Report No. CG-D-189-75
Task No. 4322.2.1

A STUDY TO EVALUATE THE FEASIBILITY OF WET OXIDATION FOR SHIPBOARD WASTE WATER TREATMENT APPLICATION

R. B. Jagow

SEPTEMBER 1975

FINAL REPORT

Prepared For

DEPARTMENT OF TRANSPORTATION
UNITED STATES COAST GUARD
Office of Research and Development
Washington, D. C. 20590

This document is disseminated under the sponsorship of the Department of Transportation in the interest of information exchange. The United States Government assumes no liability for its content or use thereof.

The contents of this report do not necessarily reflect the official view or policy of the Coast Guard, and they do not constitute a standard, specification or regulation.

This report, or portions thereof, may not be used for advertising publication, or promotional purposes. Citation of trade names and manufacturers does not constitute endorsements or approval of such products.

W. L. KING
Captain, U. S. Coast Guard
Chief, Environmental and
Transportation Technology Division
Office of Research and Development
U. S. Coast Guard Headquarters
Washington, D. C. 20590

Technical Report Documentation Page

1. Report No.	2. Government Accession No.	3. Recipient's Catalog No.
USCG-D-189-75		

4. Title and Subtitle: A Study to Evaluate the Feasibility of Wet Oxidation for Shipboard Waste Water Treatment Application.

5. Report Date: Sep[illegible]75

6. Performing Organization Code:

7. Author(s): R. B. Jagow

8. Performing Organization Report No.: 194p.

9. Performing Organization Name and Address:
Lockheed Missiles & Space Company
1111 Lockheed Way
Sunnyvale, California 94088

10. Work Unit No. (TRAIS): CG-4322.2.1

11. Contract or Grant No.: DOT-CG-23034-A NEW

12. Sponsoring Agency Name and Address:
Department of Transportation
U.S. Coast Guard
Office of Research and Development
Washington D. C. 20590

13. Type of Report and Period Covered: Final Report

14. Sponsoring Agency Code: G-DET-1

15. Supplementary Notes

16. Abstract

The feasibility of applying wet oxidation to solve the problem of shipboard waste treatment on board a 20 man Coast Guard vessel was investigated by designing, building and testing a breadboard wet oxidation system using a non-stirred packed tower reactor. Initial tests in the tower reactor indicated favorable results but lead to catalyst screening tests in a one gallon stirred reactor. The selected catalyst was tested in a one liter packed tower and finally in the full scale breadboard system. Final process conditions were selected and the preliminary design of a shipboard system was developed. Additionally, chemical oxygen sources were investigated as a means of simplifying the system hardware and eliminating the catalyst. Results were promising. Plastic model flow tests were conducted on a variety of tube configurations. A four tube reactor design was then fabricated and tested with and without chemical oxygen sources.

17. Key Words	18. Distribution Statement
Waste Water Treatment, Shipboard, Wet Oxidation, Laboratory Test, Catalyst, Preliminary Design	Document is available to the public through the National Technical Information Service Springfield, Virginia 22151

19. Security Classif. (of this report)	20. Security Classif. (of this page)	21. No. of Pages	22. Price
Unclassified	Unclassified		

Form DOT F 1700.7 (8-72) Reproduction of completed page authorized

TABLE OF CONTENTS

TABLE OF CONTENTS (continued)

ILLUSTRATIONS

ILLUSTRATIONS (continued)

TABLES

LIST OF SYMBOLS & ABBREVIATIONS

(listed in order of appearance)

BOD	Biological Oxygen Demand (mg/liter)
pH	Negative Logrithum of Hydrogen Ion Concentration
USCG	United States Coast Guard
COD	Chemical Oxygen Demand (mg/liter)
TOC	Total Organic Carbon (mg/liter)
MPN	Most Probable Number
ΔP	Differential Pressure (psi)
C_v	Valve Flow Coefficient
SCFM	Standard Cubic Feet Per Minute of Air Flow
KW	Kilowatts
$\dot{V}air$	Volumetric Flow Rate of Air (SCFM)
$\dot{V}_s$	Sewage Volumetric Flow Rate (psia)
P_i	Upstream Pressure (psia)
T	Temperature (^oR)
GPM	Gallons Per Minute of Liquid Flow
Ptotal	Total Reactor Pressure (psia)
δ	Corrosion Specimen Deflection (inches)
r	Corrosion Specimen Bend Radius (inches)
ℓ	Corrosion Specimen Leg Length (inches)
E	Corrosion Specimen Modulus of Eleasticity (psi)
f_b	Corrosion Specimen Desired Stress Level (psi)
t	Corrosion Specimen Thickness (inches)
BK	Powdered Black Form of Noble Metal
MS 5X	5X Size of Molecular Sieve
VAC	Volts-Alternating Current
V	Volts
NEMA	National Electrical Manufacturers Association

INTRODUCTION

Wet oxidation is a flameless combustion process that has been used for several decades in chemical process and domestic sewage applications for the disposal of sludge. It involves the pumping of the sludge and air into an oxidation reactor where the organic materials are converted into water vapor and gases. Wet oxidation offers the advantages over dry incineration, one of the most widely used alternatives, in that much lower temperatures are required (450 to 600°F) vs (1000 to 1500°F) and the nitrogen, sulphur and phosphorous compounds are converted to salts that remain in the water instead of becoming air contaminating oxides. Wet oxidation also requires that very little of the water change phase which results in far less power than that required for incineration. Wet oxidation operation does require that the system be operated at elevated pressures of from 600 to 3000 psi depending on the application.

The use of wet oxidation for a twenty man Coast Guard vessel was the subject of the laboratory investigations covered by this report. In order to be practical for a twenty man vessel the wet oxidation plant must process all of the dilute waste generated from the commodes, urinals, gallery, scullery, showers, laundry and below water deck drains in order to avoid the complications of using multiple waste treatment systems. The processing of dilute sewage requires that a relative large amount of water must be pumped and heated to reactor conditions in order to oxidize a small amount of waste material. If process refinements could be made to reduce reaction pressure, temperature and contact time, the wet oxidation process could be an attractive single step, relatively simple approach to vessel waste water treatment. The work presented in this report was directed to that end, i.e., investigating wet

oxidation reactor configurations, process conditions, catalysts, and chemical oxygen sources to evolve the best possible wet oxidation process plant for the Coast Guard shipboard application and then to prepare a preliminary design of a 20 man shipboard system.

WASTE WATER SUPPLY & ANALYSIS

One of the initial tasks of the program was to define the waste model to be used in system testing. The system input hydraulic loading, BOD, suspended solids, pH, temperature, and salinity were specified in the program work statement but the specific contents of the input waste were to be determined. It was also recognized that collection of the wastes particularly fecal wastes in sufficient quantities to support full scale breadboard testing would present a problem. It was required that fecal and urine wastes be collected free of flush water or toilet paper so that exact amounts of each could be added to maintain a consistent input waste model. A third consideration was measurement of the influent and effluent water quality. The primary indicator of water quality was specified in the work statement to be Biochemical Oxygen Demand (BOD). BOD measurements consume a considerable amount of time and would hamper the test program schedules so alternate means of rapidly determining water quality to insure that BOD requirements could be met were studied and implemented during the program. This section describes the work conducted in these three areas.

Waste Model Definition

Early in the program arrangements were made to visit the New York Coast Guard facility on Governor's Island to discuss vessel waste water plumbing, possible locations for a waste treatment plant onboard a typical vessel, the sources of waste water and the type, quantity and frequency of wastes introduced into the ships waste water drains. These problems were discussed with the Captain and crew of the USCG MANITOU (WYTM 60). The following list of drains was compiled:

Officer's Head - 3 men

1 - Commode
1 - Sink
1 - Shower
1 - Deck Drain

Crew's Head - 18 men

1 - Commode
1 - Urinal
2 - Sinks
2 - Deck Drains
1 - Shower

Galley

2 - Deck Drains
1 - Double Sink

No garbage disposal as garbage is bagged and put ashore

Scuttlebutts - 1

Laundry

1 - Washer
1 - Deck Drain

As a result of discussions concerning the waste types, quantities and frequency of use the following list was prepared.

Commodes

Feces	0.35 lb/man-day [1]
Toilet Paper	125 rolls/month
Cigarette Butts	10 per day
Cleanser	1 cup/day
Disinfectant	1 pint/day

Sinks

Soap	20 bars/month
Shaving Soap	20 cans/six months

Urinal

Urine	3.2 lb/man-day [1]

Showers - Included in sink soap supply

Deck Drains - Included in commode cleanser

Galley

Soap	8 oz/day of liquid detergent
Cooking Oil	1 gallon/week
Food Scraps	1 cup/day
Animal Fats	2 cup/day
Coffee Grounds	1 cup/day

Clothes Washer

Laundry Detergent	5 cups/day
Bleach	5 cups/day

These data were converted into a waste model by converting all waste rates to a per day basis and combining this information with the average hydraulic loading of 720 gallons per day. The quantities of waste materials that must be added to 60 gallons of water were then computed by ratioing by 60/720. The resultant waste model is presented by Table 1.

Table 1

Initial Waste Model

Waste Material	Quantity
Feces	263 gm
Urine	2400 cc
Toilet Paper	164 gm
Cigarette Butts	1
Powdered Cleanser	1 1/3 Tbl.
Liquid Disinfectant	2 2/3 Tbl.
Bar Soap	7 1/2 gm
Shaving Soap	4 Tbl.
Liquid Soap	1 1/3 Tbl.
Cooking Oil	3 Tbl.
Food Scraps	1 1/3 Tbl.
Animal Fat	2 2/3 Tbl.
Coffee Grounds	1 1/3 Tbl.
Laundry Detergent	6 2/3 Tbl.
Bleach	6 2/3 Tbl.

Sixty gallons of water were selected as an input waste volume, because it is the size of the system input hold tank. In operating the system the waste quantities listed in Table 1 were flushed down the garbage disposal and into the waste hold tank. The tank was then filled to the sixty gallon mark. The

contents of the tank were then emptied into the pump/grinder assembly to make them available to the high pressure wet oxidation pump and a new batch of wastes were prepared in the hold tank.

Having established a preliminary waste model, the next step was to prepare a batch in accordance with the model and analyze for BOD in order to establish what adjustments were required to obtain the required BOD of 500 mg/liter as specified in the program work statement. Analysis of the BOD of the mix resulted in a value of 730 mg/liter. The waste material quantities listed in Table 1 were reduced in the ratio of 730/500 to produce the waste model used during the test program. Table 2 presents the resultant waste model.

Table 2

500 mg/liter BOD Waste Model

Waste Material	Quantity
Feces	171 gm
Urine	1600 cc
Toilet Paper	110 gm
Cigarette Butts	1
Powdered Cleanser	1 Tbl.
Liquid Disinfectant	1 3/4 Tbl.
Bar Soap	5 gm
Shaving Soap	3 Tbl.
Liquid Soap	1 Tbl.
Cooking Oil	2 Tbl.
Good Scraps	3/4 Tbl.
Animal Fat	1 3/4 Tbl.
Coffee Grounds	3/4 Tbl.
Laundry Detergent	4 1/3 Tbl.
Bleach	4 1/3 Tbl.

Measurements of the input BOD for five of the early breadboard system test runs showed values of 442, 465, 555, 522 and 503 mg/liter. Inability to obtain a truly representative sample probably accounts for most of the variation in readings. It is very difficult to obtain a homogeneous mixture of the wastes particularly with animal fat and coffee grounds. Waste collection, mixing, grinding, introduction into the system and analysis of the input proved to be a relatively smooth operation throughout the program.

Later in the program, it was observed that the model seemed to be very high in toilet paper in comparison to the other wastes particularly with respect to the fecal input. The model was adjusted to reduce toilet paper and increase the other wastes to retain the 500 mg/liter BOD. Table 3 presents the lower toilet paper waste model used for final breadboard system testing following catalyst studies in the one gallon injection reactor.

Table 3

Reduced Toilet Paper 500 mg/liter BOD Waste Model

Waste Material	Quantity
Feces	228 gm
Urine	2130 cc
Toilet Paper	22 gm
Powdered Cleanser	1 1/3 Tbl.
Liquid Disinfectant	2 1/3 Tbl.
Bar Soap	6.7 gm
Shaving Soap	4 Tbl.
Liquid Soap	1 1/3 Tbl.
Cooking Oil	2 2/3 Tbl.
Food Scraps	1 Tbl.
Animal Fat	2 1/3 Tbl.
Coffee Grounds	1 Tbl.
Laundry Detergent	5 3/4 Tbl.
Bleach	5 3/4 Tbl.

Waste Water Analysis

The contract work statement identified BOD as the measure to be used to indicate waste water quality. It was undoubtably selected because of it's wide acceptance and use as such an indicator. It's use, however, makes it difficult to conduct a parametric test program where subsequent tests and program decisions are based on the results of previous runs because of the five day incubation period inherent in the measurement technique. Chemical Oxygen Demand (COD) and Total Organic Carbon were considered early in the program as attractive alternatives to BOD if a correlation between either of them and BOD could be found. Analysis time for COD ranged between 2 to 4 hours and for TOC between 10 and 30 minutes depending on standardization and preparation requirements. It was assumed that for a given waste input the correlation between BOD, COD and TOC should be fairly consistent.

A literature survey was made to investigate the variations in these parameters based on analysis of domestic sewage. References 2 through 8 were studied and it was concluded that for domestic sewage the ratio of TOC/BOD ranged from 0.8 to 1.0 and COD/BOD from 1.2 to 2.5. In order to check these conclusions and to obtain the correlation values for the specific sewage model developed for this contract, BOD, COD and TOC measurements were made on the original waste model presented by Table 1. BOD, COD and TOC were analyzed to be 730, 1980 and 590 mg/liter, respectively, producing ratios of TOC/BOD = 0.81 and COD/BOD of 2.72. These ratios compared favorably with the published data. It was decided to use TOC as a primary water quality measurement and to further check the correlations during the breadboard test program. TOC and BOD correlations were made on breadboard system test runs 1, 3, 5, 6, 7, and 8 during the early portion of test program with the following results:

Run No.	TOC (mg/liter)			BOD (mg/liter)		
	Inlet	Outlet	% Reduction	Inlet	Outlet	% Reduction
1	360	36	90	442	51	88
3	311	138	56	465	209	55
5	540	68	87	-	86	84*
6	538	122	77	555	105	81
7	315	64	80	522	65	88
8	370	66	82	503	83	83

*Based on inlet BOD of Run 6

As can be seen by comparing % reduction in TOC and BOD, TOC is a good indicator of percent reduction in BOD. Correlation checks were also made between TOC and COD using both dilute and concentrated wastes processed in a one liter stirred reactor with the following results:

Input TOC	Output TOC	% Red. TOC	% Red. COD
420	32	92	87
466	86	82	82
20900	1910	91	87
20900	1925	90	90
18600	1549	92	92
20000	3650	83	83

In general the correlation between COD, TOC and BOD were found to be amazingly good considering the nature of the waste input, the difficulty of obtaining a truely representative sample, and the nature of the measurement techniques. Because the correlations were favorable and the TOC measurements were so much easier to obtain, TOC was used as the primary system performance measure throughout the program.

Waste Collection Facility

Preparation of wastes in accordance with the waste model presented no particular problem except for fecal collection. Previous experience had shown that collecting urine was not a problem, however, collection of fecal matter that was not mixed with urine or toilet paper was difficult. A camper stool with attached plastic bag was used, but it was very difficult to get donors to use the camper stool except for several motivated project team members. Since relatively large quantities of feces were required for full scale breadboard system tests (as much as 690 gm/day) as compared to previous smaller scale tests, it was decided to construct a more sophisticated fecal collection facility. Figure 1 presents a drawing of the facility that evolved from design studies conducted by the program design engineer and human factors specialists. The facility referred to as "Super John" was constructed in a rest room near the test system. The conventional toilet was removed and the raised floor structure installed. On the raised floor were placed a toilet seat and toilet paper disposal hole and lid. Ducts below the raised floor directed the fecal matter and toilet paper into buckets located below the toilet seat and toilet paper disposal hole. The toilet seat duct contained a urine collector and tube that collected the urine and directed it to a third bucket. A vent fan located in the ceiling area as part of the building ventilation system was connected to the closed area under the toilet by a large duct. Air drawn through the toilet seat and toilet paper disposal hole purged the collection area prior to exhausting through the duct and roof.

Feces collected in "Super John" was frozen for later use in the breadboard system. Fecal collection rates varied during the collection periods and

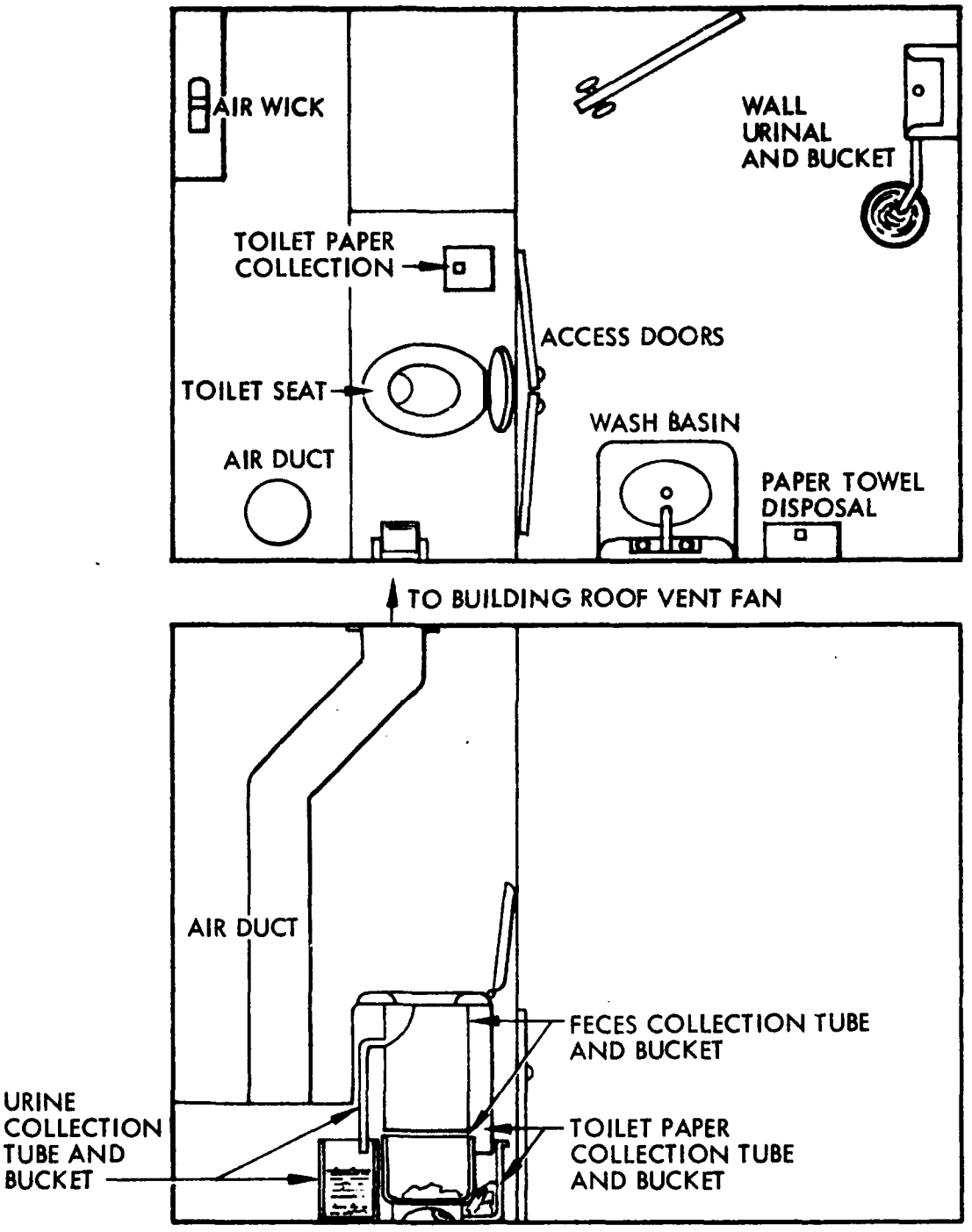


Fig. 1 Waste Collection Facility

advertizing several times during the program was required to increase use of the facility. Figure 2 presents the weight of fecal matter collected as a function time for a one month period. Urine and toilet paper collected in "Super John" were discarded because ample supplies of these materials were readily available. A conventional urinal without flush connection and a bucket were used for urine collection. The "Super John" satisfied the needs of the program very well throughout all testing.

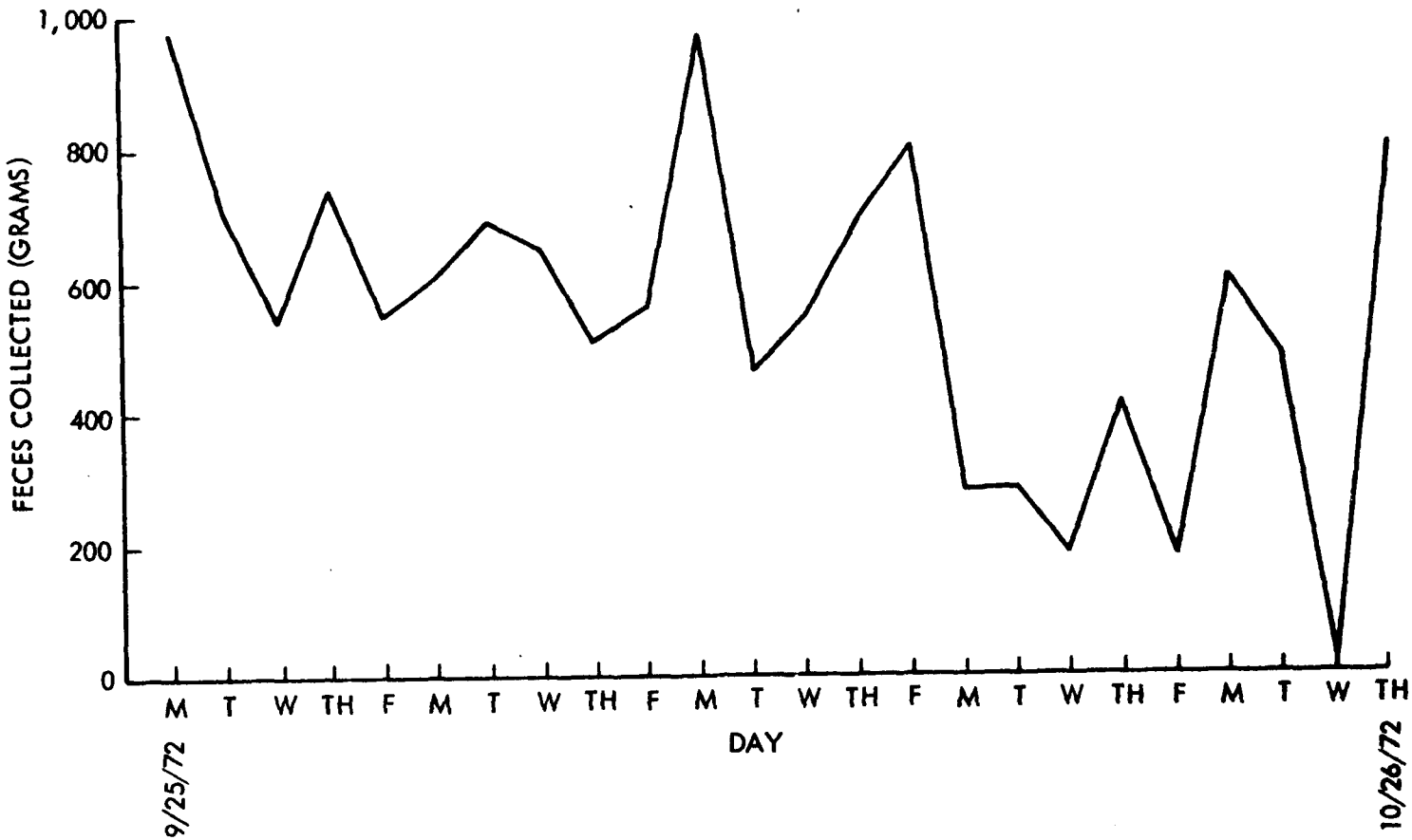


Fig. 2 Super John Performance

BREADBOARD SYSTEM DEFINITION

The breadboard system used during the test program was a combination of newly designed and fabricated equipment and hardware residuals from in-house sewage treatment developments. This section of the report describes the previous hardware developments, the new hardware designs, and the operation of the breadboard system.

Previous Development

Many elements of the breadboard test system were made available from a previous in-house development program that investigated the use of a chemical recirculation flush toilet system and a vacuum reduced-flush toilet system as means of concentrating toilet and urinal wastes for a 200 man ship. The program also investigated the integration of the collection equipment with a wet oxidation processing plant. The collection systems including toilets, urinals, drains, collection tank, recirculation pump and controls, grinder and sewage pump were assembled and tested including full scale one and three day operations with as many as 250 volunteer employee donors. Integration of the collection facility with the wet oxidation system was studied analytically and key elements of the system were designed and procured including a five gallon tower reactor, recirculation hot pump, five gallon stirred reactor, hydraulic pump and bladdered tank. A review of the contract work statement requirements showed that most of this equipment could be directly applied to the contract effort. Principal missing elements were an air compressor, regenerative heat exchanger, heater, and cooler.

United States Coast Guard System Requirements

The following system requirements were specified in the contract work statement or were generated based on previous wet oxidation laboratory investigations.

Max. System Design Temperature	650°F
Max. System Design Pressure	4500 psig
Crew Size	20 men
Hydraulic Loading	
Average	700 gpd
Average Peak	1400 gpd

Waste Sources

Sanitary, galley, scullery, shower, laundry, lavatory, and below water deck drains

Influent Characteristics

Suspended Solids (mg/liter)	
Minimum	200
Average	500
Maximum	4900
BOD	
Minimum	150
Average	500
Maximum	1800
ph	
Minimum	6
Maximum	9
Temperature	
Minimum	28°F
Maximum	95°F
Salinity	
Minimum	0
Maximum	35,000 ppm

Effluent Characteristics

Suspended Solids	50 mg/liter
BOD	50 mg/liter
Total Coliform	240 MPN/100 ml

These requirements were used to support calculations sizing the components of the breadboard system and later in the program to support the preliminary design of a shipboard system.

Breadboard System Schematic & Operation

The breadboard system shown schematically by Figure 3 was comprised of; (1) a waste collection system including two toilets, one urinal, one sink with garbage disposal, input mixing tank with water supply valve and drain valve, and a pump/grinder assembly; (2) a high pressure sewage pumping system comprised of hydraulic pump, hydraulic reservoir, hydraulic cylinder, two bladdered tanks, and two motorized sewage shutoff valves; (3) an air supply system consisting of air compressor, filters, drier, accumulator and flow controls; (4) heating and cooling equipment including regenerative heat exchanger, electrical heater and water cooler; (5) reactor system consisting of reactor vessel, electrical heaters, insulation, internal packing and flow distribution chambers, and safety relief rupture disc; and (6) miscellaneous temperature and pressure controls and sampling provisions.

The system operation is described as follows: Waste materials were measured into a bucket and dumped into the garbage disposal where water was added to flush them into the input mixing tank. Although the toilets and urinal were not used to introduce wastes into the system they were used to flush the lines. The input mixing tank was then filled to capacity by opening and closing the city water supply valve. The input mixing tank drain valve was then opened to dump the sixty gallon waste supply into the pump/grinder assembly. The drain valve was then closed and a new charge of wastes was flushed into the input mixing tank. Meanwhile, the pump/grinder picked upwastes from the bottom of the pump/grinder tank and recirculated them back into the tank for regrinding. Pump/grinder outlet pressure was adjusted by closing the recirculation valve to obtain a reading of 10 psi for normal operation and 60 psi while filling the high pressure pump system bladdered tank. The pump-grinder assembly contained a low level cutoff that shutoff the pump/grinder when sewage level in the tank approached the pump inlet.

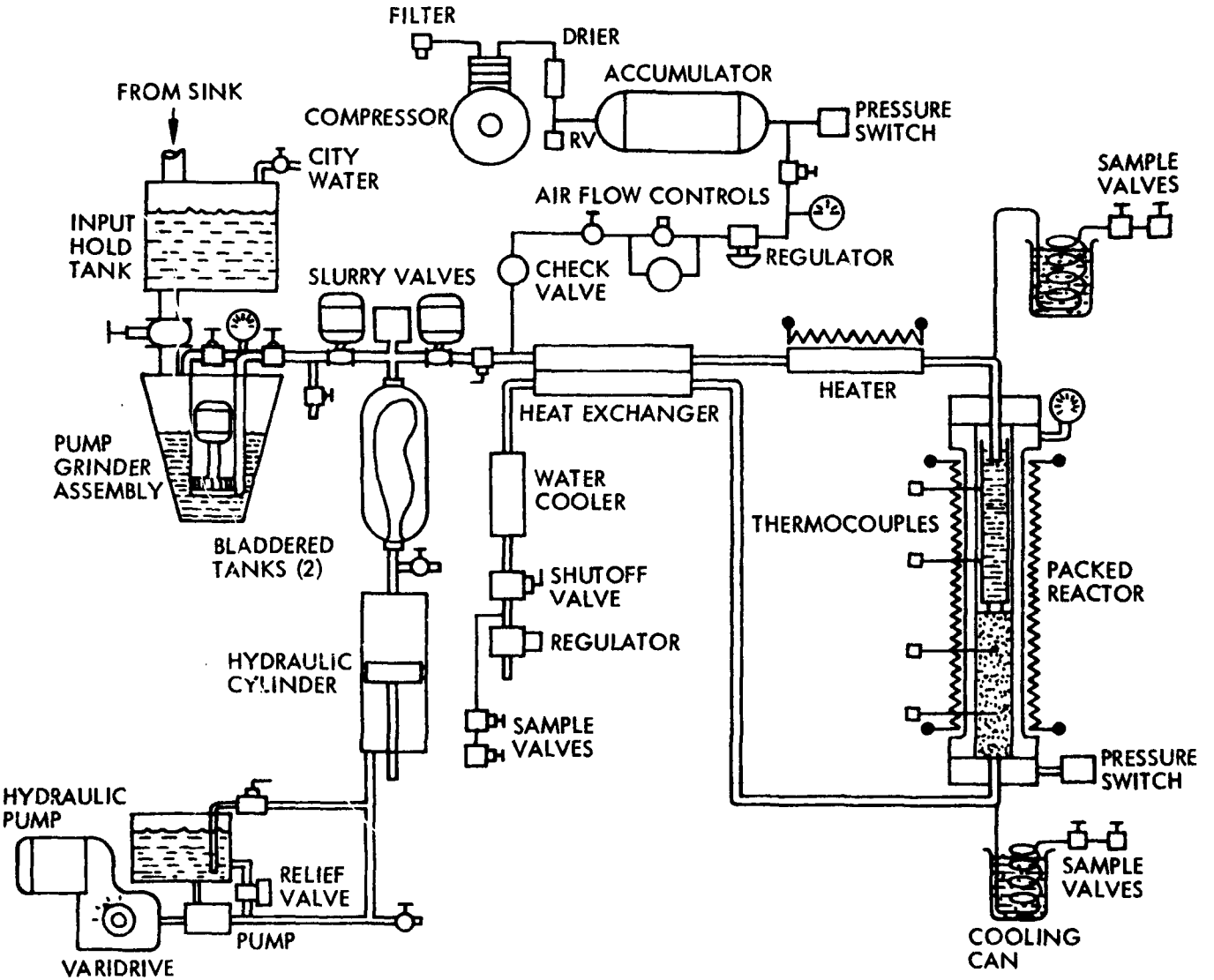


Fig. 3 Breadboard System Schematic

The sewage from the pump grinder was forced through a motorized sewage fill valve and into two bladdered tanks which in turn forced water from the back side of the bladder out of the tank and into the top end of a hydraulic cylinder. Water flowing into the hydraulic cylinder in turn forced the piston down, pushing hydraulic oil in the bottom of the cylinder back into the hydraulic reservoir through a manual bleed valve. When the bladdered tank was full of sewage, which was indicated by the hydraulic cylinder piston rod position, the motorized sewage fill valve, and the manual hydraulic oil bleed valve were closed. The hydraulic pump then automatically pressurized the hydraulic cylinder and bladdered tank to reactor pressure plus 500 psig at which point the hydraulic pump output was directed back to the hydraulic reservoir through the adjustable hydraulic relief valve. The motorized sewage delivery valve was then opened. The adjustable positive displacement hydraulic pump would then pump sewage in the reactor at a constant flow rate.

Air from a high pressure air compressor, accumulator, and air flow controls was mixed with the sewage downstream of the motorized sewage delivery valve. The air compressor was controlled by a dual set on-off pressure switch to maintain the air accumulator at or near 5000 psig. An air pressure regulator maintained a constant pressure on two air flow control valves. Air flow was established by maintaining a 200 psi ΔP across the valves and setting the valves at a predetermined setting by use of the digital handles. One flow control valve provided for high flows (flow coefficient C_v = .008) and one provided for low flows (C_v = .0008).

The sewage and air mixture was forced through a tube in tube regenerative heat exchanger where hot effluent from the reactor was used to heat the incoming sewage and air. The hot sewage and air then passed through a tubular heater where they were heated to reactor temperature using electrical energy. The hot mixture then entered the top of the tower reactor which was essentially a five inch pipe with flanges. Inside of the reactor were a hydrolysis pot in the top half and porcelain burl saddle packing material in the bottom half. Sewage filled the hydrolysis pot which provided a short

hold time for breakdown of the sewage, then overflowed into the packing, trickled through the packing where oxidation took place and then exited the reactor through a fitting at the bottom of the reactor along with the reacted gases. The effluent liquid, gases, and ash produced by the oxidation process then passed through the other leg of the regenerative heat exchanger and through a water cooler. The ambient temperature effluent was then vented through an adjustable dome loaded back pressure regulator that maintained total system pressure by controlling the vent flow. Effluent was vented down the laboratory floor drain. Sample lines and valves were provided at the heater outlet, reactor outlet and system outlet.

Electrical controls for the system consisted of the following:

On-Off manual switch for the garbage disposal

On-Off manual and automatic low level shutoff for the pump/grinder assembly

On-Off manual switch for the hydraulic pump

On-Off manual and on-off automatic pressure switch control for the air compressor

Manual open and closed switch for the motorized sewage fill and delivery valves with pressure switch safety to prevent inadvertant valve opening with pressure on the line.

On-Off manual switch, automatic relay and backup automatic relay controlled electrical heater. Normal and emergency temperature controllers operated the two relays independently so that complete redundancy of control was provided.

On-Off manual switch, automatic relay and backup automatic relay controlled reactor heaters. Normal and emergency temperature controllers operated the two relays independently so that complete redundancy of control was provided.

BREADBOARD SYSTEM DESIGN

The system defined in the previous section was converted into a working system by sizing and selecting components that would meet the requirements of the breadboard system. This section presents a brief description of the major system components, pertinent performance data, calculations and a description of the system layout in the laboratory.

Component Descriptions

Input Mixing Tank - The input mixing tank shown by Figure 4 was a 2 feet cube made from 1/8 carbon steel plate and angle iron with a 3" plastic sewer line connected to the top lid, a 1 1/4" plastic city water line connected to the side and a 3" drain line and hand operated gate valve in the bottom. The tank capacity was 60 gallons but was filled to 56 gallons to avoid overflow of the pump grinder assembly during dumping.

Pump/Grinder Assembly - The pump/grinder assembly as shown by Figures 4 and 5 was housed in a tapered fiberglass tank 30 inches in diameter at the top, 24 inches in diameter at the bottom and 36 inches tall. Tank capacity was approximately 75 gallons. The pump/grinder located in the center of the tank was suspended from the lid and was 35 inches long and 8 inches in diameter. Sewage pulled from the bottom of the tank passed through the grinder into the progressive cavity type wobble pump. The pump outlet directed the flow out of the unit lid through a 1 1/4 inch discharge pipe. The pump/grinder was driven by a one horsepower 220 volt single phase motor and the pump produced flows up to 4 gpm and pressures to 60 psi.

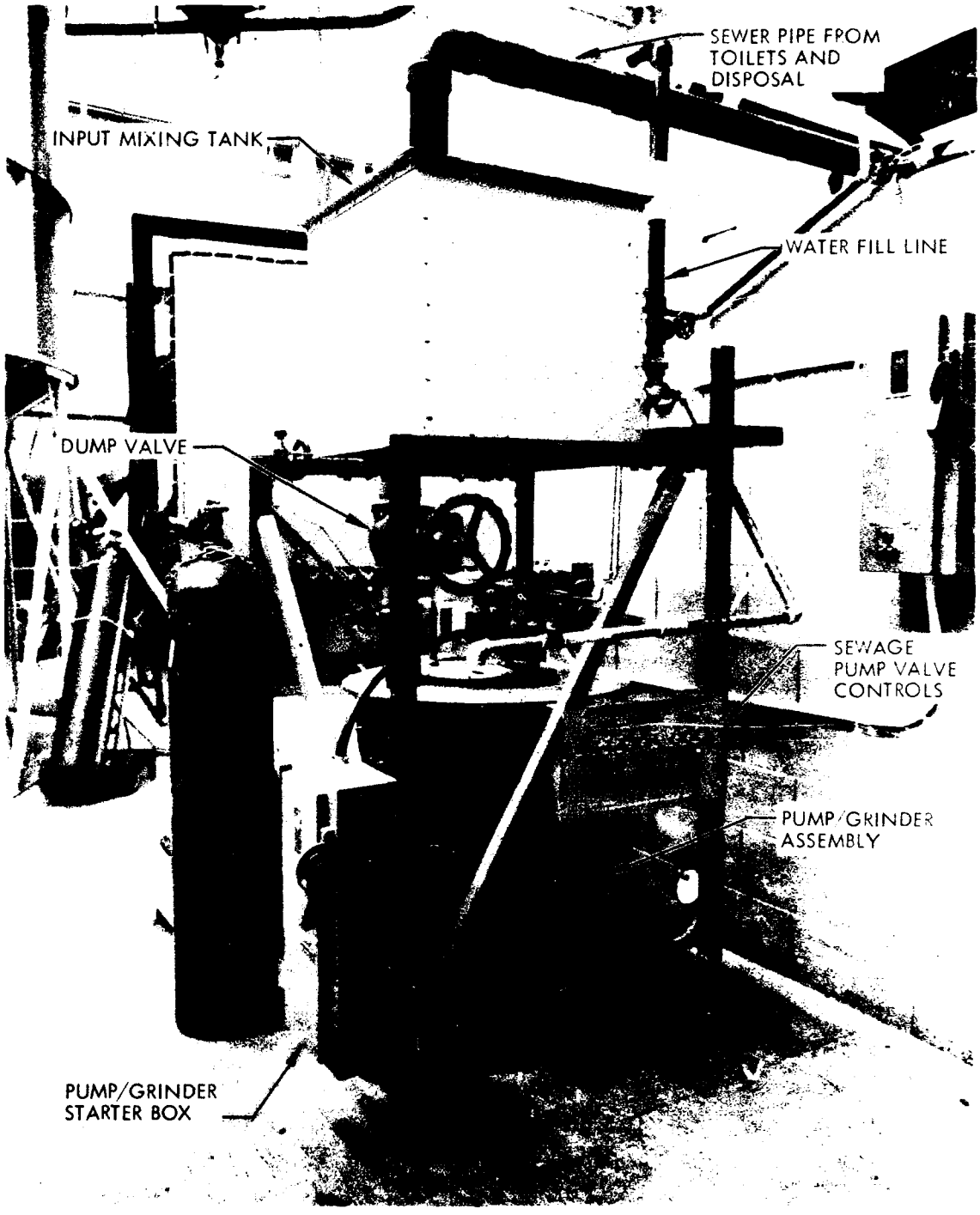
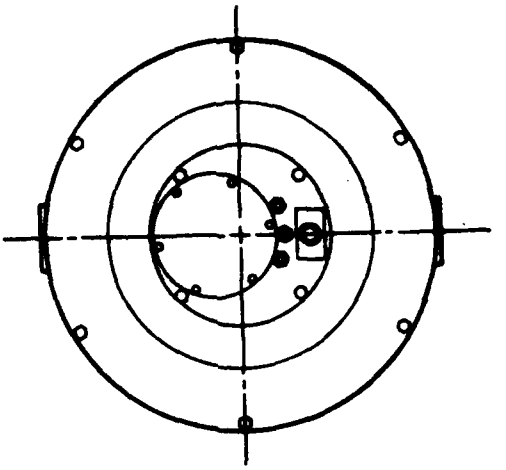


Fig. 4 Input Mixing Tank and Pump/Grinder



ALL DIMENSIONS ARE IN INCHES

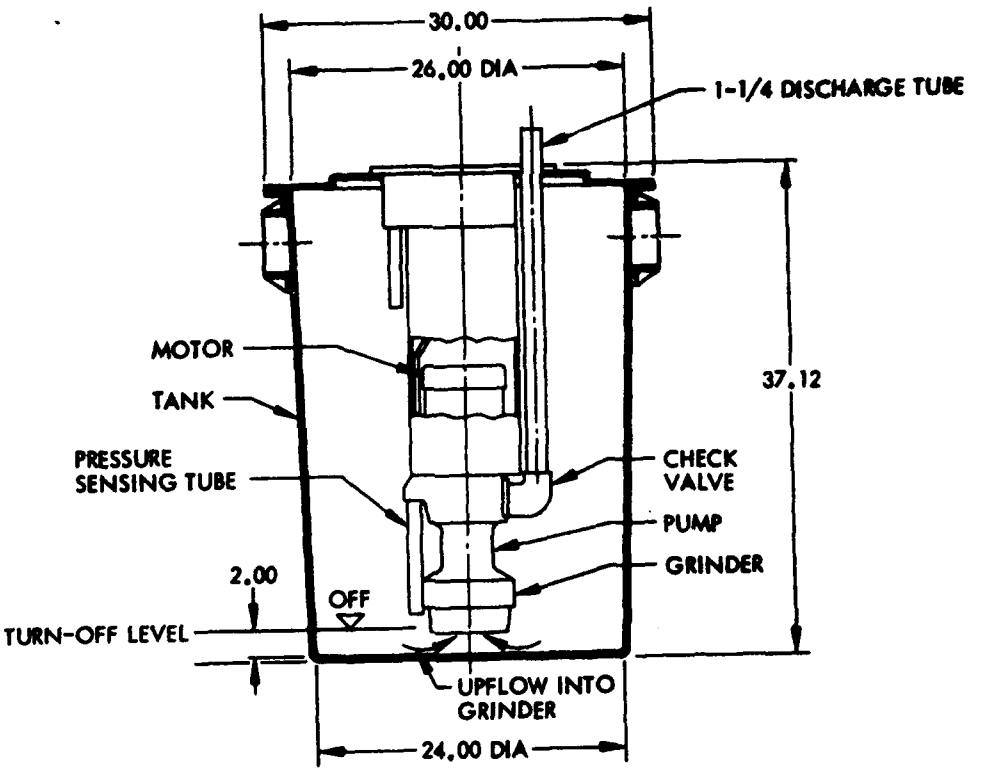


Fig. 5 Pump/Grinder Assembly

Motorized Sewage Valves - The motorized sewage valves were 3/4" Jamesbury HP36GT, 316 stainless steel ball valves rated for 4500 psig service. The EJ-20 electrical motor actuators provided cam stops for 90 degree rotation of the valve for opening and closing and also provided visual indication of valve position. The valve in the open position provided a 7/16" diameter unobstructed hole for sewage flow.

Bladdered Tanks - One 5 gallon and one 2 1/2 gallon bladdered tanks were used to provide a 7 1/2 gallon sewage capacity as shown by Figure 6. This size was selected so that at the design sewage flow of 0.5 gpm, 15 minutes of operation could be sustained before refilling of the tanks was required and also because this tank capacity represented approximately three times the liquid volume of the system so that in one pump cycle the entire system could be purged. The tanks utilized a buna-n bladder and were rated for 6000 psi service.

Hydraulic Cylinder - The hydraulic cylinder shown by Figure 6 had a 7.8 gallon capacity with an 8" bore and 36 inch stroke. It was rated at 5000 psi and contained double piston seals so that leakage of hydraulic oil or water from either side of the piston was vented through the piston and piston rod vent hole thereby preventing contamination of either fluid. The 1" diameter piston rod was used as a position indicator.

Hydraulic Pump - The hydraulic pump assembly shown by Figure 7 contained a rotary vane positive displacement pump rated for a maximum flow of 0.9 gpm at 5000 psi. A varidrive belt arrangement allowed for pump flow adjustment. A 2 horsepower, 220 volt, 3 phase motor drove the pump. The hydraulic reservoir had a useful capacity of approximately 9 gallons. An adjustable relief valve allowed adjustment of the maximum pump output pressure from ambient to 5000 psig.

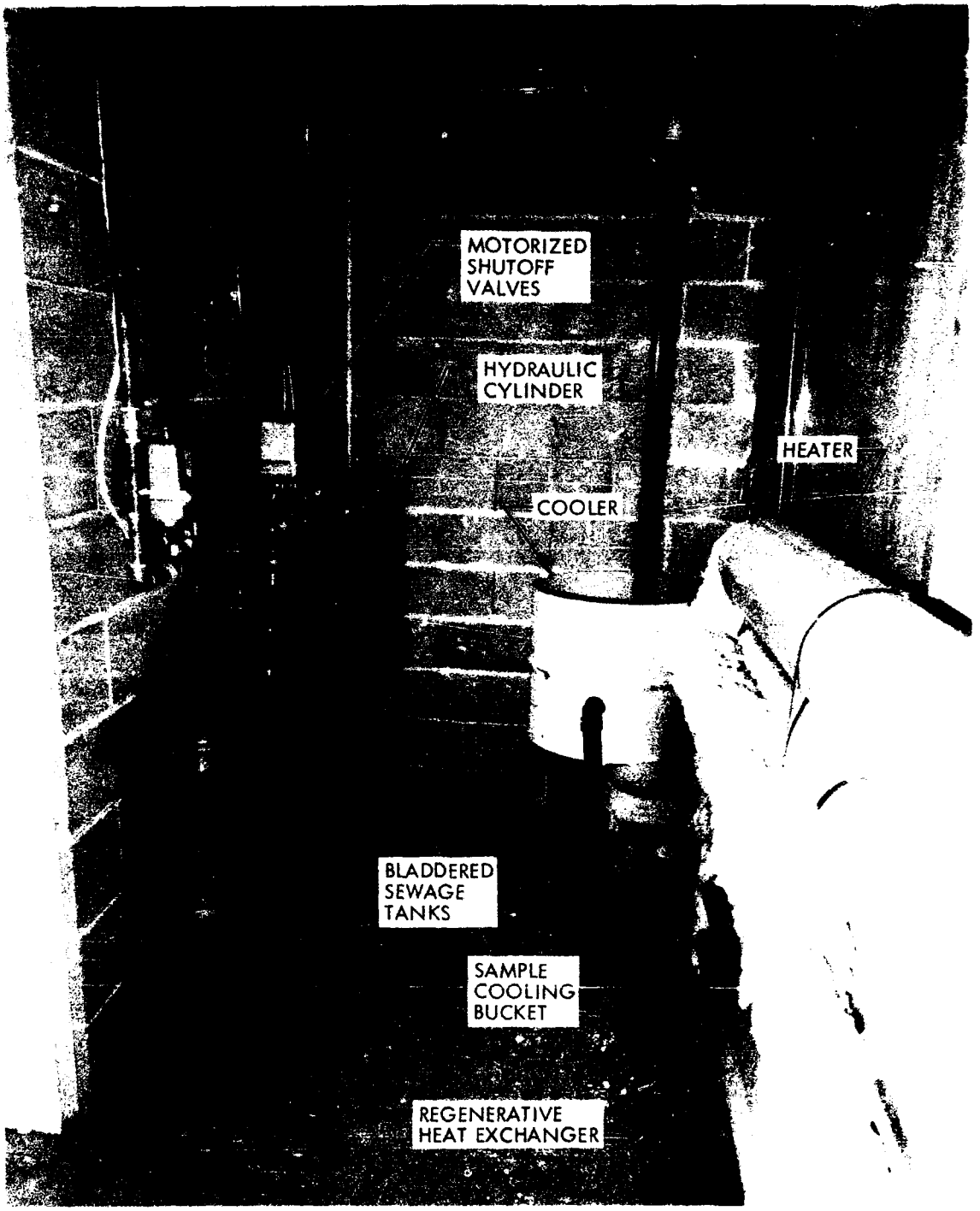


Fig. 6 Bladdered Tanks and Hydraulic Cylinder

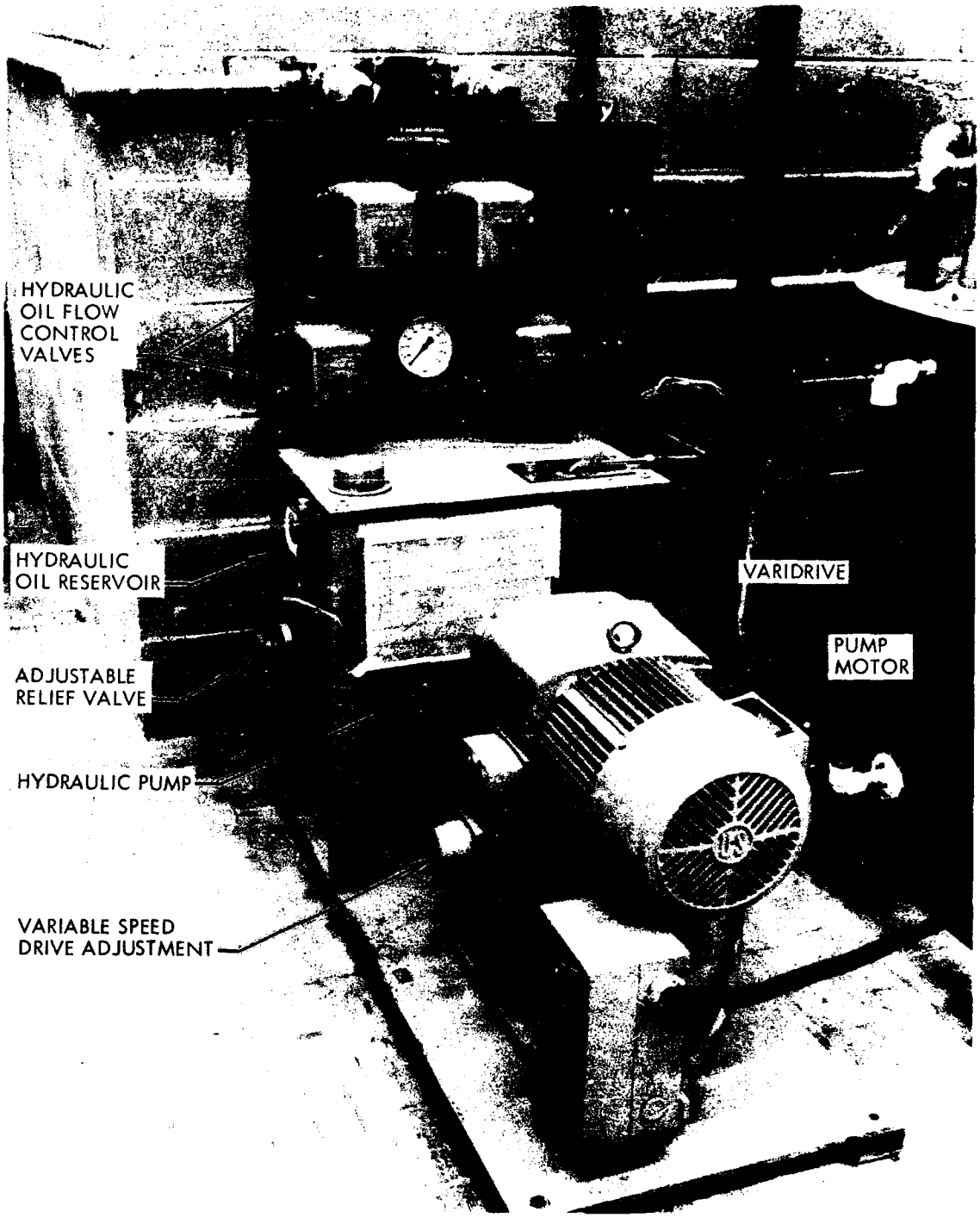


Fig. 7 Hydraulic Pump

Regenerative Heat Exchanger - The regenerative heat exchanger shown by figure 8 was a 1/4" schedule 80, 316 stainless steel pipe inside of a 1" schedule 160, 316 stainless steel pipe with 120 feet of heat transfer length. The length was obtained by bending the tube to provide six - 20 foot long sections with "U" bends at each end resulting in an envelope 6" wide, 40 inches tall and 22 feet long including support stand,"U" bends and 3 inches of insulation on all heated pipes. The pipe sizes were such that the inside pipe which carried sewage and air had an inside diameter of 0.302" and the annulas between pipes which carried effluent water and gases was 0.137". Insulation was a commercial grade of pipe insulation.

