17,500 gal, 12 ft diam x 21 ft high, carbon steel, atmo- spheric pressure	3
V-7 Spray nozzle test tank	3 ft diam x 10 ft high, 15 lb/in ² (gage), 250°F, with view port, carbon steel	2
Pumps		
P-1 Black liquor recirculation pump	100 gal/min, 75-ft head, 5 hp, 480 V, 3 phase, 1750 r/min, double mechanica seal, carbon steel	2 1
P-2 Greén liquor recirculation pump	100 gal/min, 170-ft head, 10 hp, 480 3 phase, 1750 r/min, double mechanica seal, carbon steel	V, 2 1
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Table 4. Equipment List (Sheet 1 of 4)

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Item	Description	Source*
P-3 Black liquor return pump	20 gal/min, 80-ft head, 1-1/2 hp, 480 V, 3 phase, 1750 r/min, single internal mechanical seal, carbon steel	2
P-4 Black liquor feed pump	5 gal/min, 300-ft head, (175 lb/in ²) ~2 hp, 480 V, 3 phase, 304 stain- less steel rotary pump, variable speed drive	2
Compressors and Blowers		
C-1 Main feed air compressor	Joy Twistair, 470 cfm at 100 lb/in ² (gage), 100-hp electric motor drive	3
C-2 Back-up feed air compressor	Ingersol-Rand vane type compressor, 365 cfm at 100 lb/in ² (gage), diesel engine drive	3
C-3 Combustor air fan	North American Turbo Blower, 2332- 21-2-30, 2000 cfm at 2 lb/in ² (gage), 30 hp	3
Filters and Strainers		
S-1 Baghouse	MikroPul Model 495-8-20, stainless with 3-in. insulation, Nomex 14-oz felt bags, 500 ft ²	4
S-2 Black liquor loop strainer	Rotary strainer, Dorr Oliver, 4 in., 9 r/min, 1/4 hp	2
S-3 Black liquor feed strainer	Rotary strainer, Dorr Oliver, 2 in., 16 r/min, 1/8 hp	2
S-4 Black liquor unloading strainer	In-line 3-in. pipe strainer, Y-type, coarse screen	2
S-5 Green liquor product strainer	In-line l-in. pipe strainer, Y-type	2
S-6 Quench tank strainer	Circulation pump inlet strainer, coarse screen, in tank	2

Table 4. Equipment List (Sheet 2 of 4)

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	Item	Description	Source*
S-7	Black liquor tank vent filter	Activated carbon, replaceable canister type, for odor removal, l-in. pipe connections, l-atm pressure	2
S-8	Nozzle test tank vent filter	Activated carbon, replaceable canister type, for odor removal, l-in. pipe connections, l-atm pressure	2
S-9 A&B Heat	Green liquor storage tank vent filters	Activated carbon, replaceable canister type, for odor removal, l-in. pipe connections, l-atm pressure	2
neat	ers, coorers, and burners		
H-1	Product gas combustor	Custom J. T. Thorpe, Inc., rated 600-scfm product gas at 150 Btu/scf, inlet temperature 1600 to 2000°F	3
H-2	Feed air heater	180 kW, 480 V, 3 phase, in-line with SCR controller, 110 lb/in2 (gage), at 1100°F and 15 lb/in2 (gage) at 1500°F, ASME Section VIII, Hynes Electric Heating Company	3
H-3	Melt discharge line burner	75,000 Btu/h, natural gas, 2000% excess air to 30% excess fuel, 85-lb/in2 (gage) operating pressure	2
H-4	Black liquor tank heater	40 kW, 480 V, 3 phase, in tank heater with SCR controller, 0 to 250°F, 8-in. A300 flanges	2
H–5	Black liquor recirculation heater	40 kW, 480 V, 3 phase, in-line heater with SCR controller, 0 to 250°F, 2-in. A300 flanges	2
H-6	Green liquor heater	15 kW, 480 V, 3 phase, in-line heater with SCR controller, 0 to 250°F, 2 in., A300 flanges	2
H–7	Vessel preheat burner A	75,000 Btu/h, natural gas, 2000% excess air to 30% excess fuel, 85-1b/in2 (gage) operating pressure	2

Table 4. Equipment List (Sheet 3 of 4)

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Item	Description	Source*
H-8 Green liquor cooler	Shell and tube, stainless tubes, 150,000-Btu/h cooling, 70° cooling water	2
H-9 Product gas cooler	6-15 ft lengths of 8 in. Schedule 20 carbon steel pipe, spaced 2 ft apart in vertical parallel arrangement, with interconnecting fittings, bypass, and drains	1
H-10 Vessel preheat burner B	~1 million Btu/h oil burner, maximum capacity 72 kg/h (159 lb/h), 20,000 Btu/lb oil, normal operation 18 kg/h (40 lb/h) oil and 800 lb/h air (50% excess)	2
Agitators		
A-1 Black liquor tank agitator	6 hp, 300 r/min, dual-propeller stainless with double mechanical seal, 8 in., Al50 flange top entry, 50-lb/in ² (gage) operation	· 2
A-2 Quench tank agitator	6 hp, 300 r/min, dual-propeller stainless with double mechanical seal, 8 in., Al50 flange top entry, 150–1b/in ² (gage) operation	2
<u>Special Hardware</u>	· · · · · ·	
Z-1 Soot blower	Nitrogen or steam feed, pneumatic drive reciprocating hollow shaft, l in. OD, 9-ft thrust, local control	4

Table 4. Equipment List (Sheet 4 of 4)

*Equipment Sources:

 Special equipment, to be designed and fabricated
Standard equipment, to be purchased
Existing Rockwell equipment, relocation, refurbishing, and installation may be required

4 - Existing DOE equipment, requires DOE approval for use. Relocation, refurbishing, and installation required.

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are the two feed air compressors, the feed air heater, and the product gas combustor, including its air fan. Two existing large tanks have also been earmarked for use in the pilot plant as storage tanks for green liquor. These tanks will require relocation. The list does not cover instrumentation and control items, which represent an important part of the existing facility to be used in the pilot plant. These are described in a later section.

4. Existing DOE equipment. At least two items of equipment currently installed on an inactive DOE-owned facility adjacent to the pilot plant site can be effectively used in the black liquor gasification pilot plant. These are the baghouse and soot blower. The latter was designed specially for operation in a pressurized gasifier product gas outlet line.

2. Gasifier

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The gasifier represents the key element of the process, and considerable effort has gone into its design. As presently configured, the gasifier is a carbon steel pressure vessel, 5 ft 7-1/2 in. OD and about 24 ft 6 in. total height. The walls are lined with a 2-in. layer of bubble alumina insulating refractory, an 8-in. layer of dense alumina refractory, and, in the bottom portion, an 8-in. layer of fusion cast alumina blocks. The selected refractories are manufactured by the Carborundum Company and are designated Alfrax B1, Alfrax 66, and Monofrax M, respectively. The gasifier is shown in Figure 5 (engineering drawing MOO5-68964-M3) and the refractory configuration within the vessel is shown in Figure 6 (engineering drawing MOO5-68964-M4).

The basic configuration and functions of the gasifier are unchanged from the previous design, which has been described in detail in prior reports.^{5,7} The major changes which have been made are represented in the following:

- The design pressure has been increased to 150 lb/in² (gage) 1. (for operation at 90 lb/in² (absolute) instead of 45 lb/in² (absolute)).
- The lower melt discharge nozzle has been deleted. 2.




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- 3. The air feed nozzles have been relocated and increased in size.
- 4. New nozzles have been added for preheat burners (2), drain, and pressure relief.
- 5. Thermowell nozzles have been relocated.
- Refractory installation details have been developed for all nozzles.

In the previous design, two melt discharge nozzles were provided at different heights so that the effect of melt depth could be evaluated. However, use of the second discharge nozzle would require rotating the gasifier vessel 180 deg and raising (or lowering) the quench tank subsystem to match the new nozzle height. In the present design, the effect of melt depth can be evaluated by installing a layer of filler blocks on the gasifier floor, a much simpler and more economical procedure. Hot product gas leaves the top of the gasifier at temperatures up to 921°F during normal operation at reference design conditions; however, it is estimated that gas temperatures as high as 1200°F may occur during off-normal operating periods. The gasifier outlet line is, therefore, refractory lined. The product gas flows into the separator shown in Figure 7 (drawing MOO5-6894-M5). Because of the high operating temperature, the material specified for this unit is Type 304 or 316 stainless Design conditions for the separator are 1200°F and 150 lb/in² (gage). steel. A 6-in. diameter stainless steel liner is provided in the 8-in. gas entry nozzle of the separator to minimize thermal effects at this point. It is anticipated that the product gas will cool approximately 100°F while proceeding through the uninsulated separator vessel.

