CR 151324

DEVELOPMENT & TESTING OF A WET OXIDATION WASTE PROCESSING SYSTEM

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31 March 1977

Prepared Under Contract NAS 9-13824
by
Biotechnology Organization
Lockheed Missiles & Space Company, Inc.
Sunnyvale, California
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SUMMARY

The wet oxidation process has been of interest to NASA as a potential treatment method for wastes aboard manned spacecraft for these reasons:

- Fecal and urine wastes can be processed to sterile water and CO_2 gas. However, the water requires post-treatment to remove salts and odor.
- The residual ash is negligible in quantity, sterile and easily collected.
- The product CO_2 gas could be processed through a reduction step to aid in material balance if needed.

Reaction of waste materials with oxygen at elevated temperature and pressure also produces some nitrous oxide, as well as trace amounts of a few other gases.

The present contract follows three previous ones. The most important milestones of those contracts were: a continuous flow system evolved from a batch reactor design including continuous separation of product water and gas; semi-automatic controls were developed and demonstrated; corrosion and reliability problems were resolved consistent with laboratory demonstration hardware; a means for suppressing ammonia generation was demonstrated. Significant features of the wet oxidation demonstration system developed prior to the present contract included: a regenerative heat exchanger to reduce energy, a reactor stirring mechanism, and a powered vortex phase separator to separate effluent gas from liquid. However a number of problem areas remained:

- The bearings of the reactor stirring device failed.
- A satisfactory means of shredding all dry wastes had not been found.
- Slurries composed of shredded dry wastes as well as pulverized dry wastes, when mixed with water, could not be pumped.

The current contract originally called for fabricating hardware which would shred, pulverate and transport dry trash. However, the investigation, including consultations, vendor services and laboratory tests led to the conclusion that the problem is very much greater than the present funding and technology priority. Also understanding of the applicable technology supports the projection of system penalties too great to justify continuing development effort in this area at this time.

Development work in other sectors proved more fruitful. The contract was revised several times, and the effort redirected depending upon progress and test results. The most significant results were:

- Component reliability was improved.
- System function and safety were improved.
- It was found that stirring is not required in the reactor for fecal, urine wastes.
- Vacuum distillation plus charcoal filtering cleaned up the effluent water to potable quality.
- Reactor pressure could be reduced to 6,200 kPa (900 psi) by reducing the slurry feedrate, and accepting a slight decrease in water quality.
- Oxygen feedrate can be reduced, depending upon the standards set for the effluent water.

A definitive material balance test was performed to quantitatively analyze all influents and all effluents of the system. Among the effluents was an undesirable gas, nitrous oxide. A two stage reaction was developed in which a second catalyst was used specifically to suppress the formation of nitrous oxide. This method worked successfully in a batch reactor, but could not be duplicated in a continuous flow system.

A study was made of several methods to generate oxygen by electrolysis from the liquid being processed.

INTRODUCTION

The wet oxidation process is being used to dispose of wastes in the chemical and paper making industries because of its advantages over dry burning. Dry burning generates large volumes of gas, often with associated particulate matter (smoke) which is odorous and sometimes toxic. Also, a substantial amount of ash is usually produced. Many types of liquid wastes are not suitable for dry burning either. By comparison wet oxidation is well suited to the disposal of liquids and slurries (mixtures of solid particles in liquids). The amount of effluent gas is relatively small, and usually has very little noxious content. There is very little ash produced. The application of this process in manned spaceflight is potentially two-fold: 1) to recycle water while disposing of (and sterilizing) urine, feces and wash water; 2) reclaim dry wastes such as paper and plastics.

LMSC completed three previous contracts for NASA which were lineal antecedents of the present one. Contract NAS 1-6295 employed a batch type of reactor. Measured amounts of sewage slurry and pressurized oxygen were charged into a reactor, which was then heated. Various ratios of sewage to oxygen were run at various temperatures, pressures and durations. An internal stirring device was built into the reactor to assess the efficacy of stirring. The results showed:

- o A 95% reduction of COD (Chemical Oxygen Demand) was achieved.
- o Pure oxygen is preferable to air.
- o The solids concentration (feces) is not an important variable in the completeness of oxidation.
- o Stirring enhanced oxidation
- o Higher temperatures improve oxidation; 288 to 315°C (550 to 600°F) give good results.
- o Significant quantities of ammonia were found in the effluent water.

Contract NAS 1-9183 also began with the use of a batch type reactor. A series of 34 tests were run with various combinations of temperature, time, oxygen partial pressure, percent excess oxygen, solids concentra tion and reactor stirring rate.

The results of these tests showed that PO_2 is very important, but that the amount of excess oxygen is not. Also, good oxidation can be achieved at 288°C (550°F) with no improvement at higher temperatures if there is a sufficiently high PO_2 in all cases. Therefore a PO_2 of 7,584 kPa (1,100 psi) and a total reactor pressure of 15,168 kPa (2200 psi) were selected. Amount of oxygen required was taken to be 0.8 gm/gm solids. A reaction time of 1 ½ hours was found to achieve COD reductions up to 99%. Content of solids in the raw slurry could be at least 30%.

A search was initiated for a catalyst to suppress ammonia formation, but none was found. A test was run with a mixture of paper towels, toilet paper, wet wipes, aluminized food bags and photographic film. A high degree of oxidation was achieved.

Based on the operating conditions mentioned above, a continuous flow wet oxidation system was designed, fabricated and tested. 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 various controls and gages. A 100 hour design verification test was run on this system, with generally favorable results. Evaluation of individual parts follows:

- Slurry Pump: Low flowrates, slurry mixtures and high pressures make this a difficult application. Two alternate pumping systems were developed. One used a motor driven hydraulic piston pump to pump water to the backside of a bladdered tank to force slurry out of the tank into the reactor. The other utilized the reactor effluent fluids to force the slurry into the reactor using a double-ended slide valve piston pump.

- Corrosion Resistant Metals: Various candidate metals were kept in the wet oxidation environment under bending stress for extended periods. The best results were shown by Hastelloy C.
- Oxygen Source Aboard Spacecraft: Trade-off studies compared high pressure gas, super critical cryogenic, chemical, and high pressure water electrolysis. Water electrolysis is the most advantageous.
- Post-treatment of Wet Oxidation Effluent Liquid: The dry boiler was disqualified because of doubts about its ability to function in a zero gravity environment. Electrodialysis, vapor diffusion, reverse osmosis, air evaporation and VCD (vapor compression distillation) were considered in a trade study. This analysis favored VCD.
- Ammonia Removal. A survey was made to find candidate catalysts. The most promising of these were tested and ruthenium trichloride was found to be the most effective, as well as improving the effluent COD.
- Dry Trash Shredder. A lab model was built and tested, and design recommendations were prepared for a prototype.
- Reactor Design: Reactor disassembly was a tedious job; simplification was indicated. The bearings of the stirring unit corroded.
- Valves and Regulator: After surveys and tests, suitable components were found.

The next contract, NAS 1-11748 provided for the detail design, fabrication and testing of the reactor and product recovery portions of a wet oxidation system. This design consisted of the following subsystems and components:

- A laboratory type dry trash shredder subsystem to produce a pumpable slurry, and oxygen supply.
- A regenerative heat exchanger using the hot effluent to heat the incoming slurry and oxygen.
- A slurry pumping subsystem to accumulate raw slurry and continuously inject it into the high pressure heat exchanger/reactor. A piston type water pump was used to pressurize a bladder tank.
- A subsystem to deliver metered quantities of catalyst into the feed slurry.
- A heated, stirred reactor.
- Pressure controls to maintain the reactor at the desired operating pressure.

- A filter to remove the fine ash which is entrained in the effluent.
- A phase separator to separate the effluent gas and liquid.
- Controls and instrumentation to provide automatic operation and fail-safe shutdown.