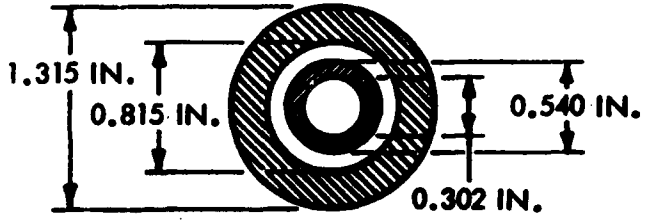
The heat exchanger length was selected based on trade offs between heater size and heat exchanger size. A 120 foot heat exchanger resulted in a 15 KW heater requirement and a heat exchange outlet temperature of 520°F based on the following design data:

0.5 gpm sewage flow
2 scfm air flow
650°F maximum reactor temperature
4500 psi maximum reactor pressure
100* BTU/HR-Ft.^{2}-°F overall heat transfer coefficient
167,000 BTU/HR total heater and heat exchanger heat load (70°F to 650°F)

The heating apportionment was 117,000 BTU/HR in the heat exchanger and 50,000 BTU/HR in the electrical heater. This selection was made to produce a reasonable balance between heater power and heat exchanger size for the breadboard system.

Heater - The electrical heater consisted of a 0.5" o.d. 316 stainless steel tube 20 feet long with a 0.065" wall with a nichrome wire ceramic core heater element and 3 inches of commercial pipe insulation wrapped around it. A 20 KW heater was used based on the 15 KW load calculated above to provide an allowance for losses from the insulation plus a safety factor considering the difficulty in establishing two phase flow heat transfer coefficients. The heater element

*estimated based on data in reference 9



TUBE CROSS SECTION

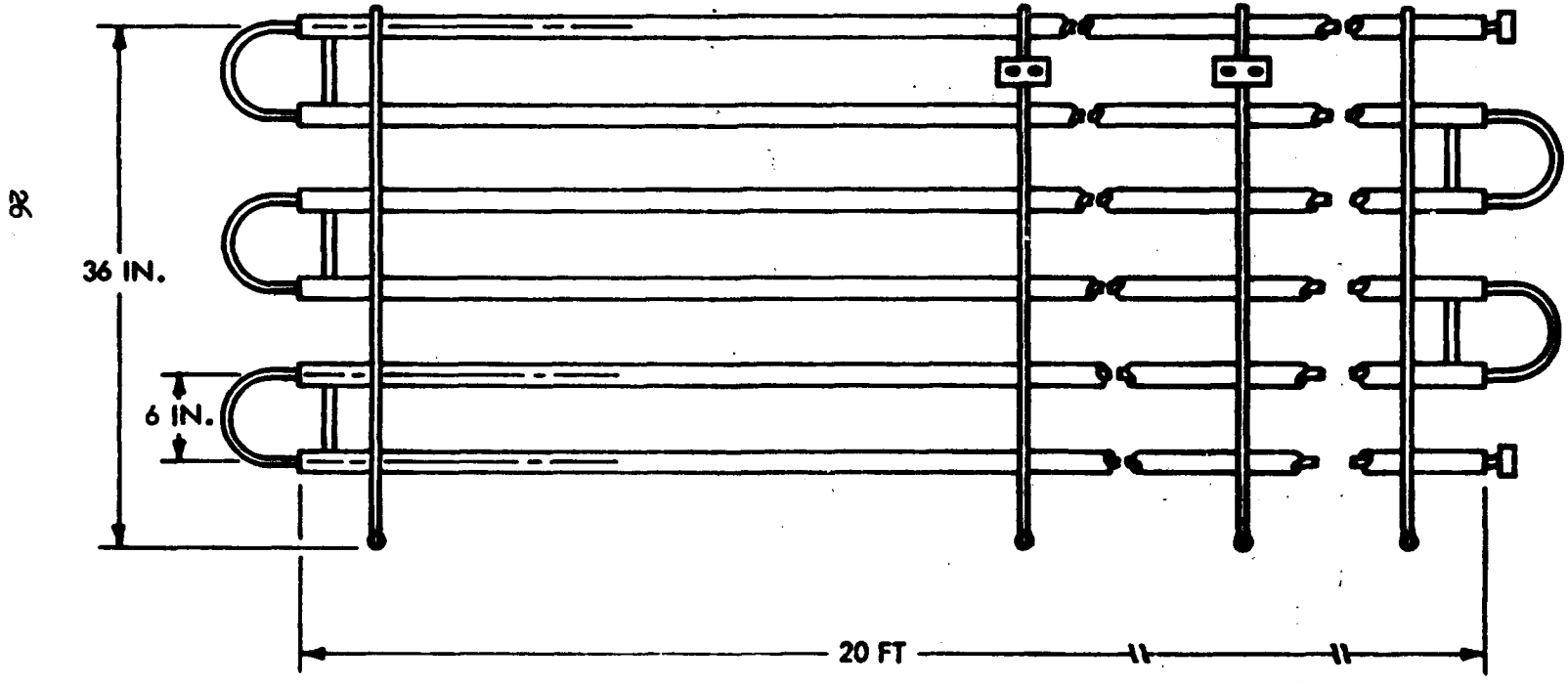


Fig. 8 Regenerative Heat Exchanger

and heater tube wall temperatures were calculated to be 1700°F and 920°F respectively for the worst case assuming radiation as the only heat transfer method.

Reactor - The reactor as shown by Figures 9 and 10 was based on the trickling tower principle as opposed to mechanical stirring of the contents. It had a 5 inch internal diameter and 60 inches internal height with an internal volume of 5 gallons. One inch pipe fittings in the top and bottom heads provided for tube connections. The vessel was rated for 5000 psig at 650°F. Five gallons were selected to provide contact times in the range of 10 to 30 minutes with corresponding sewage flows of 0.5 to 0.17 gpm. Contact times in this range were required to produce a feasible system design and seemed achievable based on laboratory test results of previous wet oxidation experiments. The reactor had four thermocouples that pierced the side wall in even intervals down the side of the vessel. Two 2.1 kilowatt 220 volt tape heaters wrapped around the vessel wall provided warmup and sustaining heat. An aluminum sheathed asbestos insulation jacket surrounded the reactor. The various reactor internal configurations used during the test program are described later in the report in the section dealing with breadboard system testing.

Air Compressor - The air compressor shown by Figure 11 was a three stage, 10 SCFM, 5000 psig, Model RIX unit, with water intercoolers and self-contained radiator cooling system. An inlet air filter and three outlet air driers were provided for cleaning the air supply. The compressor was controlled by a Barksdale pressure switch and electrical contactor. The accumulator connected to the compressor was a 10 cubic foot A. O. Smith double wall vessel rated for 6000 psi. It was provided with 6000 psi relief valve and drain valve. The compressor and accumulator like most of the breadboard system hardware were much larger and greater capacity then needed in order to accommodate a wide variety of test conditions. The compressor and accumulator were also on hand at Lockheed.

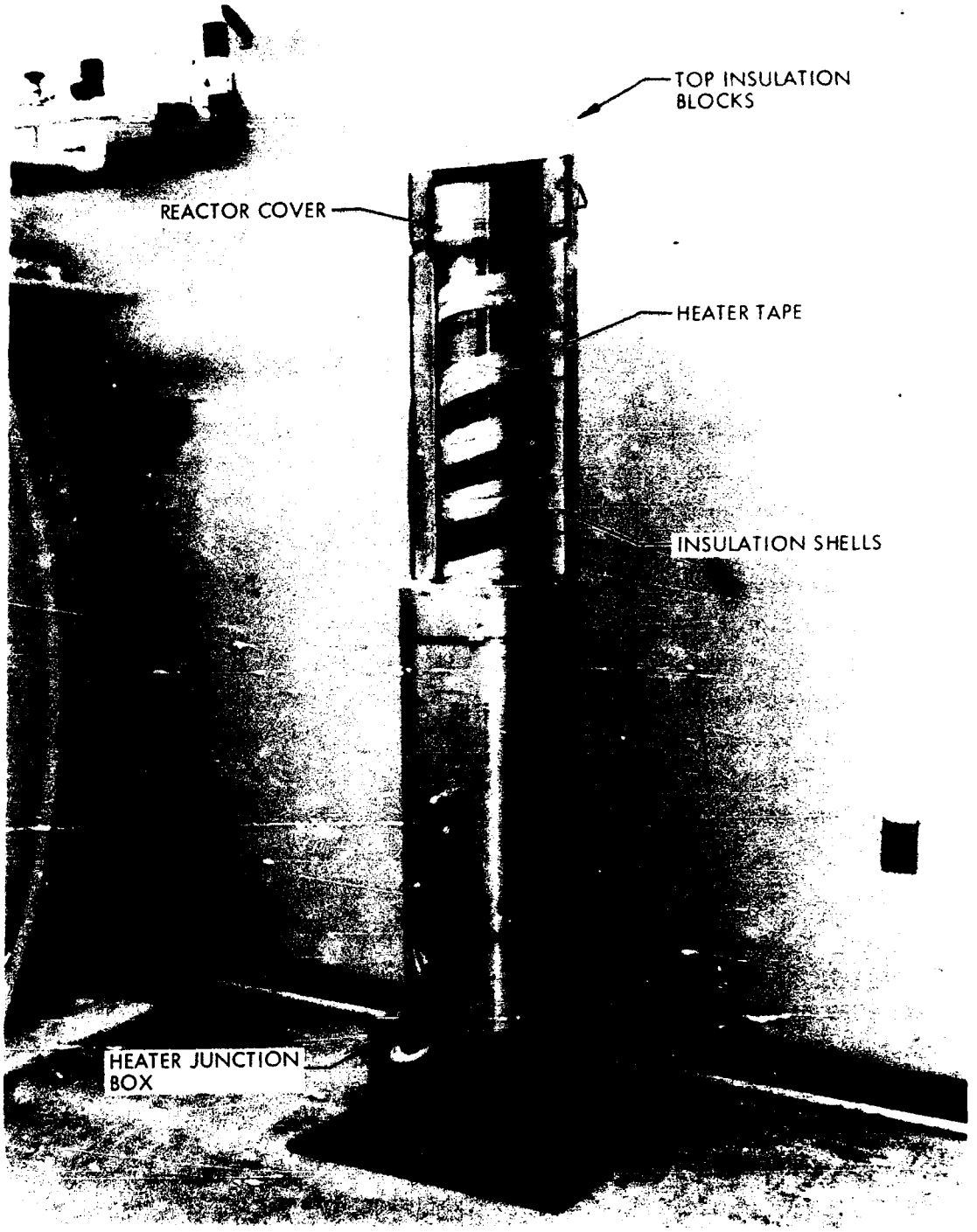


Fig. 9 Wet Oxidation Tower Reactor

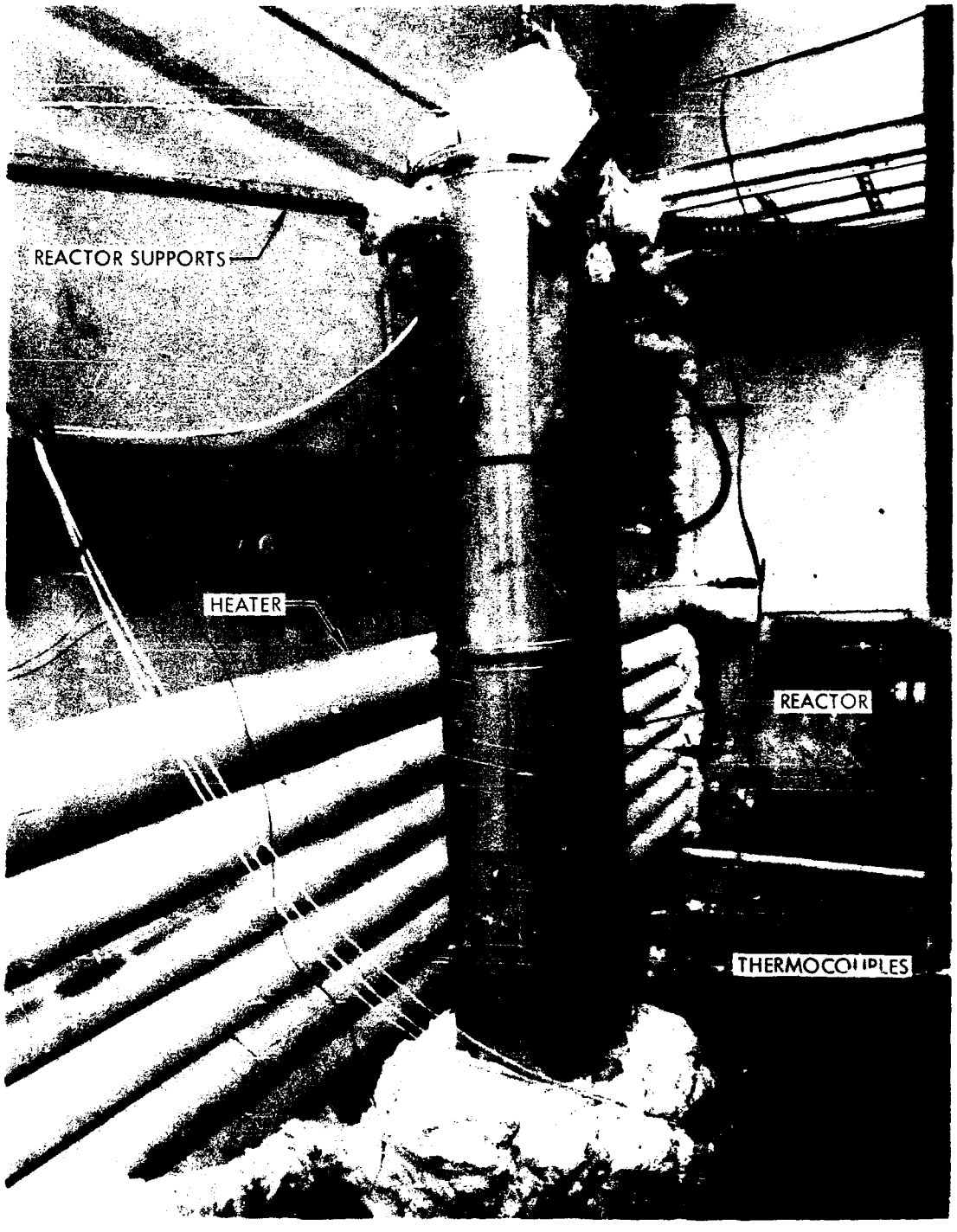


Fig. 10 Wet Oxidation Tower Reactor-Installed

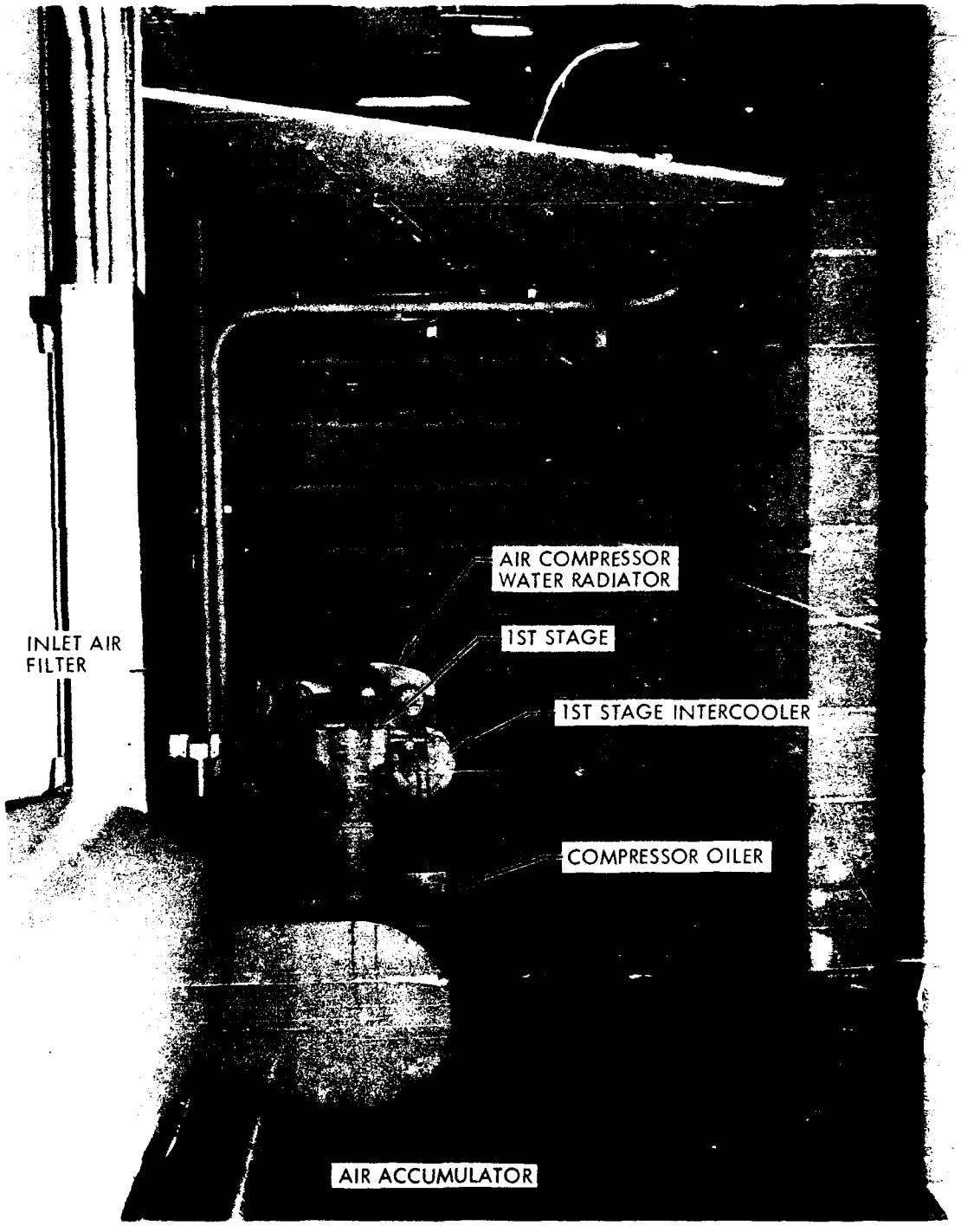


Fig. 11 Air Compressor

Air Flow Control Valves - The air flow controls (Figure 12) were selected to provide a wide range of controlled flows under a wide range of pressure conditions. Based on a maximum COD of 6500 mg/liter and a maximum sewage flow of 0.5 gpm a maximum air flow requirement of 1.65 SCFM was calculated.

$$\dot{V}_{air} = 0.51\ (COD)\ \dot{V}_s$$

where:

$\dot{V}_{air}$ = Volumetric Flow Rate of Air (SCFM)
COD = Chemical Oxygen Demand of Sewage (gm/liter)
$\dot{V}_s$ = Sewage Volumetric Flowrate (GPM)

$$\dot{V}_{air} = 0.51\ (6.5)(0.5) = 1.65 \text{ SCFM}$$

The flow control system was sized to deliver flows from 0.1 to 2.0 SCFM at pressures from 3000 to 5000 psig. The concept selected for flow control was to maintain a constant ΔP of from 200 to 500 psi across an adjustable metering flow valve with a digital handle for fine valve adjustment. Valve flow coefficients were calculated based on the following formula and extreme conditions:

$$C_v = \frac{\dot{V}_{air}}{22.7\sqrt{\frac{\Delta P(P_1)}{T}}}$$

where:

C_v = Valve Flow Coefficient
$\dot{V}_{air}$ = Volumetric Flow Rate of Air (SCFM)
ΔP = Valve Pressure Drop (psi)
P_1 = Upstream Pressure (psi)
T = Air Temperature (^oR)

C_v ranges were calculated for extreme and nominal cases:

$$C_v = \frac{0.1}{22.7\sqrt{\frac{200(5000)}{535}}} = .0001$$

$$C_v = \frac{0.97}{22.7\sqrt{\frac{500(3000)}{535}}} = .0008$$

$$C_v = \frac{2}{22.7\sqrt{\frac{200(3000)}{535}}} = .0025$$

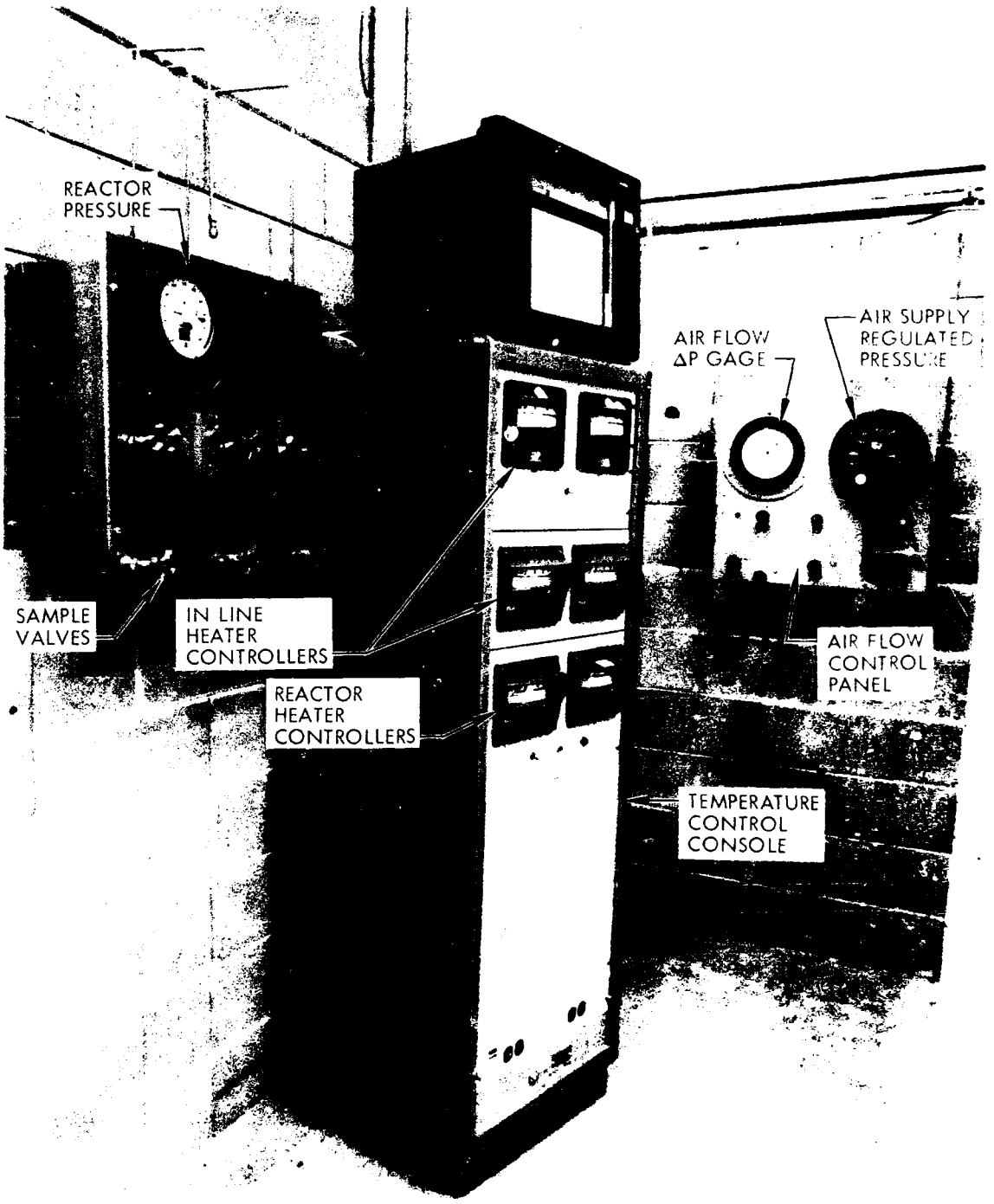


Fig. 12 Air Flow Controls

To cover these ranges and provide additional flow control capability two valves were included in the air system; one with a C_v between 0 and .0008 and one with a C_v between 0 and .008. The valves were adjusted to provide the desired air flow for each run by setting a given ΔP across the valve (generally 200 psi) and turning the valve stem until the desired flow was obtained on a flowmeter attached to the air panel bleed valve.

Cooler - The cooler was a 55 gallon drum through which city water was circulated. Two coils of 1/4" Schedule 80,316 stainless steel pipe 18 inches in diameter were immersed in the water to cool the effluents from 120°F coming out of the regenerative heat exchanger to ambient.

Back Pressure Regulator - The back pressure regulator was a gas dome loaded diaphram type regulator.

Recirculation Hot Pump - A certrifical pump capable of 15 psi pressure rise at 14 gpm and 650°F with a case pressure rating of 5000 psig was provided as a means of increasing flow through the reactor. If early tests of the packed tower reactor had shown the process was limited by lack of oxygen in solution or by inadequate liquid agitation the hot pump was available for use to recirculate liquid from the bottom of the tower back to the top for reprocessing. The pump was driven by a 2 horsepower, 220 volt, 3 phase motor acting through a belt and magnetic drive to eliminate a rotating pump shaft seal and to allow changing of pump flow by varying pump speed.

System Layout

The system arrangement in the Lockheed building 151 annex is shown by Figure 13. All high pressure sewage, effluent water, and high pressure air lines were located behind a protective wall except for the air flow controls. The toilets, urinal and garbage disposal were located on the mezzanine above the system. General location of the equipment behind the wall is indicated by Figure 13 and specific installation can be seen on photographs presented previously with the component descriptions. The input mixing tank, pump/grinder

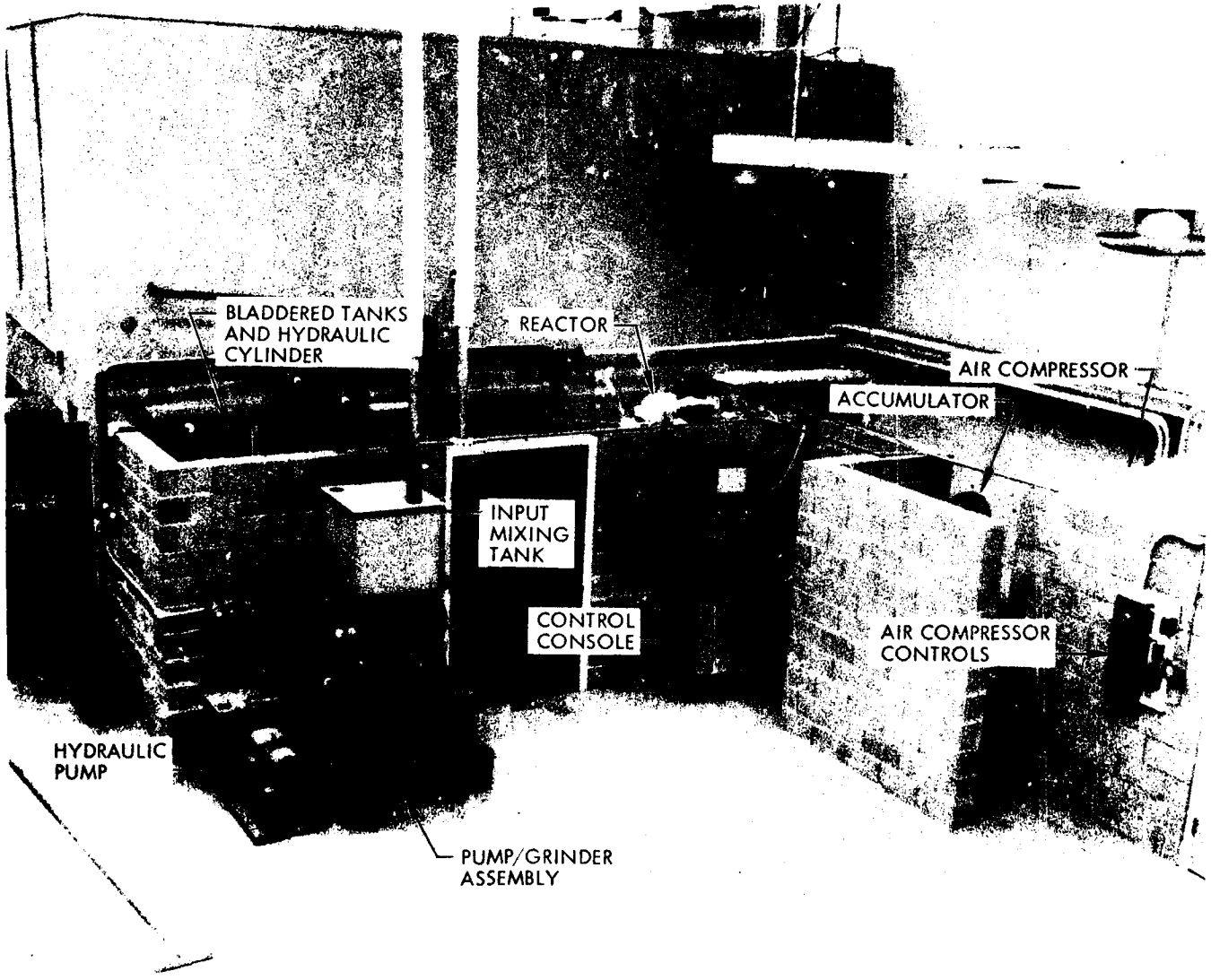


Fig. 13 Breadboard System Layout

assembly, hydraulic pump, sample valves, temperature control console, air flow control panel and air compressor control panel were located outside the protective wall because operator attention to these items was required.

TEST PLANS & PROCEDURES

Test Theory

The following three steps were recognized to be extremely important in the wet oxidation process:

Hydrolysis of solid materials
Mass transfer of oxygen from gas to solution
Reaction of oxygen with the hydrolized waste products

Of the above process steps, no definite answer could be made as to which was going to be rate controlling. Calculations of mass transfer requirements showed that it was feasible to transfer all required oxygen in a single pass through a packed tower having a height of less than three feet. Because of this, it was decided to start testing without using the recirculating hot pump originally planned for initial tests. In the event that the shipboard wastes were far more concentrated than anticipated, recycle of waste through the tower would have proven necessary to get the required oxygen into solution. Therefore, the hot pump was prepared for installation, but was scheduled for use only if initial tests proved it to be necessary.

The hydrolysis of raw wastes takes place at a slow rate under neutral or basic conditions. A review of the chemistry of wastes[10] showed that this rate could be significantly changed as conditions of pH and temperature were altered. For cellulose materials, the rate of hydrolysis is increased two orders of magnitude as acid (HCl) concentration is increased from 0.2% to 2%. A 50°F rise in temperature will increase the rate by one order of magnitude. Thus, both pH and temperature must be considered if a small system with short contact time is desired. Previous tests had also shown a strong effect of oxygen partial pressure on reaction rates. This was likely due to two factors; early tests were with concentrated slurries with oxygen demand requiring higher mass

transfer. Further, the higher partial pressure increases the availability of oxygen in solution, thus increasing the rate. The initial test program considered all of these variables.

In the preparation of the test plan, two reactor configurations were considered. These are shown in Figure 14. In configuration (a), the waste water first enters a hold tank at the top of the system, where waste hydrolysis occurs. It then passes into a packed tower where it is saturated with oxygen and chemical reaction takes place. It then flows from the reactor. The advantages of the flow path are the reduction in solids which will reduce the potential for tower clogging and the continuing supply of oxygen during the reaction phase.

It was planned that if the rate of reaction with oxygen proved limiting, configuration (b) would be adopted. In this case, the wastes are saturated with oxygen and then pass into a hold pot where hydrolysis and oxidation takes place. Recirculation is provided by the hot pump. Care must be taken in this scheme to select a packing which will not clog easily.

Test Sequence

The test sequence was based on the primary importance of temperature, acidity, contact time and oxygen partial pressure (total pressure). A series of tests to assess the performance of configuration (a) was planned with variations of these four key variables. A decision was then to be made to switch to configuration (b) or continue with configuration (a).

Estimates were made of the ranges of these key variables that should be investigated if a shipboard system is to be feasible. The ranges selected for test were as follows:

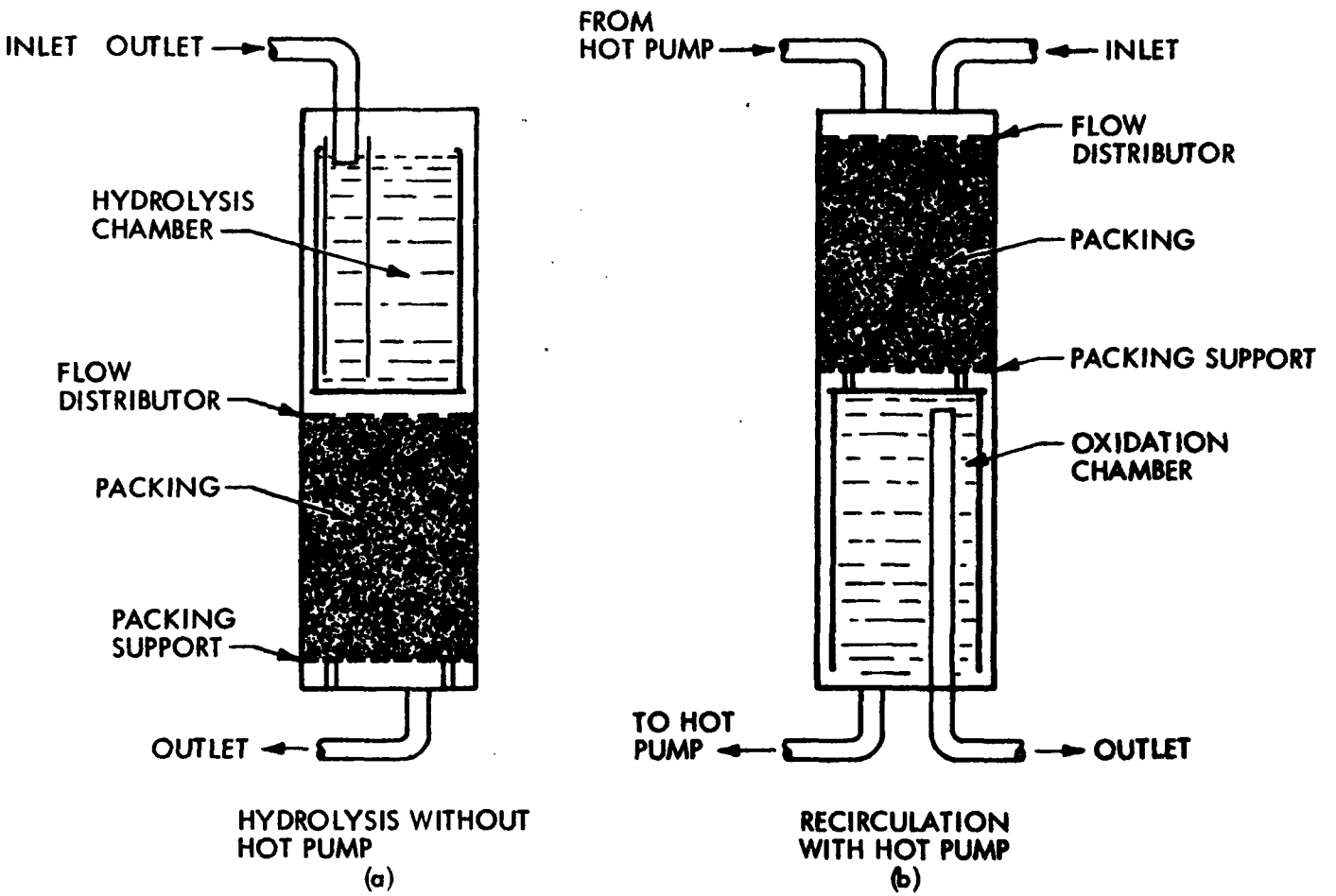


Fig. 14 Reactor Configurations

Temperature	450	550	650	(°F)
Acid (HCl)	1%	0.1%	Neutral	
Time	3	6	12	(Minutes)
Pressure	3000	3750	4500	(PSIA)

The approach selected to obtain the maximum useful optimization data was to determine the influence of each parameter individually holding all others constant. The initial operating point for all parameters was selected on the basis of previous studies and was a best estimate of an operating point which would yield a feasible system. The test program was planned to proceed in the following manner using a fresh water waste mixture.

1. Set total pressure at 4500 psia; contact time at 12 minutes; no acid needed.
   Run three temperatures; 450, 550 and 650°F.
2. Set acid at 0.1%.
   Run three temperatures; 450, 550 and 650°F.
3. Set acid at 1.0%.
   Run three temperatures; 450, 550 and 650°F.

It was planned that these data would demonstrate the influence of both acid concentration (pH) and temperature. On the basis of these data an operating temperature and acidity was to be selected to evaluate the effects of pressure and contact time. A check of these results was then to be made by running the best data point using sea water waste mixture.

The following runs were then to be made:

4. Set pH and temperature based on previous runs and P_{total} = 3000 psia.
   Run three contact times; 3, 6 and 12 minutes.
5. Set P_{total} = 3750 psia
   Run three contact times; 3, 6 and 12 minutes.
6. Set P_{total} = 4500 psia
   Run three contact times; 3, 6, and 12 minutes

Following these tests and based on the test results, a selection of configurations (a) or (b) was to be made. If the test results were promising the preferred configuration (a) was to be selected, if not the tests were to be repeated using configuration (b).

Making the assumption that the relations developed in the test program to this point had general validity, a tentative performance map was to be generated in the areas of interest to select a new estimate of the optimum operating point. This evaluation was to be based on the following criteria, which are listed in their order of importance.

1. No acid addition is desirable to limit logistic problems and a potential safety hazard.
2. Low contact time is desirable to limit reactor size and decrease system size.
3. Low pressure is desirable to minimize compressor power and reduce system weight.
4. Low temperature is desirable to reduce fuel consumption.

A new test series was then to be initiated to confirm performance at the new operation point and to develop more precise data in the region of the selected operating point. This was to be accomplished by selecting four specific operating points from the performance map. This test series would lead to selection of an optimum temperature, pH, pressure, and contact time. The effects of changes in BOD, waste input composition and salinity would then be checked, followed by chemical oxygen and catalyst tests. Figure 15 presents the logic flow for the test plan.

Breadboard System Operations

The tests of the breadboard system were run by following the procedures outlined below:

Startup - Sewage in accordance with the waste model was dumped into the garbage disposal and flushed into the hold tank. Water was added to the hold tank to

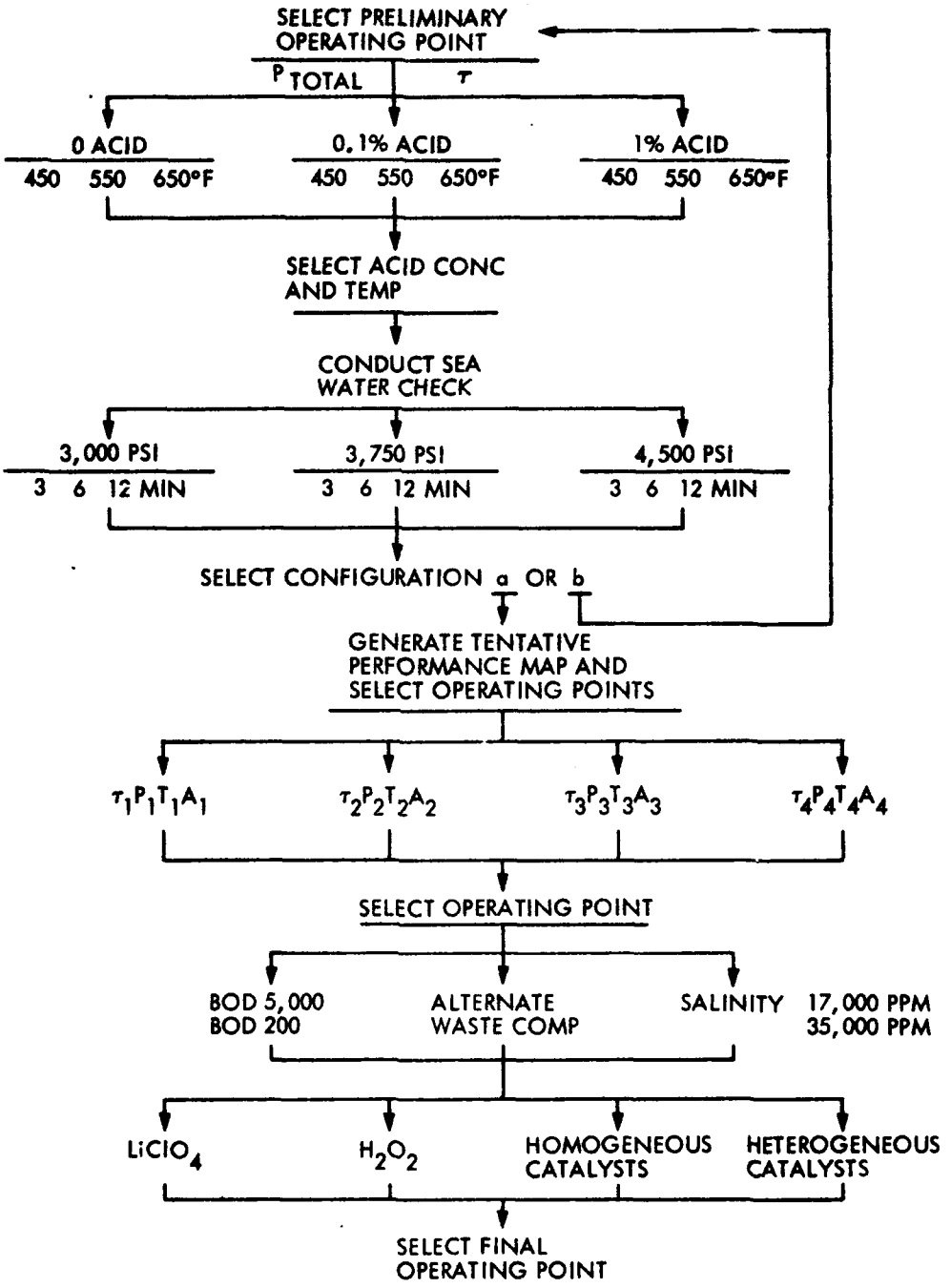


Fig. 15 System Test Logic

the full mark. The wastes were dumped into the pump grinder and it was energized. Pump outlet pressure was set to 60 psi and the bladdered tanks were filled with sewage. The hydraulic pump was then energized and the bladdered tanks pressurized to a pressure 500 psi above the reactor run pressure for the first test of the day. The air compressor and controls were energized and the reactor was pressurized to operating pressure. The reactor heater and the tube heater were energized and air flow initiated. The back pressure regulator was loaded to system pressure and the outlet valve was opened. When the line heater temperature reached 400°F sewage flow was initiated. Approximately two hours was required to reach 550°F operating temperature. The bladdered tanks required filling periodically during warmup and each time flow was interrupted for tank filling the in-line heater was de-energized to prevent overheating of the heater tube due to residual heat in the heater element.

After reaching operating temperature the system temperature, pressure and flow controls maintained steady state conditions except for periodic refilling of the sewage tanks. Adjustments to the test conditions were made by:

Changing reactor temperature by moving the in-line and reactor heater temperature controller set points.

Changing reactor pressure by adjusting the back pressure regulator setting and adjusting the air supply regulator.

Changing the contact time by adjusting the hydraulic pump speed.

Changing air flow by adjusting the flow control valves or ΔP.

Changing input slurry BOD or salinity.

CORROSION TESTING

There has been considerable question in the past with regard to metals selection for wet oxidation reactors. Previous experience with undiluted urine/fecal mixtures has shown that high temperature portions of the system require special metals to resist the combined effects of chloride ion, oxidation, and temperature. Hastelloy C-276 and Inconel 625 have been shown by corrosion testing to be superior metals for the fecal/urine application. The USCG requirement of sea water flush and the possible process advantages of acidifying to low pH added significantly to the problem of metals selection. The test plan and procedures defined a test program to evaluate selected corrosion-resistant metals for the USCG wet oxidation system.

Test Plan

The test plan covered metal selection, specimen preparation, reactor operation, specimen exposure and specimen examination.

Metal Selection - The following metals were selected for their known resistance to sea water, high temperature oxidation environments and formed the basis of the test program:

- Zirconium
- Titanium 6-4
- Titanium 6-2-1-1
- Titanium Commercially pure
- Titanium 1% Nickel
- Zircaloy
- Elgiloy
- Hastelloy C-276
- Inconel 625
- Titanium 2% Nickel
- Titanium .2 Pd
- Tantalum
- Tantalum 40% Columbium
- Columbium

Specimen Preparation - The metals were obtained in 4" x 9" sheets in thicknesses from .015 to 0.125" in the fully annealed condition. "U" bend specimens were prepared that mounted to the one liter stirred reactor head as shown in Figure 16.

The specimen were formed of 3/8" wide, 9" long strips and bent to form a tab at the top for mounting through ceramic washers to the support ring. A 1/2" diameter "U" bend was formed at the bottom 3 inches in length from bolt hole to bolt hole as measured along the "U" bend. A bolt through the legs of the "U" bend isolated from the specimen by ceramic spacers was tightened to produce a calculated 80 percent of yield in the "U" bend area. The support plate and specimens were arranged around the reactor body so that five specimens were exposed in one test run.

Reactor Operation - Figures 17 and 18 present a schematic and a photograph of the corrosion test setup. A one liter, 316 stainless steel, stirred reactor was used. The reactor provided an internal stirring shaft, thermocouple for process temperature control, air motor drive to obtain adjustable agitation speed, heaters for rapid warmup and temperature control, rupture disc, and process valves. For safety purposes, the reactor was placed in a sand bag area with controls and instrumentation remotely located. Air motor, gas supply, and pressure controls; cooling water supply and drain; air charging tank and valves; pressure gage and venting valve; and temperature controller and relays were provided. A high pressure and high temperature heater safety cut-off was also provided.

After mounting the specimens to the reactor head and tightening the bolts to obtain the desired "U" bend deflections, the reactor was filled with 460 cc of sea water/sewage mixture. The reactor head was installed and the reactor charged to 1000 psig with air. The air motor pressure was adjusted to obtain 1200 RPM agitator speed, the heater was energized, and the temperature controller set at 600°F. A log of process time, temperature, pressure, and agitator speed was maintained throughout the run.