3. <u>Quench System</u>

The melt discharge line and quench tank designs have been modified to accommodate the higher operating pressure and the new fixed elevation of the melt discharge nozzle. They are shown in Figures 8 and 9 (drawings M005-68984-M6 and 174000003, respectively). One significant change in the quench tank design is the addition of a stainless steel liner in the vertical extension of the main tank. The liner is spaced about 2 in. from the vessel wall and is designed to protect the portion of the wall that is directly in line with the melt discharge port from direct contact with high temperature melt and gases. Both the liner and unlined portions of the vertical extension will be continuously washed with recycled green liquor during operation to provide additional protection.

Gas entering the quench tank will be cooled and washed by contact with the wall wash liquid and the shatter jet spray. The gas will flow from the quench tank to a small separator, which will remove entrained droplets of green liquor or other particles. The design of this separator is shown in Figure 10 (drawing M005-68964-M8).

It is planned that pressure vessels in the pilot plant will be designed and fabricated in accordance with the ASME Code, Section VIII, which requires that corrosion be considered in the design. The quench tank and vent gas separator will be contacted by green liquor, water, and gas during plant operation. Hot green liquor is believed to represent the most corrosive medium that will be encountered. The current maximum design operating temperature is 220°F. The green liquor temperature can be prevented from exceeding 220°F (or reduced to a lower value if desired) by operation of the green liquor cooler, injection of more makeup water, or reducing the flow of melt and/or hot gas from the gasifier. The vent gas separator is expected to operate at the same temperature as the quench tank; however, it can be cooled independently by the injection of makeup water through a nozzle provided at the gas inlet port.

The corrosion of metals in green liquor was evaluated by Rocketdyne in a previous program.⁸ Although the green liquor used was not identical to that which will be encountered in the black liquor pilot plant program, it was quite similar. The reported test results for carbon steel and Type 304 stainless steel are given in Table 5. To estimate the corrosion rate at 220°F, the carbon steel data are plotted on semilog paper in Figure 11.



	NOZZLE SCHEDULE									
Б	DESCRIPTION	БÆ	TYPE RATHER		en	REHARKS				
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z	645 OUTLET	8"		1						
з	ENTRY NOZZLE	8-		-			1			
4	SOLIDS DISCHARGE	6.					1			
5	CLEANOUT PORT	4-	1	1			1			

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Figure 7.

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NOZZLE SCHEDULE											
ŝ	DESCRIPTION	SIZE	TYPE	erring	ĢТ	REMARKS					
1	GAS OUTLET	4"	SLIP-DA	150	1						
2	INLET	3.	5-10-04	150	1						
3	DRAIN	2.	SLIP-ON	150	1	22					
4	LEVEL MIDICATOR	11	NPT	3000	2	a, b (courcing)					

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CONCEPTUAL DRAWING -

10.000 6 0 mm FACILITIES ENGINEERING AS BUILT DRAWING REQUIRED SSFL **BLDG 005** Figure 10. BLACK LIQUOR GASIFICATION PILOT PLANT VENT GAS SEPARATOR R HOOD **11** .... 4-20-88 -----JAARDY M005-68964-MS 0

### FILE Nº 303-005-M78

	Carbon	Steel	Type 304 Stat	inless Steel				
	175°F	350°F	175°F	350°F				
Days	• -	Corrosion Rate	(mils per year)					
9.5	2.1	271.6, 250.0	<0.1	8.7, 2.0				
18.5	4.4	-	0	-				
29.5	-	80.8, 84.0	-	2.8, 1.0				
32.5	11.5	-	0	-				
55.0	-	45.0	-	<1.0				
78.5	9.3	-	<0.1	-				
Average	6.8	146.3	<0.1	3.0				
	Арр	earance of Stres	arance of Stressed U-Bend Samples					
	No cracking after 48 days	No cracking after 34 days	No cracking after 78 days	Cracked after 29.5 days				
	<u>Test Conditions</u>	Liquo	or Composition (T	ypical)				
Pres	sure: Atmospher	ic Na ₂	CO ₃ 21.2%	(wt.)				
Agit	ation: None	Na ₂	S 3.5					
Aera	tion: Atmospher	ic air Car	bon 0.5					
		Ash	5.6					
		Wat	er Balanc	Balance				
		рН	11.5 t	:0 12				

Table 5. Corrosion of Metals in Green Liquor*

*Data of Reference 8.

The results indicated a maximum corrosion rate of 26 mils per year (MPY) for carbon steel at 220°F and less than 1 MPY for Type 304 stainless steel at 220°F.

The currently planned pilot plant program includes five runs. If it is assumed that high-temperature green liquor is in the system for 7 days during each run, the total hours of exposure will be  $5 \times 7 \times 24 = 840$  h. Using a factor of safety of 10 on the exposure time (i.e., 8,400 h) and the maximum indicated corrosion rate of 26 MPY, the total indicated depth of corrosion is 0.025 in.



Steel in Green Liquor

This is an acceptable amount of corrosion for the pilot plant equipment, and carbon steel has, therefore, been selected as the material of construction for the quench tank and vent gas separator vessels. It is recommended that a corrosion allowance of at least 1/8 in. be added to the wall thickness. To minimize corrosion between runs, it is also recommended that the vessels be drained and filled with inert gas, or preferably drained, rinsed, and thoroughly dried after each run and that the wall thickness be monitored periodically during the period of test operations by appropriate means.

Stainless steel is probably an acceptable alternative to carbon steel, but additional data are needed to prove that it is not susceptible to stress corrosion cracking in green liquor at 220°F.

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### 4. Equipment Arrangement

The pilot plant will be located in and adjacent to Building 005 at Rockwell International's Santa Susana Field Laboratory. The general arrangement of equipment is shown in Figures 12 and 13 (drawings M005-68964-M1 and M005-68964-M2) representing plan and elevation views, respectively.

The gasifier will replace an existing refractory-lined vessel in the high bay of Building 005 and use the same support structure and platforms to the extent feasible. The quench tank will be located in a new pit to be excavated in the floor of the building adjacent to the gasifier support structure.

Product gas and vent gas separators will be located inside the high bay close to the gasifier and quench tank vessels, respectively. The existing feed air heater and the control room with its data acquisition and control (DAC) equipment are also inside Building 005. All other major facility and equipment items are located outside.

Gas leaving the product gas separator at temperatures up to about 1100°F flows into an 8-in. stainless steel pipe, which passes through a penetration in the building wall en route to the pressure control valve. Approximately 50 ft of uninsulated stainless steel line is provided to cool the gas to 800°F or less before it enters the control valve to minimize problems in procuring and operating this valve. The high-temperature, high-pressure stainless steel line is supported on the outside wall of the building at an elevation of about 20 ft above grade.

The gas pressure is reduced from as high as 90 lb/in² (absolute) to approximately atmospheric in the pressure control valve. After passing through the valve, the gas flows through low-pressure carbon steel pipe and passes through the product gas cooler, baghouse, and product gas combustor in series.

As shown in the elevation drawing, the gas cooler consists simply of six vertical passes of 8-in. carbon steel pipe, totaling about 100 ft in length, which will lose heat by convection and radiation to the atmosphere. Control is provided by a bypass line and valve.

The baghouse will be installed on an existing structural support system about 11 ft above grade. This will permit collected dust to be discharged readily into a drum or other vessel. The product gas combustor (incinerator) and its blower are currently in place at the location shown. The combustor installation is complete with regard to controls and utilities; however, refurbishment will be required prior to use for product gas combustion.

The black liquor feed system and green liquor handling system are not shown on the plan and elevation drawings because the optimum location for the required tankage has not yet been established.

## D. PIPING

Table 6 presents a preliminary list of the main pipe lines in the black liquor gasification pilot plant. The line numbers are identified in the process flow diagram, Figure 1.

Pipe diameters given in Table 6 are based on a consideration of published correlations for economic pipe diameters⁹ adjusted to take into account special requirements of the pilot plant. Thus, a 2-in. pipe is selected for the black liquor recirculation loop to give a flow velocity of about 10 ft/s and provide good mixing while 3/4-in. pipe (or 3/4-in. ID tubing) is specified for the black liquor feed line to avoid plugging problems although the resulting flow velocity is only about 2 ft/s. Green liquor and water pipe sizes are selected to give flow velocities of about 7 ft/s; while pipes carrying gases are sized to give maximum velocities ranging from about 50 ft/s for high-pressure gas to about 130 ft/s for low-pressure gas.









