DESIGN AND DEVELOPMENT OF A PROTOTYPE WET OXIDATION SYSTEM FOR THE RECLAMATION OF WATER AND THE DISPOSITION OF WASTE RESIDUES ONBOARD SPACE VEHICLES

(NASA-CR-112151) DESIGN AND DEVELOPMENT OF A PROTOTYPE WET OXIDATION SYSTEM FOR THE RECLAMATION OF WATER AND THE DISPOSITION OF WASTE R.B. Jagow (Lockheed Missiles and Space Co.) 26 May 1972 146 p CSCL 06I G3/05

N72-33115

Unclas
44081

PREPARED UNDER CONTRACT NAS1-9183

by Biotechnology Organization

Lockheed Missiles & Space Company, Inc.

Sunnyvale, California

NATIONAL AERONAUTICS
AND
SPACE ADMINISTRATION
LANGLEY RESEARCH CENTER, LANGLEY STATION
HAMPTON, VIRGINIA

146

DESIGN AND DEVELOPMENT OF A PROTOTYPE WET OXIDATION SYSTEM FOR THE RECLAMATION OF WATER AND THE DISPOSITION OF WASTE RESIDUES ONBOARD SPACE VEHICLES

Prepared Under Contract NAS1-9183

by

Biotechnology Organization
Lockheed Missiles & Space Company, Inc.
Sunnyvale, California

R. B. Jagow

26 May 1972

NATIONAL AERONAUTICS AND SPACE ADMINISTRATION
LANGLEY RESEARCH CENTER
LANGLEY STATION
HAMPTON, VIRGINIA

LIST OF CONTRIBUTORS

Name	Area of Contribution
R. B. Jagow	Project Leader
R. W. Joy	System Design & Test
R. J. Jaffe	Laboratory Process Investigations
R. C. Tuttle	Chemical Analysis
R. L. Gorman	Water Recovery Studies
R. A. Lamparter	Ammonia Removal Studies
J. F. Rynewicz	Metallurgy

NASA TECHNICAL MONITOR

V. G. Collins

SPACE SYSTEMS DIVISION
NASA, Langley Research Center

CONTENTS

Page

LIST OF CONTRIBUTORS iii

ILLUSTRATIONS vii

TABLES xi

SUMMARY 1

INTRODUCTION 5

INITIAL LABORATORY PROGRAM 7

FINAL LABORATORY PROGRAM 9

PROTOTYPE SYSTEM DESIGN 21

- SLURRY PUMP 21
- REACTOR 23
- DRY BOILER 30
- SYSTEM DESCRIPTION 30

PROTOTYPE SYSTEM TESTS 39

- SLURRY FEED CALIBRATION TEST 39
- OXYGEN FEED CALIBRATION TEST 42
- DESIGN VERIFICATION TEST 45
- POST DVT INVESTIGATIONS 51
- INITIAL PROTOTYPE SYSTEM TEST CONCLUSIONS 52

SLURRY PUMP DEVELOPMENT 55

- DOUBLE-ENDED SLIDE VALVE SLURRY PUMP 55
- HYDRAULIC PISTON PUMP 61

MATERIALS CORROSION STUDIES 67

OXYGEN SUPPLY SYSTEM 77

- HIGH PRESSURE GAS 77
- SUPERCRITICAL CRYOGENICS 77
- CHEMICAL OXYGEN SUPPLY 81
- HIGH PRESSURE ELECTROLYSIS 81

ALTERNATE WATER RECOVERY DEVICES 83

- SYSTEM COMPARISONS 83
- ELECTRODIALYSIS SYSTEM DESCRIPTION 87
- VAPOR COMPRESSION DISTILLATION SYSTEM DESCRIPTION 89

Page

VAPOR DIFFUSION SYSTEM DESCRIPTION 92
REVERSE OSMOSIS SYSTEM DESCRIPTION 94
AIR EVAPORATION SYSTEM DESCRIPTION 96
DATA SOURCES 98
EFFLUENT WATER RECOVERY TESTS 100
AMMONIA REMOVAL 101
ACID NEUTRALIZATION 101
DIRECT REDUCTION 101
CATALYTIC OXIDATION 101
REGENERABLE ABSORPTION 101
ELECTROCHEMICAL 102
ELECTRODIALYSIS 102
WET OXIDATION REACTION 102
SOLIDS GRINDER 105
ASH FILTER 113
REACTOR DESIGN MODIFICATIONS AND SYSTEM TESTS 117
CARBON BEARING LIFE TEST 117
90-DAY BALL BEARING LIFE TEST 124
CONCLUSIONS 133
REFERENCES 135
LIBRARY CARD ABSTRACT 137

ILLUSTRATIONS

Figure		Page
1	Typical Commercial Continuous Wet Oxidation System	6
2	Wet Oxidation Batch Reactor Laboratory Test Setup	10
3	Effects of Temperature on COD Reduction for Wet Oxidation of Fecal/Urine Slurries	13
4	Plot of Ammonia Conversion for Vapor Phase Catalyst	19
5	Effluent Water and Ash from Proof Test and Mixed Solids Test Runs	20
6	Slurry Pumping Approaches	22
7	Static Mixer Reactor Model	24
8	Stirred Reactor Model	26
9	Assembly Drawing of Stirred Reactor	27
10	Assembly Drawing of Dry Boiler	31
11	Initial Prototype Spacecraft Wet Oxidation System Schematic	32
12	Slurry Feed Valve Control Cycle	34
13	Initial Prototype Spacecraft Wet Oxidation System Electrical Schematic	35
14	Initial Prototype Spacecraft Wet Oxidation System Arrangement	36
15	Initial Prototype Spacecraft Wet Oxidation System Control Panel	37
16	Slurry Feed Calibration Test Schematic	40
17	Slurry Feed Rate vs Nitrogen Regulator Pressure	41
18	Oxygen Feed Calibration Test Schematic	43
19	Oxygen Feed Rate vs Oxygen Regulator Pressure	44
20	100-Hour Design Verification Test Results	47
21	Double-Ended Slide Valve Slurry Pump Schematic	57
22	Double-Ended Slide Valve Slurry Pump	58
23	Double-Ended Slide Valve Slurry Pump Test Schematic	60
24	Assembly Drawing of Hydraulic Piston Pump	62
25	Hydraulic Piston Pump Test Schematic	63

ILLUSTRATIONS
(Continued)

Figure		Page
26	Hydraulic Piston Pump Test Setup	64
27	Typical "U" Bend Corrosion Test Specimen	69
28	Corrosion Test Specimens Installed on Reactor Head	69
29	Corrosion Test Schematic	72
30	Corrosion Test Specimens	74
31	Corrosion Test Specimens	75
32	Oxygen Supply System Trade Study Results - No Oxygen Credit	79
33	Oxygen Supply System Trade Study Results - Oxygen Credit	80
34	Water Recovery System Equivalent Weight vs Mission Duration	85
35	Electrodialysis System Schematic	88
36	Vapor Compression Distillation System Schematic	90
37	Vapor Diffusion System Schematic	93
38	Reverse Osmosis System Schematic	95
39	Air Evaporation System Schematic	97
40	Urshel Laboratories Comitrol Cutter	106
41	Laboratory Test Model Grinder	108
42	Grinder Assembly	109
43	Laboratory Model Grinder Test Setup	110
44	Spacecraft Trash Grinder System	111
45	Cartridge Type Filter	114
46	Filter Bag	115
47	Modified Reactor Design	118
48	Carbon Bearing Life Test Setup	119
49	Carbon Bearing Life Test Schematic	120
50	Carbon Bearing Life Test Results	122

ILLUSTRATIONS
(Continued)

Figure		Page
51	Flodyne Motorized Ball Valves	123
52	Reactor Back Pressure Regulator	125
53	Reworked Flodyne Motorized Ball Valve	126
54	Whitey/Crocker Motorized Ball Valve	127
55	90-Day Ball Bearing Life Test Results	129
56	Reactor Parts After 90-Day Test	131

PRECEDING PAGE BLANK NOT FILMED

TABLES

Table		Page
1	Effects of Oxygen Partial Pressure and Percent Excess Oxygen on Wet Oxidation of Fecal/Urine Slurries	11
2	Effects of Temperature on Wet Oxidation of Fecal/Urine Slurries	13
3	Proof Test Results	14
4	Catalysts in Wet Oxidation	17
5	Liquid Phase Catalyst Experimental Results	18
6	Initial Prototype Spacecraft Wet Oxidation Parts List	38
7	Slurry Feed Calibration Test Data	39
8	Oxygen Feed Calibration Test Data	42
9	Reactor Metals Color Test Results	49
10	Design Verification Test Results - Reactor Effluent Water Characteristics	50
11	Reactor Effluent Water Quality vs Drive Speed	51
12	Candidate Reactor Metals	68
13	Corrosion Test Specimen Deflections	70
14	Oxygen Supply System Trade Study - Waste Model	78
15	Oxygen Content of Oxygen Source Chemicals	81
16	Candidate Water Recovery System Characteristics	84
17	Ammonia Removal Test Results - One Liter Batch Reactor	103
18	Spacecraft Waste Model for Grinder Development	105

Preceding page blank

DESIGN AND DEVELOPMENT OF A PROTOTYPE WET OXIDATION SYSTEM FOR THE RECLAMATION OF WATER AND THE DISPOSITION OF WASTE RESIDUES ONBOARD SPACE VEHICLES

By R. B. Jagow
Biotechnology
Lockheed Missiles & Space Company, Inc.

SUMMARY

Wet oxidation has been used commercially for sewage sludge oxidation for several decades. NASA interest in the process for spacecraft application resulted from the fact that a high degree of waste oxidation can be achieved by burning the wastes in the presence of oxygen and steam at temperatures of 450 to 550°F (corresponding pressures from 1000 to 3000 psi) without producing the gaseous oxides of nitrogen, sulphur and phosphorous, while producing a clear, salty, sterile effluent water and white phosphate ash. This allows recovery of useful water and gases from spacecraft wastes and elimination of all overboard venting.

The first step in the development effort was a laboratory investigation to optimize the process conditions. A series of 34 runs using fecal/urine slurries in a one-liter agitated batch reactor resulted in selection of the following process conditions:

Reaction temperature	550°F
Oxygen partial pressure	1100 psi
Reactor total pressure	2200 psi
Waste processing time	1-1/2 hours
Slurry solids content	1.7 to 30.8%*
Oxygen required	0.8 gm oxygen/gm solids

Catalyst tests were made, but none were found that would improve process conditions. A test was also run with a mixture of paper towels, toilet paper, wet wipes, aluminized food bags, and photographic film. A high degree of oxidation was achieved showing that the wet oxidation process works well with most any waste material.

---

* Range tested - Process can probably handle any solids concentration that is still fluid.

A prototype spacecraft wet oxidation system was designed, fabricated, and tested, based on the results of the laboratory program. The system consisted of an oxygen supply tank, oxygen flow controls, slurry feed tanks and controls, a continuous flow stirred reactor, back pressure controls, dry boiler, and miscellaneous controls and gages. Slurry and oxygen were forced into the reactor at controlled rates under pressure. The reactor was designed to stir the mixture and force it through an internal baffling system so that the mixture passed from the inlet to the outlet in 1-1/2 hours, minimizing the mixing of influent and effluent streams. A magnetic coupling with external electric motor drove the stirring mechanism to eliminate a dynamic shaft seal. The reaction products (water, gases, and ash) were vented from the reactor through a cooling coil and back pressure control valve into the dry boiler. The water was sprayed on a hot finned plate to vaporize the water leaving the salt and ash adhering to the plate. The water vapor passed through a membrane to prevent carryover of any solid particles before entering a condenser where the product water was collected for analysis.

A 100-hour design verification test (DVT) was run on the system. Results were generally favorable and a number of areas for further development were identified which formed the basis for the additional work conducted after completion of the DVT. The work accomplished in each work area is summarized briefly below:

Slurry Pump - The low flow rates, slurry mixtures, and high pressures made it difficult to locate a slurry pump for the spacecraft wet oxidation system. Two pumping systems have been developed, one using a motor driven hydraulic piston pump that pumps water to the back side of a bladdered tank to force slurry out of the tank into the reactor, and a second that utilizes the reactor effluent liquid and gas to force the slurry into the reactor using a double-ended slide valve piston pump. The slide valves operate similarly to a steam locomotive slide valve.

Materials Corrosion - Selection of a suitable metal for the reactor and other high temperature portions of the system resulted in a materials test program that exposed metal samples to the wet oxidation environment for extended periods. Hastalloy C was selected as a preferred metal with Inconel 625 a close second choice.

Oxygen Supply - Trade-off studies compared high pressure gas, supercritical cryogenic, chemical, and high pressure water electrolysis oxygen supply techniques. Water electrolysis was selected.

Water Recovery Devices - Electrodialysis, vapor compression distillation, vapor diffusion, reverse osmosis, and air evaporation water reclamation systems were compared for the salt water cleanup of the wet oxidation reactor effluent as alternates to the dry boiler. The trade study resulted in selection of vapor compression as a first choice and electrodialysis as a second choice. Concern about the zero gravity aspects of the dry boiler resulted in elimination of it as a candidate.

Ammonia Removal - Chemical analysis of the filtered reactor effluent water that had been boiled and condensed to simulate the vapor compression distillation process showed ammonia as the only contaminant. Its appearance in larger quantities (0.55% by weight) presented an important development problem. Surveys of ammonia removal methods resulted in emphasis on finding a catalyst that could be added to the wet oxidation reactor that would prevent ammonia formation. Tests in the one-liter batch type laboratory reactor resulted in selection of Ruthenium Trichloride as a very effective ammonia removal catalyst that, when introduced in small quantities, completely eliminated the ammonia formation.

Solids Grinder - A grinder that could pulverize the personal hygiene, medical, waste food, and other biologically contaminated wastes onboard a spacecraft so that they would be pumped into a wet oxidation reactor for burning was investigated. A laboratory model was built and tested and design recommendations for a flight prototype were evolved.

Reactor Design - The DVT showed areas for improvement in reactor design to greatly enhance assembly and disassembly techniques as well as to improve bearing life and drive motor performance. Reactor re-design, a 40-day carbon bearing life test, and a 90-day ball bearing life test were conducted. The carbon bearings failed after 40 days and the ball bearings were still operating at the conclusion of the 90-day test. This test also served the purposes of a final checkout of the new system design.

Valves and Regulator - Some difficulties were encountered in finding suitable slurry shutoff valves and a back pressure regulator. The nature of the sewage with solid particles including fine fiberous material and the finely divided effluent ash coupled with the high pressure requirement made it very difficult to find valves and regulators that would meet requirements. After testing several models, satisfactory components were found.

As a result of these development efforts, a program directed to fabricate and test a spacecraft prototype system suitable for manned chamber testing can logically be pursued.

INTRODUCTION

The wet oxidation process has been developed for commercial use in the chemical, paper, and sewage treatment industries. It provides a low temperature, sometimes self-sustaining means of oxidizing wastes to relatively safe inert effluent liquid and gaseous products. Figure 1 is a schematic of a typical commercial wet oxidation process. The waste liquid is pumped to reactor pressure by a positive displacement slurry pump. Air from an air compressor is mixed with the slurry at the pump outlet, and the mixture passes through two regenerative heat exchangers before entering the reactor. Reactor conditions are generally in the range of 450 to 550°F with corresponding pressures from 1000 to 3000 psi. The effluent passes through the high temperature regenerative heat exchanger and to a liquid/vapor phase separator. The liquid is passed through the low temperature regenerative heat exchanger to heat the incoming slurry air mixture before discharge. The vapor and gases are fed to a mixed gas turbine that drives the feed air compressor allowing recovery of process energy. The primary advantage of the process for commercial applications is the low oxidation temperature that allows more economical burning of wastes and longer equipment operating life while producing a sterile, nearly inert liquid phase and a relatively clean gaseous phase. Although the commercial units designed for low installation and operation costs are never operated to obtain extremely high degrees of oxidation, the process is capable of achieving 99 percent or greater reduction in chemical oxygen demand (COD) without producing the oxides of sulfur, phosphorous, and nitrogen that are associated with the more conventional high-temperature dry incineration. These elements appear as sulfate and phosphate ions in the liquid phase and can, therefore, be more readily handled. The purity of the liquid and gas phases are the primary reasons for interest in the process for manned spacecraft. The recovery of useful water and gases from urine and fecal matter coincident with processing of wastes to a low volume sterile, nondegradable ash, and the elimination of overboard venting of waste liquids and gases are the promises of wet oxidation. The penalties that must be paid are the weight and power required for a moderate temperature, high pressure process, and the equipment problems of handling sewage sludge and salt water effluent.

This report describes the laboratory process investigations; the design, fabrication, and testing of an initial prototype system; and the results of several critical development efforts that resulted from the tests run on the initial prototype.

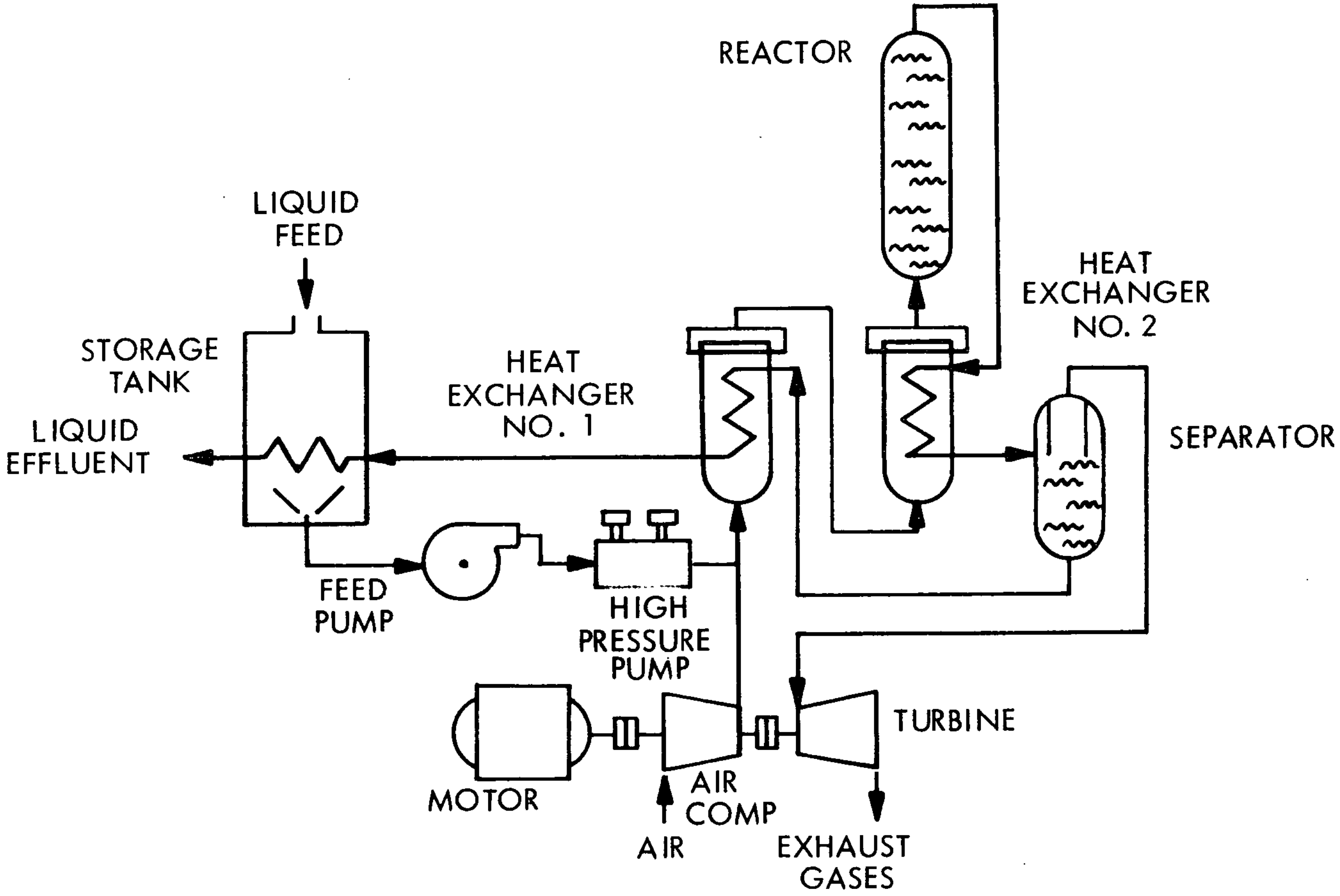


Fig. 1 Typical Commercial Continuous Wet Oxidation System

INITIAL LABORATORY PROGRAM

The spacecraft wet oxidation process varies from the commercial process in a number of significant ways. The process flows are minute, pure oxygen is the preferred input gas, and the process must be optimized to yield the highest practical water and gas purity. It was recognized by NASA that a laboratory program was needed to investigate the feasibility of wet oxidation for spacecraft waste treatment.

Contract NAS 1-6295 was let to this end, and the results are reported (Ref. 1). The most significant conclusions are summarized in the following:

1. 95 percent or greater COD reduction was achieved.
2. Pure oxygen was preferable to air.
3. A base metal oxide catalyst showed promise for reducing temperatures and promoting more complete oxidation at the higher temperature.
4. The effluent consisted of a "dark, rapidly settling precipitate and a clear colorless to pale yellow supernatant. The precipitate was entirely composed of inorganic ash. The supernatant contained small amounts of dissolved organic matter and inorganic salts."
5. The gas and vapors were relatively free of contaminants (small amounts of acetone vapor, carbon monoxide, and hydrogen were detected).
6. Slurry feed solids concentration was not an important variable in the completeness of oxidation.
7. Continuous stirring of the slurry enhanced oxidation.
8. Temperatures from 550 to 600°F were preferred.
9. Significant quantities of ammonia were found in the effluent water.

PRECEDING PAGE BLANK NOT FILMED

FINAL LABORATORY PROGRAM

Based on these results, it was decided to initiate a program for the design, fabrication, and testing of a prototype spacecraft wet oxidation system, covered by Contract NAS 1-9183. The program included a laboratory investigation to further study the effects of temperature, time, oxygen partial pressure, percent excess oxygen, and solids concentration prior to conduct of detail design of the unit.

The laboratory investigations were conducted in a one liter, batch type, stirred reactor, showed by Figure 2. Several initial runs were made to establish the slurry volume and agitation speed that were required to achieve good mixing of the liquid and gas phases. Three hundred milliliter initial slurry charge volume and 1200 rpm agitator speed were selected. Reaction time was tentatively set at 1-1/2 hr. It was concluded from the previous laboratory program that those conditions that produced the greatest reduction in COD also produced the best quality effluent, and, therefore, COD was used throughout the program as an indication of effluent purity. The effects of input solids concentration on reaction effluent purity were investigated early in the program to establish whether fresh or concentrated urine would be used in the process. The results of four runs at 5.3, 10.6, 15.5, and 30.8 percent solids by weight in a fecal/urine mixture showed that solids concentration was not a significant variable with reduction in COD of 95.6, 98.2, 98.4, and 97.3 for the four runs, respectively. The significance of this data is that if solids content is not important to the process, then solids content can be selected to provide the most advantageous design; i.e., because water reclamation is required for the wet oxidation effluent anyway, why not eliminate the urine processing system and introduce a mixture of feces and unprocessed urine directly into the wet oxidation system? Such an approach would undoubtedly reduce overall system weight and complexity. Therefore, further system studies and laboratory test runs were conducted based on a seven percent solids content in the feed slurry. This concentration is equivalent to the generally agreed upon urine and fecal production rates and solids content published for system design.

During these early runs, the importance of oxygen partial pressure was observed, and, therefore, a series of runs were made to investigate the effects of oxygen partial pressure and percent excess oxygen. Table 1 presents the results of these runs.

Runs 11, 12, 13, and 14 combine the variables of oxygen partial pressure (PO_2) and percent excess oxygen in all four combinations of extreme high and lows. High PO_2 and high percent excess oxygen and low PO_2 and low percent excess oxygen can easily be achieved by charging more or less oxygen initially. The other two combinations of these two variables, i.e., high-low and low-high required adjustments in solids concentration and/or slurry volume to achieve them. High solids concentrations and large slurry volumes required larger amounts of oxygen, thereby achieving low percent excess with high PO_2. The converse is also true. The data from these runs show very clearly the

Preceding page blank

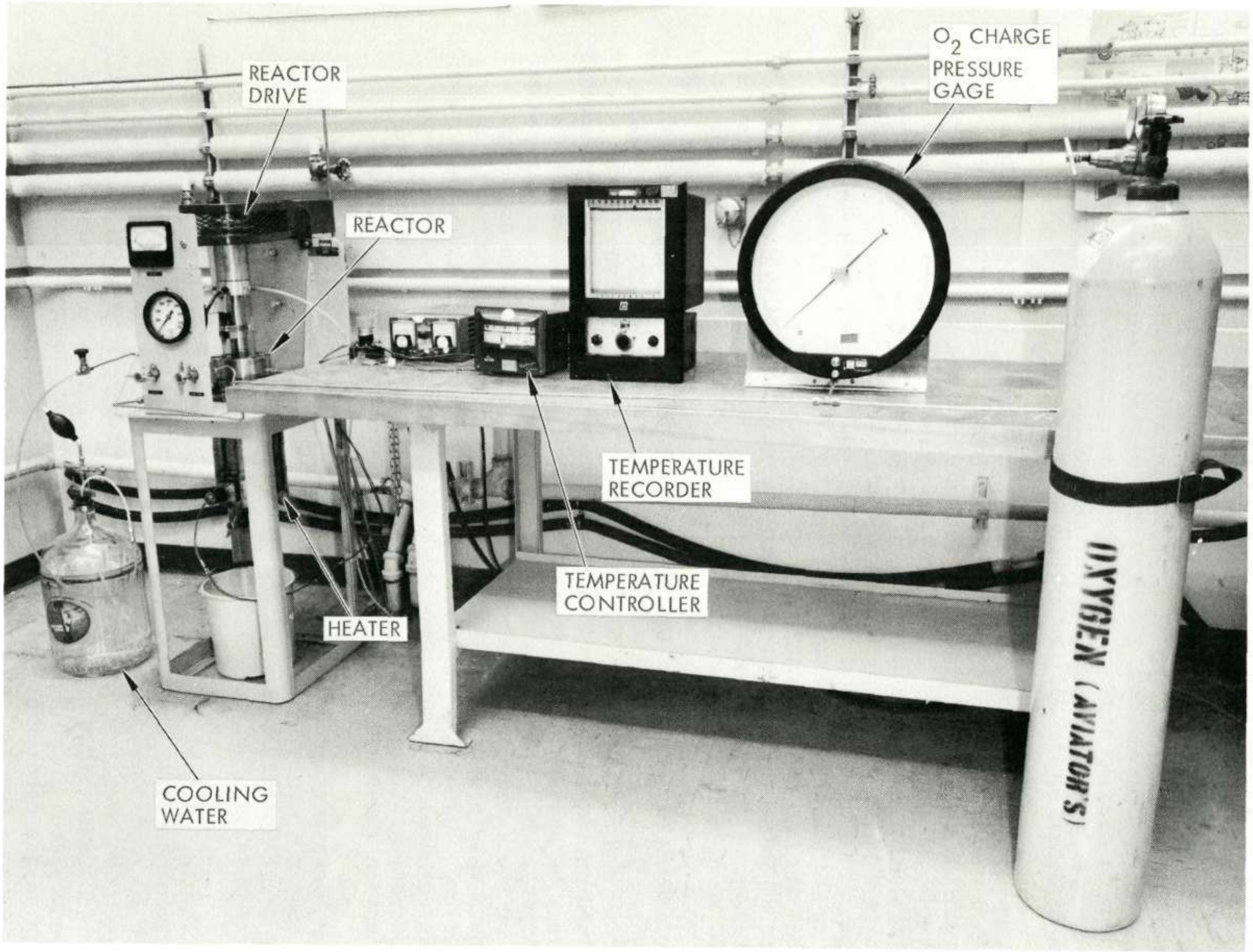


Fig. 2 Wet Oxidation Batch Reactor Laboratory Test Setup

Table 1 - Effects of Oxygen Partial Pressure and Percent Excess Oxygen on Wet Oxidation of Fecal/Urine Slurries

Run No.	% Solids	Temp. (°F)	Oxygen to Solids Ratio (gmO_2/gm Solids)	Initial O_2 Charge Pressure (psia)	Slurry Volume (cc)	% COD Reduction
10	5.3	550	5.3	1215	300	99.5
11	5.3	625	5.7	1295	300	98.7
12	5.3	625	1.2	282	300	94.7
13	14.3	625	1.1	1295	460	98.8
14	1.7	550	5.3	400	300	94.7
15	17.1	550	0.8	800	360	99.1

importance of PO_2 and the unimportance of percent excess oxygen. Runs 10 and 11 show the fact that good oxidation can be achieved at 550°F with no gain whatsoever at higher temperatures if a sufficiently high PO_2 is used. Run 15 checks the value of an intermediate PO_2 of 800 psia. The results of this run were favorable, so 800 psia was selected for the process design and future laboratory runs.

Runs 16 through 20 evaluated the effects of process temperature at the selected solids concentration and at the selected PO_2 and percent excess oxygen. Conditions and results of the runs are presented in Table 2 and are plotted in Figure 3.

These data substantiate our previous observation that temperatures above 550 degrees are not needed. Run 16 was repeated (Run 21) because the results did not check with previous data. Run 21 does correlate with the previous data and fits the data curve obtained from Runs 17 through 20. Later data also substantiates this conclusion. A process temperature of 550°F was selected, based on these results.

Time was the next variable to be investigated. Based on some initial runs, a process time at temperature of 1-1/2 hr. was tentatively selected for the laboratory tests. It was always assumed up to this point that 1-1/2 hour was a much longer time than would eventually be required for the prototype design. This assumption was based on commercial wet oxidation experience and reported data in Reference 1. Runs 21, 22, and 23 investigated the effects of 1/2, 1, and 1-1/2 hr and resulted in corresponding reduction in COD of 84.5, 91.2, and 99.1 percent, respectively. The results were surprising, indicating that 1-1/2 hr. was a good selection. A check of commercial operations led to the conclusion that short reaction periods could be used because generally a high degree of oxidation is not desired due to cost considerations. Commercial plants generally are interested in 30 to 70 percent reduction in COD. Reference 1 data runs with feces and urine were maintained at temperature for only 15 minutes, but a four-hour warm-up time was used. For the test runs presented in this report, warm-up time to temperature was generally 45 to 50 min, and this difference, plus the higher degree of oxidation achieved, might account for the longer time required. A reaction time of 1-1/2 hr. was selected for the prototype design.

Having selected all reaction conditions for the prototype design, a proof test was run and a detail analysis of the feed slurry, effluent water, filtered effluent water, head space gas, and precipitate was made. The filtered effluent water was also distilled, condensed, and analyzed to establish the difficulty of reclaiming water from the reaction by dry boiling. Table 3 presents the results of the chemical analysis for the proof run. Reaction conditions were 6.5 percent solids, 550°F, 1.2 gm of oxygen per gm of solids, 800 psia initial oxygen charge pressure, 460 cc slurry volume, and 1-1/2 hr at temperature. COD reduction was 99.2 percent. The effluent water was clear and colorless after allowing the white to light grey precipitate to settle. Although the head space

Table 2 Effects of Temperature on Wet Oxidation of Fecal/Urine Slurries

Run No.	% Solids	Temp.(°F)	O_2 Ratio (gmO_2/gm Solids)	Initial O_2 Change Pressure	Slurry VOl (cc)	% COD Reduction
16	7.3	550	1.2	800	460	97.2
17	7.3	530	1.2	800	460	98.5
18	7.3	490	1.2	800	460	97.4
19	6.5	450	1.2	800	460	81.2
20	6.5	625	1.2	800	460	99.2
21	6.5	550	1.2	800	460	99.1

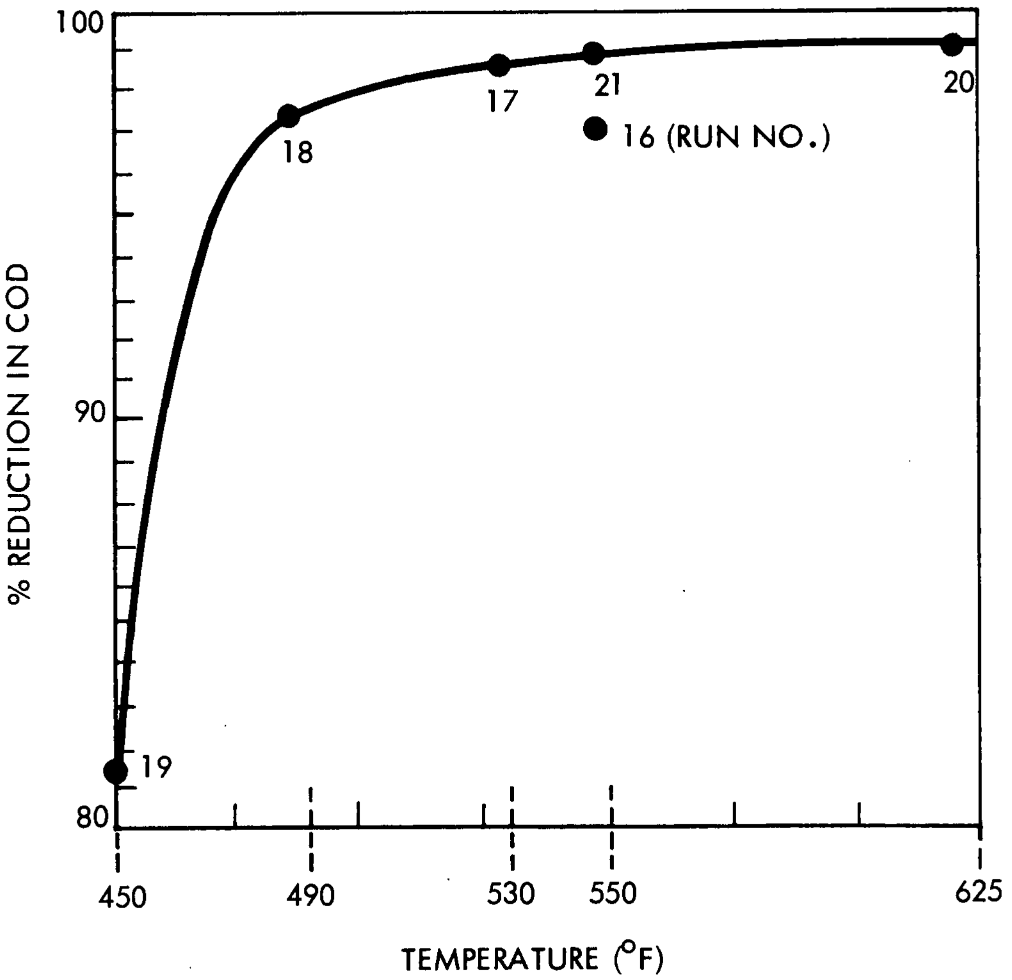


Fig. 3 Effects of Temperature on COD Reduction for Wet Oxidation of Fecal/Urine Slurries

Table 3

PROOF-TEST RESULTS

Measurement	Result	Comment/Method
A. Feed Slurry		
Chemical Oxygen Demand	55.8 mgO_2/gm slurry	Dichromate digestion
Total Solids	6.5%	Dry to constant wt. @ 100°C
Total Nitrogen	13.8 mgN_2/gm slurry	
B. Effluent Slurry		
Chemical Oxygen Demand	0.44 mgO_2/gm slurry	99.2% COD Reduction
Total Suspended Solids	0.18%	Filtration
C. Filtered Effluent		
Water Soluble Solids	1.7%	Dry 1 hour @ 100C
Ashed Solids	1.4%	Dry 1 hour @ 550C
pH	8.4	
Total Alkalinity	11.0 mg/ml as $CaCO_3$	
Conductivity	Infinite	
Total Nitrogen	4.6 mgN_2/gm	
Ammonia Nitrogen	3.6 mgN_2/gm	
Organic Nitrogen	1.0 mgN_2/gm	
Dissolved Ions		
Ammonia	0.55%	
Sodium	0.29%	
Potassium	0.16%	
Calcium	0.00024%	
Magnesium	0.00017%	
Chloride	0.75%	
Sulfate	0.11%	
Phosphate	0.045%	
Acetate	0.30%	

Table 3 (Cont.)

Measurement	Result	Comment/Method
Carbonate	0.05%	
Acetamide		
Pyridine Derivatives		
Silicon		
Copper		
Chromium		
Dissolved Solids Analysis by Emission Spectrograph		
Sodium	Major	Greater than 10%
Potassium	Major	
Silicon	Minor	0.1 to 10%
Magnesium	Minor	
Calcium	Minor	
Phosphorous	Minor	
Copper	Trace	Less than 0.1%
Chromium	Trace	
Headspace Gas Analysis		
Carbon dioxide	37%	
Oxygen	48%	
Nitrogen	9.6%	
Carbon Monoxide	0.2%	
Ammonia	5 ppm	
Oxides of Nitrogen	None Detected	1 ppm LLD*
Oxides of Sulphur	None Detected	1 ppm LLD*
Hydrogen Sulphide	None Detected	1 ppm LLD*
Total Hydrocarbons (including methane)	132 ppm	Based on methane std.
Methane	84 ppm	
Propylene	5 ppm	
Ethane	1 ppm	
Hydrogen	None Detected	250 ppm LLD*
Organic Acids C_1 thru C_4	None Detected	75 ppm LLD*
Acetone	None Detected	5 ppm LLD*
Formaldehyde	None Detected	50 ppm LLD*
Pyridine	None Detected	5 ppm LLD*

---

*LLD = Lower Limit of Detectability

Table 4 Catalysts in Wet Oxidation

Type of Catalyst	Catalyst Containment	Slurry Agitation	Condition of Effluent Water	Condition of Catalyst & Substrate
3% Cobalt-15% Molybdenate Oxides on Alumina	In Liquid	Stirred	A thick light blue paste in blue solution.	Almost complete destruction of catalyst and substrate.
Mixed Metal Oxide	In Liquid	Stirred	A deep blue clear liquid with large amounts of thick dark precipitate.	Almost complete destruction of catalyst and substrate.
Mixed Metal Oxide	In Liquid	Unstirred	A deep blue clear liquid with moderate amount of dark grey precipitate.	Significant quantity of catalysts and substrate erroded.
19% Manganese Dioxide on Alumina	In Liquid	Unstirred	A light green clear liquid with small amount of dark grey precipitate.	Substrate in good condition, significant quantity of catalyst erroded.
5% Nickel on High Fired Alumina	Retain in a Stainless Screen Basket	Stirred	A pale yellow clear liquid with small amount* of light grey precipitate.	Catalyst and substrate in good condition.
0.5% Ruthenium on Alumina	Retain in a Stainless Screen Basket	Stirred	A pale yellow clear liquid with small amount* of light grey precipitate.	Substrate in good condition, catalyst erroded.
Platinum on corregated high fired ceramic	Honeycomb substrate wired to reactor coiling coil.	Stirred	A pale yellow clear liquid with small amount* of light grey precipitate.	Catalyst and substrate in good condition.

* Amount and color of precipitate similar to uncatalyzed runs.