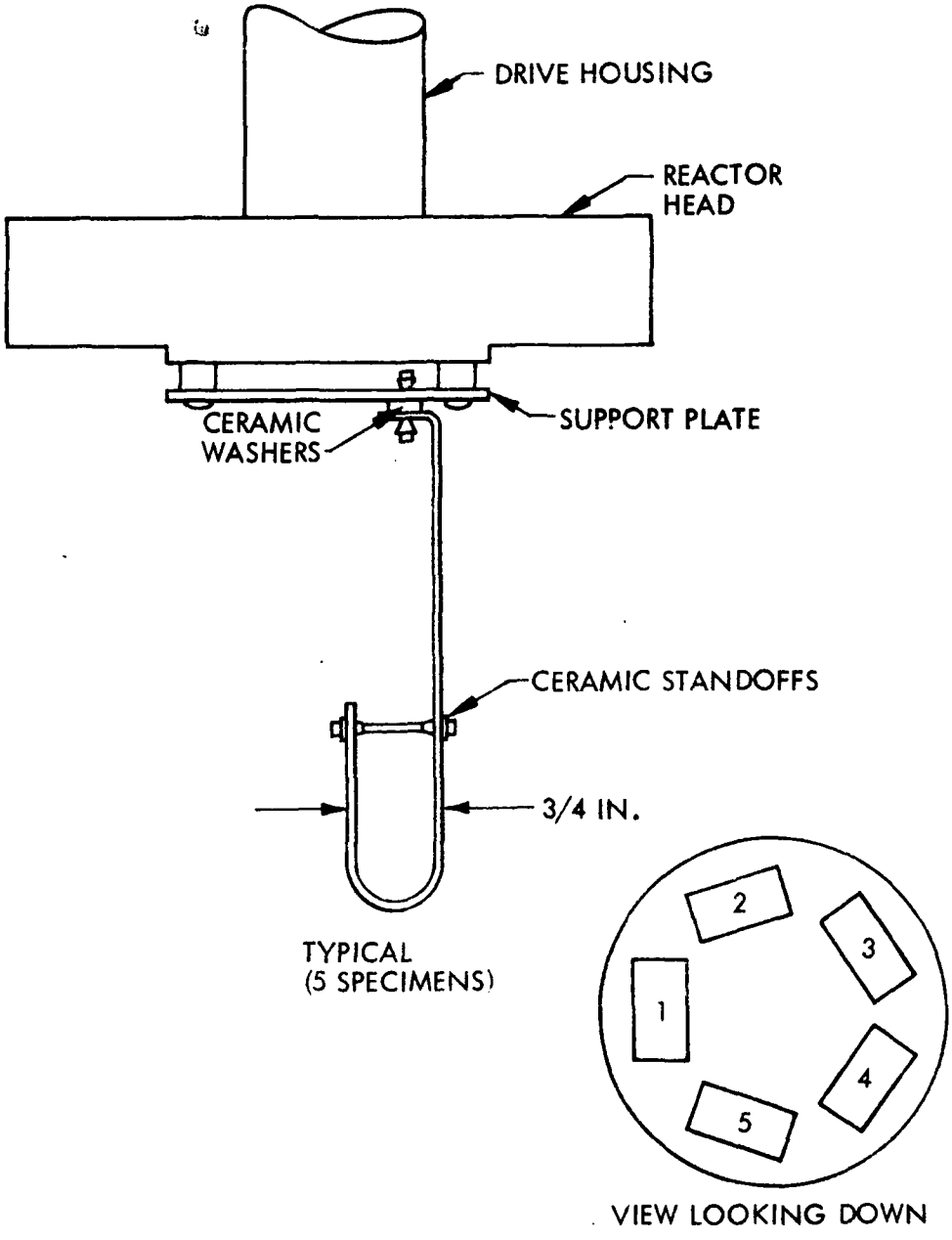


Fig. 16 Corrosion Specimen Support

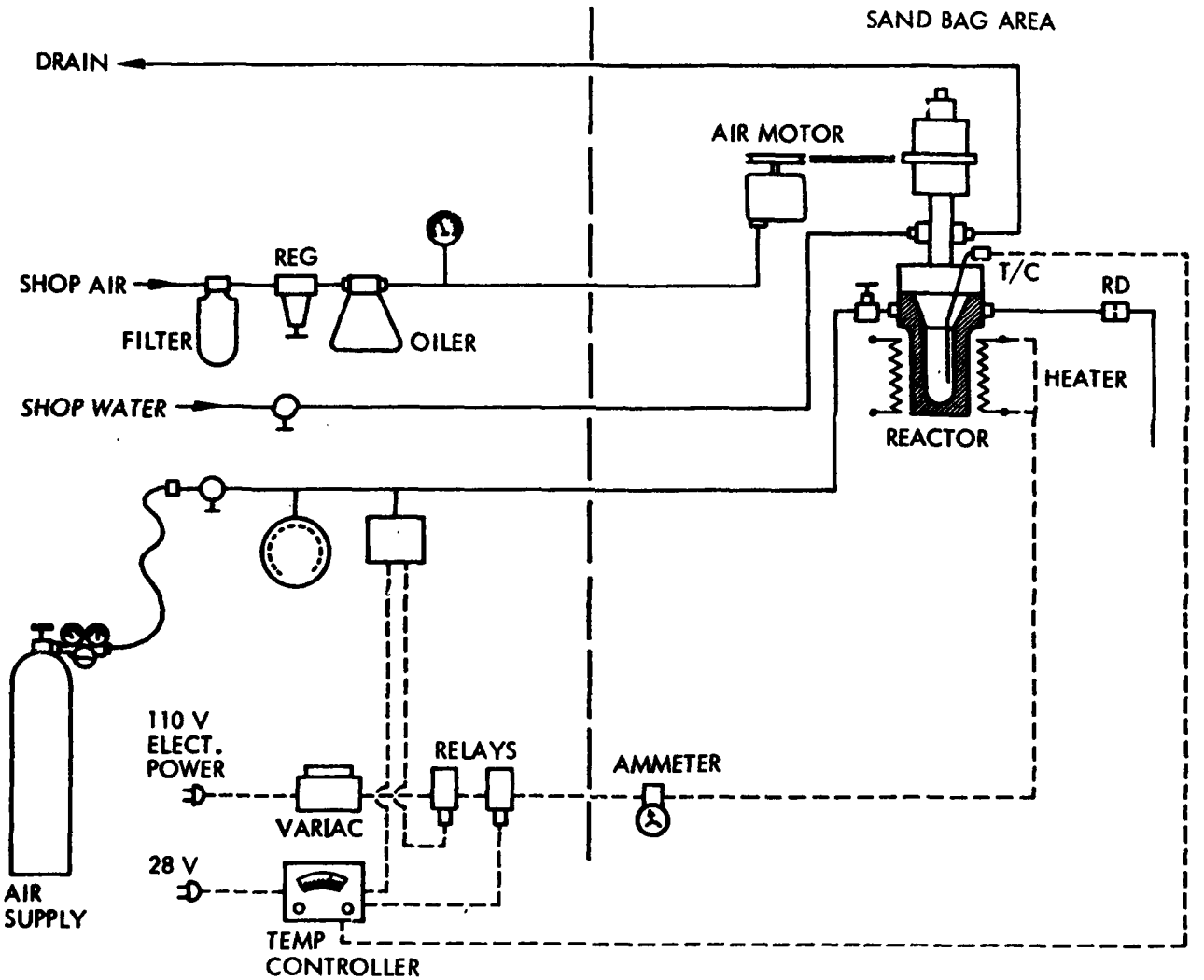


Fig. 17 Corrosion Test Schematic

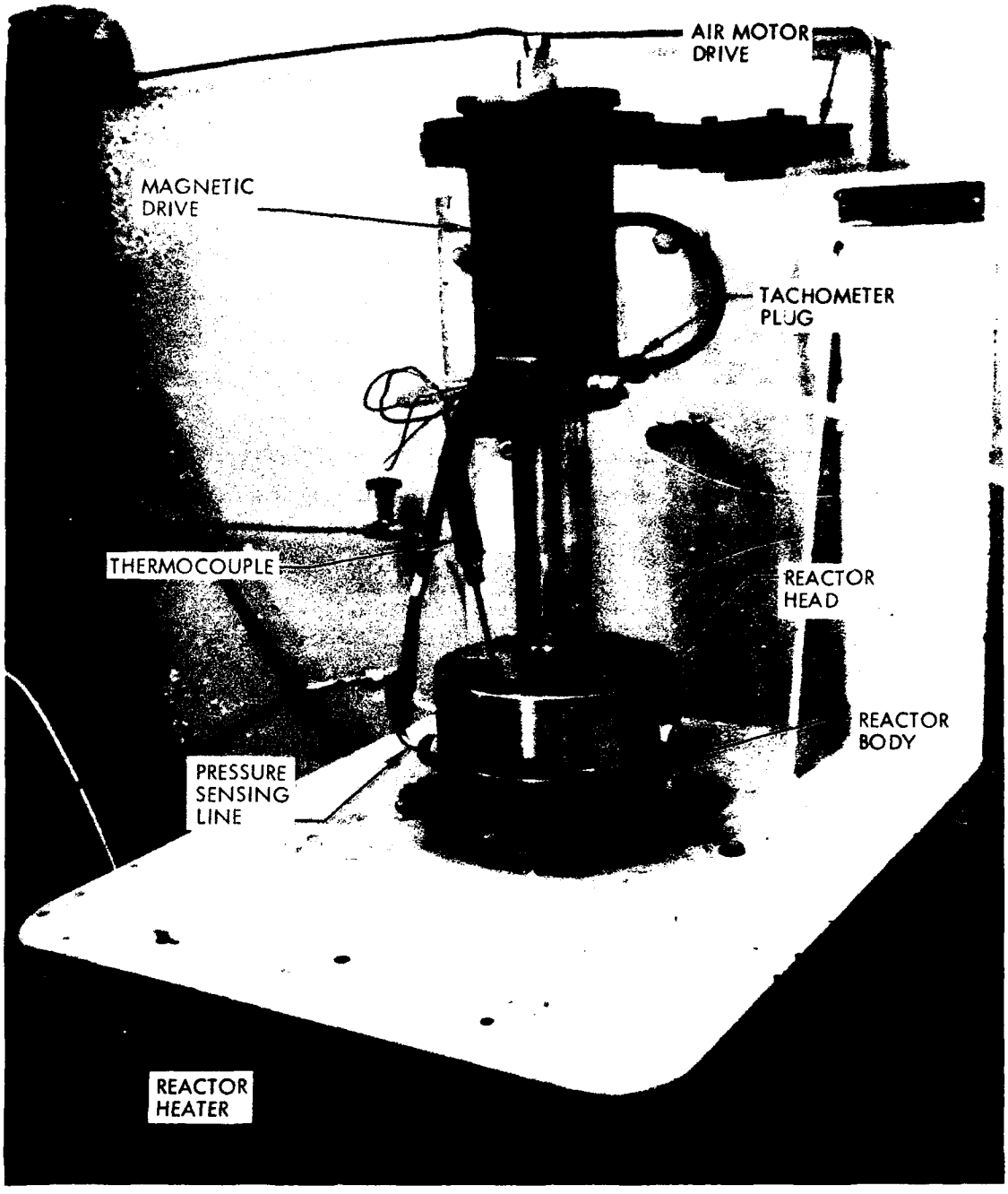


Fig. 18 Corrosion Test Reactor

Specimen Exposure - A maximum of five metals were tested in the reactor at one time. Three five-day screening tests were planned using a sea water sewage mixture of 500 mg/liter BOD acidified with hydrochloric acid to obtain a pH of 2 at a temperature of 600°F and total pressure of 3200 psig. Air was used as an initial charge gas. Based on a visual examination of all specimens run in the screening tests, five metals were selected for a planned eight-week exposure test under the same conditions. If none of the metals passed the screening tests, it was planned to repeat the tests without acid addition.

Specimen Examination - Specimen examination included visual and microscopic inspection. Dye penetrant, x-ray and fluoroscopic examination were planned if test results warranted.

Screening Test Results

Three screening tests were run with the fourteen metals grouped as listed below:

	Metal
Test No. 1	Titanium (6-4)
	Zircaloy 2
	Elgiloy
	Inconel 625
	Titanium (2% Nickel)
Test No. 2	Zirconium
	Titanium (6-2-1-1)
	Titanium (Commercially Pine)
	Hastelloy C-276
	Titanium (0.2% Pd)
Test No. 3	Titanium (1% Nickel)
	Tantalum
	Tantalum - 40% Columbium
	Columbium

The test specimens were prepared in accordance with Figure 19 and were installed on the reactor head as shown in Figure 20. Total deflections of the "U" bends specimens were calculated using the following formula:[11]

$$\delta = \frac{1}{r^3}\left[\frac{\ell^3}{3} + r\,\frac{\pi}{2}\,\ell^2 + \frac{\pi}{4}\,r^2 + 2\ell r\right]\frac{1.52\,f_b}{Et}$$

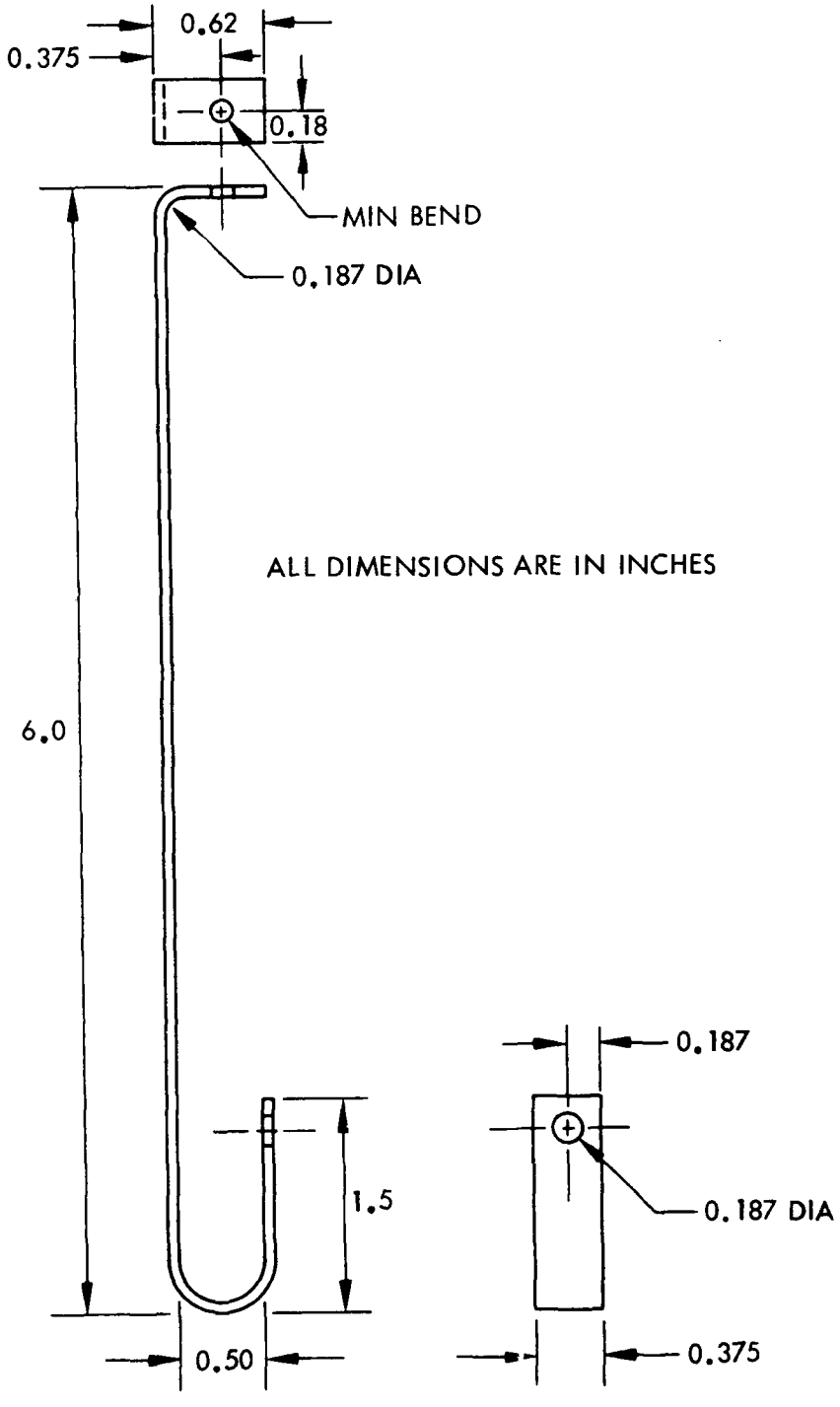


Fig. 19 Corrosion Test Specimen Configuration

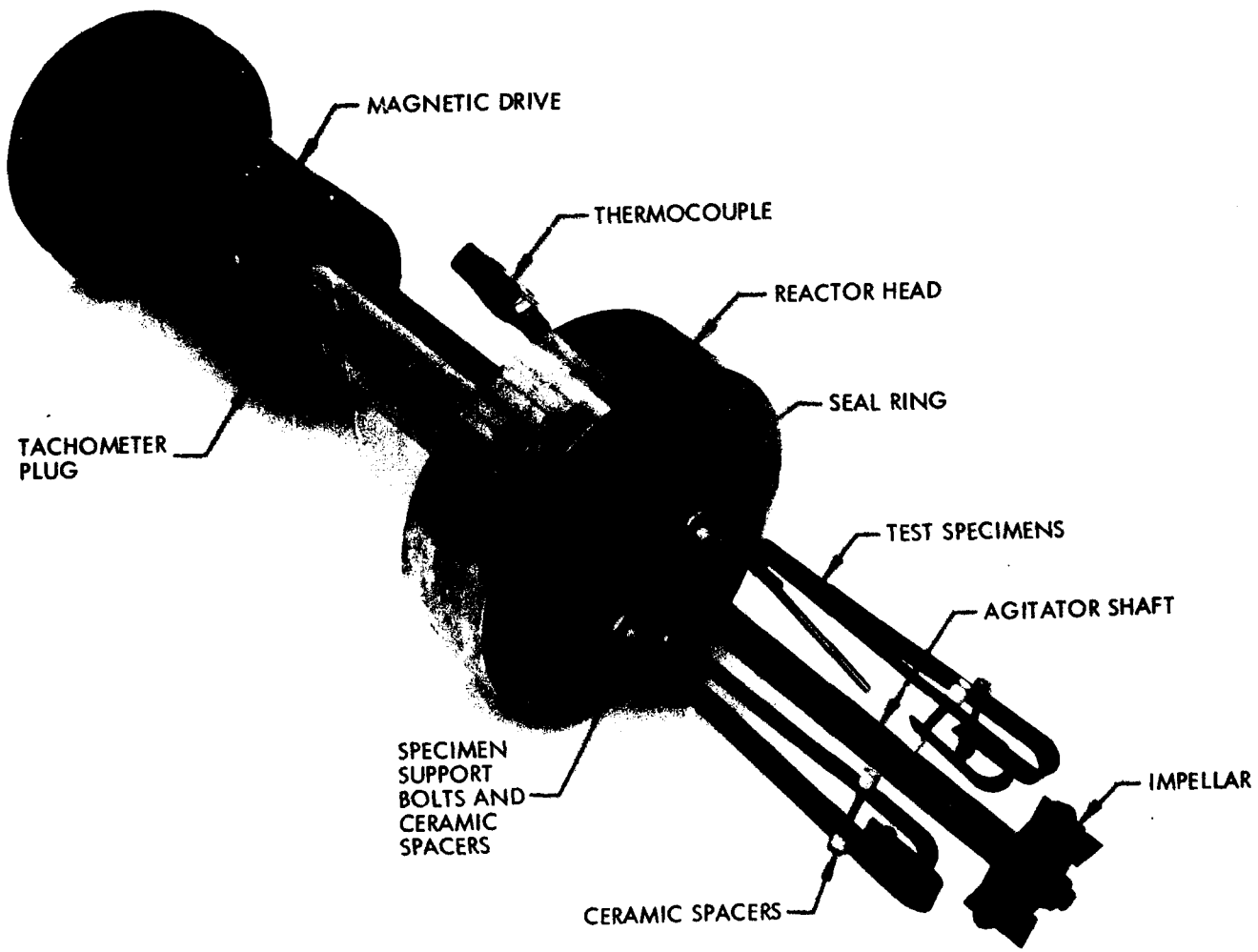


Fig. 20 Corrosion Test Specimen Installation

where:

δ = Total "U" deflection (inches)

r = "U" bend radius (inches)

ℓ = Straight length of "U" bend leg to bolt hole center (inches)

E = Modulus of elasticity of metal specimen (psi)

f_b = Desired stress level (psi)

t = Specimen thickness

Results of the calculations are listed as follows:

Specimen	Thickness (inches)	Deflection (inches)
Titanium (6-4)	0.062	0.069
Zircaloy 2	0.041	0.034
Elgiloy	0.024	0.337
Inconel 625	0.041	0.058
Titanium (1% Ni)	0.051	0.040
Zirconium	0.046	0.019
Titanium (6-2-1-1)	0.115	0.034
Titanium (CP)	0.045	0.023
Hastelloy C-276	0.093	0.024
Titanium (0.2% Pd)	0.050	0.033
Titanium (2% Ni)	0.063	0.032
Tantalum	0.016	0.045
Tantalum - 40% Columbium	0.016	0.075
Columbium	0.031	0.017

Screening Test No. 1 - Test No. 1 was run with a sewage/sea water mixture in accordance with the test plan. After two days of exposure the thermocouple failed resulting in automatic shutdown. Inspection of the reactor, thermocouple and specimens showed extensive corrosion. The top of the 316 stainless thermocouple was badly corroded and the body of the vessel was pitted to a depth of approximately .040". This reactor had been used in previous tests, so all of the corrosion did not result from this two-day exposure. Significant corrosion was observed on the Elgiloy and Inconel 625 Specimens. The Titanium 6-4 cracked in the "U" bend. The Zircolloy did not show evidence of corrosion except for some very strange surface bubbles or raised spots randomly distributed over the specimen. The 2% Nickel Titanium was corrosion free.

Although five days of testing had not been completed test no. 1 was not restarted because the two-day exposure did an excellent job of screening these five metals. In preparation for the second test, the reactor vessel wall was dressed down by removing .060" of material.

Screening Test No. 2 - The second screening test was conducted in accordance with the test procedures. After three days of exposure the test was terminated because of thermocouple failure. The Hastelloy C-276 was pitted along the edge of the specimen in the "U" bend area and the straight portion. The Zirconium specimen showed strange surface bubbles or raised spots very similar to those encountered with the Zircaloy 2 during the first test. The Titanium 6-2, Titanium .2 Pd, and commercially pure Titanium did not appear to have any evidence of corrosion.

Screening Test No. 3 - The third screening test was run in accordance with the test plan except that the test duration was shortened to three days to be consistent with the first two tests. All five specimens passed the test with no evidence of corrosion on any of the specimens.

The fourteen corrosion test specimens from the screening tests were examined in detail at 30X magnification to determine if microcracking was occurring in the bend radii of each specimen and to search for initiation of small pits not visible without magnification. The results of these examinations are listed below:

Hastelloy C

Approximately ten small pits .010" to .020" in size were distributed along the specimen edges and flat surfaces.

Zirconium

Blisters were located in the U-bend area. These were few in number and were no more than .005" high x .020" to .030" in diameter. No corrosion products were visible.

Inconel 625

Extensive pitting with 5 to 10 pits per cm^2. No cracking visible.

Elgaloy

Numerous cracks over length of specimen.

Zircaloy-2

Blisters along edge with white corrosion products barely visible at 30X.

Titanium 6AL-4V

Severe pitting and crevice corrosion with stress corrosion cracking at U-bend.

Titanium C.P., Titanium 6-2-1-1, Titanium 0.2 Pd, Titanium 2% Ni, Titanium 1% Ni Tantalum, Tantalum 40% Columbium, Columbium

No evidence of corrosion

Based on these test results and because only five specimens could be tested, it was decided to run the long duration test using commercially pure titanium, 2% Nickel Titanium, 0.2% Pd Titanium, Pure Tantalum, and Tantalum - 40% Columbium. Pure Columbium was eliminated because of it's very low strength and poor machinability. Titanium 6-2-1-1 was eliminated because of concern over the long term corrosion resistance of aluminum alloyed Titanium, and 1% Nickel Titanium was assumed to be very similar to the 2% Nickel Titanium.

Long Duration Test

Prior to initiating the long duration corrosion test several attempts were made to find a suitable coating for the reactor or to use cathodic protection of the reactor to reduce problems of reactor corrosion. Polyimide and flame spray coatings of Titanium and mixed metal oxides were tested, but all separated from the vessel walls and internal parts when exposed to the wet oxidation environment. It was decided to conduct the long duration test with a loose fitting Titanium liner, shaft and impellar installed in the corrosion test reactor. The test was started on June 8, 1973, and by July 19, 1973 approximately twenty-five days of testing at temperature had been completed but not without considerable difficulty. Leaks in the tubing connected to the reactor head and failure of the thermocouple, probably caused by acid vapor attack, caused numerous shutdowns. Test durations were as follows:

Test Duration (Days)	Reason for Test Interruption
2	Tube Leak
1 1/2	Tube Leak
5	Thermocouple Failure
9	Thermocouple Failure
1	Tube Leak
2	Tube Leak
1 1/2	Tube Leak
1 1/2	Tube Leak
1	Tube Leak
1 1/2	Thermocouple Failure
25	

The test was terminated because of the difficulty of maintaining the reactor in operation and the fact that after approximately twenty-five days of testing none of the specimens showed any evidence of corrosion. Although it was desired to test for a longer period of time it was felt that sufficient evidence of corrosion resistance had been demonstrated to allow selection of a reactor material with a high level of confidence that it would withstand the wet oxidation environment for long periods of time.

Based on the corrosion test program, commercially pure Titanium was selected as a preferred reactor metal. Tantalum and Tantalum/Columbium were eliminated because of cost and poor machinability. The higher strength alloys of Titanium were eliminated because of concern over the long term attack of the alloy constituents.

Fresh Water and Partial Sea Water Tests

During the preliminary design, both fresh and salt water flush concepts were considered, and the requirement for acid addition was eliminated.

Hastelloy C-276 was tentatively selected as best for a fresh water flush system, and Titanium was selected for a salt water flush system. In order to verify these choices, a series of additional corrosion tests were run.

Five metals were evaluated in both a fresh water - sewage mixture, and a 50% salt water - 50% fresh water - sewage mixture. 50% fresh and 50% sea water represents the anticipated concentration of salt on a vessel using sea water for toilet and urinal flush and fresh water for all other shipboard water uses. No acid was used in either test. As in previous tests the environment was air at 600°F and 3200 psi total pressure. Planned test duration was 5 weeks.

The metals evaluated were Hastelloy C-276, Hastelloy C-4, 316L stainless steel, Carpenter 20-B, and commercially pure Titanium. They were fabricated into U-bend specimens and stressed to the same level as in the previous tests. The specimens were mounted to the reactor head as shown in Figure 20.

The fresh water test was terminated at the end of the five week period. None of the specimens showed any signs of corrosion.

The salt water test was terminated after 219 hours due to mechanical malfunction of the reactor. At this time all specimens except titanium showed some corrosion so the test was terminated. The Hastelloy C-276 and C-4 experienced significant pitting, while the Carpenter 20-B had severe pitting and corrosion. In some areas the thickness was reduced up to 50%. The corrosion, however, did not appear to be stress related, as it was uniform over the specimen. The 316L specimen had severe corrosion and broke at the bottom of the U-bend, the point of maximum stress. Corrosion of this specimen appeared to be stress related, as corrosion elsewhere was slightly less severe.

BREADBOARD SYSTEM TESTS

Reactor configuration (a) as presented by figure 14, i.e., a two gallon hydrolysis pot on top of burl saddle packing was tested in accordance with the test plan. Runs 1 through 6 investigated the effects of temperature and acid concentration on reduction in TOC with a fixed total pressure and contact time. Because the acid attacked the 316 stainless steel breadboard system plumbing to a greater degree than anticipated it was decided to postpone the higher 1% acid runs until later in the program. Runs 7 through 13 investigated the effects of system total pressure and contact time in the reactor holding system temperature at 650°F and acid concentration at zero. 650°F and 4500 psi total pressure were used early in the program to identify promising candidates with the philosophy that if high percent reduction in TOC could not be achieved under these conditions then the particular reactor configuration, catalyst, or set of reaction conditions being tested did not warrant further work to reduce the system operating pressure and temperature. This philosophy was used throughout the program, i.e., screen concepts to find promising approaches under extreme temperature and pressure conditions and then work to reduce the pressure and temperature.

Table 4 presents the results of the first thirteen breadboard system runs. Air flow on all breadboard system tests was held constant at 1 SCFM.

Table 4

Breadboard System Test Results - Runs 1 through 13

Run No.	Temp. (°F)	Pressure (PSIG)	Acid Conc. (%)	Contact Time (min)	TOC (mg/liter) Inlet	TOC (mg/liter) Outlet	% Reduction
1	650	4500	0	12	360	36	90
2	550	"	0	"	357	74	79
3	450	"	0	"	311	138	56
4	650	"	0.1	"	322	17	95
5	550	"	0.1	"	540	68	87
6	450	"	0.1	"	538	122	77
7	650	4500	0	6	315	64	80
8	650	4500	0	3	370	66	82
9	650	3000	0	12	298	54	82
10	650	3000	0	6	325	66	80
11	650	3000	0	3	350	73	79
12	650	3750	0	3	327	66	80
13	650	3750	0	6	-	64	80*

*Based on Inlet TOC of Run 12

BOD measurements were made on several of these runs and are listed below in Table 5.

Table 5

Comparison of TOC and BOD Measurements

	TOC (mg/liter)			BOD (mg/liter)		
Run No.	Inlet	Outlet	% Red.	Inlet	Outlet	% Red.
1	360	36	90	442	51	88
3	311	138	56	465	209	55
5	540	68	87	-	86	84*
6	538	122	77	555	105	81
7	315	64	80	522	65	88
8	370	66	82	503	83	83

*Based on Inlet BOD of Run 6

Figure 21 is a plot of the effects of temperature and acid concentration on percent reduction in TOC. The effects of pressure and contact time were not plotted because no significant change was noted in the range of variables tested with percent reduction in TOC running around 80 for all runs. As a result of these runs it was concluded that:

As expected, temperature had an important influence on percent reduction in BOD

Acid addition significantly increased reduction in BOD at lower temperatures

Contact times from 3 to 12 minutes had very little effect on performance

Pressures between 3000 and 4500 psi had very little effect on performance

Although the results with the packed tower reactor were encouraging, the required 90 percent or greater reduction in BOD had been achieved only under the highest temperature, pressure and/or acid condition. It was decided to evaluate two additional reactor configurations. Figure 22 presents the reactor configuration tested during runs 14 through 18. It eliminated the hydrolysis pot and utilized a fully packed tower, gas phase continuous, reactor. This configuration was selected to determine whether or not oxygen getting into solution was limiting the process, because it provides the greatest area for mass transfer of the oxygen into the sewage water trickling through the packing. Figure 23 presents the reactor configuration tested in runs 19, 20, and 21. It provides a mass transfer packing at the top of the reactor followed by an oxidation pot at the bottom. The oxidation pot provided much greater hold time in the reactor than that achieved in the fully packed tower. Tables 6 and 7 present the results for these two reactor configurations.

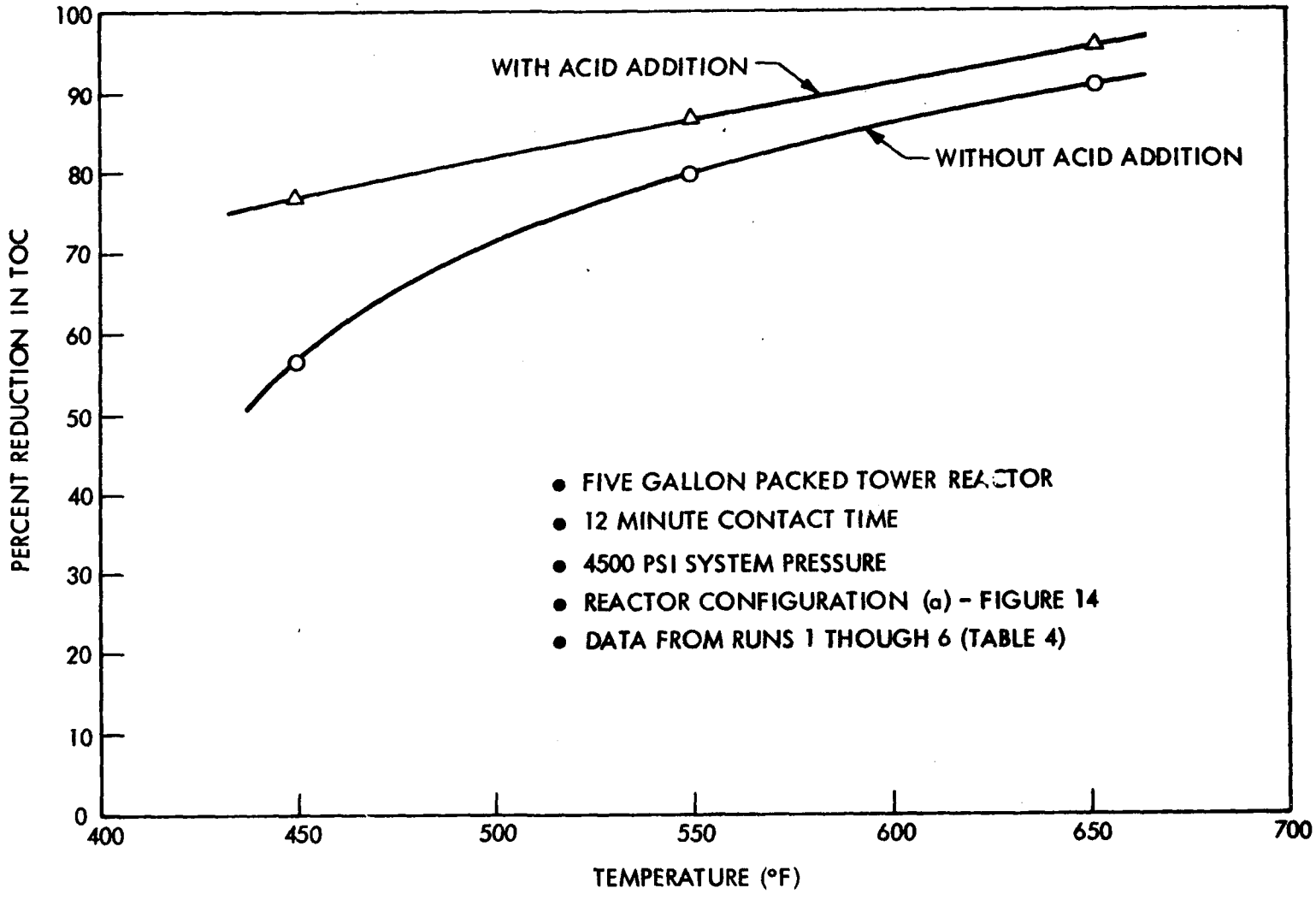


Fig. 21 Effect of Temperature and Acid Addition

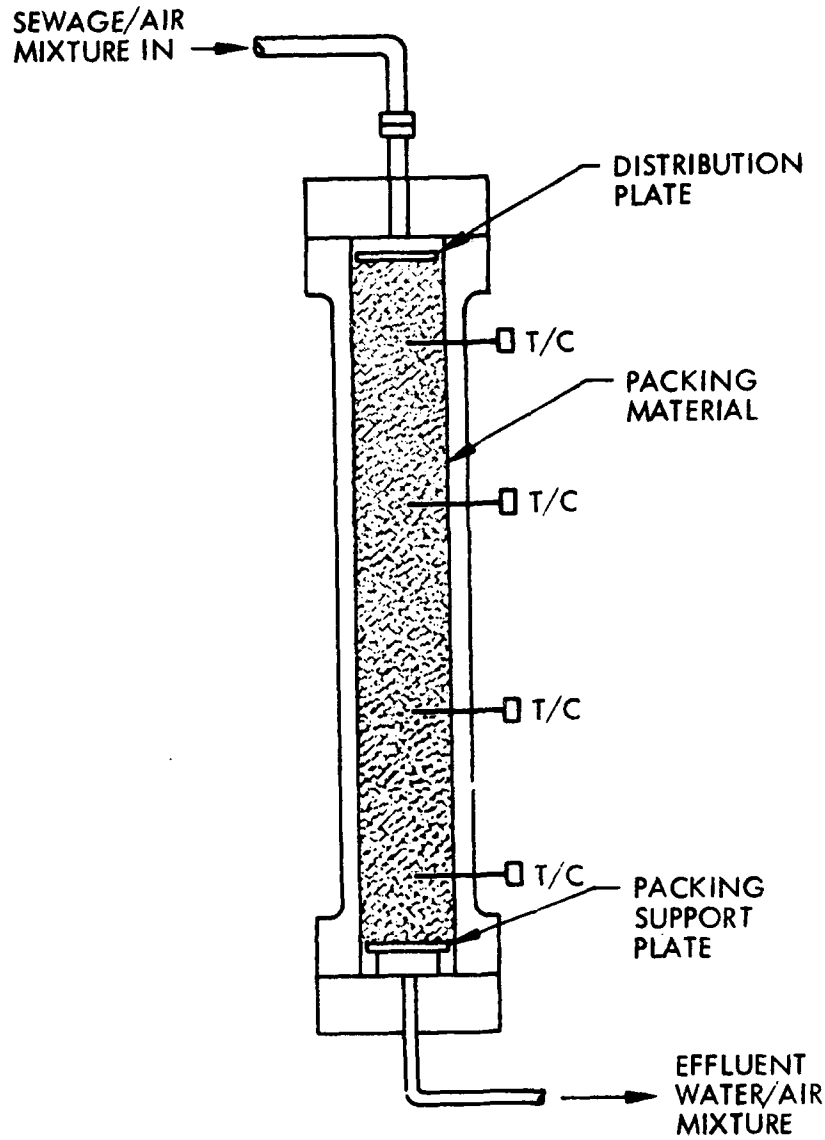


Fig. 22 Packed Tower Configuration for Runs 14 through 18

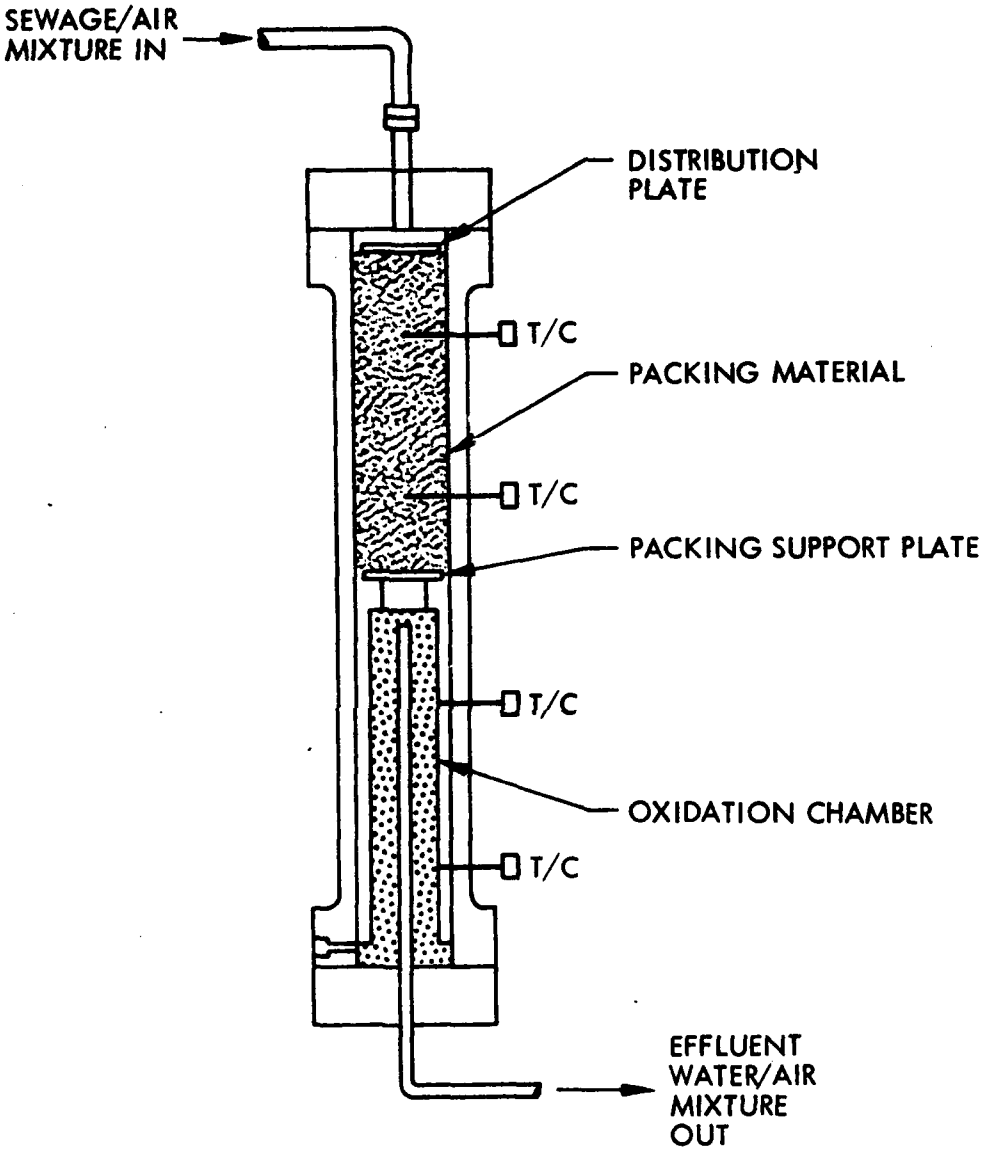


Fig. 23 Packed Tower Configuration for Runs 19, 20 and 21

Table 6

Gas Phase Continuous Reactor Performance

Run No.	Temp (°F)	Pressure (PSIG)	Acid	Slurry Flow	TOC (mg/liter) Input	Output	% Red.
14	650	4500	0	1/6 GPM (12 min*)	403	51	87
15	650	4500	0	1/3 GPM (6 min)	295	90	69
16	650	4500	0	2/3 GPM (3 min)	375	82	78
17	650	3000	0	2/3 GPM (3 min)	369	115	69
18	650	3000	0	1/6 GPM (12 min)	406	87	81

*Contact time is difficult to determine in fully packed towers - these flows correspond to the listed contact times for the first reactor configuration.

Table 7

Packed Tower Plus Oxidation Pot Reactor Performance

Run No.	Temp (°F)	Pressure (PSIG)	Acid	Contact Time (min)	TOC (mg/liter) Input	Output	% Red.
19	650	4500	0	12	402	64	84
20	650	4500	0	6	355	66	81
21	650	4500	0	3	359	73	80

Performance did not vary significantly with any of the three reactor configurations tested. Therefore it was concluded at this point in the program that a means of measuring the efficiency of the packed tower reactor was needed, i.e., perhaps 80 to 90 percent was all that was possible even under ideal conditions of oxygenation and agitation. Severe corrosion of the breadboard 316 stainless steel system was also encountered during the three acid runs, and an attempted sea water run following Run 13. For these reasons it was decided to conduct fresh water comparison runs as well as acid and salt water runs in a batch type, Titanium lined, one gallon stirred reactor. The stirred batch reactor provided the best possible conditions for oxidation and was used as a measure of the efficiency of the packed tower reactor.

Injection Reactor Tests

A one gallon, stirred reactor rated for 650°F, 5000 psi service with a loose fitting Titanium liner and Titanium stirring shaft and impellar was procured and installed in the laboratory adjacent to the breadboard system. Figures 24 and 25 present photographs of the reactor installation and the control panel, respectively. Figure 26 presents a schematic of the test setup. The reactor drive was powered by an air motor and tachometer to allow a wide range of speed control. The air motor was connected to a shop air supply and regulator. A sample injection system was used so that the reactor could be brought to temperature by the 3 KW electrical heater and temperature control circuit prior to injection of the wastes. The injection system consisted of a 3/8" diameter injection line, two 90 cc tubes with removable end caps for charging, an injection valve and an air supply valve. A separate reactor air pressurization line was provided with a vent valve, pressure gage and high pressure cutoff switch connected. A 1/8" O.D. sample line was connected to an internal sample tube that was routed through the reactor head to the bottom of the vessel. The sample line passed through a cooling coil immersed in a can of water to two high temperature sample valves.

The reactor was operated in the following manner:

The reactor liner was filled with 1320 cc of tap water.

The heater was energized.

Pressure on the air motor was adjusted to provided agitator speed of 1200 RPM.

One charging chamber was filled with 90 cc of concentrated Coast Guard wastes. (Waste model per Table 2, page 6)

The other charging chamber was filled with 90 cc of rinse water.

The reactor was heated to operating temperature.

The reactor pressure was adjusted to 300 psi below final operating pressure.

The sewage and flush water were charged into the reactor using high pressure air by opening the gas and fluid charge valves.

Samples were taken as desired (typically at 1, 3, 6, 12, 30 and 90 minutes after injection).

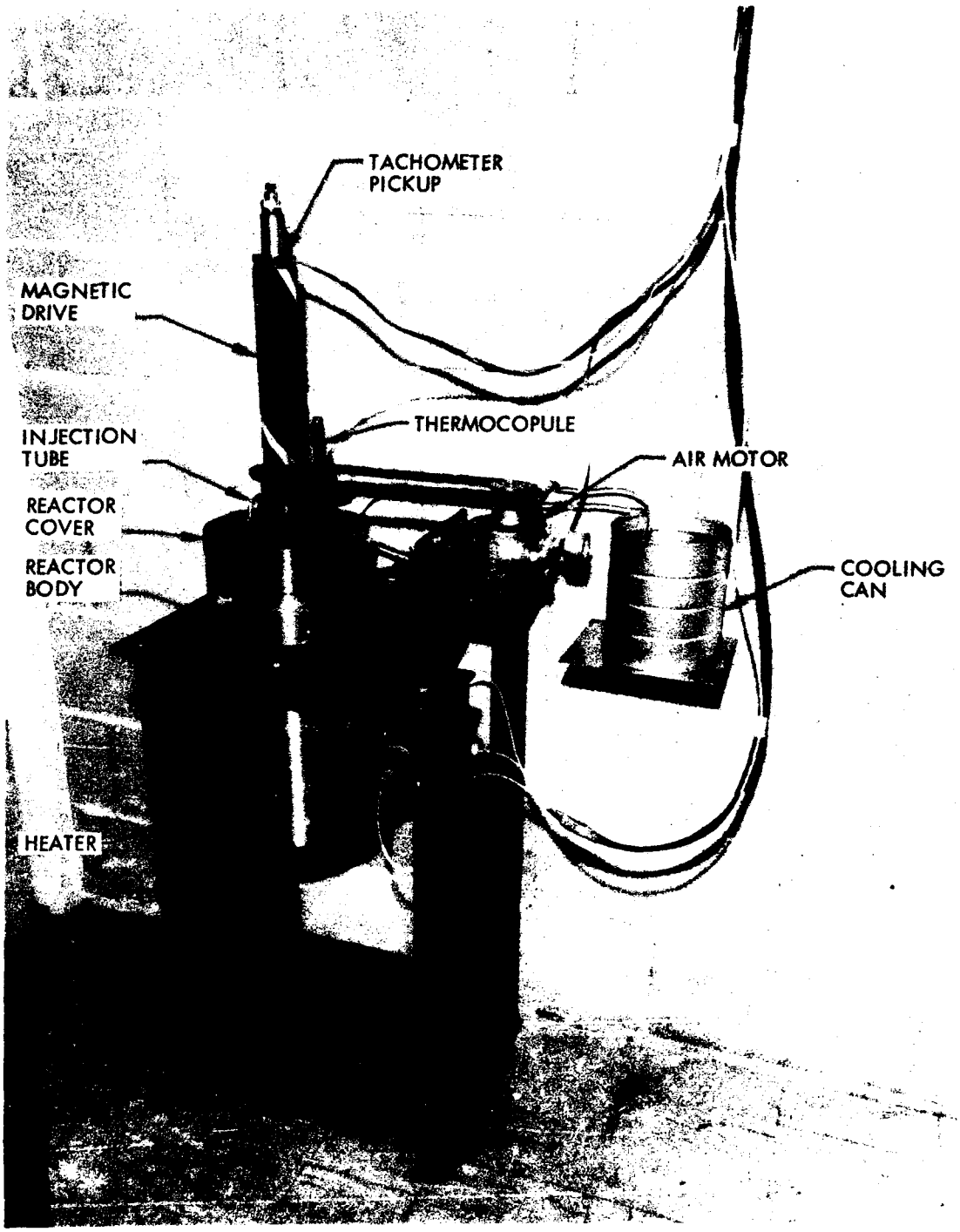


Fig. 24 One Gallon Injection Stirred Reactor

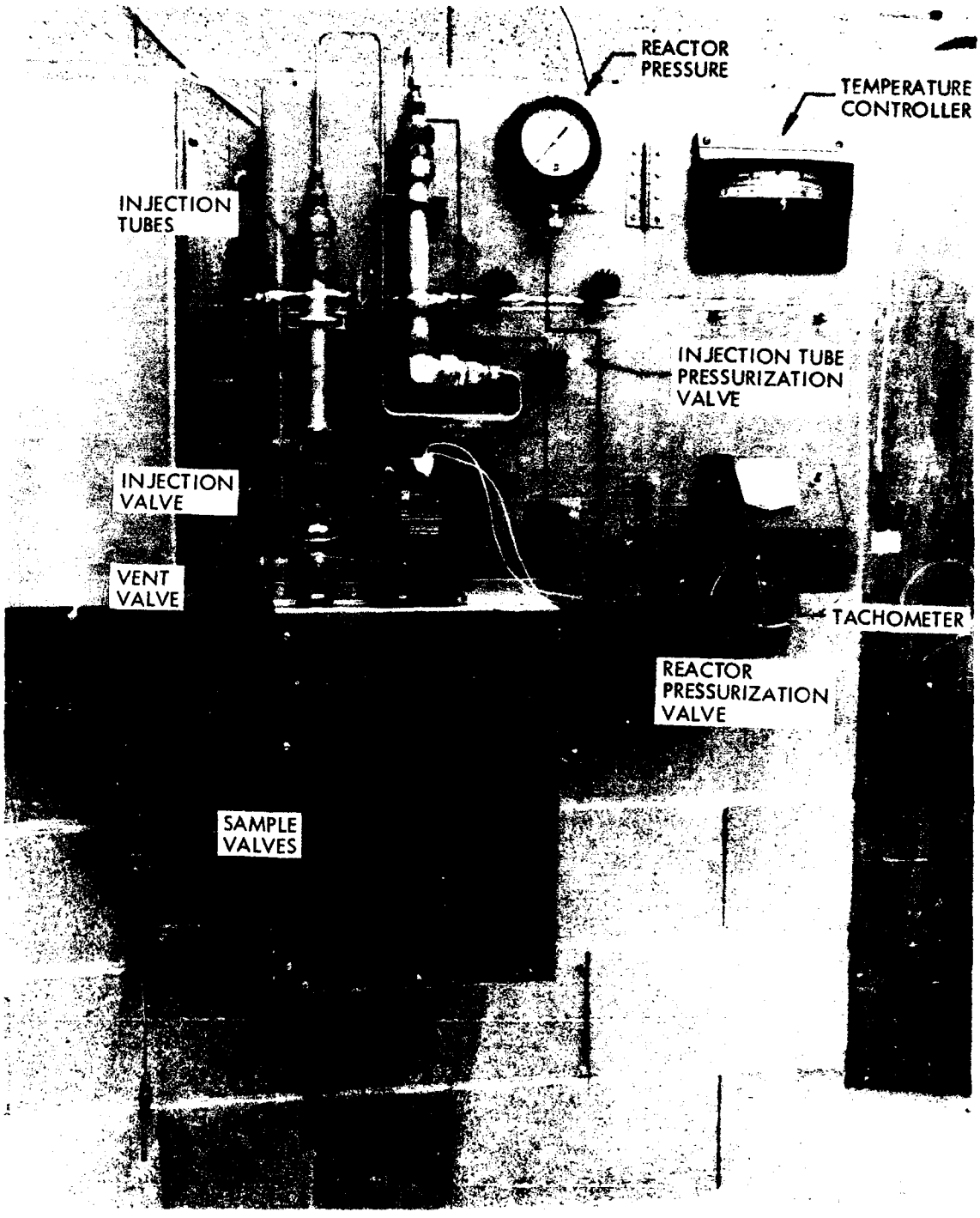


Fig. 25 One Gallon Injection Stirred Reactor Control Panel

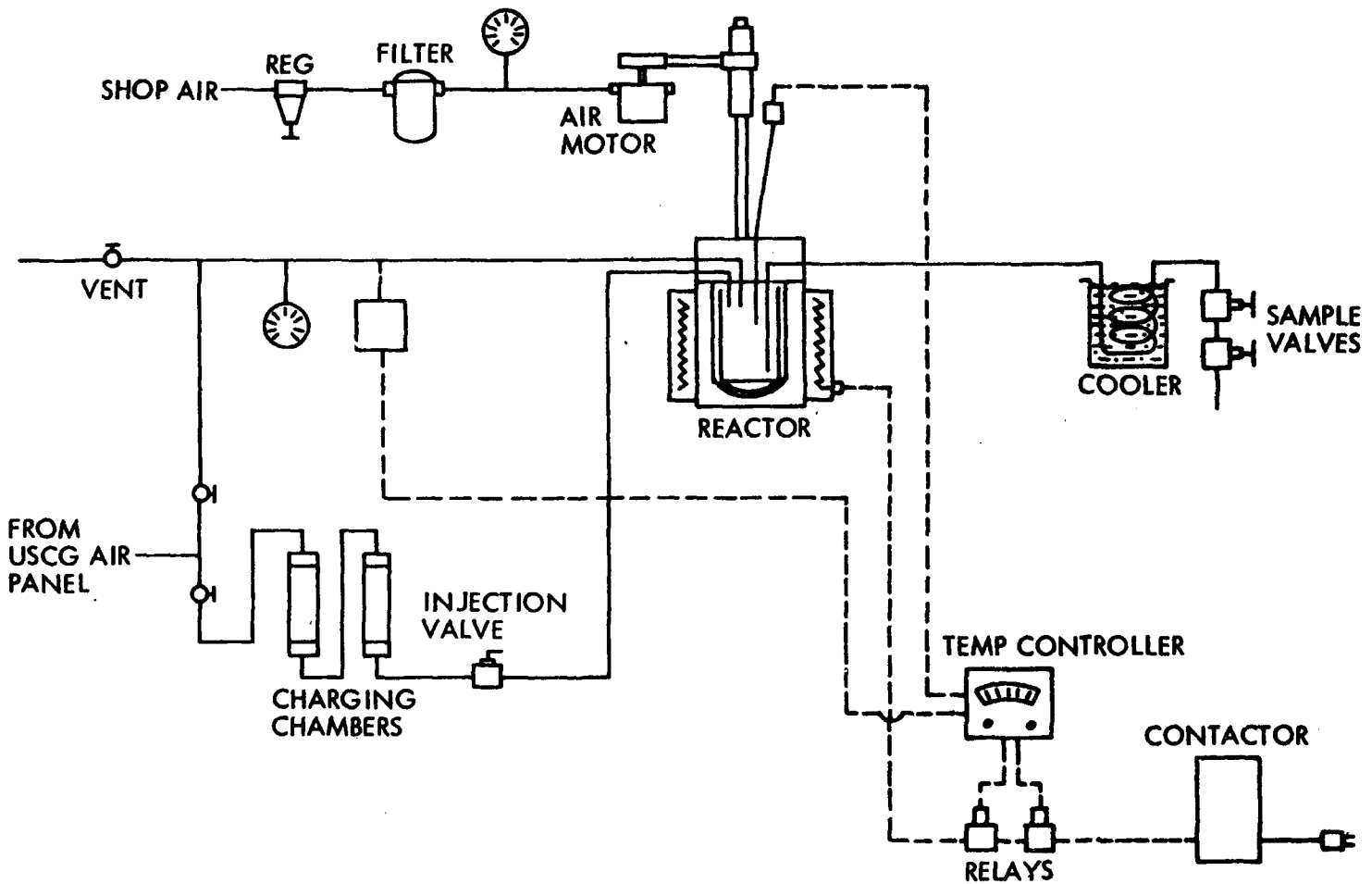


Fig. 26 One Gallon Injection Stirred Reactor Test Setup

The reactor drive and heater were de-energized.