_	Operating Conditions				Flow Rat	e	Exten	t of Line	Li	ne Descripti	on	
Line No.	Fluid	Pressure (lb/in ² Temperature [gage]) (°F)		1b/h gal/min		acfm	From	To	Size (in.)	Schedule or psi	Material	
System 100	) <u>- Gasifier</u>											
101	Air	78	700	2 674	_	208g	Air beater	Flow divider	A	Sch 80	23	
102	Prod ass	76	021	3 822	_	736g	facifier	Senarator - see di	r naina	501 00	CS & SS	
102	Prod gas	()	300	4 201	_	1689	Ranhouse	Combustor	R	Sch 20	CS & 33	
103	Gas/melt	76	500	9,201	-	592	Gasifier	Burner port	18	Sch 80	CS	
105	Air	78	250	2.674	_	127	C-1	Air beater	3	Sch 40	CS	
106	Air	78	250	~1.500	-	~71	C-2	Line 105	2	Sch 40	CS CS	
107 (A.B)	Air	78	700	~1.500	-	104ª	Flow divider	Nozzle lines	2	Sch 80	CS CS	
108	Air	78	250	~1.240		121	Line 105	Burners	3	Sch 40	CS CS	
109	Prod gas	76	900	3.822	-	430 ^a	Separator	Pressure control	8	Sch 40	SS	
110	Prod gas	<1	<800	4,201	-	2800	Pressure control	Baghouse	8	Sch 20	CS CS	
111	Nat gas	30	100	6.	3 -	0.9	Gas header	Burner H-3, H-7	3/4	Sch 40	cs	
112 (A-H)	Air	78	700	~400	-	~30	Line 107	Gasifier nozzles	1	Sch 80	CS	
System 201	<u>) – Quench Ta</u>	<u>nk</u>										
201	Burner gas	76	500 ^b	85	-	123.6 ^c	Burner H-3	Melt dis. line	10	Sch 80	CS	
202	Gas & melt	76	. 500 ^b	920	-	211.6 ^c	Burner port	Quench tank	18	Sch 80	CS	
203	Water	136	60	3,228	6.5	-	Water header	Quench tank	3/4	Sch 40	CS	
204	GL	136	220	46,600	84.0	-	Pump P-2	Shatter nozzle	2	Sch 40	CS	
205	GL	136	220	9,986	18.0	-	Line 210	Wall wash nozzle	1-1/2	Sch 40	CS	
206	GL	6	220	3,819	6.9	-	Control valve	Line 211	3/4	Sch 40	CS	
207	GL	76	220	60,405	108.8	-	Quench tank	Pump P-2	4	Sch 40	CS	
208	Vent gas	<1	220	379	-	131.4	Control valve	Line 110	4	Sch 40	CS	
209	Steam	150	366	50	-	2.5	Steam header	Quench tank	3/4	Sch 40	CS	
210	GL	136	220	13,805	24.9	-	Line 204	Control valve	1-1/2	Sch 40	CS	
211	GL	6	220	20,000	36.0	-	Line 204	Tank V-6	1-1/2	Sch 40	CS	
212	GL	~10	220	~50,000	90.1	-	Tank V-6	Truck connection	3	Sch 40	CS	

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Table 6. Pipeline List (Sheet 1 of 2)

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	Оре	rating Condi	tions		Flow Rate		Extent	Extent of Line			Line Description		
Line No.	Fluid	Pressure (1b/in ² Temper luid [gage]) (°f		1b/h	gal/min	acfm	From	Το	Size (in.)	Schedule qr psi	Material		
<u>System 2</u>	00 – Quench T	<u>ank</u> (Continu	ed)				·	· ·					
213 [°] 214 215 216 217 <u>System 3</u>	Waţer Vent gas Water Vent gas Water 00 <u>- Black Li</u>	136 76 76 150 guo <u>r Feed</u>	60 220 220 220 60	0-3,000 379 0-3,000 379 0-50,000	0-5.4 - 0-5.4 - 0-100	81.7 ^c 81.7 ^c	Line 203 Quench tank Separator tank Separator V-4 Water supply line	Separator V-4 Separator V-4 Quench tank Control valve Shatter nozzle	3/4 6 2 4 2	Sch 40 Sch 40 Sch 40 Sch 40 Sch 40	CS CS CS CS CS		
301 302 303 304 305 306 307 308 309 310	8L 8L 8L 8L 8L 8L 8L 8L 8L	10 5 106 15 15 0 10 106 106 15	220 220 220 220 220 220 220 220 220 220	64,985 64,594 2,068 2,068 64,985 ~10,000 ~10,000 0-2,068 2,068 ~50,000	100 99 3.2 100 ~15.4 ~15.4 0-3.2 3.2 ~76.9		BL tank V-5 End of line 305 Pump P-4 End of line 305 Pump P-1 Test tank V-7 Pump P-3 Pump P-4 Line 303 Truck connection	Pump P-1 BL tank V-5 Gasifier Pump P-4 Start of line 304 Pump P-3 BL tank V-5 Line 302 Test tank V-7 Line 301	3 2 3/4 ^d 2 1-1/2 1 3/4 ^d 3/4 ^d 3	Sch 40 Sch 40 Sch 40 ^d Sch 40 ^d Sch 40 Sch 40 Sch 40 Sch 40 ^d Sch 40 ^d Sch 40 ^d	CS CS ^d CS ^d CS CS CS CS ^d CS ^d CS		

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Table	6.	Pi	ре	1	in	e	Li	st	
,	Char		2	_	2	21			

Notes: ^AAcfm will be 40 to 50% higher during operation at 1.0 atm pressure ^bMetal pipe temperature (refractory lined) ^CAcfm given is for 1.0 atm pressure with Burner 3 in operation ^d304 SS tubing may be substituted (3/4-in. ID)

Abbreviations: acfm = actual cubic feet per minute GL = green liquor BL = black liquor CS = carbon steel SS = stainless steel Type 304

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All pipe is carbon steel with the exception of elevated pressure lines operating at temperatures above 700°F, which are Type 304 stainless steel. These are primarily the lines carrying hot product gas from the product gas separator to the pressure control valve. The piping, which conveys the small stream of black liquor feed from the feed pump to the nozzles of the gasifier, is specified to be 3/4-in. carbon steel pipe with the option of using 3/4-in. ID Type 304 stainless steel tubing for this service.

The piping is generally Schedule 40. Exceptions are large size, lowpressure pipes where significant cost and weight savings can be realized by using Schedule 10, and high-temperature/high-pressure applications where the additional strength and rigidity of Schedule 80 pipe is believed to be justified.

Flanged connections will be used on all lines larger than 1 in. and on Schedule 80 1-in. lines. Flanges will be installed at the connections to valves, strainers, pumps, and equipment items and in piping runs, as required, to provide easy installation and disassembly. Welding may be used elsewhere on these lines.

Unless otherwise specified, 150-1b flanges will be used on Schedule 40 pipe and 300-1b flanges on Schedule 80 pipe connections. Schedule 40 carbon steel pipe 1 in. and smaller will be assembled using conventional threaded joints. Tubing will be connected using standard compression or bite-type fittings.

Instrument air and sample lines are not included in the pipe line list. These will generally be 1/4-in, Type 304 stainless steel tubing connected with compression fittings and field routed.

### E. INSTRUMENTATION AND CONTROLS

### 1. <u>Design Guidelines</u>

This section presents the preliminary requirements for designing the instrumentation and control system for the Black Liquor Gasification Pilot Plant. Overall guidelines for the design are as follows:

- 1. The existing computer data acquisition and control system (DAC) will be used to the maximum extent practical.
- 2. Control loops will be as simple as possible with the emphasis on operating flexibility rather than fully automatic plant operation.
- 3. Remote (control room) operation and control will be provided for critical parameters, including black liquor feed rate, air feed rate, make-up water feed rate, quench tank vent gas flow rate, melt shatter steam flow, gasifier pressure, baghouse inlet gas temperature, and quench tank liquid level.
- 4. Local instrumentation and control systems will be used for secondary parameters and subsystems, including black liquor heaters, green liquor heater and cooler, feed air heater, oil and gas burners, air compressors, and steam generator.
- 5. Adequate instrumentation, alarms, and controls will be provided to ensure timely recognition of potential problems, permit rapid remedial action when needed, and ensure personnel safety under all foreseeable operating conditions and events.
- 6. The extent and precision of measurements will be consistent with the R&D nature of the program and provide adequate data for the calculation of heat and material balances, and the evaluation and correlation of plant performance.

### 2. Instrumentation and Control System Description

A preliminary list of instrumentation requirements generated on the basis of the above guidelines is given in Table 7. Where no specific function is noted for instrumentation in the table, the measurements will provide data to aid in operation or in the evaluation and correlation of plant performance.