Preceding page blank

Table 5 Liquid-Phase Catalyst Experimental Results

	Catalyst			
	Nickel	Ruthenium	Platinum	Uncatalyzed
Initial COD (mg/gm)	60.9	60.9	60.9	55.8
Effluent COD (mg/gm)	2.1	2.7	3.9	.44
COD Reduction	97%	96%	93%	99%
Effluent Liquid Ammonia (mg/gm)	2.3	2.2	3.5	5.5
Effluent Liquid pH	8.8	7.2	8.8	8.4
Head Space Gas Analysis (Post Run)				
Oxygen (% by volume)	44.2	34.4	40.3	48.3
CO_2 (% by volume)	26.7	Not Avail.	31.	37.
CO (ppm)	1135.	560.	1125	2000.
Methane (ppm)	43	80	50	84
Ammonia (ppm)	Not detected (1 ppm detection limit)			5
Nitrogen Oxides	Not detected (1 ppm detection limit)			

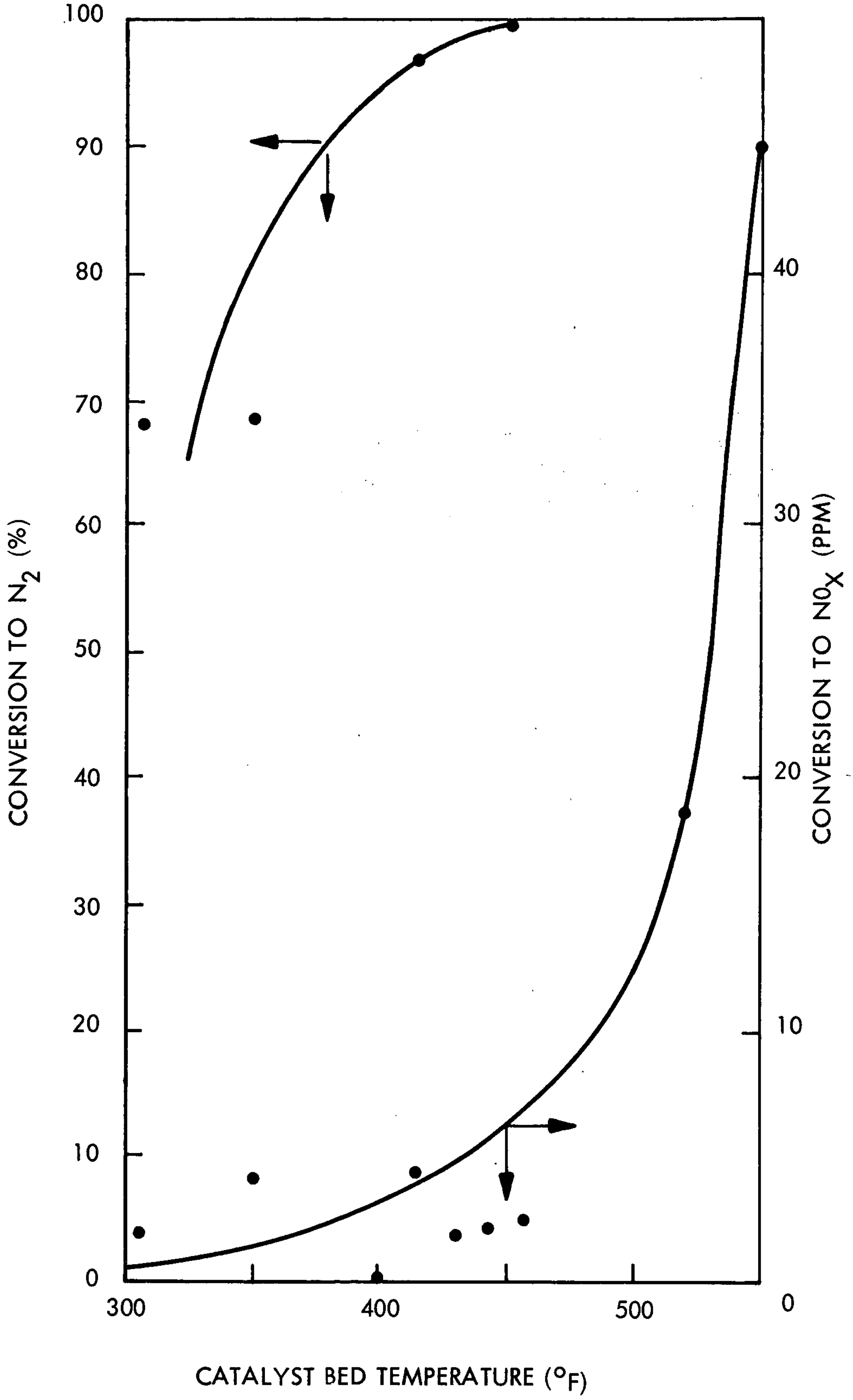


Fig. 4 Plot of Ammonia Conversion for Vapor Phase Catalyst

Fig. 5 Effluent Water and Ash from Proof Test and Mixed Solids Test Runs

PROTOTYPE SYSTEM DESIGN

Early in the program, the prototype system was envisioned as containing a slurry pumping system, oxygen flow control components, a heated pressurized reactor, venting controls, and a salt removal device. The slurry pump, reactor, and water recovery device represented significantly new design problems which will be discussed separately in the following paragraphs.

Slurry Pump

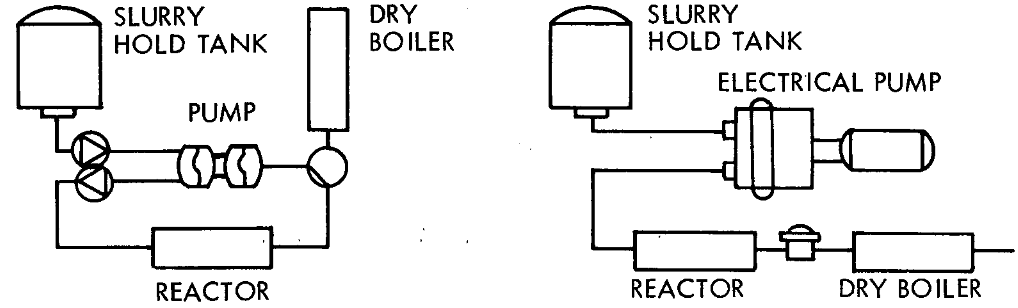
After an extensive vendor survey covering a wide variety of pumping approaches, it was concluded that a special slurry pump must be developed for the spacecraft application. The problem stemmed from the requirements for a very low flowrate (5 cc/m design flow), pumping of a slurry mixture, and high delivery pressure - in the range of 2200 to 3000 psi. The pump was also required to act as a flow control device metering the slurry into the reactor to avoid the complications of slurry flow controls. Numerous pumping approaches were considered and the most attractive ones are schematically shown by Figure 6.

The first concept presented the lowest weight and power penalty by employing a boot strap slurry pump that used reactor effluent liquid and gases to operate a double-ended diaphragm or piston pump. The volume of reactor effluent gases and liquid are greater than the slurry pumped because of the oxygen bled into the reactor from the high pressure accumulator. This difference meant the pump must have a volumetric efficiency of 72 percent or greater, which was achievable. The boot strap pump involved the greatest pump development, requiring a pump that handled slurry through check valves, pumping from cabin pressure to 2200 psi, and using effluent liquid and gases as a power source.

The second concept eliminated the need for developing a pump that uses reactor effluent fluids by employing an electrical motor as a power source. Power penalties are higher and the design problems of pumping slurry against full reactor pressure are still present.

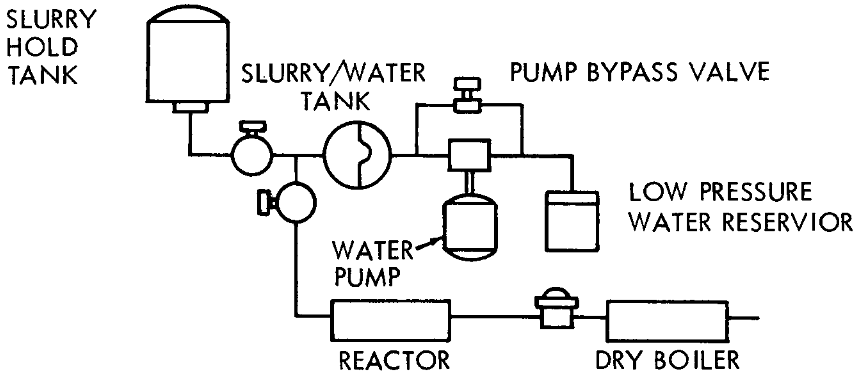
The third concept was similar to the second except that a full head rise water pump was used to force water at a constant rate to the back side of a bladdered tank, thereby forcing slurry into the reactor. This approach eliminated the need to pump slurry, but still required a very low flow, high pressure water pump.

The fourth concept reduced pump head rise to a minimum by using reactor effluent fluids to pressurize slurry feed tanks. The pump must be designed to handle slurry but need only provide in the range of 0 to 10 psi head rise. The weight and complexity of the system increased, however.

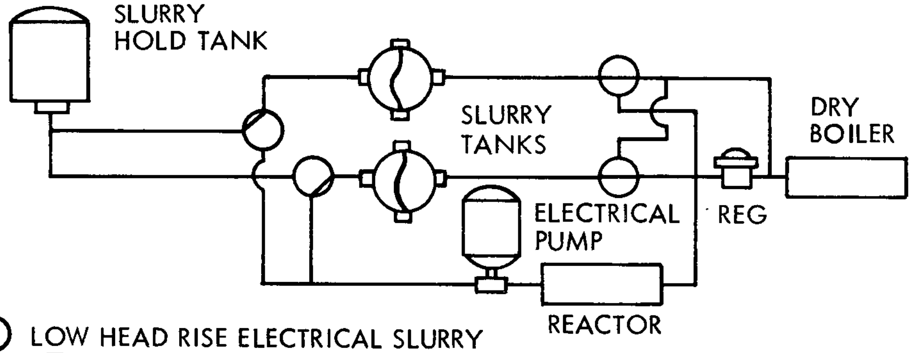


① BOOTSTRAP SLURRY PUMP

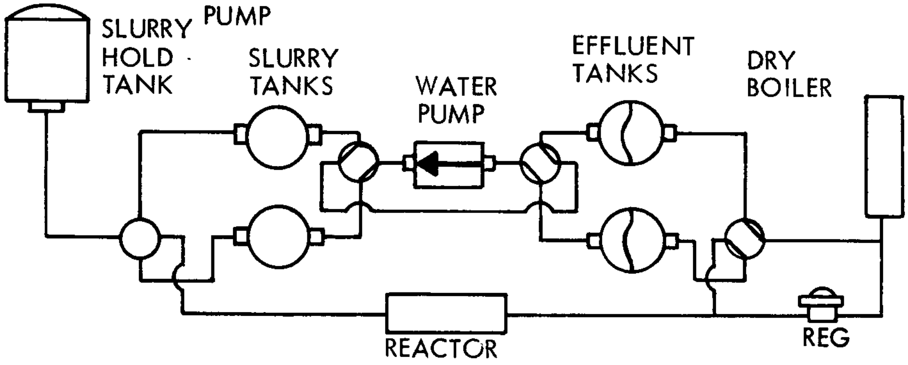
② FULL HEAD RISE
ELECTRICAL SLURRY PUMP



③ FULL HEAD RISE, ELECTRICAL WATER PUMP



④ LOW HEAD RISE ELECTRICAL SLURRY PUMP



⑤ LOW HEAD RISE ELECTRICAL WATER PUMP

Fig. 6 Slurry Pumping Approaches

Concept five added two effluent fluid tanks and placed the pump between them and the slurry feed tanks. The pump was designed for low head rise and for water or oil service. The system complexity and weight was increased even more by this approach.

In addition to the pumping arrangement, pump types were also studied. Centrifugal, gear, lobe, and screw pump designs were eliminated primarily because of the lack of ability to deliver a very low, known, continuous flow of liquid under varying delivery pressures. At the very low flow rates (5 cc/min) bypass flow around the gears, lobes, or screws became a very significant factor, making constant flow delivery impossible.

Three types of pumps emerged as holding the greatest promise: (1) a free-floating piston pump with slide valves on each end (similar to a steam locomotive valve) that utilized effluent liquid and gases to pump liquid slurry into the reactor (as depicted in Concept 1, Figure 6), (2) a conventional very low flow (5 cc/min) high pressure (2200 psi) water pump (as depicted in Concept 3, Figure 6), and (3) a peristaltic pump with a case designed for full system pressure, but that developed a small head for circulation of fluid through the system (as depicted in Concept 4, Figure 6).

The original contract did not provide sufficient time or funds to support the level of effort required to develop a slurry pump for the initial prototype. Therefore, it was decided to use high pressure nitrogen gas metered to the back side of a bladdered tank as a means of forcing a known flow of slurry into the reactor for the initial prototype and to develop a slurry pump in subsequent efforts.

Reactor

Early in the contract effort, reactor conceptual design and modeling studies were initiated. After consideration of a variety of continuous flow, batch, static, mechanically stirred, and recirculation reactor designs, three concepts were selected for modeling.

1) Static mixer - A 1/4" I.D. tube with reversing butterfly mixer installed as shown by Figure 7. Gas and slurry are forced through the tube and the reversing butterfly mixer forced liquid and gas to reverse direction every half inch down the tube. The tube would be heated to 550°F and sufficient tube length would be provided to achieve a 1-1/2 hour residence time in the reactor.

2) Corkscrew mixer - A thin plate corkscrew passage produced by two concentric tubes leaving a 1/8" gap with a metal rod wrapped in thread fashion down the gap to force the liquid and gas to spiral down the tube. The tube would be heated and sufficient tube length would be provided to achieve a 1-1/2 hour residence time in the reactor.

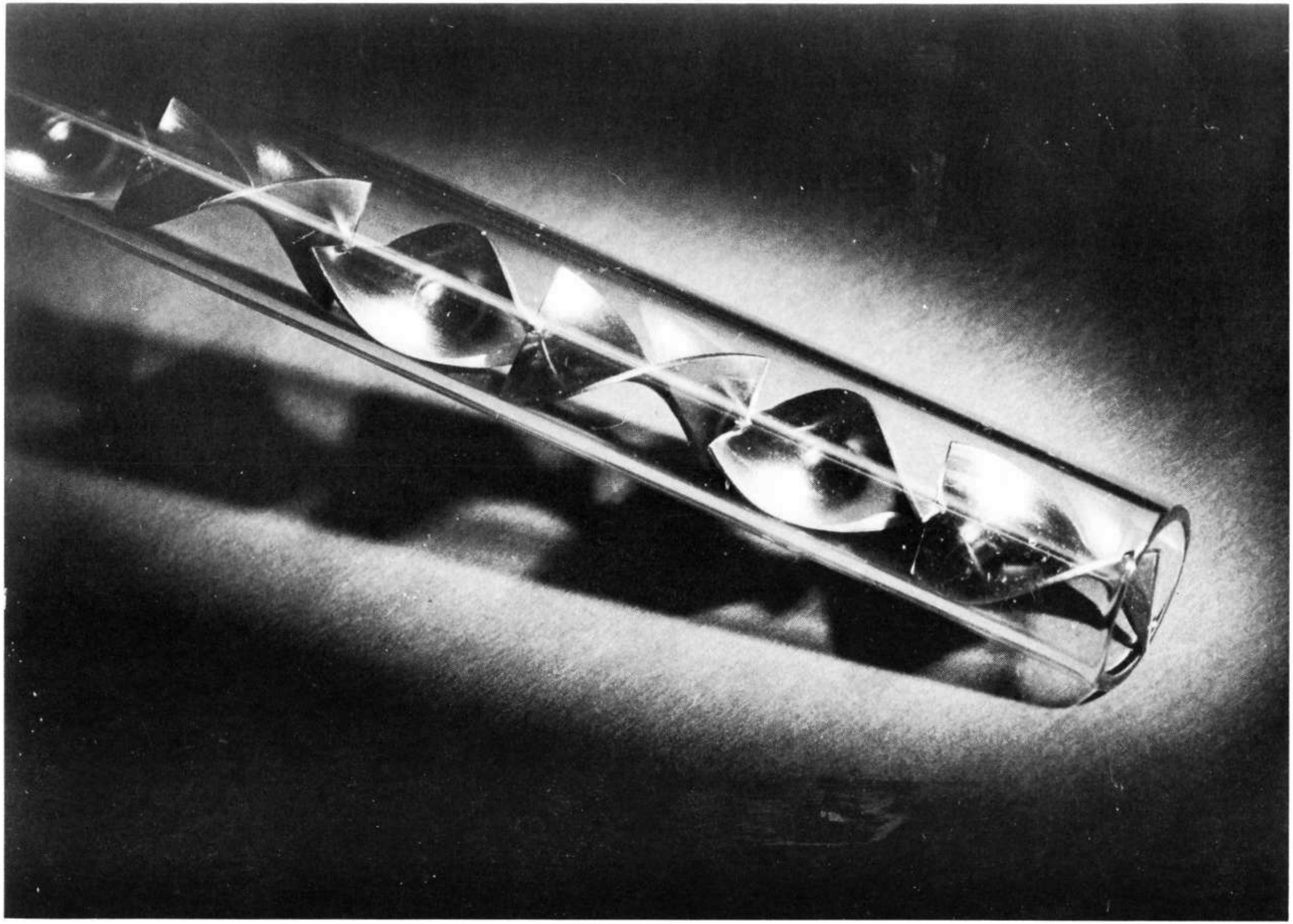


Fig. 7 Static Mixer Reactor Model

3) Mechanically stirred reactor - A reactor body divided into a series of chambers to prevent mixing of influent and effluent with several blades and baffles in each chamber to agitate the liquid/gas mixture. A magnetic coupling and external motor drive the internal stirring shaft. This design was adapted from the vertical, batch reactor used in laboratory test program. Figure 8 is a photograph of the plastic model used to evaluate this reactor design concept.

Tests were run on the three reactor design models to assess the degree of mixing achieved by each. Both static mixer and corkscrew mixer performance was very poor. The extremely low flowrates of liquid and gas (5.5 cc/min and 2.1 cc/min respectively) resulted in very little agitation of the liquid and gas phases. Bubbles of liquid and gas passed through both units without ever being broken up. The performance of the mechanically stirred reactor was excellent. Mixing was good and by introduction of dye, it was determined that little, if any, back flow from chamber to chamber was occurring. Based on these results, it was decided to proceed with the detailed design of a motor driven, magnetic coupling, stirred reactor vessel.

Figure 9 presents an assembly drawing of the reactor designed for the initial prototype system. This design was based on the results of the laboratory program and the following calculated values:

Reactor Operating Conditions

Temperature	550°F
Total Pressure	2200 psi
Water Vapor Pressure	1050 psi
Oxygen Requirement	1.2 gm O_2/gm solids
Solids in Feed	7% by weight
Process Reaction Time	1.5 hours

Reactor Design Conditions

Maximum operating pressure to allow room for experimentation	4000 psi
Burst pressure to meet laboratory test safety requirements	12000 psi
Maximum operating temperature to allow room for experimentation	625°F

Reactor Feed Rates

Assuming a 4-man capacity processing urine and fecal slurry with a 25 percent design margin, 3.2 pounds per man day of urine production at 5 percent solids content and 0.35 pounds per man day of fecal production at 25 percent solids content:

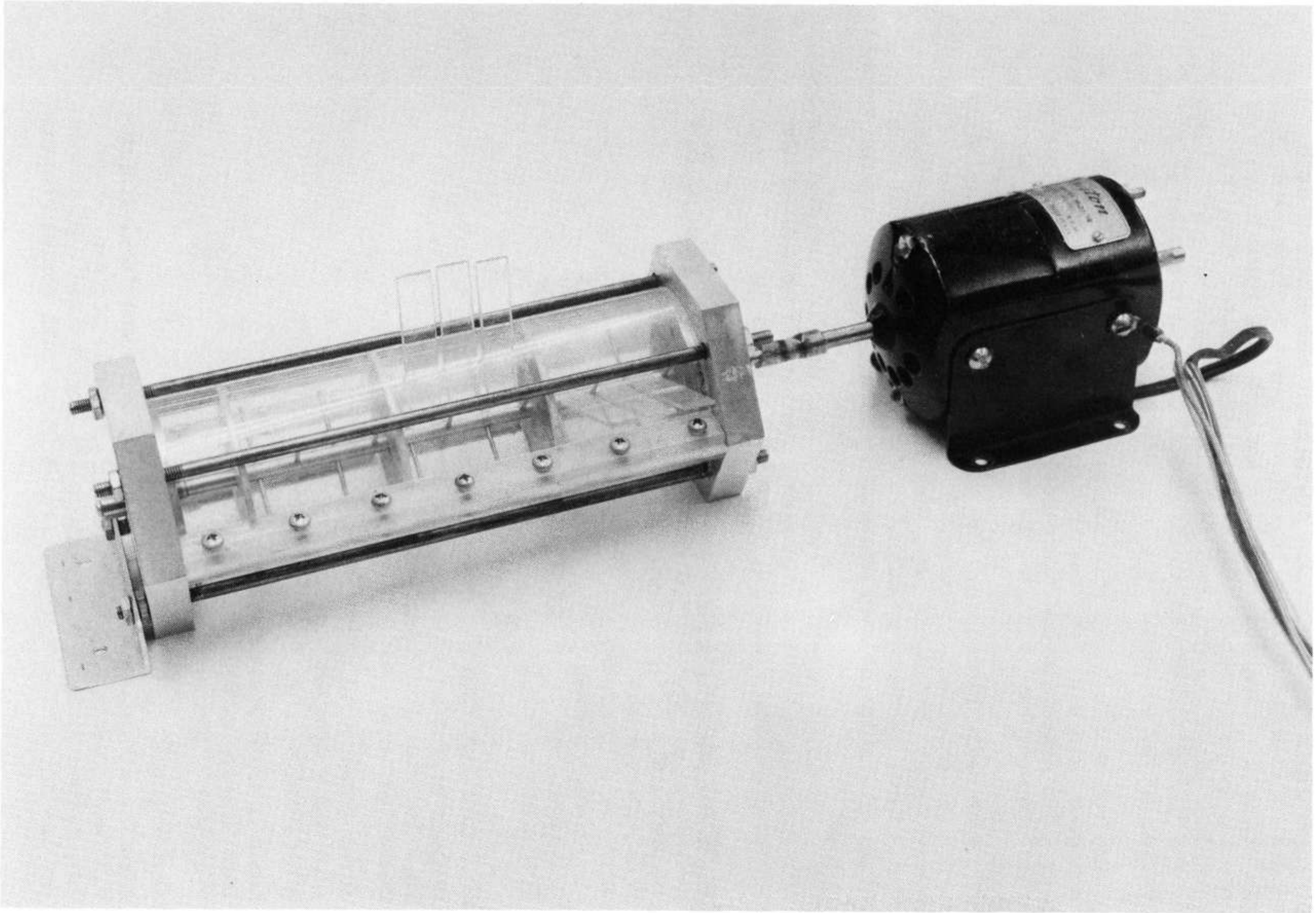


Fig. 8 Stirred Reactor Model

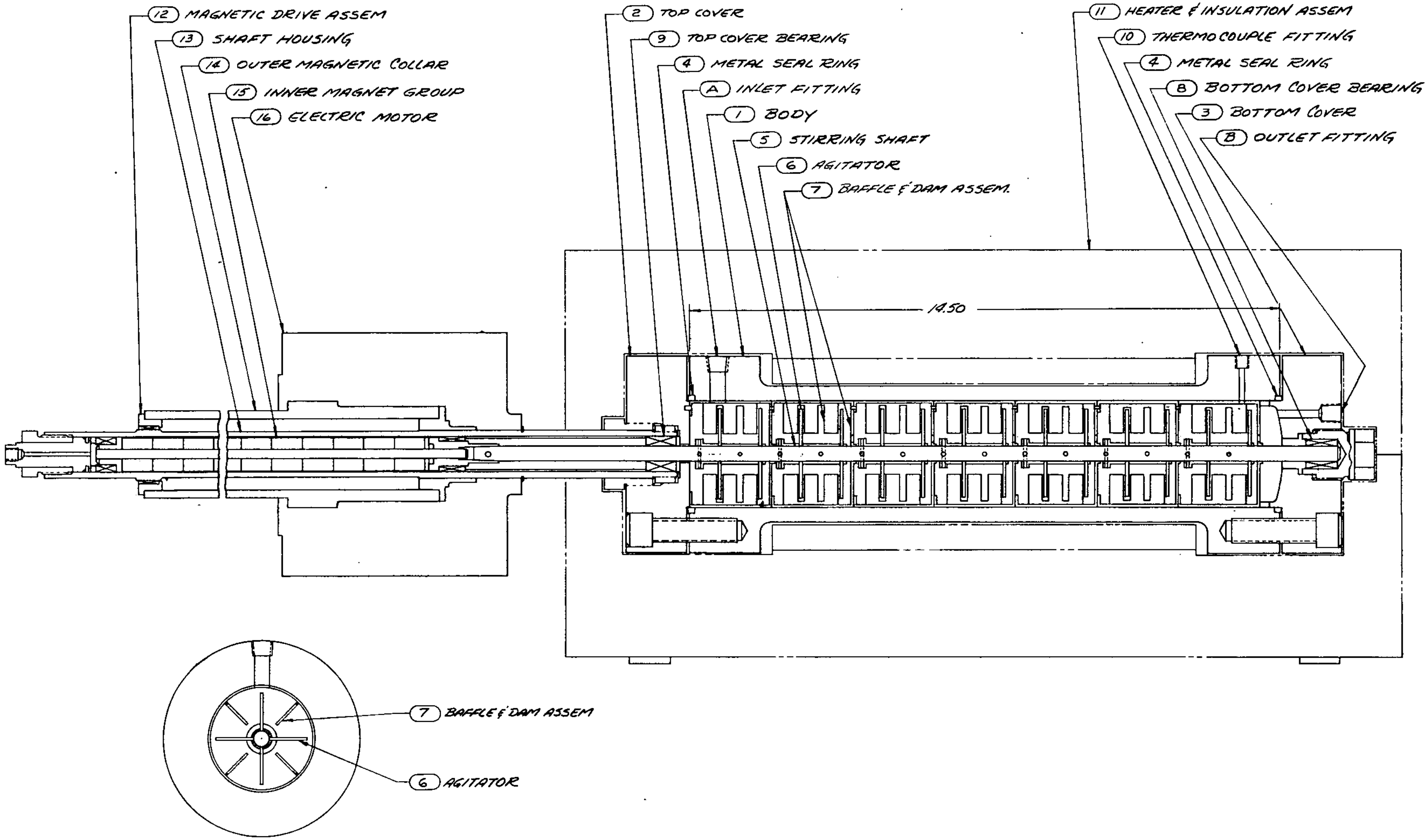


Fig. 9 Assembly Drawing of Stirred Reactor

Urine Feed Rate $4(1.25)(3.2) = 16$ lb/day

Fecal Feed Rate $4(1.25)(0.35) = 1.8$ lb/day

Combined Feed Rate $\frac{17.8(454)}{24(1.01)} = 330$ cc/hr

Oxygen Feed Rate

$$\frac{1.2(454)}{24} \quad 16(0.5) + 0.25\ (1.8) \quad = \quad 28.4 \text{ gm/hr}$$

Oxygen Feed Volume at 2200 psig = 550°F

$$\frac{0.97(28.4)(48.3)(1010)}{454(2215)(144)\ \ 28,200} = 262 \text{ cc/hr}$$

Oxygen Feed Volume at 2200 psig = 75°F

$$262\left(\frac{535}{1010}\right) = 139 \text{ cc/hr}$$

Reactor Volume (Based on laboratory test data)

Laboratory Reactor Volume 1060 cc

Laboratory Reactor Slurry Charge 460 cc

Prototype Reactor Slurry Charge $1.5\ (330) = 495$ cc

Prototype Reactor Volume $1060\left(\frac{495}{460}\right) = 1140$ cc

Prototype Reactor Liquid Volume at 550°F

495 cc of slurry charge at 75°F

at 550°F $495\left(\frac{.0218}{.01606}\right) - 24 = 647$ cc

Expansion due to temp

Evaporated Water

Prototype Reactor Gas Volume

$$1140 - 647 = 493 \text{ cc}$$

Reactor Effluent Rates

Based on laboratory test data, liquid and gas effluent rates are nearly identical to liquid and gas feed rates on a weight basis. Therefore, assuming equal density of liquid:

Effluent Liquid Volume = 330 cc/hr

Effluent Gas Weight = 28.4 gm/hr

Effluent Gas Volume at a calculated gas constant of

41.5 and 2200 psig and 75°F

$$\frac{28.4(41.5)\ 535(28,200)}{454\ (144)\ 2200} = 127 \text{ cc/hr}$$

Referring to Figure 9, the reactor consisted of a body(1); a top cover (2), and bottom cover (3) with metal seal rings (4) for vessel closure; internal stirring shaft (5) with attached agitators (6) for stirring the slurry/oxygen mixture; baffle and dam chambers(7) to assist in agitation of the contents of the reactor and to minimize end flow from chamber to chamber; bottom cover bearing (8) and top cover bearing (9) to support the stirring shaft; thermocouple (10) for reactor temperature control; heater and insulation assembly (11) to provide support for the reactor, heating of the body and to minimize heat loss from the reactor; and magnetic drive assembly (12) to rotate the stirring shaft without requiring a dynamic shaft seal.

The magnetic drive assembly consisted of the shaft housing (13); outer magnetic collar (14); inner magnet group (15); electric motor (16) built around the outer magnet collar; and miscellaneous bearings, spacers, and seals.

In operation, the slurry/oxygen mixture entered the first chamber of the hot reactor through the inlet fitting (A). It slowly passed through the seven chambers in the reactor as fresh slurry was pumped in and oxidation products were vented. The liquid and gas effluents vented through outlet fitting (B) in the reactor bottom cover. The slurry/oxygen mixture in all seven chambers was stirred to promote oxidation by the stirring shaft and baffle systems. Flow from chamber to chamber passed through small holes in the dams across the reactor body as well as through clearance holes between the dams and the stirring shaft. The magnetic drive operated continuously to rotate the stirring shaft at 1200 RPM.

Dry Boiler

The concept of the dry boiler was evolved from the indication during the laboratory test program that if the wet oxidation reactor effluent was boiled to dryness, little, if any, carryover of organics or salts would occur. This meant that salt removal and 100% recovery of the water would be possible in a single step. It was envisioned that the salty water from the wet oxidation reactor would be sprayed on a hot surface to immediately produce water vapor, leaving the salts adhering to a replaceable boiler extented surface.

Figure 10 presents an assembly drawing of the dry boiler. It consisted of the boiler body (1); replaceable extented surface boiler chamber (2) for water boiling and salt and ash collection; a replaceable boiler chamber cover (3) with membrane (4) to block the passage of any unboiled water, salt or ash; a water inlet tube (5) with deflector (6) to direct the incoming water to the hot boiler surfaces; a hot plate (7) with attached heaters (8) to transfer heat to the replaceable boiler chamber; a boiler cover (9); steam outlet (10); outer cover (11) to support the boiler and insulation (12); and miscellaneous brackets, supports, and connectors. The volume of the replaceable boiler chamber was calculated to hold a 30-day supply of salt and ash, assuming complete utilization of the chamber to the top of fins. Pallflex Products teflon on fiberglas membrane TV201960 was used in the dry boiler.

System Description

Based on the laboratory test program results, and the reactor, slurry pump, and dry boiler design studies, a prototype system design was established. Figure 11 presents a schematic of the initial prototype system. Gaseous oxygen at a maximum pressure of 2900 psi bled from a high pressure storage tank was supplied to the wet oxidation reactor at a metered rate by a pressure regulator and flow control solenoid valve. The regulator maintained a constant pressure of 2440 psig on the upstream port of the three-way solenoid valve. One leg of the three-way solenoid was connected to a closed tube of known volume (26 cc) which was alternately filled by oxygen flow from the regulator and then emptied to the reactor which was at a lower pressure of 2200 psi. A timer energized the three-way solenoid valve for forty-five seconds and then de-energized it for forty-five seconds to provide an oxygen flow rate of 28.4 gm/hr. Oxygen tank pressure and regulated oxygen pressure gages were provided as well as oxygen supply shutoff and system bleed valves. An oxygen relief valve set at 2500 psig was located in the line between the solenoid valve and reactor fitting.

Fecal/urine slurry was supplied to the slurry feed tanks from a hold tank pressurized to 20 psig. Four solenoid valves connected to the high pressure slurry feed tanks allowed filling of the tanks, isolation from the

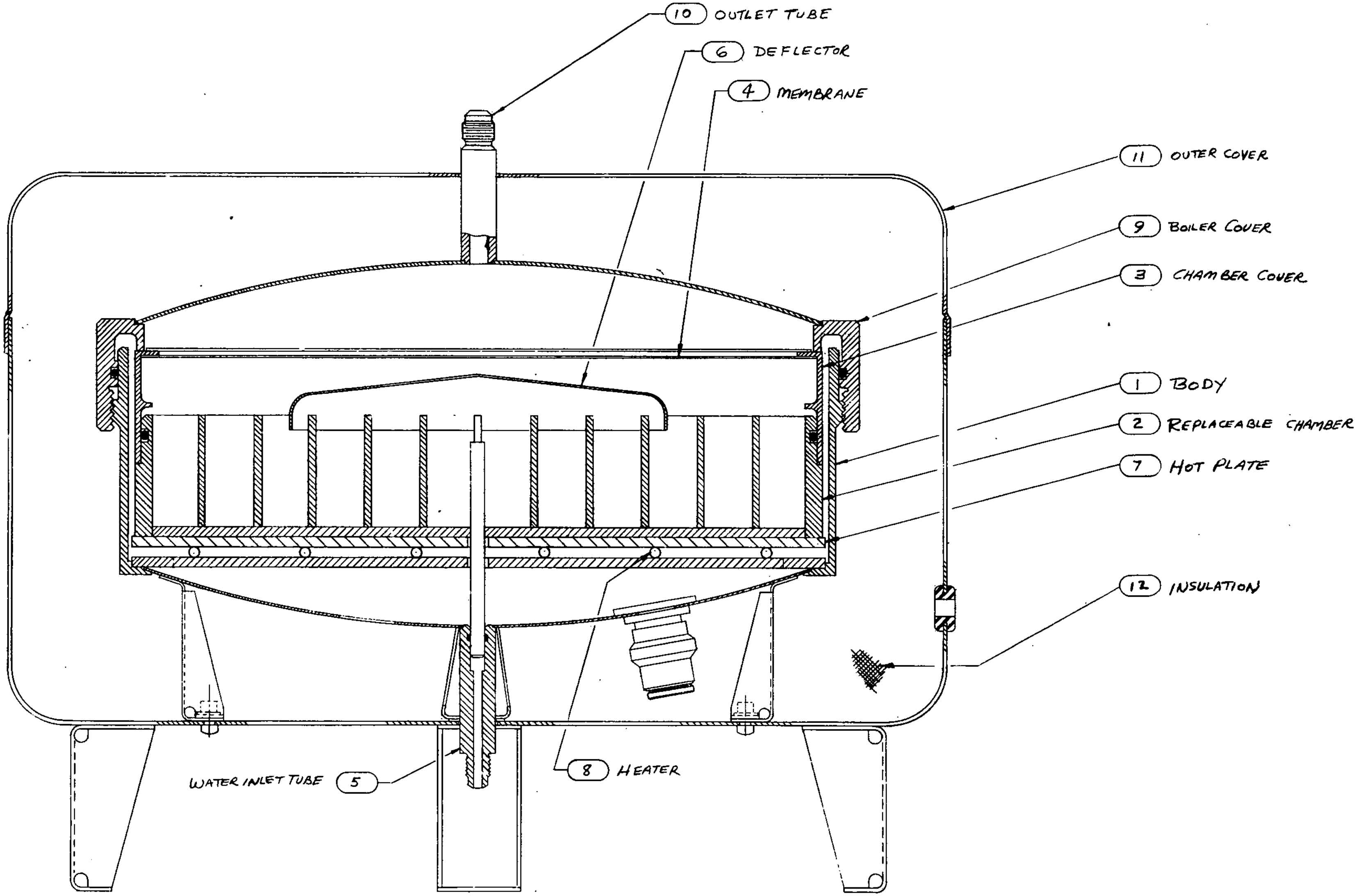


Fig. 10 Assembly Drawing of Dry Boiler

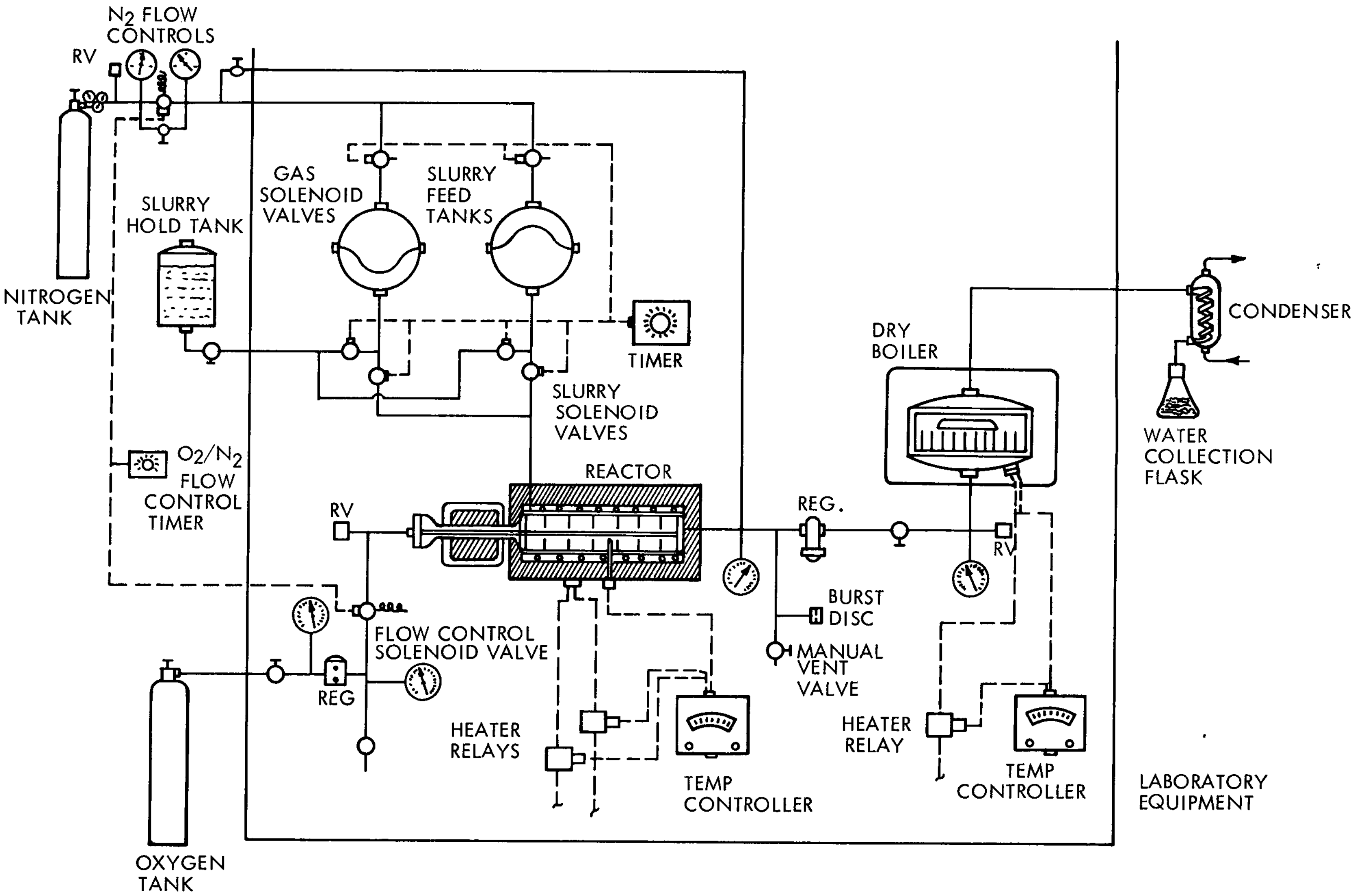


Fig. 11 Initial Prototype Spacecraft Wet Oxidation System Schematic

reactor or hold tank, and for emptying of the tanks into the reactor. Two three-way solenoid valves, one for each slurry feed tank, controlled the flow of nitrogen for feed tank pressurization. Nitrogen metered to the feed tanks in an identical manner as the oxygen was metered to the reactor, was used to force slurry at a known flowrate from the slurry feed tanks into the reactor. The nitrogen supply tank was charged to 6000 psi, and the nitrogen regulator delivered 2570 psig gas to the closed tube whose volume was 23 cc. A bypass valve around the nitrogen three-way solenoid valve allowed the slurry feed tanks to be pressurized to reactor pressure after filling from the slurry hold tank. Sequencing of the six solenoid valves surrounding the slurry feed tanks was controlled by a 24-hour timer. A two day control cycle is illustrated by Figure 12.

Slurry and oxygen entering the reactor mixed and passed through the reactor chambers, producing salty effluent water, essentially free of organics, a white phosphate ash suspended in the water, and product gases primarily comprised of CO_2, O_2, N_2 and water vapor. The reactor was initially brought to 550°F in 50 minutes by two heaters connected in parallel to supply 1550 watts of power. The heaters were automatically connected in series to provide 380 watts for sustaining temperature. The effluent mixture leaving the reactor passed through a cooling coil and back pressure regulator which maintained the reactor at 2200 psig. The back pressure regulator discharged the mixture into the dry boiler. A manual vent valve, rupture disc and reactor pressure gage were connected to the reactor outlet.

The water/ash/gas mixture entering the dry boiler struck the deflector plate which directed the solids down against the hot boiler surfaces. The water turned to steam and with the gases passed around the deflector, through the membrane and outlet fitting to the condenser, where processed water and gases were collected separately for analysis. The ash and salt remained in the boiler adhering to the hot boiler surfaces. The hot boiler surfaces were maintained at 250°F by a two-element 350 watt heater, operated by a temperature probe and controller.

Figure 13 presents an electrical schematic for the prototype system. One 25 amp circuit provided power to the reactor heaters through two relays that automatically connected the heaters in series after warmup. A second circuit provided power to the dry boiler temperature controller and heater elements. A third circuit provided power to the six slurry feed solenoid valves through the control timer and manual override switches, to the reactor drive motor, and to the reactor temperature controller and relays.

The prototype system components were arranged on an aluminum support structure as shown by Figure 14. The control panel (Figure 15) located on the front of the support structure housed the gages, switches, temperature controller, slurry valve control timer, reactor drive tachometer, and oxygen shutoff valves. Table 6 presents a parts list of the most significant elements of the initial prototype system.