After overnight cool down, the reactor was flushed through the injection tubes and sample line and prepared for the next run.

Three runs in the one gallon injection reactor were made to allow comparison of the stirred reactor performance with the packed tower reactor performance and to indicate the upper limit of percent reduction in TOC that can be achieved with wet oxidation of the wastes being tested on this program without the use of acid or catalyst. The upper limit was chosen to be that percent reduction achieved at 650°F, 4500 psig, 90 minutes contact time in a stirred reactor.

The first run was made to compare packed tower performance with stirred reactor performance, so a 650°F, 4500 psig, no acid run was made with samples taken at 3, 6, 12 and 90 minutes. Results of Run 1 are tabulated below:

Sample Time (min.)	Sample TOC (mg/liter)	% Reduction (TOC)
Input	420	-
3	149	65
6	116	72
12	96	77
90	32	92

Since these results did not equal or exceed the packed tower performance results as was expected, it was decided to try a run with Fe Cl_2 addition to check the value of the Fe^{++} ion which was no doubt present in the packed tower and absent in the stirred reactor. Run Number 2 was therefore made under exactly the same conditions as Run 1, except that 1 1/2 gms of Fe Cl_2 were added to the slurry. Results were as follows:

Sample Time (min.)	Sample TOC mg/liter	% Reduction
Input	420	-
3	116	72
6	101	76
12	96	77
90	53	87

The Fe CL_2 assisted at low contact times, but inhibited at 90 minutes and did not increase the overall performance at all.

In conjunction with another program, tests were run with undiluted fecal/urine slurry that assisted in establishing further direction of the test program and are therefore reported here. A batch run in the one gallon reactor[1] (Run 3) produced the following results after ninety minutes exposure.

	Run No. 3	
	TOC	COD
Input Slurry	20,925	51,100
Effluent (90 min.)	1,910	6,700
% Reduction	91	87

Several batch undiluted fecal/urine slurry runs were made in a one liter 316 SS stirred reactor under these same conditions to provide additional support to the data. Results of two runs produced the following data:

	Run No. 13*		Run No. 15*	
	TOC	COD	TOC	COD
Input	20,923	51,100	18,600	51,500
Effluent	1,925	5,258	1,549	4,365
% Reduction	90	90	92	92

*One liter reactor test run numbers

A review of all of the data taken to this point in the program yielded the following conclusions:

TOC correlation with COD were very favorable indicating further that TOC can be used as an indicator of system performance.

Approximately 80 percent reduction in TOC, BOD and COD was all that can be achieved in 3 to 12 minutes of contact time and 92 percent is the approximate limit without catalyst or acid addition.

The packed tower reactor system worked as well as, if not better than the stirred reactor system, indicating that the recirculation hot pump was not needed.

Future efforts should be directed toward finding a catalyst that improves system performance and allows reduction in pressure and temperature.

Catalyst Screening & Performance Tests

A total of thirty six catalyst screening and performance tests were run. Tables 8 and 9 present the outlet TOC and the percent reduction in TOC that resulted from these runs. Catalysts were selected on the basis of their general use in oxidation processes.

The catalysts were flushed into the reactor in powdered form along with the initial tap water charge. It was assumed early in the catalyst screening test program that if a noble metal catalyst were selected it would be deposited on a substrate and packed into the tower reactor for operational use. The use of powdered catalyst at this point of the program was an expedient means of conducting stirred reactor tests. Catalyst quantities of 1.5 grams per run were used with the following exceptions.

Run No.	Catalyst & Amount (gm)
11	1.2 gm $CoCl_2$ $6H_2O$ + 1.08 gm Mo O_3
13	3.0 gm Ground MnO_2 on Alumina
17	1.09 gm CuO + 0.75 gm Cr_2O_3
21	0.75 gm Pd BK + 0.81 gm Ru BK
22	0.75 gm Pd BK + 0.85 gm Pt BK
23	0.75 gm Pd BK + 0.75 gm Pt BK
24	0.75 gm Pd BK + 1.76 gm Ru Cl_3
25	0.75 gm Pd BK + 0.75 gm Ru BK
27	0.75 gm Pd BK + 1.85 gm Ru Cl_3 + 0.55 gm NaOH
35	0.75 gm Pt BK 0.75 gm Ru BK

Table 8 One Gallon Stirred Reactor Test Results
Catalyst Evaluation

Run No.	Catalyst	Sample TOC (mg/liter) in 1	3	6	12	90 Mins.	Temp. (°F)	Pressure (PSIG)
1	None	-	149	116	96	32		
2	$FeCl_2$	-	116	101	96	53		
4	Ru BK	128	72	54	10	4		
5	Pt BK	19	12	11	12	4		
7	Pt BK	62	44	32	19	13		
9	$Mn\ O_2$	164	124	107	83	31		
10	Ni Oxides	225	164	135	111	36	650	4500
11	$CoCl_2+MoO_3$	248	155	118	91	27		
12	CoO	200	154	139	118	65		
13	MnO_2+Alumina	184	147	117	97	19		
14	Pd BK*	15	15	15	15	15		
15	Pd BK*	7	6	15	7	7		
17	$CuO+Cr_2O_3$	107	79	60	50	10		
18	Powdered MS 5X	191	140	108	80	-		
19	Ir BK	132	83	53	35	15		
20	Rh BK	159	106	84	55	5		
16	Pd BK*	226	154	93	60	10		
19A	Ir BK	316	277	200	143	-	550	2250
20A	Rh BK	297	261	226	141	-		
21	Pd BK*+Ru BK	160	91	66	46	9		
23	Pd BK*+Pt BK	129	89	76	66	42		
24	Pd BK*+$RuCl_3$	110	72	63	42	9		
25	Pd BK*+Ru BK+HCl	112	88	80	55	36		
26	Pd BK** Sea Water	156	122	112	101	47		
27	Pd BK**+$RuCl_3$+NaOH	58	24	8	5	8	650	3500
28	Pd BK**	278	233	158	117	86		
29	Pd BK***	349	334	235	193	78		
30	Pd BK***	279	226	115	120	70	550	2250
31	Pd BK***	253	228	186	123	53		
32	Pd BK**	221	132	119	111	69		
33	Pt BK	62	33	12	4	4	650	4500
34	Pt BK	88	62	57	44	25		
35	Pt BK+Ru BK	71	51	41	25	14	550	2250
36	Pt BK (Lot No. 2)	88	71	64	52	18		

*Lot No. 1
**Lot No. 2
***Lot No. 3

Table 9 One Gallon Stirred Reactor Test Results
Catalyst Evaluation

Run No.	Catalyst	Percent Reduction in TOC at 1	3	6	12	90 Min.	Temp. (°F)	Pressure (PSIG)
1	None	-	65	72	77	92		
2	Fe Cl_2	-	72	76	77	87		
4	Ru BK	70	83	87	98	99		
5	Pt BK	95	97	97	97	99		
7	Pt BK	85	90	92	95	97		
9	MnO_2	59	69	73	79	92		
10	Ni Oxides	44	59	66	72	91		
11	$CoCl_2$+MoO_3	38	61	71	77	93	650	4500
12	CoO	50	62	65	71	84		
13	MnO_2 + Alumina	54	63	71	76	95		
14	Pd BK*	96	96	96	96	96		
15	Pd BK*	98	98	96	98	98		
17	CuO+Cr_2O_3	73	80	85	88	97		
18	Powdered MS 5X	52	65	73	80	-		
19	Ir BK	67	79	87	91	96		
20	Rh BK	60	74	79	86	99		
16	Pd BK*	44	62	77	85	97		
19A	Ir BK	21	31	50	64	-		
20A	Rh BK	24	35	44	65	-	550	2250
21	Pd BK*+RU BK	60	77	84	89	98		
23	Pd BK*+Pt BK	68	78	81	84	90		
24	Pd BK*+$RuCl_3$	73	82	84	90	98		
25	Pd BK* + Ru BK + HCl	72	78	80	86	91		
26	Pd BK** Sea Water	61	70	72	75	88		
27	Pd BK**+$RuCl_3$+ NaOH	86	94	98	99	98	650	3500
28	Pd BK**	31	42	61	71	79		
29	Pd BK***	13	17	41	52	81		
30	Pd BK***	30	43	61	70	83	550	2250
31	Pd BK***	37	43	54	69	87		
32	Pd BK**	45	67	70	72	83		
33	Pt BK	85	92	97	99	99	650	4500
34	Pt BK	78	84	86	89	93		
35	Pt BK + Ru BK	85	90	92	95	97	550	2250
36	Pt BK (Lot No.2)	82	85	87	89	96		

*Not No. 1
**Lot No. 2
***Lot No. 3

In general two classes of catalysts were tested, i.e., noble metal black and metal oxides or salts. The metal oxides or salts did little if anything to decrease the contact time required to achieve a high degree of oxidation. In some cases they seemed to inhibit the reaction. Although several noble metal blacks exhibited catalytic effects, three stood out as most promising early in the catalyst testing: Ruthenium, Platinum and Palladium. Since these three showed a high degree of oxidation at 650°F and 4500 psig even at low contact times (1 to 12 minutes), it was decided to evaluate their performance at lower temperatures and pressures, i.e. 550°F and 2250 psig. It appeared in early runs (4, 5, and 14) that perhaps the Ruthenium, Platinum and Palladium were working on different components of the waste mixture because of the relative reaction rates. At 650°F and 4500 psi, Ruthenium was slow to start, but finished well. Platinum was faster than Ruthenium, but Palladium showed almost instant effects. It was decided to test mixtures of the noble metal blacks which promised to give improved results.

Tests through run 25 showed Palladium to be a superior catalyst, however, the original supply of Palladium was depleted and a second lot did not produce nearly as good results. A third lot also failed to provide good performance.

A check with the catalyst manufacturer showed that the first lot of Palladium was very highly active and that the second and third lots were what could normally be expected with respect to active sites. Emphasis in further testing was shifted from Palladium to Platinum which proved to be superior to lot 2 and 3 Palladium in later tests. A mixture of Platinum/Ruthenium proved to be the best combination as shown during run 35 and was selected as the catalyst to be tested in the packed tower.

Although final catalyst tests were run at 550°F and 2250 psig and the Pt/Ru catalyst produced 90 to 95 percent reduction in TOC between 3 and 12 minutes, the final selection of process conditions was planned to be made in the

five gallon packed tower breadboard system. It was decided to conduct tests to determine preliminary process conditions and to evaluate the performance of the catalyst deposited on a substrate in a continuous flow one liter packed tower system prior to conducting tests in the full scale system. The one liter packed tower system had been built for other purposes and was made available for this program. Tests conducted in the one liter system will be discussed later in the report after a discussion of chemical oxygen source studies in the one gallon, injection, stirred reactor.

Chemical Oxygen Source Evaluation

The use of chemicals as a source of oxygen instead of air offers several advantages for wet oxidation. The chemicals could be mixed with the sewage prior to pumping thereby eliminating the relatively heavy bulky and power consuming air compressor. Chemical oxygen sources are generally more active than gaseous oxygen for two reasons; the oxygen is released in solution where the oxidation takes place and the breakdown of the chemicals can produce transient species of highly active oxygen which should lead to more effective oxidation. Chemical oxygen sources should provide a high degree of oxidation without the need for a catalyst. Three chemical oxygen sources were tested; sodium hypochlorate, calcium hypochlorite and sodium chlorate. One gallon, injection, stirred reactor test runs listed in Tables 10 and 11 were made to investigate the use of chemical oxygen sources with and without catalyst. On all runs a 90 cc charge of chemical oxygen solution was substituted for the 90 cc rinse water in the second injection tube. The chemical solution followed the concentration sewage down the tube and into the reactor during injection at time zero. The catalyst used on runs 37, 39, 40 and 41 was flushed into the reactor with the initial water charge prior to warmup. Table 10 presents the effluent TOC for each sample time of 1,3, 6, 12, 30 and 90 minutes after injection and Table 11 presents percent reductic in TOC for the same samples.

Table 10 One Gallon Stirred Reactor Test Results -
Chemical Oxygen Sources

Run No.	Catalyst or Oxidant	Sample TOC (mg/liter) in 1	3	6	12	30	90 Min.	Temp. (°F)	Pressure (PSIG)
37	Pt + 5% NaOCl	40	24	13	13	13	13	550	1300
39	Pt + 5% NaOCl	170	164	157	151	147	143	450	500*
40	Pt + 14% NaOCl	72	67	61	62	51	19	450	500*
41	Pt + 14% NaOCl**	45	47	50	46	11	11	450	500*
42	14% NaOCl	84	77	75	68	60	34	450	500*
43	14% NaOCl+Carbon	174	178	182	187	174	150	450	500*
44	14% NaOCl**	64	68	75	83	74	33	450	500*
45	14% NaOCl***	49	46	45	43	40	26	450	750
46	14% NaOCl	124	106	85	52	22	12	500	700*
47	14% NaOCl****	175	38	38	35	13	11	500	700*
48	14% NaOCl	141	101	54	21	12	14	550	1100*
49	14% NaOCl*****	284	202	-	113	79	42	500	700*
50	18 gm $Ca(OCl)_2$	208	170	143	123	121	113	550	1100*
51	27 gm $Ca(OCl)_2$	80	68	55	45	44	42	550	1100*
52	36 gm $Ca(OCl)_2$	60	55	50	50	50	50	550	1100*
60	10 gm $NaClO_2$	385	326	34	14	17	43	550	1100*

*No air used on these runs (entire O_2 demand supplied by hypochlorite)

**A 10 minute sewage hydrolysis without air preceded NaOCl injection

***A 10 minute sewage hydrolysis with air preceded NaOCl injection

****Two 90 cc NaOCl injections were made

*****Two 45cc NaOCl injections were made

Table 11 One Gallon Stirred Reactor Test Results - Chemical Oxygen Sources

Run No.	Catalyst or Oxidant	% Reduction in TOC at 1	3	6	12	30	90 min.	Temp. °F	Pressure (PSIG)
37	Pt + 5% NaOCl	92	95	97	97	97	97	550	1300
39	Pt + 5% NaOCl	65	66	67	69	70	71	450	500
40	Pt + 14% NaOCl	85	86	87	87	89	96	450	500
41	Pt + 14% NaOCl	91	90	90	91	98	98	450	500
42	14% NaOCl	83	84	85	86	88	93	450	500
43	14% NaOCl + Carbon	64	63	63	62	64	69	450	500
44	14% NaOCl	87	86	85	83	85	93	450	500
45	14% NaOCl	90	91	91	91	92	95	450	750
46	14% NaOCl	75	78	83	89	95	98	500	700
47	14% NaOCl	64	92	92	93	97	98	500	700
48	14% NaOCl	71	79	89	96	98	97	550	1100
49	14% NaOCl	47	62	-	79	85	92	500	700
50	18 gm $Ca(OCl)_2$	61	68	73	77	77	79	550	1100
51	27 gm $Ca(OCl)_2$	85	87	90	92	92	92	550	1100
52	36 gm $Ca(OCl)_2$	89	90	91	91	91	91	550	1100
60	10 gm $NaClO_3$	28	38	94	97	97	92	550	1100

Four runs (37, 39, 40 and 41) were made with sodium hypochlorite and platinum catalyst to establish the combined effects. The sewage and hypochlorite were injected using air in run 37 so it represents the combined effects of air and chemical oxidation and catalyst. Run 39 wastes and hypochlorite were injected using a high pressure nitrogen tank as a gas source and it was discovered that 90 cc of 5% NaOCl solution does not provide sufficient oxygen without assistance from the air. Further testing was accomplished using 90 cc of 14% NaOCl solution. Runs 40 and 41 showed that extremely low temperatures (450°F) and pressures (500 psig) can be used with catalyst and hypochlorite addition without the use of air. One of the biggest hopes for chemical oxygen sources was, however, the elimination of the need for catalysts. Subsequent runs were made in search of a chemical oxygen source that resulted in a practical quantity of expendable material, and reasonably low process conditions. Sodium hypochlorite proved to be a very effective oxidant in wet oxidation. It's use presents some serious problems, however, because it is very unstable in high concentrations. 14% NaOCl is about the upper limit for reasonably long term storage at ambient temperatures. This means that a large quantity of water must be stored to provide the required amount of oxygen. The generation of hypochlorite from sea water was considered but the power and equipment size required to generate the required quantities were prohibitive.

Calcium hypochlorite offers the advantages that it is readily storable and although it does not seem to be as effective at producing the extremely low TOC's at long contact times as sodium hypochlorite, it does provide promising results as seen by the results of runs 50, 51 and 52.

Sodium chlorate is also a stable chemical that is readily available and easily stored. It is highly soluble in water, making injection easy and has more available oxygen per unit weight and volume than either hypochlorite chemicals. Run 60 showed very favorable performance for sodium chlorate.

In order to reduce the quantities of expendable chemicals required to achieve 90% or greater reduction in BOD, it was decided to add chemicals to the injection reactor after a 6 or 12 minute period of air oxidation. The theory behind this idea was to let the air complete the easy part of the oxidation process which it does rapidly without catalyst at reasonable pressures and temperatures and then let the chemicals do the last 20 percent that is very difficult to accomplish with air. Runs 53 through 59 tested calcium hypochlorite, sodium chlorate, and chlorine as polishing agents. The results of these runs are presented by Tables 12 and 13. Very favorable results were obtained with all three at 550°F and 1500 psig.

Table 14 presents a summary of the results of the one gallon injection reactor chemical oxygen source tests. The quantities of chemicals and process temperatures, pressures, and contact times are presented for the four chemical oxygen sources tested assuming (1) use of the chemicals for complete oxidation without air, (2) as polishing agents to air oxidation. The quantity of chemicals and contact times are estimated based on the test data and are intended for comparison purposes only. Further testing is required to substantiate these values. It was concluded from inspecting Table 14 data, that the quantities of chemicals required for complete chemical oxidation were prohibitive for use on Coast Guard vessels. This was also true for use of sodium and calcium hypochlorite as polishing agents. Chlorine was considered too toxic for use on a vessel. The quantities of sodium chlorate were very reasonable and considering the other advantages of the chemical, it was selected for further evaluation in the breadboard and one liter packed tower systems.

One Liter Packed Tower Reactor Tests

The one liter packed tower reactor system presented schematically by Figure 27 was a small scale version of the five gallon breadboard system. It utilized the air compressor, air accumulator, input mixing tank, and pump/grinder assembly of the big system. A five gallon bladdered tank provided an average of four hours continuous operation without requiring a tank refill. A 0.33 gpm

Table 12 One Gallon Stirred Reactor Test Results

Chemical Polishing

Run No.	Catalyst or Oxidant	Sample TOC (mg/liter) in Air Oxidation 1	3	6	12	Sample TOC (mg/liter) in Oxidant Injection 1	3	6	12	30	90 min.	Temp. (°F)	Pressure (PSIG)
53	10 gm $Ca(OCl)_2$	392	180	98	*	21	14	16	14	11	10	550	1500
54	10 gm $Ca(OCl)_2$	408	394	395	*	193	165	129	92	54	30	500	1100
55	10 gm $Ca(OCl)_2$	455	339	173	*	30	20	13	10	12	10	550	1500
56	5 gm $Ca(OCl)_2$	526	498	350	181	56	38	25	12	13	11	550	1500
57	2.5 gm $NaClO_3$	490	406	304	185	110	70	30	7	7	10	550	1500
58	None	-	-	-	-	409	310	185	120	97	70	550	1500
59	Chlorine	-	431	380	268	144	93	70	41	12	10	550	1500

*$Ca(OCl)_2$ injected after six minutes of air oxidation on runs 53, 54 and 55

Table 13 One Gallon Stirred Reactor Test Results

Chemical Polishing

Run No.	Catalyst or Oxidation	% Reduction in TOC at 1	3	6	12	1	3	6	12	30	90 Min.	Temp. (°F)	Pressure (PSIG)
53	10 gm $Ca(OCl)_2$	27	66	82	-	96	97	97	97	98	98	550	1500
54	10 gm $Ca(OCl)_2$	23	26	28	-	64	69	76	83	90	95	500	1100
55	10 gm $Ca(OCl)_2$	16	36	68	-	94	96	98	98	98	98	550	1500
56	5 gm $Ca(OCl)_2$	0	5	33	65	89	93	95	98	98	98	550	1500
57	2.5 gm $NaClO_3$	8	24	43	65	79	87	94	99	99	98	550	1500
58	None	-	-	-	-	23	42	65	77	82	87	550	1500
59	Chlorine	-	19	28	50	73	83	87	92	98	98	550	1500

← Air Oxidation →

Oxidant Injection

Selected Conditions to Produce 90 Percent or Greater Reduction in BOD

Chemical Oxidant or Catalyst			Type of Oxidation	Process Conditions		
	Type	Amount (Lb/Day)		Temp. (°F)	Pressure (PSIG)	Contact Time (m
1)	14% NaOCl Solution	400	Chemical	550 500	1100 700	10 20
2)	$Ca(OCl)_2$ Powder	80	Chemical	550	1100	6
3)	Chlorine Liquid	50	Chemical	450	500	20
4)	$NaClO_3$ Crystals	25	Chemical	550	1100	10
5)	14% NaOCl Solution	80	Air Plus Chemical	550	1500	7
6)	$Ca(OCl)_2$ Powder	16	Air Plus Chemical	550	1500	7
7)	Chlorine Liquid	10	Air Plus Chemical	550	1500	7
8)	$NaClO_3$ Crystals	5	Air Plus Chemical	550	1500	15
9)	Platinum/Ruthenium on Alumina (Non Expendable)		Air Only	550	1500	12

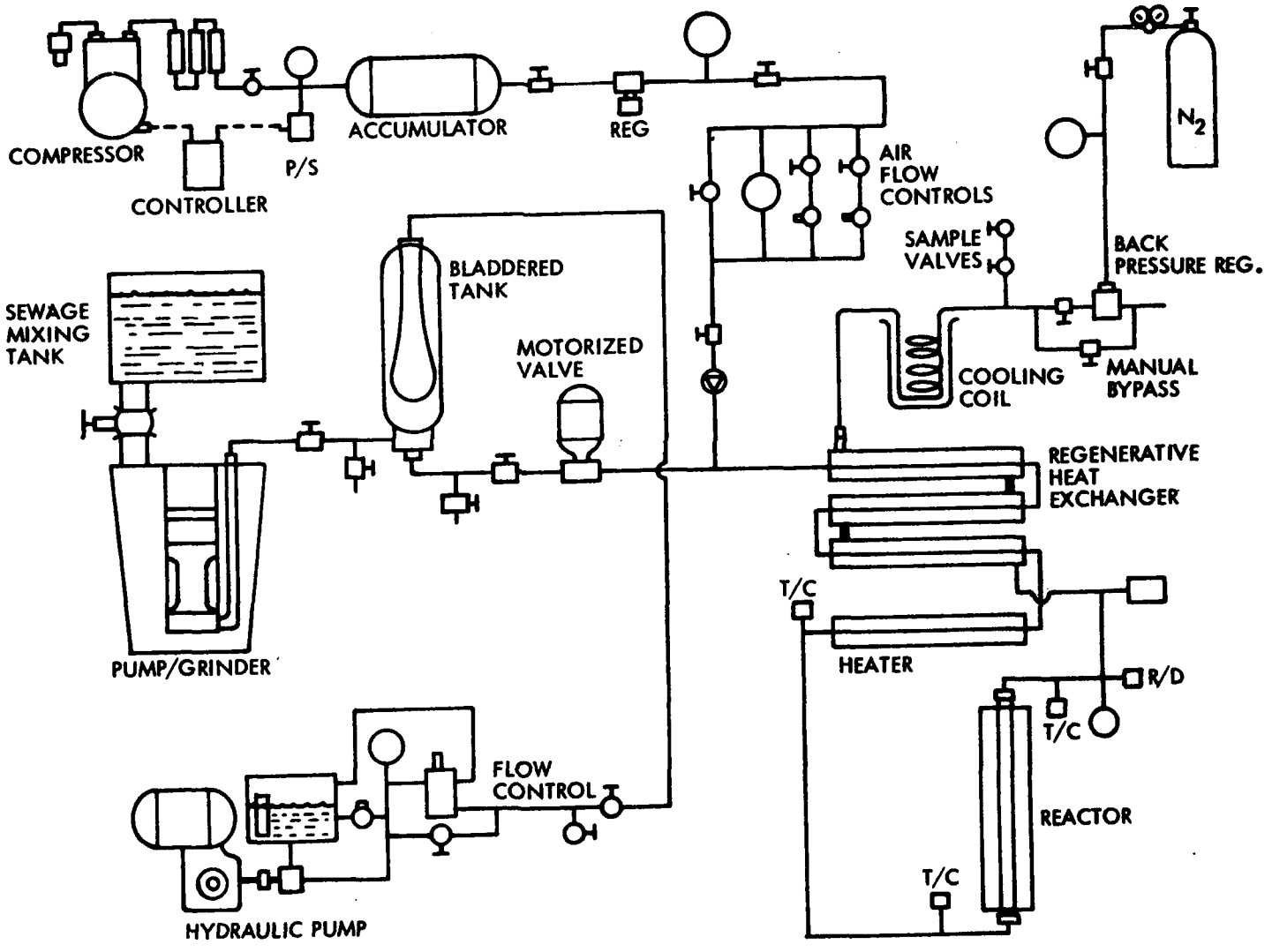


Fig. 27 One Liter Packed Tower Reactor System Schematic

hydraulic pump with varidrive and flow control valve provided sewage flows from 27 to 110 cc/min. A regenerative heat exchanger, electrically powered tube heater, air flow controls, cooling coil and back pressure regulator very similar to the corresponding components of the larger system were provided. The reactor was an electrically heated one inch O.D. by 0.120" wall 316 stainless steel tube, six feet long. For the initial catalyst tests it was filled with platinum and ruthenium on 1/8" diameter alumina cylinders. The platinum and ruthenium were deposited on separate cylinders and then mixed in equal proportions. Figure 28 is a photograph of the system components inside the protective wall and Figure 29 is a photograph of the control panels and hydraulic pump.

The one liter packed tower system was operated in a manner very similar to the breadboard system.

A total of sixty-two data runs were made using several tower configurations with and without catalyst and with and without packing material. Figure 30 presents the various tower configurations used during the test program and Table 15 presents the test data for the 62 runs. The first configuration was used in testing the Platinum/Ruthenium catalyst on 1/8" alumina substrate with an upflow liquid phase continuous reactor in order to take full advantage of the tower volume and provide the longest possible liquid contact time. Runs 1 through 17 generated the performance map shown by Figure 31 which indicated system performance far in excess of expectations based on one gallon stirred reactor test results. It appeared that 500°F and 1100 psi would produce 95% reduction in TOC in 15 to 20 minutes. Many catalysts exhibit short term break-in performance loss, so two additional days of operation at these conditions were run to check for performance loss, which did occur. One reason for such loss in performance is that a considerable amount of Pt/Ru dust coats the pellets which adds to the catalytic effect early in the use of the catalyst. As this dust is swept away catalyst performance drops until only the deposited catalyst remains.

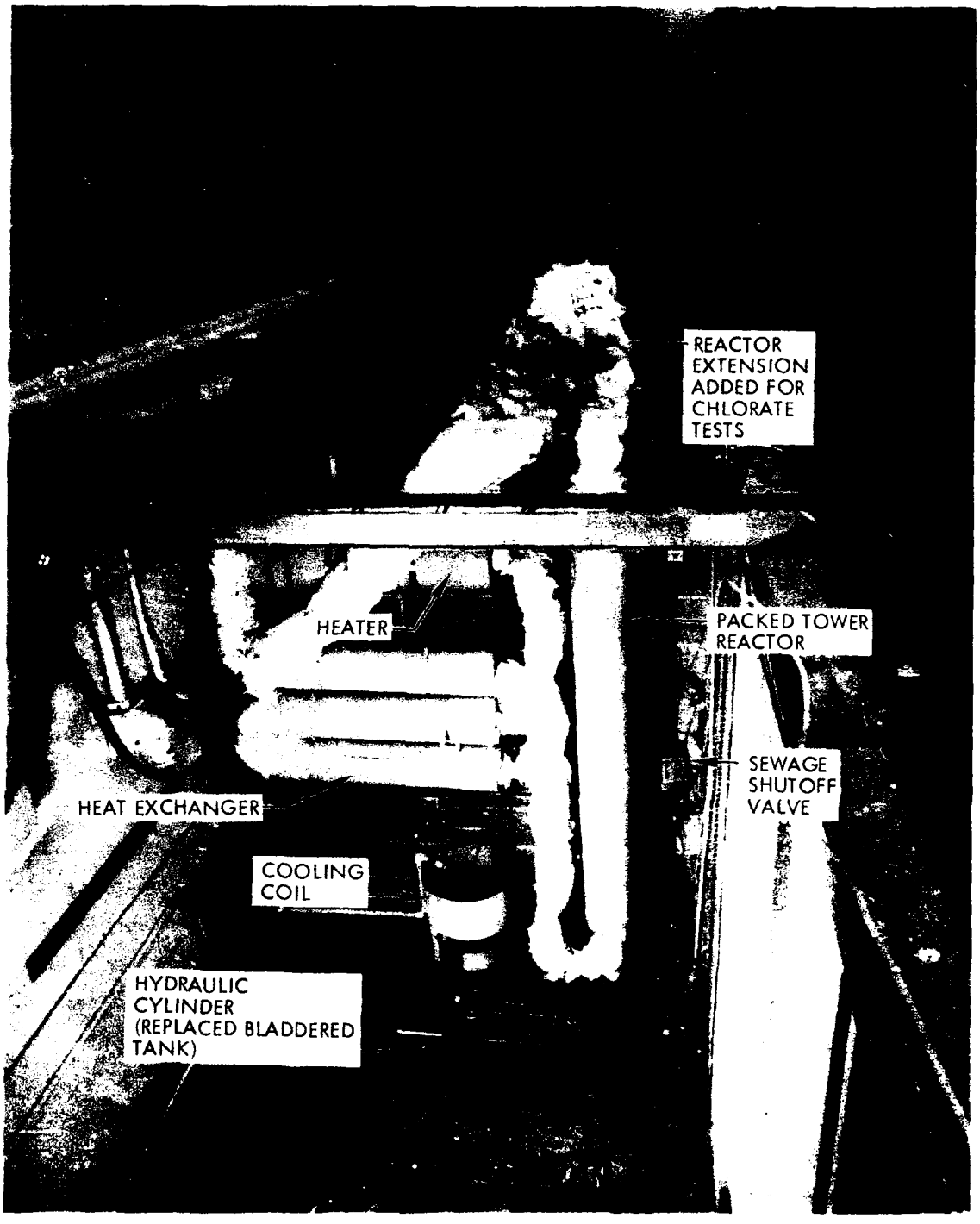


Fig. 28 One Liter Packed Tower Reactor System

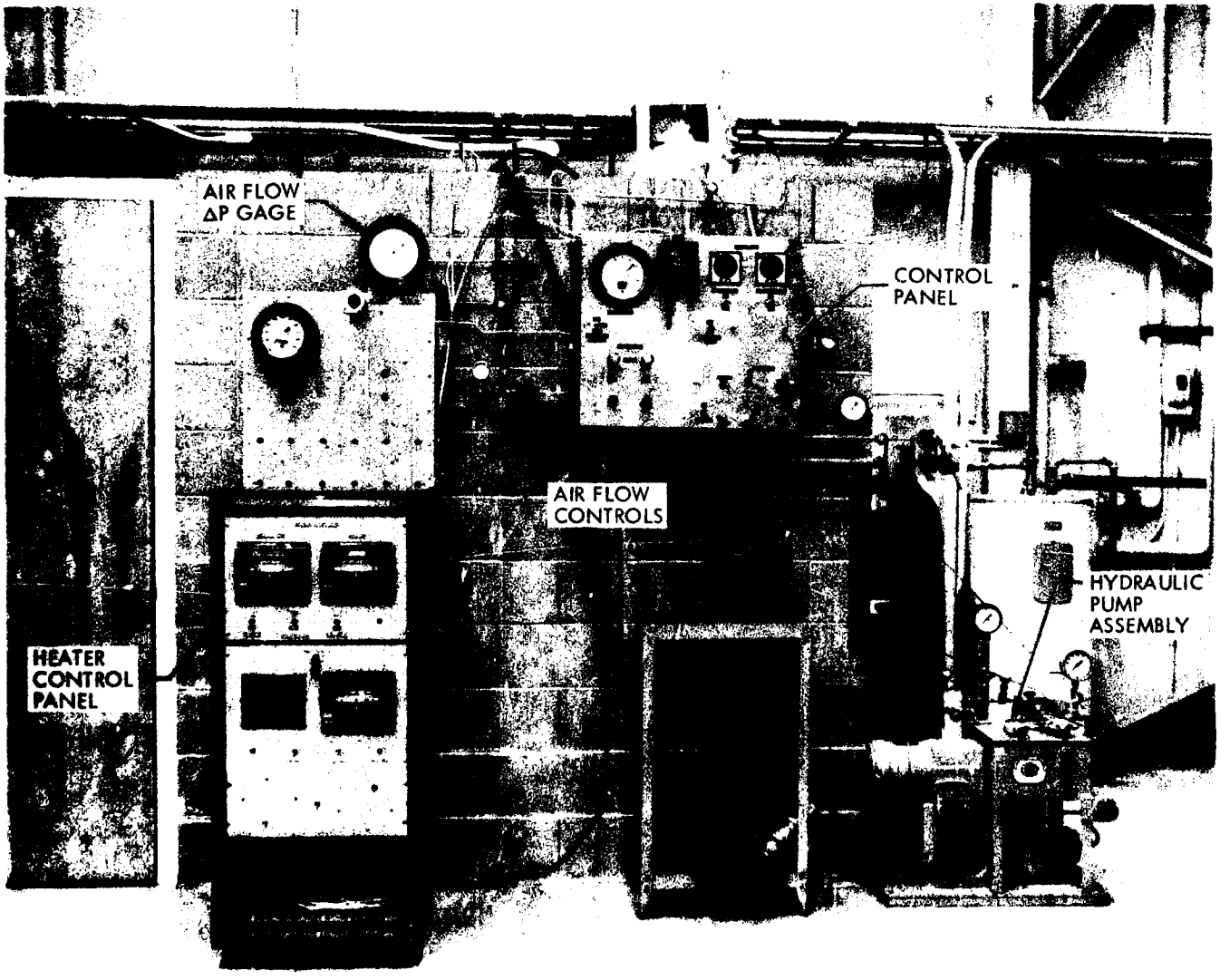


Fig. 29 One Liter Packed Tower Reactor System Control Panel and Console

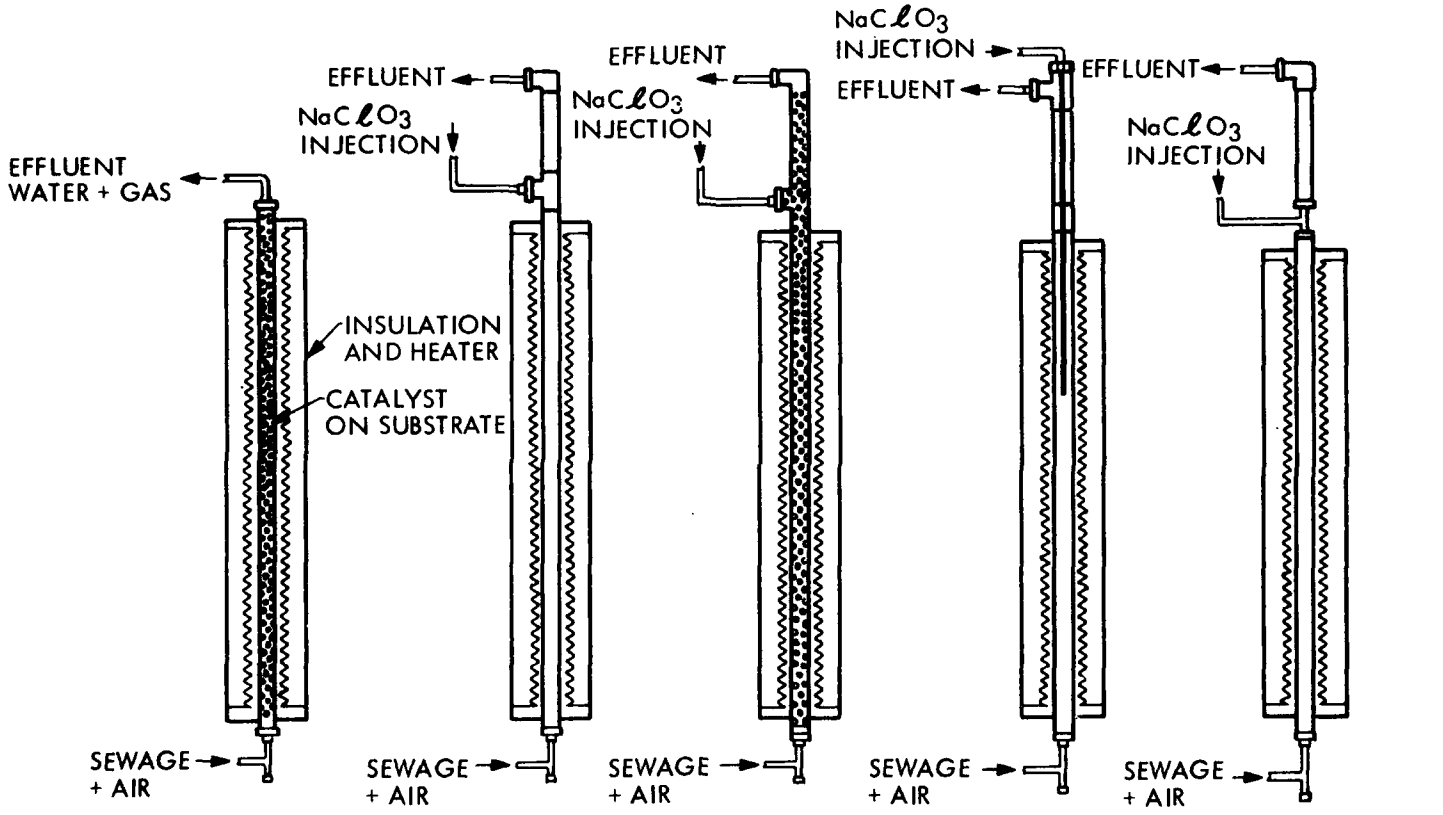


Fig. 30 One Liter Packed Tower Test Configurations

Table 15

One Liter Packed Tower Test Results

(Pt/Ru Catalyst on Alumina)

	Run No.	Temp. (°F)	Pressure (PSIG)	Sewage Flow (cc/m)	Air Flow (cc/m)	Contact Time (min)	Output TOC	% Red.
	1	550	1500	55	1200	10	11	98
	2	550	1500	27	1200	20	5	99
	3	550	1500	110	2400	5	13	98
	4	550	2000	55	1200	10	8	98
	5	550	2000	27	1200	20	6	99
	6	550	1200	55	1200	10	15	97
Input	7	550	1200	110	2400	5	34	93
Sample	8	500	1100	55	1200	10	32	93
TOC	9	500	1100	27	1200	20	18	96
457	10	500	1100	110	2400	5	74	84
	11	500	850	55	1200	10	50	89
	12	500	850	27	1200	20	36	92
	13	500	850	110	2400	5	82	82
	14	450	800	55	1200	10	92	80
	15	450	800	27	1200	20	74	84
	16	450	600	55	1200	10	99	78
	17	450	600	27	1200	20	91	80
Input Sample TOC 457	18	500	1100	55	1200	10	80	83
	19	500	1100	55	1200	10	70	85
	20	550	1500	55	1200	10	50	89
	21	550	1500	27	1200	20	24	95
	22	550	1500	110	2400	5	57	88
	23	550	1200	55	1200	10	37	92
	24	550	1200	27	1200	20	21	96
	25	550	1200	110	2400	5	55	89
	26	500	1100	55	1200	10	62	87
Input	27	500	1100	27	1200	20	41	91
Sample	28	500	1100	110	2400	5	86	82
TOC 478	29	500	850	55	1200	10	58	88
	30	500	850	27	1200	20	49	90
	31	500	850	110	2400	5	89	81
	32	550	1500	55	1200	10	36	92
	33	550	1500	55	1200	10	51	89

	Run No.	Temp. °F	Pressure (PSIG)			Sewage Flow (cc/m)	Air Flow cc/m	Contact Time (min)	Output TOC	% Red.
Input Sample TOC 478	34	550	1500			55	1200	10	39	92
	35	550	1500			27	1200	20	30	94
	36	550	1500			110	2400	5	47	90
Input Sample TOC 1623	37	550	1500			55	1600	10	107	93
	38	550	1500			27	1600	20	67	96
	39	550	1500			110	3200	5	148	91
	40	550	1500			55	1600	10	127	92

One Liter Packed Tower Test Results

No Catalyst - No Packing

				$NaClO_3$ Flow (cc/hr)	Pump Setting				
Input Sample TOC 351	41	550	1500			55	1200	10	139	60
	42	550	1500			27	1200	20	108	69
	43	550	1500			110	2400	5	152	57
	44	550	1500	30	21	55	1200	10	122	65
	45	550	1500	15	13	27	1200	20	94	73
	46	550	1500	60	39	110	2400	5	128	64

One Liter Packed Tower Test Results

No Catalyst - 1/8 Alumina Packing

Input Sample TOC 435	47	550	1500	NA	NA	55	1200	10	161	63
	48	550	1500	NA	NA	27	1200	20	142	67
	49	550	1500	NA	NA	110	2400	5	170	61
	50	550	1500	30	21	55	1200	10	156	64
	51	550	1500	15	13	27	1200	20	74	83
	52	550	1500	60	39	110	2400	5	175	60

One Liter Packed Tower Test Results

No Catalyst-No Packing-$NaClO_3$ Tube in Center of Reactor

Input Sample TOC 404	53	550	1500	NA	NA	55	1200	10	146	64
	54	550	1500	NA	NA	27	1200	20	116	71
	55	550	1500	NA	NA	15	300	37	89	78
	56	550	1500	30	21	55	1200	10	138	66
	57	550	1500	15	13	27	1200	20	86	79
	58	550	1500	15*	13*	15	300	37	107	73

*Diluted $NaClO_3$ Solution 2/1

Input Sample 404	59	550	1500	120	74	55	1200	10	141	65
	60	550	1500	30	21	55	1200	10	160	61
	61	550	1500	120	74	55	1200	10	144	64
	62	550	1500	30	21	55	1200	10	138	66

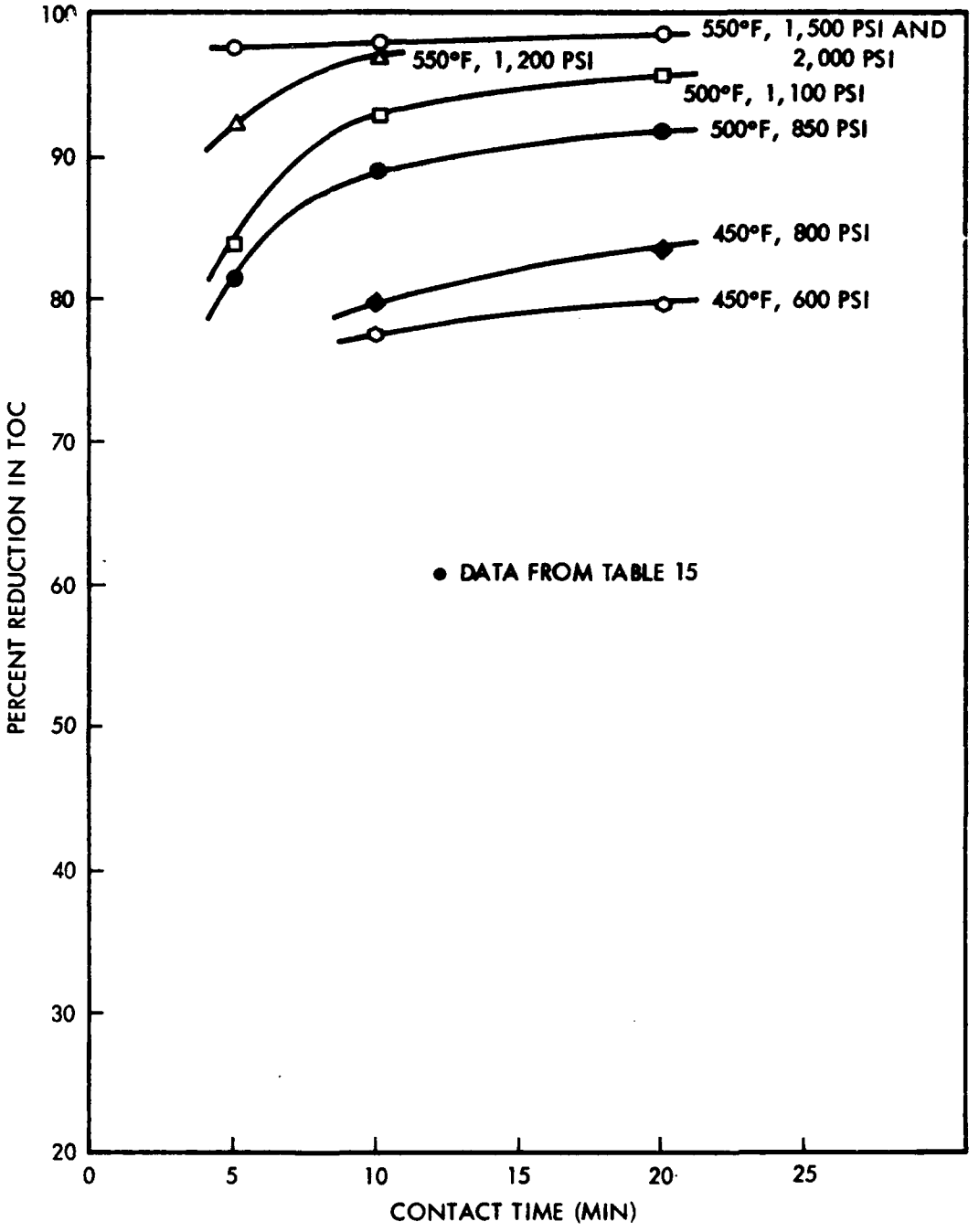


Fig. 31 One Liter Packed Tower Reactor Test Results (Fresh Catalyst)

A second performance map presented by Figure 32 was prepared from runs numbers 20 through 40. Runs 20 through 31 generated the basic data and runs 32 through 36 confirmed that catalyst stability had been reached. Runs 37 through 40 were made to evaluate system performance under high BOD/TOC loadings. Performance was slightly better at the higher concentrations as was expected. These runs completed the one liter packed tower catalyst performance tests. The backup concept using uncatalyzed wet air oxidation followed by sodium chlorate injection was tested next. Four configurations (designated configuration 2,3,4 and 5) were tested without promising results.