	Operating l	.imits		) ,
Parameter	Normal	Max	Symbol	Function
Temperature, °F				
Air from feed air heater	60 to 800	1,000	TIC	Control power to air heater
Air feed to gasifier	60 to 800	1,000	ТЈ	·
Air feed upper nozzles (4)	60 to 800	1,000	TJ	
Air feed lower nozzles (4)	60 to 800	1,000	TJA	Alarm if too high (melt backup) or too low (no airflow)
Gasifier interior (9)	1600 to 1900	2,200	ТJ	
Gasifier exterior skin (10)	300 to 600	800	TJ	
Product gas outlet	800 to 1200	2,000	TJA	Alarm if too high (no liquid spray)
Product gas outlet	800 to 1200	2,000	TI	Backup indicator in control room
Product gas separator outlet	700 to 1100	1,200	ТJ	
Product gas at control valve	500 to 800	1,000 .	TJ	
Product gas cooler outlet	400 to 500	500	TJ	
Gas to baghouse	400 to 500	500	TJC	Control cooler bypass valve
Gas to combustor	300 to 400	500	ТJ	
Combustor stack	1000 to 2000	2,200	TJA	Alarm if too high
Ambient air high bay	60 to 100	110	TJ	
Melt discharge at gasifier	1600 to 1900	2,200	TJ	
Natural Gas preheat burner	1600 to 1900	2,500	TJ	
Oil preheat burner tip	200 to 400	500	TJ	
Melt line burner	1200 to 1900	2,500	TJ	
Melt discharge at Q.T.	1600 to 1900	2,000	ТJ	
G.L. in quench tank	60 to 250	250	TIC	Control power to G.L. heater

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Table 7.	Preliminary	List	of	Instrumentation	Requirements
		(Shee	et 1	of 8)	

	Operating Limits			:
Parameter	Normal	Max	Symbol	Function
<u>Temperature, °F</u>				
G.L. in quench tank	60 to 250	250	TIC	Control valve on C.W. to G.L. cooler
G.L. from G.L. cooler	60 to 250	250	тј	
G.L. to shatter jet	60 to 250	250	TJ	
G.L. from Q.T.	60 to 250	250	TJ	
Vent gas from Q.T.	60 to 500	500	ТJ	
Vent gas from separator	60 to 250	250	• тэ	
Cooling water feed	50 to 100	100	TJ	
Steam feed	250 to 350	375	ТJ	
B.L. at tank truck	100 to 250	250	TI	Local thermometer
B.L. in B.L. tank	100 to 250	250	TIC	Control power to B.L. tank heater
B.L. in B.L. tank	100 to 250	. 250	TJ	
B.L. after recirc. pump	100 to 250	250	тј	
B.L. after recirc. heater	100 to 250	250	TIC	Control power to B.L. recirc, heater
B.L. after recirc. heater	100 to 250	250,	TJ	• • • • • • • • • • • • • • • • • • • •
B.L. feed stream	100 to 250	250	ТJ	
Cooling water return	50 to 150	200	TJ	
Pressure, 1b/in ² (gage)				
Air from main compressor	0 to 120	150	PJ	
Air from backup compressor	0 to 120	150	рJ	
Air to gasifier	0 to 120	150	РJ	
Air to gasifier	0 to 120	150	PI	Local gauge

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Table 7. Preliminary List of Instrumentation Requirements (Sheet 2 of 8)

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	Operating Limits			
Parameter	Norma]	Max	Symbol	Function
Pressure, 1b/in ² (gage)				······································
Gasifier	0 to 76	80	РЈА	Alarm on high pressure
Gasifier	0 to 76	80	PI	Backup indicator in control room
Product gas separator	0 to 76	80	РJ	•
Product gas from separator	0 to 76	80	PJ	
Product gas to PCV	0 to 76	80	PJC	Actuate pressure control valve
Product gas to PCV	0 to 76	80	PIC	Backup indicator-controller in control room
Product gas to PCV	0 to 76	80	PI	Local gauge
Product gas from PCV	0 to 2	5	рJ	
Product gas from PCV	0 to 2	5	PI	Local gauge
Product gas to baghouse, in. H ₂ O	0 to 15	20	рј	
Product gas from baghouse, in. H ₂ O	0 to 5	10	рј	、
Natural gas supply	0 to 30	45	PI	Local gauge
Quench tank	0 to 76	80	рј	
Quench tank	0 to 76	80 '	PI	Local gauge
Green liquor recirc. pump in	0 to 76	80	PJ	
Green liquor recir. pump out	40 to 116	150	рJ	
Vent gas after control valve, in. H ₂ O	0 to 20	25	рј	
B.L. intake line	0 to 15	20	PI	Local gauge
B.L. tank	0 to 15	20	PI	Local gauge
B.L. recirc. pump inlet	0 to 15	20	РJ	0

Table 7. Preliminary List of Instrumentation Requirements (Sheet 3 of 8)

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	Operating Limi			
Parameter	Normal	Max	Symbol	Function
Pressure, 1b/in (gage)	······································			
B.L. recirc. pump outlet	0 to 30	30	РJ	·
B.L. feed pump inlet	0 to 15	20	РJ	
B.L. feed pump outlet	20 to 116	150	рј	
B.L. feed nozzle, gasifier	20 to 116	150	РЈ	
B.L. feed nozzle, test tank	0 to 50	60	PI	Local gauge
Test`tank	0 to 15	15	PI	Local gauge
Process water supply	0 to 150	200	PI	Local gauge
Makeup water supply	0 to 100	150	PI	Local gauge
Nitrogen gas supply	0 to 1000	2000	PI	Local gauge
<u>Differential Pressure</u>				
Gasifier outlet/separator, psi	0 to 1	2	DPJA	Alarm on high $\Delta P$ (plugged outlet)
Baghouse outlet/separator, in. H ₂ O	<b>0 to 10</b> ,	20	DPI	Local DP gauge
G.L. product strainer, 1b/in ²	0 to 5	10	DPI	Local DP gauge
B.L. loop strainer, lb/in ²	0 to 5	10	DPI	Local DP gauge
B.L. feed strainer, lb/in ²	0 to 5	10	DPI	Local DP gauge
Flow Rate				
Total air to gasifier, 1b/h	0 to 2700	3,000	FJC	Control air feed valve
Total air to gasifier, 1b/h	0 to 2700	3,000	FIC	Backup indicator-controller
Feed air to heater, 1b/h	0 to 2700	3,000	FJ	
Feed air to lower nozzle, lb/h	0 to 1800	3,000	FJ	
Feed air to upper nozzle, lb/h	0 to 1800	3,000	FJ	

Table 7.	Preliminary	List o	of .	Instrumentation	Requirements
		(Sheet	c 4	of 8)	

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	Operating	Limits		
Parameter	Norma]	Max	Symbol	Function ,
Flow Rate				
G.L. recycle, gal/min	0 to 100	125	FJA	Alarm on low flow to shatter nozzle
G.L. wall wash, gal/min	0 to 25	35	FJA	Alarm on low flow to wall wash
G.L. product, gal/min	0 to 20	30	FJ	
G.L. product, gal	0 to 50,000	100,000	FQI	Local, measures total quantity produced
Vent gas, 1b/h	0 to 500	800	FJC	Control vent gas valve
Makeup water, gal	0 to 50,000	100,000	FQI	Local, measures total water added
Vent gas wash water, gal/min	0 to 5	1Ò	FI	Local rotometer
Shatter steam, 1b/h	0 to 50	75	FJC	Control shatter steam valve
Black liquor recirc., gal/min	0 to 100	125	FJ	Mass flow meter
Black liquor feed, lb/h	0 to 2500	3,000	FJC	Mass flow meter, feed valve control
Black liquor feed, lb/h	0 to 2500	3,000	FIC	Backup indicator-controller
Black liquor feed, gal/min	0 to 3.5	4.0	FJC	Volumetric flow, alternate control
Oil to preheat burner, 1b/h	0 to 60	100	FJ	
Air to oil burner, lb/h	0 to 1000	1,500	FJ	
Sweep air to oil burner, lb/h	0 to 1000	1,500	FJ	
Natural gas to preheat burner, SCFH	0 to 75	100	FI	Local flow meter
Natural gas to melt disch. burner, SCFH	0 to 75	100	FI	Local flow meter
Air to N.G. preheat burner, lb/h	0 to 100	150	FJ	

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# Table 7. Preliminary List of Instrumentation Requirements (Sheet 5 of 8)

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	Operating	Limits		
Parameter	Normal	Max	Symbol	Function
Flow Rate				
Air to melt disch. burner lb/h	0 to 100	150	FJ	
N.G. to product gas combustor, SCFH	0 to 100	150	FI	Local flow meter
Liquid Level				
Quench tank level, ft	3.0	1.0, 6.0	LJC	Control product G.L. valve
Quench tank level alarm, ft	3.0	2.5, 4.0	LJA	High- and low-level alarms
G.L. storage tank level, %	0 to 75	80	LJA	High-level alarm
B.L. tank level, %	0 to 75	80	LI,LJ	Local level gauge and DAC
Spray nozzle test tank level, %	0 to 75	80	LI	Local level gauge
Vent gas separator level, %	0.	50	LI,LJA	Local level gauge and high-level alarm
<u>Densitometers</u>				
Black liquor feed, g/cc	1.20 to 1.35	1.40	DMJ	
Green liquor product, g/cc	1.00 to 1.15	1.20	DMJ	
<u>Weigh Scales</u>				
B.L. tank weight, 1b	0 to 80,000	95,000	WJ	1
Spray nozzle test tank weight, lb .	0 to 5,000	6,000	WJ	
Oil tank weight, lb	0 to 1,000	1,000	WJ	

Table 7.	Preliminary 1	.ist of	Instrumentation	Requirements
		Sheet 6	of 8)	