The system described above was fabricated and assembled in two modules; the slurry and oxygen supply module, and the wet oxidation module. These two modules were placed side by side, connected to each other and to supporting lab equipment. After 737 hours of operation (starting Monday mornings and running continuously thru Friday afternoons) the following results were obtained:

- Effluent was clear, pale yellow, having an odor and TOC (Total Organic Carbon) values ranging from 200 to 300 mg/liter.
- The trash shredder was incapable of providing a pumpable trash slurry.
- Shredded dry waste plus water resulted in clogging the influent lines.
- The slurry pump, oxygen supply, catalyst introduction, heat exchanger, pressure regulator, filter, phase separator, and control systems worked well with some minor difficulties.
- The bearings of the stirring device in the reactor corroded and required replacement several times. Slight corrosion occurred to the inner surfaces of the reactor.
- The catalyst apparently coated the inner surfaces of the reactor and heat exchanger so that it was possible to run the system for more than 500 hours after turning the catalyst injector off, with no increase in effluent TOC. Also, the high concentration build-up of catalyst in the reactor initially caused the slight corrosion of the reactor.

As the consequence, the following conclusions were reached:

- The bearings for the reactor stirrer require redesign to overcome the corrosion problem.
- A major design and development effort is required to develop a dry waste shredder subsystem capable of producing a pumpable slurry.
- Dry waste slurry having a solids concentration consistent with the load model will require: conduits having a minimum of 0.95 cm (3/8 inch) diameter in cool areas; no sharp bends or sharp edges in the flow path; ball valves rather than other types; all reductions of the conduit diameter to be very gradual and smooth; and piston rather than bellows type design for variable volume tankage.

The current contract, NAS 9-13824 originally called for the design, construction and testing of a prototype wet oxidation system including a subsystem capable of shredding, holding and injecting dry wastes into the reactor. It also stipulated that electrical signals be available to monitor the controls for proper operation, and that oxygen generation by electrolysis inside the reactor be investigated experimentally. Due to problems encountered with the shredding of dry trash, and clogging of the reactor inlet, the contract was revised. The revision added reactor modification and stirring rate tests as tasks, deleted the requirement for electrical monitoring signals, and reduced the scope of electrolytic oxygen generation to obtaining design information only. In the course of lab work done on the processing of dry trash, it became more apparent that this involved more formidable problems than could be addressed on this contract. Consequently, effort was redirected to develop a more basic understanding of the shredding and transport problems as applied to this process and waste model, and the constraints of zero "g" and weight, power, and scale characteristic of manned space flight. Also, a new task was added to perform a material balance test.

After extensive lab work, analytical studies and consultations with experts concerning the shredding of trash, and the pumping and transport of slurries, it was apprent that the manned spacecraft application of wet oxidation for other than urine and feces processing is not feasible at this time. The primary conflicting constraints are: 1) a high flow velocity is needed to enable slurries to move thru conduits, 2) the design feedrate is verylow, the amount of water is limited, and the viscosity is high; 3) zero "g", and severe power, weight and scale constraints limit the acceptability of such solutions that can be envisioned at this time. Therefore, it was decided to discontinue work on dry trash. Instead, the investigation of post-treatment clean up of the liquid effluent, was initiated, and additional tests were planned to determine lower limits ofthe design parameters: reactor temperature, pressure, stirring rate and feed rate.

SYSTEM DESCRIPTION

DESCRIPTION OF THE ORIGINAL SYSTEM

The wet oxidation system as it existed at the end of the previous contract (NAS 1-11748) is shown in the schematic diagram, Figure 1. The input dry trash processor module is shown at the upper left corner. Because this module could not process the dry trash satisfactorily, and because dry trash slurry clogged other portions of the system, the module was abandoned. Immediately below is the main slurry hold tank and its associated nitrogen pressurization source. This forced slurry at 20 psig into the high pressure pumping system immediately to the right. The slurry was forced into two bladdered tanks (#29) when the motor driven inlet valve (#10) was opened by the timer (#45) for a fill cycle. Simultaneously the bladder pushed water out of the bottom of the tanks (#29) thru the open solenoid valve (#20) into the water reservoir (#35). After sufficient time to ensure tank filling, timer #45 closed fill valve #10 and solenoid valve #20, allowing water pump #30 to pressurize the bladder tanks to the pressure (e.g. 2500 psig) at which relief valve #21 was set. The timer then opened motorized valve #11 to allow delivery of the slurry to the heat exchanger and reactor.

The catalyst supply system is shown directly above the slurry system. At the top of this schematic diagram is the oxygen supply subsystem, which employed a regulator to maintain a constant delivery pressure approximately 50 psi above the reactor pressure. This oxygen subsystem and the slurry pumping subsystem met at a tee which fed through motorized valve #8 into the reactor (#38) via the heat exchanger (#36), thereby heating the influent and cooling the effluent. The reactor was equipped with a motor driven stirring device having a magnetic coupling to avoid dynamic seals. Finally, the oxidized effluent left the heat exchanger, passed through a water cooler, and a dome loaded back pressure

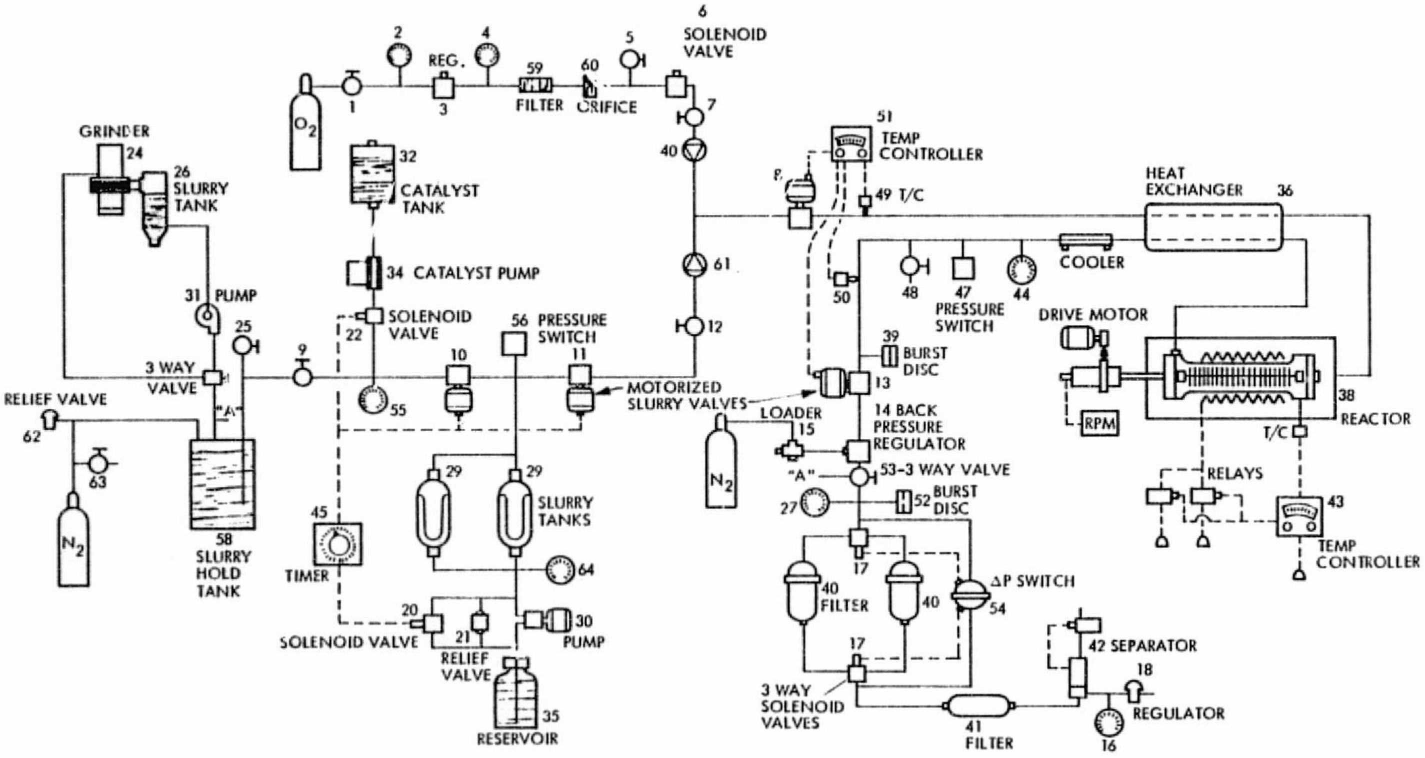


Figure 1 Schematic Diagram of Previous System

regulator (#14) which allowed flow whenever its set pressure was reached, so as to maintain the desired reactor pressure. Finally the effluent flowed thru filters (#40 and 41) to remove ash and then thru a separator (#42) to allow gas and liquid to leave via their separate effluent lines.