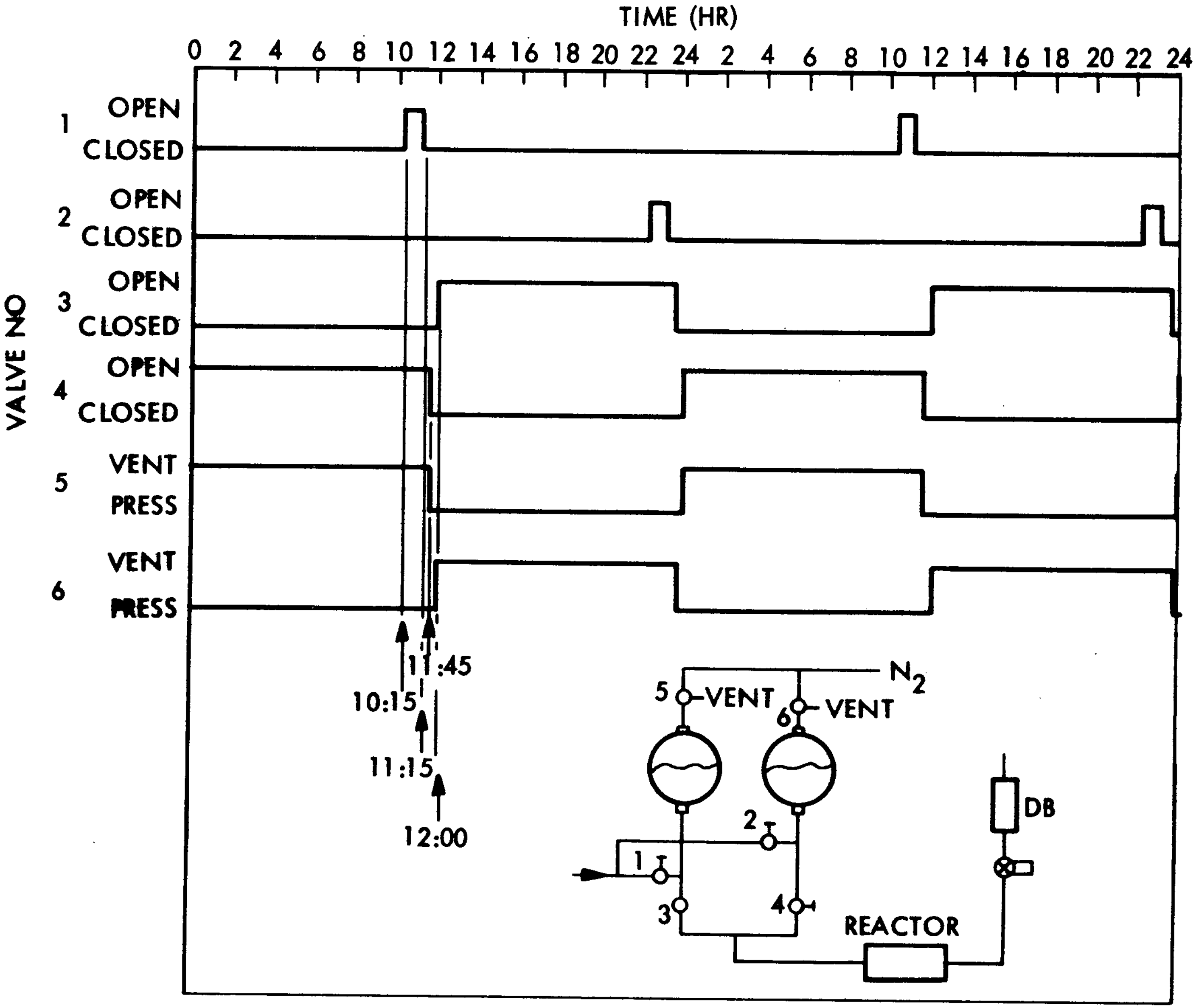


Fig. 12 Slurry Feed Valve Control Cycle

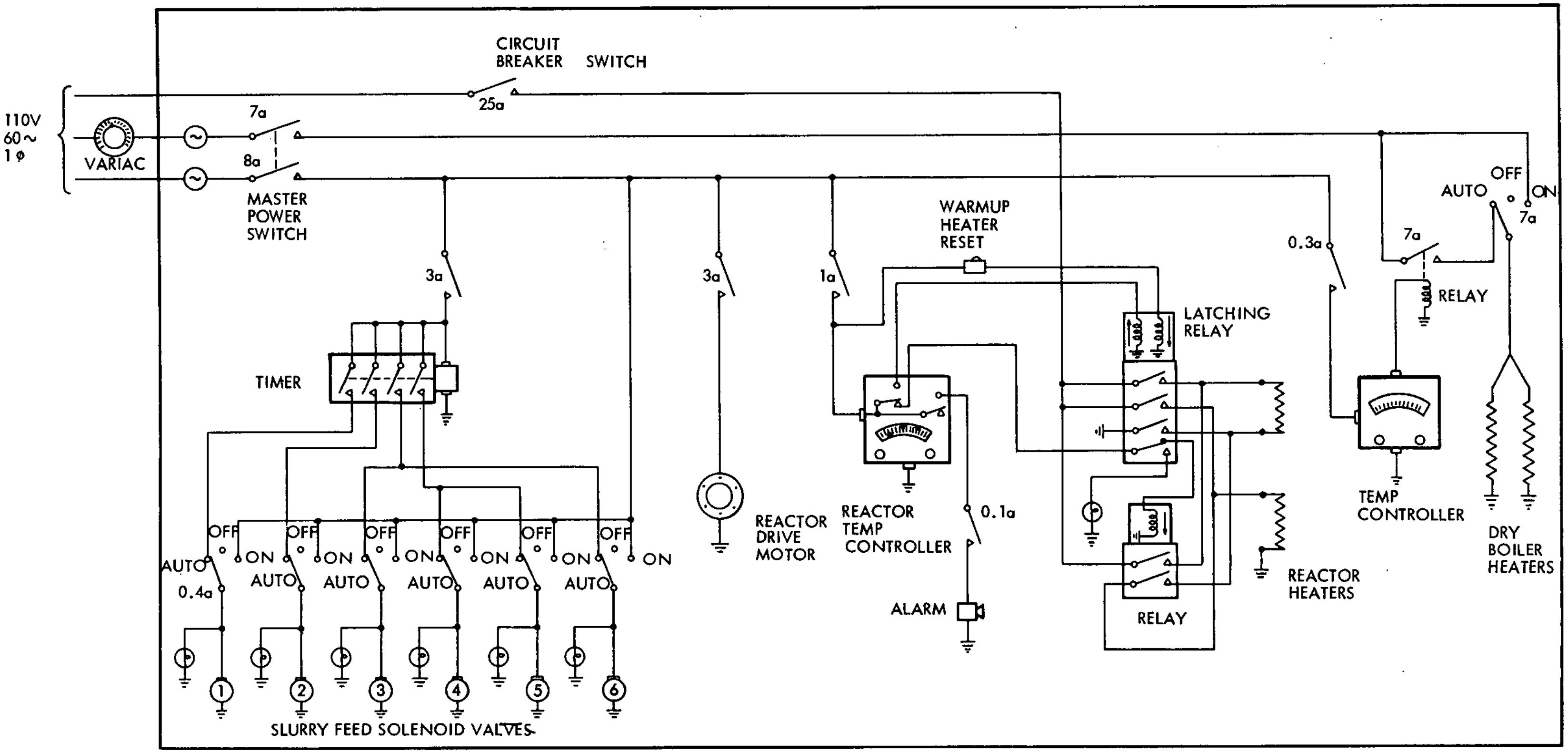


Fig. 13 Initial Prototype Spacecraft Wet Oxidation System Electrical Schematic

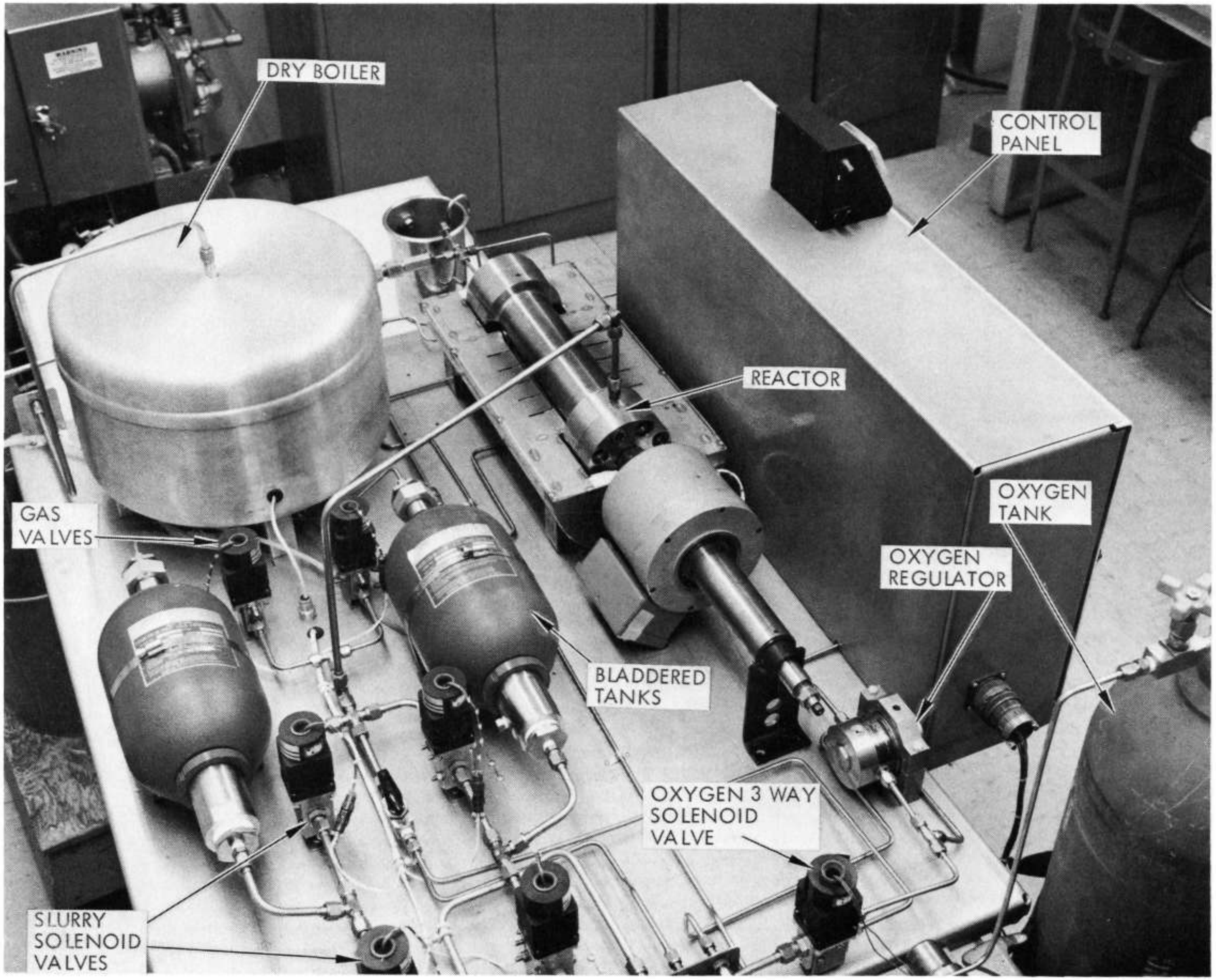


Fig. 14 Initial Prototype Spacecraft Wet Oxidation System Arrangement

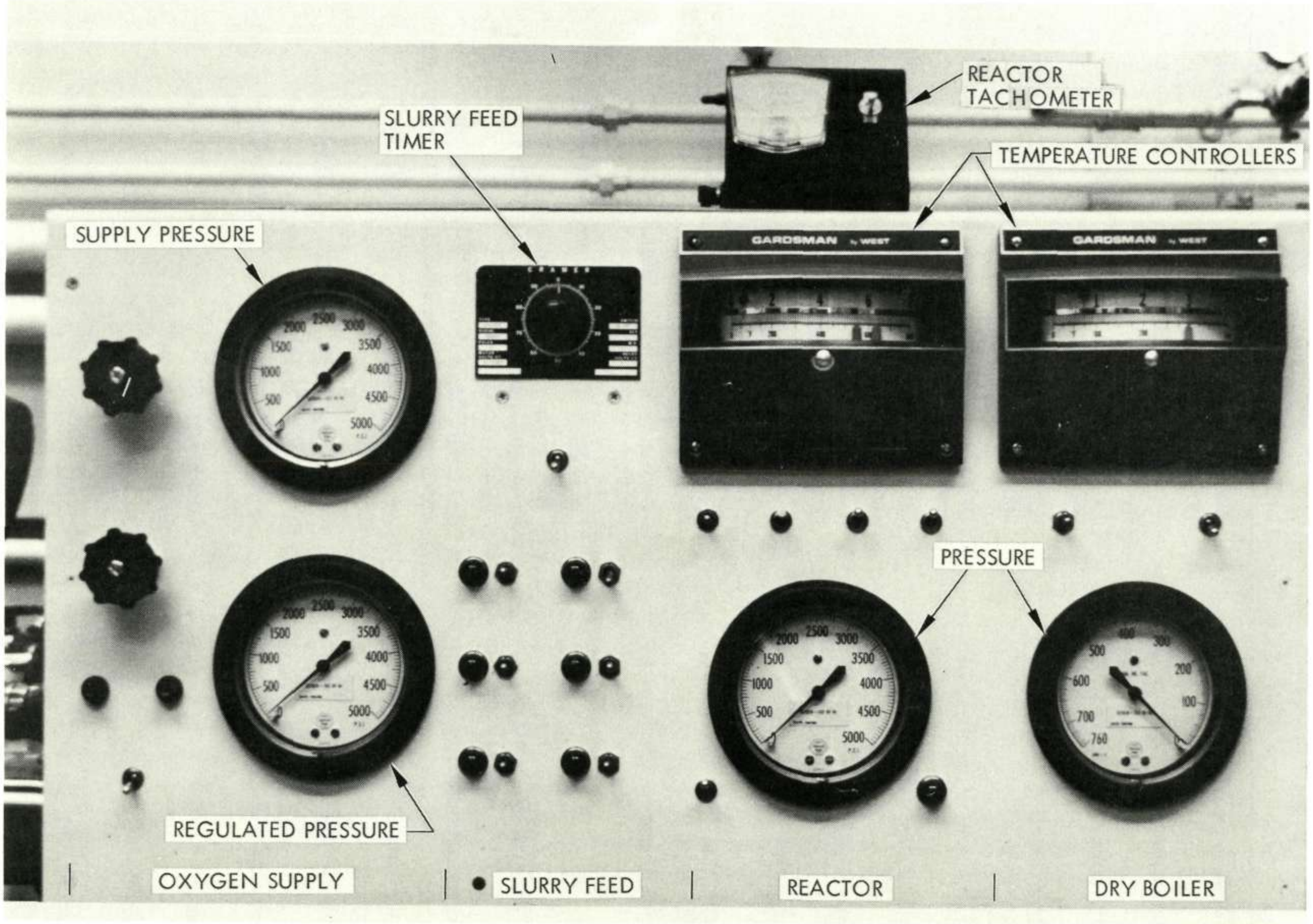


Fig. 15 Initial Prototype Spacecraft Wet Oxidation System Control Panel

TABLE 6
INITIAL PROTOTYPE SPACECRAFT WET OXIDATION PARTS LIST

Part Title	No. Req.	Supplier	Part No.
Oxygen Shutoff Valve	2	Circle Seal Anaheim, Calif.	B933T-4TT(P)
Oxygen Regulator	1	Accessory Products Co. Whittier, Calif.	1381KXTC-4T
Oxygen Pressure Gage	2	Helicoid Gage Bridgeport, Conn.	440-316
Oxygen Flow Control Solenoid Valve	1	Victor Controls Sacramento, Calif.	SV21B32 P4P
Oxygen Feed Control Timer	1	Industrial Timer Corp. Newark, New Jersey	CM 6
Oxygen Relief Valve	1	Victor Equipment Co. Santa Clara, Calif.	0630-3000
Slurry Feed Tank	2	Greer Hydraulics, Inc. Los Angeles, Calif.	A69568-200
Slurry Solenoid Valve	4	Victor Controls Sacramento, Calif.	SV31S2NC6T
Slurry Tank Gas Solenoid Valve (3-way)	2	Victor Controls Sacramento, Calif.	SV21S32P4T
Slurry Feed Control Timer	1	Giannini Controls Corp. Centerbrook, Conn.	540-24H
Reactor	1	Autoclave Engineers Inc. Erie, Pa.	40-42485
Reactor Temperature Cont.	1	West Instrument Co. Schiller Park, Ill.	JPT-3
Reactor Pressure Cage	1	Helicoid Gage Bridgeport, Conn.	440-316 0 to 5000 psi
Reactor Sys. Burst Disc Assembly	1	Autoclave Eng., Inc. Erie, Pa.	SS6600
Back Pressure Regulator	1	Accessory Prod. Co. Whittier, Calif.	1103-20
Dry Boiler Pressure Gage	1	Helicoid Gage Bridgeport, Conn.	440-316-0- 0-760 mmHg Vac
Dry Boiler	1	Lockheed MSC Sunnyvale, Calif.	BE 103-19
Dry Boiler Temp. Cont.	1	West Instrument Co. Schiler Park, Ill.	JP
Dry Boiler Relief Valve	1	Circle Seal Anaheim, Calif.	559T-60

PROTOTYPE SYSTEM TESTS

Tests run on the initial prototype system included slurry and oxygen feed calibration tests, a combined systems checkout test, 100-hour design verification test, and reactor drive RPM tests.

Slurry Feed Calibration Test

Figure 16 presents a schematic of the test setup for the slurry feed calibration test. The test was conducted by:

- Filling the slurry feed tanks, valves, and lines with water.
- Pressurizing one slurry tank with nitrogen to a pressure of 2200 psig.
- Adjusting the nitrogen pressure regulator to 2500 psig, 2600 psig, and 2875 psig, consecutively.
- Starting the three-way solenoid valve timer which operated on a 90-second cycle.
- Bleeding water from the slurry tank through solenoid valve 3 maintaining 2200 psig on the system.
- Measuring the water volume bled from the system over a fifteen minute period.

Table 7 and Figure 17 present the test results.

Table 7

Slurry Feed Calibration Test Data

N_2 Reg Outlet Pressure(psig)	Slurry Tank Pressure(psig)	No. of Solenoid Valve Cycles	Elapsed Time(min)	Measured Water Volume (cc)	Water Rate cc/cycle
2500	2200	10	15	65	6.5
2600	2200	10	15	90	9.0
2875	2200	10	15	133	13.3

Based on a design slurry flow rate of 5.5 cc/min or 8.25 cc/cycle of the timer, a nitrogen regulator supply pressure of 2570 psig was required.

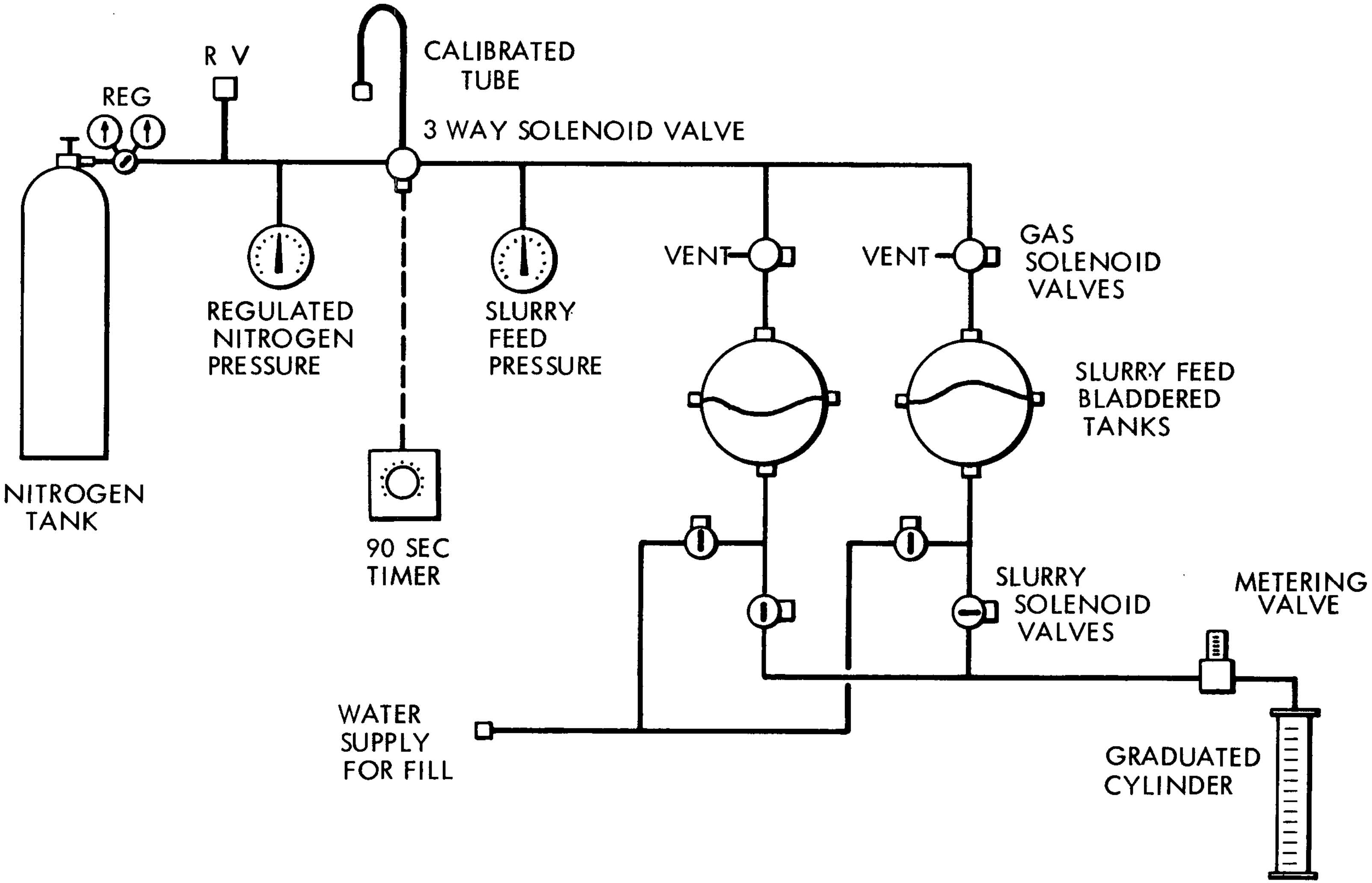


Fig. 16 Slurry Feed Calibration Test Schematic

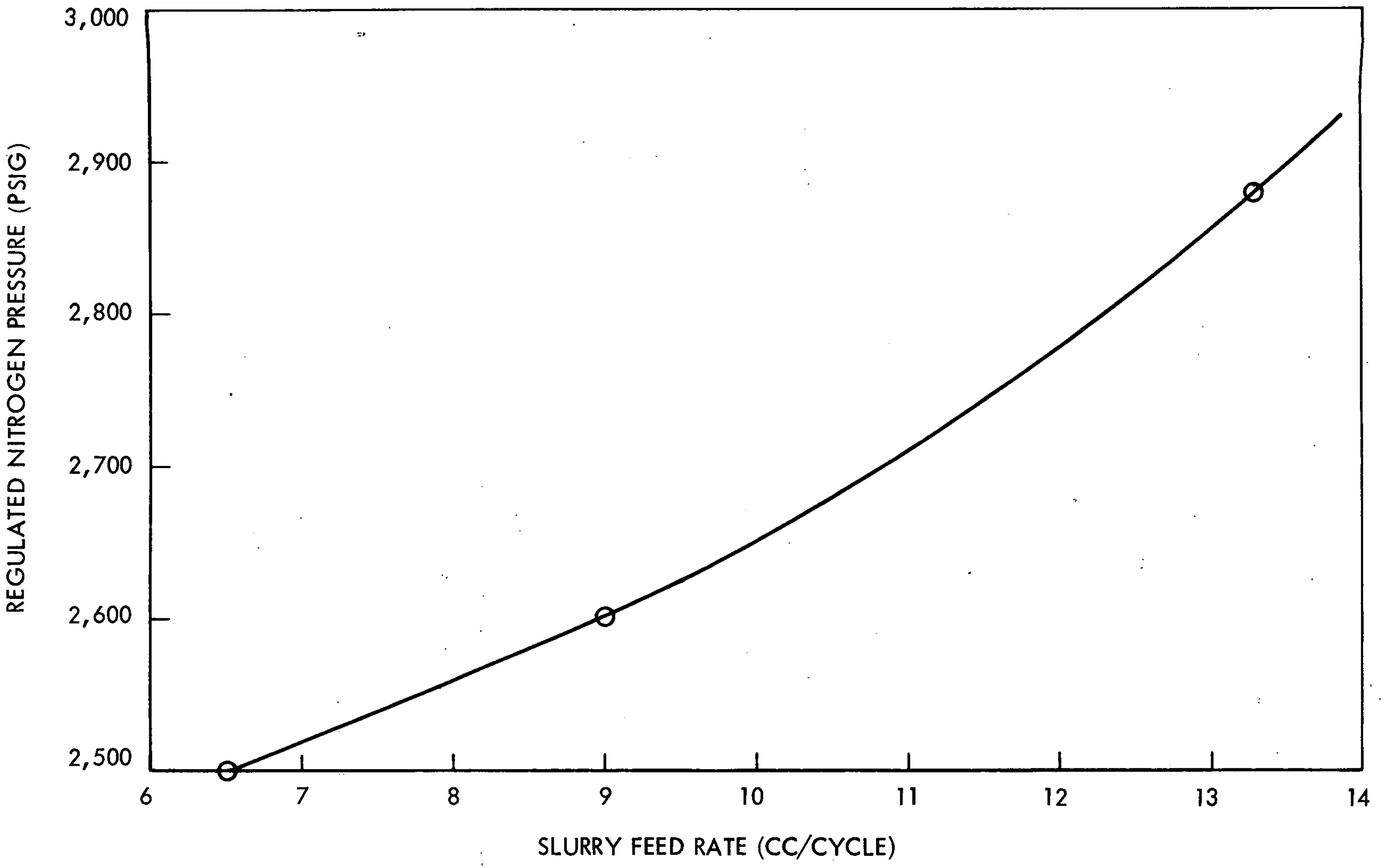


Fig. 17 Slurry Feed Rate vs Nitrogen Regulator Pressure

Oxygen Feed Calibration Test

An oxygen feed calibration test was run in a manner similar to the slurry feed calibration test. Figure 18 presents a schematic of the test setup. The test was conducted by:

- o Adjusting the oxygen regulator to various pressure levels (2400, 2430, 2460, and 2480 psig).
- o Energizing the three-way solenoid valve timer.
- o Maintaining downstream pressure at 2200 psig by bleeding the discharge tube.
- o Measuring the volume of gas bled into a graduated cylinder.

Table 8 and Figure 19 present the test results.

Table 8

Oxygen Feed Calibration Test Data

Oxygen Regulator Outlet Pressure (psig)	Oxygen Delivery Pressure (psig)	No. of Solenoid Valve Cycles	Elapsed Time (min)	Meas. O_2 Volume (scc)	Oxygen Flowrate (scc/cycle)
2400	2200	4	6	1470	367
2430	2200	2	3	900	450
2460	2200	2	3	1110	555
2480	2200	3	4.5	1885	628

Design O_2 Flowrate = 139 cc/hr at 2200 psig and 75°F.

Changing to Standard Pressure

$$\frac{139}{60}\left(\frac{2200}{14.7}\right)\underset{\text{Compressibility Factor}}{(0.94)} = 326 \text{ scc/min.}$$

For a 1.5 minute cycle time

Design O_2 Flowrate = 326 (1.5) = 489 scc/cycle

From Figure 19, the oxygen regulator should be set at 2440 psig.

F

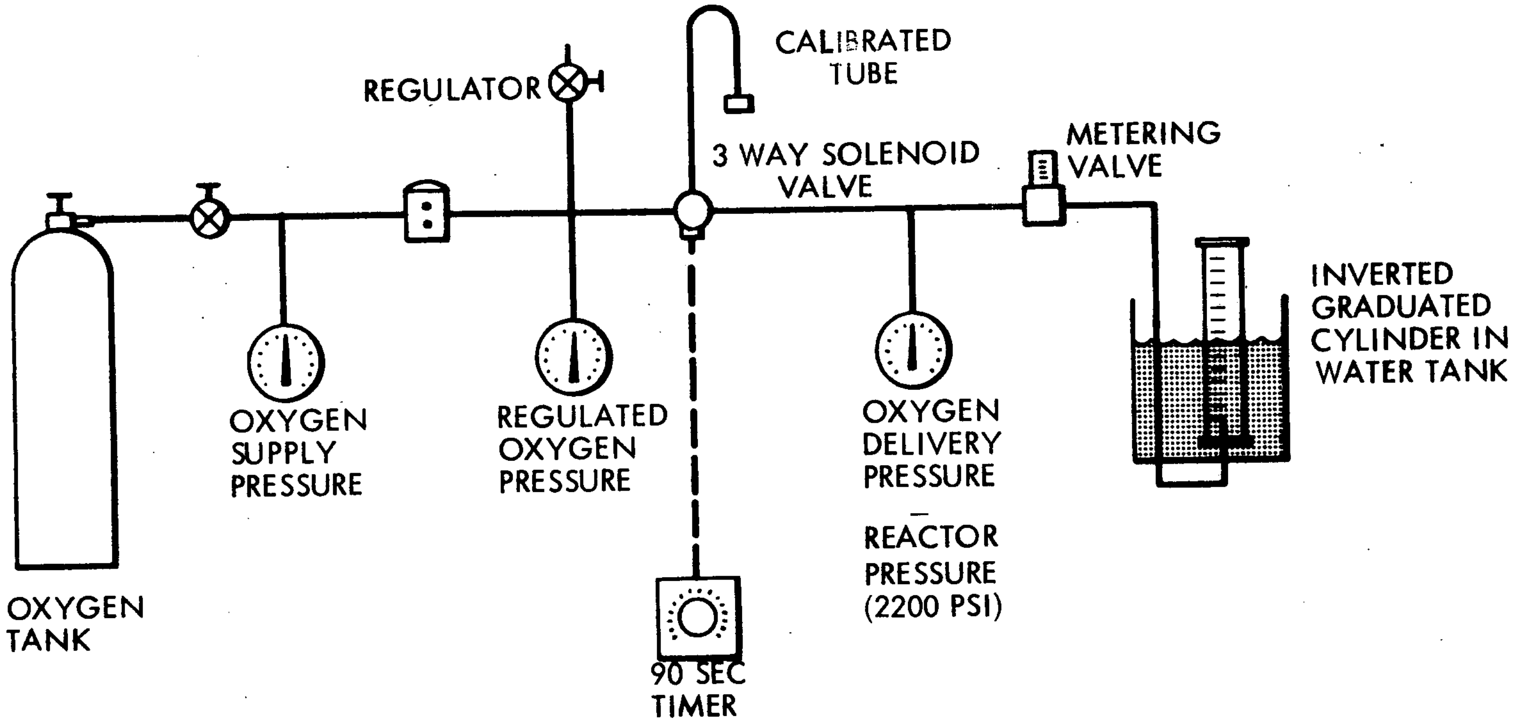


Fig. 18 Oxygen Feed Calibration Test Schematic

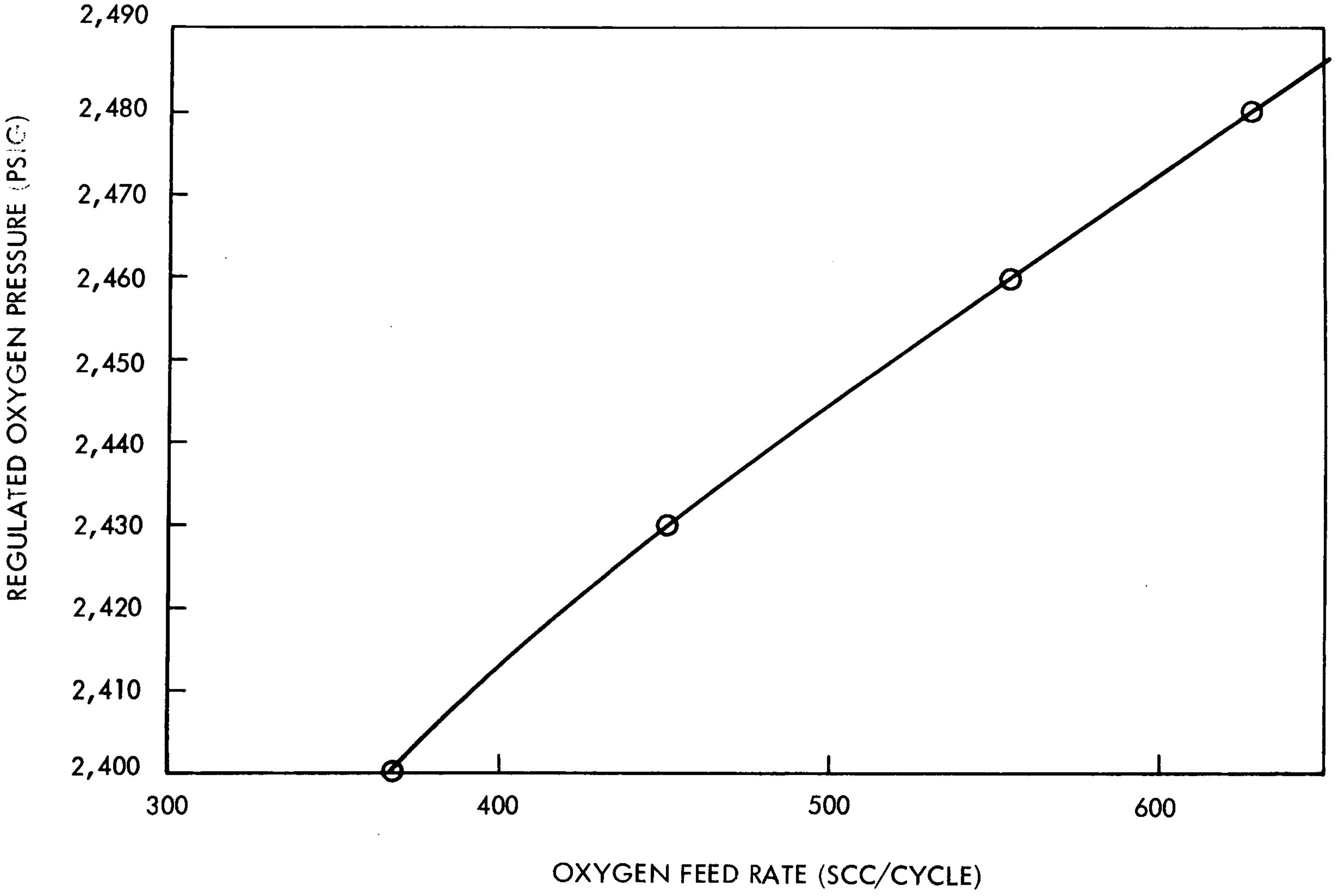


Fig. 19 Oxygen Feed Rate vs Oxygen Regulator Pressure

Design Verification Test

After several system checkout tests to verify that the system was ready, a 100-hour design verification test was run. The two significant conclusions of the checkout tests were (1) that the AVCO back pressure regulator was not satisfactory for this application, and (2) that the reactor drive required minor rework to eliminate shaft seizing. The regulator would operate satisfactorily most of the time, venting liquid, gas and ash slowly, maintaining reactor pressure at 2200 ± 25 psi, until some ash would stick under the seat. Reactor pressure would then drop as the regulator continued to vent until sufficient closing force would seat the regulator. Reactor pressure would generally fall to approximately 1700 psig. It was decided to conduct the DVT by using a manual vent valve, because repeated attempts to make the AVCO regulator work for this application failed.

The reactor drive seized several times during the checkout tests. Clearances between the motor armature and stator were increased and the end play of the shaft was adjusted to allow greater room for expansion during warmup.

The design verification test was run after these design changes were made to the system. The 100-hour test was run by:

- Filling the slurry feed tanks and lines with slurry.
- Pressurizing slurry feed tanks and reactor to 2200 psi from nitrogen tank.
- Pressurizing the oxygen system.
- Heating reactor to 550°F.
- Starting slurry and oxygen feeds by energizing control timers.
- Energizing reactor drive.
- Manually venting reactor to maintain a reactor pressure of 2200 psig.
- Collecting reactor effluent on an hourly basis.
- Monitoring system operation.
- After establishing satisfactory reactor operation, energizing the dry boiler.
- Collecting dry boiler effluent on an hourly basis.

System operation was automated so that very little manual operation was required. Manual effluent venting, filling of the slurry hold tank, and changing oxygen bottles were the major tasks required to keep the system running. The only hardware problem encountered during the test was a gauling noise created by interferences in the reactor drive system. The interference never became great enough to seize the reactor drive, but disassembly following the test showed many stirring rods and baffles bent. The gauling noise started early in the test and continued throughout the test. De-energizing the drive momentarily, generally stopped the noise for a short period of time only to have it begin again. It was concluded that clearances between the stirring rods and baffles and between the dams and shaft must be increased and that shaft end play must be better controlled to eliminate these interferences.

A summary of the test dates, samples taken, effluent liquid volumes, and description of the effluent color, which is indicative of water quality, are presented by Figure 20. Effluent volume flow rate centered around the 4-man capacity with closer control during the latter half of the test. Variations in flow occurred as a result of variations in reactor pressure caused by manual venting, by switchover of slurry feed tanks, which took 30 minutes, and nitrogen feed regulator drift. Water color and water quality varied considerably during the test, improving as the test progressed. The first half of the test was characterized by tan, brown, dark brown, blue and various shades of green. The second half of the test resulted in a light green effluent which was later to be determined as very characteristic of the Inconel 625 metal used for the reactor. The blue color was caused by a hand pump used to pressurize the slurry hold tank.

It was discovered, approximately half way through the test, that the barrel of the hand pump was made of copper and that occasionally the slurry hold tank was filled to where the tip of the pump was in the slurry. During the second half of the test, precautions were taken to insure that the pump did not touch the slurry. Wiping the pump housing produced a bright blue color. No blue samples were produced during the second half of the test.

The tan, brown, and dark brown color resulted from low oxygen flow. The oxygen was introduced through the drive tube for the first half of the test, but after dark brown water was observed at the 29th sample, it was established that some of the small passages through the drive tube were being restricted by gummy deposits. The oxygen line was re-routed so that slurry and oxygen entered the reactor through the same fitting. Brown and tan water occurred again during the 45th through 49th sample, once again due to insufficient oxygen flow. The oxygen regulator had drifted low in pressure and for some unexplained reason, slurry feed rates increased greatly. Adjusting feed rates to the design values, corrected the problem and provided consistently good water after the 50th sample.

The light green color of the effluent water was attributed to the Inconel 625 reactor body. Inconel 625 was selected on the basis of a literature review of available metals. Sea water, oxygen, and chloride stress corrosion resistance were important considerations in selecting this metal. The batch reactor used in the laboratory program was 316 stainless steel and it produced clear, colorless water. It also showed little signs of corrosion after many hours of operation. Data on the susceptability of 316 stainless to chloride stress corrosion, however, resulted in elimination of it as a possible prototype reactor material. As a further complication, because of schedule and cost impact on the reactor fabrication, the vendor (Autoclave Engineers) was permitted to fabricate the reactor internal parts of Monel 400, Hastalloy C, and 17.4 stainless steel. Although these parts did not corrode significantly, Page intentionally left blank

except for the 17-4 SS roll pins used to attach the stirring rod assemblies to the shaft, it was felt that they contributed to the effluent water color. The light green color did not present water quality problems, because it was removed along with other ions by distillation, but it caused concern about reactor corrosion.

To evaluate the color characteristics of the reactor metals, a sample of each metal was individually attached to the batch reactor cooling coil and run for 1-1/2 hours in the wet oxidation environment. The results are listed below:

Table 9

Reactor Metals Color Test Results

Metal Sample	Water Color
Inconel 625	Light green
Monel 400	Light blue
Hastalloy C	Colorless but slightly cloudy
17.4 Stainless	Colorless but slightly cloudy

It was concluded from these investigations that it was not the dissimilar metals that produced the color, but probably a characteristic of the basic Inconel 625 metal in wet oxidation. It was also concluded that materials screening tests were required to evaluate metal corrosion in the wet oxidation environment for extended periods.

Effluent water and gas samples were analyzed during the second half of the test. Table 10 characterizes effluent water samples 58 and 66. Although the water produced in the last half of the test could easily be processed to produce potable water, it was not of as high a quality as that obtained during the laboratory test program using the batch reactor. Reduction in COD was less, nitrogen and solids content were high, and ammonia concentrations were high. Additional exploratory tests were run later in the program to uncover the reasons for the differences.