_	Operating Limits		_	· · · · · · · · ·
Parameter	Normal	Max	Symbol	Function
<u>I&amp;C Package Systems</u> N.G. preheat burner, flame safety and control system			BCS	
Oil preheat burner, flame safety and control system			BCS	
Melt discharge burner, flame safety and control system			BCS	
Product gas combustor, flame safety and control system			BCS	
Main air compressor control system			CCS	
Backup air compressor control system			CCS	
Steam generator control system			SGCS	

### Table 7. Preliminary List of Instrumentation Requirements (Sheet 7 of 8)

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Testau	net Sumbola	Abbrauistiana	
	Data Acquisition and Control Center	$B \downarrow = Black \downarrow journ$	
TIC	Tomograture Indicator Controller	GI = Green Liquor	
T10	Tomporature to BAC	0.T = 0uench Tank	
τ 10	Tomporature to DAC with Alarm	CW - Cooling Water	1
TIC	Tomporature to DAC with Control	N.G Natural Gac	
TT	Temperature Indicator	N.G Natural das	
דו רם	Proceure to DAC		
רט ארם	Proceurs to DAC with Alarm		
רטא סום	Pressure to DAC with Control		
PJC	Pressure to bac with controllor		
DT	Pressure Indicator		
	Pressure Indicator		
רפח	Differential Process to DAC		
053	Differential Pressure to DAC		
DEJA	Differential Pressure to DAC with Atalm		
0F1 63	Class Pate to DAC		
гJ ET	Flow Rate to DAC		
E1C	Flow Rate Indicator		
FJC	Flow Rate to DAC with control		
FIC	Flow Rate Controller		
FUI	Flow localizer, Local Indicator		
LJ	Level to DAC		
	Level to DAC with Control		
LJA	Level to DAL with Aldim		
WJ	weigh Scale Reading to DAC		
BLS	Burner Flame Safety and Control System (Near Burner)		
005	Compressor Control System (At Compressor)		
SGUS	Steam Generator Control System (Part of Boiler)		
DMJ	Densitometer, Signal to DAC		

Table 7. Preliminary List of Instrumentation Requirements (Sheet 8 of 8)

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Operation of the pilot plant, with emphasis on the instrumentation and control approach, is described briefly below. Equipment numbers refer to the process flow diagram (Figure 1) and the equipment list (Table 4).

Black liquor feed is stored in the black liquor tank, V-5. The temperature in this tank is maintained at about 200°F by the black liquor tank heater, H-4, which is controlled automatically by a local temperature sensor and controller. Black liquor is continuously circulated from this tank through a pump, P-1, a strainer, S-2, an in-line heater, H-5, and back to the tank. The rate of flow in this loop is measured, but not controlled automatically since it has little effect on plant operation. The heater, H-5, augments the tank heater, H-4, to ensure that the black liquor feed stream is adequately heated. It is locally controlled by a temperature sensor in the black liquor recirculation line. The pressure drop across the strainer is indicated locally to warn the operator of possible plugging. The black liquor recirculation loop serves to mix, heat, and remove large solid particles from the black liquor prior to gasification. The density of black liquor in the loop is continuously monitored by a densitometer which transmits its signal to the DAC.

A small feed stream of black liquor flows from the recirculation loop through a fine strainer, S-3, and black liquor feed pump, P-4, which raises its pressure for injection into the gasifier. The black liquor feed rate is a critical parameter and is measured by both a mass flow meter and a volumetric flow meter, both of which provide signals to the control room. Either meter may be used as the primary source of control to actuate the black liquor feed rate control valve. Control is normally accomplished by the DAC; however, a backup indicator-controller is provided in the control room for use in case of a DAC failure. As a further check on the amount of black liquor fed into the system, the black liquor feed tank is equipped with a weigh scale, which provides a signal to the DAC. A local differential pressure gauge across the strainer, S-3, is used to provide evidence of plugging.

Air at a constant pressure (normally 120  $1b/in^2$  [gage]) is provided by either or both compressors, C-1 and C-2, which are locally controlled by existing systems. The total rate of airflow to the gasifier is measured and controlled automatically by a control valve actuated from the control room. During normal operation, this function is provided by the DAC; however, a backup indicator-controller is provided. The air and black liquor feed rates will be independently set and adjusted by the operator on the basis of the desired throughput and indicated gasifier temperature. The amount of air entering the heater, H-2, will be metered to provide data on the main stream of feed air, which enters through the upper and lower nozzles. The ratio of air feed to the two nozzle levels will be determined by flow meters in both branches of the air feed line, and adjusted by the manual operation of valves in these branch lines.

The main stream of gasifier feed air is preheated by the heater, H-2, to about 700°F. The selected preheat temperature is maintained by a local controller, which regulates power to the heater. Temperatures within the gasifier are monitored by nine thermocouples. The readings of key thermocouples are displayed continuously by the DAC for operator information, but are not used for automatic control of feed rates or other parameters. Other key gasifier temperatures that will be monitored by the DAC include the air feed nozzles (8), the gasifier vessel outside wall (~10 places), and the product gas outlet. This key temperature will also be shown on a backup temperature indicator in the control room. The temperatures at the lower air feed nozzles will be set to activate an alarm at either high or low temperatures indicating possible melt backup into the nozzles or airflow interruption, respectively. The product gas temperature will also be set to alarm at high temperature, which would be an early indication of black liquor feed flow stoppage.

Product gas from the gasifier flows to the separator, V-3, then through an uninsulated stainless-steel line, where it cools to less than 800°F before entering the main pressure control valve. This pressure control valve is set in the control room to hold the gasifier at the desired operating pressure.
It is normally actuated by the DAC; however, a separate backup pressure indicator-controller is provided. The automatic control valve is augmented by a manual bypass valve that can be opened in the event of plugging of the main valve. As added safety measures, the gasifier vessel is equipped with a pressure relief valve and a high-pressure alarm in the control room. The differential pressure across the product gas outlet port and separator is also monitored, and this measurement is set to alarm at high  $\Delta P$ , indicating possible plugging in this region.

After passing through the main pressure control valve, the product gas flows at near-atmospheric pressure through the product gas cooler, H-9, baghouse, S-1, and product gas combustor, H-1. The purpose of the product gas cooler is to ensure that the product gas temperature is below the maximum allowable temperature of the cloth bags in the baghouse. Excessive cooling is also undesirable since the condensation of water in the baghouse can cause poor flow of powder product and other operating problems. The amount of gas cooling is, therefore, controlled automatically by a temperature sensor in the feed line to the baghouse that is monitored by the DAC and actuates a valve in the cooler bypass line.

Operation of the baghouse is monitored primarily by a differential pressure measurement and by temperature and pressure readings on inlet and outlet streams, all of which are transmitted to the DAC. The product gas combustor is an existing facility completely equipped with local controls on its air supply and flame safety and burner control system. The rate of natural gas flow to the product gas combustor burner will be measured and recorded by the DAC. The stack gas temperature is also transmitted to the DAC, which is set to provide a high-temperature alarm to warn of improper operating conditions in the combustor.

Product melt and about 10% of the product gas flow out of the gasifier through the melt discharge line to quench tank, V-2. This line is equipped with a natural gas burner, H-3, which aids in maintaining the temperature inside the line above the melting point of the flowing melt (about 1550°F).

Thermocouples in the line sense the temperature and transmit the data to the DAC. If the melt temperature appears to be too low, the operator has several options including (1) initiating or increasing gas and airflow to burner, H-3. (2) increasing the flow of gasifier product gas through the line, (3) injecting air (without fuel) through burner, H-3, to react with combustible product gas in the line, and (4) increasing the temperature within the gasifier. It is planned that burner, H-3, will be used primarily during low-pressure, lowcapacity operation of the gasifier and will be turned off prior to operation at the full gasifier pressure of 90 lb/in² (absolute). Melt and gas throughput are high enough at this condition to maintain a suitably high temperature, and this operating procedure eliminates the need for a natural gas compressor on the burner feed. When in use, burner operation will be controlled by a local flame safety and control system, which meets all requirements for unattended operation. The rate of gas flow will be indicated by a local meter, however, the airflow rate will be transmitted to the DAC since total air input to the gasifier system is an important parameter for material balance calculations.

To ensure adequate breakup of melt falling into the quench tank, both steam and recycle green liquor shatter nozzles will be used. The steam flow rate will be measured and automatically controlled by an appropriate meter and control valve operable from the control room. The green liquor flow of about 84 gal/min will be measured, but not separately controlled, since the flow rate is not important as long as it is adequate to break up the melt stream. The recycle green liquor stream represents a major portion of the output of the green liquor recirculation pump, P-2. An alarm on low flow in the green liquor recirculation line will warn the operator of a blockage or pump failure, in which case he can activate an independent third shatter nozzle using process water from a high-pressure main.

Prior to melt production in the gasifier and during periods of low melt flow, it is necessary to add heat to the green liquor to maintain the desired operating temperature (180 to  $220^{\circ}$ F). This is accomplished by heater, H-6, which is controlled automatically by a local temperature sensor and power controller. At full operating capacity and during non-steady-state periods of high melt flow, it is necessary to remove excess heat from the green liquor. A cooler, H-8, is provided for this purposes. Operation of H-8 is controlled automatically by a valve in the cooling water line actuated locally by a temperature signal from a temperature sensor in the green liquor. A temperature signal is also sent to the DAC to show the temperature of the liquor leaving the cooler.