COMPONENT CHANGES AND IMPROVEMENTS

As the wet oxidation equipment was operated in the laboratory, certain component and system changes were required for functional reasons and others were made to improve reliability or safety. The shredding and transport of dry trash was unsatisfactory and this portion of the input subsystem was separated from the main system (see detailed explanation in a later section).

The two bladder tanks which had been used to provide an interface between the slurry and the pressurizing water had functioned successfully with sewage waste. However, they were not suitable for use with dry trash and so were replaced with a single hydraulic piston/cylinder. The cylinder was fabricated from 303 stainless steel tubing. A specially designed 4 way head was made of 316 stainless steel, and the piston rod was made of carbon steel, hard chrome plated.

Reactor

The Hastelloy C-276 ball bearings which supported the internal stirring rod were very costly and proved to have a short life. After replacing them three times, it was decided to switch to journal type bearings made of carbon. To increase the life of these new bearings the end closure of the reactor was lengthened, enabling the bearings to be removed from the hottest area. In addition this end closure was fitted with an external water cooling sleeve. This arrangement proved to be very satisfactory in use. Another improvement was needed to prevent clogging of the reactor inlet. The design was changed to enlarge the inlet fitting and to provide adequate internal clearance so that the stirring mechanism and internal baffles would not impede the inflow of slurry. Similarly, the internal spider assembly was anchored to preclude blockage of the outlet port.

HEAT EXCHANGER

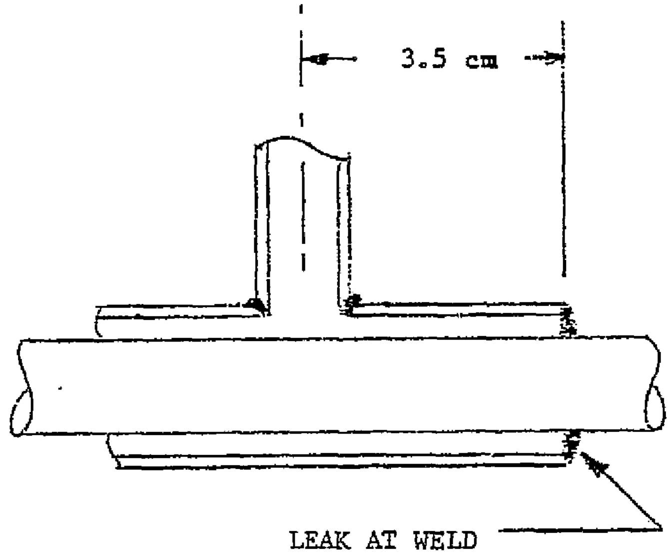
The design of the Hastelloy C-276 heat exchanger proved to be thermally satisfactory. However in February 1976 a pin point leak began at a weld near the inlet from the reactor. This is the hottest location in the entire heat exchanger. The fabricator had left an unnecessarily long overhang of the outer tube beyond the entry point of the tubing leading from the reactor. See Figure 2. The stagnant area of the flow annulus promoted inter-granular stress corrosion. See Figure 3. A repair was attempted by removing the weld metal at and around the leak, then rewelding with Hastelloy C-276 weld rod. Following this, a leak test was performed and the leakage proved to be even worse. Thereupon it was decided to have a new heat exchanger fabricated, but without the two overhangs which created the stagnant flow areas. The new heat exchanger was leak tested and installed, and then leak tested in situ. It has been operating satisfactorily ever since.

EFFLUENT FILTERS

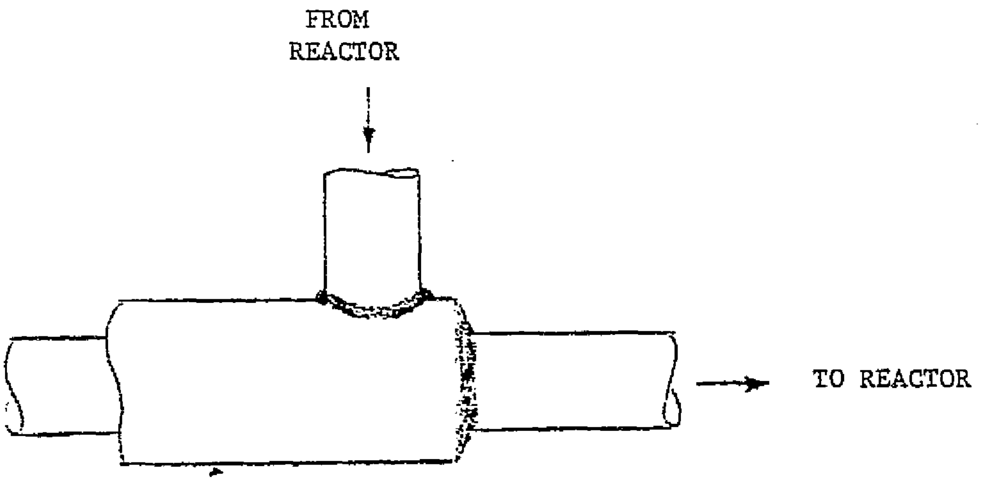
Due to the very small amount of ash produced, it became apparent that only one primary filter was needed, rather than two in parallel. It was also determined that the final filter (for finer particle size) was not needed, so it was removed. It was found that the latter filter (Pall Trinity Micro Corp. MCN 4463-VR-J) had corroded, whereas the two primary filters (GAF Corp. RBK 316 SS) had not.

OXYGEN REGULATOR

The original high pressure oxygen regulator was a Consolidated Controls Corp. 1381-KXT-C4T unit. This supplied oxygen at a constant pressure (within a regulation band), ordinary 50 psi above the nominal reactor pressure. Since the reactor pressure was controlled by a back pressure regulator, it too varied within a range (approximately ±50 psi). Consequently, the pressure difference between the oxygen supply and the reactor varied appreciably. It was even possible for the ΔP (and the flow of oxygen) to become zero. Con-



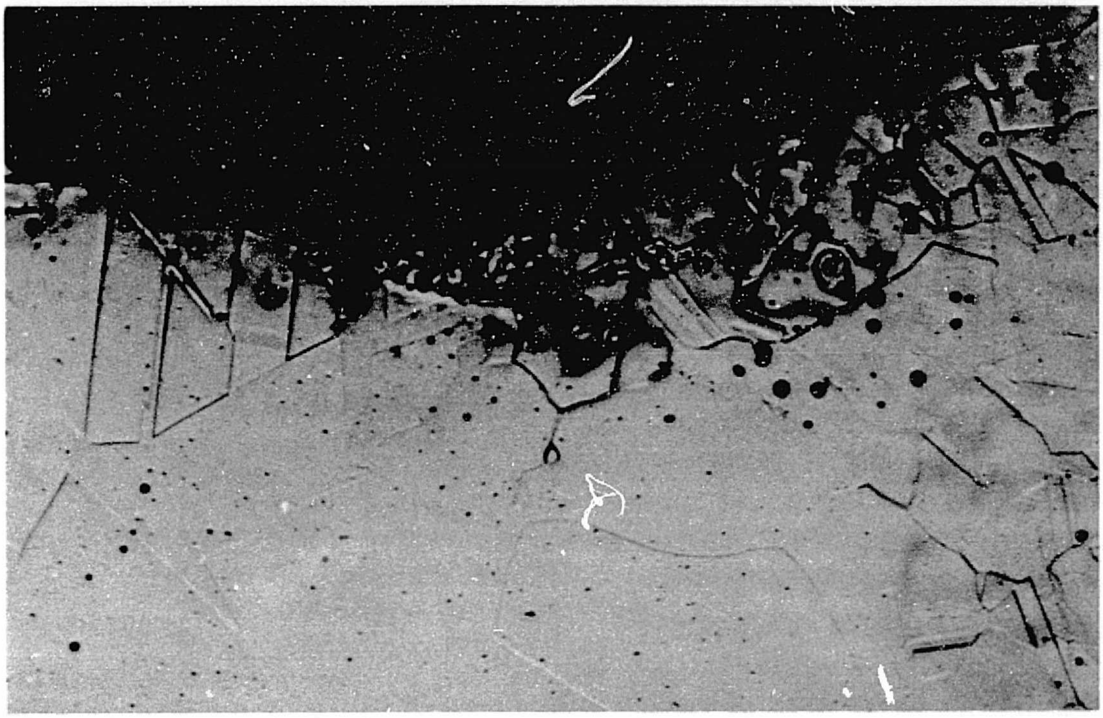
ORIGINAL HEAT EXCHANGER DETAIL



NEW HEAT EXCHANGER DETAIL

Figure 2 LOCAL DETAIL OF THE HEAT EXCHANGER LEAK AREA

2



500 X magnification, showing corrosion pit.
Inner surface of Hastelloy C-276 tube, carried
raw slurry out of heat exchanger into reactor.