Gas analysis at 72 hours showed the following gases to be present in the concentrations indicated:

Carbon Dioxide	16.7%
Oxygen	60.0%
Nitrogen	20.4%
Carbon Monoxide	0.18%
Ammonia	20 ppm
Oxides of Nitrogen	None
Oxides of Sulphur	None
Hydrogen Sulphide	None
Total Hydrocarbons	0.115%
Methane	19 ppm

Table 10

Design Verification Test Results
Reactor Effluent Water Characteristics

Sample Number	58	66
o Effluent Water		
Chemical Oxygen Demand (mgO_2/gm water)	3.9	1.0
Total Suspended Solids (% by weight)	0.63	0.25
o Filtered Effluent		
Total Water Soluble Solids (% by weight)	1.2	1.3
pH	8.4	8.4
Total Alkalinity (mg/cc)	14	10
Conductivity	Infinite	Infinite
Total Nitrogen (mg N_2/gm water)	9.3	8.0
Ammonia Nitrogen (mg N_2/gm water)	8.1	7.0
Organic Nitrogen (mg N_2/gm water)	1.2	1.0
Dissolved Ions (% by weight)		
Ammonia	1.2	1.0
Chloride	0.39	0.38
Sulphate	0.05	0.05
Phosphate	0.02	0.02
o Percent Reduction in COD	93	98

The wet oxidation reactor effluent was directed to the dry boiler after 73 hours of testing to allow evaluation of dry boiler performance. The boiler performance was satisfactory. The condensed water was analyzed and found to be free of organics, but contained 0.03 percent by weight inorganic salts (sulphates, phosphates, chlorides, etc.), indicating some carryover of salts. The water was very light grey in color. Disassembly of the dry boiler following the test showed ash and salt adhered to the boiler surface, with little ash or salt on the membrane.

Post-DVT Investigations

A number of reactor tests were run following the DVT in order to increase the quality of water produced by the prototype system. The continuous flow prototype reactor was run in batch mode to see if the continuous feed or mixing of influent and effluent were reasons for lower quality water. Batch tests of the prototype reactor resulted in water quality very similar to that produced during the DVT. It was then decided to check the efficiency of the reactor stirring system by running a test at double the speed (2400 RPM). Perhaps the stirring rods were not providing enough agitation of the liquid and gas phases. The reactor drive motor frequency was doubled to 120 cps and a six hour continuous flow test was run. Effluent water quality dropped during this test, indicating that the stirring system was running too fast, separating the liquid and gas instead of mixing them. A reduced speed test was then run at 600 RPM. Seven one-hour samples were collected during this test and water quality increased to that achieved in the laboratory batch tests. Table 11 compares the water quality achieved during tests at 2400, 1200 and 600 RPM.

Table 11

Reactor Effluent Water Quality Vs Drive Speed

	Reactor Drive RPM		
	2400	1200	600
Water Color	Blue green	Yellow green	Light green
Ash Color	Dark	Dark	Light grey
% Reduction in COD	91	94	99
Organics in Ash (mg/gm)	5.5	4.0	Nil
Organics in Distilled Effluent	10-15 ppm	10-15 ppm	None

It was now evident that low drive speeds produced the best degree of mixing. Further tests were required to select the optimum speed.

Initial Prototype System Test Conclusions

The design and testing of the initial prototype showed the wet oxidation system to be a relatively simple process, easily automated and capable of consuming a variety of waste material and producing effluent water and gases that could be easily processed to recover useful water, oxygen, and nitrogen. A number of further development tasks were identified that would improve or extend system performance. These tasks formed the basis for the remaining development work conducted under the contract and are summarized below:

- Slurry Pump Development - As discussed previously, it became apparent early in the program that special development efforts must be directed toward a slurry pump. Vendor surveys were conducted and two slurry pumping approaches were developed and tested.
- Materials Corrosion Studies - Water color observed during the DVT suggested that additional materials corrosion studies were needed. Candidate metals were selected and corrosion tests were run on samples in the one liter batch reactor.
- Oxygen Supply Tradeoffs - The source of high pressure oxygen for the wet oxidation system is an important factor in the system feasibility. Comparison data was generated on a variety of oxygen supply systems and a preferred approach identified.
- Alternate Water Recovery Methods Study - Concern that the dry boiler power requirements were high and that zero gravity operation was not obviously proven by ground test, resulted in the need to compare various salt removal devices to see which one integrated best with the wet oxidation system. Comparison data was generated for vapor compression distillation, electrodialysis, air evaporation, reverse osmosis, and vapor diffusion water reclamation systems. Ten gallons of wet oxidation effluent water were produced for tests by developers of the electrodialysis and vapor compression units.
- Ammonia Removal - Ammonia concentrations in the wet oxidation effluent water were high and presented the only developmental problem with processing the water or gaseous effluents from the system. Ammonia removal processes were analyzed and laboratory tests run to evolve a means of eliminating the ammonia.
- Spacecraft Solids Grinder - Biologically contaminated wastes in addition to feces and urine present a disposal problem onboard a spacecraft. Development of a spacecraft solids grinder would allow these wastes to be pumped into the wet oxidation system for processing. Commercially available grinders were surveyed and laboratory tests were run to evolve requirements for a prototype grinder.

3

- Slurry Shutoff Valves - High pressure motorized shutoff valves suitable for flight prototype system use were not available for the initial prototype system. Solenoid valves were used that were subject to clogging. This task consisted of locating, procuring, and testing of motorized, high pressure valves suitable for slurry use.

- Back Pressure Regulator - As a result of the poor performance of the AVCO back pressure regulator for wet oxidation application, the task of locating, procuring, and testing a suitable back pressure regulator was added to the new work.

- Filters - The need to filter ash from the effluent if the dry boiler was replaced by one of the alternate water recovery devices was recognized. Filter vendor surveys and testing of candidate filters was scheduled.

- Reactor Design Studies - The design of the seven individual chambers in the reactor made it very difficult to assemble and disassemble the reactor. In order to replace the center bearing or seal, the entire reactor had to be disassembled, including removal of the stirring rod assemblies from the shaft and removal of all seven chambers. The heavy, bulky drive motor, limited life of the carbon bearings, and interferences encountered in the DVT were also of concern. Reactor design studies were conducted to improve the design in each of these areas. New reactor parts were fabricated and long duration bearing life tests were run. These tests also performed the function of updated system design verification tests. The balance of the report presents the work accomplished in each of these development areas.

SLURRY PUMP DEVELOPMENT

As a result of the vendor surveys and design studies, two pumping systems were selected for further development. One was based on the bootstrap principle using effluent liquid and gases to force the slurry into the reactor, and the other utilized a full head rise water pump to force slurry from a bladdered tank into the reactor. Both of the pumping approaches were discussed earlier in the report and were schematically described by System 1 and 3 of Figure 6, respectively. Both pumping systems were designed, fabricated, and tested during the development effort following the DVT.

Double-Ended Slide Valve Slurry Pump

The most difficult part of the bootstrap pump was the design of suitable valves. Conventional check valves could not be used, because of the solids content of the slurry. Steamlining of the valves to avoid fouling of springs did not prove successful. After a considerable amount of vendor survey work, it was decided that the use of slide valves similar in design to those used in aircraft hydraulic systems offered the greatest chance of working in this application. A specification based on this principle was prepared and Sterer Engineering in Los Angeles, California, responded with a favorable quotation. The following summarizes the pump requirements:

1) Slide Valves
    a) Materials: Basic materials shall be 316 stainless steel. Special materials may be used in the working parts of the valves if needed, but shall be compatible with the slurry and effluent fluids.
    b) Working Pressures: 10 to 3000 psia.
    c) Type of Seal: Shear Seal.
    d) Minimum Passage Size: A minimum passage size of 0.300 shall be maintained through the slide valves to prevent clogging with slurry or effluent.
    e) Inlet and Outlet Ports: All external inlet and outlet ports shall be 3/8 AN female fittings.
    f) Operating Temperature: +65 to +120°F.
    g) Actuation: It is desired that the slide valves be actuated by electrical solenoids directly without the use of gas pressure. If this is not feasible, a second choice is electric motor actuation. If this is not feasible, solenoid actuated pilot valves with gas or hydraulic oil may be used.

2) Piston and Cylinder Assembly
    a) Type: Piston.
    b) Slurry Outlet Pressure: 2200 psia.
    c) Stroke Volume: 27.5 cc (1.68 in3).
    d) Stroke Length: 1 inch.
    e) Design Pumping Rate: 330 cc/hr (20.2 in^3/hr).
    f) Pumping Speed: 12 strokes/hour.

g) Materials: Basic materials shall be 316 stainless steel. Special materials may be used in the working parts of the pump if needed, but shall be compatible with the slurry and effluent fluids.

h) Pump Case Design Working Pressure: 3000 psia.

i) Pressure Safety Factor: Burst pressure of the pump shall be four (4) times the pump case design working pressure.

j) Operating Temperature: +65 to 120°F.

k) Quantity of Pump Drive Fluids: The pump can utilize all or part of a liquid/gas mixture of 330 cc/hr (20.2 in^3/hr) of liquid and 127 cc/hr of gas at 2180 psia.

l) Pump Fluids Description: The fluid to be pumped is a sewage mixture of pulverized feces, urine, toilet paper, paper towels, and plastic bags with particles no larger than 1/16 dia. The fluids available to drive the pump are water containing dissolved salts (1.7% by weight), suspended phosphate ash (0.2% by weight), and ammonia (0.55% by weight), and gases are comprised mostly of CO_2, O_2 and N_2 with trace amounts of ammonia and hydrocarbons.

Figures 21 and 22 present a schematic drawing and photograph of the pump that Sterer designed in accordance with the specified requirements. The pump provided a free-floating two-piece piston with slurry on the upper end and reactor effluent on the lower end. The piston slid in a two-piece body with a diaphram between the two body and piston pieces. The diaphram acted as a static backup seal to the piston seals to prevent crossflow from the slurry end to the effluent end of the pump. The slides and the shear seals located on top and bottom of the body provided valving action and sealing of the valves. The slides were moved across the top of the body by four hydraulic actuators located on each side of both slides. The principle of sealing was that the pressure imbalance across the shear seal plus the spring force behind the shear seal effected a seal between the slide and the lip of the shear seal, as well as a seal between the lapped slides and the pump body. Slide and piston vent ports were provided.

In operation, slurry under slight pressure (20 psig) was applied to the slurry inlet port with the upper and lower slides pushed to the right. Slurry would then fill the upper chamber, pushing the piston down, forcing effluent liquid and gas from the bottom piston chamber out the vent port. The upper slide was then pushed to the left, connecting the upper chamber to the reactor inlet, pressurizing it to 2200 psig. The bottom slide was then pushed to the left, connecting the bottom chamber to the reactor outlet. Effluent gases and liquid would then pressurize the bottom chamber forcing the piston to rise because the bottom piston area was larger than the top piston area. Slurry was thereby forced into the reactor and effluent liquid and gas were discharged from the reactor. The top slide valve was then pushed to the left again, connecting the upper chamber to the low-pressure slurry hold tank. Pushing the bottom slide to the left then vented the high-pressure effluents from the bottom chamber and allowed slurry to fill the top chamber again, completing the pump cycle.

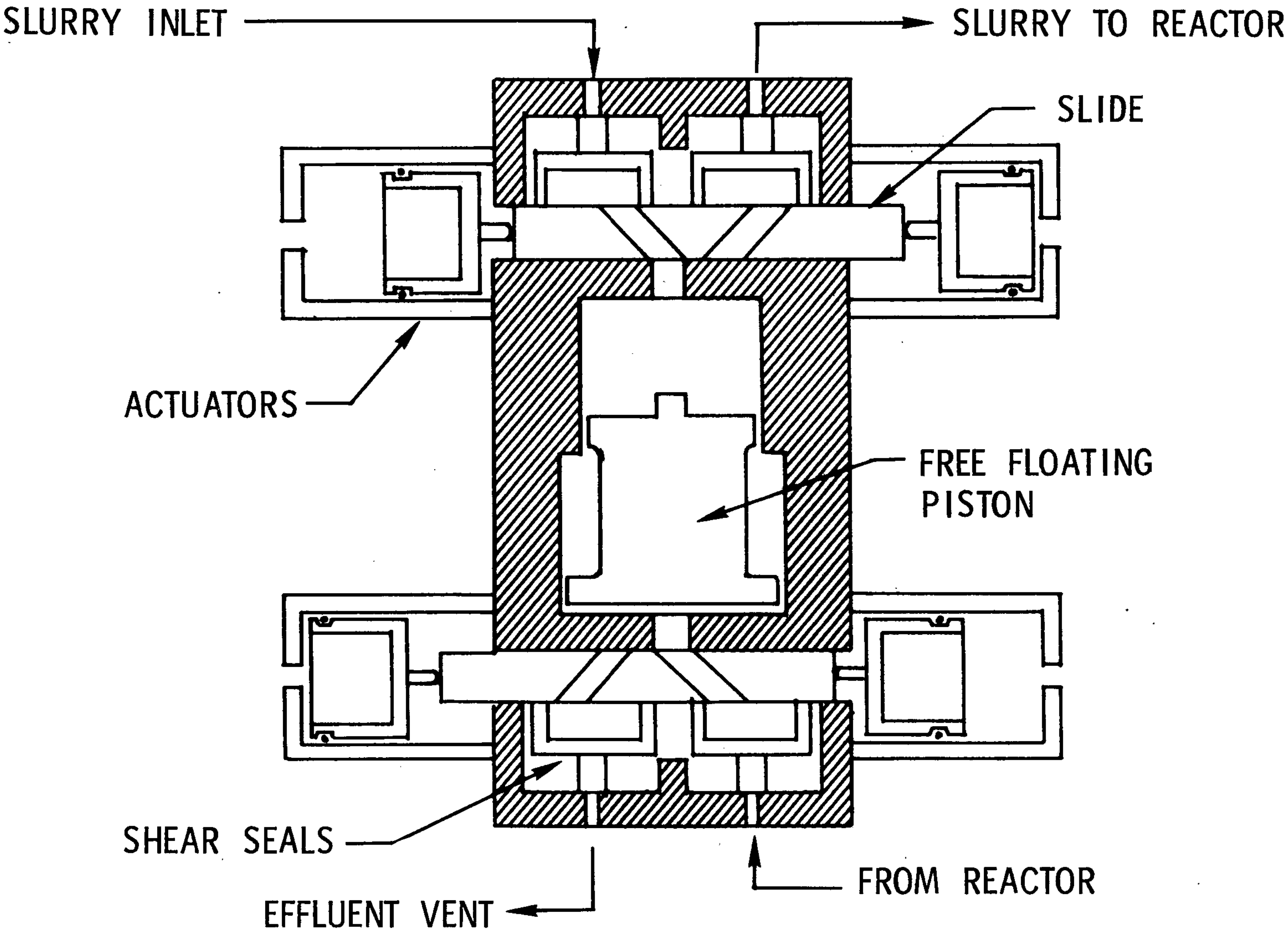


Fig. 21 Double-Ended Slide Valve Slurry Pump Schematic

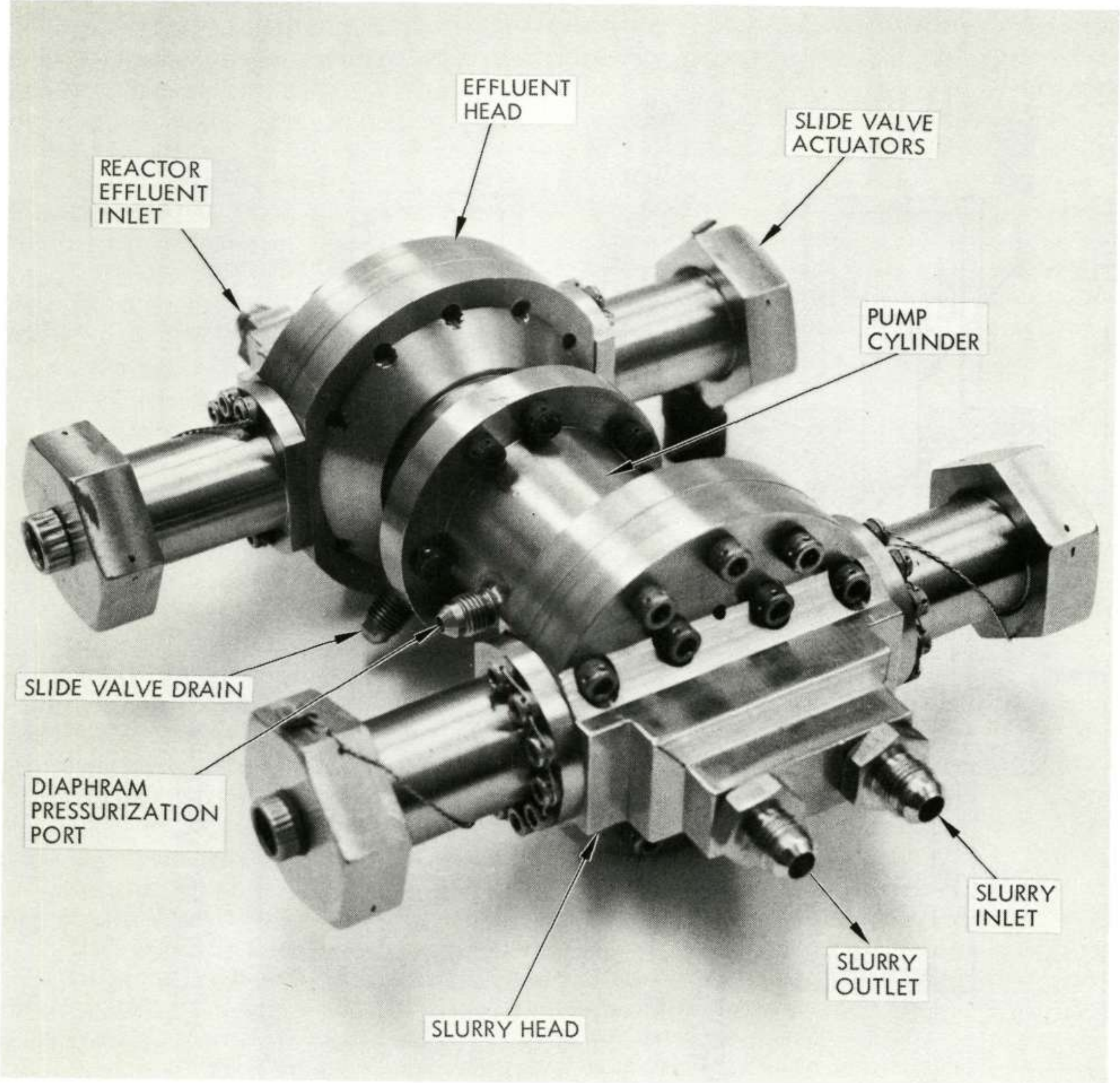


Fig. 22 Double-Ended Slide Valve Slurry Pump

All basic pump parts were 316L stainless steel except for the slides which were tungsten carbide. The shear seal tips were also tungsten carbide.

The pump developed and tested under this contract was equipped with hydraulic actuators, so that actuation forces could be varied to establish optimum slide valve performance. Hydraulic actuation provided easy adjustment of actuation force to evaluate pump performance under different slide valve velocities which had an effect on slide valve leakage. A flight pump design would incorporate electric motor driven slides.

Figure 23 presents a schematic of the test setup used to evaluate slide valve pump performance. Slurry was supplied to the pump inlet from a bladdered tank pressurized by nitrogen gas to 20 psig. The pump outlet was connected to the reactor and the reactor vent line was connected to the drive end of the slurry pump. The drive end vent was connected to a collection flask. Nitrogen gas at 60 psig was used during startup to prime the pump and purge the slurry feed lines. A hydraulic pump, three solenoid valves and timer were used to sequence operation of the hydraulic actuators that drove the slide valves. The test was run by:

- Filling the bladdered tank from the slurry hold tank.
- Priming the pump and bleeding slurry lines using nitrogen gas at 60 psig on the drive end of the pump. Hydraulic pump and solenoid valve timer operation were required.
- Heating reactor to temperature (550°F).
- Energizing oxygen feed controls to pressurize the reactor system to 2200 psig.
- Energizing the hydraulic pump and solenoid valve controls.
- Observing pump actuator operation; leakage, if any, from the slide bleed ports; back pressure regulator venting; and reactor pressure.

The pump was tested for approximately 20 hours during four test runs. Two problems arose early in the test series. Significant gas leakage from the drive and slide valve seriously depleted reactor pressure during most of the test period. Reactor pressure was maintained at 2200 psig only by bleeding in more oxygen. The second problem was that the cap screw used to connect the two piston parts together protruded beyond the end of the piston, thereby effecting a seal over the slide valve port. Therefore, with the top chamber connected to the reactor at 2200 psig, when the bottom slide was moved to the reactor position, the gases and liquid from the reactor could only push against the cap screw and not the full piston area. The piston would not rise as a result. Cutting slots in the cap screw solved this problem. Subsequent tests also resolved the leakage problem. Leakage from the drive end slide reduced steadily during the test series until it completely disappeared in the last six hour test. Apparently, wearing in of the slides and seals was required to effect a good seal.

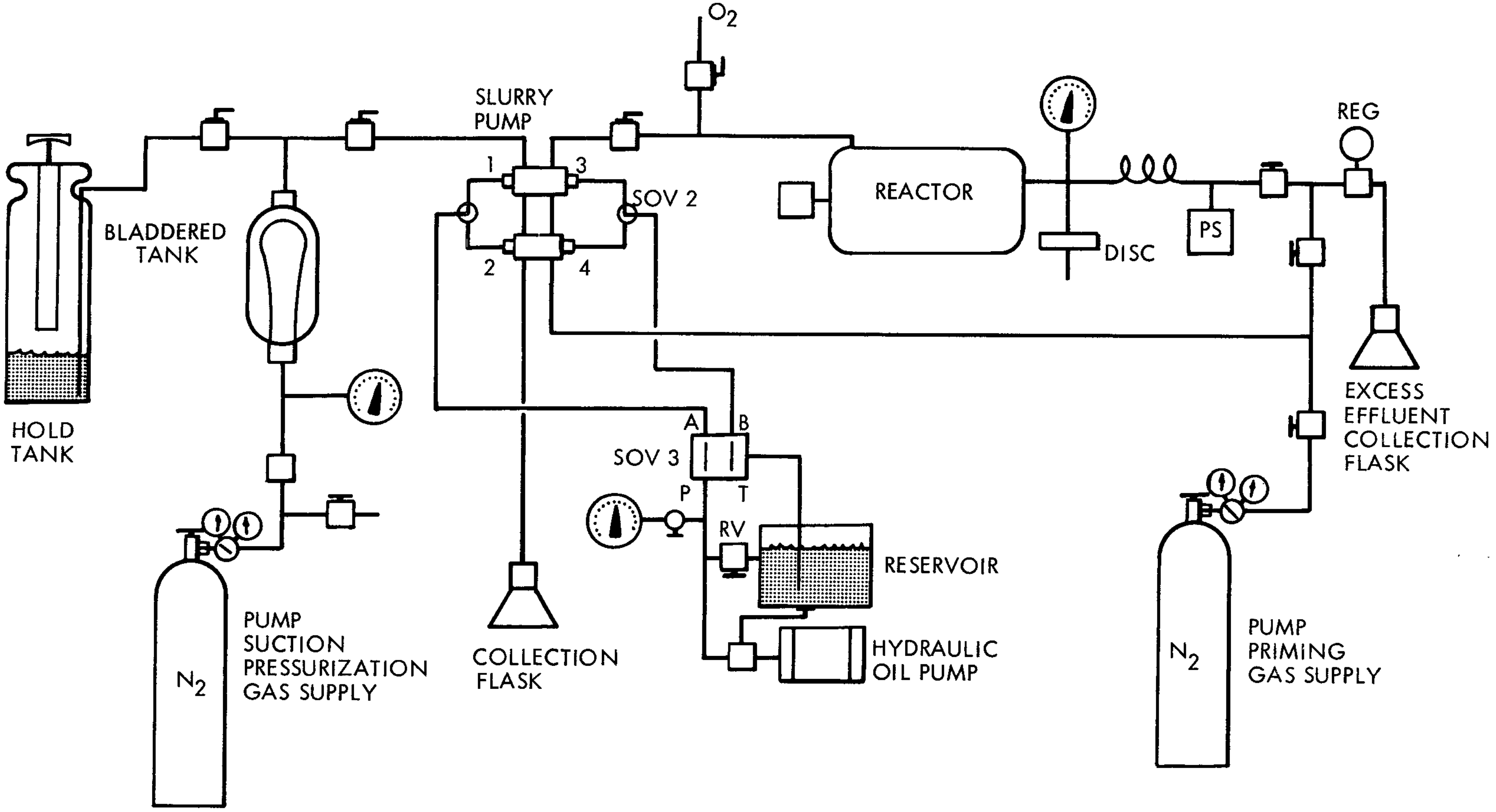


Fig. 23 Double-Ended Slide Valve Slurry Pump Test Schematic

During the last six hour test, the pump operation was very satisfactory. Slurry was pumped into the reactor, reactor effluents were vented and collected, reactor pressure was automatically maintained at 2200 psig, and excess effluent liquid and gases not needed for pump operation were vented through the system back pressure regulator. A hydraulic pressure of 950 psig was used for slide valve actuation during the test series to insure slide valve operation. During the last day, hydraulic pressure was varied to investigate pump performance as a function of hydraulic actuator pressure. Pressures as low as 350 psig would move the actuators, but 450 to 500 psig was required to insure complete and reliable operation.

Hydraulic Piston Pump

The Milton Roy piston type hydraulic pump, Model D 19-60029-0034, was located after a considerable amount of vendor survey work. The pump was ideally suited to the requirements of the prototype wet oxidation system. It was sized to deliver 0 to 540 cc/hr of water and although rated for 1000 psig service, the supplier indicated that this model of pump had been used in many applications to 2500 psig. Seal life not structual or motor size limited rating to 1000 psig. Figure 24 presents an assembly drawing of the pump. It consists of a pump head (1) containing double ball check valves (2), piston (3) and piston seals (4); a plunger (5) and flow control eccentric (6) that drives the piston; a gear box (7) for speed reduction, and electric motor (8). Pumping rate was changed by adjusting a stop that limited piston stroke to obtain flowrates between 0 and 540 cc/hr.

Figures 25 and 26 show schematically and pictorially how the pump was plumbed into the system and tested. Operation of the system was as follows: assuming the bladdered slurry tank was just filled with slurry, with valve number 1 open, valve number 2 closed, and valve number 3 open. Water was used in the checkout test, but operation with slurry was identical. Slurry has filled the bladdered tank and pushed water from the back side of the bladder through solenoid valve number 3 into the low pressure reservoir. The hydraulic pump was left running at all times and during tank filling, was pumping water back to the reservoir through valve number 3. Tank filling required approximately five minutes. Valves 1 and 3 were then closed and in approximately five minutes, the pump increased the tank pressure to 2400 psi to open the relief valve holding system pressure at that level. Valve number 2 was then opened to deliver slurry to the reactor or in case of the checkout test, to a graduated cylinder. System pressure immediately fell to reactor pressure (2200 psi) upon opening of valve number 2. Approximately twelve hours later, when the slurry tank was empty of slurry, the bladder forced the piston at the tank outlet closed to protect the bladder from overpressurization and the hydraulic pump pressurized the system to 2400 psi, opening the relief valve. Water flow was then directed back to the reservoir, until valve number 2 was closed and valve number 3 was opened, reducing system pressure to ambient. Valve number 1 was then

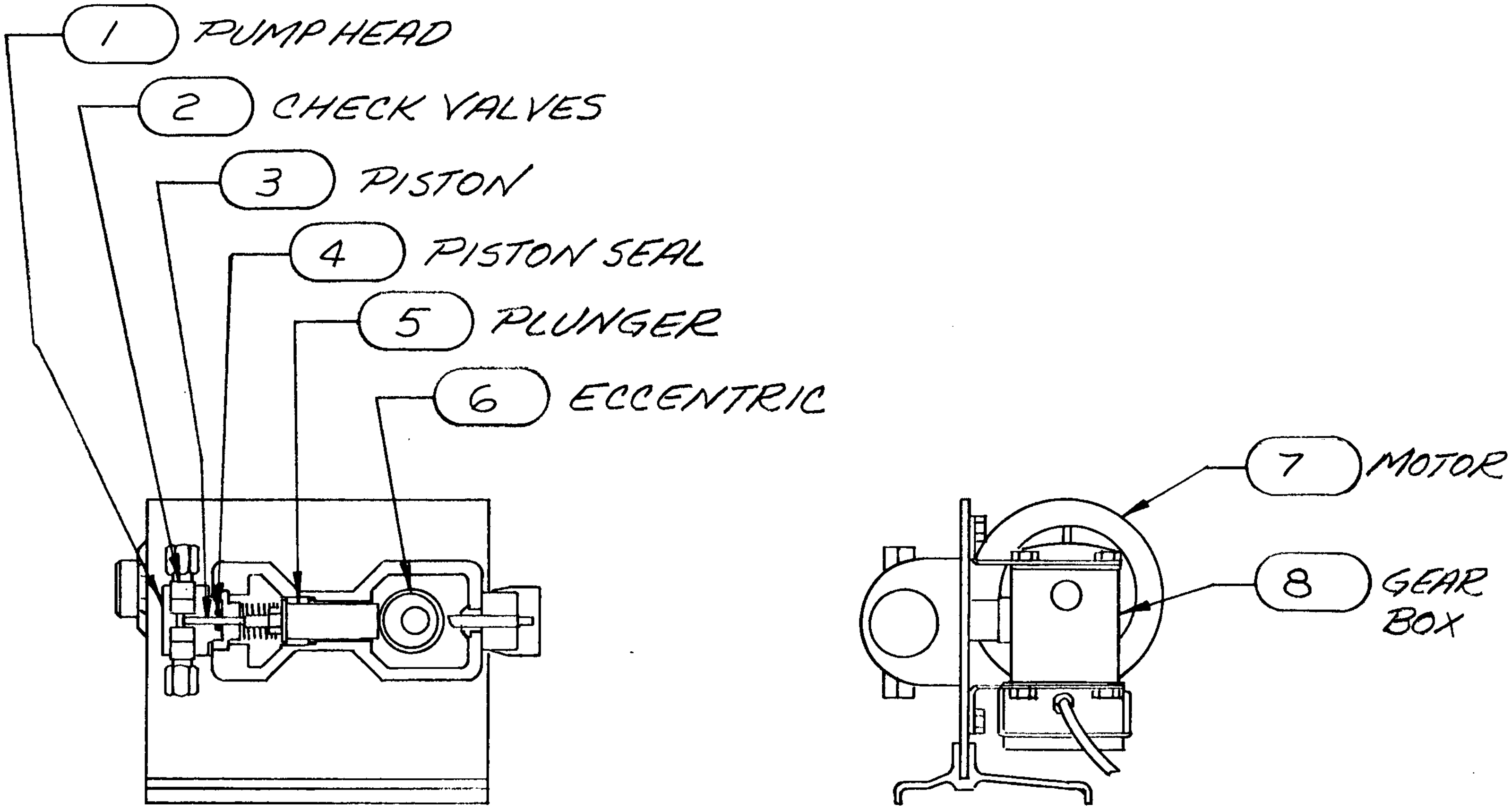


Fig. 24 Assembly Drawing of Hydraulic Piston Pump

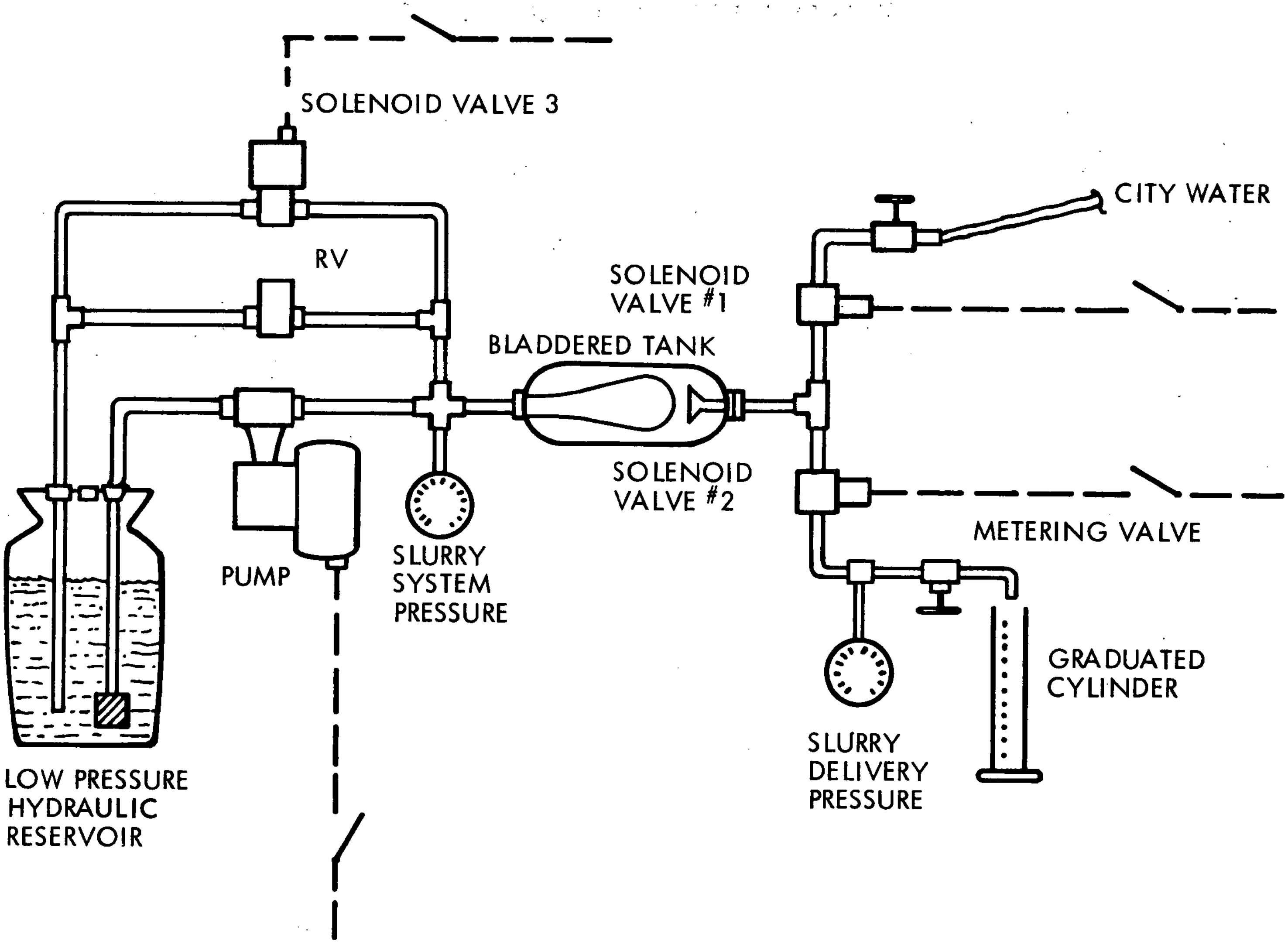


Fig. 25 Hydraulic Piston Pump Test Schematic

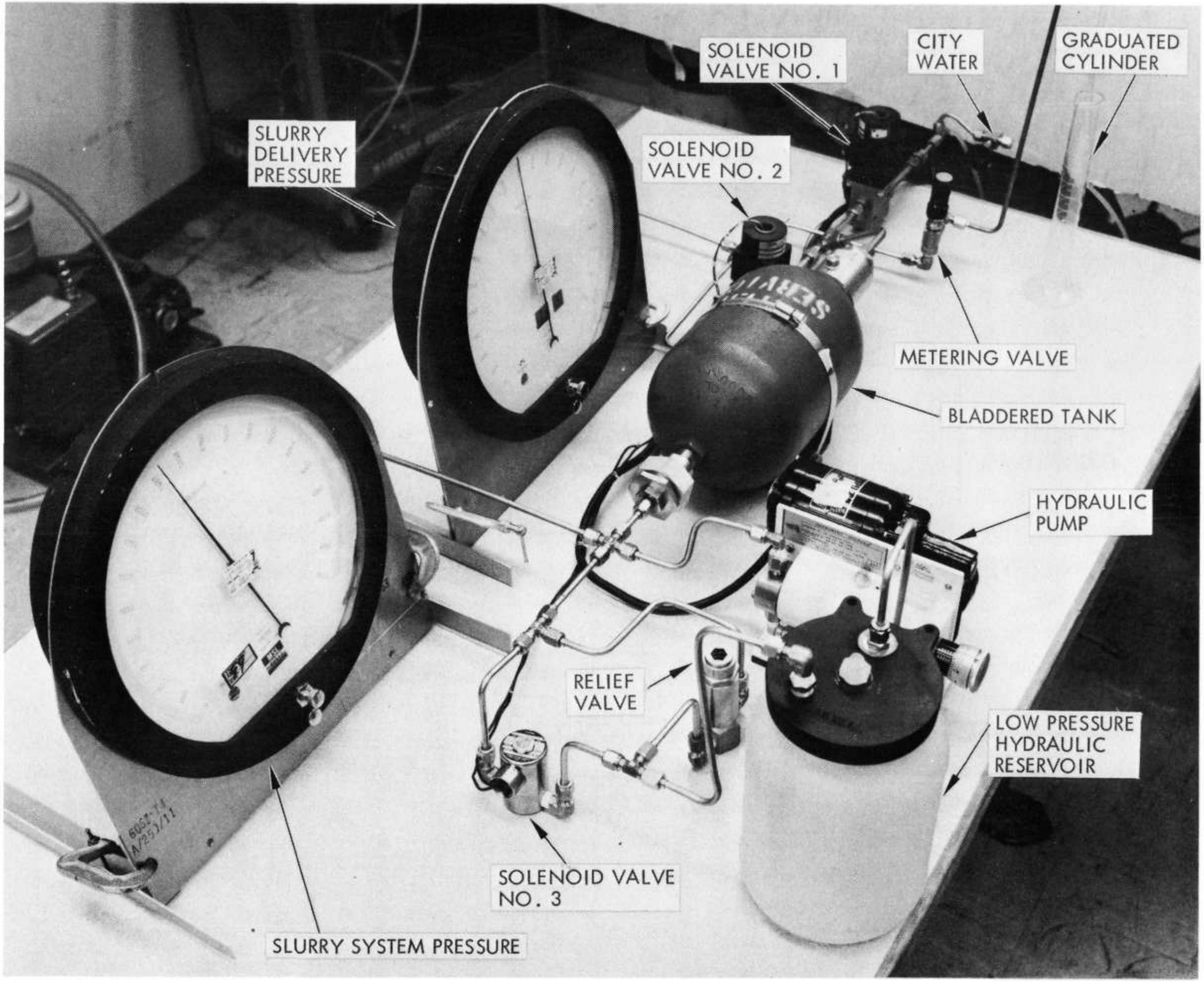


Fig. 26 Hydraulic Piston Pump Test Setup

opened, refilling the slurry tank. This entire sequence was controlled by a timer set for a twelve-hour cycle. Approximately fifteen minutes was required every twelve hours for slurry tank filling and pressurization.

A calibration and checkout test was run on the system, shown schematically by Figure 25. The system was pumped to 2200, 1000 and ambient pressures in three consecutive data runs. The water delivery valve was adjusted to maintain a constant system pressure with the pump running at 100% rated capacity. Water was collected for a ten minute period, three times at each pressure. Water flow was 9 ± 0.2 cc/min for all pressures. The pump stroke limit adjustment knob had a micrometer type scale reading from 0 to 100. A setting of 61 produced the system design water flow rate of 5.5 cc/min or 330 cc/hr.

The hydraulic pump was run extensively during reactor and system tests later in the program, accumulating approximately 30 days of operation over a twelve-month period.

MATERIALS CORROSION STUDIES

A materials corrosion study and test program was initiated as a result of DVT results. It was felt that a more extensive materials survey, followed by laboratory testing of specimens for extended periods in the wet oxidation environment, was required. The nine metals described by Table 12 were selected for test, based on their history of use in sea water and/or oxygen applications and their resistance to chloride stress corrosion. It was desired to evaluate both surface and stress corrosion, so the tests were run on "U" bend specimens, deflected by bolts through the legs of the "U" to achieve 80% of yield stress levels in the bend of the "U". The specimens were tested in the one liter batch reactor by suspending the "U" bend specimens on a 316 SS support rack attached to the reactor head. Figure 27 presents a sketch of a typical "U" bend specimen and Figure 28 shows the specimens attached to the support rack and the rack attached to the reactor head. The specimens were attached to the support rack with 302 stainless bolts and ceramic spacers were used to isolate each specimen from the bolts and support rack. Megohmeter readings taken between the sample and the rack generally ran between 1 and 3000 megohms before the test and 0.1 to 1 megohms after the test. The difference between pre and post-test readings was caused by an ash deposit on the ceramic spacers following the test. The reactor agitator shaft can be seen running down the center of the support rack with the impellar at the lower end.