A large coarse-screen strainer inside the quench tank prevents large particles of solidified melt from leaving the quench tank and entering the suction side of P-2. Since they are soluble, particles held on the screen will eventually dissolve and clear the openings. A relatively small slip stream of green liquor is continuously removed from the green liquor recirculation loop downstream of P-2 and passed through a finer strainer, S-5, which removes small particles. This strainer is equipped with a differential pressure indicator to alert the operator to possible plugging. A green liquor flow of about 18 gal/min is returned to the quench tank from a point downstream of strainer, S-5, to provide a continuous wash for the inside surfaces of the quench tank vertical extension. The flow rate in the wall wash line is monitored by the DAC with an alarm to signal low flow.

A densitometer is used to measure the density of green liquor flowing in the line downstream of S-5. The densitometer operates through the DAC to control the flow of makeup water into the quench tank. A local flow totalizer will provide a record of the amount of water added. A small amount of water may also be injected into the vent gas separator to cool and wash this gas. The flow of water used for this purpose will be manually set and indicated by a local flow meter. Since this water drains into the quench tank, it represents a portion of the total makeup water.

Net green liquor product is discharged from the system downstream of strainer, S-5, to one of the green liquor storage tanks, V-6 (A and B). The rate of discharge is controlled automatically by a control valve actuated by the liquid level in the quench tank. Since it is important that an adequate liquid level be maintained in the quench tank at all times to provide feed for pump, P-2, and the shatter nozzle, and also that the level not rise to the point where it could interfere with shatter jet operation or contact hot refractory, two separate liquid level instruments are specified for the quench tank. One liquid level detector is independent of the DAC and operates locally to open and close the green liquor discharge valve. The other operates through the DAC to provide a liquid level reading in the control room and both high- and low-level alarms. Each of the green liquor storage tanks is also provided with a level indicator operating through the DAC to alarm on high levels.

Product gas that leaves the gasifier via the melt discharge line and combustion products from burner, H-3, flows into quench tank, V-2, with the product melt and must be continuously vented. The vent gas flows from the quench tank to a small separator, V-4, which serves to remove drops of green liquor or other particles which may be entrained. The separator is equipped with a water injection port for the addition of water to cool and wash the gas, if necessary. Excess water, collected green liquor, and condensed steam drain from the separator by gravity to the quench tank. A liquid level sensor with high-level alarm at the DAC is provided to warn of drain line plugging or other possible cause of increasing liquid level in the separator.

To ensure that 10% (or other desired amount) of the product gas flows through the melt discharge line into the quench tank, it is necessary to control the flow of gas out of the quench tank. For this purpose, a line is provided from separator, V-4 (which operates at the same pressure as the gasifier and quench tank), to a point of low pressure in the product gas line. A flow control valve in this line limits the amount of gas flowing and also reduces its pressure as required. This control valve is actuated by the DAC based on a flow rate sensor located in the low-pressure (high-velocity) portion of the vent line.

Special equipment items used during start-up or intermittently during operation also require instrumentation and controls. The spray nozzle test

tank, V-7, is used to permit black liquor flow through the feed system just prior to gasifier operation and also to test spray nozzles with hot black liquor feed. It is equipped with a weigh scale with readout at the DAC, a local level indicator, and a pressure gauge.

Two preheat burners are provided to raise the temperature of the gasifier vessel prior to plant operation. The natural gas burner, H-7, will be used for initial heating and as an igniter and pilot burner for the much larger capacity oil burner, H-10. Both burners are equipped with local flame safety and control systems, which meet all requirements for unattended operation. Natural gas to H-7 is metered with a locally mounted flow indicator. Air feed to H-7, as well as burner air and sweep air to the oil burner, H-10, are metered separately and recorded by the DAC. Since air (without fuel) is fed through the burners during normal operation with black liquor, information on the quantity of this air is required for material balance calculations. The amount of oil flowing to H-10 is indicated by a locally mounted flow meter. In addition, the oil tank is mounted on weigh scales with DAC readout to provide a record of fuel oil used.

Product gas chemical analyses will be performed by using the existing instrumentation. These instruments include one gas chromatograph (GC) with nitrogen carrier gas for hydrogen; a second GC with helium carrier gas for carbon dioxide, ethylene, ethane, oxygen plus argon, nitrogen, methane, and carbon monoxide; and separate continuous analyzers for oxygen, carbon dioxide, carbon monoxide, and total sulfur. The continuous analyses will be tied into the DAC to provide data to the plant operator, but will not be used for automatic control of any plant operation. Gas sample stations will be provided to permit the sampling of gas from the gasifier product line before mixing with the vent gas, the vent gas line, and the mixed gas line between the baghouse and the combustor. No continuous chemical analyses of liquid or solid streams will be performed; however, samples will be withdrawn periodically from the black liquor feed line, green liquor product line, baghouse discharge, and product gas separator discharge for laboratory analysis.

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### IV. SPRAY NOZZLE TESTING

### A. BACKGROUND AND OBJECTIVES

The black liquor gasifier requires a spray nozzle with unique characteristics. The drops it produces must be large enough to fall through the drying zone without being entrained in the product gas stream, but small enough to dry completely before they enter the gasification zone at the bottom of the vessel. The spray pattern must provide good coverage of the small-diameter gasifier vessel without excessive impingement on the walls. The nozzle orifice must be relatively large and unobstructed to minimize plugging problems. Finally, the unit used in the pilot plant must be scalable to larger sizes.

Splash-plate and hollow-cone nozzles used for feeding black liquor in recovery boilers are unsuitable because their spray patterns are not compatible with a small-diameter cylindrical gasifier configuration. A review of other commercially available nozzles during the previous phase of the program did not locate a design that would meet all the black liquor process requirements.^{6,7} It was concluded that narrow-angle, full-cone nozzles and twofluid (black liquor/steam) nozzles were the most likely of the commercially available nozzles to be adaptable to the gasifier application. A new concept was also proposed aimed specifically at the requirements of the pilot plant. The new concept has been called a splash-tube nozzle.

The objective of this task is to evaluate the full-cone pressure nozzle, two-fluid nozzle, and splash-tube nozzle designs for applicability to the requirements of black liquor gasification by experimentally testing nozzles of each type with water and hot wax.

## B. METHOD AND APPARATUS

The technique of using molten materials for measuring droplet size distributions from spray nozzles has been used by many prior investigators.^{10,11,12} It involves the spraying of molten a liquid (typically a wax product), which will form a droplet spray characterizing the size distribution of the test nozzle. The droplets are allowed to freeze in flight and are collected for analysis. A simple sieve analysis of the collected wax particles, when fitted to a log-normal (bell shaped) distribution curve, yields the following key parameters that describe the nozzle spray product:

- 1. The mass mean particle diameter,  $D_m$ , a measure of the particle size produced by the nozzle. Fifty percent by weight (or volume) of the particle are larger and 50% are smaller than  $D_m$ .
- 2. The geometric standard deviation,  $\sigma_G$ , a measure of the breadth of the size distribution.  $\sigma_G$  is the ratio:  $D_{84.13}/D_m$  (84.13% by weight of the particles are smaller than  $D_{84.13}$ ).
- 3. The correlation coefficient, a measure of the fit of the test data to a log-normal distribution curve.

A schematic diagram of the apparatus used for this study is shown in Figure 14.

Prior to a test, the wax reservoir is filled with preheated molten wax (about 2.5 lb capacity) and the hot water tank surrounding the wax reservoir is filled with boiling water from the hot water supply tank. Hot water is continuously circulated from the supply tank on the ground level to the hot water tank overhead providing uniform heating and stabilization of the temperature in the wax reservoir and the reservoir discharge line including the two-way solenoid valve. Water from around the solenoid valve is also permitted to drain through a 1/8-in.-diameter orifice on the valve inlet to flush and help preheat the valve and discharge tubing when wax is not flowing. An electrical heating tape is used to heat the test nozzle mounted outside and below the hot water tank.

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The test is initiated by pressurizing the wax reservoir to the desired test pressure using nitrogen, and then opening the solenoid valve for a predetermined time interval. The test nozzle discharges vertically downward approximately 10 to 11 ft to a plastic sheet collection basin, which measures 10 ft x 10 ft. A horizontal spray of cold water is directed across the basin to aid in solidifying the hot wax droplets and flushing them through a hole in the center of the basin into a product collection tank. The entire quantity of wax sprayed is collected in water, separated from the water by filtration, and dried. The resulting dried wax product is weighed and sieved using six Tyler screens with aperture openings from 1,651 microns to 350 microns across.