Figure 3 Microphoto of Heat Exchanger Tube sequently it was decided to obtain a regulator which would maintain a constant Δ P. A survey disclosed that such regulators are not available, and furthermore only one company was willing to custom build such a regulator. This was Consolidated Controls. They designed and built a Δ P regulator with a diaphram/seat assembly which is loaded both by a pressure dome and an adjustable spring. The Δ P can be varied from 50 to 130 psi. This unit was designated model no. 1381 x 3900.

MOTOR DRIVEN BALL VALVES

These four valves are driven by Raymond Controls model MAR 8 actuators. Originally each actuator drove its respective valve via a coupler having set screws. Set screws occasionally loosened, causing couplers to slip on their shafts. To prevent such valve malfunctions, the set screw couplers were replaced with slotted couplers which engaged the flats on the shafts of the actuators and the valves. Thereafter, slippage was impossible.

WATER PUMPS

Pursuant to component improvement, mechanical drives were investigated as an alternative to the hydraulic pumps currently used to actuate the slurry injection pump. Screw jack actuators were considered first. These consist of a rotating nut whose outer circumference is a gear which is driven by a worm. The nut-to-jackscrew interface can be either an Acme thread or a more efficient ball bearing screw. The worm would be driven by a reversible multi-speed DC motor thru a speed reduction gear. However it was found that the manufacturer did not recommend using the screw jack in continuous duty applications at their maximum loading.

Next, a rack-and-pinion gear was considered. But for the force required, the steel rack would be 1.5 inches high X 2.5 inches wide. This, together with the other necessary drive components would result in an excessively large and heavy assembly. Consequently, it was decided to have our existing pumps rebuilt by the manufacturer (Milton Roy Co., Laboratory Data Control Division).

CONTROLS

Figure 4 is a system schematic diagram, showing primarily the mechanical components and fluid lines. Figure 5 is an electrical schematic diagram The number designation for a given component is consistent in both figures. Table I presents the function of the individual controls required during normal operation. The system was designed to operate automatically, once started, if slurry and oxygen are supplied. It was also designed to shut down safely and automatically if a hazardout condition is approached. There are a number of displays, both mechanical (e.g. - pressure gages) and electrical (e.g. - lighted pushbutton switches) which enables monitoring the condition of various components and subsystems. Similarly, there are various controls, both mechanical (e.g. - manually operated valves) and electrical (e.g. - pushbutton switches) which can be used to manually override the automatic controls. In addition to his manual override option, the operator can select either of two automatic modes: Day or night. Table II enumerates the safety controls which prevent hazardous situations. If the operator selected the "Night Control" mode of automatic operation, existence of a potentially hazardous situation causes the closing of Reactor Isolation Valves (8, 13) as well as turning off the main electrical power. This is possible because Isolation Valve power is separate from the main power source. If the operator selected the "Day Control" mode of automatic operation, the Reactor Isolation Valve (8 and 13) close and an alarm rings.

This situation requires an operator to respond, to assess the situation by inspection of the pressure gages, reactor temperature indicator and various position indicators, and take necessary action (i.e. - actuate manual controls as appropriate to the situation). Table III identifies the instruments and displays from which the operator can assess the status of the system.

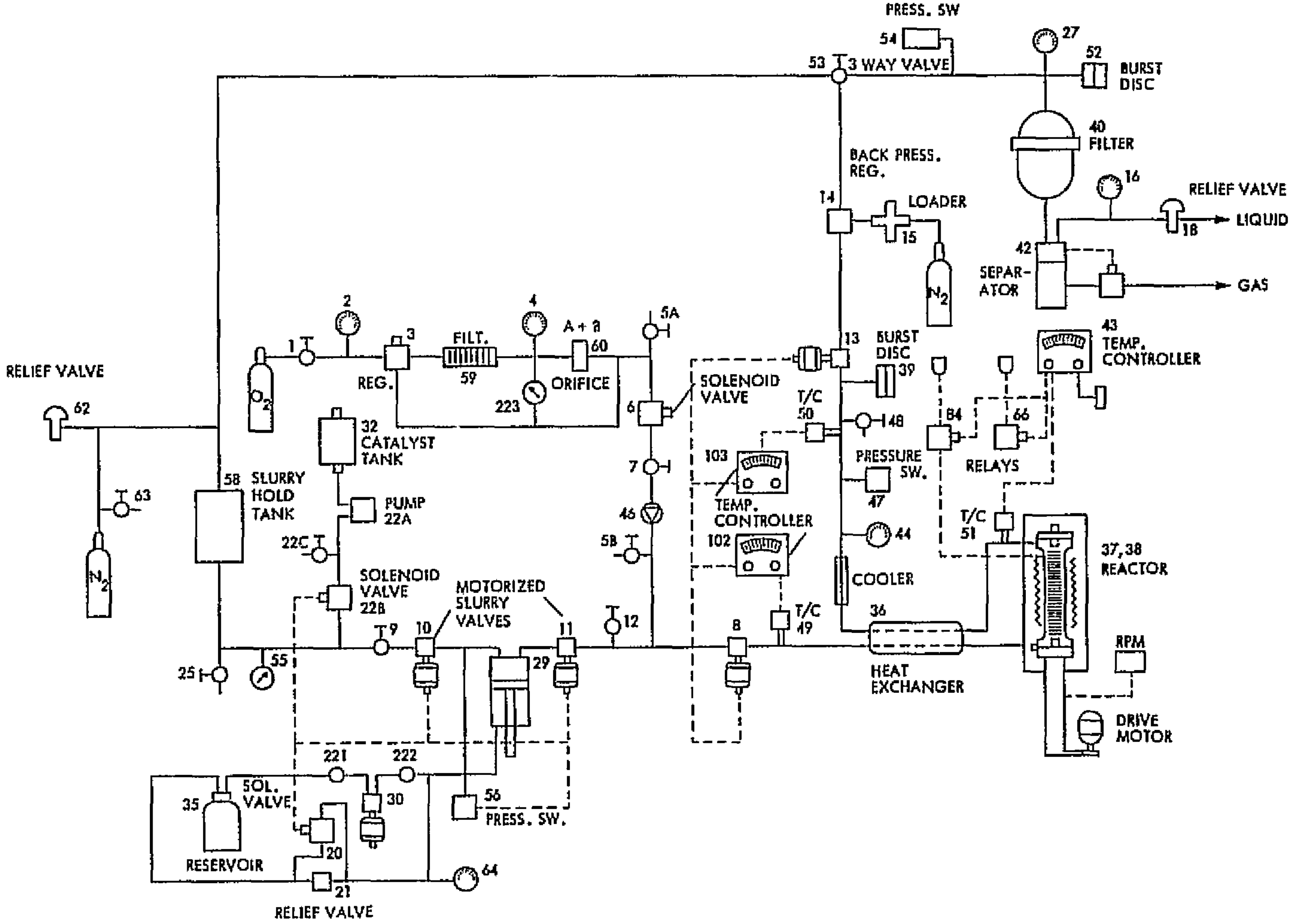
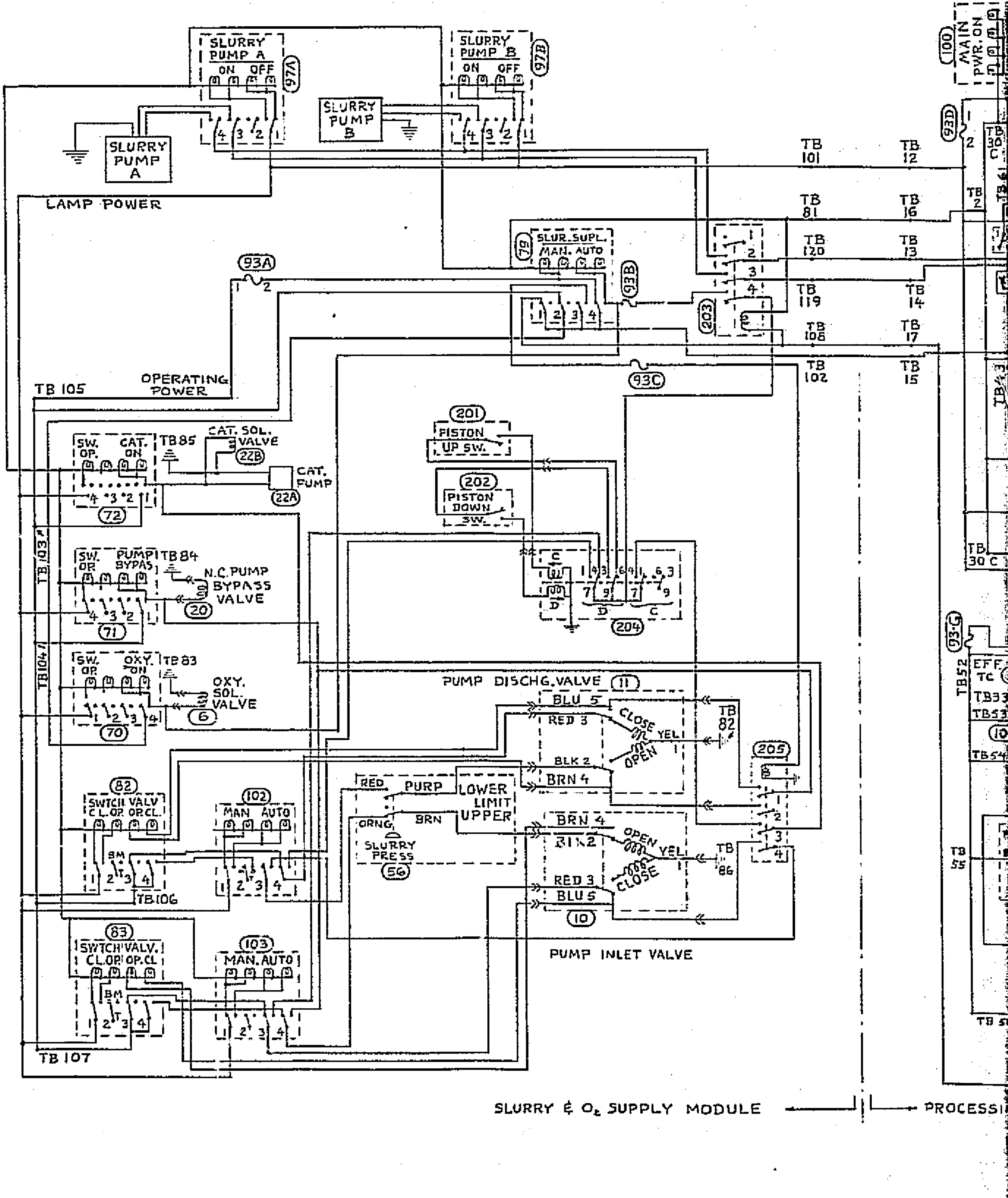