Configuration 2 - The catalyst and packing were removed from the tower and a 14 inch section of one inch OD pipe was added to the top of the tower. The $NaClO_3$ solution was injected into a tee between the tower and the extension tube. 100 grams of sodium chlorate were dissolved in water to create 500 cc of solution. This was pumped into the reactor by a Milton Roy piston pump. The pump was an instrument pump that delivered a constant known flow rate that was adjusted by varying the piston stroke. The flowrate of sodium chlorate solution was adjusted to provide sufficient oxygen to accomplish 20 percent of the oxidation required in the system. The concept was to accomplish 75 percent by air oxidation in the bottom of the tower and accomplish 20 percent in the extension tube using sodium chlorate. Runs 41, 42 and 43 were conducted without chlorate addition and runs 44, 45 and 46 were with chlorate addition. Although some chlorate oxidation took place the results were not promising.

Configuration 3 - Inert packing in the form of 1/8 inch alumina cylinders (identical to the catalyst substrate used in runs 1 through 40) was added to the tower and extension tube. Runs 47 through 52 were then run using the same conditions as the runs made on configuration 2 to see if the agitation of the sewage/air mixture and the sewage/sodium chlorate mixture would increase oxidation. The results show no improvement.

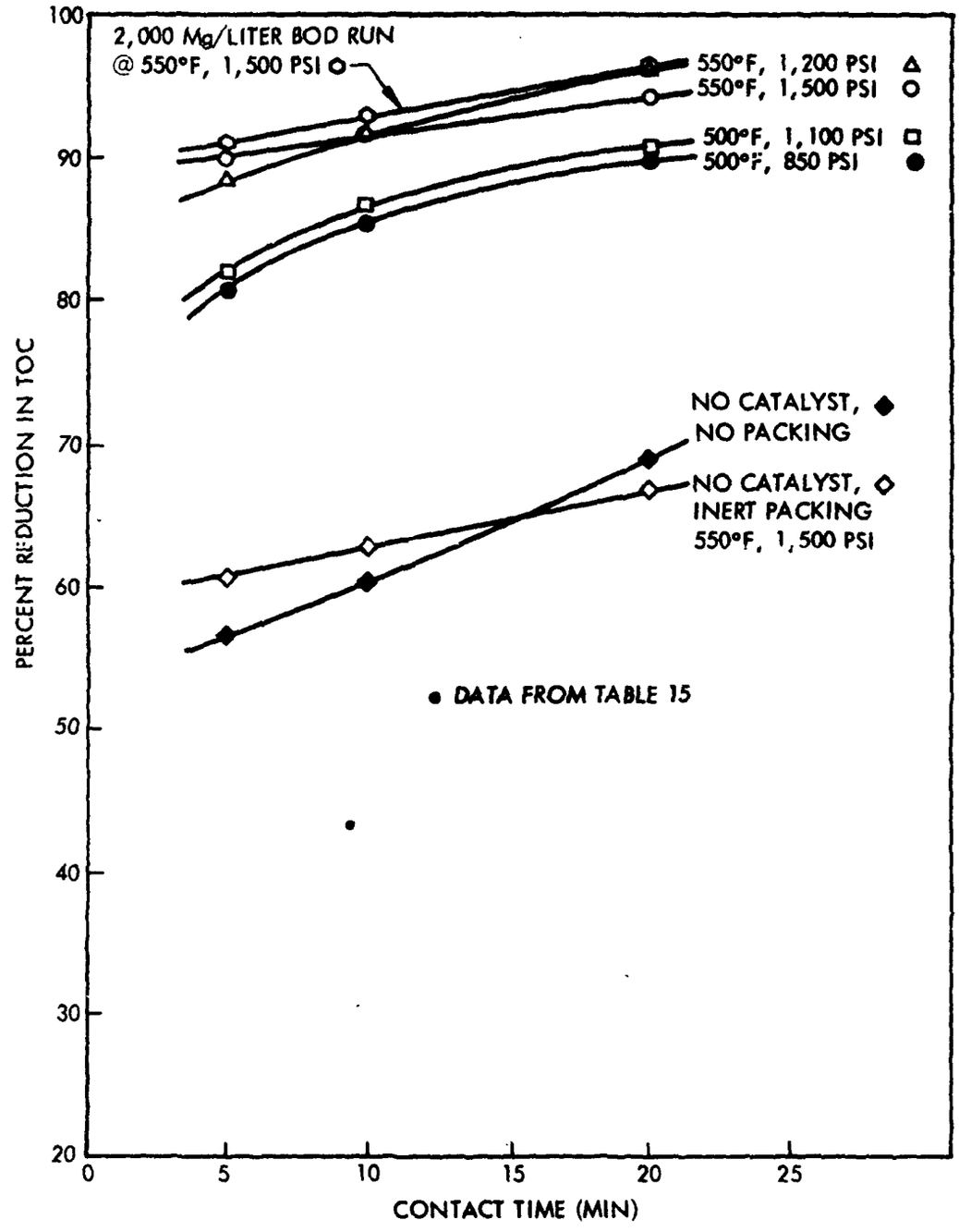


Fig. 32 One Liter Packed Tower Reactor Test Results (Stabilized Catalyst)

Configuration 4 - The packing was removed from the tower and extension tube and a 1/8" sodium chlorate injection tube was run down halfway into the reactor tube in order to increase sodium chlorate/sewage contact time. Also, the five minute contact time run was changed to a 37 minute contact time. Results again showed little or no oxidation resulting from chlorate addition.

Configuration 5 - A neckdown portion of tubing was placed between the tower and the extension tube so that higher velocities in the area would provide better mixing of the sodium chlorate and sewage. Four runs were made with the configuration, two under normal sodium chlorate flows (60 and 62) and two with four times the normal chlorate flow (Runs 59 and 61). Results indicated that no oxidation was being accomplished by the chlorate. Chlorate tests were terminated in the one liter tower. It was decided to repeat the one gallon injection reactor chlorate run to verify that chlorate can be used as an oxidant. If the results looked favorable it was planned to conduct chlorate injection tests on the full scale breadboard system. Table 16 presents a comparison of test runs 57 and 61 using chlorate as a polishing agent in the one gallon stirred reactor.

Table 16

One Liter Packed Tower Sodium Chlorate Injection Test Results

		% Reduction In Toc	
Sample Time		Run No. 57	Run No. 61
Post Sewage Injection	1	8	40
	3	24	41
	6	43	56
	12	65	69
Post $NaClO_3$ Injection	1	79	73
	3	87	84
	6	94	95
	12	99	98
	30	98	98
	90	98	98

Run 61 was a repeat of the original sodium chlorate injection test run 57 and verified the value of sodium chlorate as a chemical oxygen source. Apparently the low flow rate of sodium chlorate solution in the one liter packed tower system resulted in decomposition of the chlorate in the hot portion of the injection tube or inadequate mixing with the sewage in the reactor. The test program was next directed toward full scale system tests in the five gallon packed tower breadboard system with the tentative selection of 550°F, 1200 psig, and 20 minute contact time as process conditions for the shipboard application.

Five Gallon Packed Tower Reactor Tests

Catalyst Tests - In addition to packing the Platinum and Ruthenium catalyst on 1/4" alumina cylinders into the reactor, it was decided that several changes should be made to the slurry pumping system. By using the upflow liquid phase continuous approach as opposed to the hydrolysis pot and gas phase continuous approach used earlier in the program, the volume of liquid in the system was increased from approximately 3 1/2 gallons to 6 1/2 gallons. This meant that the 7 1/2 gallon bladdered tanks in the slurry pumping system would not adequately flush out the system with each tank emptying cycle. Since it took approximately seven minutes to fill the tanks and only 15 minutes to empty them at the design sewage flow of 0.5 gpm, it was decided to add another set of bladdered tanks to allow one set of tanks to be filled while the other was delivering sewage to the reactor. This approach provided nearly continuous sewage pumping with flow stoppage only during switchover from one tank to another which took less than thirty seconds. Two bladdered tanks, four motorized shutoff valves and electrical controls were added to the system. Figures 33 and 34 present a schematic and photograph of the revised system. The control panel and hydraulic pump valves can be seen on Figures 4 and 7 respectively. The valves 1 through 8 were sequenced to fill one set of sewage tanks from the pump/grinder assembly which would force hydraulic oil back into the hydraulic pump reservoir. Simultaneously the hydraulic pump was forcing sewage out of the other set of tanks by pushing hydraulic oil into the hydraulic cylinder or tank. Separate sewage and hydraulic oil tanks

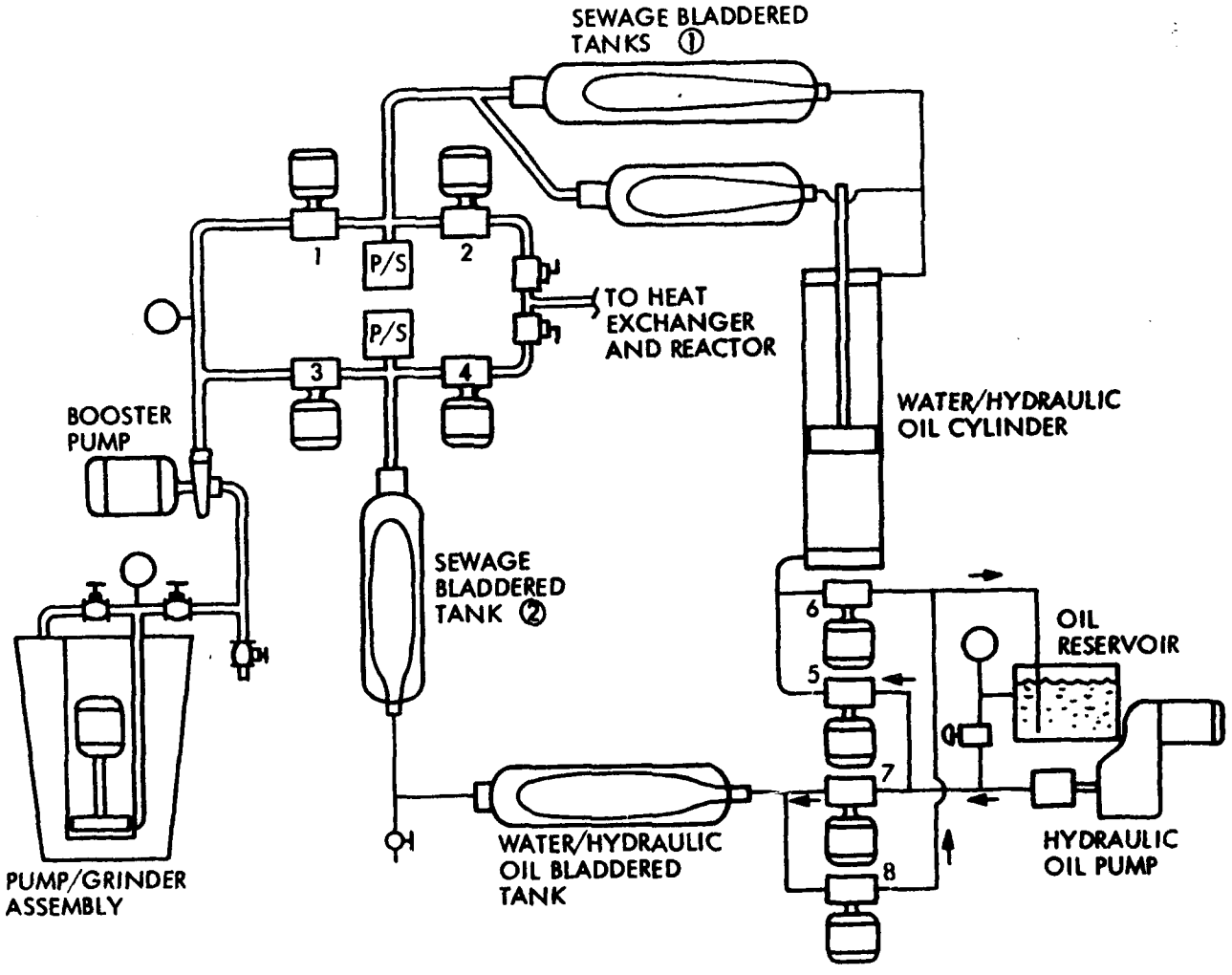


Fig. 33 Five Gallon Reactor Pumping System

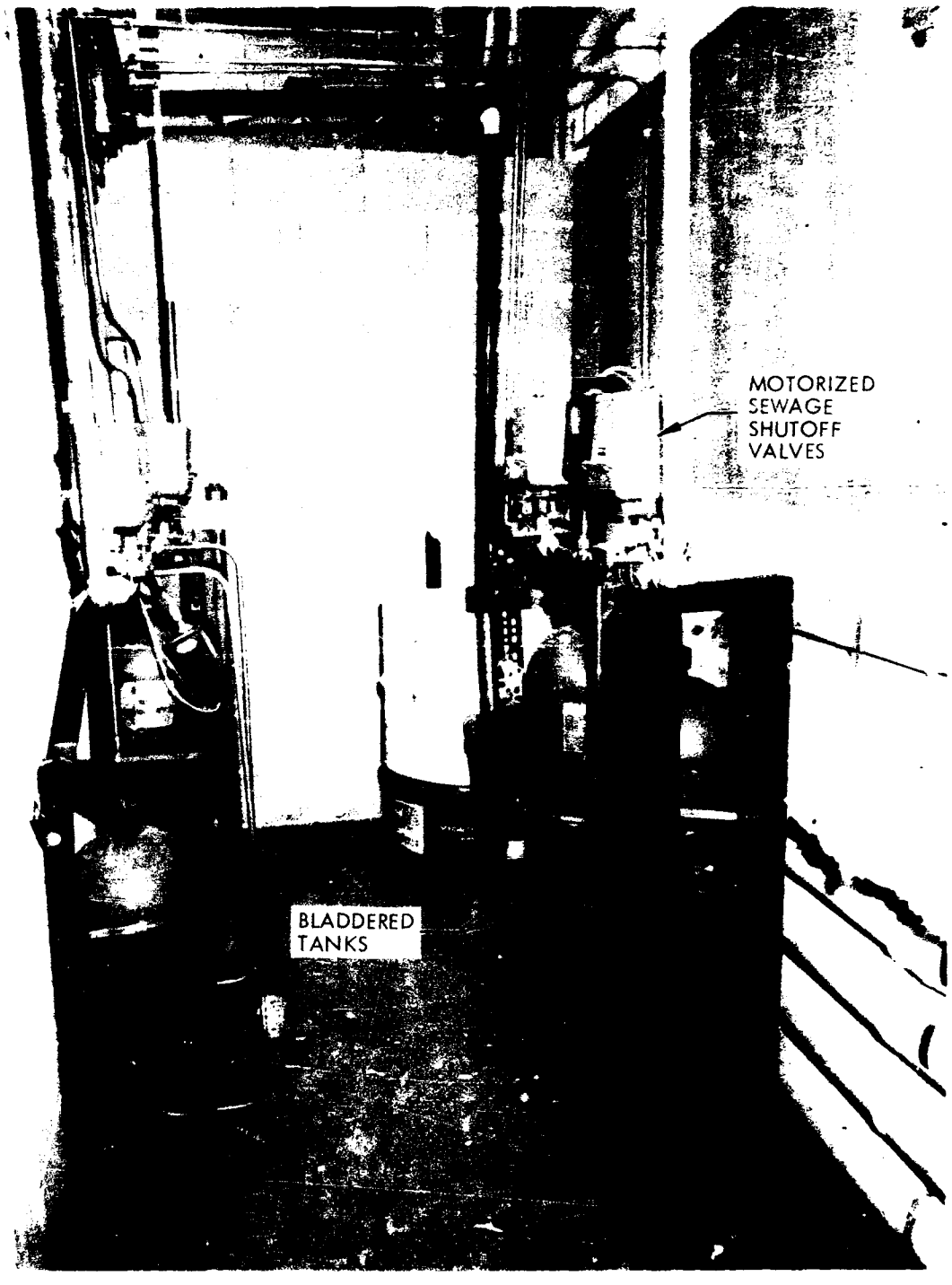


Fig. 34 Five Gallon Reactor Bladdered Tank Installation

or cylinders were used for additional safety so that failure of any single seal or bladder could not result in hydraulic oil being pumped into the hot reactor system. A booster pump was provided to assist the pump/grinder and thereby reduce tank fill time.

Thirteen test runs were made on the breadboard system with the Pt/Ru catalyst in the reactor. The tests were run in the same manner as the earlier breadboard system tests with respect to equipment operation except for the modified sewage pumping system. Air flow for these runs was maintained at 1 SCFM. Table 17 and Figure 35 present the data for the thirteen runs.

Table 17

Five Gallon Packed Tower Reactor Test Results
(Pt/Ru Catalyst on Alumina)

Run No.	Seq. This Series	Temp. (°F)	Press. (PSI)	Sewage Flow (GPM)	Contact Time (min)	Output TOC	% Red. in TOC
22	1	550	1200	0.25	20	33	93
23	2	550	1200	0.5	10	10	98+
24	3	500	1100	0.25	20	48	91
25	4	500	1100	0.5	10	84	84
26	5	500	1100	0.38	15	114	79
27	6	500	1100	0.25	20	72	87
28	7	500	1100	0.5	10	100	82
29	8	550	1200	0.25	20	41	92
30	9	550	1200	0.5	10	78	86
31	10	550	1500	0.5	10	72	87
32	11	550	1500	0.25	20	48	91
33	12	550	1500	0.17	30	36	93
34	13	550	1500	0.17	30	67	88

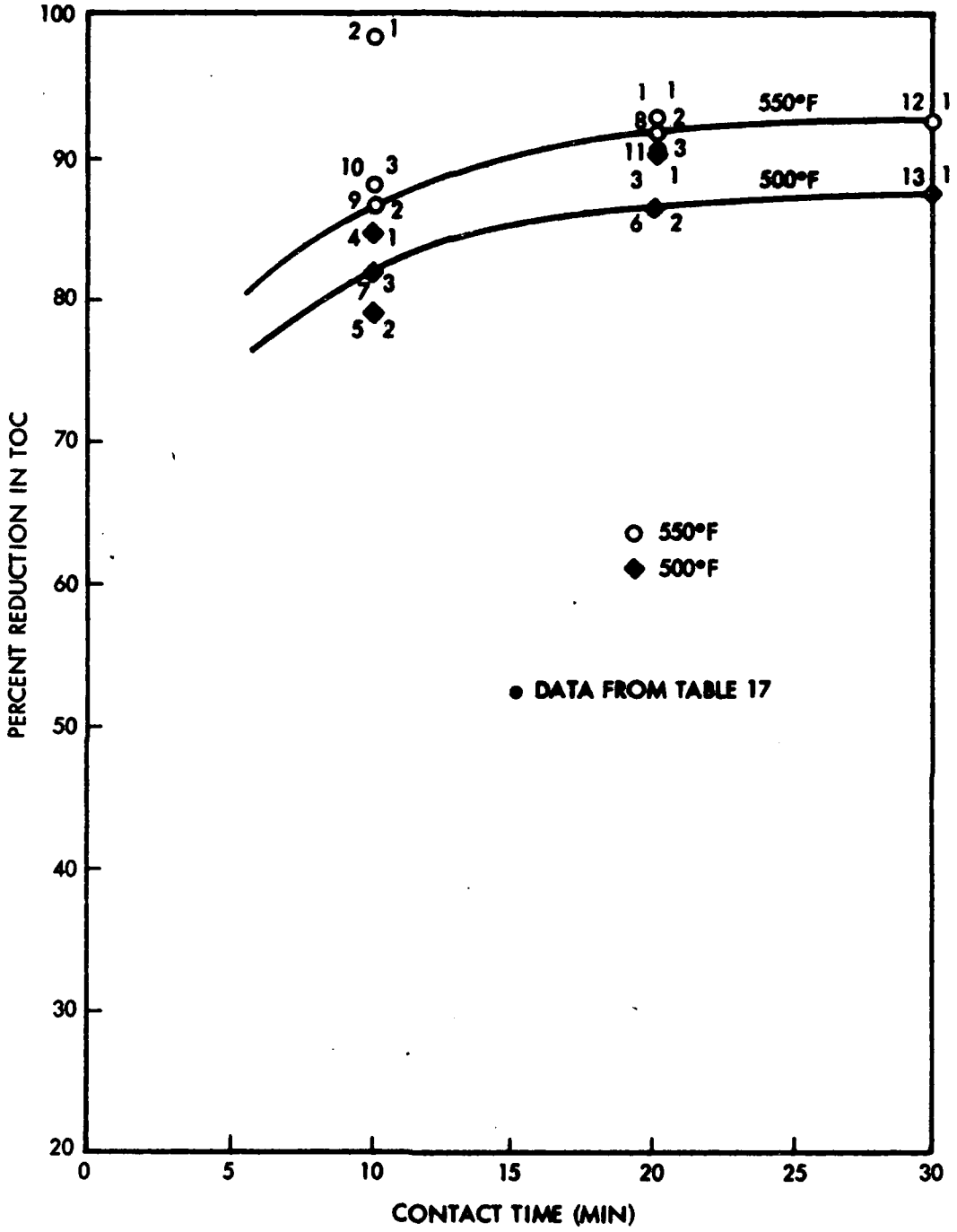


Fig. 35 Five Gallon Packed Tower Reactor Test Results

Two numbers appear by each data point shown on Figure 35. The one on the left is the sequence in which the runs were made without respect for process conditions and the one on the right is the sequence of runs at a given process condition. For example, run number 23 was the second of the series, but the first at 550°F and 10 minutes. Run number 30 was the ninth of the series of thirteen runs, but the second at 550°F and 10 minutes. Run number 31 was the tenth of the series and the third at 550°F and 10 minutes. By inspecting the sequence at each process condition and in the total series, the effect of catalyst wearin can be observed. The first data points taken at each process condition which were also the first runs of the series show higher performance than subsequent runs. The third point was either higher than the second or very close to the second indicating that the catalyst wearin had been completed. The two curves presented by Figure 35 show lower performance than identical data points from the one liter packed tower tests. These differences should be further studied and one important one was aspect ratio of the reactor. The one liter reactor was small in diameter compared to its height in comparison to the larger diameter shorter five gallon reactor.

The data from the five gallon reactor tests even though less than the one liter reactor test data did substantiate the selection of 550°F, 1200 psi and 20 minutes as the best process conditions for obtaining 90 percent or greater reduction in TOC with the lowest possible system weight volume and power.

Sodium Chlorate Tests - Following the data runs in the five gallon packed tower reactor using catalysts, the reactor was reconfigured for sodium chlorate tests. The catalyst and packing were removed and a chlorate pump was connected to the second thermocouple boss from the top of the reactor. A schematic of the test setup is presented by Figure 36. Figure 37 is a photograph of the chlorate pump installation. A chlorate solution was prepared so that when pumped into the reactor at flows in the range of 250 to 520 cc/hr the required amount of sodium chlorate would be introduced. Test runs 35 through 37 were run without chlorate injection to establish a baseline and runs 38 through 40 were run with chlorate. Although performance was not as good as

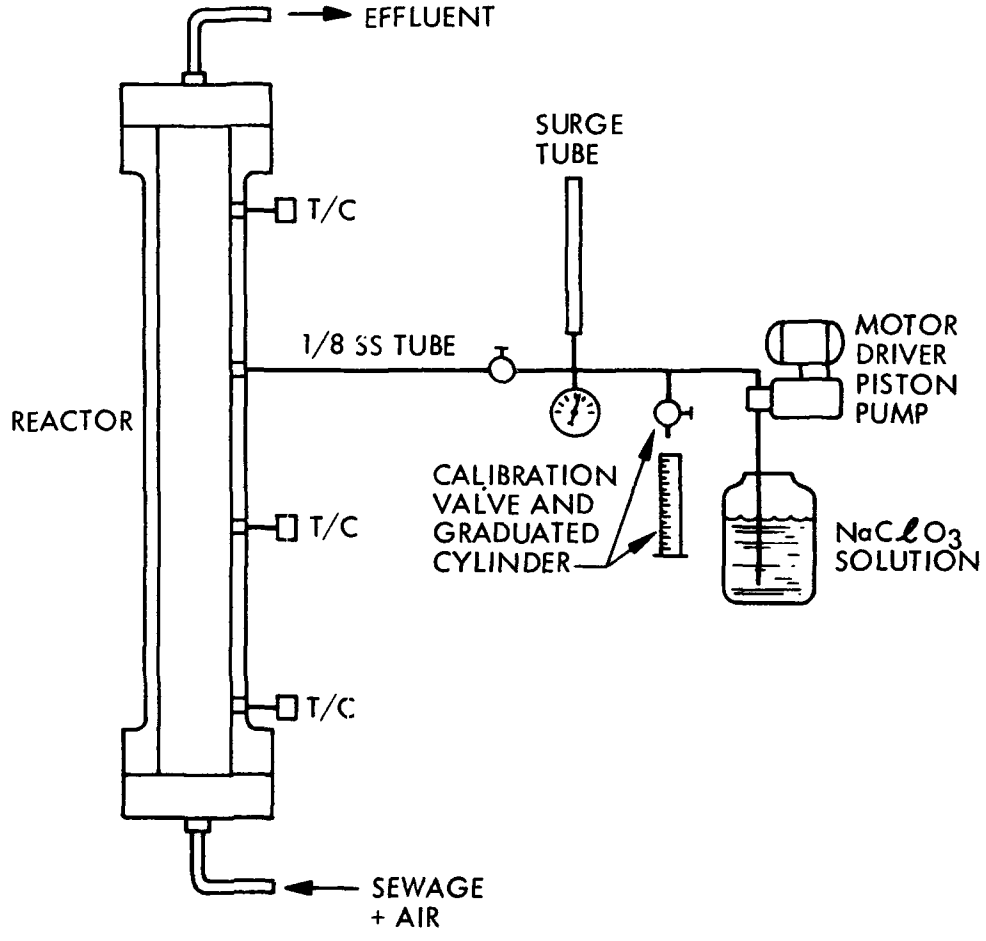


Fig. 36 Sodium Chlorate Injection System

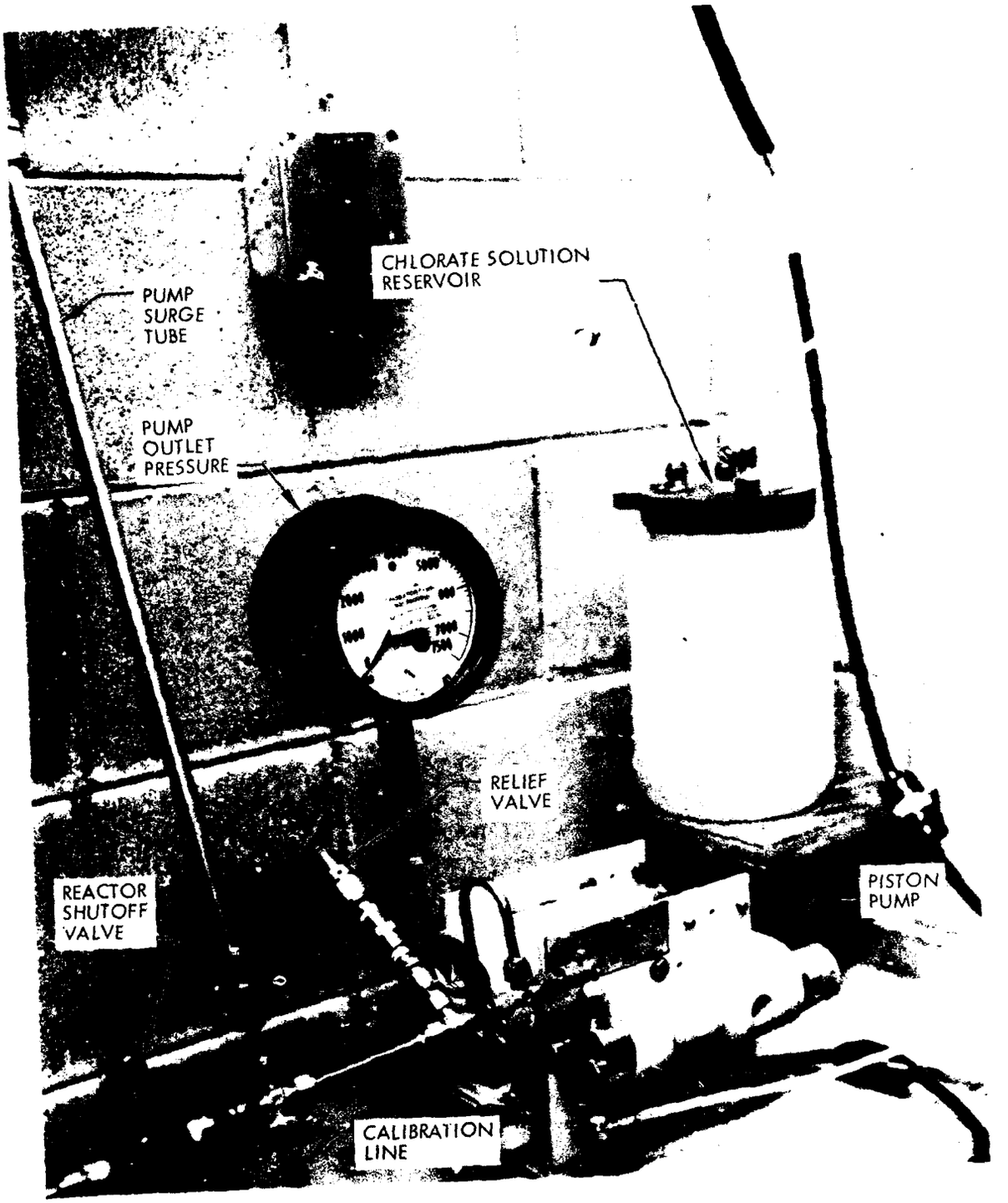


Fig. 37 Sodium Chlorate Injection Pump

that achieved in the stirred reactor (95-98 percent reduction in TOC in 20 minutes) it was extremely better than that achieved in the one liter packed tower.

Table 18

Five Gallon Packed Tower Reactor Sodium Chlorate Injection Test Results

Run No.	Temp. (°F)	Press (PSI)	Sewage Flow (GPM)	Contact Time (Min.)	$NaClO_3$ Flow (cc/hr)	Output TOC	% Red. in TOC
35	550	1500	0.33	15	0	171	67
36	550	1500	0.5	10	0	157	70
37	550	1500	0.17	30	0	145	72
38	550	1500	0.25	20	515*	58	89
39	550	1500	0.5	10	515**	101	81
40	550	1500	0.17	30	355*	42	92

*100 gm $NaClO_3$ in 500 cc solution

**200 gm $NaClO_3$ in 500 cc solution

It is interesting to note run 39 results where instead of increasing sodium chlorate solution flow the concentration of chlorate solution was doubled to get the higher chlorate input required for the highest sewage flow. This was done because 520 cc/hr was the upper limit for the injection pump, and the higher chlorate concentration avoided the necessity of installing another pump. The result, however, was not favorable indicating that higher flow rates probably assist mixing of the chemical with the sewage and/or limit decomposition in the inlet tube prior to injection. These tests give added promise to additional development work in this area on reactor configurations and chlorate mixing and injection techniques.

Wet Oxidation of Various Waste Mixes

During earlier tests on this contract and other related work in the laboratory, it was noted that the wastes mixed in accordance with the model were more difficult to oxidize than wastes comprised of feces and urine. To further investigate this a series of tests were run in the one gallon stirred reactor with the following mixes.

1) Feces and urine only
2) USCG Waste Model
3) USCG Waste Model minus feces and urine
4) USCG Waste Model minus soap, feces, and urine

The results of these tests shown by Table 19 and Figure 38 prove that the non-fecal-urine wastes are more difficult to oxidize. Additional work should be done to classify wastes, so that higher system performance can be achieved by recommending use of specific materials such as soaps, oils, detergents, etc.

Sea Water Effects

Tests using sea water sewage conducted early in the program using the breadboard system were terminated because of leakage in the reactor outlet tube. The hot salt water corroded the 316 stainless steel tube, developing cracks and pin holes in a matter of a couple of hours. In order to assess the effects of sea water on the system performance several runs were made in the titanium lined one gallon injection reactor. Figure 39 presents the results of two runs conducted during the catalyst screening tests. Both runs utilized 1.5 gpm of palladium black at 550°F and 2250 psig. Sea water used in run 26 was obtained from the Pacific Ocean by the San Francisco Coast Guard personnel. Results of these tests showed performance to be improved by use of sea water.

Table 20 and Figure 40 present data from tests run at the conclusion of the program. Fresh water and sea water runs were made with and without catalyst in the titanium lined one gallon stirred reactor at 550°F and 1500 psig. Catalysts used were 0.75 gm of platinum black and 0.75 gm of ruthenium black. The tests without catalysts produced the same results as the earlier test with palladium, i.e., sea water improves reduction in TOC for any given contact time. The Pt/Ru catalysts runs indicated a slight performance advantage for fresh water, but the curves are close enough so that the differences are not significant.

Table 19

Wet Oxidation of Various Waste Mixes

Waste Composition	Input TOC (mg/liter)	Output TOC (mg liter) at 1	3	6	12	30 min	% Reduction in TOC at 1	3	6	12	30 min
Feces and Urine Only	1058	526	401	327	257	176	50	62	69	76	83
USCG Waste Model (From Table 3)	527	303	283	211	154	114	43	47	60	71	79
USCG Waste Model Minus Feces & Urine	477	324	268	201	180	149	32	44	58	62	69
USCG Waste Model Minus Soap, Feces & Urine	436	372	278	206	151	120	15	36	53	65	73

All tests at 550°F, 1500 psi, uncatalyzed, in one gallon injection reactor.

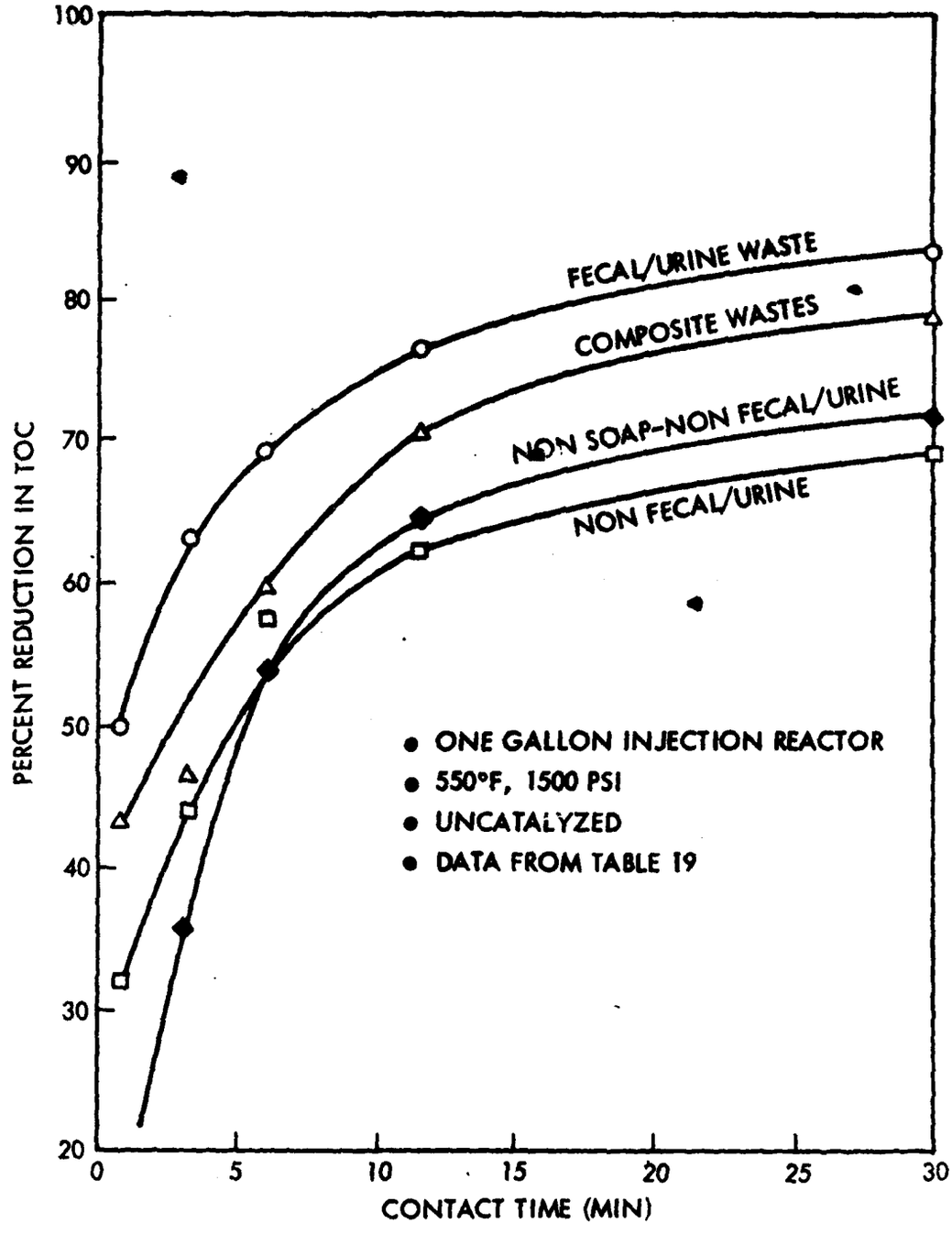


Fig. 38 Wet Oxidation of Various Mixed Wastes in a One Gallon Stirred Reactor

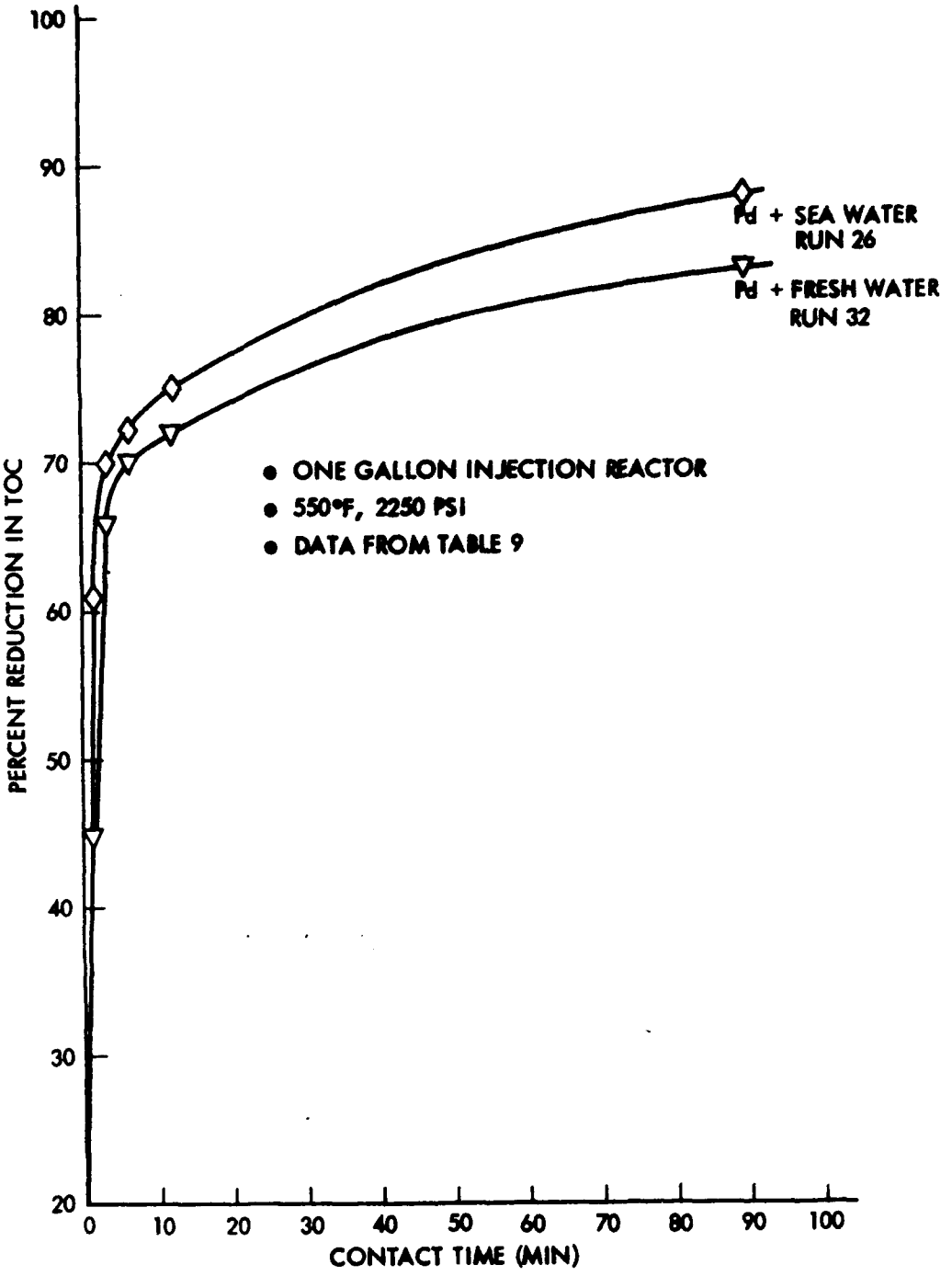


Fig. 39 Effects of Sea Water on Wet Oxidation System Performance

Table 20

Sea Water Effects

Test Fluid	Catalyst	Input TOC (mg/liter)	Output TOC (mg/liter) at 1	3	6	12	30	90 min	% Reduction in TOC at 1	3	6	12	30	90 mn
Fresh Water	None	436	389	420	239	176	123	49	11	4	45	60	72	89
Sea Water	None	436	358	304	244	179	104	39	18	30	44	59	76	91
Fresh Water	Pt/Ru	436	199	122	84	44	14	7	54	72	81	90	97	98
Sea Water	Pt/Ru	436	216	131	90	61	13	7	50	70	80	86	97	98

All tests at 550°F, 1500 psi, USCG Waste Model from Table 3, in one gallon injection reactor.

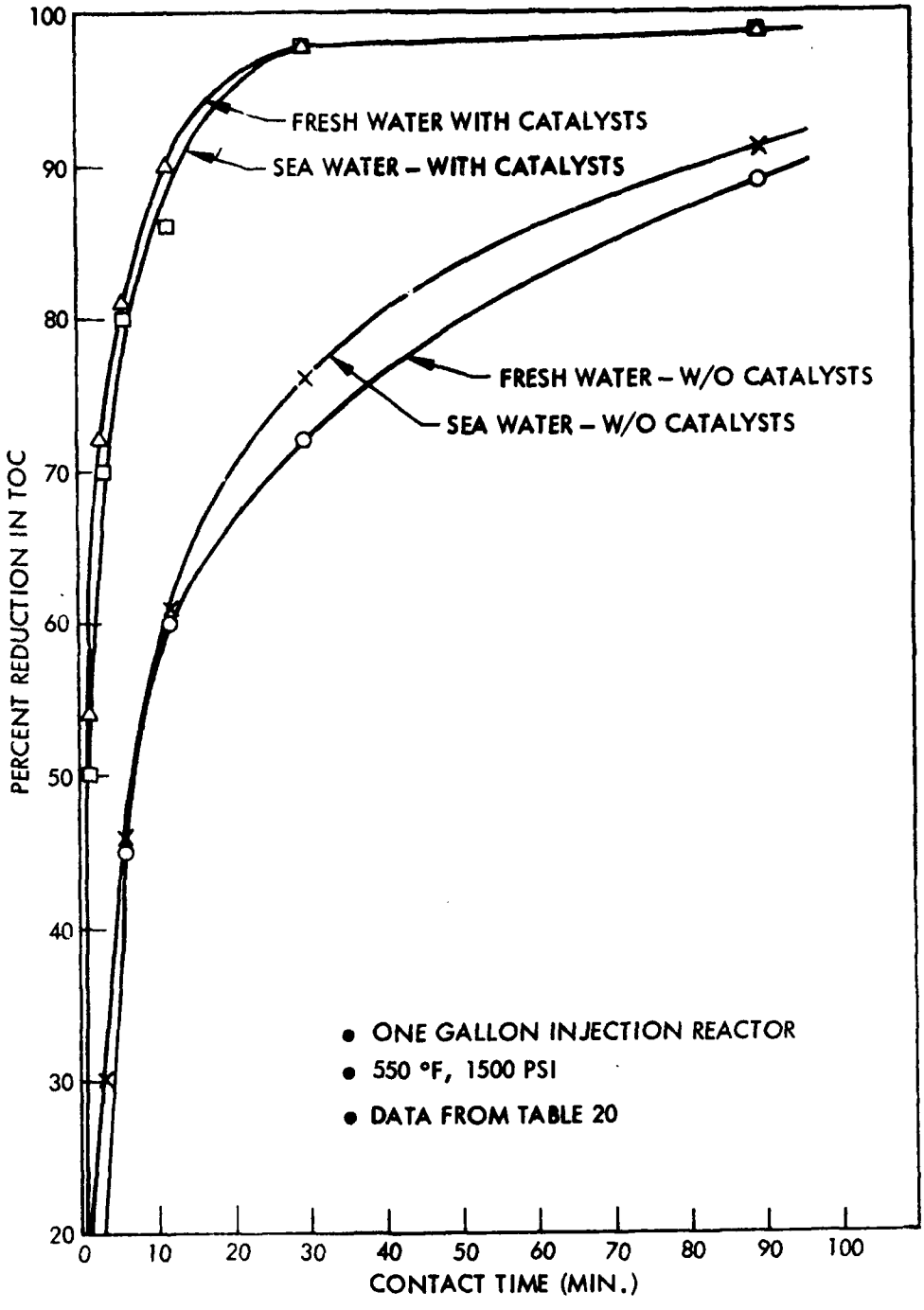


Fig. 40 Effects of Sea Water on Wet Oxidation System Performance

TUBULAR REACTOR STUDIES

The packed tower reactor used during the breadboard system test program was a five gallon, five foot high reactor. The test program showed a ten gallon reactor was required to provide the needed 20 minute contact time at the design sewage flowrate of 0.5 gpm. To maintain the same reactor length to diameter ratio in the preliminary design reactor as tested in the breadboard test system a taller reactor would have been required. Envelope constraints on a shipboard system made a taller reactor vessel impractical, so a multiple tube reactor concept was developed. Multiple tubes, three feet long to meet ship height limitations, packaged in a bundle and connected in series could be used to approximate the design of a single taller tube. The number of tubes and tube diameter would affect flow distribution and mass transfer.

Reactor Flow Models

In order to better understand the flow distribution taking place inside the reactor and to select a multiple tube reactor tube diameter and number of tubes, a series of flow model tests was devised. Acrylic plastic tubes 4 ft. high were fabricated in 2", 3", 4", 6" and 9" nominal diameters. These were incorporated into a flow bench which allowed measured quantities of water and air to be mixed together and passed through the tubes. Three types of tests were run. These were vertical bubble tests, horizontal tests, and chemical oxygen addition tests. All tests unless noted, were run at the nominal system inlet flow rate of 0.5 GPM liquid and 1.6 SCFM air. Due to pressure, temperature, and vaporization effects the actual gas flow in the reactor during operation is about 6000 cc/min; and this flow was used for the flow model test.

Vertical Bubble Tests - Vertical bubble tests were run in all sized tubes with the tubes in a vertical position. The water and gas entered the bottom of the tube, and flowed out at the top. Three types of tests were run and these are described in the paragraphs which follow.

Bubble Distribution Tests - Tests were run in all size tubes to establish under what conditions the best air distribution could be obtained. Diffusers, which consisted of metal plates with an even distribution of holes, were fabricated and placed in the bottom of the empty tubes. The hole diameter was then adjusted until satisfactory performance resulted. The same procedure was then repeated with about 6 inches of catalyst on top of the diffusers. Tests were run using a range of flow rates for both conditions and all tests were photographically recorded. Pictures of the reactor model at design flow conditions (4 inch tube - 0.5 GPM, 1.6 SCFM) both with and without catalyst are shown in Figure 41 and 42. Photographs of reactor models tested at off design conditions are presented in Appendix A. The tube inside diameter, water flow rate and gas flow rate are shown in order from top to bottom on the tag attached to each tube in the photographs.

The conclusions drawn from these tests were:

- Even distribution of gas can be ahieved in any size tube with a properly designed diffuser, although even distribution is more difficult to achieve in the larger tubes.
- Catalyst improves distribution slightly.
- At the nominal gas flow rate, bubble size is fairly independent of flow rate and tube size; but bubble size increases at very high gas flow rates.
- In smaller tubes (4" and less) distribution of gas over the cross section is adequate even without a diffuser.
- Liquid velocity (at .5 GPM) did not influence gas distribution or bubble behavior.
- Liquid carryover is small at gas flow rates below those where very large bubbles are formed.