The support rack held fourteen specimens; five on the lower tier in the liquid at the bottom of the reactor, five in the liquid/gas region near the center of the reactor, and four in the gas phase near the top of the reactor. The location of the thermocouple suspended in the reactor from the head prevented a fifth specimen from being installed on the top tier. This specimen arrangement allowed three specimens of five metals to be tested simultaneously with one specimen of each metal in each phase zone of the reactor, i.e., liquid, liquid/gas, and gas, except for the one missing gas phase specimen.

Table 13 presents the metal thicknesses used for the test specimens, their measured yield strength values and calculated total deflections to give 80% of yield stress in the "U" bend area.

All metals were recieved cold rolled and fully annealed except MP 35N which was received 50% cold worked unaged. Bending of the specimens harden them, so yield strength measurements after bending were made and their measured values used to calculate total deflections.

TABLE 12
CANDIDATE REACTOR METALS

DESIGNATION	SUPPLIER	MAJOR COMPOSITION*	CHARACTERISTICS
316 L STAINLESS	Many	Cr 16-18, Ni 10-14, Mo 2-3, Si 1, Mn 2, Bal Fe	Reference metal.
20 Cb STAINLESS	Carpenter Tech	Cr 20, Ni 34, Mo 2.5, Cu 3.5, Fe 33, Mn 2, Si 1.0, Cu 3.5	Improved SCC**.
E-BRITE 26	Airco Vacuum Metals	Cr 25, Mo 1.0, Mn 0.4, Si 0.4, Ni + Cu 0.5, Bal Fe	Improved SCC**.
INCONEL 601	Huntington Alloys	Ni 76, Cr 16, Fe 7	Excellent high temp oxidation resistance.
INCONEL 718	Huntington Alloys	Ni 53, Fe 18, Cr 19, Cb 5.2, Mo 3	High strength, good sea water history, good high temperature oxidation resistance.
INCOLLOY 825	Huntington Alloys	Ni + Co 42, Cr 22, Mo 3, Fe 28, Mn 1.0, Cu 4	Good SCC and sea water history.
INCONEL 625	Huntington Alloys	Ni 58, Cr 22, Mo 9, Fe 5, Col 4, Co 1.0	Excellent SCC, good fabricability.
HASTALLOY C	Union Carbide Stellite Division	Co 2.5, Cr 14.5-16.5, Mo 15-17, W 3 - 4.5, Fe 4-7, Mn+Si 1.0, Bal. Ni.	Excellent sea water history, excellent SCC and high temperature oxidation resistance.
MP 35N	Latrobe Steel	Co 35, Ni 35, Cr 20, Mo 10	Excellent general corrosion resistance, high strength.

* PERCENTS.

**CHLORIDE STRESS CORROSION RESISTANCE.

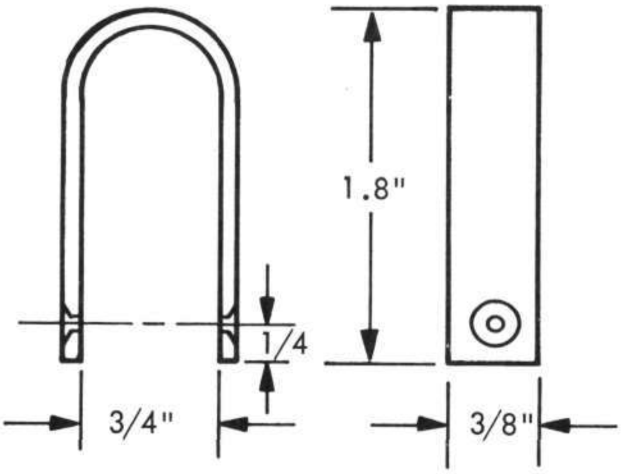


Fig. 27 Typical "U" Bend Corrosion Test Specimen

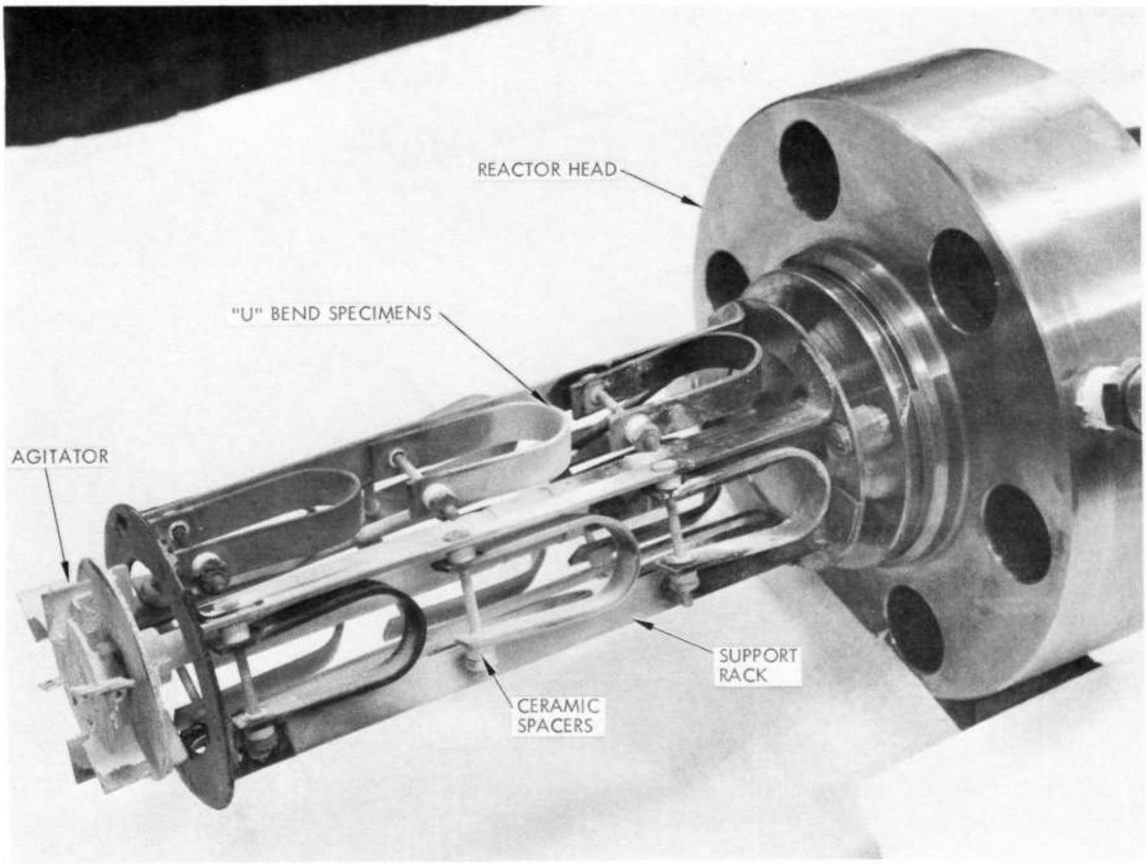


Fig. 28 Corrosion Test Specimens Installed on Reactor Head

Table 13
Corrosion Test Specimen Deflections

Metal	Thickness (in.)	Measured Yield Strength (psi)	Total Deflection (in.)
316L Stainless	0.060	42,000	0.06
20 Cb Stainless	0.062	70,900	0.14
E-Brite 26	0.040	62,900	0.20
Inconel 601	0.078	35,000	0.08
Inconel 718	0.062	175,000	0.29
Incolloy 825	0.062	35,000	0.07
Inconel 625	0.062	80,500	0.13
Hastalloy C	0.080	73,500	0.12
MP 35N	0.052	190,000	0.37

Figure 29 presents a schematic of the corrosion test setup. The one liter batch reactor was operated in batch mode by charging 460 cc of fecal/urine slurry (10 percent by weight feces), installing the reactor head with specimens attached, pressurizing the reactor to 800 psig, with pure oxygen, heating the reactor to 550°F (2200 psig pressure) and adjusting the air motor drive to 1200 RPM. The reactor was operated 24 hours a day in a series of four tests. Automatic shutdown safety provisions were provided to cut off all power on over temperature (625°F) and overpressure (3000 psig). The first two tests were 100 hours in duration and they served as screening tests to eliminate four of the nine candidate metals, so that only one long duration corrosion test could be run on three specimens each of the best five metals.

Test number one exposed three samples each of Hastalloy C, E-Brite 26, 316L Stainless and Carpenter 20 and two Inconel 601 samples. Test number two exposed three samples each of Inconel 718, Incolloy 825, Inconel 625, and MP 35. Following the 100 hour screening tests, the samples were inspected with the following results:

Inconel 718 - The upper tier specimens showed minor pitting (.001" to .003" deep) at the upper end of the "U" bend legs. The lower specimen had extensive pitting over 30 to 50% of the specimen. A large crack had initiated on the tension face of the bend and was 0.040 inches deep.

Inconel 601 - The legs of the top two specimens were pitted (0.010" deep). No evidence of cracking was observed.

Incolloy 825 - The "U" bend surface on the top two specimens was pitted. (0.001 to 0.002" deep).

All Others - No evidence of surface or crack corrosion was evident.

As a result of the screening tests, Hastalloy C, Inconel 625, Carpenter 20, E-Brite 26 and MP 35 were selected for further test. 316SS was eliminated because of its record of chloride stress corrosion (Ref. 2 and 3) and only five metals could be tested, at one time, in the reactor.

Three samples each of the five metals, excluding one top tier specimen, were then exposed to the wet oxidation environment for two weeks (336 hours). Examination after two weeks exposure showed no evidence of corrosion on any specimens, so they were tested for an additional four weeks (672 hours). Final examination showed no evidence of any pitting or crevice or stress corrosion cracking on the Hastalloy C, Inconel 625, E-Brite 26 or MP 35. Dye penetrant inspection (Flourescence) did not reveal any micro attack. The Carpenter 20 was pitted on the surface of the U bend area and on the legs (0.001 to 0.005" deep). Hastalloy C showed the most uniform color over the entire specimen indicating an even oxide protective coating.

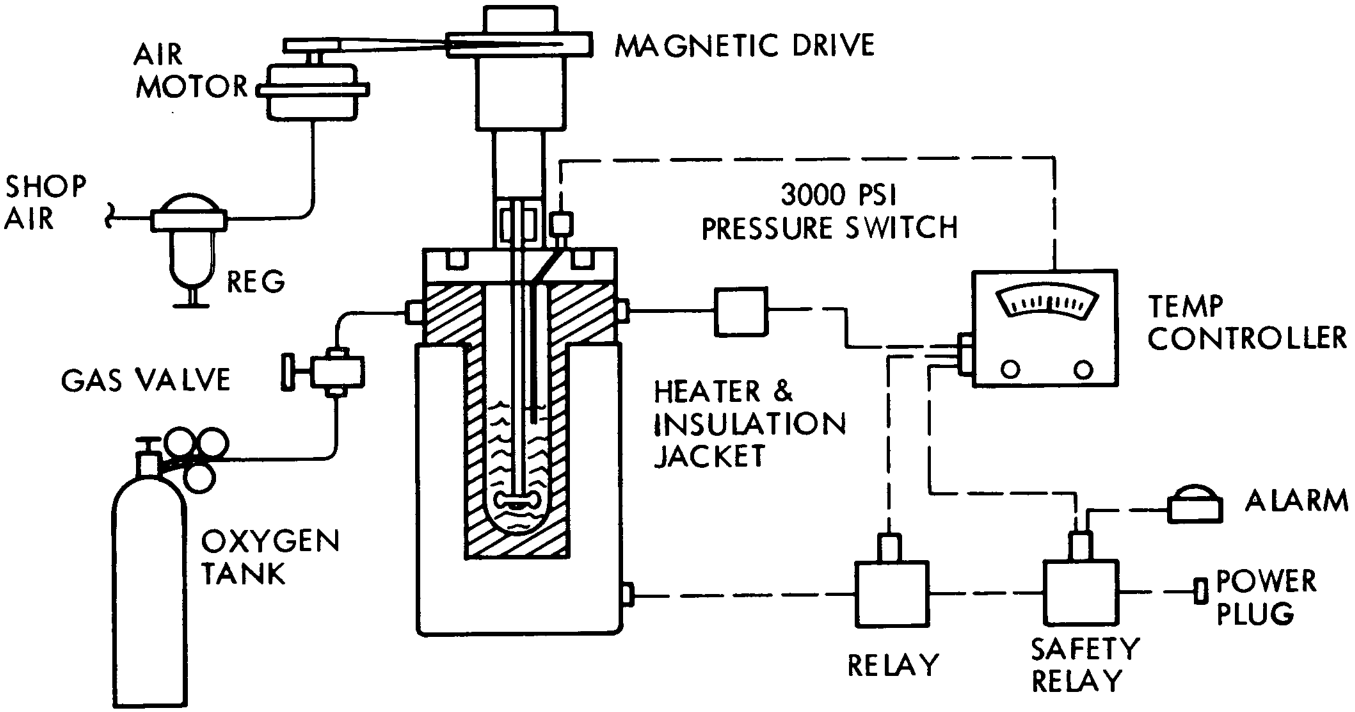


Fig. 29 Corrosion Test Schematic

Based on the results of the corrosion tests, Hastalloy C was selected as the preferred metal for the wet oxidation reactor. As a matter of added interest, the Inconel 625 reactor has been run for a total operating time of approximately five months over a period of two years, without any significant evidence of corrosion while making yellow-green water. The color can be removed in the salt removal process, so Inconel 625 has proven to be a reasonably good selection. Hastalloy C is still felt to be a better choice.

Figures 30 and 31 present photographs of the most severely corroded specimens for all metals except 316L stainless. Discoloration of the MP35, Inconel 625, Hastalloy C and E-Brite 26 was not an indication of corrosion. These are gas phase specimens that did not produce an even coloration as did the liquid phase specimens, because of splashing of liquid and ash on them during agitation. The light spots on the "U" bend of the Carpenter 20 are pits. Discoloration in the leg of the Inconel 601 and on the "U" bend of the Incolloy 825 are corrosion.

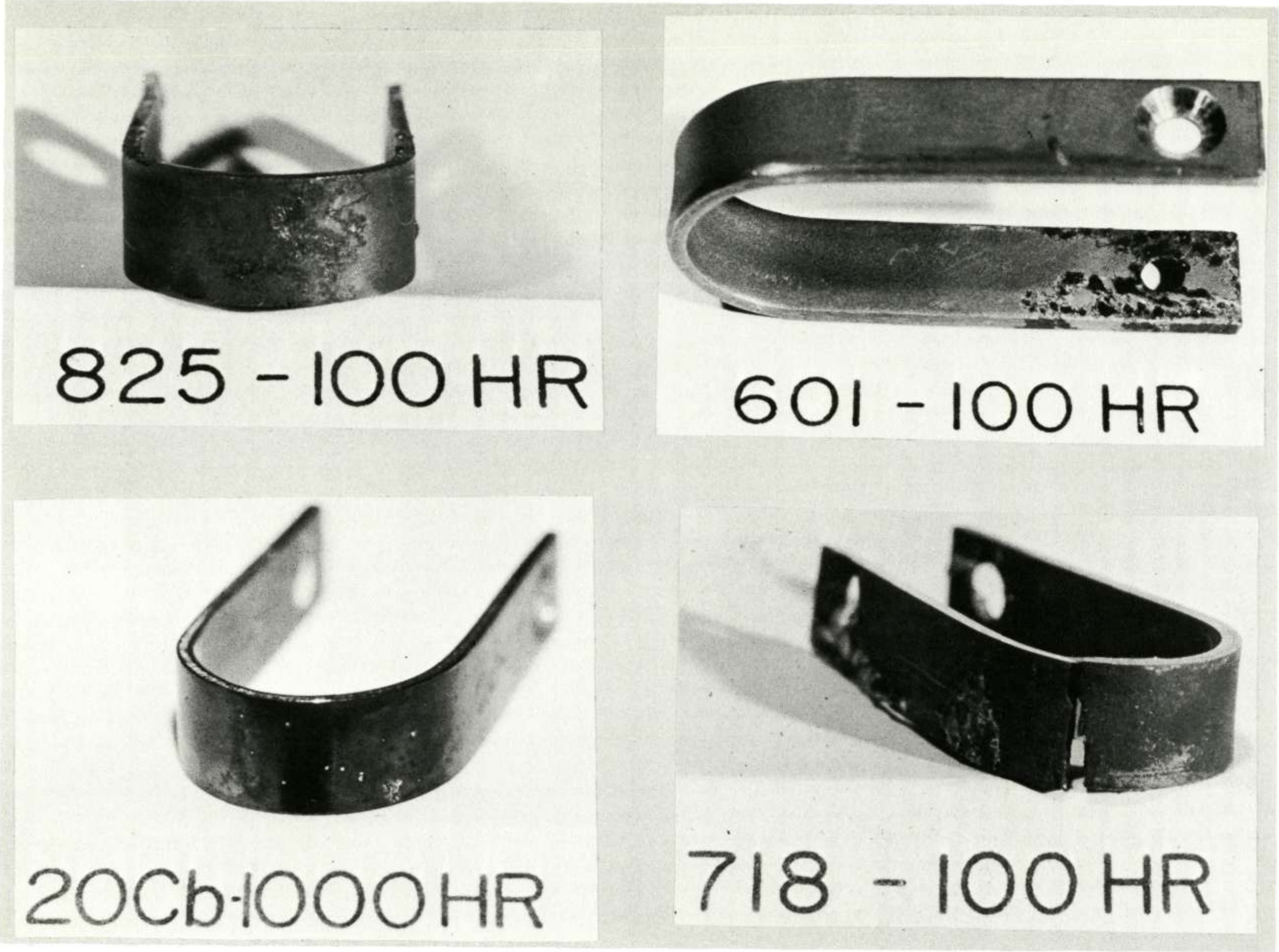


Fig. 30 Corrosion Test Specimens

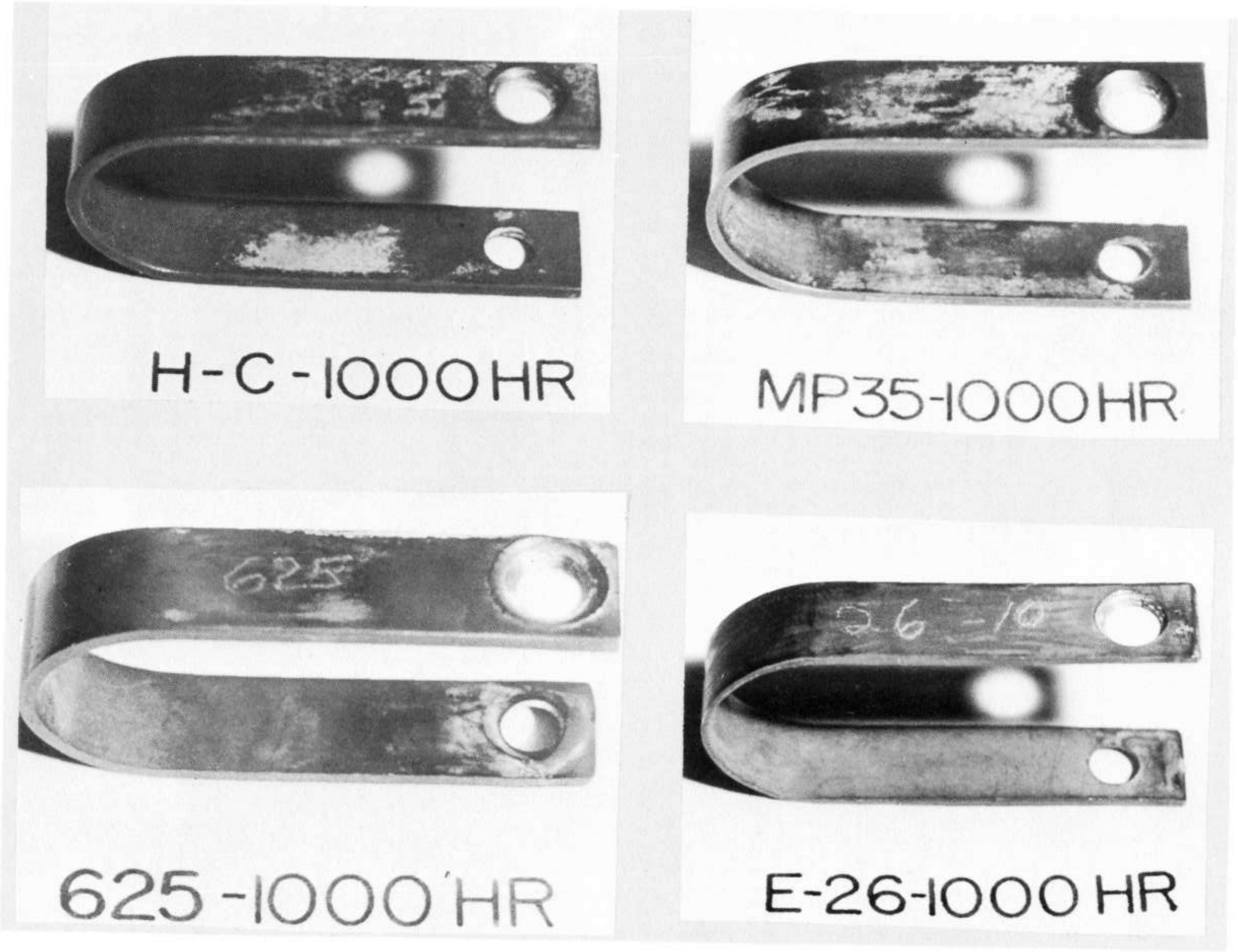


Fig. 31 Corrosion Test Specimens

OXYGEN SUPPLY SYSTEM

Oxygen required by the wet oxidation system can be supplied in a number of ways. A trade study was conducted to determine the best method for oxygen supply. High pressure gas, cryogenic and chemical storage, and high pressure water electrolysis were compared. Low pressure storage or water electrolysis combined with a high pressure pump was initially considered, but analysis of the pump requirement proved this approach to be impractical. Table 14 presents the waste model and oxygen demand used for the trade study.

Two approaches were taken in the study. One assumed that any stored oxygen must be counted as a penalty, i.e., the life support materials balance is closed needing no supplemental oxygen. The second assumed that any oxygen stored can be used by the system and the only penalty to be counted is the added storage weight to achieve high pressure.

Figure 32 presents the weight penalty including power equivalents for oxygen supply systems, assuming no credit for stored oxygen and Figure 33 presents the same data, assuming all stored oxygen can be used by the system without penalty. The data shows that a high pressure water electrolysis system is a clear choice if no credit is taken for stored oxygen. If the oxygen and water produced by hydrogen peroxide decomposition can be used to help balance the life support material requirements, the hydrogen peroxide is the lightest weight system. A brief discussion of each approach is presented below.

High Pressure Gas

A storage pressure of 7500 psi was chosen to reduce residual loss assuming a wet oxidation delivery pressure of 2500 psi. Filament wound tank weight data generated by Hamilton Standard for the Space Shuttle Program was used, resulting in a tank weight of 0.88 pounds of tank per pound of stored oxygen. Unusable oxygen was 0.71 pounds residual per pound of usable oxygen.

Supercritical Cryogenics

Operating pressure of 2500 psi was assumed. Tank weights including supporting equipment were 0.43 pounds of tank per pound of stored oxygen. The tank residual was 0.31 pounds of unusable oxygen per pound of usable oxygen. Insulation of the tanks is critical and if the assumed rate of 4.8 pounds per day is reduced much, boil-off will increase the cryogenic system penalty.

Table 14

Oxygen Supply System Trade Study
Waste Model

Material		Quantity	Oxygen Required
Feces		2.1	2.25
Urine		19.2	
Food		0.7	.05
Personal Hygiene Wipes		0.4	1.30
Utility Wipes		0.6	
Medical Supplies		0.1	
Food Containers		1.5	1.00
Plastic Gloves		0.1	
Filters & Membranes		0.2	
Disinfectant Liquids		0.2	0.2
Flush Water		12.0	nil
Wash Water Residues		12.0	nil
	Totals	49.1 lb/day	4.80 lb/day

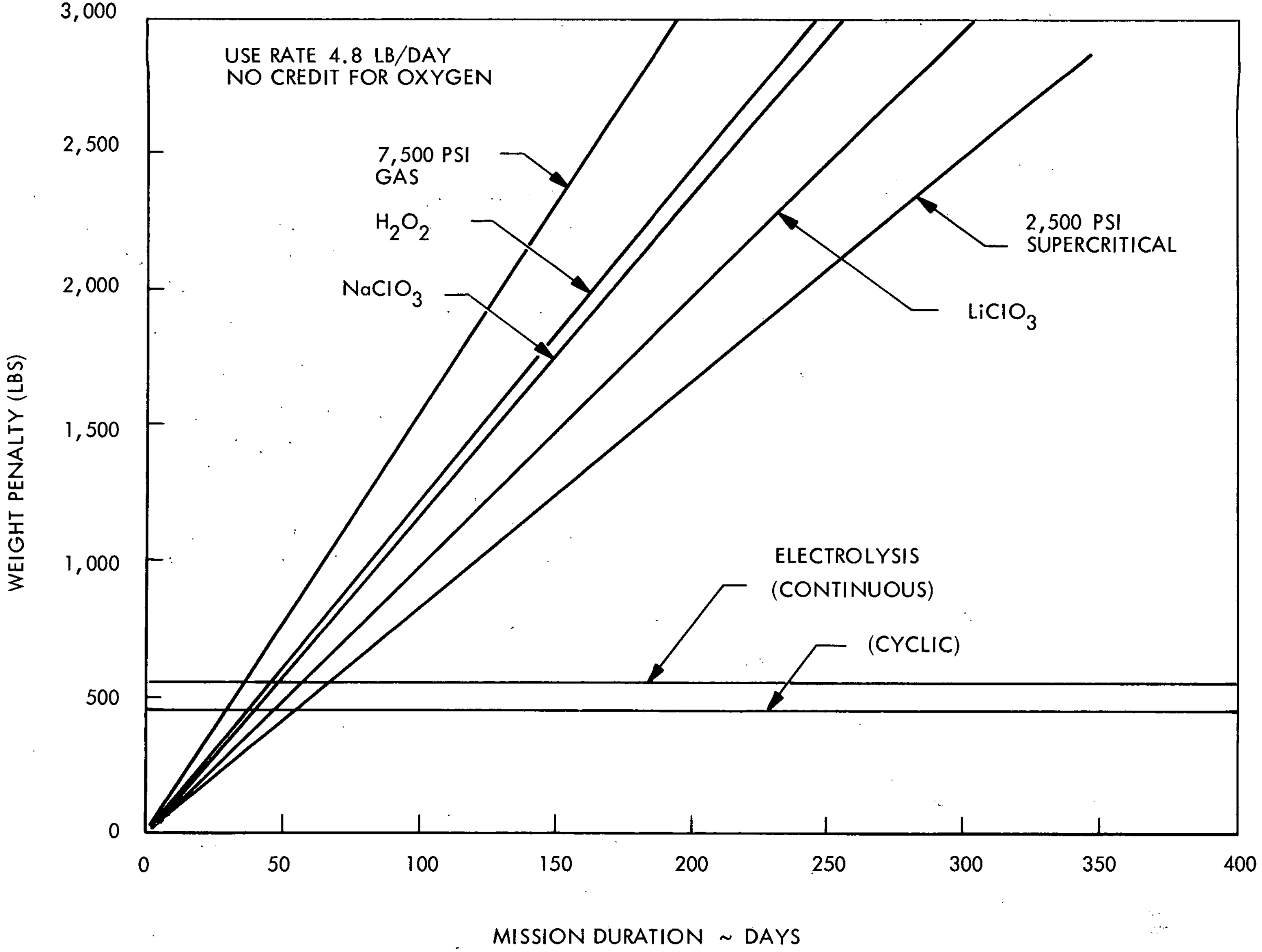


Fig. 32 Oxygen Supply System Trade Study Results - No Oxygen Credit

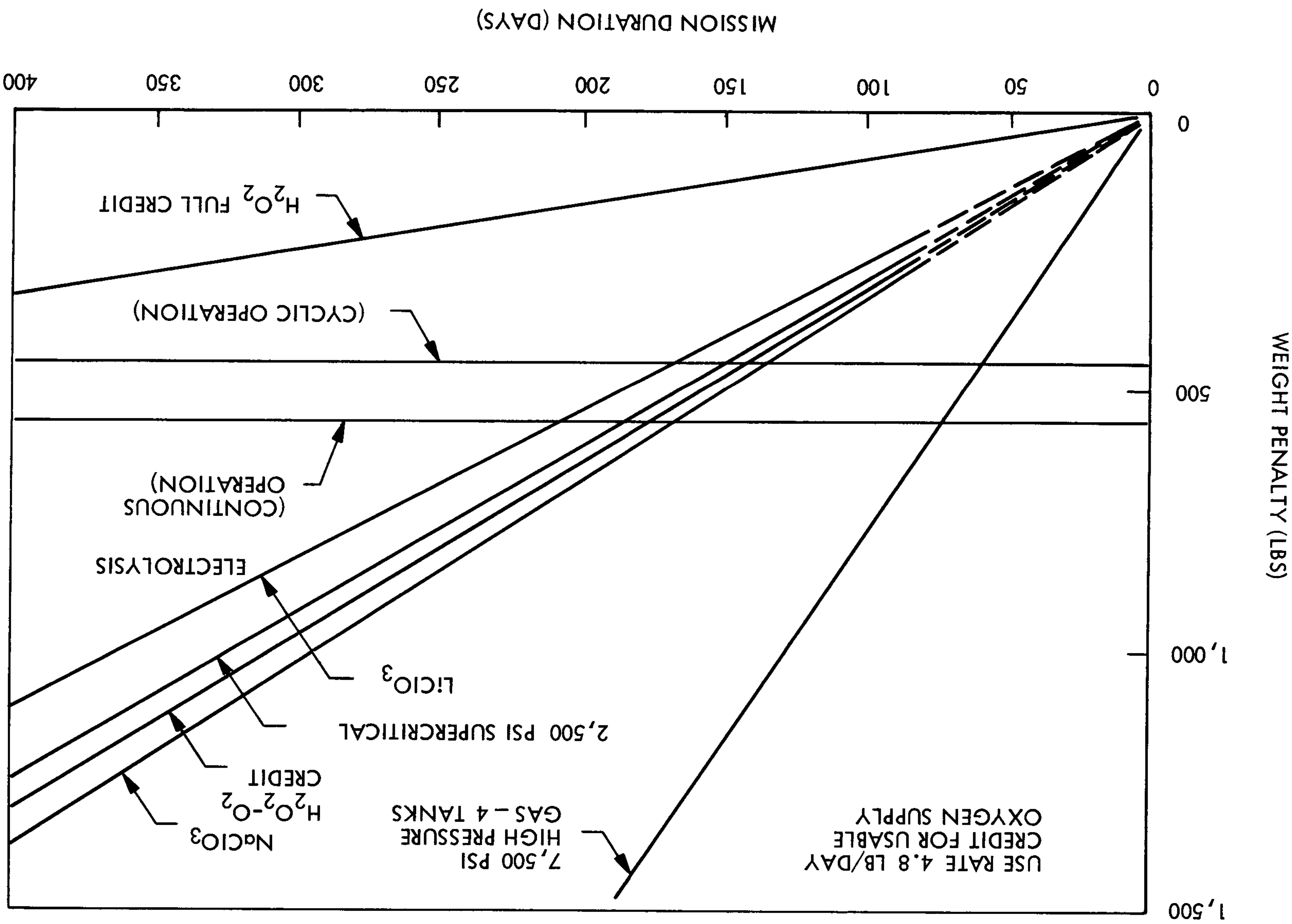


Fig. 33 Oxygen Supply System Trade Study Results – Oxygen Credit

Chemical Oxygen Supply

The chemical oxygen sources listed below were included in the trade study. The oxygen content of each is also presented.

Table 15

Oxygen Content of Oxygen Source Chemicals

Chemical	Oxygen Content (lb O_2/lb chemical)
Lithium Perchlorate	0.60
Lithium Chlorate	0.53
Sodium Perchlorate	0.52
Sodium Chlorate	0.45
Potassium Perchlorate	0.46
Hydrogen Peroxide	0.94 (Oxygen in gas & water) 0.47 (Gaseous oxygen only)

Weight penalties for the chemical oxygen sources were based on the theoretically available oxygen and an assumed storage penalty of 0.1 pound of tankage per pound of chemical for solid materials and 0.2 pounds of tankage per pound of hydrogen peroxide.

High Pressure Electrolysis

In a closed system which requires no supply of oxygen, water electrolysis shows a clear advantage as no stored supplies are required. Both Westinghouse and G.E. have been carrying out studies on the high pressure operation of the solid electrolyte and solid polymer cells, respectively. G.E. has acquired actual operating experience on these cells at pressures as high as 1500 psi. It is felt that operating pressures can easily be extended to the 2500 psi required for wet oxidation application.

ALTERNATE WATER RECOVERY DEVICES

As stated previously, although the dry boiler seemed to work satisfactorily during the DVT, there was sufficient concern about zero gravity operation to warrant a review of current available alternate water recovery devices to establish the best approach to wet oxidation effluent salt removal. The study investigated the following five most promising water reclamation systems, currently under development by NASA for use in manned spacecraft.

Electrodialysis
Vapor Compression Distillation
Vapor Diffusion
Reverse Osmosis
Air Evaporation

Included in this section of the report is a discussion of the advantages and disadvantages of each system, a recommendation as to the best system to be used with wet oxidation, a description and schematic of each system, and a discussion of data sources.

System Comparisons

In the review of the available water recovery systems, certain advantages and disadvantages of each process were evolved, sometimes basic to the process and sometimes associated with the hardware. These factors were combined with weight, volume, power and reliability data presented by Table 16 to support selection of a proposed approach. Figure 34 presents a plot of water recovery system equivalent weight versus mission duration. A brief discussion of the advantages and disadvantages of each system is presented below.

Electrodialysis.- Electrodialysis is a proven commercial process for brackish water cleanup and has been used for decades in this country as well as overseas, particularly in the Middle East. It has not been utilized for direct urine treatment because it cannot process urine organics. Two significant development efforts have successfully used electrodialysis as a salt removal second step following urine pre-treatment. Contact with Ionics and McDonnell Douglas personnel, the system developers, resulted in favorable recommendations for using electrodialysis as a wet oxidation cleanup step. Membrane clogging is the only significant problem that could present itself, but is very dependent upon the characteristics of the treated water. The relatively low weight, volume, and power and reasonable reliability placed electrodialysis in a second rating for wet oxidation cleanup.

Vapor Compression. Vapor compression received very early development as a water reclamation system. Mechanical difficulties, including bearing life, dynamics seal failures, compressor life and evaporator fouling were encountered. Recent advancements in the distillation unit design have apparently solved most of these problems, such that the current system

Table 16

Candidate Water Recovery System Characteristics
6 Man - 1 Year Mission

	Vapor Compression	Air Evap.	Vapor Diffusion	Reverse Osmosis	Electro-dialysis
Weight	203	402	159	99	115
Basic	81	82	108	45	50
Spares	90	63	28	21	25
Expendables	32	257	23	33	40
Power Penalty	44	461	533	47	100
Water Loss Penalty	292	158	438	1170	730
Radiator Penalty	24	77	101	10	10
Total Equivalent Wt.	563	1098	1231	1326	955
Power (Watts)	75	577	901	80	170
Volume (Ft^3)	18	42	13	9	12
MTBF	14000	20000	16000	21000	18000
Parts Count	16	18	18	21	17
Relative Spares	1.15	1.42	1.15	1.00	1.08
Water Recovery %	98	99	97	92	95

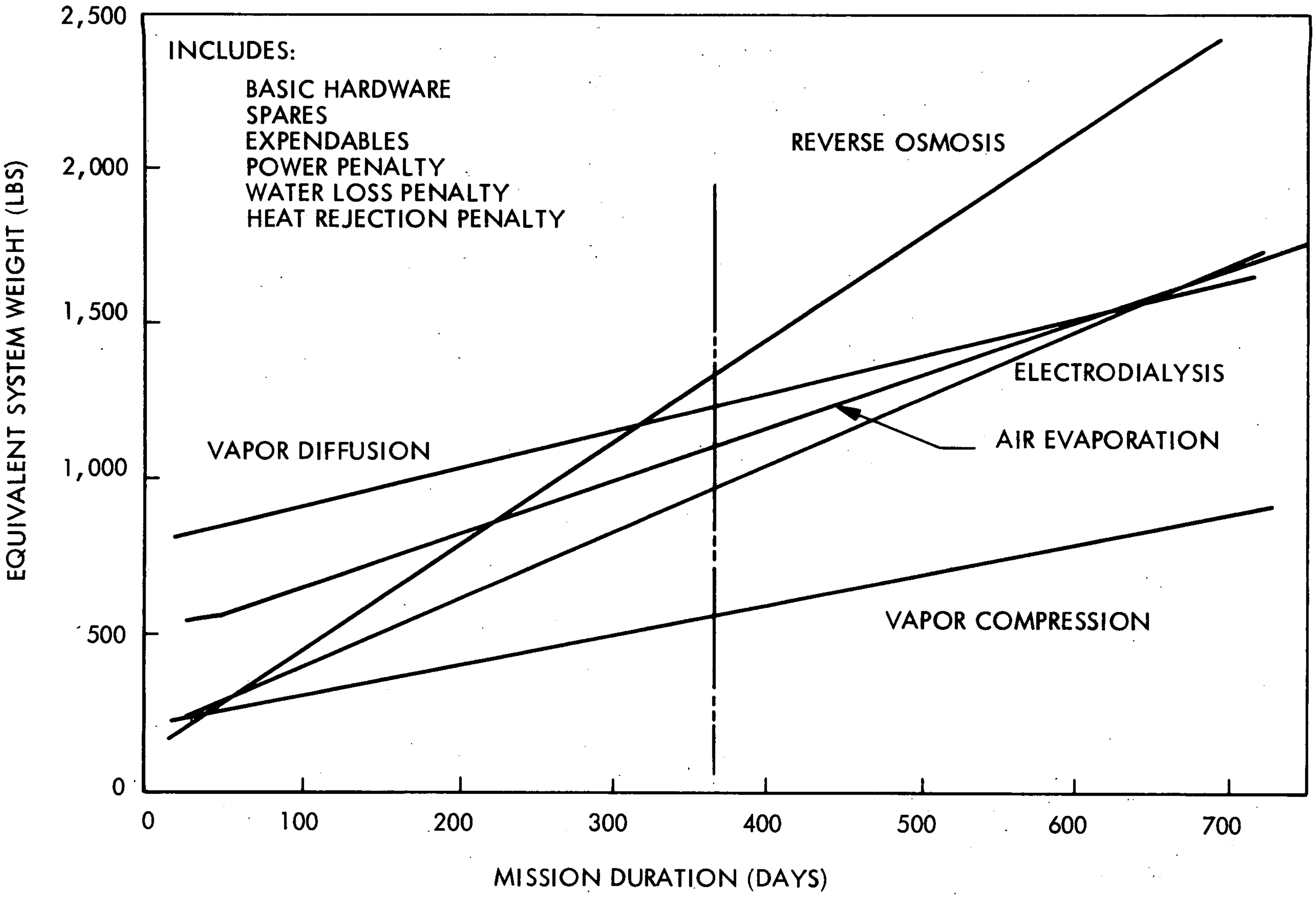


Fig. 34 Water Recovery System Equivalent Weight vs Mission Duration

could be committed to a long duration integrated systems test with a high confidence level. Contacts with Chemtric, the system developer, the NASA-MSC contract monitor, and the SSP water reclamation engineer at Hamilton Standard, resulted in consistently favorable comments. Still maintenance is the only major concern. The advanced stage of development of this system and the very favorable weight, volume, and power characteristics resulted in selection of the vapor compression system as a first choice for wet oxidation cleanup.

Vapor Diffusion.- Vapor diffusion is a relatively new water reclamation development compared to the other systems, and as such, may not have received enough development efforts to place it in a favored position. The power penalty for phase change is an important factor in making the vapor diffusion system one of the heavier approaches. Membrane development to improve life and prevent clogging is required. Nitrogen permeation of the membrane which injects bubbles into the circulating urine stream has also been encountered. These development problems and the weight penalties of the system resulted in a relatively low rating.

Reverse Osmosis.- Reverse osmosis has primarily been used to provide potable water from very dilute sources to give high recovery rates or from relatively concentrated sources to give low recovery rates. For example, a 30 percent recovery rate can be achieved with reasonable cell pressures (200 psi) for municipal water supply, where the amount of salt water that is passed through the unit presents a small penalty to the system. For wet oxidation water reclamation, a cell pressure of 1500 psi is required to achieve 92 percent water recovery. Much higher pressures are required to achieve higher recovery rates. At 92 percent, the water loss penalty is excessive, resulting in a high overall system weight.