Fig. 4 NASA Wet Oxidation System Schematic



FOLDOUT FRAME

REPRODUCIBILITY OF THE ORIGINAL PAGE IS POOR

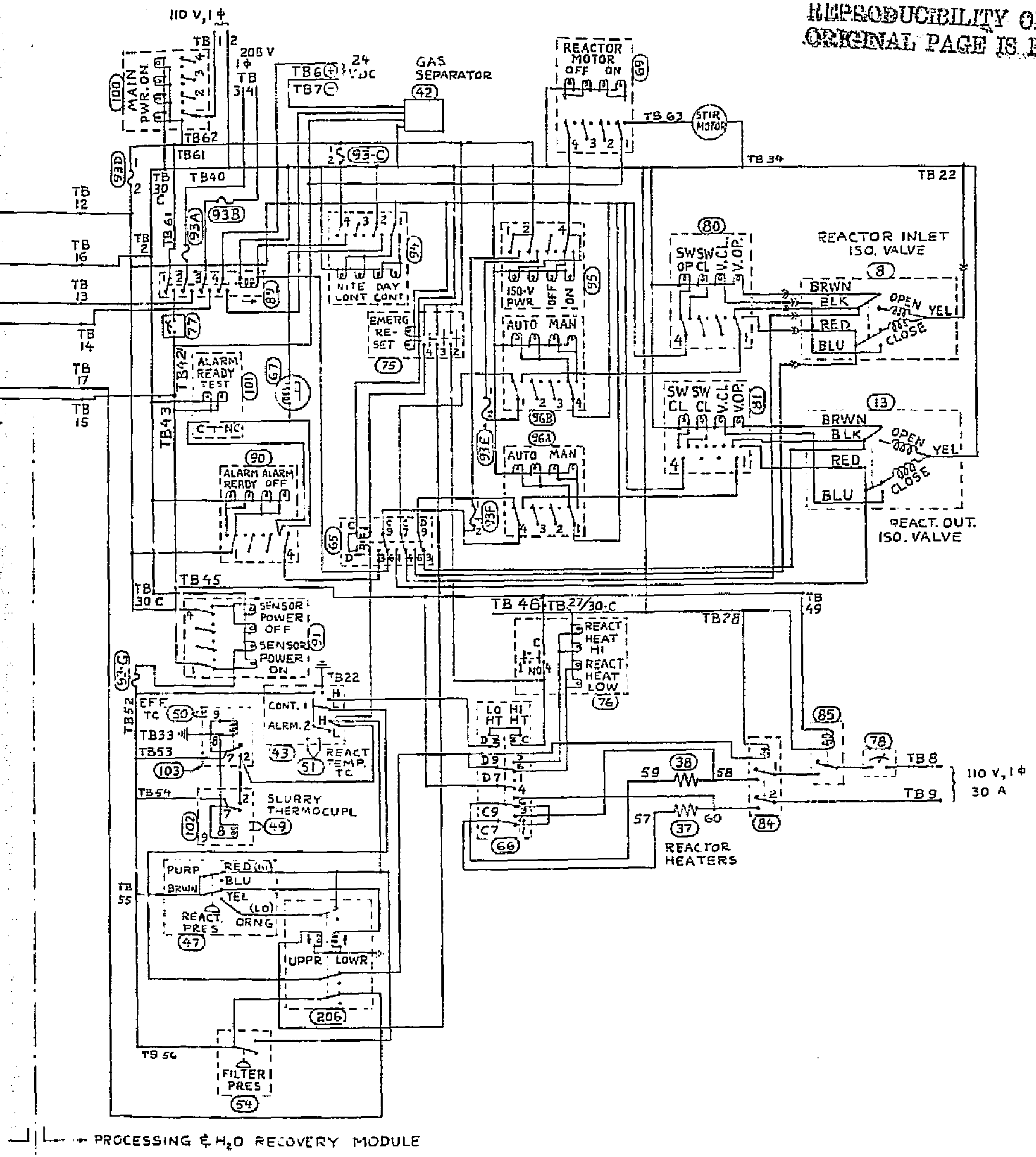


Figure 5 Electrical Schematic Diagram

FOLDOUT FRAME 2

Table I Automatic Controls for Normal Operation

Controller	Controlled Element	Controlled Condition
Oxygen Reulator (3)	-	Maintains a constant differential pressure (above reactor pressure) across orifices 60 A and B.
Main Power Switch (100)	Oxygen Solenoid Valve (6)	Oxygen supply is on or off depending upon main power switch.
Nitrogen Tank Regulator	Slurry Hold Tank Pressurization (58)	Maintains constant pressure to force slurry from Hold Tank into Pump cylinder (29).
Piston Up (201) and Piston Down (202) Switches	Catalyst Pump (22A) and Solenoid Valve (22B)	Catalyst injected into slurry supply line during filling (piston downstroke).
Switches (201, 202) and Slurry Pressure Switch (56)	Motor Driven Slurry Supply Valves (10 and 11) and Hydraulic Solenoid Valve (20)	Slurry Outlet Valve (11) closes, then Hydraulic Sol. Valve (20) opens, dropping water press. Slurry Inlet Valve (10) opens allowing pressurized slurry to force Piston (29) down. At bottom of stroke, Switch (202) triggers closing of valves (10) and (20). Hydraulic pumps (30) then increase the pressure until Slurry Pressure Switch (56) reaches it's upper trip point, above reactor pressure, at which time valve (11) opens.