Large Bubble Formation and Bed Fluidizing Tests - These tests were run over a a range of gas flow conditions in all size tubes. At lower flows per unit area the bubbles were nearly the same size regardless of gas flow rate. At very high flows, however, the bubbles merged into large bubbles. Also observed was the point at which the catalyst particles in the top 1 inch of the bed started to lift and vibrate. This phenomenon is called fluidizing, and is undesirable as catalyst damage can result. Conclusions drawn from these tests were:

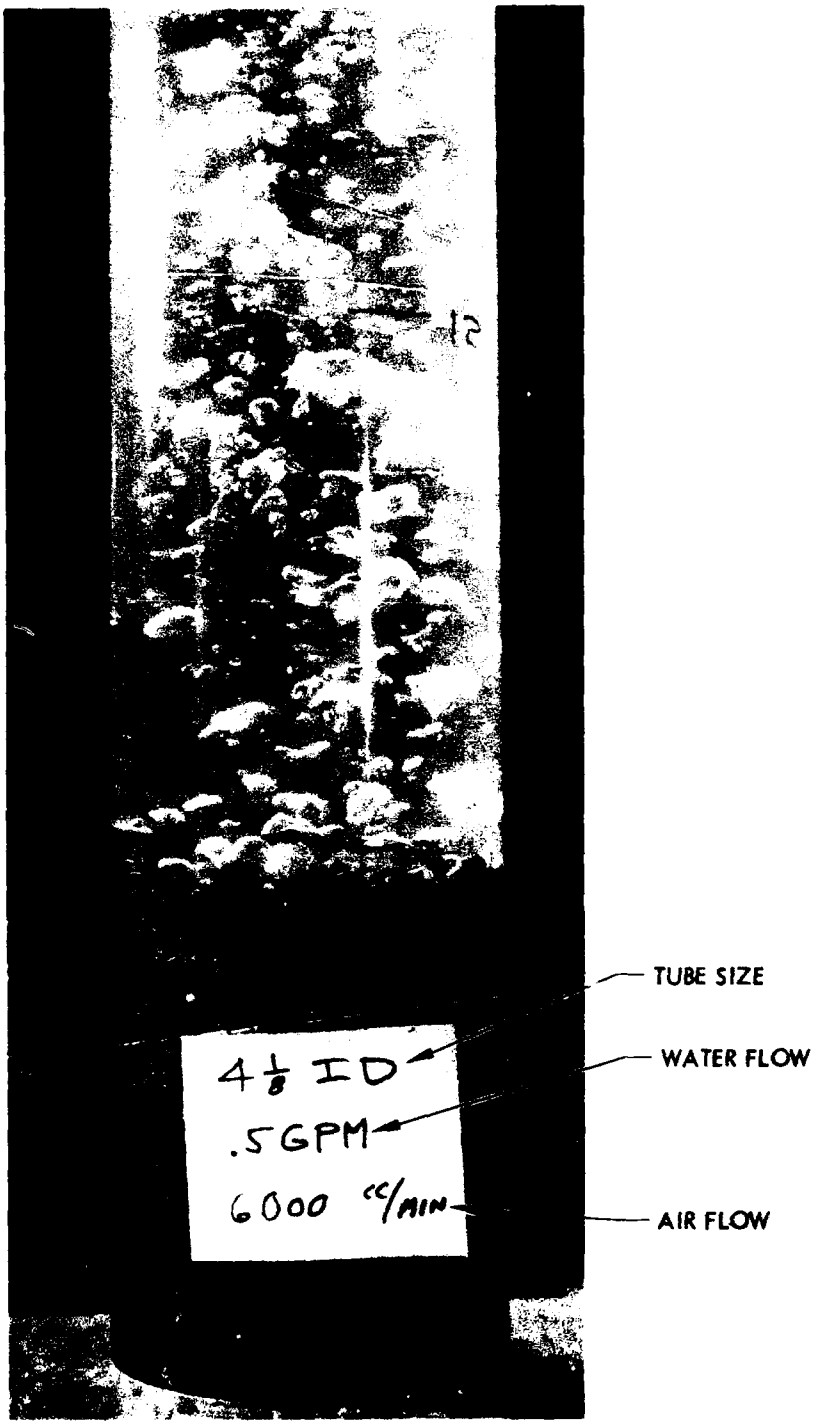


Fig. 41 Reactor Flow Model Tests (Design Flow with Catalyst)

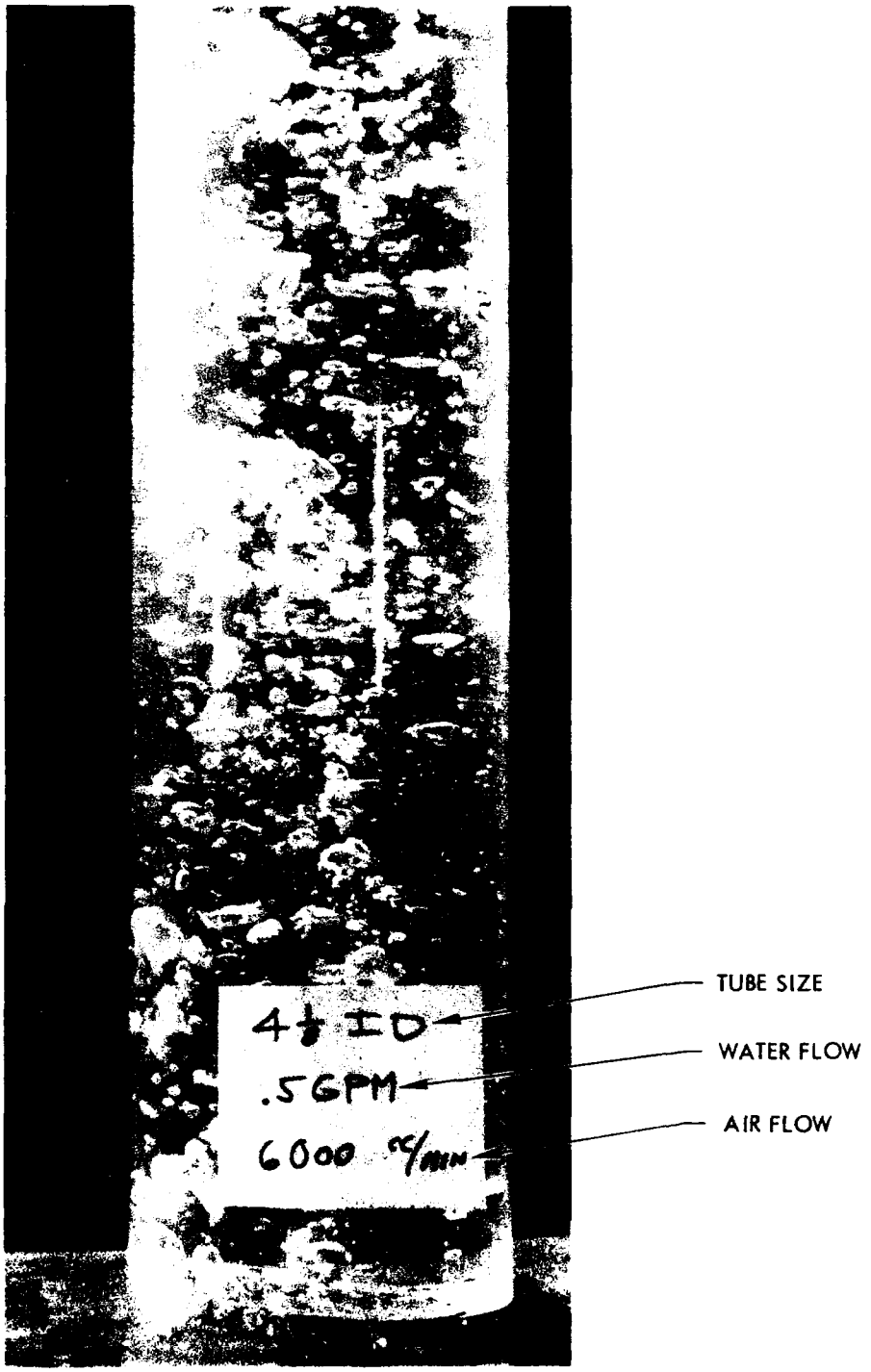


Fig. 42 Reactor Flow Model Tests (Design Flow without Catalyst)

- Large bubble formation occurs at 1000 to 1200cc/min-in.^{2} in all tube sizes.
- Fluidizing occurs at 700 to 1000 cc/min-in.^{2} in all tube sizes. Larger tube sizes fluidize at flows nearer 700 cc/min-in.^{2}, as uniform gas distribution is more difficult to achieve.
- The 4 inch tube is the minimum suitable size for the projected design flow. It has a gas flow of 440 cc/min-in.^{2}, while the 3" size is 780 cc/min-in.^{2} and is therefore close to flows where fluidizing occurs and large bubbles form.

Reactor Tilt Tests - These tests were run by tilting the flow models at various angles and observing the disruption of the flow pattern. Conclusions drawn from these tests were:

- Small tubes are less affected by tilt angle.
- Angles at which serious flow disruption occurs at the design flow rate are: 2"-15°, 3"-10 to 12°, 4"-8 to 10°, 6"-5 to 7°, 9"-3 to 5°.

It should be noted that the transient 10° pitch and 40° roll requirements, with a 10 second period of oscillation, would not affect reactor performance due to the slow liquid velocity in the tubes. The required permanent 3° trim would not affect operation, and operation at the 15° permanent list would require the addition of 2 or 3 gas redistribution plates inside the reactor tubes. These would not otherwise affect reactor performance.

Horizontal Reactor Tests - Flow tests were run to evaluate the flow distribution achieved in horizontal tubes. Baffles were fabricated and inserted into the tubes to assist in mixing the liquid and gas phases. Conclusions from these tests were:

- With tubes 2" dia and larger and air flows less than 6000 cc/min. velocities were insufficient to sustain mixing and gas-liquid stratification occurred almost immediately.
- To promote good liquid/air mixing in a horizontal tube, the tube must be broken into many short vertical passes.
- The seal between the baffles and main tube wall must be good, as even small clearances allow a considerable amount of gas leakage.

Chemical Oxygen Addition Tests - These tests investigated the mixing and diffusion characteristics of sodium chlorate, the chemical used for addition of extra oxygen to the reaction process. Previously run tests in the packed tower reactor failed to produce the favorable results obtained in the batch type stirred reactor. The flow model tests were run to determine whether poor mixing in the reactor could be the cause of poor chlorate performance in the tower reactor. Since sodium chlorate is clear, sodium dichromate ($Na_2Cr_2O_7$) was selected for the visual tests as it is almost identical in diffusion characteristics and has a bright yellow color, thus allowing visual observation of mixing.

A $Na_2Cr_2O_7$ solution equal in concentration to the sodium chlorate solution normally used in the reactor tests was injected into the flow system at several points using a metering pump. Injection points included the inlet line upstream of the large tube, the inlet line upstream of a static mixer, and directly into the large tube both upstream and downstream of the diffuser plate. In all cases mixing occurred very quickly, and therefore, any of the injection locations should be adequate in the shipboard system.

Four Tube Reactor Design

A 4 tube reactor using 4 inch nominal diameter tubes was chosen for testing in the breadboard system based on package considerations from the preliminary design, and the results of the reactor flow model tests. The unit was to have a 10 gallon capacity to provide a nominal 20 minute contact time at the 0.5 GPM liquid flow rate. However, due to availability of tubing and the preliminary design height requirement of 4 feet, the actual volume was 8.6 gallon. The reactor, as shown in Figure 43, consisted of 4 tubes mounted on a stand. The tubes were interconnected with plumbing, wrapped with heater tapes, and surrounded with insulation to minimize heat loss. The tubes were constructed of schedule 40, 316 stainless steel pipe with a 4.5 inch O.D. The straight section of pipe was 35 inches long, and hemispherical pipe caps were welded to each end. 1 inch pipe couplings welded into the pipe caps provided inlet and outlet fittings. The overall tube length was 40 inches.

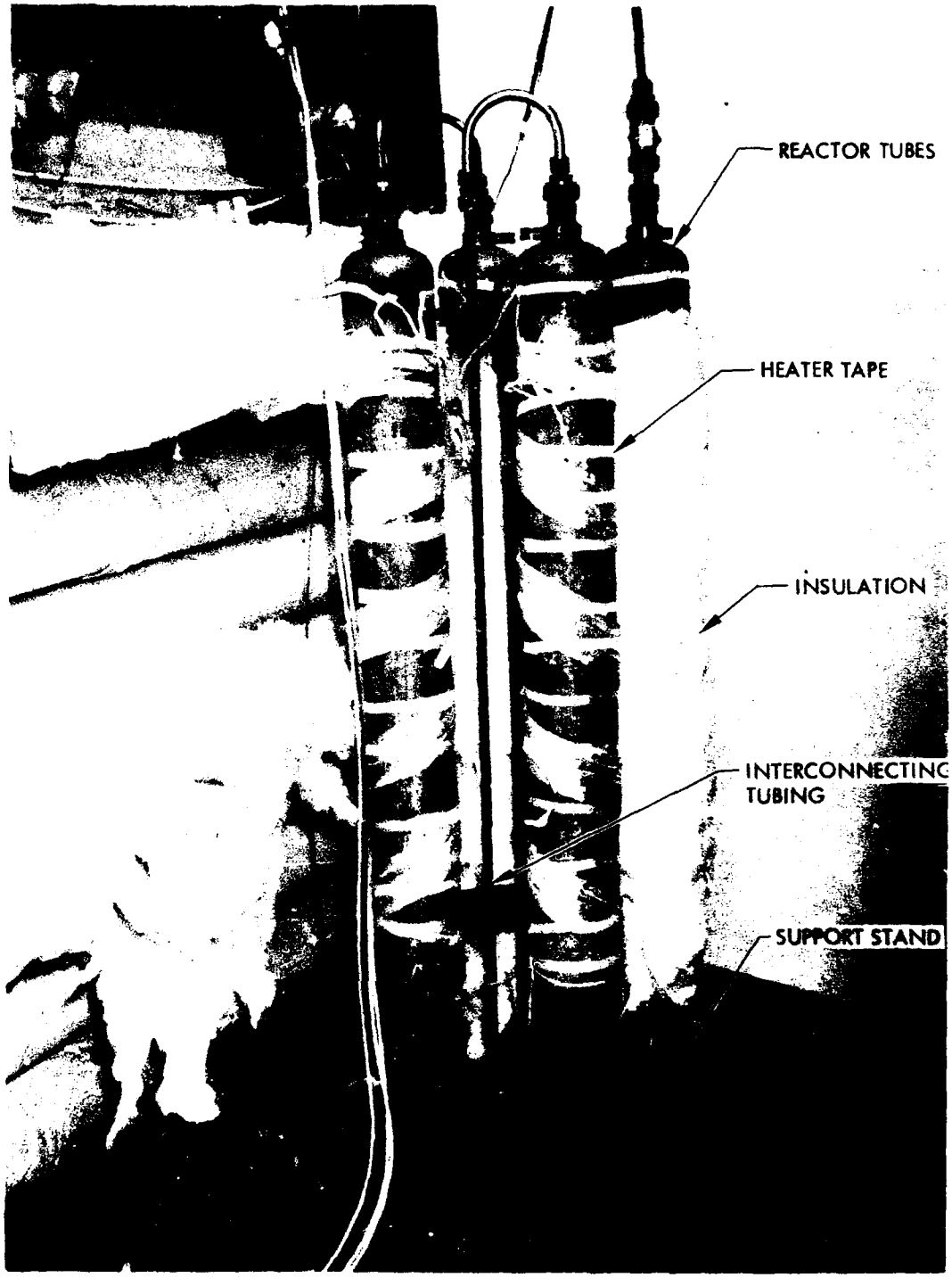


Fig. 43 Four Tube Reactor Assembly

The stand was constructed of 2" schedule 40 pipe welded to a 1/2" carbon steel plate. Supports were provided which allowed bolting of the tubes to the stand, allowing for thermal expansion and tube removal. The interconnecting plumbing was 1/2 inch 316 stainless tubing. It was fabricated in sections to allow changing of flow configurations. The heater tapes were Briseoe Mfg. Company, Part Number BWH-62 1/2 having 1296 watts each at 120 VAC. They were individually wrapped around the tubes and operated two in series at 208V, thus giving a total power of approximately 3.9 KW.

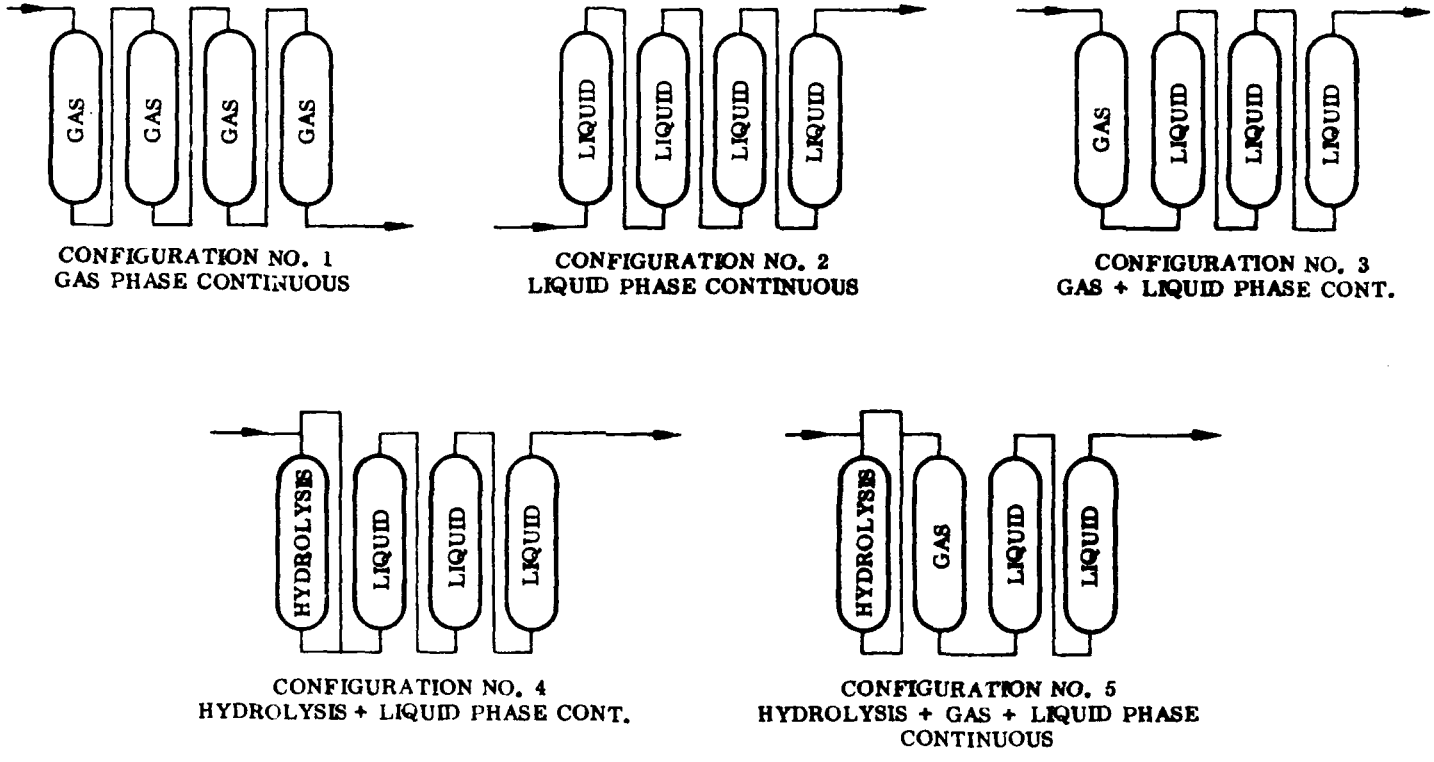
The whole assembly was insulated with about three inches of fiber glass blanket insulation, which was wrapped around the tubes, and around the whole assembly. Temperature control thermocouples were provided at the top of each tube.

Four Tube Reactor Tests

After manufacture, the 4 tube reactor was incorporated into the breadboard test system, and a series of test runs were made. These included both catalyst and sodium chlorate injection runs.

Catalyst Tests - The catalyst tests used the same half and half mixture of 0.5% platinum on alumina and 0.5% ruthenium on alumina catalysts previously used in the 5 gallon reactor. The catalyst assisted in the oxidation process, and allowed a lower operating temperature and pressure.

All possible variations of configuration using 4 tubes were identified, and the most likely five candidates were chosen for testing. As shown in Figure 44, these were (1) series flow-gas continuous tubes, (2) series flow-liquid continuous tubes, (3) 1 gas tube followed by three liquid continuous tubes, (4) hydrolysis in 1 tube followed by three liquid continuous tubes, and (5) hydrolysis in 1 tube followed by gas continuous flow in the next tube, and liquid continuous flow in the last two tubes. Catalyst was used in all 4 tubes in configurations 1, 2, and 3; but was used only in the last three tubes of configurations 4 and 5.



CONFIGURATION NO. 1
GAS PHASE CONTINUOUS

CONFIGURATION NO. 2
LIQUID PHASE CONTINUOUS

CONFIGURATION NO. 3
GAS + LIQUID PHASE CONT.

CONFIGURATION NO. 4
HYDROLYSIS + LIQUID PHASE CONT.

CONFIGURATION NO. 5
HYDROLYSIS + GAS + LIQUID PHASE CONTINUOUS

Fig. 44 Tubular Reactor Configurations

As summarized in Table 21, a series of runs was made to evaluate all configurations. The performance was divided into two groups. In the first group, runs 49 through 61, the high flow and low flow reduction in TOC were 90 to 91% and 94 to 96% respectively. In the second group, runs 62 to 69 the reductions were 85 to 86% and 91 to 93%. The step drop in system performance was unexplainable so, an investigation was undertaken to determine whether or not any uncontrolled changes in system parameters might have taken place during the testing. It was discovered that the system flow rate was about 10% high, and that some waste model ingredient substitutions had occurred. These were substitutions of another brand of soap or cleanser for the brand originally used in the sewage model.

In order to eliminate the effects of varying input model, the original mix was recreated, and the system was rerun. Performance of these runs (71 to 74) improved slightly, but the original performance was not duplicated.

The four tube reactor tests resulted in selection of configuration 3 and revision of the contact time from 20 to 30 minutes. A 15 gallon reactor was required to achieve the 30 minute contact time.

Sodium Chlorate Injection Tests - Sodium chlorate tests run in the five gallon reactor did not produce as high a percent reduction in TOC as achieved in the stirred batch reactor. To further study sodium chlorate performance, a series of 6 runs was made using the 4 tube reactor. Three different sodium chlorate injection points were tested; (1) in the line between the third and fourth tubes (tests 43 and 44), (2) in the line between the third and fourth tubes with a static mixer downstream of the injection point (tests 45 and 46), and (3) directly into the bottom of the fourth tube (tests 47 and 48). Each configuration was tested at 0.25 GPM of slurry with 0.8 SCFM of air and at 0.5 GPM of slurry and 1.6 SCFM of air. The reactor tubes were always connected in a liquid continuous series flow configuration and there was no catalyst or packing in any tube. The amount of sodium chlorate injected was the same as used previously. It was, however, diluted with twice as much water to allow a

Table 21

Four Tube Reactor Test Results

(Catalyst Runs)

Run No.	Catalyst Config.	Liquid Flow GPM	Air Flow SCFM	Outlet TOC	% Red. in TOC
49 (See Note 2)	4	.5	1.6	28	93.3
50	4	.25	.8	23	94.5
51	4	.5	1.6	60	85.7
52	4	.25	.8	32	92.4
53	4	.5	1.6	49	88.3
54	5	.25	.8	24	94.3
55	5	.25	.8	26	93.8
56	5	.5	1.6	50	88.1
57	3	.25	.8	14	96.6
58	3	.5	1.6	42	90.0
59	3	.25	.8	15	96.4
60	3	.5	1.6	39	90.7
61	2	.25	.8	19	95.5
62	2	.5	1.6	63	85.0
63	1	.25	.8	33	92.1
64	1	.25	.8	37	91.2
65	1	.5	1.6	72	82.8
66	2	.25	.8	30	92.8
67	2	.5	1.6	63	85.0
68	3	.25	.8	35	91.6
69	3	.5	1.6	61	85.5
71	3	.25	.8	34	91.9
72	3	.5	1.6	56	86.6
73	3	.25	.8	35	91.6
74	3	.5	1.6	56	86.6

NOTES: 1. All tests run at 1200 psi and 550°F
2. Break-in run with fresh catalyst
3. All % reductions based on inlet TOC of 420

more accurate control over the inlet flow rate, to increase mixing and to minimize decomposition in the inlet line. The high and low chlorate flow rates were 2064 cc/hr and 1032 cc/hr respectively, using a solution of 50 grams sodium chlorate in 500 cc of water. The solution was injected by a metering pump.

As shown in Table 22 results for all configurations were disappointing as the reduction in TOC was only 60 to 70%.

Table 22

Four Tube Reactor Test Results

(Sodium Chlorate Injection)

Run No.	Chlorate Injection Config.	Liquid Flow GPM	Air Flow SCFM	Outlet TOC	% Reduction in TOC (inlet = 420)
42	1	.5	1.6	195	53.6
43	1	.25	.8	143	65.9
44	1	.5	1.6	158	62.4
45	2	.25	.8	123	70.7
46	2	.5	1.6	155	63.1
47	3	.25	.8	124	70.4
48	3	.5	1.6	164	60.9

There was very little difference in performance between the various injection locations and reduction was little better than without chlorate injection (run 42).

In order to determine why the reduction had not taken place, an analysis of the effluent was made. From this analysis, it was determined that residual chlorate was present in the effluent at concentrations that would indicate little, if any, decomposition was taking place. It was, therefore, obvious that the chlorate was entering the system and was being mixed with the process fluid, but was not breaking down to release oxygen. The reason why chlorate worked so well in a batch stirred reactor and not in a packed tower remains a problem to be resolved.

SHIPBOARD PRELIMINARY DESIGN

A shipboard system preliminary design based on the results of the breadboard system tests was prepared. This section describes the system operation and design characteristics and presents weight, envelope and power requirements for the shipboard system.

System Operation

The shipboard wet oxidation system shown schematically in Figure 45 is designed to process 700 gallons per day of sewage with suspended solids and biological oxygen demand ranging between 4900 mg/l and 200 mg/l, and 1900 mg/l and 150 mg/l, respectively. It processes the wastes to provide an effluent with a maximum of 50 mg/l suspended solids and BOD, and a maximum total colliform content of 240 MPN/100 ml.

The system designed to meet these requirements, provides a surge tank and grinder to condition the sewage, a hold tank to accommodate variations in system flow rate, a sewage feed pump to force the sewage into the reactor, an air compressor to supply process air, a regenerative heat exchanger to recover effluent heat, a heated reactor to support the oxidation process, liquid level and gas vent controls to maintain the system pressure, and various other controls to assure safe operation. The system is fully automated, and provides alarms if an emergency condition occurs.

During system operation, sewage from the ships drains pass into the surge tank, through the grinder, and into the hold tank. The grinder runs continuously, but is flooded with liquid only as the drains are used. An overflow into the hold tank is provided in the event that the capacity of the grinder is

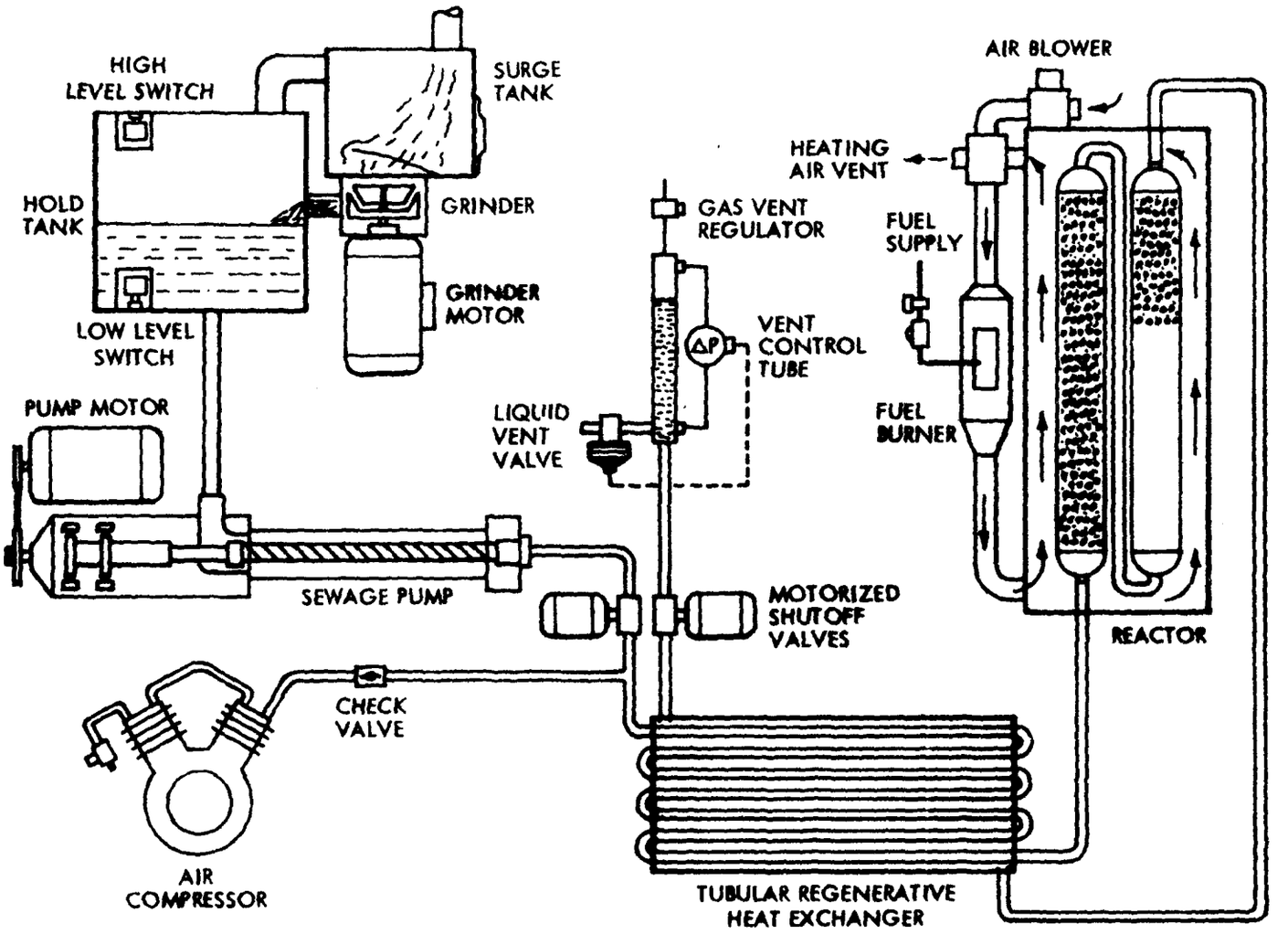


Fig. 45 Shipboard System Schematic

momentarily exceeded. When the level in the hold tank is above the low level shut off switch, the sewage pumps, and air compressor are started, thus initiating system flow. If the level exceeds the high limit set point, an alarm is sounded. The sewage pump and air compressor run continously during operation, and feed is at a constant rate. Sewage discharge from the pump enters the regenerative heat exchanger at a nominal temperature of 50°F where it is heated to 525°F. It then enters the reactor where it is heated to 550°F, and the sewage oxidation takes place. The reactor is filled with a catalyst to promote the oxidation. The nominal reactor pressure is 1200 psi, and the air and sewage flow rates are 1.6 SCFM and 0.5 GPM respectively. The contact time in the reactor is 30 minutes. Two types of reactor heaters are presented; a fuel fired system which meets the specified electrical power consumption of 4 KW, and consumes 0.8 lb/hr of light fuel oil and an electrically heated system which is simpler but consumes 6.9 KW of power during steady state operation.

In the fuel fired version, 60 lb/hr of air from the fan passes through the regenerative heat exchanger where it is heated to 446°F. It then passes into the burner and is raised to 1365°F by the combustion of 0.8 lb/hr of light oil. Leaving the burner, it passes into the reactor transferring heat to the liquid, and exits at 570°F. Transfer of heat to the inlet air stream, which takes place in the heat exchanger, reduces the exhaust air temperature to 175°F. The electrically heated reactor is similar, except that heaters fastened directly to the reactor tubes supply 3.4 KW of heat, and therefore the air to air regenerative heat exchanger, fan, and air circulation loop are not required. A system heat balance for both reactor heater systems is presented in Figure 46.

Control of both reactor concepts is independent of other system operations. In the fuel fired version, the burner pilot is controlled by a flame sensor. Lack of pilot flame closes the pilot fuel valve and prevents opening of the main fuel valves. Two controllers are used in series to control the burner flame level. One senses the fluid temperature at the reactor outlet, and

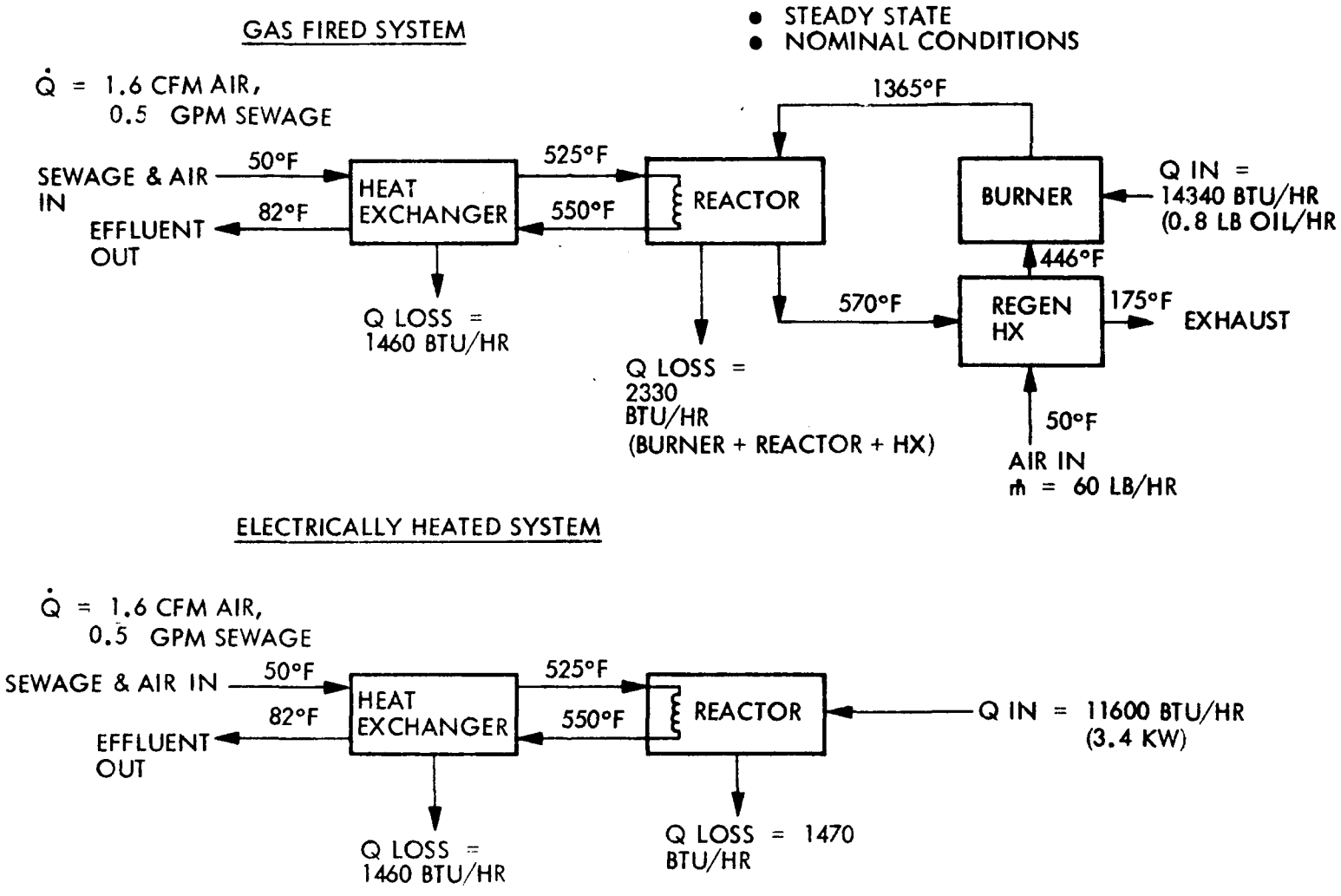


Fig. 46 System Heat Balance

the other senses the air temperature leaving the burner. High limits are built into both controllers, and a high limit signal from either shuts down both the burner and the rest of the system, and rings an alarm. In the electrically heated version, one controller modulates the electrical power to the heaters.

After leaving the reactor, the effluent passes through the heat exchanger, transferring its heat to the influent. It then passes into the level control system which consists of a pipe, 1 inch in diameter and 46 inches high, which serves as a phase separator for the effluent gas and liquid. System pressure is controlled by a back pressure regulator vent control. Liquid level in the column is measured by a ΔP sensor which modulates a liquid vent valve to control the level in the column.

This control concept is utilized, because it eliminates the need for a two phase flow regulator, with its attendant-leakage problems.

If the sewage level in the hold tank reaches the low level shutoff switch, the compressor and sewage pump are de-energized. Motorized shutoff valves are provided at the inlet and outlet of the heat exchanger to prevent bleed-down of the pressure. These close automatically when system feed is stopped.

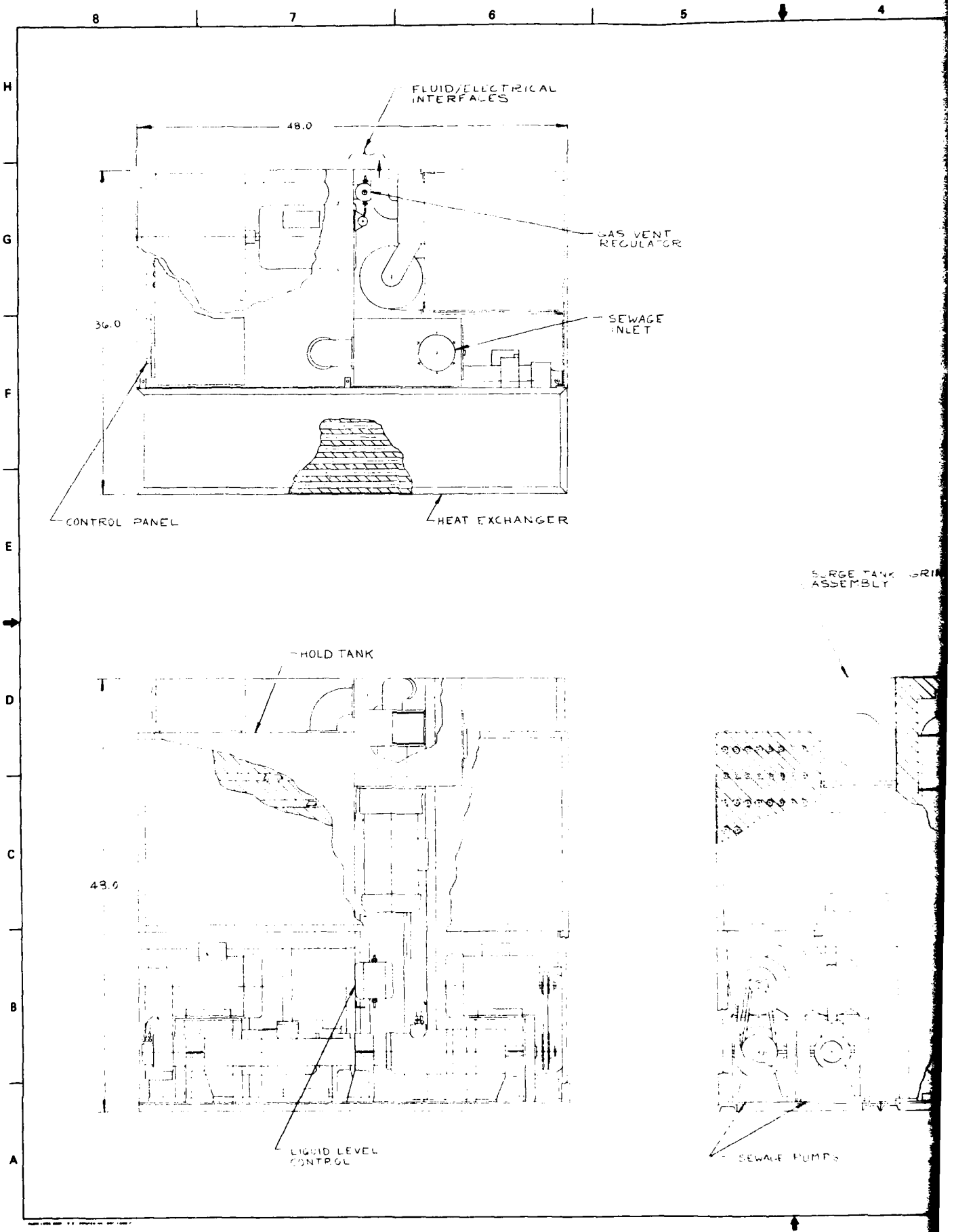
Controls provided for emergency purposes include reactor high and low pressure switches which turn off the system if pressure deviates from a specified range, a sewage feed pump ΔP sensor which initiates a warning if the ΔP across either pump exceeds the specified value, and rotation sensors on the pumps and compressor which shut off the system if any of these components stop. Also, thermal overloads are provided on each component with a motor.

System Design

Figure 47 presents the system assembly. All components are packaged into a 3x4x4 ft envelope. Additionally, the components are grouped as subassemblies where possible, and the subassemblies are designed so they will pass through a 24 x 24 inch hatch or 22 x 66 inch door. Required subassemblies are the reactor assembly, the heat exchanger, the surge tank and grinder assembly, the hold tank, the sewage pumps and motors, and the air compressor. A mounting base plate is also provided and all components are mounted to this base and/or each other. It should be noted that due to the modular construction of the subassemblies, the system could easily be packaged in arrangements other than the 3'x4'x4' module. For instance, subassemblies could be arranged individually in a ship on a space-available basis.

The surge tank-grinder assembly is located as high as possible in the package to assure gravity flow of sewage into the hold tank. Flooding of the grinder occurs only during sewage feed, reducing the overall power consumption. The unit is attached directly to the hold tank assembly which is located just above the sewage pumps, assuring gravity feed and a continuous prime of the pumps. This location is also ideal for the pumps, as they are supported directly by the base plate, and a minimum of mounting structure is required.

The reactor assembly, heat exchanger, and air compressor have no particular mounting requirements other than vertical orientation, and are therefore distributed throughout the package where space permits. The reactor assembly includes a burner, a fan which provides a forced draft, and a regenerative air to air heat exchanger to reduce fuel consumption and the circulation loop air temperature. All components are mounted to the reactor itself, or to the surrounding insulation and structure. The subassembly is provided with its own base assembly, and this mounts directly to the system base plate.


8
7
6
5
4
H
G
F
E
D
C
B
A
FLUID/ELECTRICAL INTERFACES
48.0
GAS VENT REGULATOR
SEWAGE INLET
36.0
CONTROL PANEL
HEAT EXCHANGER
SURGE TANK GRID ASSEMBLY
HOLD TANK
43.0
LIQUID LEVEL CONTROL
SEWAGE PUMPS

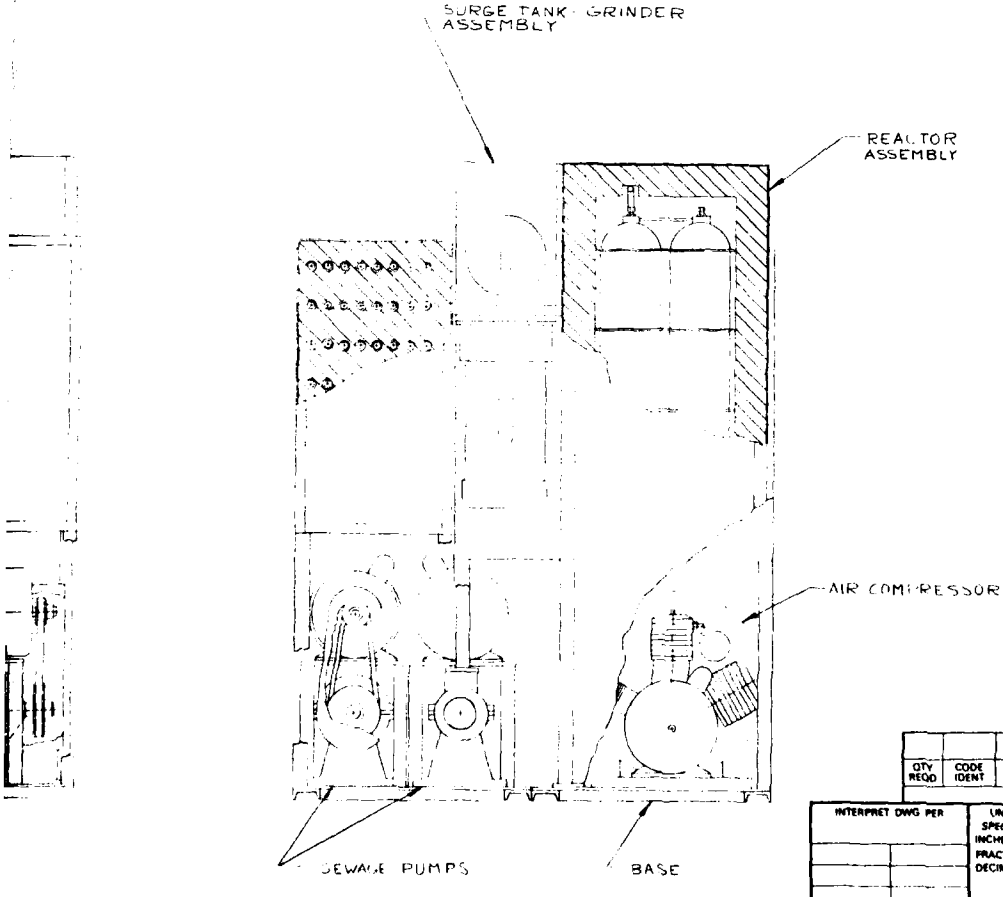


Fig. 47 Shipboard System Layout Drawing

The heat exchanger is a completely integral unit. It is supported by the hold tank at one end, and on the other end by braces which attach directly to the base plate. The reactor controls, and manual override switches are located in a panel above the hold tank. The burner pilot controls are located on the side of the reactor assembly, the gas vent and liquid level controls are located next to the reactor, and the pump ΔP controls are located near the pumps.

The system design utilizes commercial practice of low cost, medium weight, rugged construction. It employs prefabricated metal shapes and simple frame construction where possible; and stresses simple, maintainable components.

Hastelloy C-276 was chosen as the material of construction for all elevated temperature system components exposed to the influent, based on the use of fresh water for the flush system. Other high temperature parts are stainless steel. Metal parts not exposed to high temperature are constructed of carbon steel. If salt water were used, a titanium reactor, heat exchanger, and plumbing would be required. This would not affect the system weight, but would result in higher costs and possible increased delivery schedule. Reactor temperature control would also become critical in a fuel fired reactor, due to the rapidly decreasing strength of titanium at temperatures above 600°F.

The overall system weighs approximately 1470 lbs, dry and 2000 lbs with a maximum load of sewage. A detailed weight breakdown is presented in Table 23. Major components such as the reactor assembly, heat exchanger, sewage pumps, and air compressor contribute the majority of the weight. Structure and related items have been kept to a minimum through efficient design, and therefore do not contribute significantly to the overall weight. It should be noted that a lower weight could be achieved in some areas, with no sacrifice in strength, by utilizing more sophisticated construction techniques and alloys. This however, would add appreciably to the cost and complexity.

Table 23 System Weight Summary

Reactor Assembly	447
Heat Exchanger	242
Surge Tank/Grinder	55
Sewage Pump Assembly	269
Hold Tank	111
Level Control System	50
Air Compressor	120
Base Assembly	134
Plumbing	14
Controls	28
Total Dry Weight	1470 Lbs.
Total Wet Weight (Hold Tank Full)	2020 Lbs.

The system power summary is presented in Table 24. It shows electrical power and fuel heating requirements for both fuel fired and electrically heated systems. As can be seen, the pumps and air compressor consume the bulk of the non-heater power. This is due to the necessity of raising the process influents to the 1200 psi system pressure. The pumps consume large amounts of power because they have inherently high fluid slippage and friction losses. They, however, successfully pump sewage containing solids and abrasive particles at a controlled flow rate.

The steady state heater power is necessary to make up for losses incurred in the regenerative heat exchanger, and through the insulation. These losses have been minimized by utilizing the largest heat exchanger and thickest insulation feasible, consistent with system volume and weight goals.

When the system is started cold, extra power is required to bring it up to operating temperature within a reasonable length of time. With the heatup power shown, either electrically heated or fuel fired systems will reach normal temperature in about 6 hours.

Component Descriptions

Reactor Assembly - The reactor assembly, as shown in Figure 48, consists of the reactor tubes with catalyst, the heating system, and the structure and insulation. The overall package is 22.7" x 15.5" x 47.0" and weighs approximately 447 pounds.

The reactor tubes have an outside diameter of 5.5 inches and a straight section length of 36 inches. They are constructed of Hastelloy C-276, are 0.237 inches thick, and have hemispherical end caps with one inch pipe couplings which provide inlet and outlet fluid connections. The platinum-ruthenium catalyst is disposited on 1/4" dia x 1/4" long alumina cylinders that are inserted into the reactor through the pipe fittings in the tube end caps. Four tubes are provided to obtain an internal volume of 15 gallons, producing a 30 minute contact time. The tubes are grouped closely together, held by interconnecting structure and are interconnected with 1/2 O.D. tubing. Liquid, and air flow in the tubes is upward.