A survey of R.O. membrane suppliers resulted in the conclusion that a workable 1500 psi membrane is not currently available. Ionics and Abcor are currently developing 1500 psi membranes but do not consider them as available. Dupont, Dow, Aero-Jet, Eastman, Hercules, Monsanto and Westinghouse are working with pressures below 1000 psi. One significant problem encountered with high pressure membranes is compaction of the surface which impedes flow of water through the membrane. Membrane clogging has also been encountered.

Even if high pressure membranes can be developed, pressures of 4000 psi produce water recovery rates of only 95 percent, which still results in a large water loss penalty for the system. Based on these conclusions, reverse osmosis received a poor rating compared to the other systems.

Air Evaporation.- Air evaporation has come closer to operational use than any other water reclamation system by being used in two long duration manned chamber tests. The system operated satisfactorily and in many respects, is a simple, reliable device, capable of extremely high water recovery. Its primary disadvantages are very limiting, however, The power for phase change, the large volume of wicks and the need for wick handling present severe problems for the system. Feed control of waste water to the wicks to obtain high utilization of the wicks without carryover of waste water to the condenser is another problem.

These operational disadvantages plus the weight penalties resulted in a relatively poor rating for this system.

Electrodialysis System Description

Figure 35 presents a schematic of the electrodialysis system. The following discussion describes operation of the system.

System Charging.- Wet oxidation effluent water* is delivered to the bladder storage tank. A limit switch stops supply if the tank reaches full capacity. If the storage tank is depleted, the waste reclamation unit is shut down. Pressure transfer at approximately 17 psia is utilized to fill and expel to the circulation accumulator.

Filling of the electrodialysis loop is controlled by a limit switch in the bladdered circulation accumulator. A signal is sent to the controller to close the recharge valve and place the potable diverter valve in the recirculation position. Wet oxidation effluent water is thereby pumped from the effluent storage tank through the potable diverter valve, electrodialysis cell, and into the circulation accumulator. When the accumulator is at full charge, the limit switch sends a signal to the controller that deactuates the recharge valve to the open position. The gas side of the accumulator is vented to ambient so that pressure within the accumulator will remain nearly constant.

Process Operation.- The system is now ready for processing. Pump operation, which is continuous unless the E/D system is shut down, provides circulation through the cell in a closed loop. This circulating flow is diluted as salts are removed. When the conductivity falls to a prescribed level, the conductivity sensor sends a signal to the controller to end processing and to initiate potable water delivery.

Brine is delivered from the cell to the brine accumulator by electrochemical potential. A limit switch on this accumulator provides the signal to the controller for brine dump. Dumping is accomplished by a brine pump and shutoff valve. When the accumulator is empty, the limit switch deactivates the pump and shutoff valve.

---

* "Effluent Water" used many times in the report refers to the clear salty water produced by the wet oxidation system which is an input to the water reclamation system.

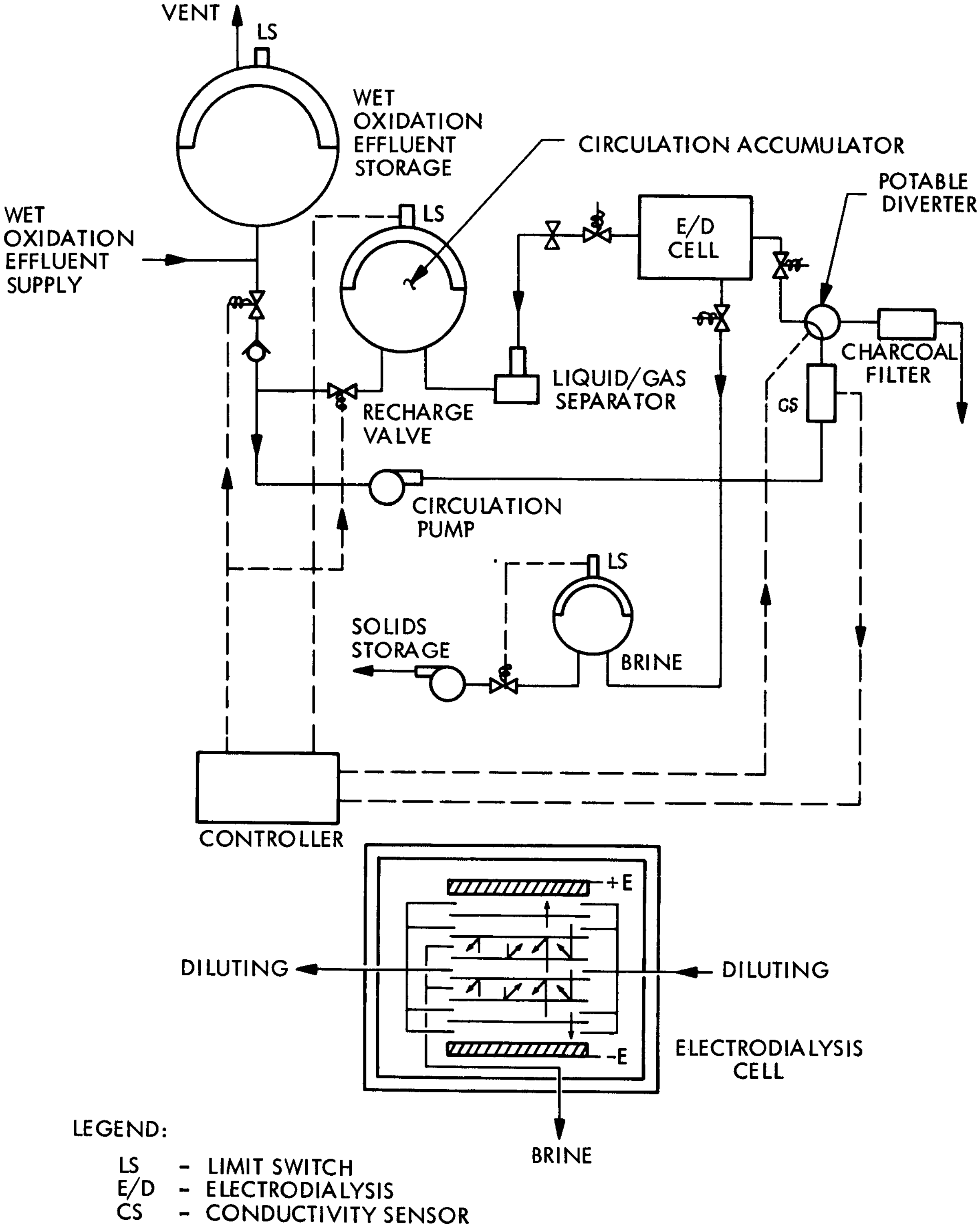


Fig. 35 Electrodialysis System Schematic

An orifice immediately downstream of the cell provides a slightly higher pressure on the dialysate circulation side of the membranes. This has been provided to ensure that if a leak should develop, leakage will be from potable supply to concentrate.

Potable Water Delivery.- Potable water delivery is initiated when the conductivity sensor senses the proper low conductivity in the circulating fluid loop. The controller then actuates the potable diverter valve and potable water is delivered to storage. If, at any time prior to complete dialysate expulsion, the conductivity of the potable water delivery exceeds a specified limit, the controller deactivates the diverter valve so that additional water processing can be accomplished. Expulsion is resumed when proper conductivity is reached.

Potable delivery is terminated by a limit switch in the dialysate accumulator. The controller receives the signal and deactivates the potable diverter valve and simultaneously actuates the re-charge valve. The system is then ready for another cycle.

Vapor Compression Distillation System Description

Figure 36 presents a schematic of the vapor compression distillation system. The following discussion describes operation of the system. Vapor compression is an efficient distillation process to separate potable water from brine. The heart of the system is the distillation unit. The remaining components supply and dispose of liquids, as necessary.

Still Operation.- The still consists of an evaporator, compressor, and condensor. The evaporator and condensor are back-to-back heat exchangers. The compressor isentropically compresses the saturated vapor coming from the evaporator, and delivers it to the condensor. Since the condenser and evaporator are back-to-back, heat of condensation is conducted from the condensor to evaporator where it serves as heat of vaporization. This eliminates the need to supply external sources of energy for heating and cooling.

The evaporator and condenser active surfaces are formed by a single cylindrical drum. The inside surface acts as the evaporator and the outside surface acts as the condensor. This drum is rotated to provide an artificial surface so that evaporative liquid will remain in contact with the evaporator surface and condensation will be readily removed. A second rotating drum that surrounds the condenser collects condensate on its inside surface.

Both drums are integral to one another and rotate on common bearings about a non-rotating shaft. Small tubes routed through the shaft serve as pickup and supply lines. All mechanisms are surrounded by a stationary housing that is integral to the shaft. External dynamic seals are, therefore, precluded.

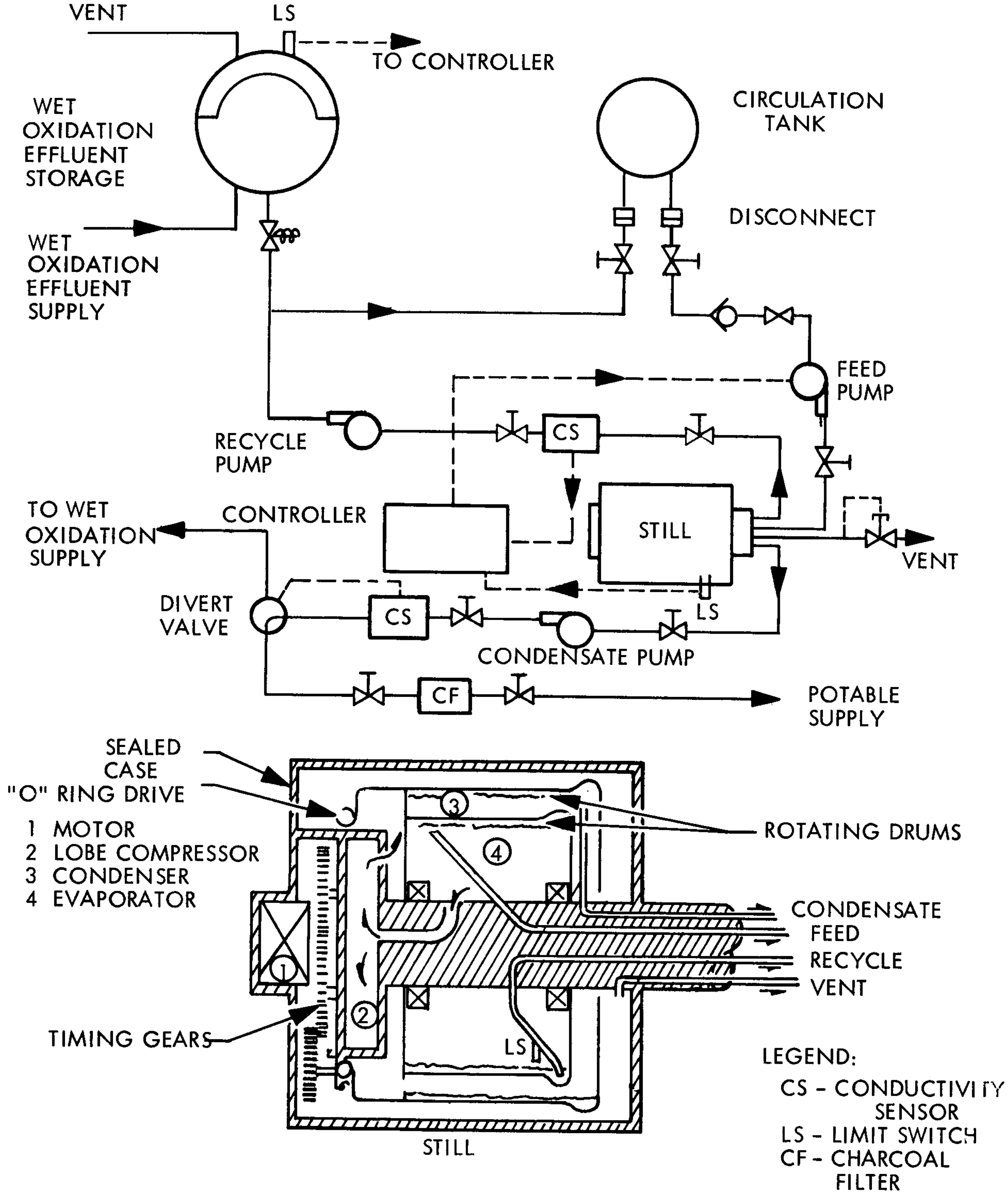


Fig. 36 Vapor Compression Distillation System Schematic

Provisions have been made to remove non-condensable gases from the condenser in order to maintain a low distillation unit pressure to allow evaporation and condensation at near ambient temperatures. A back pressure regulator vents non-condensables and maintains still pressure.

Circulation System.- Processing of effluent water is accomplished on a batch basis. The system is initially filled and as water is processed, make-up waste water is added to maintain the processing liquid at constant volume. Since solutes remain in circulation, the concentration will increase. At a solids concentration of approximately 50 percent, the process is complete.

Brine is continually circulated through the evaporator of the still. This is done to maintain a thin film on the evaporator to achieve high boiling rates and to facilitate dumping of the concentrated brine when a batch run is completed. Removal of all of the concentrated brine from the evaporator is therefore not necessary, since it represents a small portion of the total circulated brine. At the end of a batch cycle when the brine has reached 50% solids concentration, the circulation tank is removed and placed in storage. A new circulation tank is installed and waste water from the wet oxidation effluent storage tank is supplied to the circulation tank. The circulation tank is maintained full by constant feed from the wet oxidation effluent storage tank.

Circulation is provided by peristaltic pumps driven by a common motor. A circulation pump pulls liquid from the evaporator at less than 1 psia and delivers it to the circulation tank at ambient pressure. The feed pump does not supply a head but serves to meter flow into the evaporator. The circulation pump capacity exceeds the feed pump capacity so that the evaporator remains nearly dry. A thin film is maintained by a small dam at the end of the evaporator drum. The circulation pump sump is therefore many times dry and the circulation pump cavitates. This presents no problem to the peristaltic pump, which will reprime itself when water is sucked from the evaporator sump.

Potable water is removed from the still with a peristaltic pump driven by the same motor as the circulation and feed pumps. This pump is operated at a rate in excess of the maximum condensation rate. This assures that condensate will be removed in the event of a buildup in the condenser. Normal operation for the potable pump is to ingest vapor as well as liquid, since it pumps at a rate greater than the condensation rate. When vapor is ingested, it is compressed to a higher pressure where it condenses to form liquid. The peristaltic pump is capable of this two-phase pumping action.

Brine conductivity is continually monitored in the circulation loop. When 25 percent concentration is reached, a signal to the controller activates a timer. After the proper elapsed time, corresponding to 50 percent concentration, processing is terminated by the controller. At this time, the circulation tank is replaced and the system reactivated manually.

Potable Water Delivery.- Potable water is continually removed from the still. Water quality is monitored with a conductivity sensor. If conductivity becomes too high, the diverter valve is actuated to deliver the impure water to effluent storage.

Vapor Diffusion System Description

Figure 37 presents a schematic of the vapor diffusion system. The following discussion describes operation of the system.

Effluent Circulation.- Wet oxidation system effluent water is supplied to the bladder storage tank as it is produced. A limit switch stops supply if the tank reaches full capacity. If the storage tank is depleted, the water reclamation unit will be shut down. A back pressure regulator on the gas side of the bladder permits ullage gas bleedoff during fill and a pressure regulator in the pressurization line maintains pressure during expulsion. This tank is continuously being filled, even while water is being fed to the circulation tank.

Fill and drain of the circulation tank are controlled by position indicators located in the tank that direct a feed shut-off valve for fill and a solids dump valve for drain. Initial charging is terminated when the full position indicator is activated. During run operation, the circulation pump draws fluid from the circulation tank through the evaporation passage of the vapor diffusion unit where water wets the membrane and vapor diffuses through nitrogen gas to the porous cold plate condensor. Condensed water is forced through the porous plate by a slight positive pressure in the nitrogen passage. The condensed water enters the product water chamber and is drawn from the cell by a product water delivery pump. A conductivity cell measures product water salt content and, if satisfactory, allows delivery to the potable water storage. If not, the product water is recycled to the effluent storage tank.

Wet oxidation effluent water leaving the vapor diffusion cell passes through the pump to a conductivity cell, a solids diverter or dump valve and back to the circulation tank. The circulation tank is maintained full by feed from the wet oxidation effluent tank, which is controlled by a sensor in the circulation tank. When the conductivity cell indicates 25% concentration by weight has been achieved, the controller disables circulation tank fill. The circulation tank will then empty as waste is processed by the system. A tank position indicator corresponding to approximately 50% concentration sends a signal to the controller that activates a solids divert valve. As this highly concentrated salt water solution is removed, the tank will further empty until a tank empty position indicator signals the controller to start a new batch cycle by refilling the circulation tank.

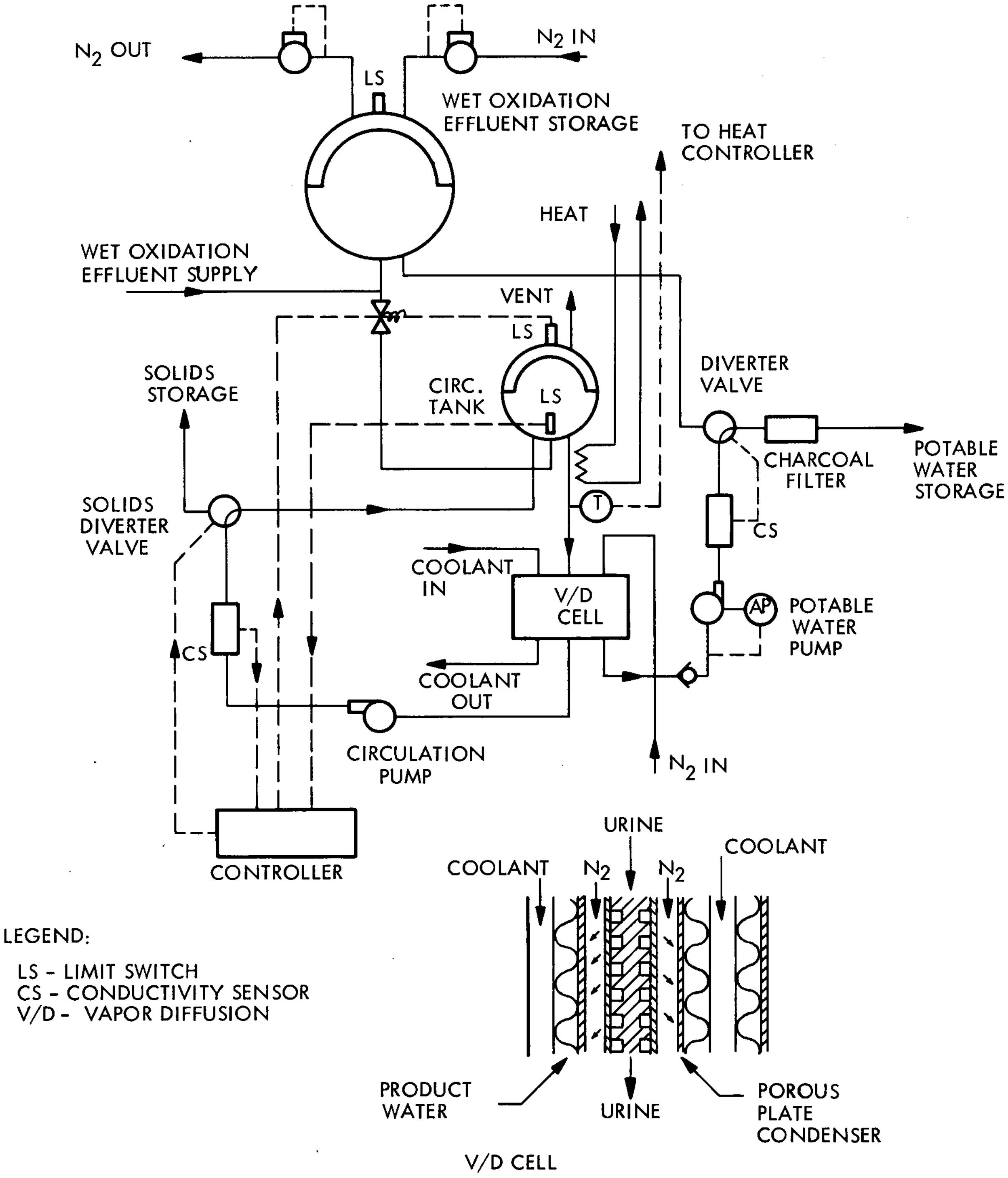


Fig. 37 Vapor Diffusion System Schematic

During all of these operations, the circulation pump is in operation. It provides the driving force for circulation that is necessary to prevent local concentration buildup. Circulation takes place in a continuous loop from the tank, to cells and back to tank. During the solids divert operation, it provides the suction required to expel the tank.

Potable Water Circuit.- Potable water is removed from the cell by a pump. Pump suction pressure is kept below diffusion gap pressure (ambient) and above potable water vapor pressure (1 psia) by use of a pressure sensor. Pump operation is begun when the upper limit of the pressure band is reached and terminated when the lower limit is reached. Charcoal cleanup is provided in the potable line to remove odors.

Nitrogen Gap Circuit.- Regulated nitrogen gas is introduced to the diffusion gap. Gap pressure is maintained as close to module effluent pressure as practical.

Temperature Control.- The distillation driving force with this system is the difference in vapor pressure between the feed water and potable water. Effluent water is heated to 150°F and potable water is cooled to 50°F to effect a reasonable vapor pressure differential. Effluent water is heated at the circulation tank outlet. Temperature control is maintained by a temperature sensor. Potable water is cooled inside the module on the condenser plates. A fluid coolant loop is used for heat transport. Temperature sensors regulate coolant flow.

Reverse Osmosis System Description

Figure 38 presents a schematic of the reverse osmosis system. The following discussion describes operation of the system.

Reverse osmosis is a method of separating water from water/salt solution. Osmosis will normally produce a flow from the dilute solution to the concentrated solution. In order to reverse this process, to obtain potable water from effluent water, the effluent water pressure is increased to a value above the osmotic pressure. The requirement for a successful system is that the solution be sufficiently pressurized and then dumped when proper concentration is achieved. The system shown also includes solution recirculation to prevent local concentration and consequent reduced flux.

Pressurization System.- Pressure is exerted on the effluent water by use of a hydraulic system. High pressure hydraulic fluid (water) is injected on the back side of the bladder in the recirculation tank. A reversible hydraulic pump supplies pressure and an air accumulator is placed in the high pressure circuit to reduce pressure surges and reduce pump duty cycles.

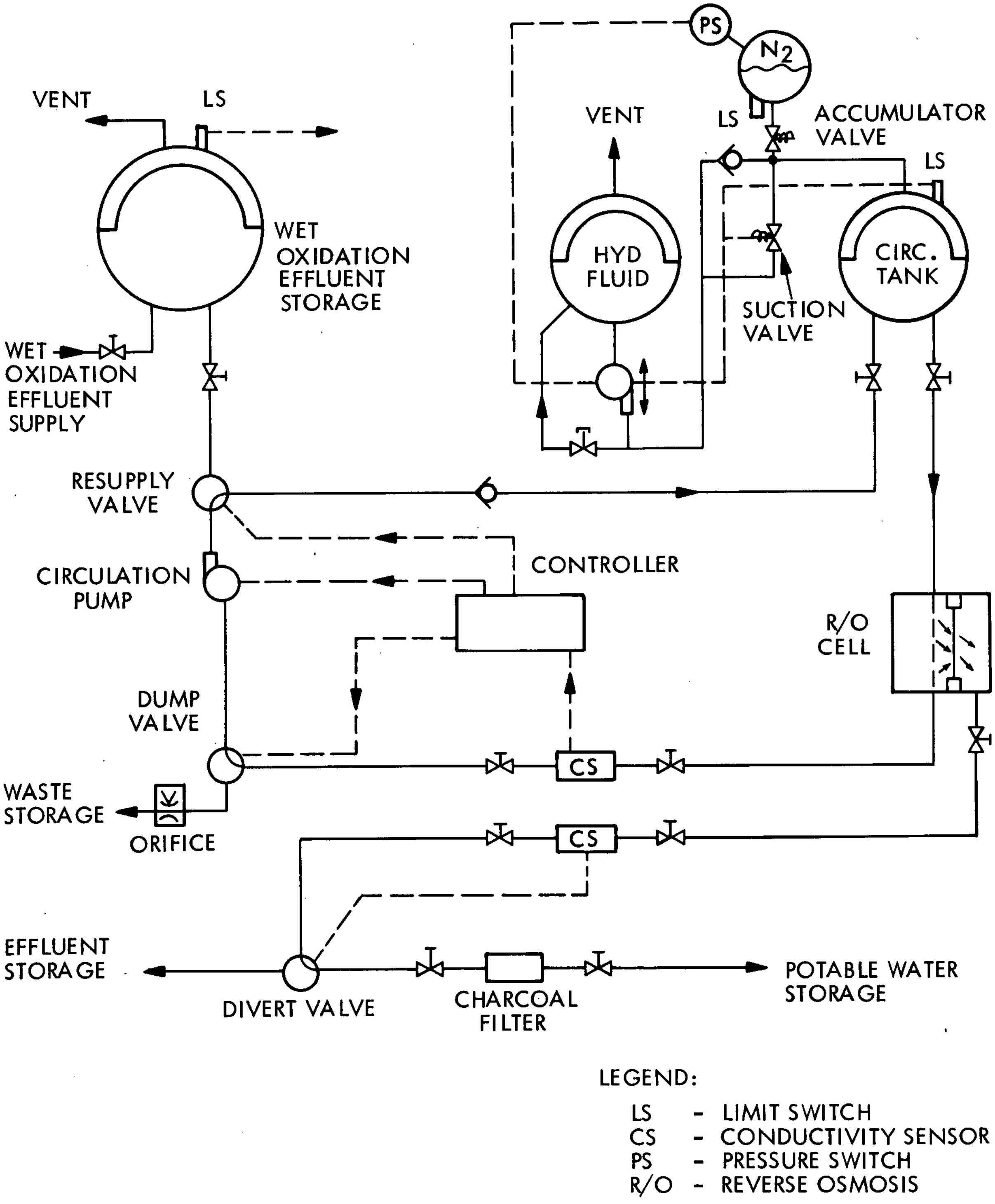


Fig. 38 Reverse Osmosis System Schematic

System pressure is maintained within an operating band of approximately 1550 ± 50 psia. As water is drawn from the concentrated effluent water side, hydraulic fluid from the accumulator flows into the recirculation tank as replacement. The loss of accumulator fluid results in lower pressure. When the lower pressure limit is reached, the hydraulic pump is energized by the accumulator pressure switch. The same pressure switch also shuts down the pump when the upper limit is reached.

Circulation System.- Effluent water is processed in batch form, that is, the circulation tank is charged and processed to completion prior to injecting additional fluid into the circulation system.

During processing, the brine is continually circulated within a closed loop from the circulation tank, through the R/O cell, to a circulation pump and back to the tank. Concentration is monitored with a conductivity sensor. When 25 percent concentration is reached, the conductivity sensor activates a timer within the controller. After the proper elapsed time, corresponding to 50 percent concentration, processing is terminated by the controller. This is done by actuating the dump valve. The air accumulator will provide the driving force for expulsion. A limit switch in the circulation tank signals the controller that dumping is completed. The system is now ready for a new batch.

Recharging is initiated by the controller. The accumulator valve is closed, the suction valve is opened, the dump valve is deactivated, the recharge valve is actuated, and hydraulic pump is reversed. Hydraulic fluid will then be drawn from the circulation tank and delivered to the hydraulic tank. Suction of hydraulic fluid draws effluent water from the wet oxidation effluent storage tanks. A limit switch in the circulation tank is actuated when the tank is full. The controller then returns all of the valves and hydraulic pump to the process position and the cycle is completed. During all operations, the circulation pump runs continually.

Potable Water Delivery.- Potable water is continually drawn from the cell by a peristaltic pump except for those short periods of time when the recirculation tank is being recharged. The pump will cavitate when flow stops, but will not result in adverse affects. Water quality is continually monitored with a conductivity sensor. If conductivity becomes too high, the divert valve is actuated to deliver the impure water to effluent storage for reprocessing.

Air Evaporation System Description

Figure 39 presents a schematic of the air evaporation system. The following discussion describes operation of the system.

The air evaporation system provides water reclamation by passing heated air over fiberous wicks laden with waste water to achieve evaporation of the water with condensation of the vapor in a coolant fed heat exchanger. Condensed water is removed from the circulating air stream and is delivered to potable water storage.

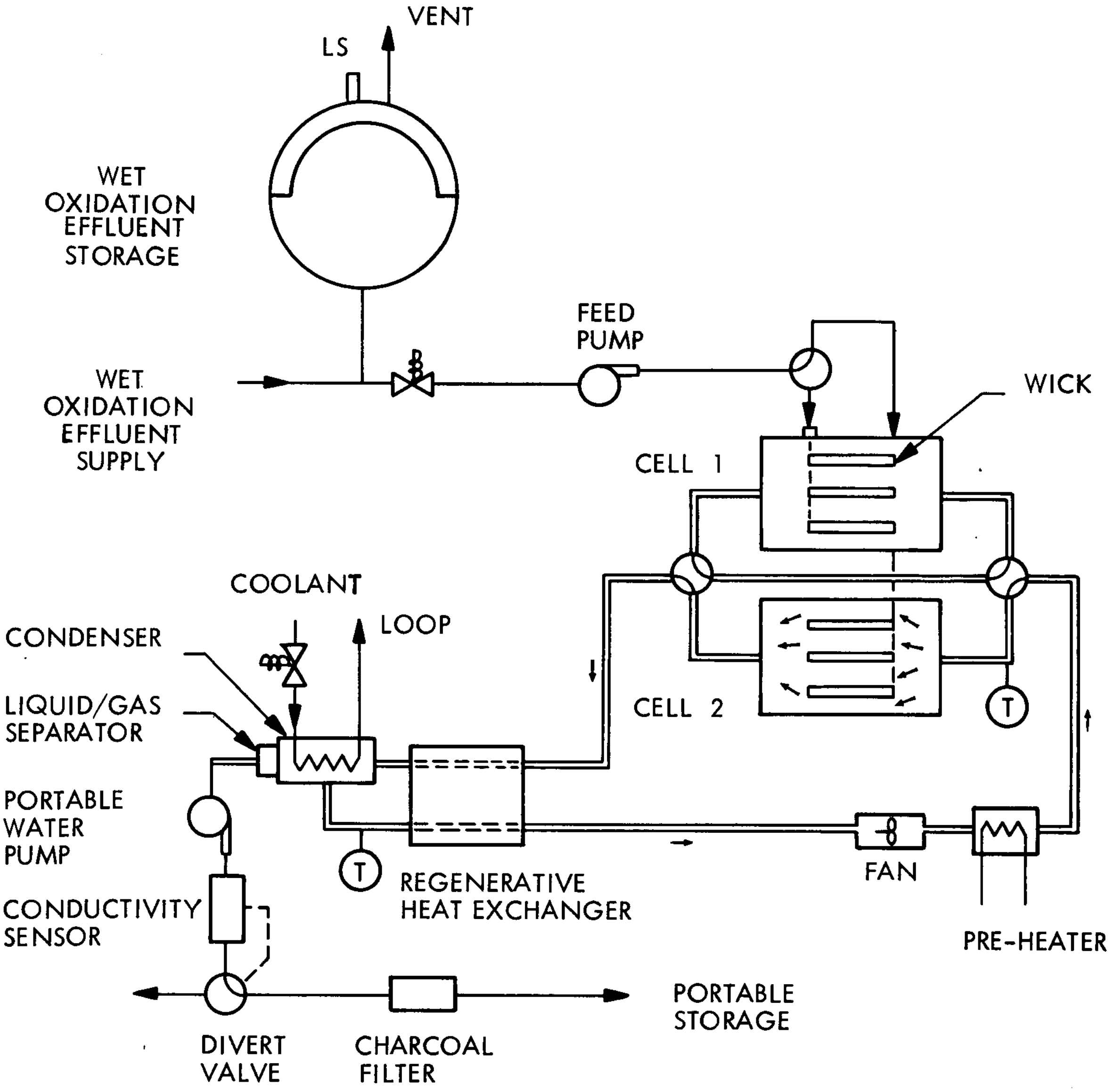


Fig. 39 Air Evaporation System Schematic

Air Circulation Loop.- The air circulation system is a closed loop. Air from the fan is heated electrically before it flows through the wicks where water is absorbed. The moisture laden air then passes through the hot side of a regenerative heat exchanger where partial condensation and cooling takes place, to the condenser where final condensation takes place, to the cold side of the regenerative heat exchanger, where reheat is accomplished and back to the fan.

The pre-heater is used to increase the water holding capability of the air. The regenerative heat exchanger, a recent addition to the system, reduces thermal power requirements below that of any present prototype. Warm, water-saturated air upstream of the condenser is cooled in this heat exchanger and partially condensed by the chilled air leaving the condenser. This recovery of thermal energy results in reduced pre-heater energy and reduced condenser load.

The condenser consists of a heat exchanger with a water separator and spacecraft coolant loop. A temperature control valve modulates coolant flow as a function of air outlet temperature.

Feed System.- Wet oxidation system effluent water is stored in a bladdered tank that is vented to ambient. A peristaltic pump provides feed to the wicks. Feed rate is pre-determined and independent of upstream and downstream pressure. It is set to prevent wick flooding or drying.

Potable Water Delivery.- Potable water is continually removed from the water separator by use of a pump. Water quality is monitored with a conductivity sensor. If conductivity becomes too high, the divert valve is actuated to deliver the unpotable water to effluent storage for reprocessing.

Wick Replacement.- Wick replacement is currently accomplished by manual removal and installation of fresh wicks. Complete wick usage as well as waste water feed rates to the wicks during operation are accomplished by maintaining a record of total water processed through a given set of wicks. Overfeeding will result in carryover of contaminated water to the condenser. Underfeeding will result in poor utilization of the wicks.

Data Sources

In order to establish a basis for system comparison, a mission module was synthesized. A compromise on duration was made between the short early applications and longer, more distant future applications. Important parameters are listed below:

Mission duration	1 year
Crew Size	6 men
Heat rejection penalty	
direct to coolant	.054 lb/BTU/hr
Power penalty	
continuous regulated DC (solar cell)	591 lb/KW
Water	
Process time	18 hr/day
Process rate	40 lb/day
Solute concentration	1.7%
Organic solutes	neg.
Particulates	neg.
Ammonia content	neg.

The systems described in the previous section were based on the above mission data and the latest configuration data from the water recovery system developers. The most significant factors characterizing the wet oxidation water recovery problem is the absence of organic material in the effluent water and absence of potentially hazardous particulate matter.

The summary of the systems characteristics, shown in Table 16, was developed from the following data and listed references:

Electrodialysis (Ref. 4, 5, 6, 7)
Vapor Compression Distillation (Ref. 5, 6, 7)
Vapor Diffusion (Ref. 5, 6, 7, 8, 9)
Reverse Osmosis (Ref. 5, 6, 7)
Air Evaporation (Ref. 5, 6, 7, 10)

System weight is presented as basic system hardware without redundancy, plus spares that include redundant components and expendables. Weights as presented in the referenced literature were scaled to reflect differences in process rate, mission length, solids content, and required system modifications. The high weight of vapor compression spares is primarily due to still components and the high air evaporation expendables are due to wick replacement.

Power requirements were calculated and compared with data obtained from the literature and verbal conversations. High power consumption for both air evaporation and vapor diffusion systems is due to the fact that heat of vaporization and condensation must be supplied thermally. It should be noted that the air evaporation penalty is significantly less than the vapor diffusion penalty because:

- The large penalty for heating and cooling circulation air has been drastically reduced for the regenerative heat exchanger, and
- Heat leak from the vapor diffusion evaporator to condenser is high.

Water loss penalty is that water that cannot be recovered by the process during the entire mission duration. It can have a large influence upon total effective weight and is the reason that the reverse osmosis system is so heavy.

Volume includes basic hardware weight, spares, and expendables. These values were scaled down from the literature and adjusted for mission duration and process rate. The significant fact is that the volume of wicks and holders comprises most of the volume of the air evaporation system, which is quite bulky compared to the other systems.

Mean time between failures has been taken from reference. Since there have been modifications to the systems from that shown, these numbers serve to indicate relative rankings.

Parts count includes only those components that are active in the respective systems. Redundant components are not included. Relative spares indicate the number of redundant and spares components required for the mission.

Water recovery factors, upon which water loss is based, are the result of discussions with equipment designers and the literature. They have been adjusted to the solute content of wet oxidation effluent water and are present day state of the art.

Effluent Water Recovery Tests

Based on the results of the trade study, it was recommended that the vapor compression unit and an electrodialysis unit be tested with wet oxidation effluent water. To support such tests, ten gallons of wet oxidation effluent water were produced in a five gallon, batch type stirred reactor vessel very similar in design to the one liter batch reactor. After numerous attempts to effect a seal on the pressure vessel cover, ten gallons of water were produced from urine/fecal mixtures. Five gallons have been sent to NASA-Langley for evaluation in an electrodialysis unit and five gallons have been sent to Chemtric, the current developer of the vapor compression distillation unit.

AMMONIA REMOVAL

Product water from the prototype reactor during DVT was analyzed and found to contain only small amounts of acid organic salts, 1.7 percent by weight inorganic salts, and relatively large quantities of ammonia (0.55% by weight). After boiling and condensing, to simulate a water cleanup step, the condensate was analyzed and found to contain only ammonia. Its presence in large quantities represented the only apparent problem in producing potable water by the wet oxidation process. An analytical and experimental study of ammonia removal was conducted to resolve this problem. A review of ammonia removal methods resulted in consideration of the following approaches.

Acid Neutralization

For a one-year, six-man mission, 67 pounds of ammonia must be removed. Hydrochloric acid could be used to neutralize the ammonia with subsequent removal of the NH_4Cl salt product. Forty pounds of HCl would be required each 90-day resupply period. The storage of the hazardous acid presents a severe problem with this approach. Other possible problems which were verified by laboratory tests are that regardless of adjusted pH, some breakdown of the ammonia compounds and acid organic salts occurs on distillation.

Direct Reduction

Ammonia can be directly reduced to H_2 and N_2 by high temperature, catalytic processes. High temperatures (1800°F) are required to achieve nearly complete ammonia reduction resulting in high power demands. Formation of oxides of nitrogen from trace quantities of oxygen in the feed gas is very likely. Shell 405 I2 or Englehard 1/2% Ruthenium are attractive catalysts for this process.

Catalytic Oxidation

Ammonia can be oxidized to N_2 and H_2O over a catalyst in the presence of oxygen. Work in this area has shown some promise, (Ref. 11), but formation of oxides of nitrogen is highly probable.

Regenerable Absorption

In contaminant removal systems, ammonia is removed by absorption on treated charcoal or other treated substrates. Indications are that a chemical equilibrium exists which permits the adsorption of ammonia at low temperatures with decomposition of the chemical intermediary formed in the absorption process by a rise in temperature. The ammonia desorbed from the bed would be dumped to space, which presents a major disadvantage of this process.

Electrochemical

Ammonia can be converted to N_2 and H_2O vapor in an electrochemical cell. Tests were run using platinum electrodes at a number of current densities. Strong NO_2 formation accompanied the ammonia conversion. Indications (Ref. 12) are that black platinum electrodes have been successful but that process control is very "touchy". The low probability of success eliminated this approach from further consideration.

Electrodialysis

Electrodialysis treatment of water will remove ammonia ions. Increased batch times over those required for Na^+ ion removal would be required. Higher voltages as solution conductance falls would also be required. It is unlikely that 1 ppm can be achieved and 10 ppm would be difficult. If the process were carried to 100 ppm, with final cleanup in a fixed non-regenerative ion exchange bed, a feasible system may result.

Wet Oxidation Reaction

Catalyst tests conducted early in the laboratory test program showed a reduction in ammonia produced by the wet oxidation process. Catalysts used in these tests were selected to investigate reduction in process temperature and pressure, not ammonia reduction. Since a small quantity of powdered or liquid catalyst added to the feed slurry offered the simplest solution to the ammonia problem, the greatest effort was expended in this area. Table 17 presents the results of 15 test runs in the one liter batch reactor that evaluated ammonia removal catalysts. The tests were run by charging 460 cc of fecal/urine slurry (10 percent by weight feces), adding the catalyst, installing the reactor head, charging to a pressure of 800 psi with oxygen, heating to 550°F (2200 psi), reacting for 1-1/2 hours and cooling to room temperature. The resultant water was checked for pH and was distilled and filtered through charcoal when test results warranted. The distillate and filtered water were analyzed for organics and pH. Runs 6 through 14 evaluated solid catalysts and showed Ruthenium Black to be a very effective catalyst for ammonia removal. Ammonia concentrations in the baseline run 4 were very high (approximately 0.6% by weight). Ammonia concentrations in the catalyzed run 12 effluent water were 17 mg/liter (0.0017% by weight), showing up as ammonium ion because of the acidic nature of the water. Analysis of this distillate showed 19 mg/liter of acetic acid as the only contaminant. This was readily removed by charcoal filtration.