Table I (continued)

Pressure Switch (47)	Reactor Heaters (37, 38) Water Pumps (30)	During system startup, it is necessary for press. switch (47) to reach the lower of its two set points to turn the Reactor Heaters (37, 38) and Water Pumps (30) on. This ensures that there will be fluid flow thru the reactor so that thermocouple (51) will be heated by effluent fluid. This precludes the possibility of over heating the reactor, which could happen if the heaters were on before any hot fluid raised the temperature of thermocouple (51).
Temperature Controller (43)	Reactor Heaters (37, 38)	Temp. Controller (43) has 2 set points. During startup, this controller turns on Reactor Heaters (37, 38) in parallel for hi heat input. When the thermocouple (51) reaches the lower set point temp., Controller (43) turns the Heaters (37, 38) off. Thereafter, each temperature excursion below the lower set point will turn on the Heaters connected in series for low heat input.
Back Pressure Regulator (14)	-	Maintains constant reactor pressure by releasing effluent fluid whenever reactor pressure exceeds the pressure inside the regulator's nitrogen pre-charged dome.
Slave Impeller rpm of Phase Separator (42)	Gas solenoid valve of Phase separator (42)	Opens gas solenoid valve when gas accumulates, reducing rpm of slave impeller.
Relief Valve (18)	-	Opens liquid effluent line when its set pressure is exceeded.

Table II Safety Controls to Prevent Hazardous Situations

Controller	Controlled Element	Controlled Condition
Relief Valve (62)	Slurry Hold Tank (58)	Pressure cannot exceed a safe valve.
Relief Valve (21)	Water pressure loop	Water Pumps (30) cannot build up excessive pressure.
Press. Switch (56) low set point.	Slurry Inlet Valve (10)	Valve (10) will not open unless slurry pump cylinder (29) pressure is below low set point.
Press. Switch (56) high set point	Slurry Outlet Valve (11)	Valve (11) will not open unless slurry pump cylinder (29) pressure is greater than reactor pressure.
Temp. Controllers (102, 103)	Reactor Insulation Valves (8, 13)	If any of these controllers sense that one of its set points have been exceeded, it will act to close valves (8) and (13). If the system has been placed in "Day Mode" it will also sound an alarm. If in "Night Mode" it will turn off the main electrical power.
Temp. Controller (43)		
Min. & Max. limits of Press. Switch (47)		
Filter Press. Switch (54)		
Burst Disc (39)	Reactor, Heat Exchanger (36) and associated high pressure plumbing.	When the maximum safe pressure is reached, Burst Disc (39) ruptures, reducing pressure to atmospheric.
Burst Disc (52)	Filter (40), Phase Separator (42) and associated low pressure.	When the maximum safe pressure is reached, Burst Disc (52) ruptures, reducing pressure to atmospheric.

Table III System Instruments and Displays

Direct Reading

- Oxygen Supply Pressure (2)
- Regulated Oxygen Pressure (4)
- Slurry Hold Tank Pressure (55)
- Slurry Tank Fill Pressure (55)
- Slurry Pump Pressure (64)
- Slurry Hold Tank Level (sight glass)
- Hydraulic Reservoir Level (visual)
- Slurry Pump Piston Position (visual)
- Motorized Valve Position Indicators (10), (11), (8), (13)
- Reactor Drive RPM (tachometer)
- Reactor Temperature (43)
- Reactor Heater Current (ammeter)
- Reactor Heater Voltage (voltmeter)
- Reactor Pressure (44)
- Filter Inlet Pressure (27)
- Effluent Water Pressure (16)
- P Across Oxygen Orifices (223)

Panel Light Indicators

- Reactor Isolation Valve Position (80,81)
- Reactor Heater Power (High or Low Heat Indication - 43)
- Catalyst Pump Power On or Off (72)
- Hydraulic Pumps "A" Power On or Off (97A)
- Hydraulic Pumps "B" Power On or Off (97B)
- Hydraulic Pump Bypass Solenoid Power On or Off (71)
- Oxygen Solenoid Valve Power On or Off (70)
- Slurry Inlet Valve Position (83)
- Slurry Outlet Valve Position (82)

ELECTRICAL POWER SYSTEM

The electrical system schematic is presented by Figure 5. All controls and electrical equipment use 110V, 60Hz, 1ø ac power, except for the phase separator which also requires 28V dc and the hydraulic pumps which require 208V, 60Hz, 1ø ac. All components are wired for automatic control with manual override. The hydraulic pumps, phase separator, reactor stirring drive motor, and fail safe shutdown controls are wired to operate any time that the main power switch is closed. The reactor heaters are controlled by the reactor temperature controller. The fail safe shutdown controls turn off all power and close the isolation valves if the night mode is selected and ring an alarm and close the isolation valves if day control is selected.

SYSTEM PACKAGING

The system was packaged and arranged in four modules for laboratory testing. The oxygen and nitrogen supply tanks and slurry hold tank formed one module. The pulverizer subsystem consisting of pulverizer, hold tank, recirculation pump and three way valve constituted the second module. These two modules were not as sophisticated with respect to hardware design or arrangement as the remaining two and were considered to be experimental. The remaining two modules, representing the heart of the system, were packaged to simulate a spacecraft installation. The smaller module on the left of Figure 6 housed the slurry, oxygen and catalyst supply subsystems. The larger module on the right contained the isolation valves, heat exchanger, reactor, cooler, back pressure regulator, filter, phase separator, effluent regulators, and associated hardware. All displays and controls associated with the components in a given module were located on the front of that module.