Table 24 System Power Supply

Component	Power Required: Fuel Fired System	Power Required: Electrically Heated System
Air Compressor	900 watts	900 watts
Pumps (2)	2000 watts	2000 watts
Grinder	400 watts	400 watts
Fan	55 watts	55 watts
Controls	100 watts	100 watts
Reactor	None	3400 watts
Total Steady State Electrical Power	3455 watts	6855 watts
Startup Electrical Power	None	2000 watts
Total Startup Electrical Power	3455 watts	8855 watts
Fuel Required (Light Oil)		
Steady State	0.8 lb/hr	None
Startup	1.18 lb/hr	None

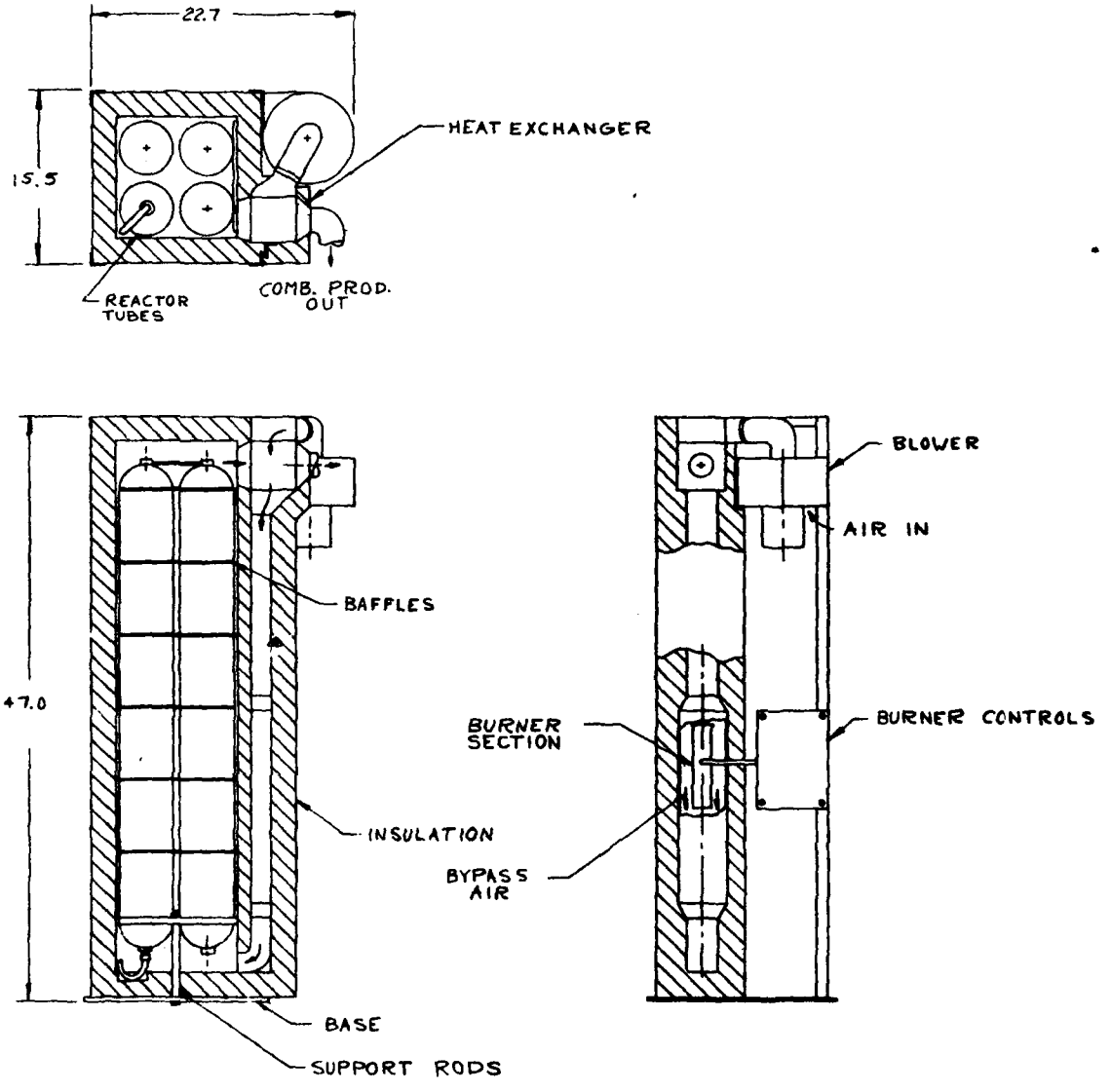


Fig. 48 Fired Reactor Assembly

The fuel fired reactor heating system consists of a burner section, fan, regenerative heat exchanger, ducting and baffles, and burner controls. A circular burner section is provided in an oval air circulation duct. In order to assure a reasonable burner outlet air temperature, excess air is provided by the fan assembly. The Rotron Model MLPM type 6502-K4 multistage blower produces in excess of 10 inches of water at 60 lb per minute flow, is approximately 7 3/4 inches in diameter and 7 1/2 inches long, and operates on 110 volt, 60 cycle power. It is extremely long wearing, quiet, and is very compact and light compared to conventional multistage turbines.

A portion of the air from the fan is drawn through the burner section and combustion takes place at a high temperature, ensuring complete combustion. The burner is controlled by both normal and emergency controls and burns 0.8 lb/hr of light oil during steady state operation, and 1.18 lb/hr during startup. The rest of the air passes around the burner and mixes with the hot burner outlet air. The heated air stream, which reaches a temperature of approximately 1400°F during normal operation, passes over the reactor. The reactor is baffled, to increase heat transfer, and an overall heat transfer coefficient of 100 BTU/hr°F results from the effects of convection, gas radiation due to flue gas products, and reradiation from the baffles and insulation. In the reactor assembly the air temperature decreases to 570°F while transferring 10150 BTU/hr to the process fluid and 2330 BTU/hr to the surroundings through the insulation. After leaving the reactor assembly, the air passes into the regenerative heat exchanger, which is a stainless steel plate-fin counter flow design with a core size of 6 1/4" x 3.7" x 3.7". It has an effectiveness of 76%, resulting in 446°F air returning to the burner and 175°F air being exhausted. The total pressure drop for both sides is 2 inches of water.

For the electrically heated reactor the fuel burner, regenerative heat exchanger, and blower are replaced by strip heaters which are clamped directly to the reactor tubes. Two 3/4 KW electrical heaters are provided on each tube, which results in a low watt density and uniform heating.

The insulation and structure supports the reactor assembly, and minimizes heat loss to the surroundings. The four reactor tubes are welded to a plate at the bottom end. Support rods are then used to tie this structure to the base. The baffles slip over the reactor and are secured by rods and spacers. Slabs of Johns-Manville Thermo-12 insulation of 2 1/2 nominal thickness surround the unit. The inside is surfaced with furnace cement to prevent erosion by flue gas and the outside is covered with .020 carbon steel. The sheet steel and insulation are held in place by 1"x1"x1/8" angles located at the corners of the unit.

Regenerative Heat Exchanger - The regenerative heat exchanger is used to conserve power by transferring heat from the effluent fluids to the influent sewage and air. As shown in Figure 49 it consists of 200 ft. of 1/2 inch O.D., .028 wall tube inside of a 3/4 inch O.D. .035 wall tube. Both tubes are Hastelloy C-276 and are arranged in 56 interconnected rows. 1 1/2 inch radius bends, the minimum advisable for this material, connect the rows. The tubes are manufactured in flat panels of 7 tubes each, and 8 of these panels are joined together together at 1 1/4 inch increments in the final assembly. Johns-mansville Thermo-12 slab insulation is used between the tubes for support. A nominal insulation thickness of two inches is used on the hot side, top, bottom, and ends and 1 inch is used on the cold side. The insulation is covered with .020" thick sheet steel, and it is supported by 1"x1"x1/8" angle located at the corners of the unit.

During operation, heat transfer results from a combination of conduction and convection. It should be noted that the heat content of the liquid/gas mixture is not linear with temperature, because of the heat of vaporization of the liquid into the gas phase, as well as the heat content of the liquid and gas. The regenerative heat exchanger and heater designs must be considered together because the combination must provide the energy to bring the input fluids to reactor temperature. Figure 50 presents a curve of heater power requirements as a function of heat exchanger size. The larger the heat exchanger the less heater power is required. The curve does not account for losses through the

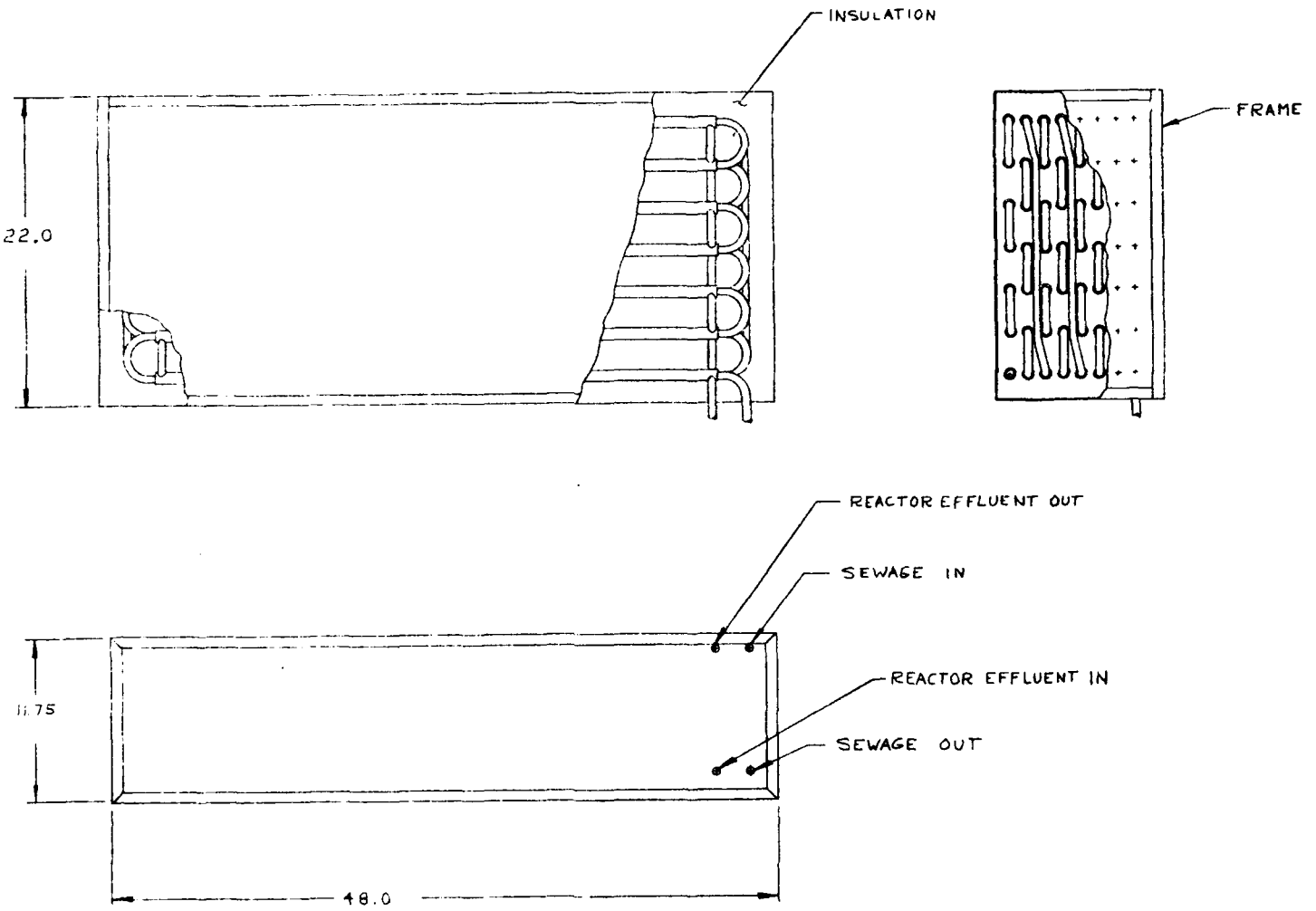


Fig. 49 Heat Exchanger Assembly

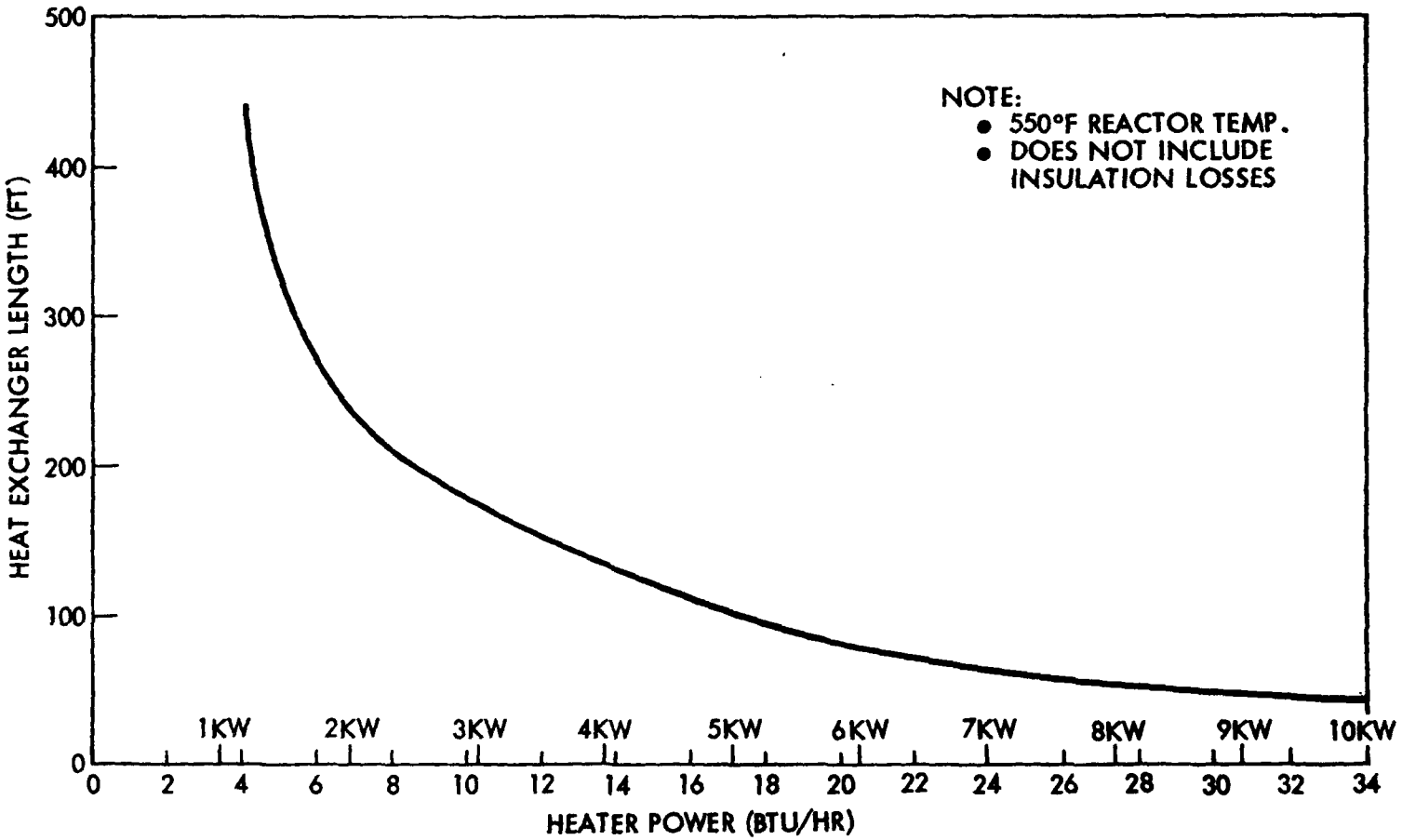


Fig. 50 Heat Exchanger Length vs Heater Power

insulation. A heat exchanger tube length of 200 ft was chosen, as a compromise between heat exchanger size and heater power requirements, resulting in a no-loss heater power requirement of 8700 BTU/hr. The overall heat transfer coefficient is 200 BTU/hr ft^2 ^oF, and the heat transfer between fluid passes is 130,200 BTU/hr. The losses to ambient through the insulation are 1460 BTU/hr.

Sewage Pumps - The high pressure sewage pumps are used both to meter the inlet sewage flow, and to raise the flow to the nominal 1200 psi system pressure.

The pumping system utilizes two Robbins and Meyers "Moyno" pumps operated in series. Two pumps are used to limit pressure rise per pump to less than 1000 psi in order to increase pump life and to limit pump length to 4 feet. The "Moyno" pump provides positive displacement flow combined with the ability to pump liquids containing high percentage of solids and abrasives.

The pumps utilized in the design are a slight modification of an existing standard design, with the only appreciable changes being the use of an increased number of stages and revised thrust bearing and shaft seal designs in the second stage pump inlet.

As shown in Figure 51, the pumps consist of a rigid rotor and an elastomeric stator assembly. Ball bearings support the shaft, and a gland type packing is used to seal the shaft against leakage. Fifteen stages are used in each pump with a 50 psi design head per stage, giving the capability of 750 psi ΔP per pump. The stator contour length is 28 inches and the overall dimensions of the pump are 48 inches long x 7 inches wide x 10 inches high.

The metal parts exposed to the fluid are 316 stainless steel, and the rotor has heavy layers of hard chrome plating to increase resistance to abrasion. Each pump is fitted with a 1 horsepower capacitor start, enclosed, fan cooled motor. Drive is by belts and pulleys, and the motors are supported by stands constructed of 1"x1"x1/8" steel angle and 1/16 inch plate. The pumps operate at a nominal 900 RPM.

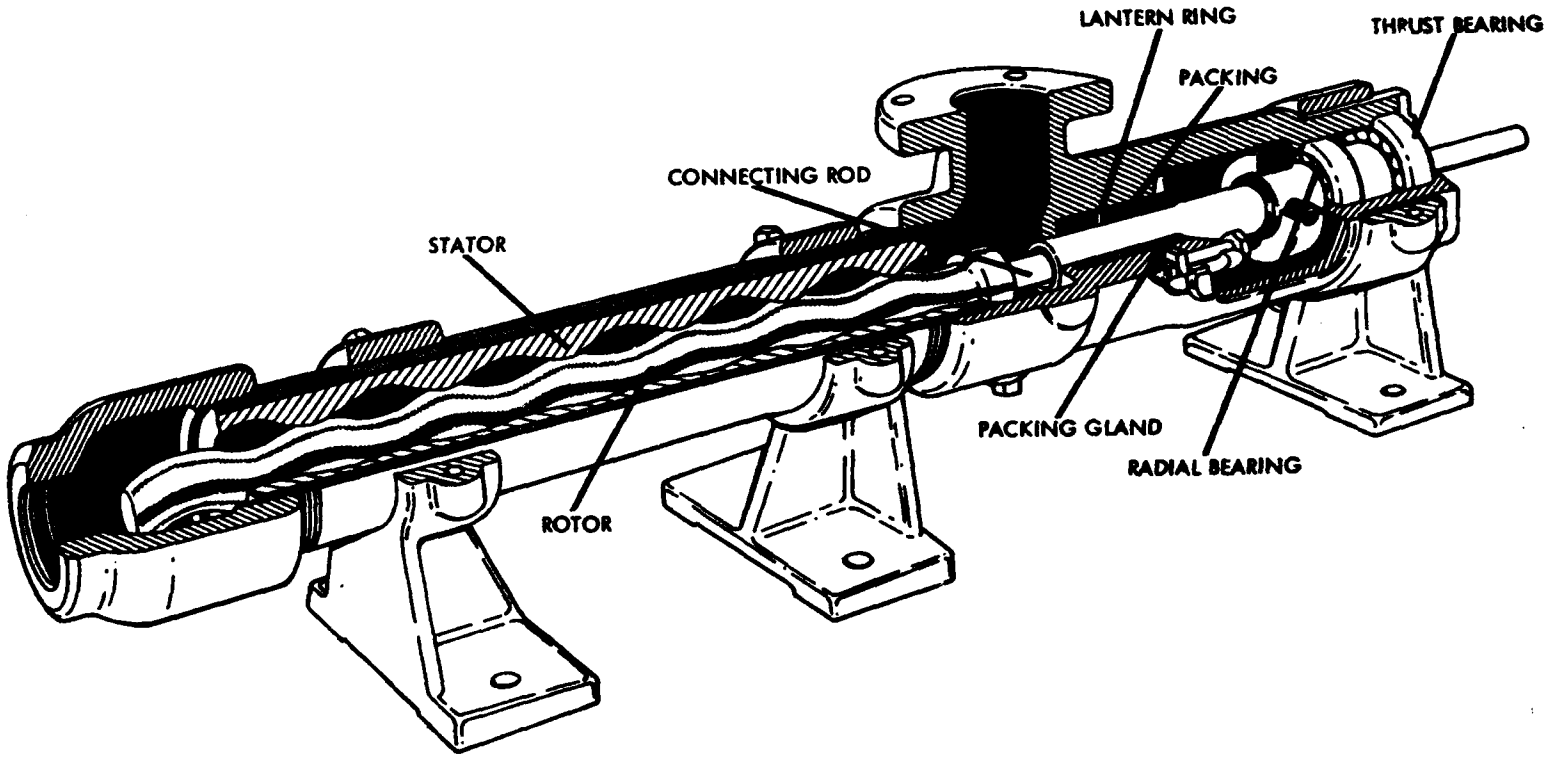


Fig. 51 Sewage Pump

Hold Tank Assembly - Due to the specified fluctuations in the sewage feed rate, a hold tank is necessary to accumulate excess sewage thereby allowing the system to operate at a constant rate below peak demands. The tank incorporates high and low level switch assemblies to control system operation.

The tank, as shown in Figure 52, has a 60 gallon capacity and is fabricated from 1/16" steel plate. The bottom is reinforced with 1"x1"x1/8" angle. The tank provides connections for the pump grinder assembly, which mounts directly to it. Flange mounted, float operated, level switches are provided at the top and bottom of the tank. Protective screen covers prevent blockage of the float assemblies. A one inch threaded outlet is provided for the sewage pump suction line.

Surge Tank-Grinder Assembly - As shown in Figure 53, the surge tank-grinder assembly consists of a surge tank, a grinding stage and a motor. It mounts directly to the hold tank assembly. The surge tank-grinder assembly reduces inlet sewage solids to a size acceptable to the sewage pumps. It incorporates a screen assembly which collects metal and other particles which could damage the grinder or other system parts. The grinder is located in the surge tank to reduce power consumption because liquid is not continually present in the grinder. Overall dimensions are 12.5"x7.5"x26". The 4.5 gallon surge tank is constructed of 1/6 inch steel internally coated to prevent corrosion. It contains a perforated screen and has a clean out port through which foreign objects can be removed. A 4 inch sewage inlet, and 3 inch overflow to the hold tank are provided.

The grinder is an adaptation of a unit designed for spacecraft application. It uses a commercially available cutter head which has both cutting and grinding stages. It utilizes carbide and tool steel cutting edges. A NEMA Frame 56C, 1/2 HP, flange mounted, enclosed fan cooled motor mounted to the bottom of the unit drives the grinder. The grinder discharges ground sewage directly to the hold tank.

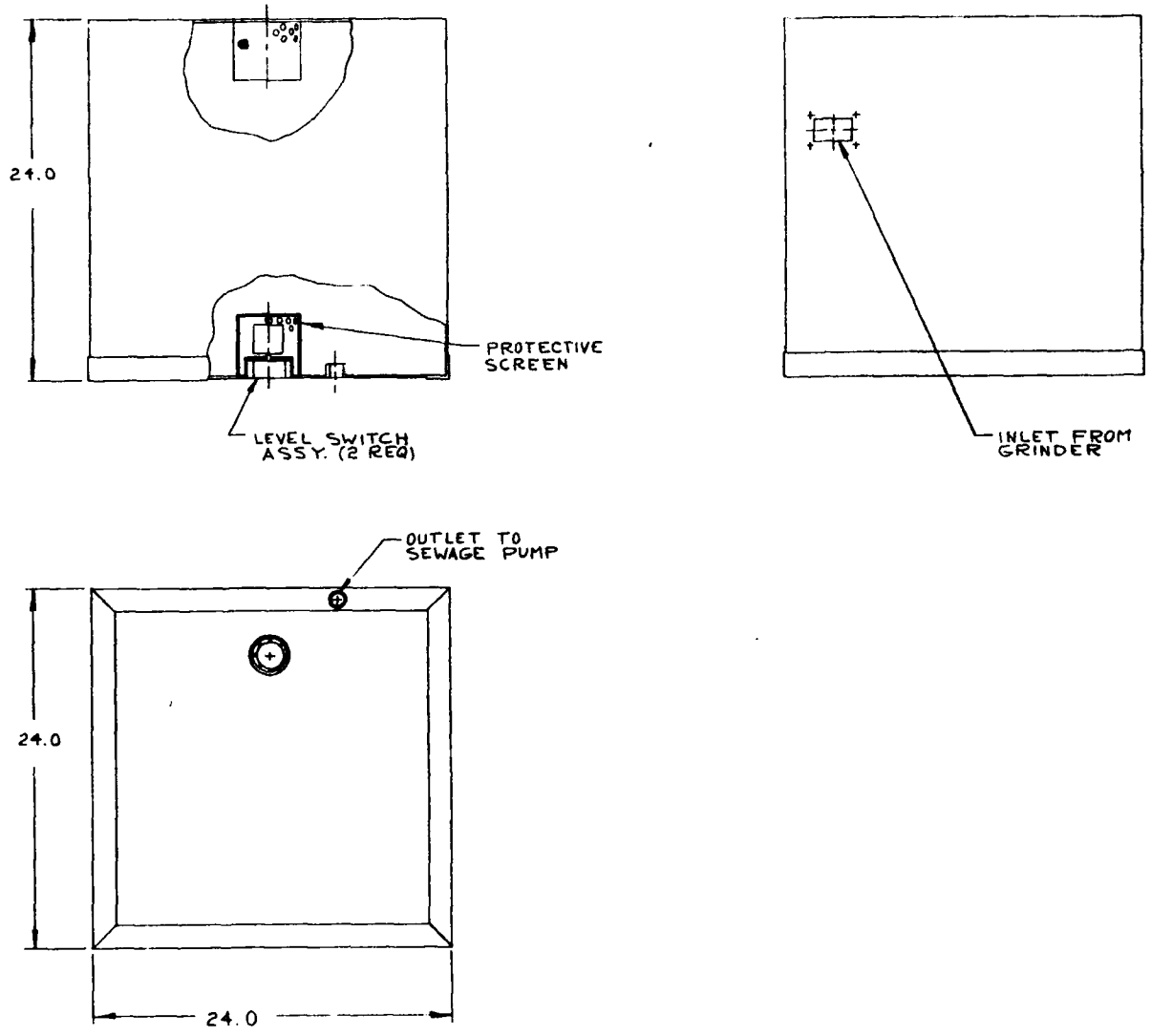


Fig. 52 Hold Tank Assembly

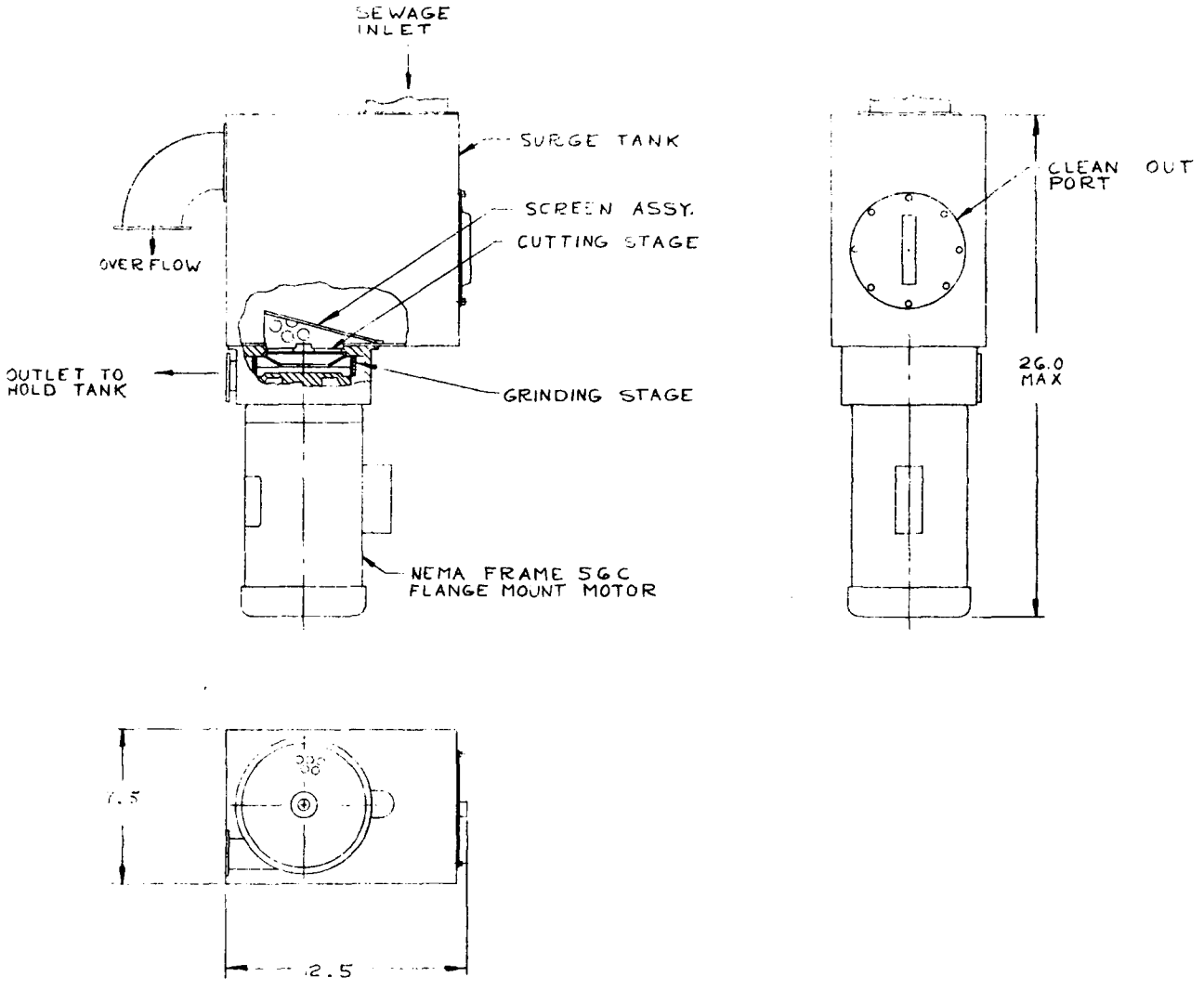


Fig. 53 Surge Tank-Grinder Assembly

Influent sewage from the ship passes directly into the surge tank. Metallic or other solid particles are trapped by the screen assembly. If the capacity of the hold tank is momentarily exceeded, the liquid portion of the sewage flows through the overflow, directly into the hold tank. Solids and the remaining liquid pass through the continuously running grinder, and into the hold tank.

Air Compressor - The air compressor supplies 1.6 SCFM of air at 1200 psi, to react with the influent sewage.

The compressor, shown in Figure 54, is a Purus 3 cylinder, 3 stage unit designed for 1.8 SCFM output at 3200 psi. The unit is somewhat larger than required but is the smallest commercially available. Although a special design might be slightly more efficient, development could prove costly and time consuming. Derating the unit to 1200 psi will increase operating life. A one horsepower electric motor drives the compressor.

The unit is air cooled by a blower which is located in the center of the compressor near the front. Intercooling and safety valves are provided after all three stages. The entire rotating and reciprocating assembly is equipped with needle or ball-bearings, and lubrication is by a splash spray system. The total package is 25 x 14 x 17 inches and weighs 120 pounds.

Controls - Normal and emergency controls are provided to ensure the safe, automatic operation of the system. As summarized in Table 25, these include controls for system feed reactor temperature, system pressure and liquid level and safety.

Also, a complete system electrical schematic is presented in Figure 55.

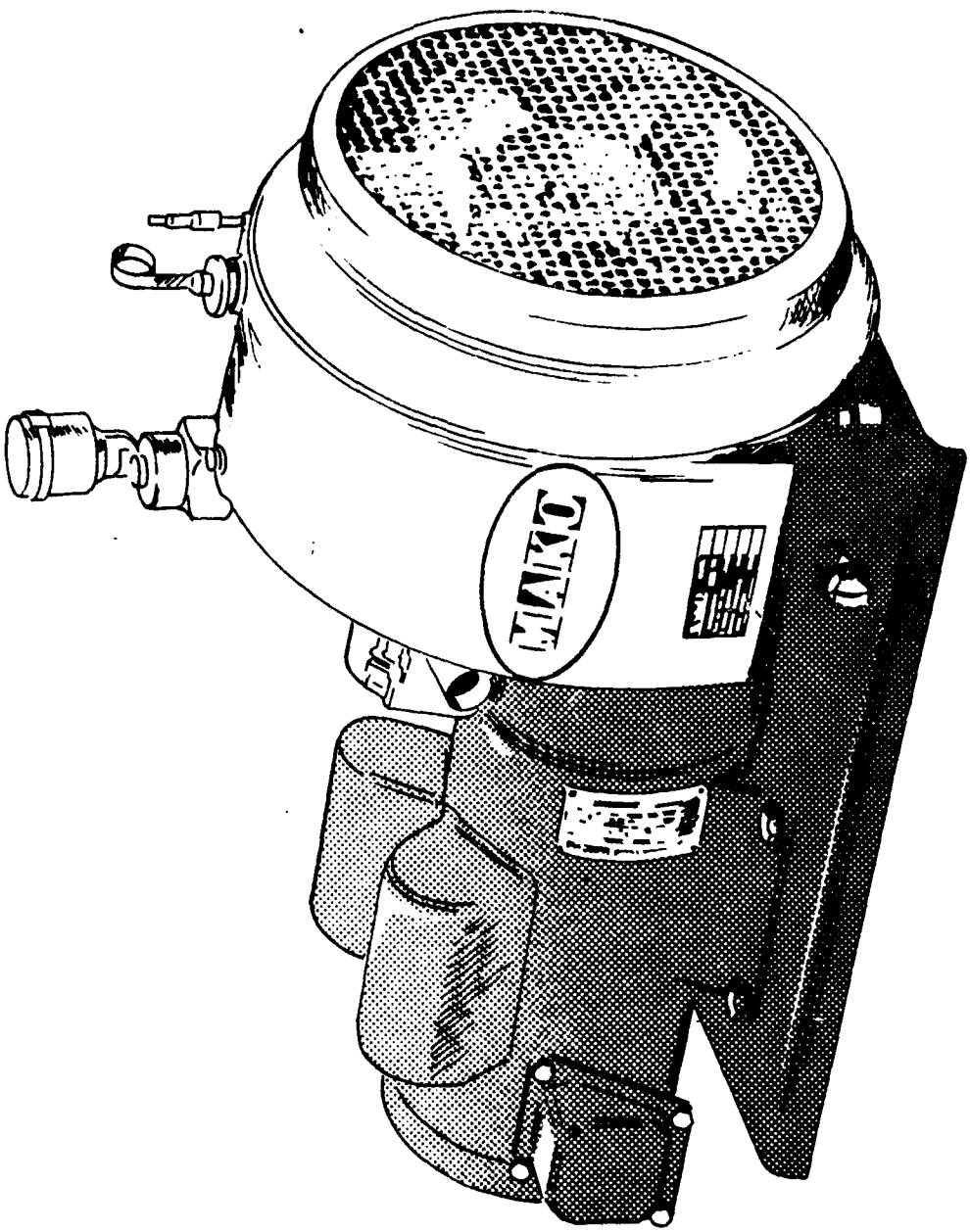


Fig. 54 Air Compressor

Table 25 System Controls Summary

TYPE OF CONTROL	FUNCTION	
	NORMAL OPERATION	EMERGENCY OPERATION
Hold Tank Low Level Switch	Senses liquid level in hold tank - starts pumps, compressor, opens motorized valves when level is high enough. Stops pump and compressor and shuts valves when level drops.	-
Hold Tank High Level Switch	-	Initiates warning signal if high hold tank liquid level is sensed.
Burner Pilot Control (Fuel System Only)	Senses pilot flame and allows pilot valve and main burner valve to open. Manual override provided for startup.	If pilot malfunctions, shuts pilot valve and prevents main burner valve from opening.
Reactor Fluid Outlet Temperature Control	Senses effluent temperature at outlet of reactor; keeps it at desired temperature by modulating burner main fuel valve to control flame level (controls power to heater on electrically heated version).	If preset high temperature limit is exceeded, shuts off burner (or heaters) completely; also shuts off rest of system and initiates warning.
Reactor Air Temperature	Works in series with reactor fluid outlet temp. control; senses air temperature entering reactor and modulates main fuel valve to control flame level and keep temperature below preset level.	If preset high temperature limit is exceeded, shuts off burner completely; also shuts off rest of system and initiates warning.

Table 25 (continued)

TYPE OF CONTROL	FUNCTION	
	NORMAL OPERATION	EMERGENCY OPERATION
Gas Vent Control	Senses system total pressure and controls it by venting gas if preset pressure level is exceeded.	-
Liquid Level Control	Senses liquid level in column with ▲P sensor; opens liquid vent if liquid level is high; closes valve when level drops.	-
System High Pressure Switch	-	Senses total system pressure; shuts down system and initiates warning if preset pressure level is exceeded.
System Low Pressure Switch	-	Senses total system pressures shuts down system and initiates warning if pressure drops below preset level.
Sewage Pump ▲P Switch	-	Senses ▲P across sewage pumps; initiates warning if preset maximum ▲P is exceeded.
Thermal Overloads	-	Senses component motor temp; shuts off component if preset limit is exceeded.
Rotation Sensors on Pumps and Air Compressors	-	Senses rotation; shuts off system air and liquid feed if rotation ceases on any component.

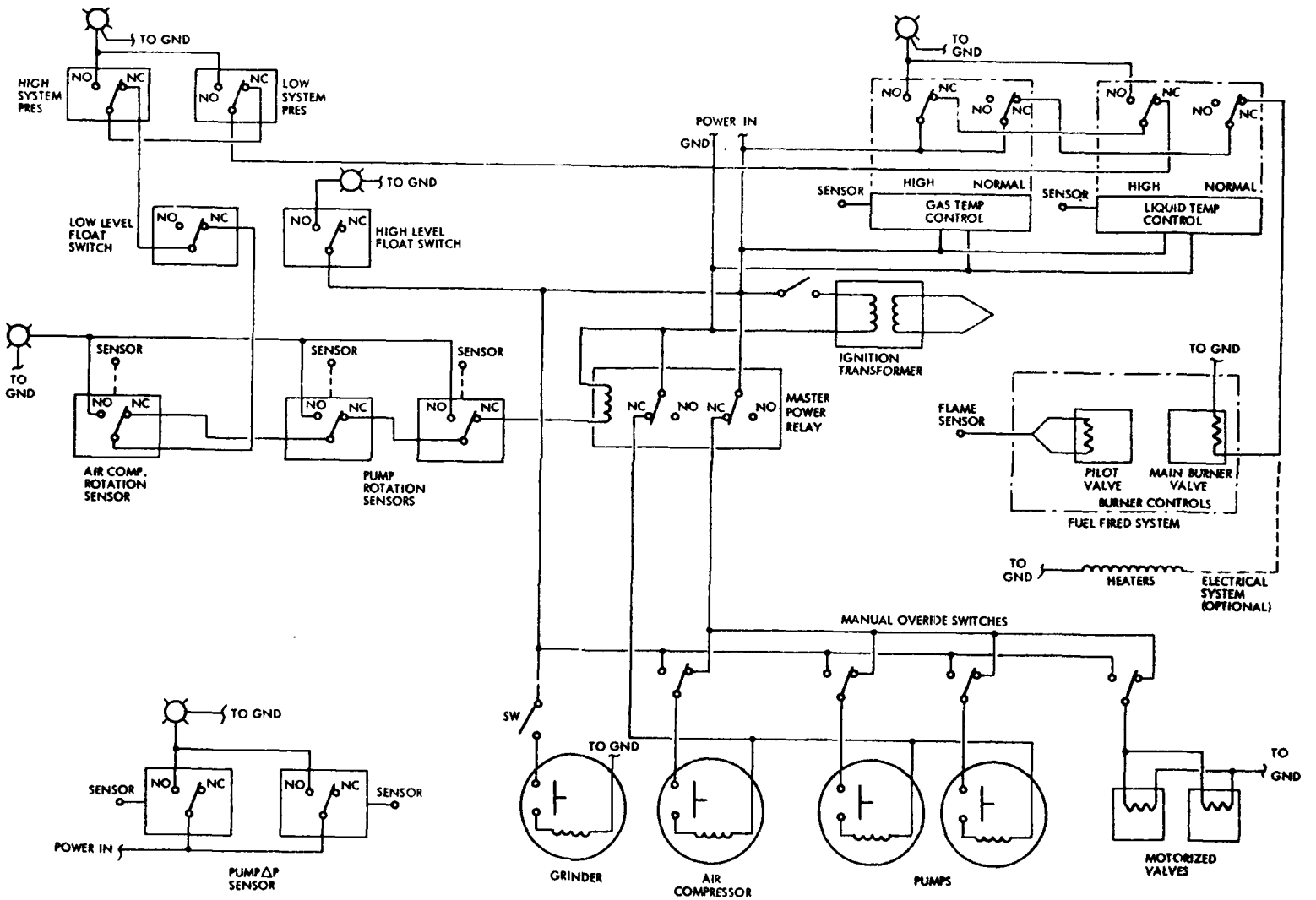


Fig. 55 System Electrical Schematic

System feed is controlled by the low level float switch in the hold tank. When the sewage level in the tank is sufficient to allow system operation, the switch actuates the sewage pump and air compressor, and opens the motorized valves. The valves are ball type with electrical actuators, and are spring loaded to close if system electrical power fails. When the hold tank level drops, the air compressor and pumps are stopped, and the valves are closed. If, for some reason, the hold tank becomes full, the high level float switch initiates an overflow warning signal.

The fuel burner control is independent of other system operations. On the fuel fired unit, two separate controls are used in series. The first control senses the reactor liquid outlet temperature, and the second senses the reactor inlet air temperature. During normal operation, the first control modulates the burner main fuel valve to hold the outlet temperature at 550°F. When flow stops, or at other times where very little heat is needed to maintain the 550°F outlet temperature, the other controller limits the inlet air from the burner to about 1500°F by overriding the first control and modulating the main fuel valve. Each controller also has a high limit set point, and if either high limit set point is exceeded the entire system is shutdown and a warning is initiated. On the electrically heated version only the reactor outlet fluid control is used, and it modulates the electrical heater power.

Thermoelectric Model 100 indicating, proportioning, controllers have been selected for both reactor control functions. These have solid-state electronics with thermocouple sensing and incorporate both a normal set point circuit with proportioning control and a high limit set point circuit with digital control. The normal set point is fully adjustable over the entire operating range, and the high limit circuit is actuated if the normal set point is exceeded by a percentage adjustable from 1 to 10%.

System operating pressure and the venting of effluent are controlled by the gas vent control and liquid level control. A liquid column is used as a phase separator to separate the gas portion of the effluent from the liquid portion. A gas regulator then controls the system pressure by venting gas as required. Liquid level in the column is sensed by a ▲P sensor, and the liquid control valve is actuated when the level exceeds a preset value. When the level drops, the control valve is closed.

The gas vent control is a standard dome loaded regulator. It has a stainless steel body and nylon seat. This type of control is used successfully on the breadboard system. The liquid level sensor is a diaphram type ▲P sensor which provides a signal to the liquid level control valve.

Controls utilized for safety purposes include the system high and low pressure switches, the sewage pump ▲P sensor, electrical overloads, and rotation sensors on the pumps and air compressors.

The system high and low pressure switches sense the system operating pressure and shutdown the system if the pressure deviates from the desired control band. A warning is also initiated. A Barksdale Model B2T pressure switch is used for both control functions. It is of the bourdon tube type, and has two separately adjusted switches. The switch is located near the level control system.

The sewage ▲P switch senses the ▲P across the first stage sewage pump. If either pump wears appreciably, the load will shift from one pump to the other. If 750 psi ▲P is exceeded, a warning signal is initiated. The switch is a Barksdale Model B2T, and is located near the pumps.

Thermal overloads are built into the motors on the pumps, compressor, and grinder. They sense motor temperature and cut power to the motors if an overheat condition exists. The pump and compressor rotation sensors are magnetic proximity sensors. They sense the rotation of a ferrous protrusion on a portion of the rotating equipment. If motion stops, a solid state circuit opens a relay, thus shutting down the system and initiating a warning.

System Maintenance

The system has been designed to require a minimum of attention. Well proven, long life components have been selected where possible, and others have been designed for maximum accessibility and life. Items which must be replaced regularly are easily accessible without disassembly of the system. In addition, the modularization of components allows easy removal of subassemblies for servicing. Planned maintenance items and maintenance frequency are shown in Figure 15.

Tasks have been broken down into 3 months and 1 year intervals, on the basis of the best information presently available. Actual maintenance schedules on unproven items would be established during development.

Table 26 System Periodic Maintenance Summary

INTERVAL	ITEMS
3 mo.	- clean out metallic objects from surge tank through cleanout port - check and adjust pump belts; replace if necessary - check burner controls - check oil level in air compressor and clean air filter - clean air filter at fan inlet
1 yr.	- disassemble reactor, clean and inspect baffles and burner, replace burner nozzles and ignition electrodes if necessary; inspect and replace catalyst if required. - disassemble sewage pumps; grease bearings, inspect rotor and seals and replace if required - clean out hold tank and surge tank, inspect float level switches - inspect packing in motorized valves and fluid level controls and replace if necessary.

Performance Parameters for Other System Sizes

Weight, volume and power have been determined for systems with hydraulic loadings from 700 to 3500 gallons per day. These correspond roughly to systems sized for from 20 to 100 men. The influent biological oxygen demand ranges between 150 mg/liter and 1800 mg/liter, with an average of 500 mg/liter. The effluent BOD is a maximum of 50 mg/l.

The 20 man system parameters are those of the proposed preliminary design system. The larger system parameters are based on design of 50 and 100 man systems using commercially available components. Parameters for systems between these sizes were determined by extrapolation between the three points. Although all parameters are shown as smooth curves it should be noted that some parameters, especially weight and volume, have discrete steps due to the availability of certain key components in only certain size ranges.

As shown in Figure 56, the system weight increases fairly linearly with size. Larger systems, however, are more efficient in terms of pounds of system weight per gallon processed. This figure also shows that the ratio of system volume to capacity decreases for larger systems. Both effects are due to the more efficient structural design possible with a larger system, and the number of fixed components such as the temperature and level controls.

As shown in Figure 57 the electrical power required for both fuel fired and electrical systems increases fairly linearly with system size. The difference in slope is due to the nature of the electrical loads. The electrical loads in a fuel fired system are comprised of motors and controls. Control power remains almost fixed with system size, and larger motors and motor driven components are more efficient, thus the power to capacity ratio decreases fairly rapidly.

In the electrically heated system, a large percentage of the load is heat supplied to the process fluid. Since this has almost a constant power to capacity ratio, the overall ratio decreases less rapidly.

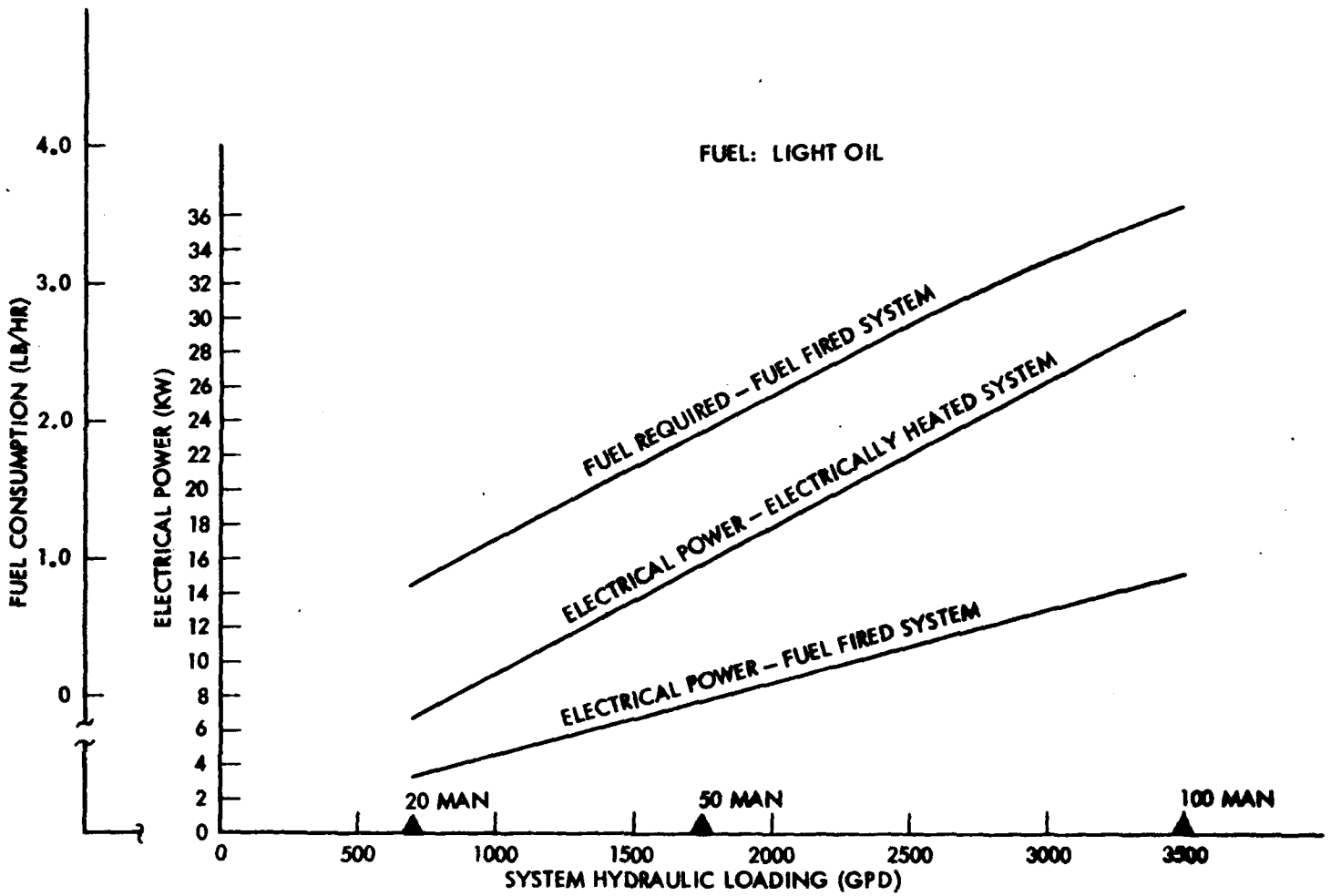


Fig. 56 Electrical Power & Fuel Consumption vs Shipboard System Capacity

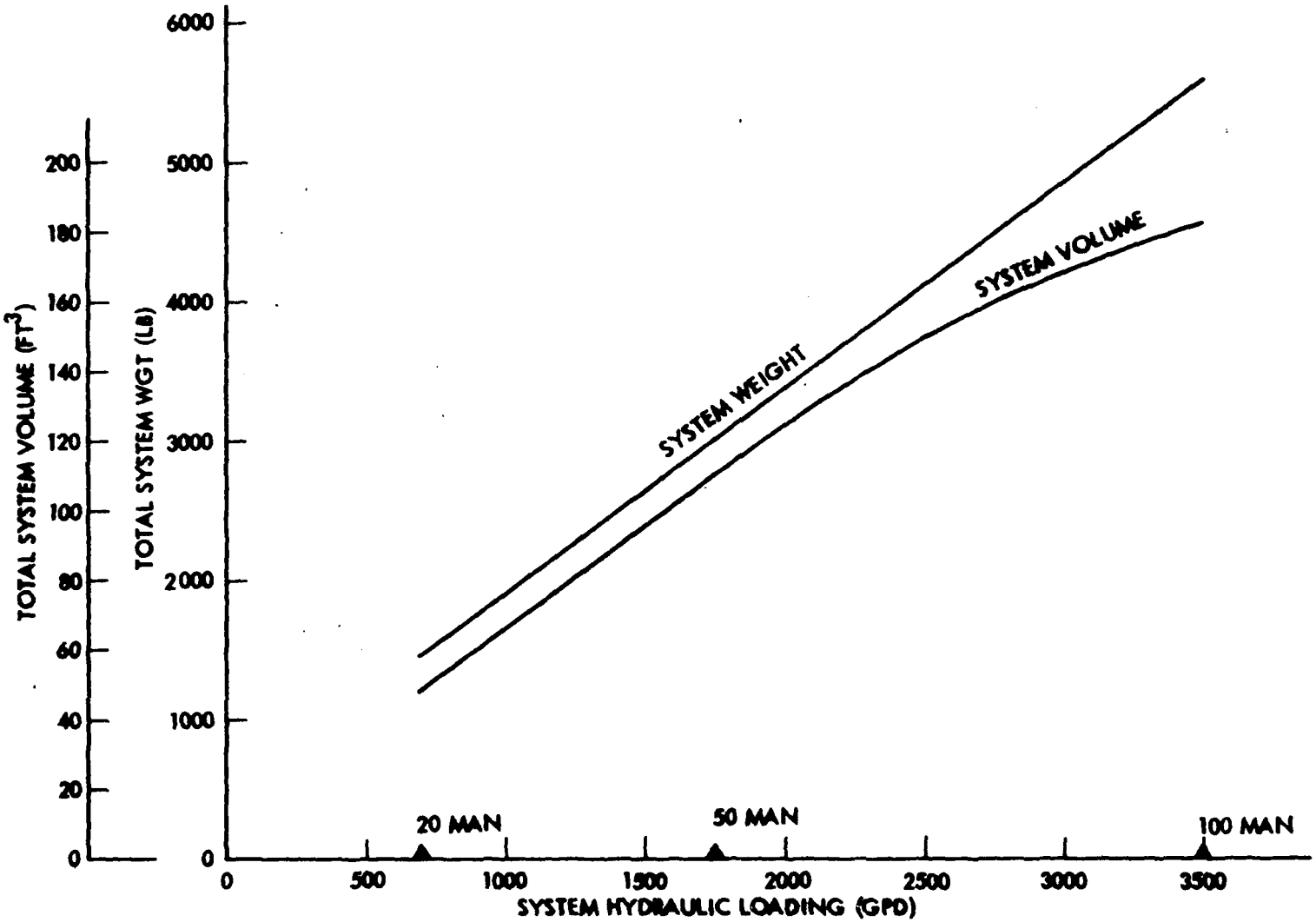


Fig. 57 System Weight and Volume vs Shipboard System Capacity

As shown in Figure 57, the ratio of fuel required to system capacity decreases only slightly with an increase in system size, because the fuel is used for heating the process fluid. The slight decrease is due to reduction of heat losses in the larger systems.