The five temperature sensing elements (designated TE) shown in Figure 14 are used to measure the wax reservoir internal temperature (upper and lower), hot water tank internal temperature, wax temperature at the inlet to the test nozzle, and exterior skin temperature of the test nozzle. The pressure on the wax reservoir is set using a precision test gauge, PI, and monitored using a O- to  $100-1b/in^2$  (gage) pressure transducer, PE. An IBM XT computer coupled with a Keithly 500 Data Acquisition System (DAS) is used to record and display the temperature and pressure test measurements every 0.5 s.

The hot-wax tests were conducted with Petrolite Polywax 500. This is a crystalline aliphatic hydrocarbon with a molecular weight of about 500 and a melting point of 187°F.¹³ A comparison of the properties of the wax, water, and black liquor at typical spraying conditions for the three fluids is given in Table 8.

Table 8.	Properties of	Liquids	
	Polywax 500	Black Liquor 65% Solids (typical)	Water
Temperature, °F (°C)	195 (91)	212 (100)	70 (21)
Viscosity, Cp	7.5	200	1.0
Density, g/cc	0.83	1.34	1.0
Surface tension, dyn/cm	30	40	73

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The nozzles tested are listed in Table 9. They include three commercial nozzles and the new splash tube nozzle that was fabricated in Rocketdyne's Test Support Machine Shop.

No.	Туре	Manufacturer	Spray Pattern	Part Name/Number					
1.	Pressure	Spraying Systems Company	Solid Cone30°	Fulljet 1/4 G3009					
2.	Pressure	Spraying Systems Company	Solid Cone30°	Fulljet 1/2 G3030					
3.	Two Fluid	North American Manufacturing Company	Two Fluid30°	Series 5622 O2A/O2B, Oil Nozzle No. 3-3289-1, and Steam Nozzle No. 3-3289-2					
4.	Pressure	Experimental Rocketdyne	Hollow Cone30°	Splash Tube					

In addition to the hot wax tests, tests were conducted on all of the above nozzles with cold water. These tests were used to observe the spray patterns and measure flow capacity data under various nozzle operating conditions. Flow capacity data were obtained by presetting the upstream nozzle pressure and collecting and weighing the total water stream emerging from the nozzle over a measured time interval.

#### с. TEST RESULTS

A total of 24 test runs were made with hot wax during this test series using the spray nozzles noted in Table 9. Flow and pressure data obtained with wax are presented in Table 10. The first test runs with the smaller (3009) full cone nozzle proved to be a series of checkout runs for the test apparatus and for refining the experimental techniques.

		Tank Noz Pressure Pres		e Nozzle	Wax Flow (Calc)		Wax Flow (Meas)		Air
Run No.	Run Nozzle** (16/in² (16/in² Tei No. No. [gage]) [gage])	Temperature (°F)	GPM	1b/h	GPM	1b/h	Flow (Meas)		
1	1	15.7		198	_	_			NA
2	1	49.1	· _	188	-	-	-	-	NA
3	1	48.8	· _	- 187	-		-	-	NA
- 4	1	36.7	33.5	194	0.898	373		-	NA
4 ·	1	15.0	13.7	192	0.574	239	-	-	NA
6	1	48.2	44.0	192	1.03	428	-		NA
7	1	49.0	44.7	194	1.04	431	0.487	203	NA
8	· 1	23.4	21.4	194	0.717	298	0.376	156	NA
9	2	47.9	26.2	195	2.34	973	-	-	NA
10	2	57.3	31.4	191	2.56	1064	-	-	NA
1]*	2	9.97	5.46	195	1.07	444	1.25	520	NA
12	2	14.2	7.77	201	1.28	530	1.43	594	NA
13	2	23.9	13.1	204	1.65	687	-	-	NA
14	3	4.37	1.94	204	0.785	326	-		51.2
15	3	8.0	3.54	201	1.06	441	-	-	71.2
16	3	9.20	4.08	200	1.14	473	1.24	516	46.8
17	3	8.63	3.82	202 '	1.10	458	1.13	470	46.4
18	3	8.76	3.88	200	1.11	462	1.52	632	46.4
19	3	9.28	4.11	202	1.14	475	-	-	46.4
20	3	8.87	3.93	202	1.12	465	1.28	531	37.1
21	3	8.94	3.96	203	1.12	466	1.10	456	55.7
22	4	24.3	11.8	203	1.78	740	-	-	NA,
23	4	48.4	23.4	203	2.51	1044	-	-	NA
24	4	72.6	35.1	203	3.08	1279	-	-	NA

# Table 10. Summary of Flow/Pressure Data with Wax

*Spray nozzles heated externally starting with Run 11. **See Table 9.

An evaluation of the results for tests 1 through 10 indicated that the wax flow rates were lower than predicted on the basis of manufacturer's data with water. Two potential causes of the low flow rates were identified: (1) partial freezing of wax within the test nozzles was occurring and (2) the flow resistance of the wax flow path from the wax reservoir to the nozzle inlet was not insignificant.

Prior to Run 10, the test nozzles had been heated only by flowing hot water through them immediately before initiating wax flow. Water flow also occurred immediately after the test. Examination of the internals of the nozzles after each test showed them to be clear of wax; however, the flushing action of the hot water after the wax flow had stopped would have removed the evidence of flow blockage. As a precautionary measure, a heating tape was therefore installed on the test nozzles for all runs starting with number 11. A skin thermocouple was also installed to monitor and control the nozzle temperature.

A series of water flow capacity measurements was made on the test setup to determine if the wax flow path resistance was significant when compared to the flow resistance of the spray nozzles. The measurements indicated that the flow resistance in the line and solenoid valve could not be neglected. An accurate value of this was, therefore, measured using water resistance so that the wax reservoir pressure could be corrected to give nozzle inlet pressure as a function of flow rate.

A check of the measured wax flow rates versus the calculated flow rates was made with the nozzle heater in operation and using corrected pressure data. It was found that, rather than being about 50% low, the new flow rate measurements were approximately 10 to 15% higher than predicted. However, this result can be explained by the inherent error in timing the wax flow for the short discharge intervals required by the tests. Once the above modifications to the test setup and procedure were made, wax flow rate checks gave reasonable results.

Water flow capacity data were obtained on each of the test nozzles to determine throughput as a function of differential pressure. For low viscosity liquids (less than 100 to 200 Cp), the nozzles should all behave like orifices with the volumetric flow rate affected only by pressure and density. This was born out by the consistent values of "K" observed for each nozzle. Table 11 presents the data obtained. The results for the commercial nozzles were within 10% of the data reported by the vendors.

The sieve analysis results on wax samples collected are presented in Table 12. Not all tests run were sieved. Examination of the wax collected during some runs revealed that a significant fraction of the particles were nonspherical; e.g., flattened out, or agglomerated and shapeless. These results generally occurred when low nozzle pressures were used; i.e., conditions that favored very large droplets. The large droplets were not cooled sufficiently in their 10- to 11-ft drop to the collection basin to form solid spheres. Figure 15 shows photographs of typical wax products from a run that produced predominately spherical particles (Run 6) and one that produced large nonspherical shapes (Run 22).

### D. DISCUSSION OF RESULTS

The calculated drop size requirements for the pilot plant gasifier are summarized in Table 13. The values given are based on the calculation procedure described in the Phase 2B Final Report.⁵ The minimum and maximum allowable drop sizes shown in the table are estimated by averaging the calculated results for the extreme cases of zero black liquor expansion and 100% expansion during the drying period.

No satisfactory method applicable to all types of nozzles has been developed for accurately extrapolating nozzle performance from one liquid to another. In general, the drop size can be expected to increase with increased viscosity and increased surface tension and decrease with decreased density. For pressure nozzles, available correlations indicate that water and hot wax will give about the same size drops, while black liquor (at 100°C) will yield drops which are 40 to 100% larger.

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	Table 11.	Water	Capacity Data		
	Water Pressure	Water	Flow Rate		
Nozzle*	P (1b/in ² [gage])	Q (gal/ min)	₩ (1b/h)		K**
1 (3009)	7.93 13.9 24.2 34.5 43.0 49.5	0.390 0.525 0.685 0.842 0.934 1.008	195 263 343 421 467 504	0.1389 0.1413 0.1392 0.1433 0.1433 0.1425 <u>0.1435</u>	
2 (3030)	9.8 9.75 20.5 30.1 39.9 48.7	1.324 1.327 1.891 2.265 2.600 2.867	662 664 946 1133 1300 1434 -	0.1414 0.4229 0.4250 0.4176 0.4128 0.4115 <u>0.4109</u>	average
3 (02A/02B)	5.0 6.15 6.95 8.0 9.15 10.45 20.7	1.085 1.233 1.319 1.427 1.509 1.613 2.280	543 617 660 714 755 807 1140	0.4168 0.4851 0.4971 0.5003 0.5046 0.4987 0.4989 0.5073	average
4 (splash-tube, initial con- figuration)	2.1 4.1 6.0 8.0 10.0 13.9 18.0 21.9 30.0	1.193 1.665 2.050 2.368 2.663 3.117 3.581 3.986 4.597	597 833 1025 1184 1332 1559 1791 1993 2299	0.4989 0.8235 0.8223 0.8367 0.8371 0.8421 0.8360 0.8441 0.8518 0.8392	average
4 (splash-tube, as tested)	18.0	2.008	1004	0.8370	average
*See Table 9. *K = Q $(\rho_{L}/P)^{1/2}$					
	Δ	T = DOF = 1	3566		