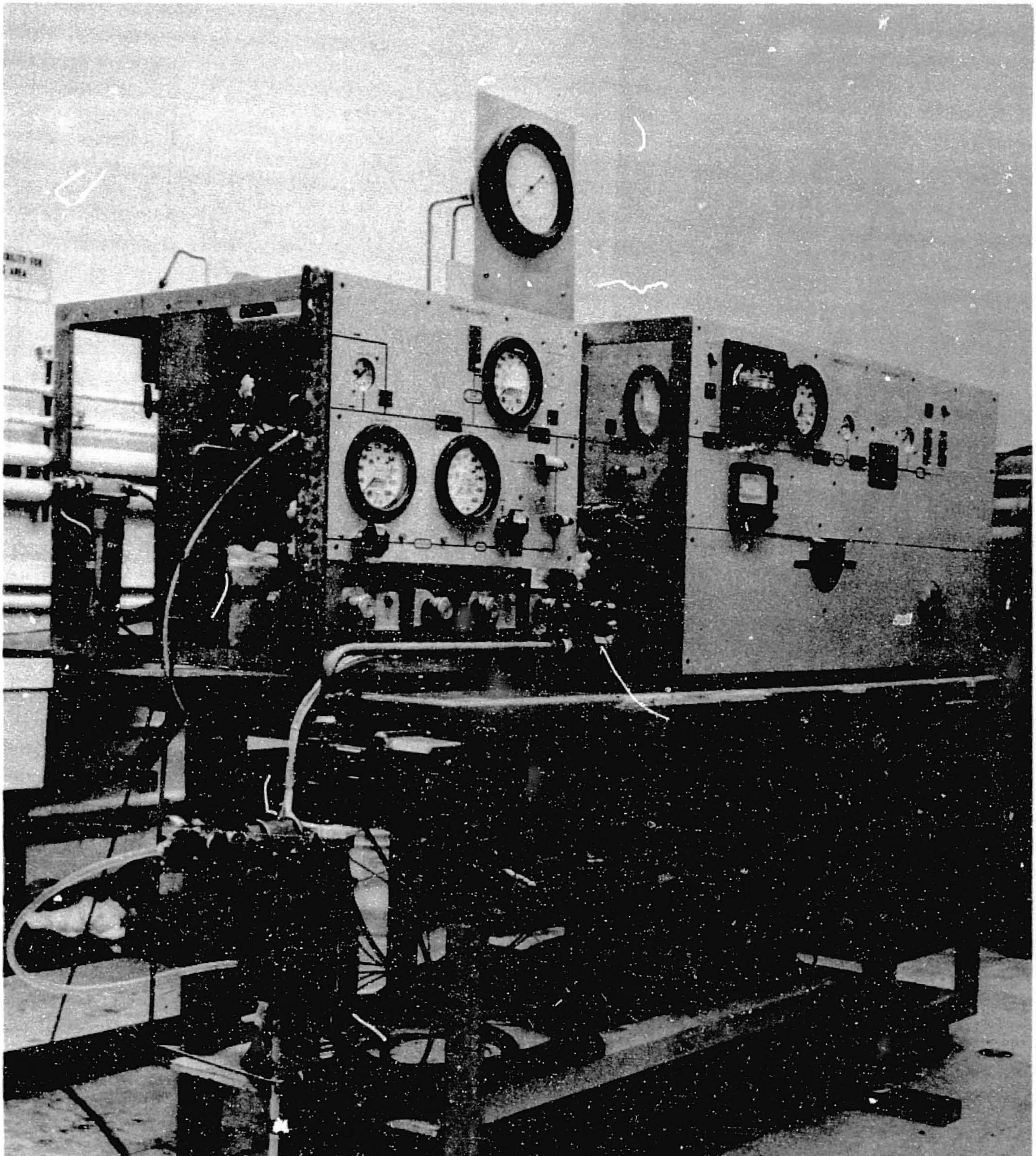


Fig. 6 Photo of Wed Oxidation Modules

TESTING

ELECTRICAL CHECKOUT TESTING

This testing was done to verify operation of all electrical equipment. The procedure was to first verify continuity of all electrical circuitry by measuring all wiring point to point for proper resistance values. Secondly, all power resistances to ground were checked for proper values. All switches were then put in open condition, fuses removed, and power distribution terminals disconnected. Power was then introduced sequentially to small sections of the circuit and component operations verified until all sections were checked.

MECHANICAL CHECKOUT TESTING

These tests were performed to determine whether flow paths were installed as planned, and whether there were any leaks. The method used was to open all flow paths (except oxygen and catalyst loops), pressurize to two atmospheres (gage) with nitrogen and check for leakage with soap solution. Next, oxygen was introduced into the isolated oxygen loop and joints were checked using Leak-Tec solution.

Water was introduced into the Slurry Hold Tank (58) and the system was filled with water. All gases were bled off. The back pressure regulator (14) was charged to system proof pressure while operating the Slurry Pump (29). At progressively higher pressures, the system was observed for leaks until proof pressure (20,700 kPa = 3000 psi) was reached. At this point, pressure was released, water drained, and regulator (14) set for operating pressure. All mechanical devices such as pressure relief valves, rupture discs, check valves, gages and regulators were checked for correct installation and adjusted to proper values. Then the liquid pumping subsystem including the four Water Pumps (30) and the Slurry Pump (29) was operated (using water instead of slurry) to verify that it could deliver a pressure up to 21,400 kPa (3100 psi) at which point the relief valve (21) opened.

SYSTEM TESTS

Some of the component tests were actually conducted within the system. This mode of operation obviated the requirement for separate component test setups. At a system level, the controls were tested for their adequacy in providing safety as well as semi-automatic operation.

CONTROLS TESTS

The purpose of this test was to verify that the controls subsystem provided for safe automatic or manual system operation. The mechanical and electrical subsystems had earlier been checked out and adjusted. The procedure was to confirm the safety aspects of the subsystem, then verify the correct automatic operation, then verify correct manual operation.

SAFETY VERIFICATION

Certain out-of-tolerance critical parameters will cause the system to shutdown. Night shutdown consists of turning off all power to the Slurry and O_2 Supply Module except for the indicator lamp power bus. This effectively stops and isolates the catalyst supply, oxygen supply, and slurry supply. At the same time all power is turned off to the Processing and Water Recovery Module except for the indicator lamp power bus, and isolation valve actuators (8) and (13), which close their respective isolation valves. Day shutdown consists of ringing an alarm bell and closing isolation valves (8) and (13). This alerts the day shift operator who has the opportunity to correct the fault. At night while the system is unattended, the preferred mode is to accomplish an alarmless full shutdown.

Six parameters will cause a shutdown and they were tested as follows:

Isolate and vent all systems as follows:

- Close-off N_2 Supply to Holding Tank (58)
- Place 3-way Valve (53) in Filter Position
- Close Valve (9)
- Close Drain Valve (25)
- Open Vent Valve (63)
- Open Vent Valve (22C)

Close Valve (12)

Close Valve (1)

Close Valve (7)

Open Valve (5A)

Open Valve (48)

Open Valve (5B)

Energize and test sensors in sequence as follows:

A. Disconnect terminals (TB-52), (TB-53), (TB-54), (TB-55), and (TB-56).

B. Open circuit breaker (77).

C. Plug in 110 VAC to wall.

D. Place MAIN POWER switch (100) ON.

E. Place DAY/NITE CONTROL switch (94) in DAY position.

F. Place ALARM READY/OFF switch (90) in READY position.

G. Momentarily press EMERGENCY RESET switch (75).

H. Close circuit breaker (77).

I. If alarm did not sound at step F, momentarily press ALARM READY TEST and verify alarm working.

J. Place SENSOR POWER ON/OFF switch (91) in OFF position.

K. Reconnect terminal (TB-52).

L. Place SENSOR POWER ON/OFF switch (91) in ON position.

M. Set right red pointer of reactor temperature controller (43) at 600°F.

N. Touch hot soldering iron to thermocouple (51) and observe green temperature indicator needle rise to 600°F and observe alarm ring.

NOTE: If soldering iron not hot enough the test can be performed at a lower temperature.

O. Place ALARM OFF and SENSOR POWER OFF. Disconnect terminal (TB-52) and reconnect terminal (TB-53).

P. Place SENSOR POWER ON, and momentarily press EMERGENCY RESET.

Q. Adjust controller (103) to 120°F.

R. Subject thermocouple (50) to 120°F heat source and observe alarm ring.

S. Repeat steps O through R with controller (102), thermocouple (49) and terminal (TB-54).

NOTE: If 120°F heat source not available, the test can be performed by adjusting controllers to ambient temperature. Return controllers to 120°F setting afterward.

T. Place ALARM OFF and SENSOR POWER OFF. Disconnect terminal (TB-54) and reconnect terminal (TB-55).

U. Place SENSOR POWER ON, and momentarily press EMERGENCY RESET.

V. Remove cover of pressure switch (47) and manually move bourdon tube until alarm rings.

W. Repeat steps T through V with red conductor of pressure switch (47) disconnected.

X. Place ALARM OFF, SENSOR POWER OFF, reconnect red conductor of pressure switch (47), disconnect terminal (TB-55), reconnect terminal (TB-56).

Y. Place SENSOR POWER ON, and momentarily press EMERGENCY RESET.

Z. Temporarily apply an 80 psig source to filter pressure switch (54) observe alarm ring.

ZZ. Place ALARM OFF, SENSOR POWER OFF, reconnect terminals (TB-52), (TB-53), (TB-54) and (TB-55). This completes shutdown sensor test.