Runs 15 through 23 searched for a soluble catalyst to make catalyst addition easier in the final system. Very small quantities of Ruthenium Trichloride (Run 21) produced results very similar to Ruthenium Black (Run 12). As little as 0.5 pounds of Ruthenium Trichloride is required for the urine/feces trash loading for a 6-man one-year mission.

Table 17

Ammonia Removal Test Results – One Liter Batch Reactor

Run No.	Type of Run	Water pH Reactor Eff.	Water pH Distillate	Water pH Charcoal Filtered	Remarks
4	Baseline-No catalysts	8.2	9.9	---	High ammonia in distillate.
6	1.0 gm Ruthenium Black	3.8	3.5	4.5	Distillate contained small amount of organics - charcoal removed them.
10	0.5 gm Ru Bk, Pd Bk, $CuCl_2$	5.6	4.5	---	18 mg/l acetic acid in distillate.
11	0.5 gm Platinum Black	6.0	5.3	---	16 mg/l acetic acid in distillate.
12	0.3 gm Ruthenium Black	5.1	4.0	5.6	19 mg/l acetic acid in distillate. Removed by charcoal.
13	1.0 gm Manganese Dioxide	8.1	9.4	7.7	High ammonia in distillate.
14	1.0 gm Fumed glass	8.2	9.0	7.5	High ammonia in distillate.
15	1.0 gm $CuCl_2$, 1.15 gm $FeCl_2$	8.0	---	---	High ammonia - not analyzed further.
16	1.0 gm $CuCl_2$, 0.6 gm $FeCl_2$, 0.76 gm $NiCl_2$	9.0	---	---	High ammonia - not analyzed further.
18	0.6 gm Ruthenium Trichloride	4.1	3.2	4.3	18-20 mg/l acetic acid in distillate. Removed by charcoal.
19	0.6 gm Platinum Chloride	6.4	9.3	8.0	High ammonia carryover in distillate.
20	0.1 gm Ruthenium Trichloride	3.7	3.8	6.3	18-20 mg/l acetic acid in distillate. Removed by charcoal.
21	0.01 gm Ruthenium Trichloride	4.4	3.5	5.0	18-20 mg/l acetic acid in distillate. Removed by charcoal.
22	3.0 gm LiOH, 0.6 gm Ruthenium Trichloride	7.6	9.3	---	High ammonia - not analyzed further.
23	1.0 gm Pt Triphenylphosphine	6.4	8.4	---	High ammonia - not analyzed further.

SOLIDS GRINDER

The development of a spacecraft solids grinder was initiated during this contract. The purpose of the grinder was to pulverize biologically contaminated spacecraft wastes and deliver them to a storage tank so that they could be pumped into the wet oxidation reactor for processing. The work described in this report consisted of a survey of available grinder designs, design and fabrication of a laboratory model grinder and tests to define a grinder system approach and design requirements for a spacecraft grinder.

The survey of available grinder designs included cutters, ball mills, choppers, hammer mills, and shredders. The grinder design that seemed the most adaptable to spacecraft design was the Urschel Laboratories Comitrol Cutter. Figure 40 presents a photograph of the commercially available cutting head, impellar and housing. The material to be ground is fed into the center of the rotating impeller, which forces it outward through the cutting head to the outlet duct.

The first step in the development program was to establish a waste model, prepare a sample, send it to Urschel Laboratories and have them run the wastes through a number of their cutting heads to select a preferred cutter design. A waste model was established from published spacecraft housekeeping study results (Ref. 13) and is presented by Table 18.

Table 18

Spacecraft Waste Model for Grinder Development

Material	Six-Man Crew Quantity (lb/day)
Feces	2.1
Urine	19.2
Flush Water	12.0
Waste Food	0.7
Food Containers	1.5
Personal Hygiene Wipes	0.4
Utility Wipes	0.6
Medical Supplies	0.1
Plastic Gloves	0.1
Disinfectant Liquids	0.2
Filters and Membranes	0.2
Wash Water Residue	12.0
Total	49.1

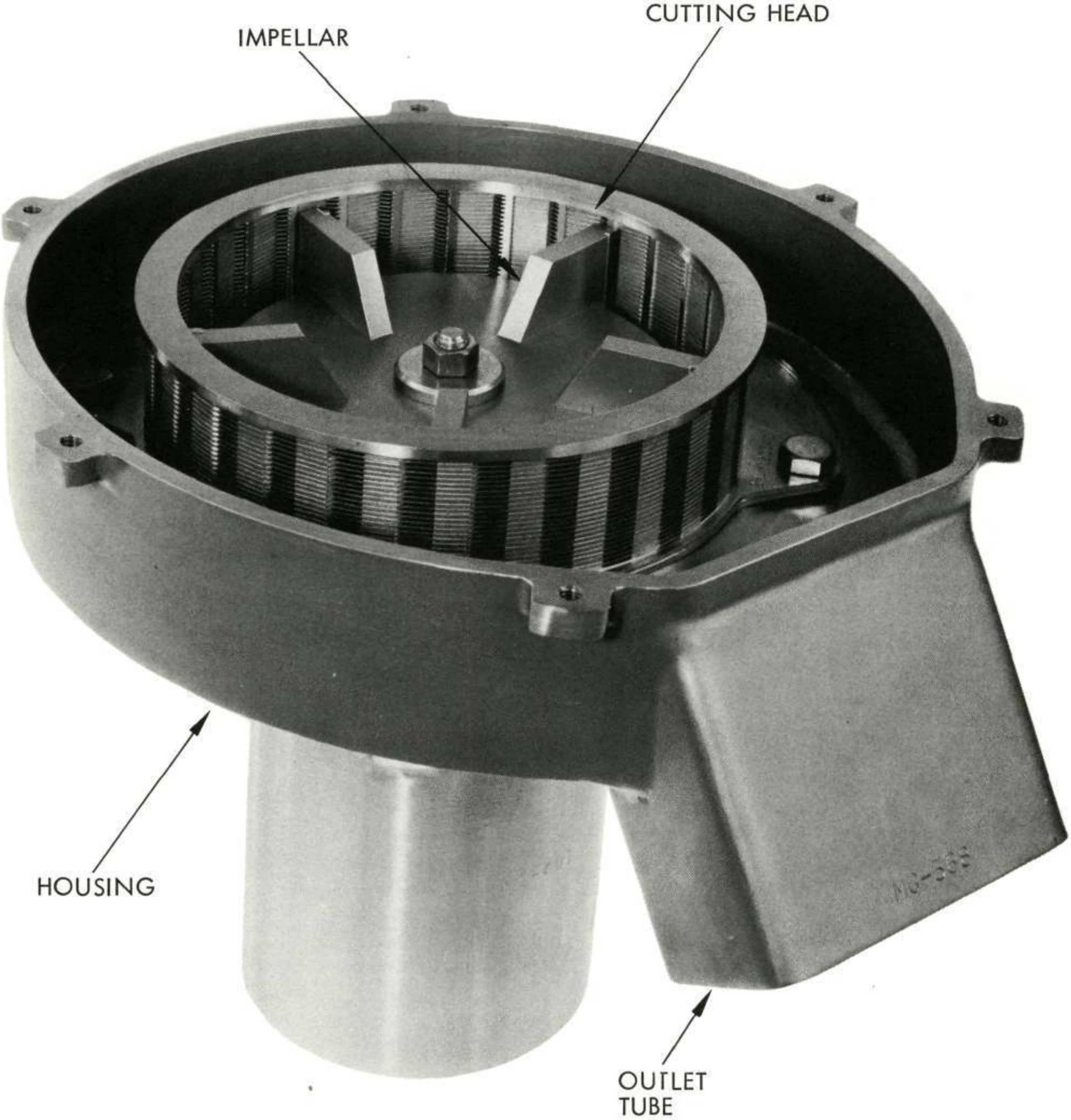


Fig. 40 Urshel Laboratories Comitrol Cutter

A sample of spacecraft waste was then prepared and sent to Urschel for evaluation. They recommended use of Urschel cutting head 3-K-015025, based on the tests run at their laboratory. The next step in the development program was to design and fabricate a laboratory test model to support evaluation tests at LMSC. Figure 41 presents a photograph of the unit and Figure 42 is a sketch of the assembly. The original intent of the grinder was that it be used as a combination blower and grinder. Relatively dry wastes would be introduced into the grinder, pulverized, blown into a phase separator where water would be introduced to make a slurry, slurry would be pumped to the wet oxidation hold tank and gases vented to the air conditioning system. The possibility of having the grinder blow ground trash into the Hydro-John flushing toilet using its phase separator and flushing capability were also considered.

The laboratory grinder operation was evaluated in a series of tests using dry and wet wastes. These tests produced the following results:

- Dry wastes were ground satisfactorily using the grinder as a combination blower and grinder.
- Damp and moist waste materials clogged the grinder, even when various volute and outlet shapes and forced feeding were tried.
- Relatively large quantities of water were required to prevent clogging. Wet wastes with solids content of 0 to 5 percent by weight were grindable without clogging of the cutting head or outlet tube.
- Wet wastes with solids content of 5 to 10 percent were grindable only by recycling wet ground wastes to assist in the grinding operation as illustrated by Figure 43, which is a schematic of the laboratory grinder test setup. Six quarts of water, simulating wash water concentrate, were charged into the hold tank. The recirculation pump was energized to establish a flow of water to the grinder inlet. Waste materials in accordance with the waste model were continuously fed into the grinder inlet. test was terminated when the slurry became so thick that the pump would no longer recycle the slurry. Solids content was then measured by weighing, drying, and reweighing a sample of the slurry. Solids content, when the test terminated, varied from 9 to 11 percent. Higher solids content could undoubtedly be achieved by a better pump design since it was the pump that limited solids content, not the grinder.

Since it can be assumed that not all wastes to be ground are dry and that solids content of from 10 to 20 percent are desired to match available waste waters with available solids wastes, a spacecraft grinder system based on recycling was envisioned. The schematic of such a spacecraft trash grinder system is presented by Figure 44. The system would operate in the following manner:

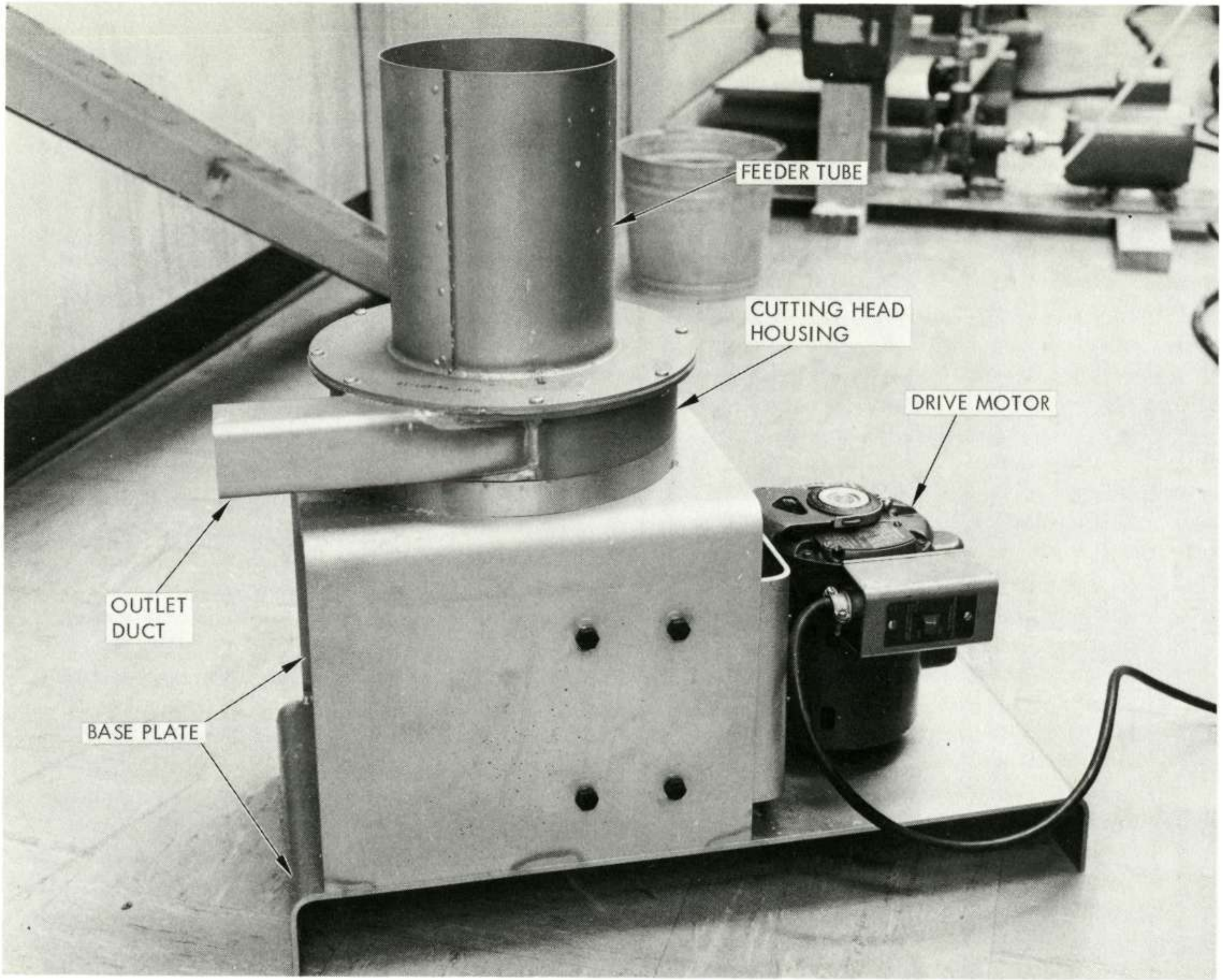


Fig. 41 Laboratory Test Model Grinder

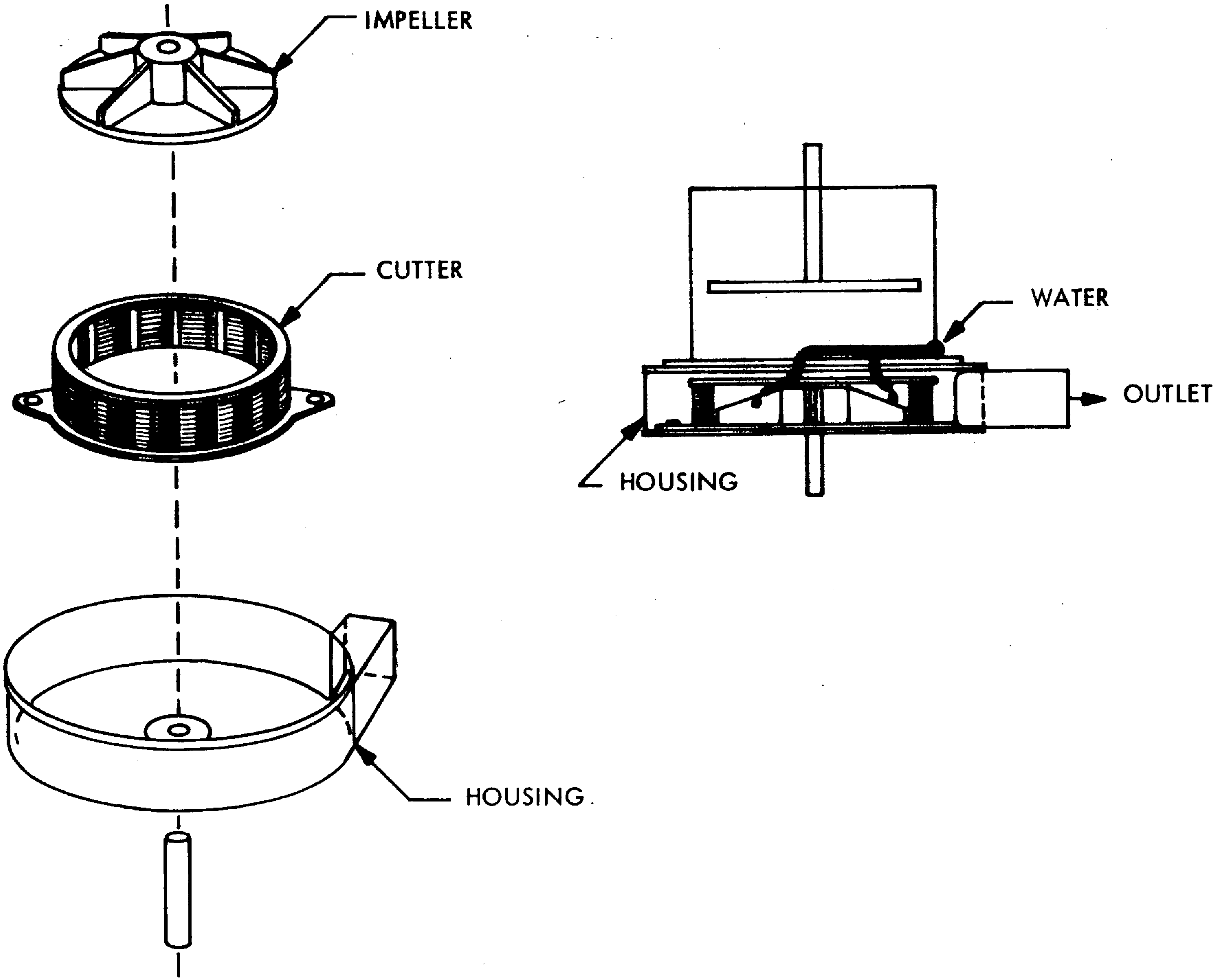


Fig. 42 Grinder Assembly

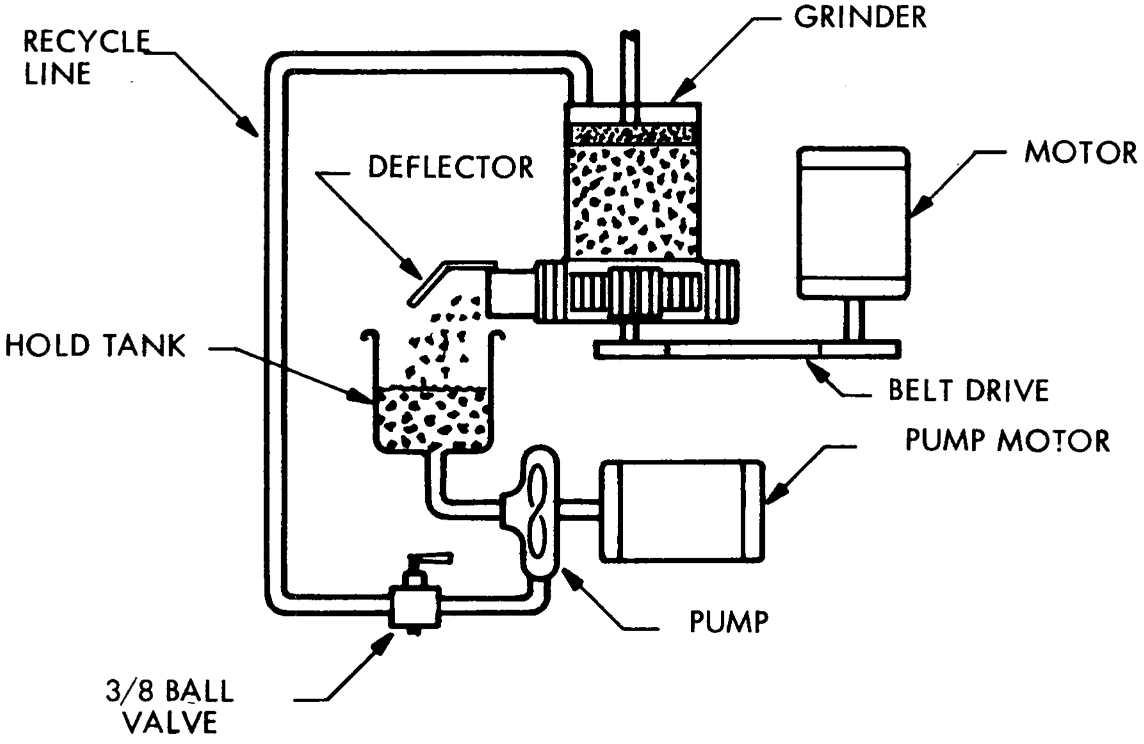


Fig. 43 Laboratory Model Grinder Test Setup

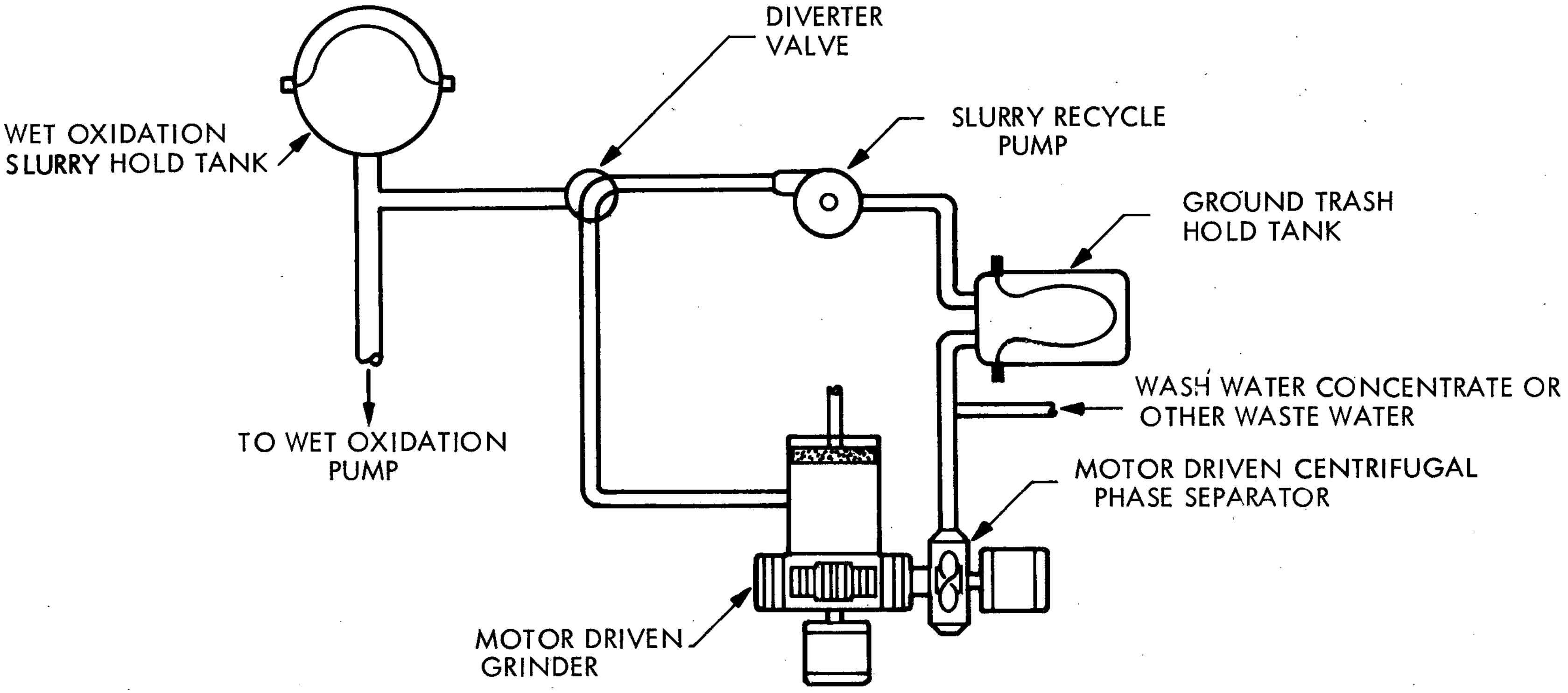


Fig. 44 Spacecraft Trash Grinder System

- Fill trash hold tank partially with waste water (wash water concentrate)
- Energize recycling pump
- Load grinder feeder with trash
- Energize grinder and phase separator
- Feeder automatically pushes trash into grinder impeller
- System is shutdown and trash feeder is reloaded as many times as needed
- When trash hold tank becomes full, the diverter valve is switched to deliver wet ground trash from the ground trash hold tank to the wet oxidation slurry hold tank
- When the trash hold tank is emptied, the diverter valve is switched back to the recycle position and the trash hold tank is partially refilled with waste water.

The tests described previously were run with a 1/2 horsepower motor with a grinder speed of approximately 3000 RPM. A series of tests were run to determine the effects of varying motor horsepower and grinder speed. The following variations were run:

1/2 HP @ 3500, 2650, 2100 RPM
1/4 HP @ 3500, 2650, 2100 RPM
1/6 HP @ 3500, 2650, 2100 RPM

The 1/6 horsepower motor was satisfactory and the 2100 RPM speed produced the best results. The grinder did not bog down as readily at the lower RPM's.

It is recommended that a spacecraft grinder development program be initiated based on the results of the laboratory investigations.

ASH FILTER

Use of water recovery systems other than the dry boiler established the requirement for an ash removing filter. A survey of available filters resulted in selection of two types. Two Montgomery Brothers cartridge type SSBX 10-3/4 filters shown by Figure 45, were procured and tested with wet oxidation effluent from the five gallon batch reactor discussed previously in the section entitled "Alternate Water Recovery Devices". Photographs of ash samples showed particles sizes ranging from 0.5 to 1 micron. Tests were run with a 5 micron element downstream of a 75 micron element to determine the percent of ash that passed through each cartridge. Ten gallons of wet oxidation effluent containing approximately 68 grams of ash were pumped through the two filters. All of the ash collected on the 75 micron filter forming a filter pack which removed the fine particles.

Pressure drop through the filters during the tests was less than 1.0 psi at rated flow. However,it was intended that a cartridge would be changed every thirty days in a spacecraft system and the 68 grams of ash available for this test represented only a 1.4 day supply. Production of a thirty day supply of ash and conduct of long duration tests were beyond the scope of the effort that could be devoted to the filter problem during this contract. Additional filter tests to determine the best filter pore size and system arrangement that will provide adequate filtration with high filter loading are recommended.

The second type of filter that looked promising for wet oxidation application was the GAF Corporation AFCO polypropylene filter bag shown by Figure 46. This filter works on the filter cake principle of building up a layer of ash on the surface and then letting the pack act as its own filter thereafter. Considering the relatively low flow rates involved in a spacecraft system, such an approach holds promise of providing a simple means of achieving high ash loadings. This filter was studied, but no tests were run during the contract period.

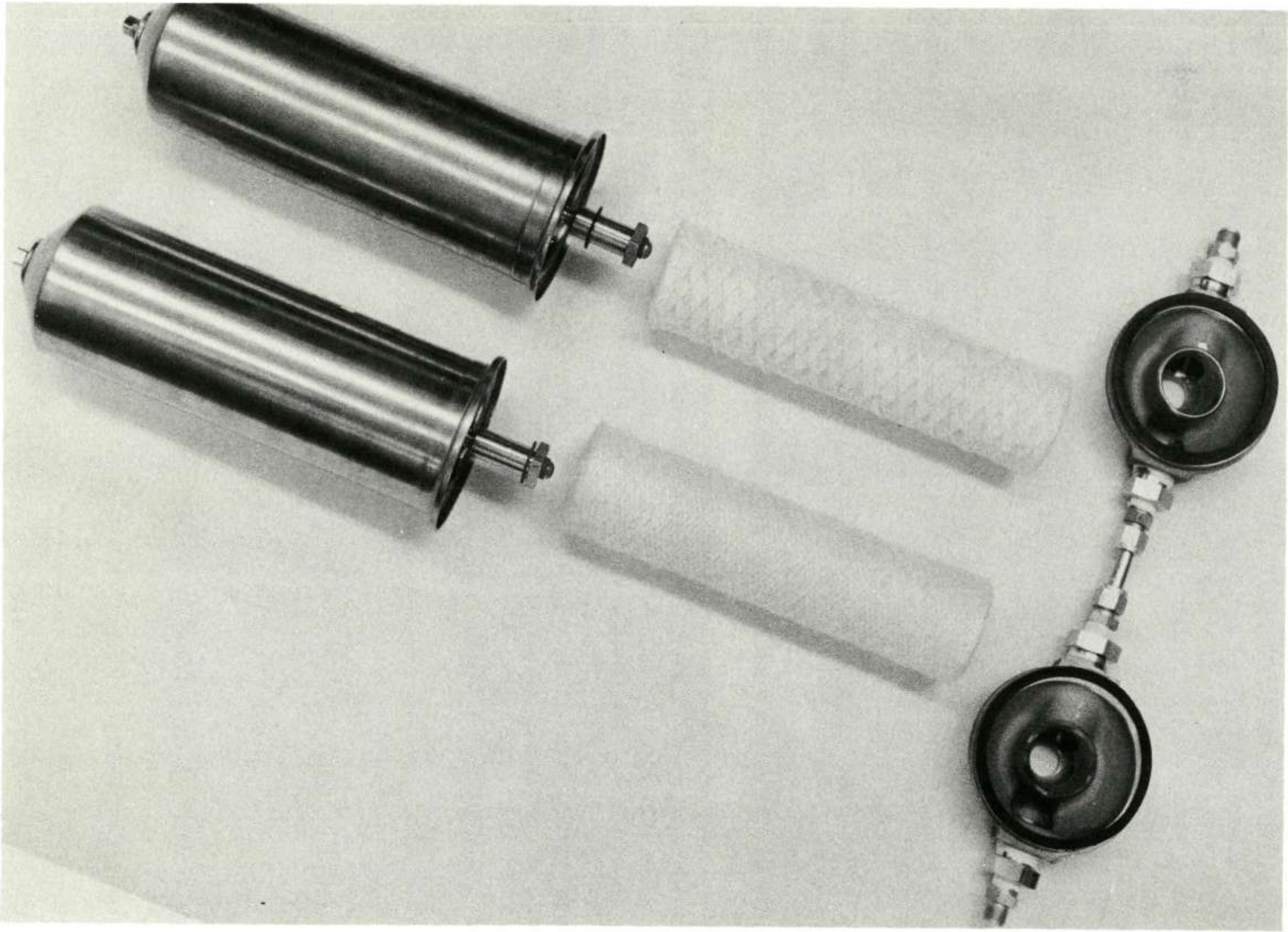


Fig. 45 Cartridge Type Filter

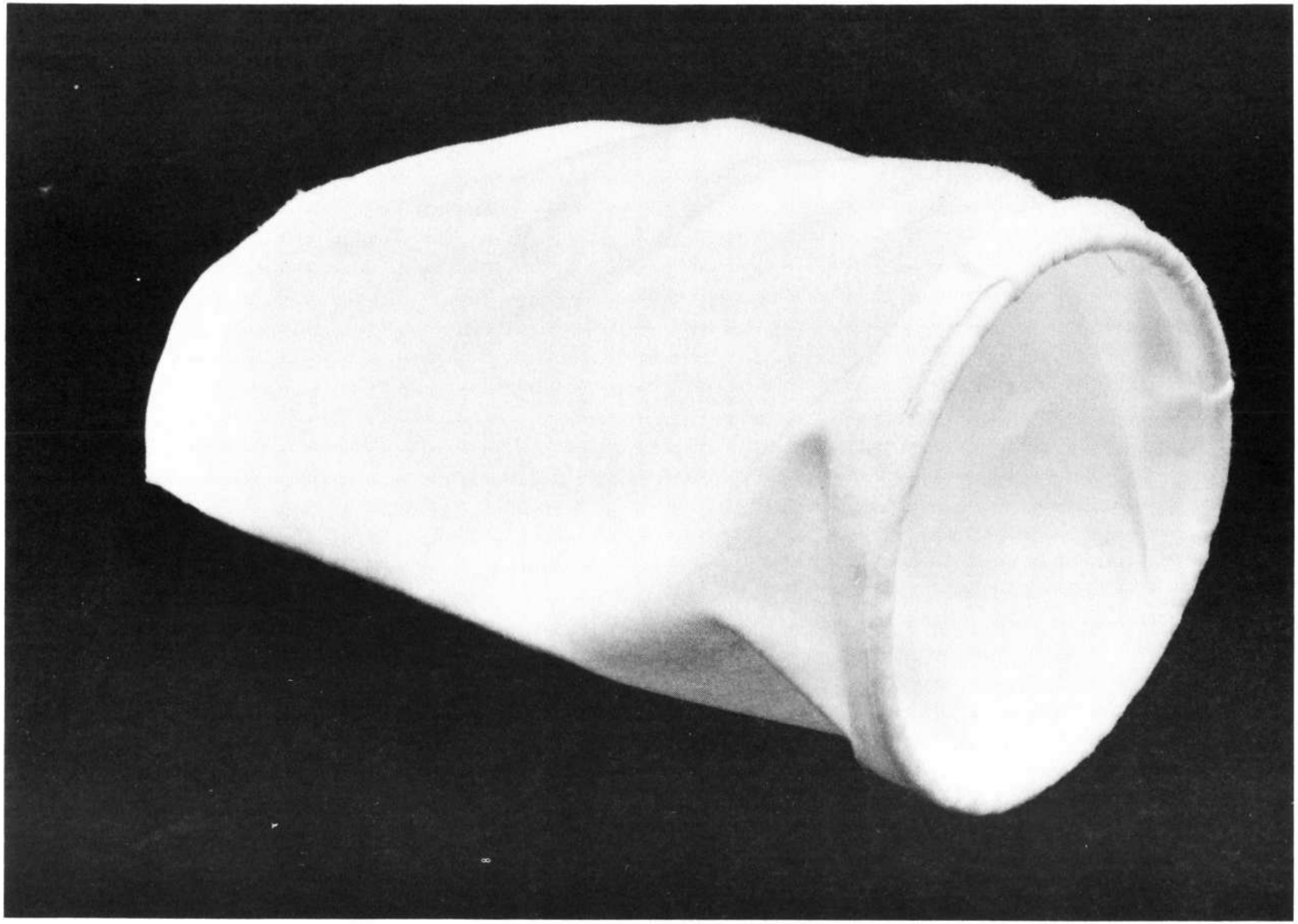


Fig. 46 Filter Bag

REACTOR DESIGN MODIFICATIONS AND SYSTEM TESTS

The DVT showed several problems with the reactor design. Assembly and disassembly were not easy, requiring removal of stirring rod and baffle chamber assemblies for any significant reactor rework or repair. The heavy bulky drive motor, limited chamber bearing life, and interference between stirring rods and baffle assemblies were also of concern. Reactor design studies were conducted to evolve solutions to these problems. The following modifications were made:

- New stirring shaft and new baffle assembly designs were adopted. Figure 47 presents a drawing of the modified reactor design. The stirring rod assemblies of the old design were eliminated, and the stirring rods were pressed directly into a hollow 3/8 inch O.D. shaft. This design stiffened the shaft and eliminated the excess weight of the stirring rod assemblies. The seven baffle chambers were eliminated and a two-piece clam shell design with internal dams and baffles was used. This new design allowed the reactor stirring shaft to be connected to the drive tube and cover, the baffle assemblies to be placed in position one at a time, and clearances between the stirring rods and baffles checked. The stirring rod position could then be adjusted to provide adequate clearance between the stirring rods and baffles. The reactor body was then slipped over the baffle parts and the end covers bolted to the body to complete the assembly. The new internal reactor parts were fabricated from 316 SS instead of Inconel 625 in the interests of cost and schedule. It was recognized that significant corrosion would be encountered but would not be so bad as to interfere with the conduct of the proposed testing.
- A new drive motor concept was also adopted. The motor wrapped around the magnetic drive tube proved to result in a heavy bulky motor design. An externally mounted motor with belt drive proved to be a much smaller, lighter weight drive method. The motor drove the reactor outer magnetic drive tube through a belt and pulleys.
- The concern about carbon bearing life led to a carbon bearing life test. This test also served as a combined systems test in that the new hydraulic pump, double-ended slide valve pump, motorized ball valves and back pressure regulator were checked concurrently with bearing life.