	Run Number									
	4	6	10	14	15	· 19	20	21	23	24
Nozzle*	1	1	2	3	3	3	3	3	4	4
<u>Sieve Uala</u> Weight per screen g										
1651 microns	0,923	1.360	12,208	5,624	14.320	6.927	8 701	3 303	16 199	16 252
1410	3,422	0.887	4,984	2,126	2.345	2.471	3.926	1.788	3.563	3 691
1000	3.902	7.238	21.077	5.232	4.378	5.211	8.419	4.302	7,139	7.932
840	19.764	23.607	14.845	4.870	4.096	4.401	6.513	3.568	4.146	5.006
425	10.164	59.037	30.225	18.161	17.200	14.509	21.431	14.456	12.450	13.801
350	25.142	8.380	8.041	5.979	6.620	5.085	6.095	5.143	3.031	2.780
PAN	9.038	14.614	7.019	43.298	42.449	24.457	35.209	21.729	7.200	6.872
Total	72.355	115.123	98.399	85.390	91.408	63.061	90.294	54.289	53.728	56.334
Calculated Results**										
Normalized % undersize										
1651 microns	93.995	98.819	87.593	93.414	84.334	89.015	90.364	93.916	69.850	71.151
1410	88.602	98.048	82.528	90.924	81.769	85.097	86.016	90.622	63.218	64.599
1000	61.287	91.761	61.108	84.797	76.979	76.834	76.692	82.698	49.931	50.518
840	47.239	71.255	46.022	78.976	72.498	69.855	69.479	76.126	42.214	41.632
425	12.491	19.973	15.305	57.708	53.681	46.847	45.744	49.498	19.042	17.134
350	-	12.694	7.133	50.706	46.439	38.783	38.994	40.025	13.401	12.199
Mass mean diameter, microns	805.51	598.16	831.82	347.99	379.44	470.51	476.49	437.27	1007.33	1001.62
	<u>+</u> 27.35	<u>+</u> 20.49	<u>+</u> 16.78	<u>+</u> 6.22	±14.84	<u>+</u> 6.84	<u>+</u> 11.82	<u>+</u> 6.28	<u>+</u> 5.82	+4.42
Geometric standard dev.	1.625	1.538	1.821	2.837	4.087	2.834	2.699	2.403	2.635	2.463
	<u>+</u> 0.042	<u>+</u> 0.035	<u>+</u> 0.038	±0.052	<u>+</u> 0.236	<u>+</u> 0.054	<u>+</u> 0.085	<u>+</u> 0.036	<u>+</u> 0.024	<u>+</u> 0.016
Correlation coefficient	0.9943	0.9945	0.9975	0.9994	0.9967	0.9993	0.9980	0.9994	0.9998	0.9999

Table 12. Size Distribution Results with Hot Wax

*See Table 9. **Based on least squares fit of test data to log-normal distribution curve.

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a nu% mort troduct from Run 6



b) Sample of unsieved product from Run 22

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•	Design Case					
	A B C					
Black liquor feed rate, lb/h	391	982	2,068			
Required drop size*						
Minimum size, microns	300	400	500			
Maximum size, microns	1,300	1,700	2,200			
D _m	600	800	1,100			

Table 13. Black Liquor Spray Requirements

*Approximate values based on average of drop sizes calculated by assuming zero and 100% expansion of sprayed black liquor drops during drying.

The hot wax drop size results for the four nozzles tested are summarized in Table 14. Typical size distribution curves for three different nozzle types are given in Figure 16.

For the specific nozzles and flow rates tested, the solid cone pressure nozzles appeared to give the particle size nearest to the desired value and also the narrowest size range. However, it was observed that this nozzle ejects the liquid with a very high downward velocity. In the wax spraying operations, this limited the tests to conditions which produced relatively small drops because the large drop sprays apparently did not have time to harden prior to collection. In the gasifier application, the high downward velocity would limit the time available for drying.

The two-fluid nozzle using hot wax and air was observed to develop a mist of very small particles in addition to the main stream of medium size drops. This nozzle also showed the greatest breadth of size distribution for the particles collected and sieved. The main drawback of the two-fluid nozzle, however, is its large requirement for air or steam. The minimum amount of gas which will produce a relatively stable spray is one-tenth the amount of liquid by weight. In a gasifier processing black liquor containing 35% water, the use of 0.1 lb of steam per pound of feed would increase the water content of

Run No.	Nozzle No.ª	Identification	Wax Pressure (1b/in ² )	Wax Flow Rate ^b (1b/h)	Air Flow Rate (lb/h)	D ^C (microns)	°G ^d
4	1	Fulljet 3009	33.5	<373	-	806	1.6
6	1	Fulljet 3009	44.0	<428		598	1.5
10	2	Fulljet 3030	31.4	<1,064	-	832	1.8
14	3	NA 02A/02B	1.9	326	51.2	348	2.8
15	3	NA 02A/02B	3.5	441	71.2	379	4.7
19	3	NA 02A/02B	4.1	475	46.4	471	2.8
20	3	NA 02A/02B	3.9	465	37.1	476	2.7
21	3	NA 02A/02B	4.0	466	55.7	437	2.4
23	4	Splash tube	23.4	1,044	-	1,007	2.6
24	4	Splash tube	35.1	1,279	-	1,002	2.5

Table 14. Summary of Hot Wax Spray Test Results

^aSee Table 9.

^bCalculated on the basis of water flow tests. Actual rates for Runs 4, 6, and 10 probably less than calculated values due to possible wax cooling and solidification within the nozzles. ^CMass mean diameter =  $D_{m}$ .

^dGeometric standard deviation =  $\sigma_{G}$ .

the product gas by almost 30% and would represent a significant consumption of process steam.

The splash-tube nozzle appeared to give an excellent spray pattern and the wax drops left the nozzle with a relatively low downward velocity. The observed drop sizes for the unit tested are somewhat larger then desired, but probably similar to those of a solid cone pressure nozzle with the same capacity (i.e., 1,279 lb/h at 35.1 psi). In general, the drop size of pressure nozzles increases with increased nozzle capacity (orifice size) and, for a given nozzle, decreases with increased pressure drop. The results of Runs 23 and 24 indicate that the splash-tube nozzle differs from conventional pressure nozzles in giving an almost constant drop size with changing pressure (and throughput). It is anticipated that smaller (or larger) drops can be obtained with this type of nozzle by changing the orifice size and splash tube configuration.





The results of the spray tests and published data indicate that all three types of nozzles can be designed and operated to give black liquor drops in the desired size range. The two-fluid nozzle is considered to be undesirable because of its high consumption of steam. The solid-cone and splash-tube nozzles are recommended for testing with black liquor in conjunction with the pilot plant program.

The splash-tube design is preferred at this time over the solid-cone nozzle because it does not contain a vane insert that may be subject to plugging by black liquor solids; it provides a lower exit velocity; and it can be cleaned and/or adjusted while in service.

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## V. CONCLUSIONS AND RECOMMENDATIONS

The black liquor gasification pilot plant design has been upgraded and improved to increase its operating pressure and capacity by a factor of 2 (to  $90 \text{ lb/in}^2$  (absolute) and 2,068 lb/h black liquor feed, respectively). The capacity increase causes the predicted heating value of the product gas to increase from 101 to 114 Btu/scf (dry basis).

All major pilot plant design uncertainties have been resolved and the required hardware and systems have been defined in sufficient detail to provide a firm basis for proceeding with the final design, construction, and operation of the facility.

No significant problems have been uncovered during the pilot plant design activities which would preclude satisfactory operation or attainment of the key design objectives of producing gas with a higher heating value (HHV) of 100 Btu/scf and melt in which over 90% of the sulfate is reduced to sulfide.

It is concluded that the black liquor gasification pilot plant as now configured represents a well-integrated, cost-effective facility for demonstrating all critical operations of the process and providing data for the design of much larger units.

Tests of three types of nozzles with hot wax indicated that all three can be designed and operated to spray black liquor into the gasifier with the desired drop size distribution and narrow cone pattern. One type, a two-fluid (steam/black liquor) nozzle, has been removed from further consideration because of its indicated high consumption of steam.

It is recommended that two of the nozzles, a narrow-angle, full-cone design and a specially designed splash-tube concept, be tested with black liquor as part of the pilot plant experimental program and that the final reference design be selected on the basis of the results. The splash-tube nozzle is preferred at this time because it is less subject to plugging (the full-cone nozzle contains internal vanes); ejects the black liquor with a lower initial downward velocity; and can be cleaned and/or adjusted while in service. However, the full-cone nozzle design warrants further consideration as a backup design because it provides a solid-cone rather than hollow-cone spray pattern and appears to give a somewhat narrower size distribution.

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