CONCLUSIONS AND RECOMMENDATIONS

The following conclusions and recommendations have been compiled based on the conduct of the test program and preparation of the preliminary design.

- The wet oxidation principal can be feasibly applied to solving the sewage treatment problem on board United States Coast Guard vessels resulting in a system that requires no more than a 3'x4'x4' envelope, 3.4 kilowatts of power and 0.8 pounds per hour of fuel oil and weighs no more than 1500 pounds. It is recommended that a dock-side demonstration system be built and tested to prove the feasibility of the wet oxidation application.

- Achievement of 90% or greater reduction in BOD at reasonable temperatures, pressures and contact times is not possible without the use of catalysts or chemicals. A packed tower reactor operating at 550°F, 1200 psig, with Pt/Ru catalyst and 30 minute contact time is recommended.

- The packed tower reactor principle without the need for internal agitation or hot recirculation pump was demonstrated to be as effective as a stirred reactor.

- Corrosion tests showed commercially pure titanium and alloyed titanium (0.2% Pd, 1% and 2% Ni, and 6-2-1-1) to be suitable reactor materials for the extreme environment of sea water and acid (pH of 2) service. The less severe fresh water sewage without addition of acid

for pretreatment would probably permit the use of Hastelloy C-276 which offers advantages of higher strength, higher temperature operation, and greater availability. It is recommended that additional corrosion tests be run using fresh water sewage with pH between 5 and 8.

- Sea water in the influent sewage does not adversely affect system performance, but does markly affect materials selection. It is recommended that fresh water flush be used, if at all practical from a ships operations standpoint.

- Chemical oxygen sources are very effective in wet oxidation as either a complete source of oxygen or as a polishing agent to air oxidation. Additional work should be done to evolve a chemical injection and distribution system to take full advantage of the potential of chemical oxidation.

- ph adjustment by acid addition to values below 2 markly increases reduction in BOD particularly at low temperatures. The need for carrying an expendable acid source, the added reactor corrosion problems, and the need for neutralizing the effluent from the system result in the recommendation not to use acid pH adjustment in design of the shipboard system.

- Operation of the breadboard system during the test program showed that the system is easy to automate and maintain in operation.

- Leakage of high pressure reactor seals and fittings proved to present the most frequently encountered system maintenance problem. Final system design should incorporate welded construction to eliminate reactor sealing problems. Leakage of standard tube fittings was not a problem during the test program.

- TOC has proven to be a good indicator of BOD and COD for the input wastes used during the test program.

REFERENCES

1. Webb Associates, "NASA Life Sciences Data Book". First Edition, June 1962

2. Helm, J. W., "Rapid Measurement of Organic Pollution by Total Organic Carbon and Comparison with Other Techniques", United States Dept. of Interior, Geological Survey, Open File Report, 1970.

3. Ostendorf, R.B., and Byrd, J.F. "Modern Monitoring of a Treated Industrial Effluent", Journal of Water Pollution Control Federation, V. 41, 1, 1969.

4. Stenger, V.A., and Van Hall, C.E., "Analysis of Municipal and Chemical Waste Waters by an Instrumental Method for COD Determination", Journal of Water Pollution Control Federation, V. 41, 10, 1968.

5. Blackmore, R.H., and Voskel, D., "Rapid Determination of Total Organic Carbon in Sewage", Water and Sewage Works, 1967

6. Robbins, J.W.P., Kriz, G.T. and Howells, D.H., "Total Organic Carbon Determinations in Swine Waste Effluents". Paper presented at American Society of Agricultural Engineers, Chicago, Ill., Dec. 9-12, 1969.

7. Ford, D.L., "Application of the Total Carbon Analyzer for Industrial Waste Water Evaluation", 23rd Industrial Waste Conference, Purdue University, 1968.

8. Waste Water Engineering, Metcalf and Eddy, Inc., McGraw-Hill Book Company, New York, New York 1072

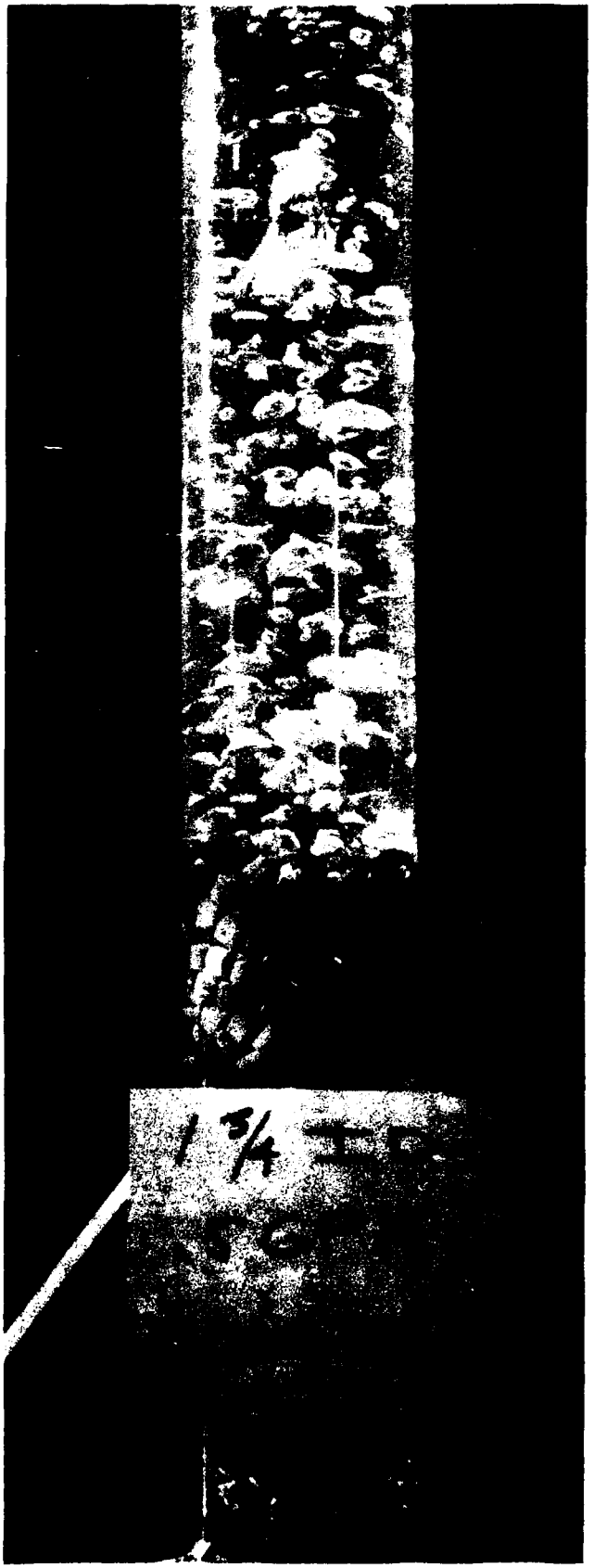
9. Perry, John H., "Miscellaneous Over-all Coefficients of Heat Transfer", Chemical Engineers Handbook, Third Edition, McGraw-Hill Book Company, New York, Toronto and London, 1950.

10. Wise and Jahn, "Wood Hydrolysis", Wood Chemistry, ACS Monograph, Reinhold, 1952.

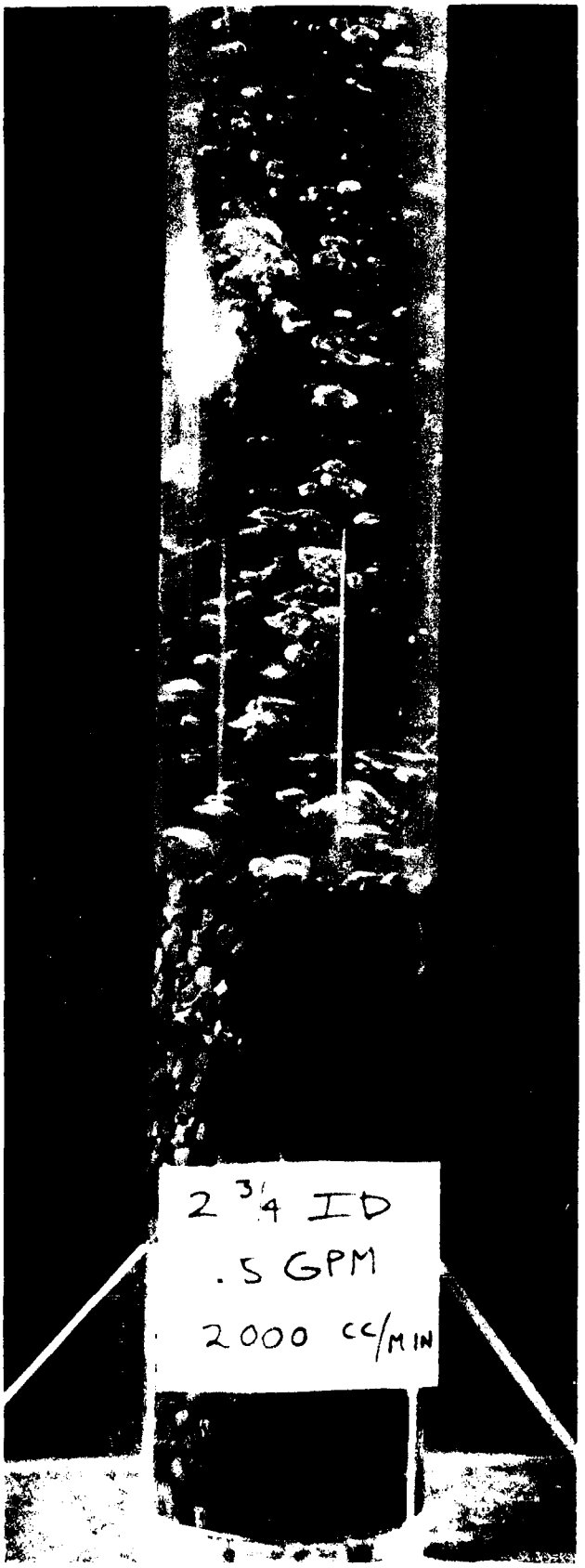
11. Timoshenko, "Strength of Materials - Part II," D. Van Nostrand Co., Princeton, New Jersey, 1930.

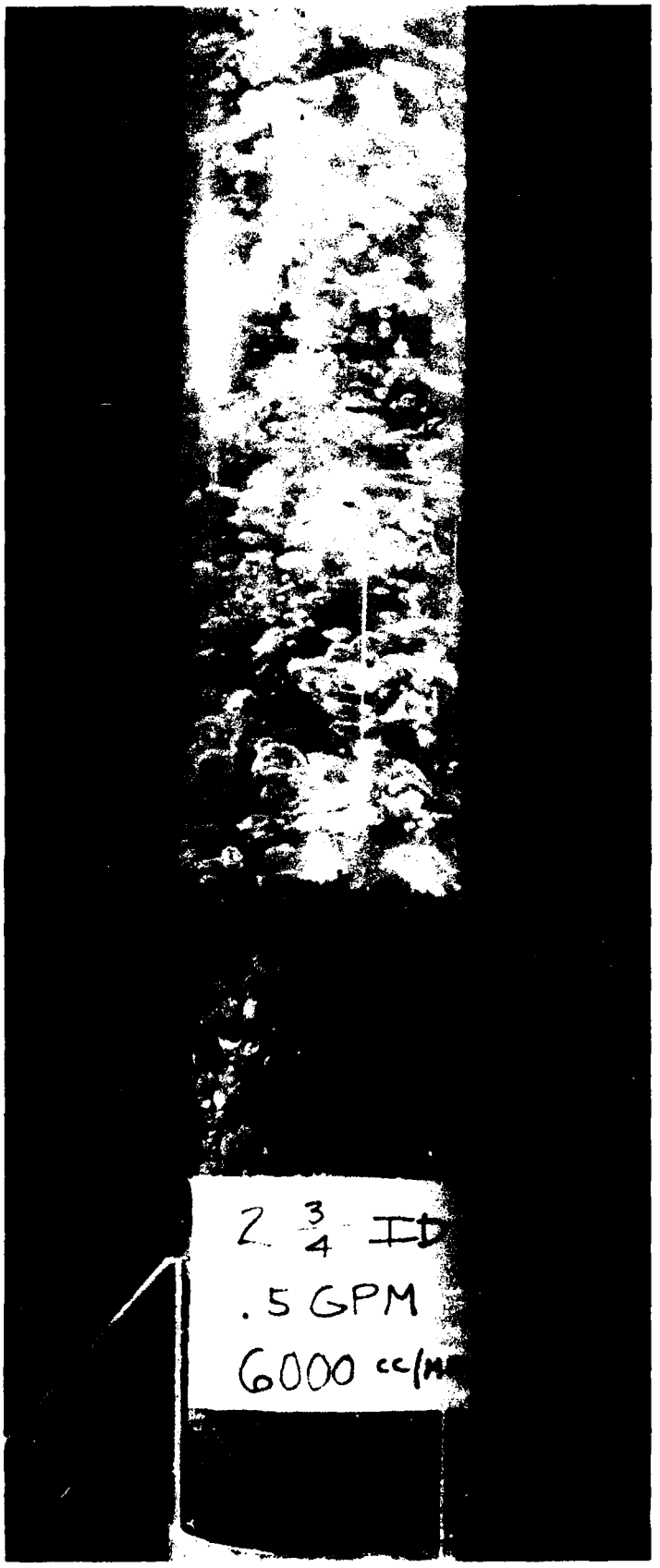
APPENDIX A

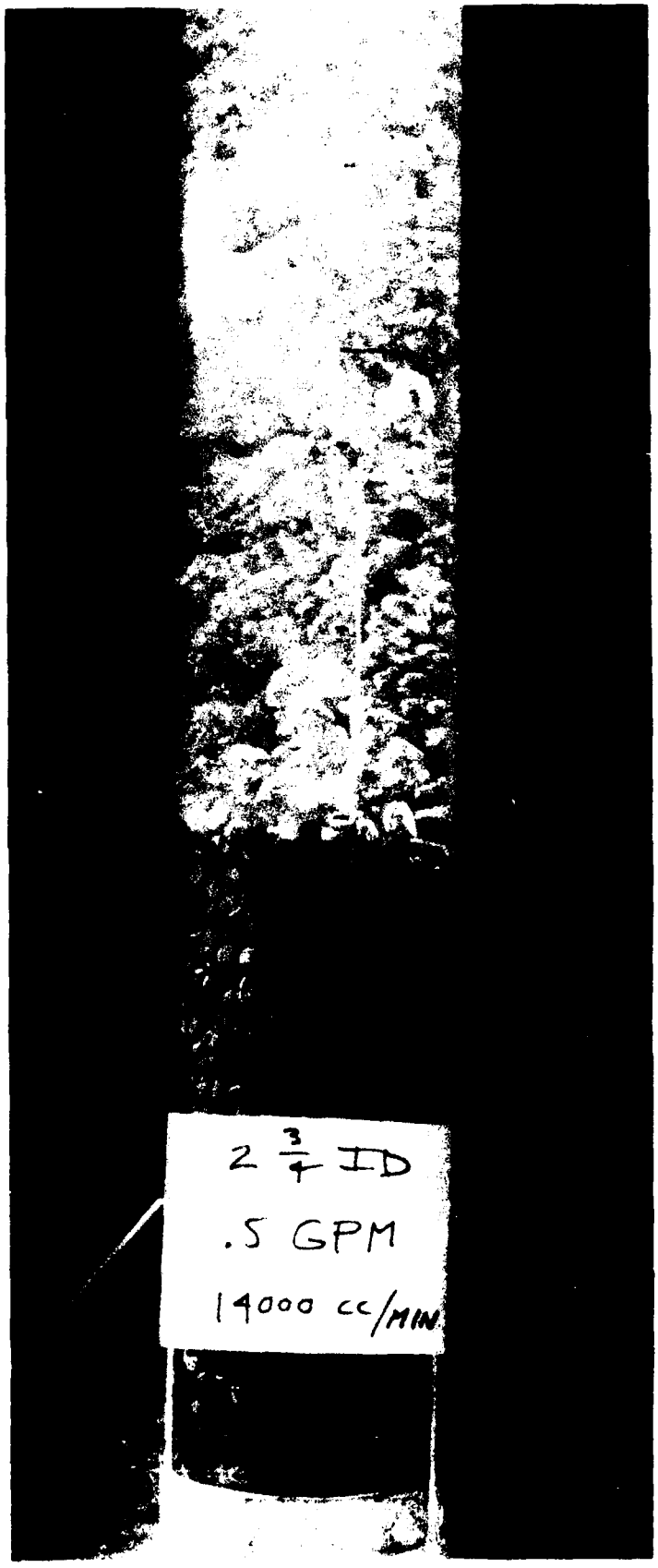
REACTOR FLOW MODEL BUBBLE TEST RESULTS

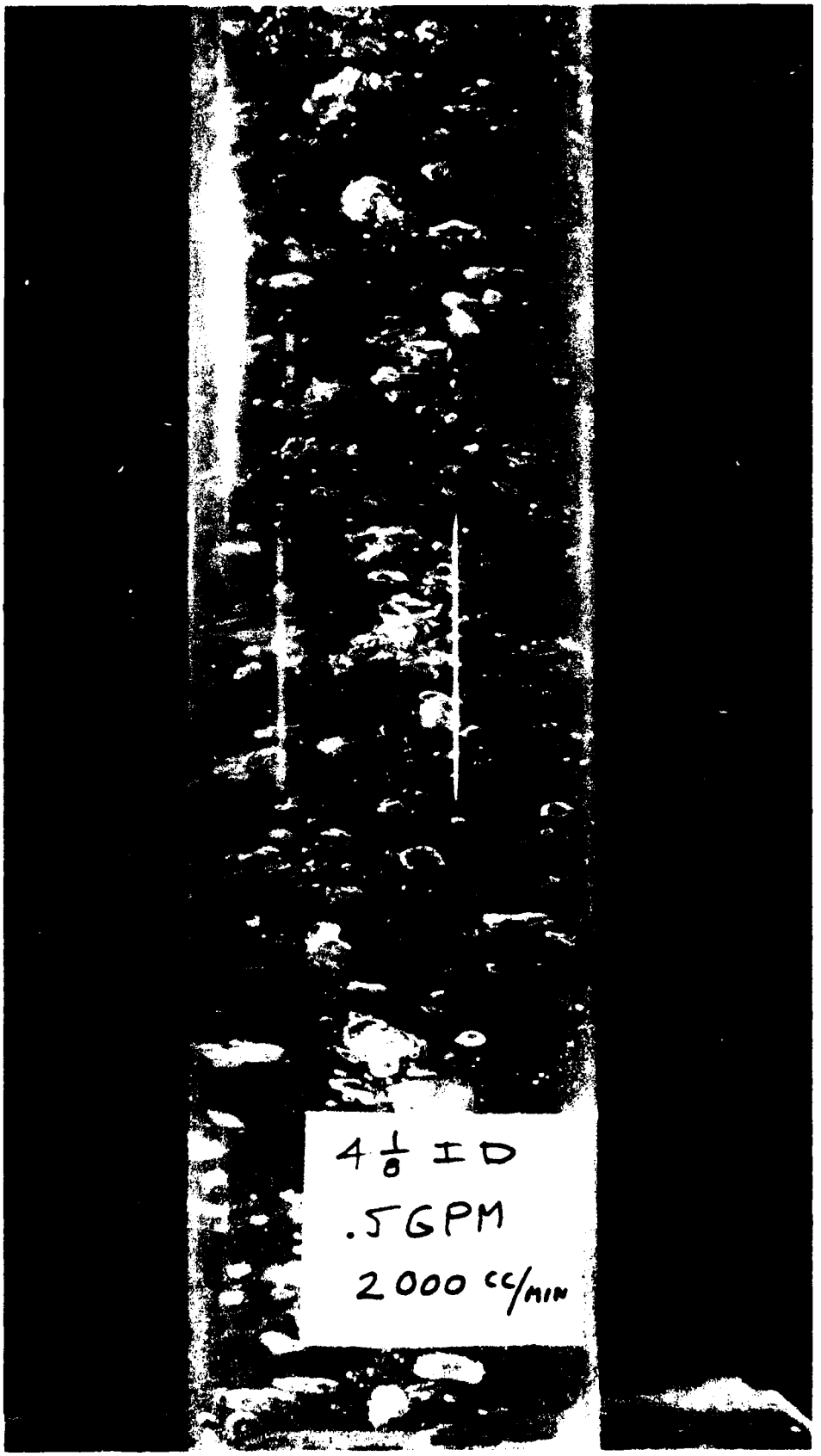

1 3/4


1 3/4


2 3/4 ID
.5 GPM
2000 cc/MIN


2 3/4 ID
.5 GPM
6000 cc/M

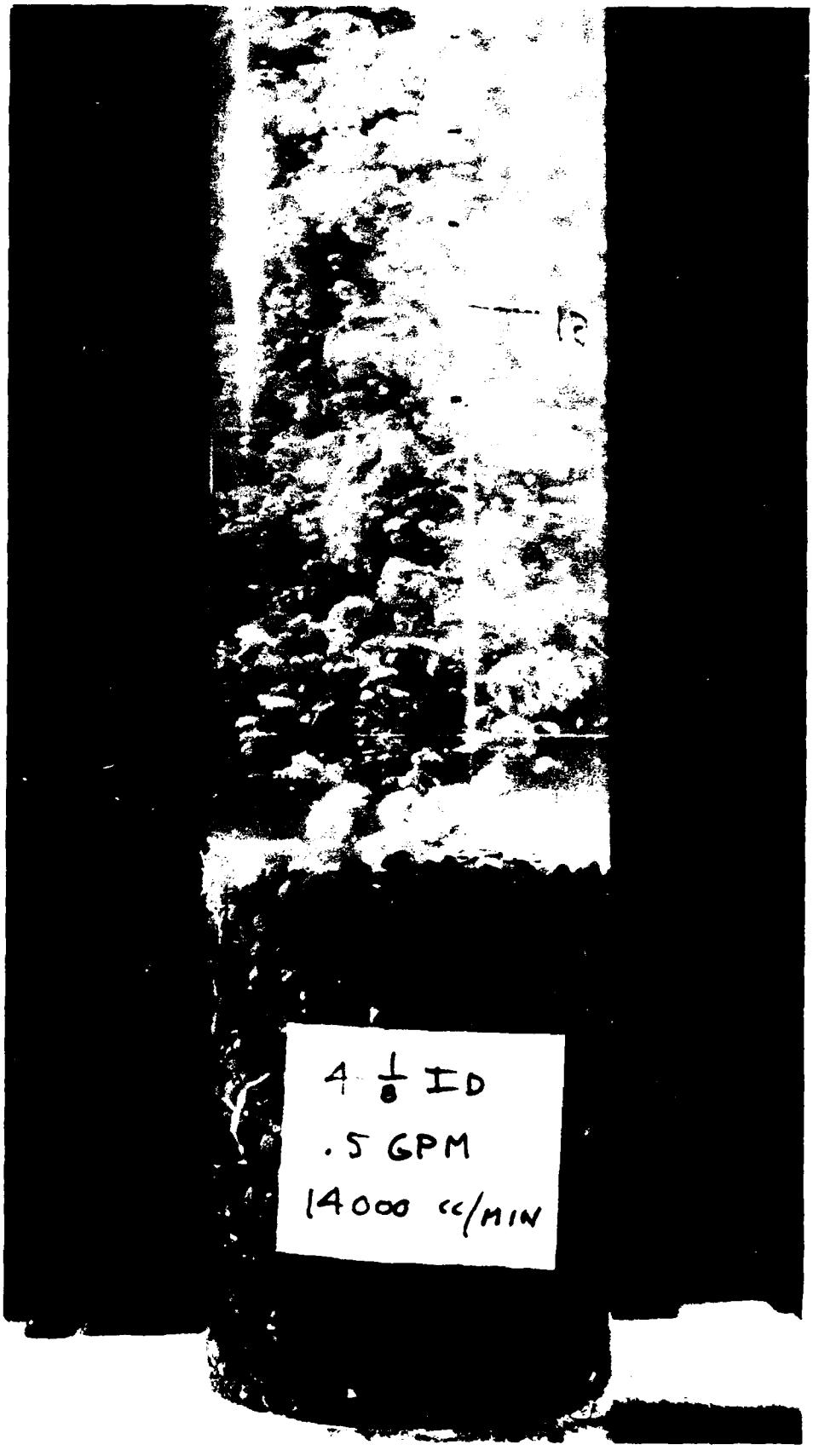

2 3/4 ID
.5 GPM
14000 cc/MIN.

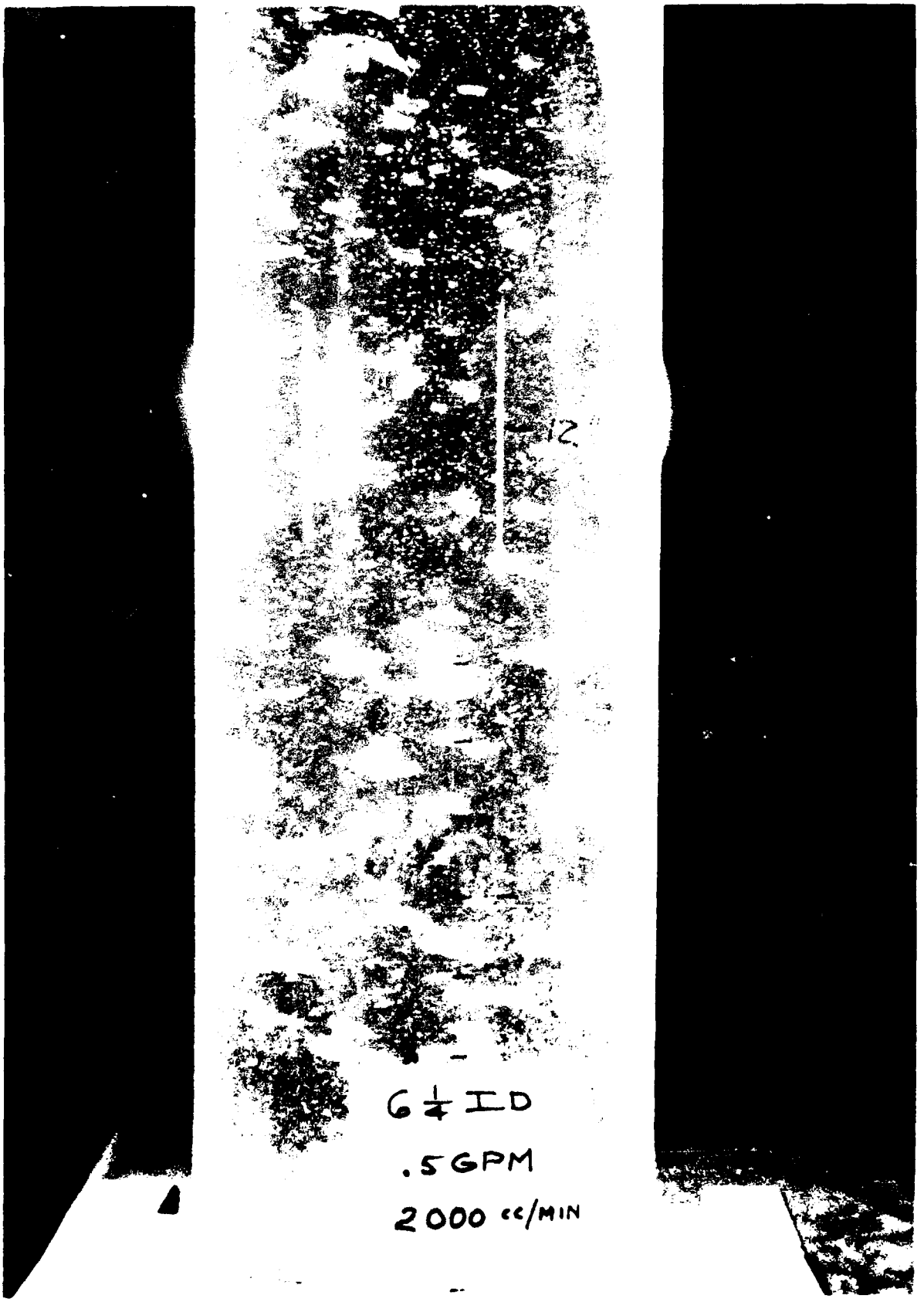

4 1/8 ID
.5 GPM
2000 cc/MIN


4½ ID
.5GPM
6000 %/min

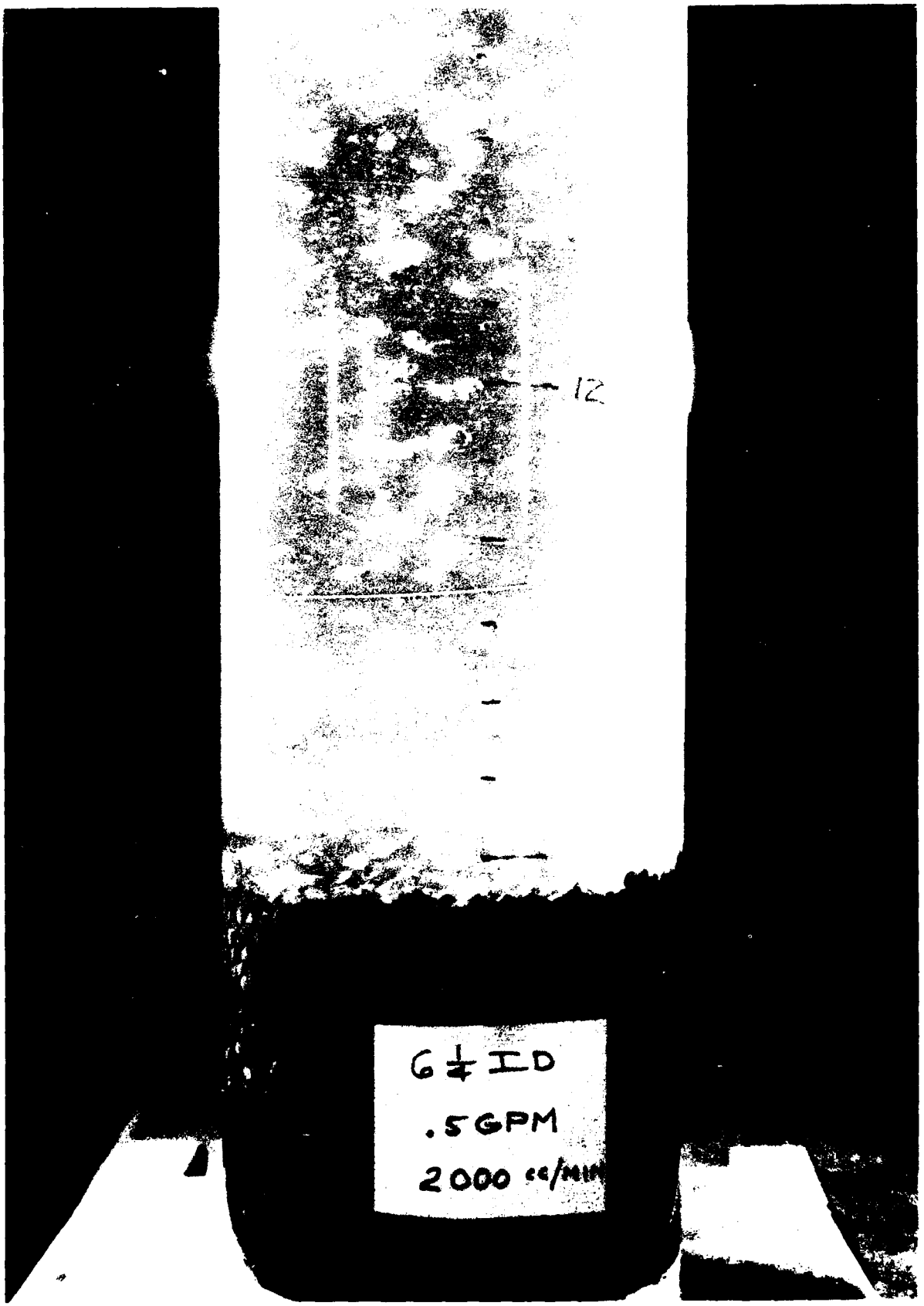

4 1/8 ID
.5 GPM
2000 cc/min

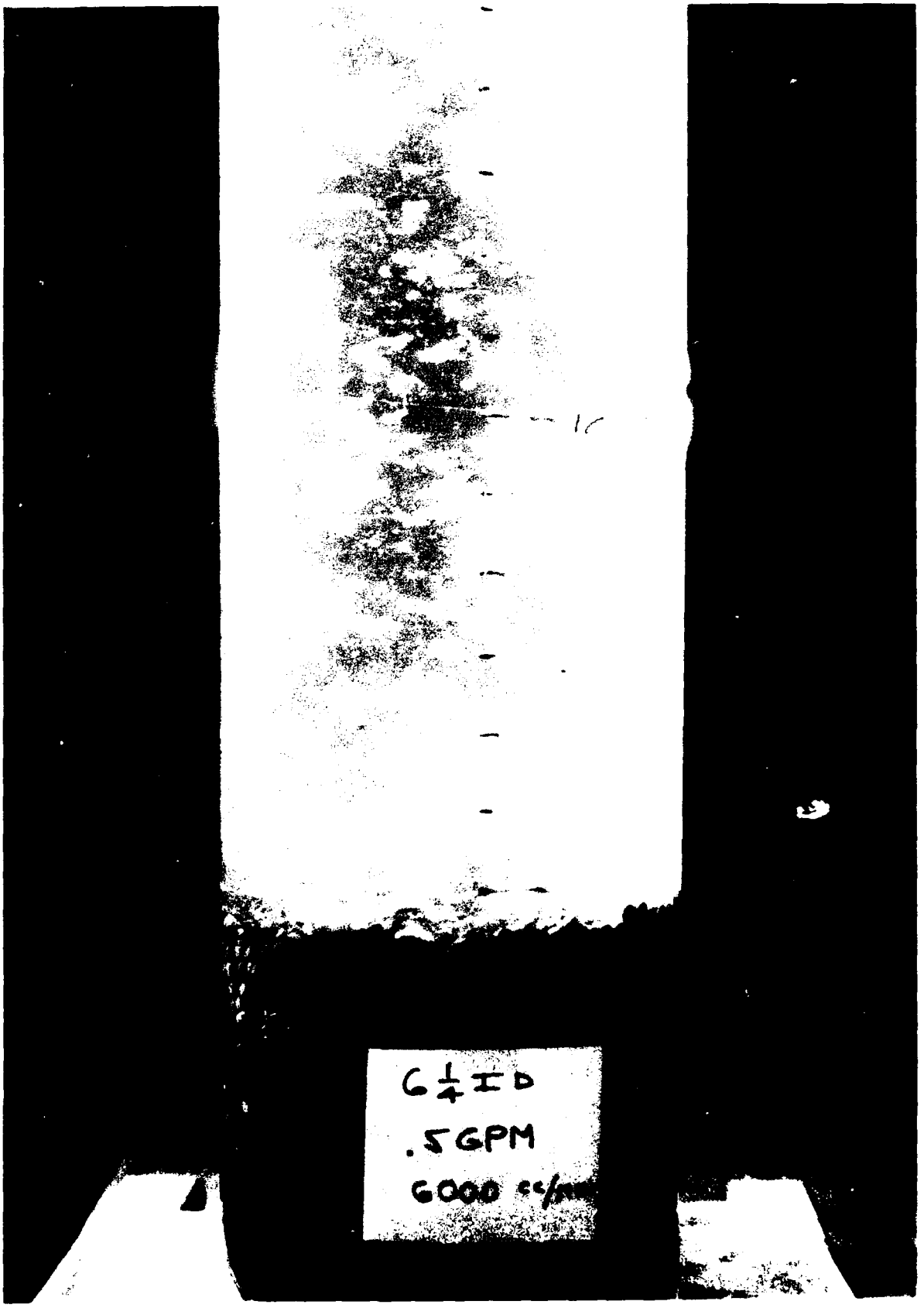

12
4½ ID
.5GPM
6000 #/min

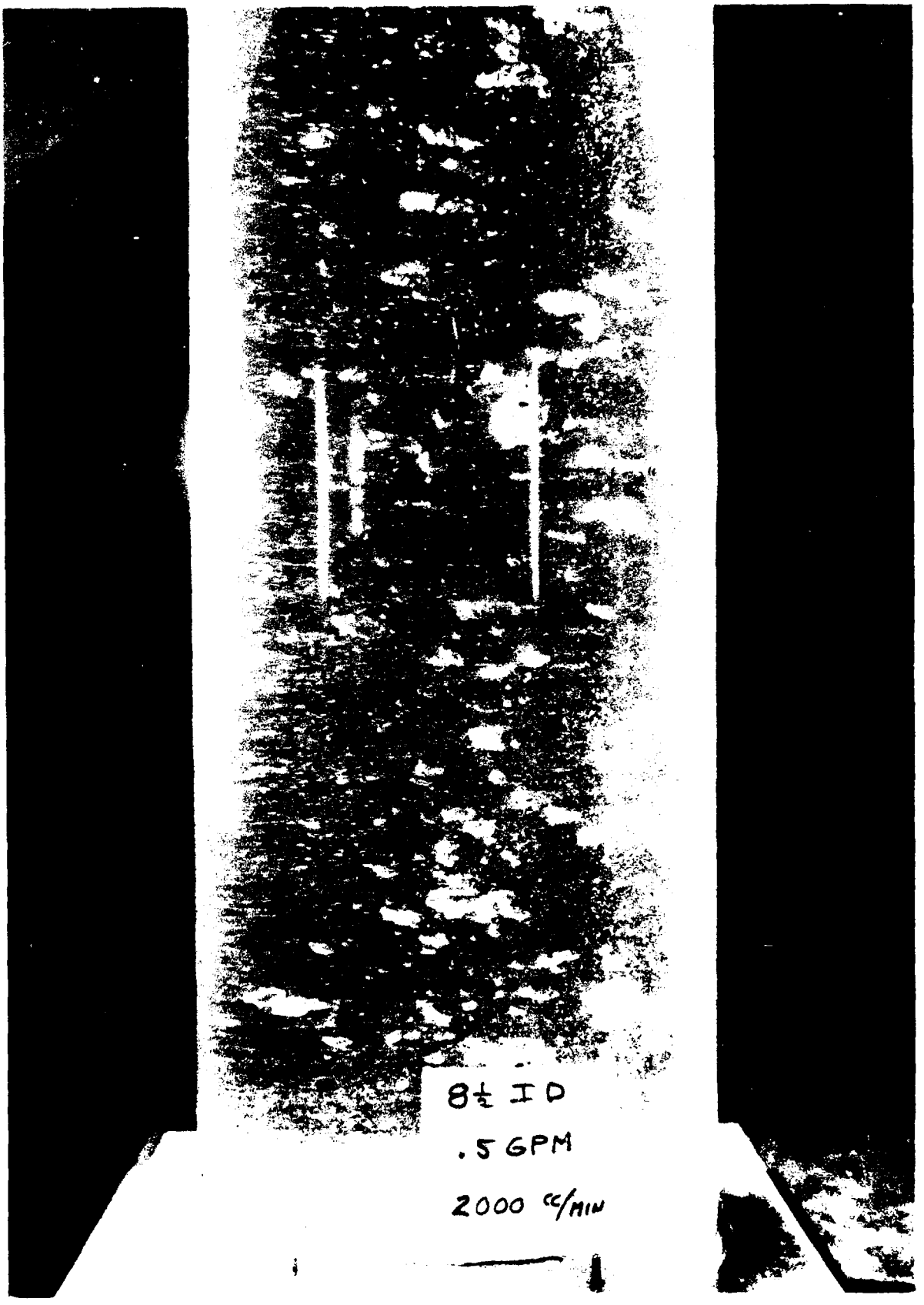

4 1/8 ID
.5 GPM
14000 cc/MIN

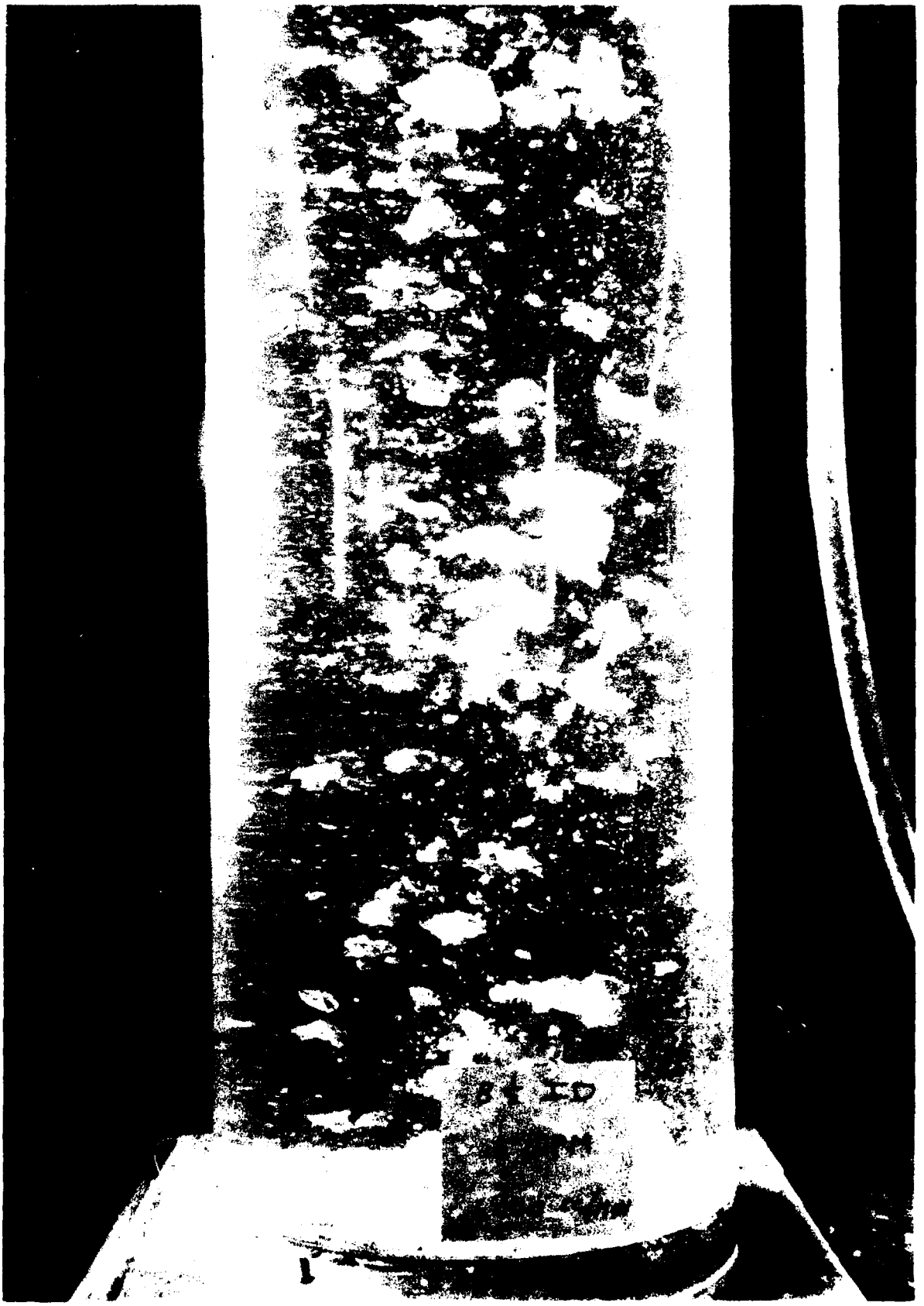

12.
6¼ ID
.5 GPM
2000 cc/MIN

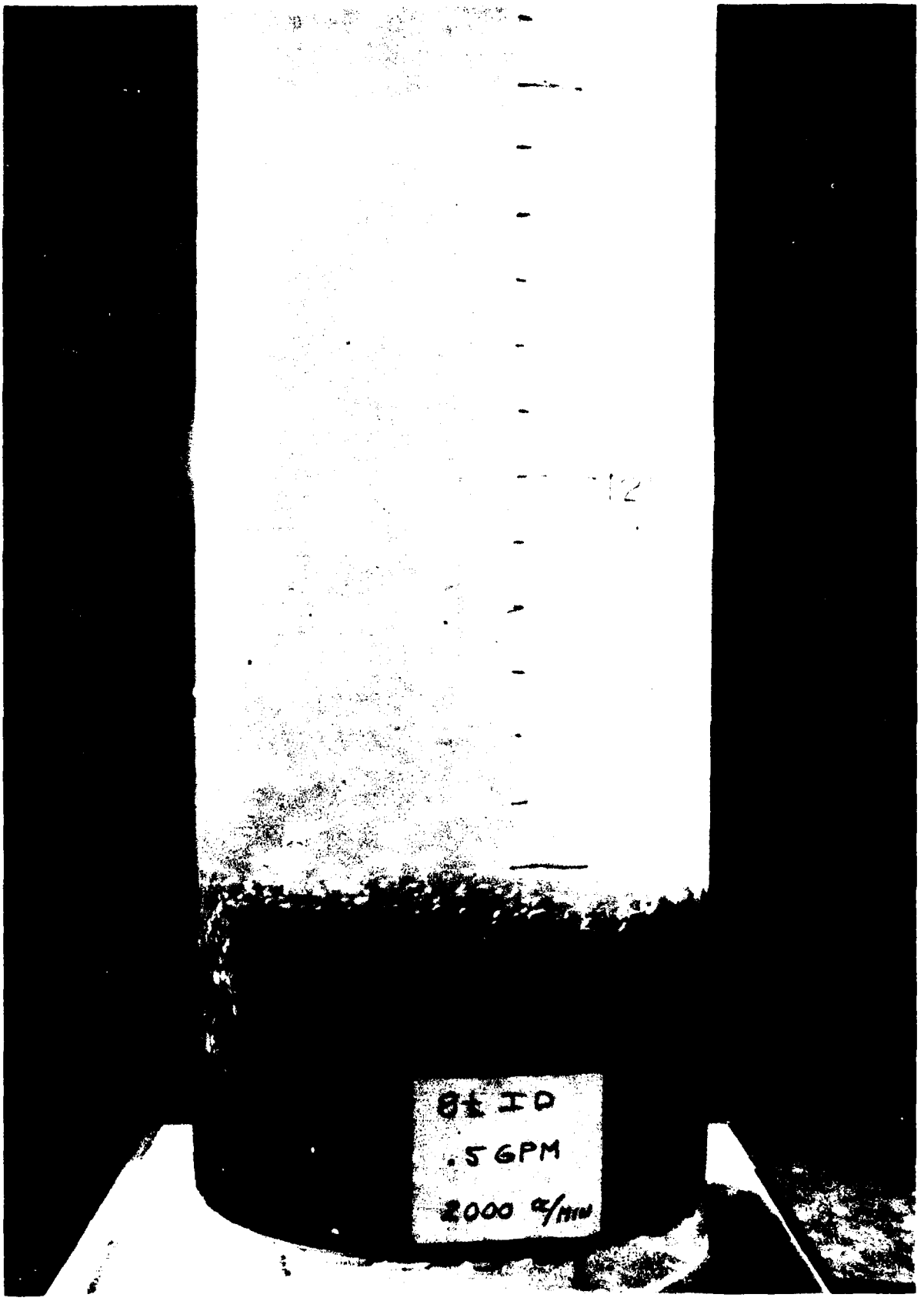

6 1/4 ID
.5 GPM


12
6 1/4 ID
.5 GPM
2000 cc/MIN


6 1/4 ID
.5GPM
6000 cc/m


8½ ID
.5 GPM
2000 cc/MIN




8t ID
.5 GPM
2000 q/min

GPO 889-318