Carbon Bearing Life Test

Figures 48 and 49 present a photograph and a schematic of the carbon bearing life test setup. In preparation for the test, the reactor was assembled with new carbon bearings in both drive tube and reactor covers and the new stirring shaft and clam shell baffle assemblies were used. The test was run in the following manner:

B

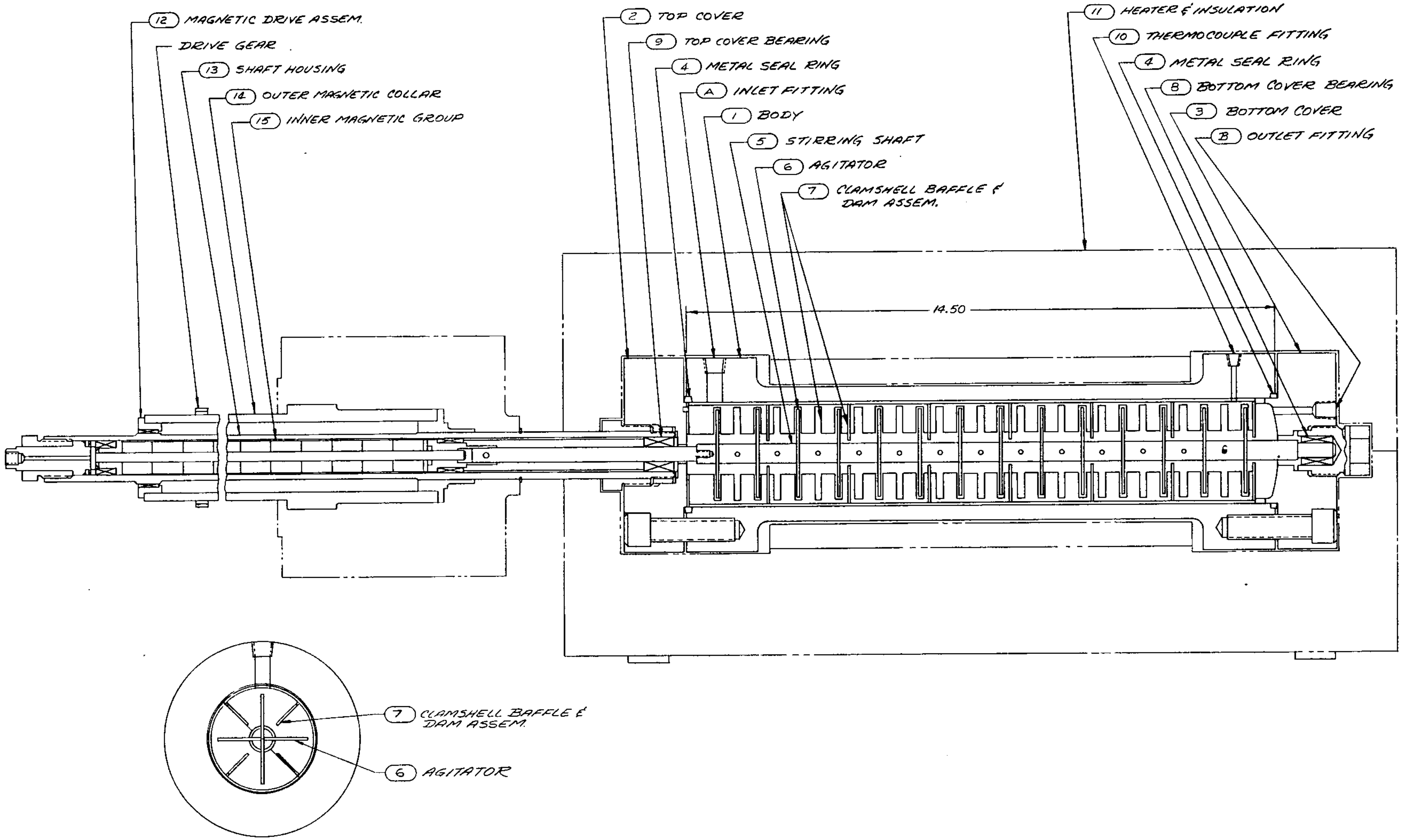


Fig. 47 Modified Reactor Design

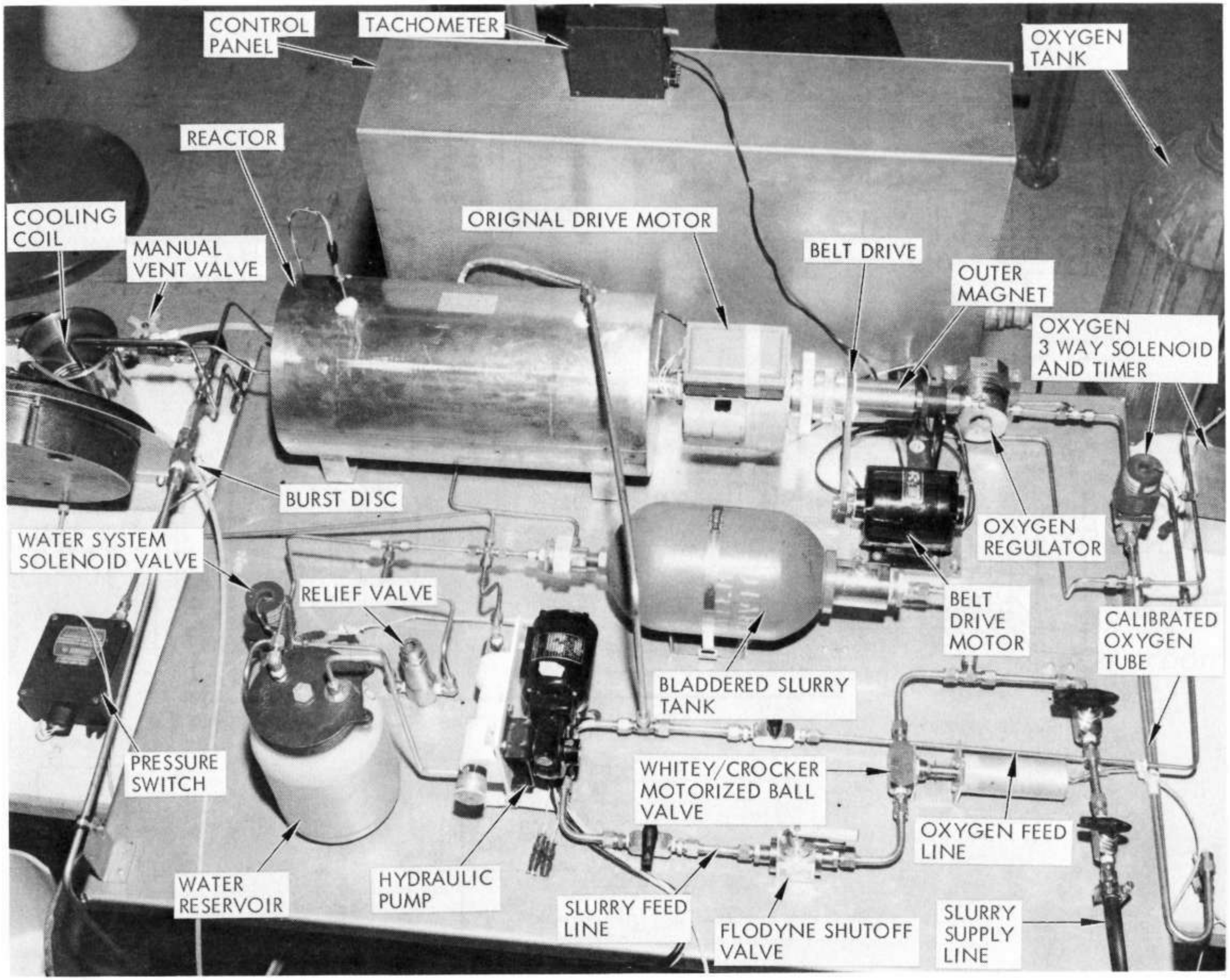


Fig. 48 Carbon Bearing Life Test Setup

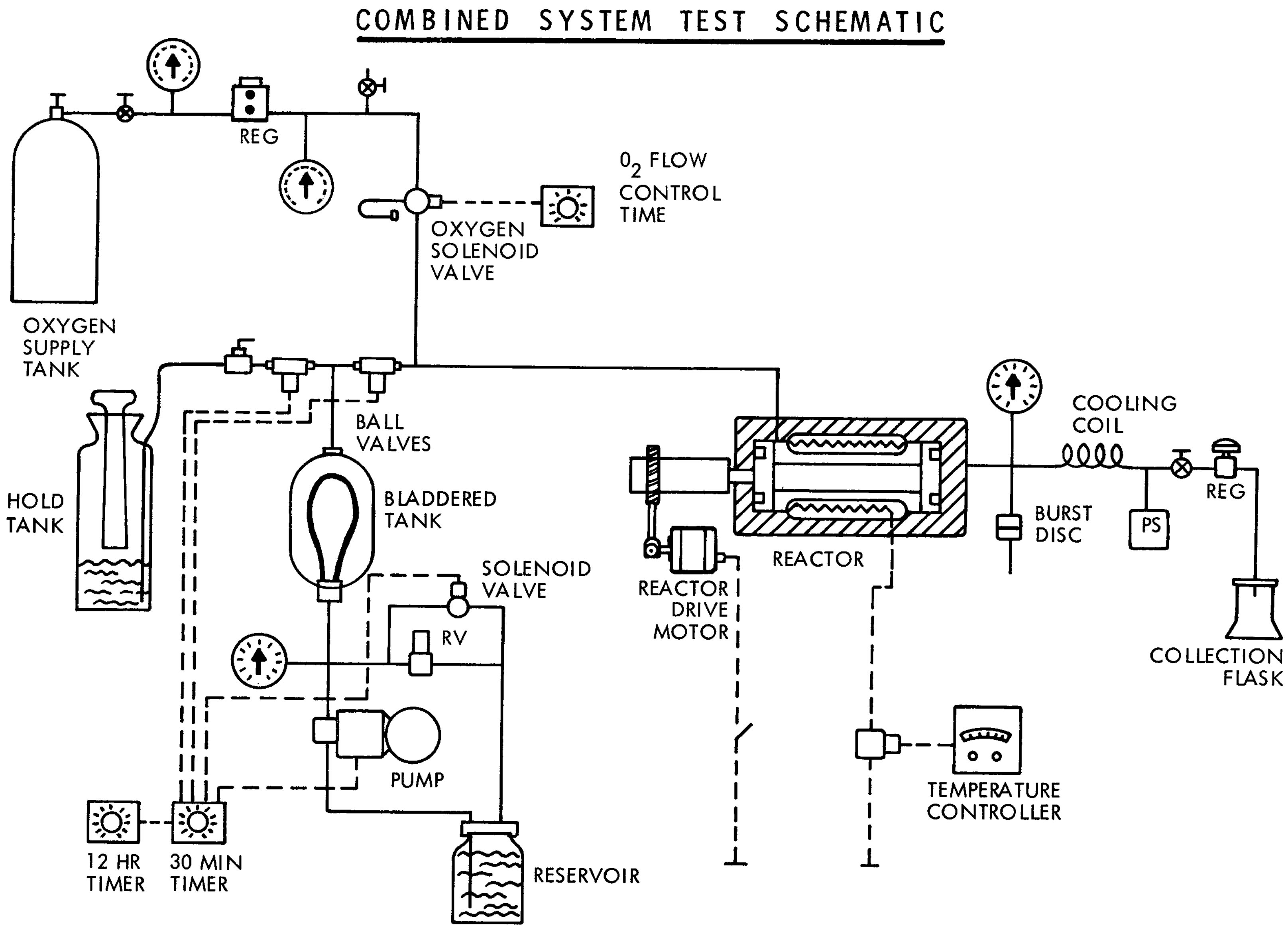


Fig. 49 Carbon Bearing Life Test Schematic

- Slurry tanks and lines were filled with slurry from the slurry hold tank
- Reactor was charged with 400 cc of slurry
- Oxygen system was energized and the oxygen regulator adjusted
- Reactor temperature controller, warmup heaters, and reactor drive motor were energized
- When reactor reached temperature, the hydraulic pump was energized and the slurry feed control timers and the oxygen feed control timer were energized
- Samples were then collected at the effluent port
- Reactor stirring rod speed was then varied to determine the RPM that gave the best water quality. 600, 436, 328, and 200 RPM test runs were made. Speed variations were achieved by changing pulleys on the drive motor and reactor outer magnetic drive tube.

Figure 50 presents a plot of the drive speed and reactor temperature and pressure, and indicates significant events that occurred during the test. Also shown are times that the reactor was testing in a continuous flow mode and when it was closed off. The numbers above the continuous flow plot indicate the number of hours run in that mode on that day. The most significant problem encountered during the test was pin hole leakage from the reactor outlet tube. This occurred five times during the test, showing that 316 stainless steel tubing is not an acceptable choice for the high temperature portion of the system. No problems have been encountered over the entire program with leakage from the cool portions of the oxygen, slurry, or effluent lines. Leakage from the outlet tube interfered with sample collection for the RPM tests because several hours of operation were generally required to reach steady state after a change in RPM or overnight close off of the reactor. Sufficient water samples were collected to show that 436 RPM was the lowest speed that produced high quality water. Effluent liquid and ash color was used as the primary indicator. The water became slightly yellow and the ash darkened at 328 and 200 RPM. Therefore, 436 RPM was selected for all future testing.

Two motorized ball valves shown by Figure 51 were obtained from Flodyne Controls, Inc., Murray Hill, New Jersey, for slurry feed shutoff application. They were included in the early portion of the carbon bearing life test, but both failed relatively early in the test. Shaft seal leakage allowed slurry to fill the motorized actuator causing binding of the gear train by salt deposits. The valves were returned to the vendor for rework to provide an improved shaft seal and to incorporate an actuator shaft seal with the space between them vented to prevent leakage from entering the actuator. In order to evaluate the re-designed valve at minimum expense, handles were placed on them for manual operation, since the problems were with the valve, not the actuator. Whitey manual ball valves were used for the remainder of the carbon bearing life test and the modified Flodyne valves were evaluated in subsequent tests.

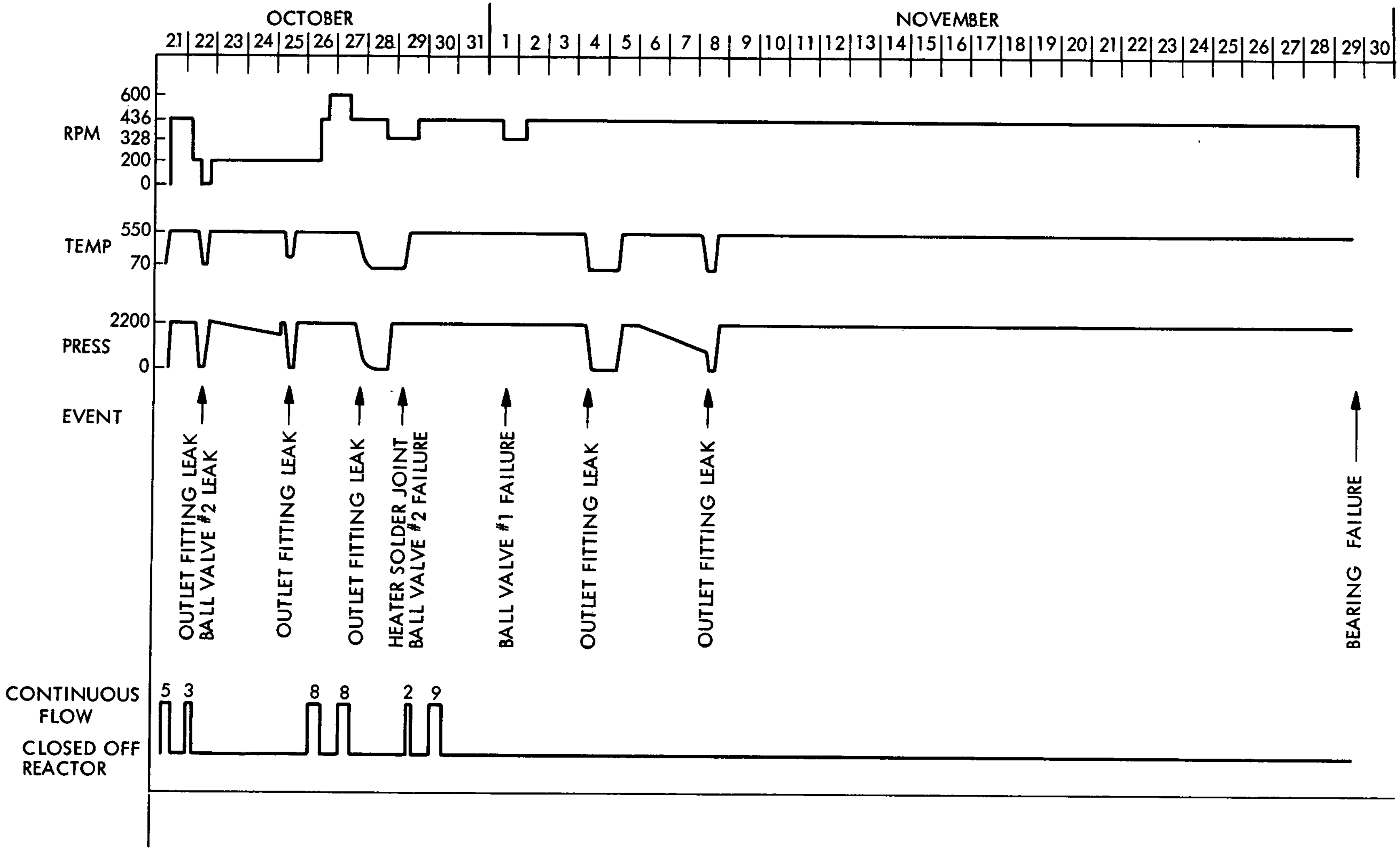


Fig. 50 Carbon Bearing Life Test Results

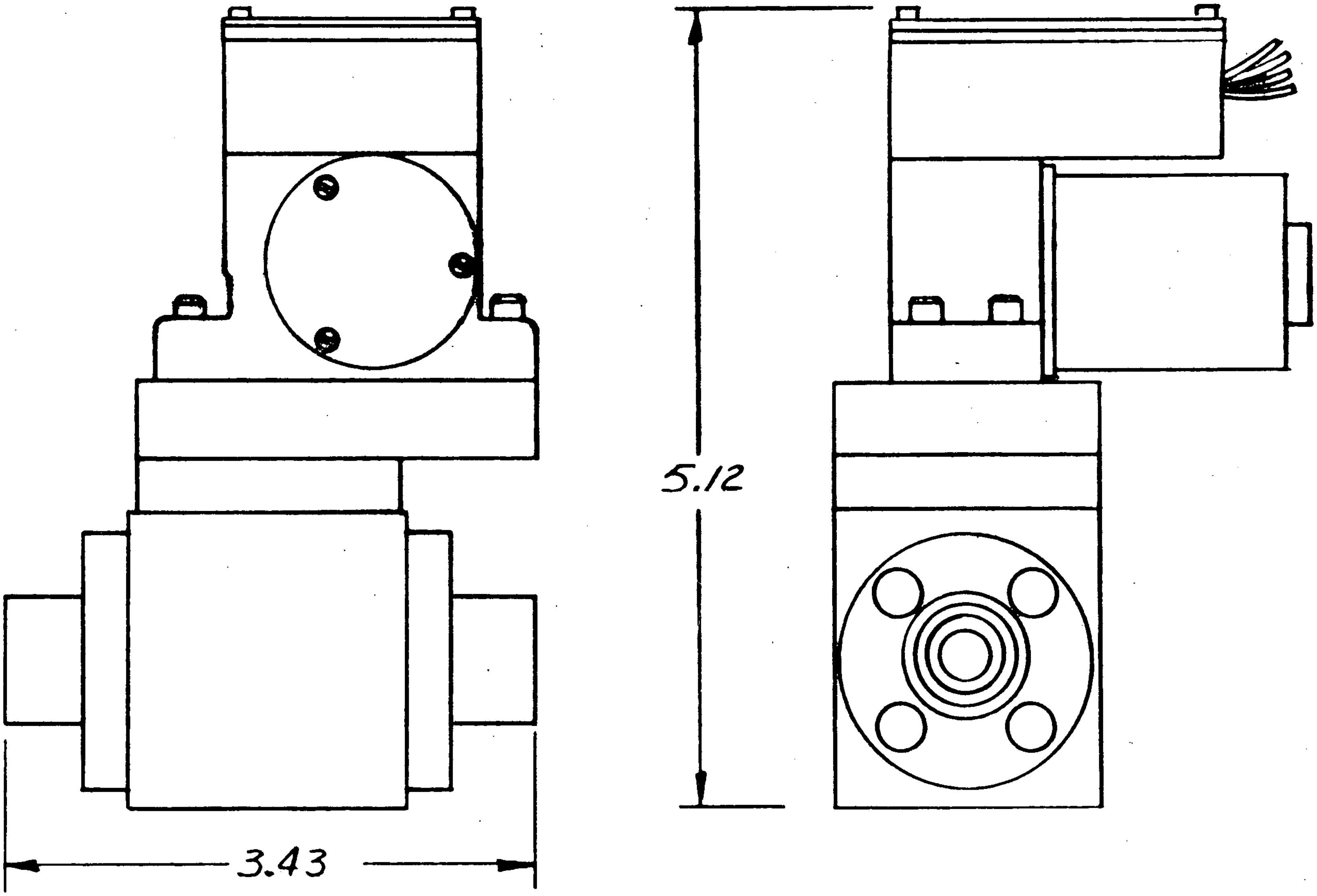


Fig. 51 Flodyne Motorized Ball Valves

On the fortieth day of the test, a clicking noise indicated that the stirring rods must be striking the baffles, so the test was stopped and the reactor disassembled. The carbon bearings had obviously failed with a considerable amount of wear taking place on the bottom of the horizontally mounted bearings. Apparently, the weight of the shaft produced the wear, not any imbalance in the shaft. The ends of the bearings open to the reactor were also mushy, showing some attack by the slurry/oxygen mixture in the reactor.

As anticipated, the reactor baffle assembly was badly corroded. The stirring shaft was not corroded, however, indicating that electrolytic corrosion between the close fitting baffle assembly and reactor took place. The type of deposits, in ridges and valleys on mating parts, also substantiated this conclusion, giving further evidence to the fact that the reactor and all its parts should be made of the same metal.

Based on the results of the carbon bearing test and a vendor survey of corrosion resistant ball bearings, it was decided to run a 90-day ball bearing life test using Fafnir's Cōballoy bearing AAP4WDD5 FT. The reactor was outfitted with the ball bearings and reassembled, and the test was run in a similar manner as the carbon bearing life test.

90-Day Ball Bearing Life Test

The 90-day ball bearing life test was also used as a checkout test for several other items in the test setup. A new back pressure regulator (Grove Mity Mite SD-91-XW) shown by Figure 52, the reworked Flodyne ball valves shown by Figure 53, and a motorized ball valve using a Whitey 44F4316 valve and a Crocker Manufacturing Company actuator 40B250 shown by Figure 54 were evaluated. Figure 55 presents a plot of the drive speed, reactor temperature and pressure, reactor flow condition and significant events that occurred during the 90-day test. The back pressure regulator was used during the entire test to maintain reactor pressure at 2200 psi. It did an excellent job venting liquid, gas, and ash during all continuous flow portions of the test and holding pressure during closed reactor operation. It is a gas dome loaded balanced diaphram type regulator.

The reworked Flodyne valves failed early in the test with leakage from the shaft seal. They were removed from the test setup and the test was continued. Because of the problems encountered with the Flodyne motorized ball valves, and the very favorable performance of the Whitey manual ball valves used in the initial prototype tests and the 60-day carbon bearing life test, it was decided to find an actuator for a 3/8 Whitey ball valve and evaluate its performance in the system. The Crocker actuator was found mated to the valve and run in the ball bearing life test. It performed very well. For a final prototype design some changes would be made, however. The Crocker actuator rotated 90 degrees in one second. Rotating the valve this fast produced excessive torque that tried to stall the motor. For long life, an actuator with a rotation time of between 5 and 10 seconds with the same torque of 50 inch pounds would be much better.

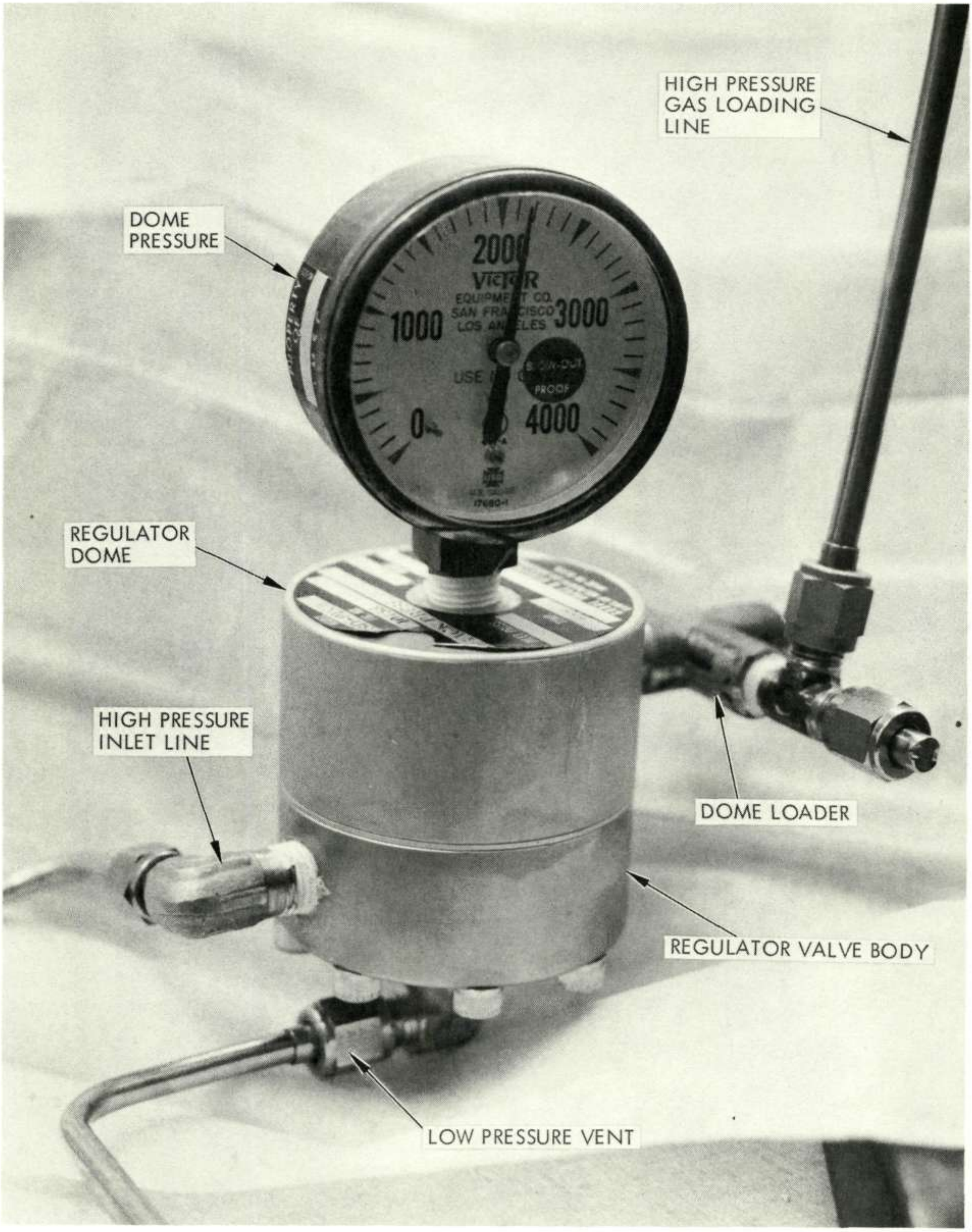


Fig. 52 Reactor Back Pressure Regulator

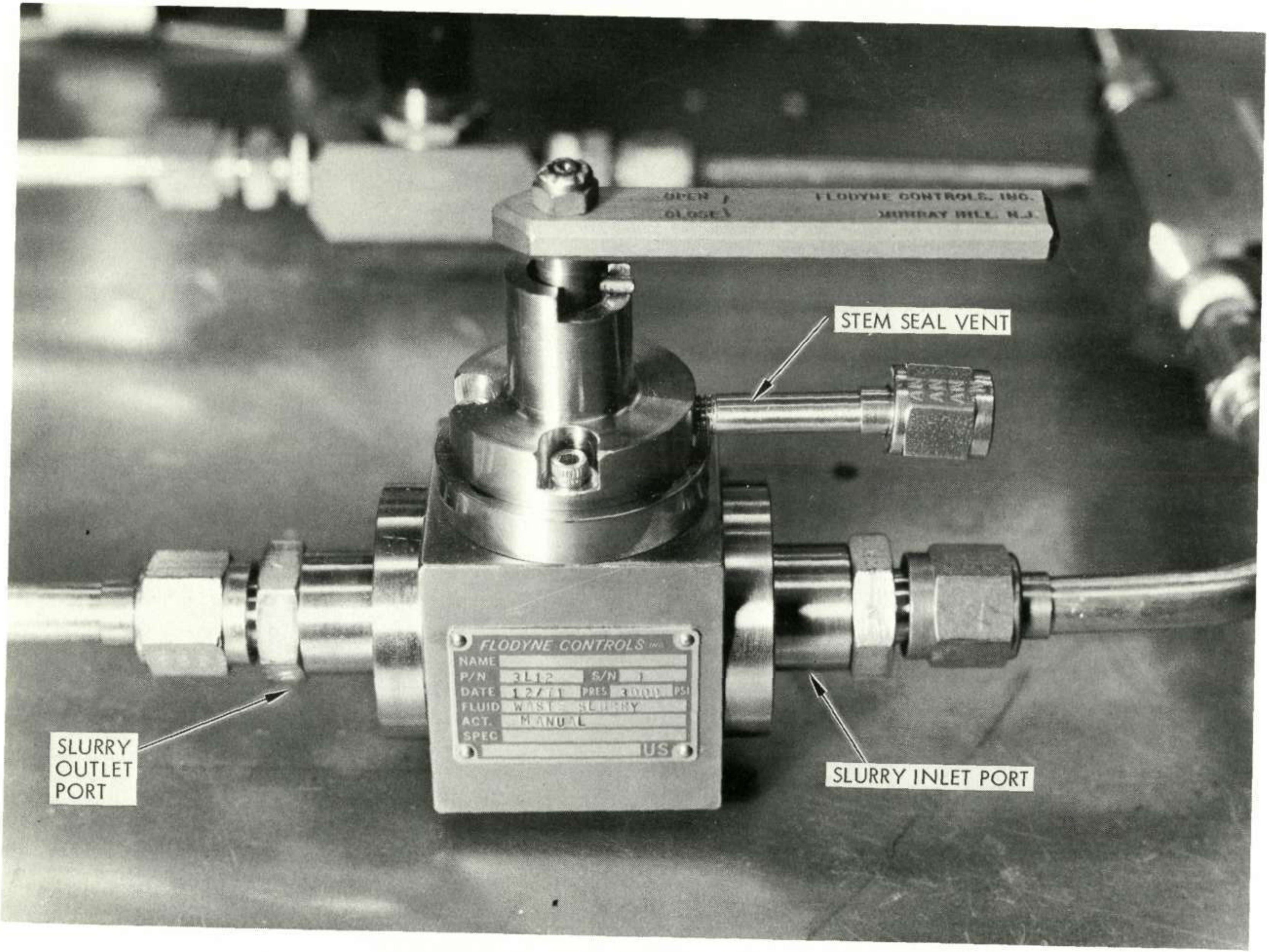


Fig. 53 Reworked Flodyne Motorized Ball Valve

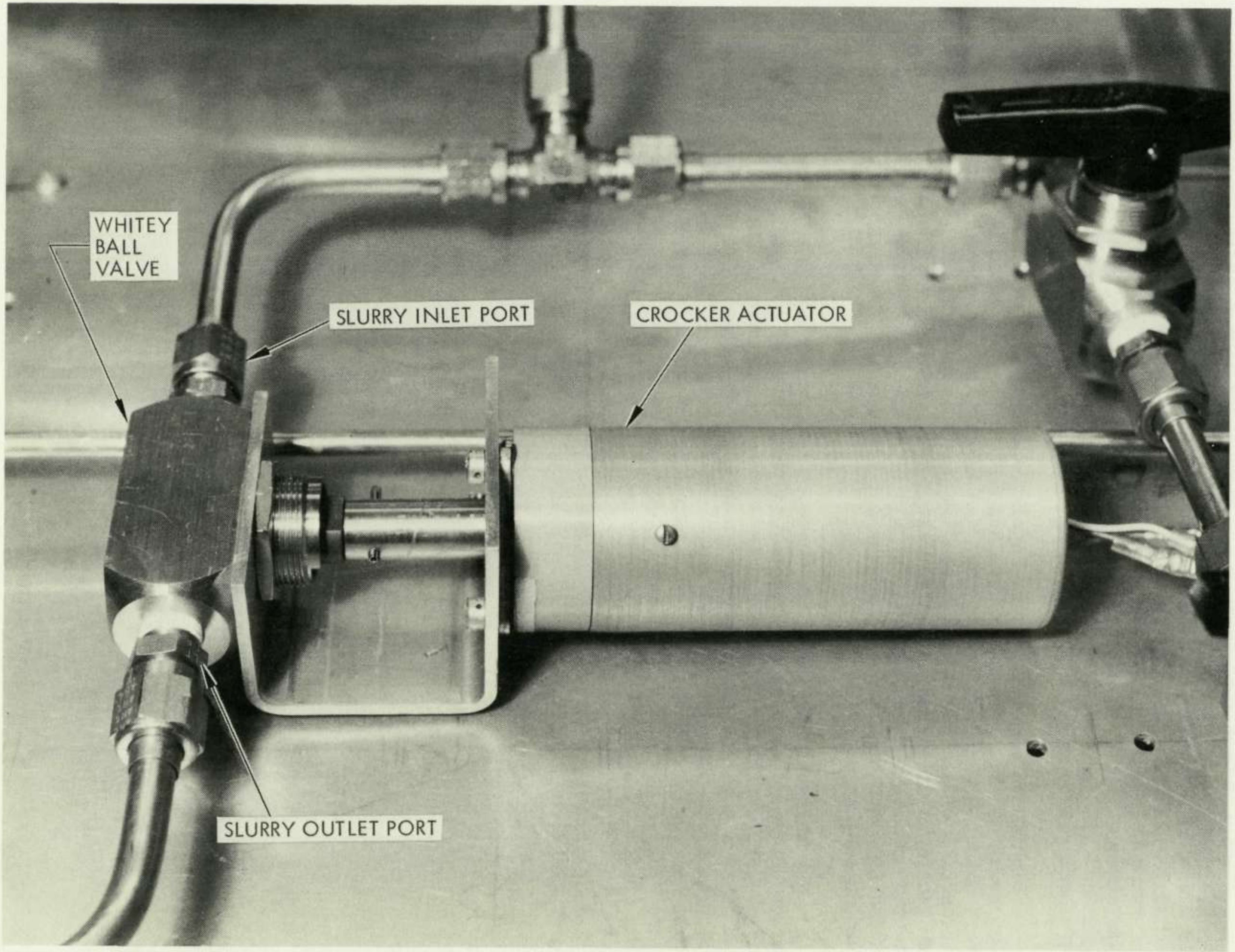


Fig. 54 Whitey/Crocker Motorized Ball Valve

Reactor outlet tube leakage was encountered again during this test, as was expected. 316 SS tubing was used repeatedly, because Inconel or Hastalloy C tubing required a mill run and presented an impossible schedule problem. The slurry feed system was replumbed to allow testing of the double-ended slide valve slurry pump and to allow testing system performance with a slurry tank full of ground trash from the laboratory grinder. The reactor effluent was identical in color and COD using trash or fecal slurry feed.

Liquid and gas samples were analyzed during the test with the following results. No nitrogen oxides, sulphur oxides, or hydrogen sulphide were detected. The effluent water was boiled and condensed to simulate the action of a vapor compression distillation unit and analyzed for hydrocarbons and organics. None were found. Ammonia was the only detected impurity in the water. Ammonia removal catalysts were not introduced into the continuous flow reactor, because the two test programs ran concurrently.

The ball bearing life test was completed at the end of 90 days with all equipment operating. The internal parts of the reactor looked in good shape except for the baffle assemblies which some additional corrosion above that encountered during the 60-day carbon bearing life test. Figure 56 presents a photograph of the disassembled reactor following the 90-day ball bearing life test. The ball bearings were pitted and corroded but were still functioning. They broke when pressed from the end covers. The corrosion appeared to be electrolytic in nature because of the porous look to the surface and the fact that it occurred where metals touched. It is recommended that future efforts investigate the feasibility of Hastalloy C ball bearings in a Hastalloy C reactor.

c

Page intentionally left blank

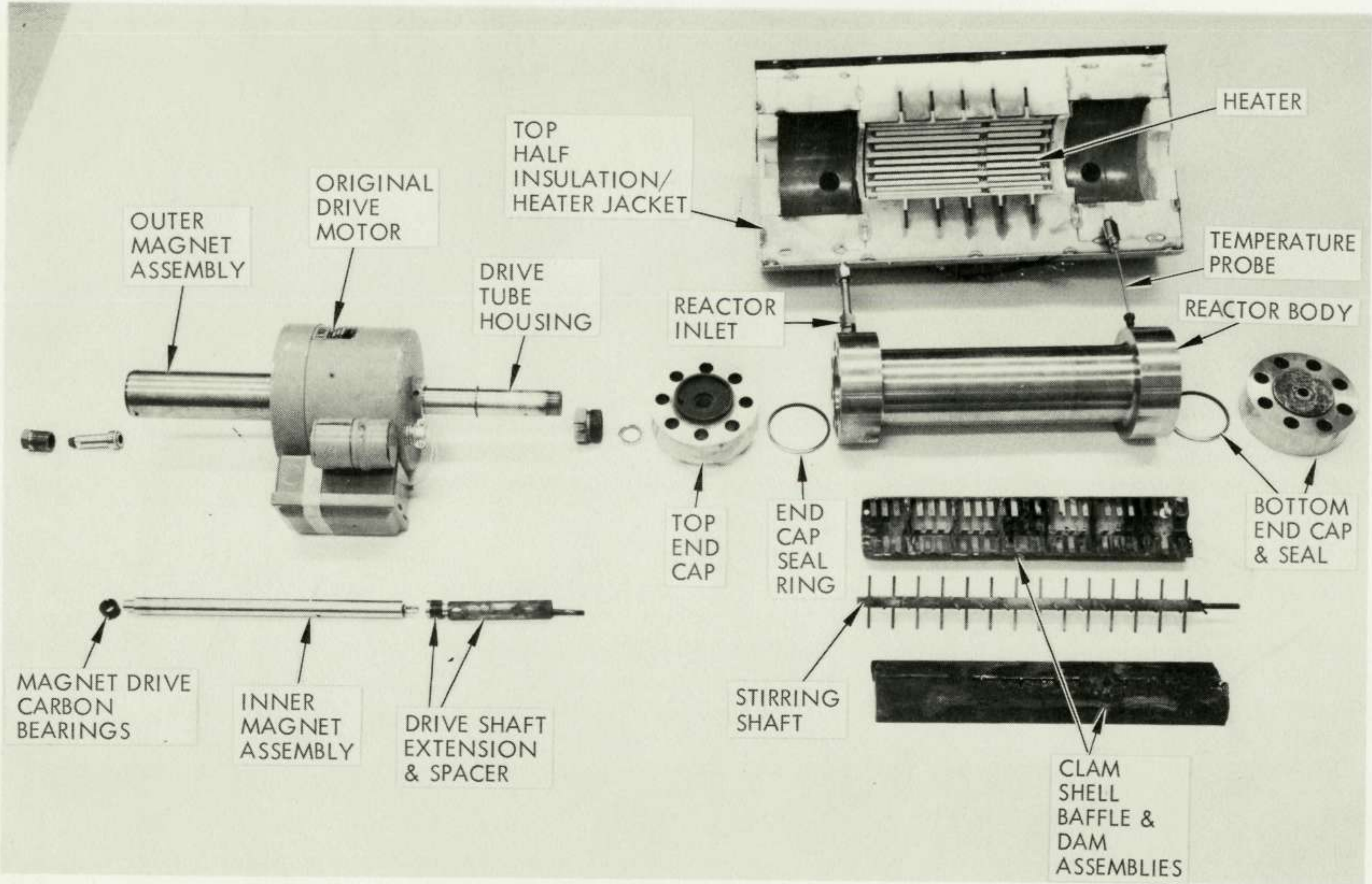


Fig. 56 Reactor Parts After 90-Day Test

PRECEDING PAGE BLANK NOT FILMED

CONCLUSIONS

The laboratory process study and prototype system tests proved wet oxidation to be a very effective means of providing a high degree of waste oxidation while producing effluent liquids and gases that are relatively easy to reclaim by currently developed devices. Satisfactory solutions to all of the problems encountered in the initial prototype system were evolved in the subsequent development effort to a point where a prototype system suitable for manned chamber testing can be fabricated and tested with a high degree of confidence that it will perform successfully.

It is recommended that the double-ended slide valve slurry pump, outfitted with motor actuators, be used in future systems and that the hydraulic piston pump be considered a backup approach.

The wet oxidation reactor body and all internal parts should be constructed of Hastalloy C and all tubing in hot portions of the system should also be of Hastalloy C. The feasibility of Hastalloy C ball or bushing-type bearings should be investigated.

Development work on high pressure water electrolysis cells should be emphasized and integration of the oxygen supply and wet oxidation system should be studied.

The vapor compression distillation and electrodialysis water recovery systems are the most attractive for wet oxidation effluent water processing. Additional development tests using wet oxidation system effluent should be run to establish system performance and integration methods.

Ruthenium Trichloride catalyst addition to the wet oxidation feed slurry will provide an effluent free of ammonia. It is envisioned that the catalyst salt would be desolved in water and metered into the slurry feed line or hold tank.

The investigations of the solids grinder resulted in a recommended system arrangement and grinder requirements. The grinder problem is formidable enough; however, to justify a separate development contract. The problems of loading the grinder, feeding trash to the cutting head and recirculating ground wet trash to the grinder to achieve high solids content require careful attention. The design of a phase separator that will provide high separation efficiencies with a trash slurry gas mixture is also a significant development problem.

The continuous flow reactor with internal baffles, dams and stirring rods proved to be very effective in achieving the desired agitation and preventing mixing of influent and effluent.

Filter tests run under the contract were exploratory in nature and additional filter arrangement and pore size tests are required.

Preceding page blank

REFERENCES

1. Robert B. Wheaton, et al, "Investigation of the Feasibility of Wet Oxidation for Spacecraft Waste Treatment", Final Report NASA Contractor Report 66450, Contract No. NAS 1-6295, Whirlpool Corporation.
2. ASTM Special Publication No. 264, "Stress Corrosion Cracking of Austenetic Chromium - Nickel Stainless Steels", American Society of Testing Materials, Philadelphia, Pa., 1960.
3. W. G. Ashbaugh, "Stress Corrosion Cracking of Process Equipment", Chem. Engr. Prog. 66, 44-7, Oct. 1970.
4. David F. Putnam and Richard L. Vaughan, "Design and Fabrication of a Flight Concept Prototype Electrochemical Water Recovery Subsystem", NASA CR-111961, September 1971.
5. G. Kleiner, et al, "Trade-Off Study and Conceptual Design of a Water and Waste Management Subsystem (WWMS)", Final Report NASA Contract NAS 1-10515, June 1971.
6. Anon, "Trade-Off Study and Conceptual Designs of Regenerative Advanced Integrated Life Support Systems (AILSS)", Final Report NASA Contract NAS 1-7905, Hamilton Standard, July 1969.
7. David F. Putnam, "Composition and Concentrative Properties of Human Urine", NASA Contract NAS 1-8954, June 1970.
8. William A. Blecher, "Development of a Prototype Vapor Diffusion Water Reclamation System", Unpublished, Hamilton Standard Division of UAC, 16 June 1971.
9. Henry J. Kolnsberg and Donald R. McCann, "Design and Fabrication of a Flight Concept Prototype Vapor Diffusion Water Reclamation System", NASA Contract NAS 1-8943, April 1971.
10. D. F. Putham, "Water Management for Extended-Duration Manned Space Missions", Douglas Aircraft Co., Paper presented to Conference on Bioastronautics, Virginia Polytechnic Institute, Blacksburg, Virginia, 14-18 August, 1967.
11. A. J. Gully, et al, "The Catalytic Removal of Ammonia and Nitrogen Oxides from Space Cabin Atmospheres", Interim Report NASA Contract NAS 1-9506, 1 September 1969 through 31 August 1970.
12. D. L. Brown, "The Electro-Oxidation of Ammonia in Sewage to Nitrogen", Final Report, U. S. Department of Interior Contract 14-12-432.
13. Anon, "Housekeeping Concepts for Manned Space Systems", Data Book Volumes IIA and IIB, Document No. MS 124 7002, NASA Contract NAS 9-10662, Fairchild Hiller, 30 October 1970.

LIBRARY CARD ABSTRACT

This report covers the development of a spacecraft wet oxidation system. Laboratory investigations to define optimum process conditions for oxidation of fecal/urine slurries were conducted in a one-liter batch reactor. The results of these tests formed the basis for the design, fabrication, and testing of an initial prototype system, including a 100-hour design verification test. Areas of further development were identified during this test. Development of a high pressure slurry pump, materials corrosion studies, oxygen supply trade studies, comparison of salt removal water recovery devices, ammonia removal investigation, development of a solids grinder, reactor design studies and bearing life tests, and development of shutoff valves and a back pressure regulator were undertaken. The development work has progressed to the point where a prototype system suitable for manned chamber testing can be fabricated and tested with a high degree of confidence of success.