Correct operation of shutdown is verified by placing the system in operation, triggering one of the shutdown parameters and observing shutdown events as follows:

A. With system isolated and vented and the filter pressure switch (54) shutdown parameter still in effect.

   1. Connect 110 VAC to wall plug.

   2. Connect 208 VAC to wall plug.

   3. Connect 110 VAC 30 AMP to wall plug.

B. Place MAIN POWER switch (100) ON. Observe indicator lights come on.

C. Close circuit breaker (77).

D. Place DAY/NITE CONTROL switch (94) in NITE position.

E. Place SENSOR POWER ON/OFF switch (91) to ON position.

F. Place phase separator (42) in operation. Refer to phase separator operation manual.

G. Place ALARM READY/OFF switch (90) to READY.

H. Place ISOLATION VALVE POWER ON/OFF (95) to ON position.

I. Place AUTO/MANUAL switches (96A) and (96B) to AUTO position.

J. Place REACTOR INLET ISOLATION VALVE switch (80) to SWITCH CLOSE position.

K. Please REACTOR OUTLET ISOLATION VALVE switch (81) to SWITCH CLOSE position.

L. Place REACTOR MOTOR ON/OFF switch (69) to ON position.

M. Close circuit breaker (78).

N. Momentarily press REACTOR HEAT HI/LOW switch (76).

O. Place SLURRY PUMP A ON/OFF switch (97A) to ON position.

P. Place SLURRY PUMP B ON/OFF switch (97B) to ON position.

Q. Place SLURRY SUPPLY MAN/AUTO switch (97) in AUTO position.

R. Close CATALYST switch (72). Switch OPEN indicator goes out.

S. Close PUMP BYPASS VALVE switch (20). SWITCH OPEN indicator goes out.

T. Close OXYGEN SOLENOID VALVE switch (6). SWITCH OPEN indicator goes out.

U. Place SLURRY INLET VALVE switch (83) in SW CLOSE position.

V. Place SLURRY OUTLET VALVE switch (82) in SW CLOSE position.

W. Place SLURRY INLET VALVE switch (103) in AUTO position.

X. Place SLURRY OUTLET VALVE switch (102) in AUTO position.

Y. Momentarily press EMERGENCY RESET switch (75).

Observe:

1. Reactor isolation valves (8) and (13) open.
2. Reactor stirring motor run.
3. Phase separator (42) run.
4. Reactor heaters come on in high heat mode.
5. Metering pumps (30) start running.
6. If slurry pump piston rod is fully retracted, pump inlet valve (10) will open. Catalyst pump (32A) will start pumping. Pump bypass valve (20) will open. Oxygen solenoid valve (6) will close.

Z. Trigger any one of the six parameters tested before and observe automatic night shutdown events.

1. Reactor isolation valves (8) and (13) close.
2. Reactor stirring motor stops.
3. Phase separator (42) stops.
4. Reactor heaters go off.
5. Metering pumps (30) stop.
6. If slurry pump piston rod is fully retracted: catalyst pump (22A) will stop, pump bypass valve (20) will close, and oxygen solenoid valve (6) will close.

ZZ. Repeat the above steps except that before step Z, place DAY/NITE CONTROL SWITCH (94) in DAY position, then an automatic day shutdown would occur with the following observations:

1. Alarm (67) rings.
2. Reactor isolation valves (8) and (13) close.

Automatic Operation. Automatic operation is accomplished by closing all manual vent valves and opening all manual input valves so that automatic components may take over system control. Then the system is powered up and started. When proper operating conditions are attained, the process will proceed until a shutdown, either real or simulated, is incurred. The procedure is as follows assuming all supply tanks are filled.

Close vents and open input manual valves as follows:

A. Place 3 way valve (24C) in grinder recirculate position.
B. Close drain valve (25).
C. Place 3 way valve (53) in filter position.
D. Close vent valve (63).
E. Open N_2 supply to holding tank (58) adjust regulator to 20 psig.
F. Close vent valve (22C).
G. Open valve (9).
H. Open valve (12).
I. Close valve (5A).
J. Close valve (5B).
K. Close valve (48).
L. Open valve (1).
M. Open valve (7).

Repeat Electrical Steps (A) through (Y).

Observe:

1. Reactor isolation valves (8) and (13) open.
2. Reactor stirring motor run.

3. Phase separator (42) run.
4. Reactor heaters come on in high heat mode (76).

When reactor temperature reaches operating set point, slurry pump (29) will commence pumping. Catalyst and oxygen will be introduced depending on the position of slurry pump piston rod.

Manual Operation. At any time, the system may be operated manually. Numerous safeguards are installed which prevent casualties from occurring. In general, each electrically operated component can be turned on or off independently except where an installed safeguard interlock prohibits. Assuming the system to be in automatic operation, the following elements may be functioned manually.

Phase Separator (42). Switches on control panel. Refer to phase separator operation manual.

Reactor stirring motor. REACTOR MOTOR ON/OFF switch (69).

Reactor Isolation Valves (8) and (13).

1. Place ISOLATION VALVE AUTO/MANUAL switches (96A) and (96B) in MANUAL position.
2. Operate valves open and closed using switches (80) and (81).

Reactor Heaters. Heaters may be turned off by opening circuit breaker (78). High heat mode is set by momentarily pressing REACTOR HIGH/LOW HEAT switch (76). Low heat mode may be set by adjusting left control flag of (43) to cross green temperature indicator.

Metering Pumps (30). SLURRY PUMP A and B ON/OFF switches (97A) and (97B).

Slurry pump inlet and outlet valves (10) and (11). These valves have pressure switch (56) override which prevents their opening when too large a pressure differential exists. To operate valves:

1. Place SLURRY SUPPLY AUTO/MAN switch (79) in MAN position.
2. Place MANUAL/AUTO switches (102) and (103) in MANUAL position.

3. Place SW OPEN/CLOSE switches (82) and (83) in CLOSE positions. Observe valves closed.
4. Operate PUMP BYPASS VALVE switch (71) to close switch. Observe SW OPEN indicator go out. Observe accumulator pressure drop. When pressure less than 100 psig proceed to next step.
5. Place SW OPEN/CLOSE switch (83) in OPEN position. Observe valve (10) open and pump (29) fill. When pump full, return switch (83) to CLOSE.
6. When accumulator pressure has risen to 2000 psig, place SW OPEN/CLOSE switch (82) in OPEN position.

Oxygen solenoid valve (6). Operate oxygen switch (70).

Catalyst solenoid valve (22B) and pump (22A). Operate catalyst switch (72).

CALIBRATION

The pressure gages mounted on the control panel of the system as well as the O_2 supply pressure gage were calibrated in the Measurement Standards Laboratory of LMSC to verify that they were within the manufacturer's specified tolerances. Similarly the temperature controller was checked to verify that its trip points were within tolerance for various indicated settings. A stroboscope was used to calibrate the tachometer indicating the reactor stirring shaft. The calibration graph is shown in Figure 7.

Prior to running the material balance test, it was essential to have a calibration for the oxygen feedrate vs. oxygen supply pressure. A large pressure gage having a 10 inch diameter dial graduationedin 5 psi increments was calibrated using two wet test meters in series. The data was plotted as shown in Figure 8 "Oxygen Delivery vs. Supply Pressure Decrease". Both of the high pressure oxygen tanks were connected in parallel, and are always used in this manner. The best calibration of the slurry feed system was obtained as a result of the material balance test. With two of the reciprocating water pumps set at 37%, the slurry pump piston moves at a rate of 1 inch per 3 minutes and 40 seconds during its delivery stroke. This results in an average slurry delivery rate of 470 ml/hr. This slurry feedrate was half of the "standard rate of 940 ml/hr."

The LMSC report for the preceding contract (Report #1-11748, page 81) has a graph of the delivery characteristic of the Precision Control Products Model 10641-11 metering pump which was used to deliver the catalyst solution. At a 20 psi delivery pressure and at 40% setting, delivery is 0.78 ml per minute (two pulses per minute).

PARAMETRIC TESTING

In order to determine optimum operating conditions, a series of tests was run. It was desired to change only one independent variable at a time, and to keep all of the other independent variables constant so that the cause of changes in dependent variables could be clearly determined. All of these tests were performed on a slurry having the same recipe: 500 gm of human feces, 7,608 gm

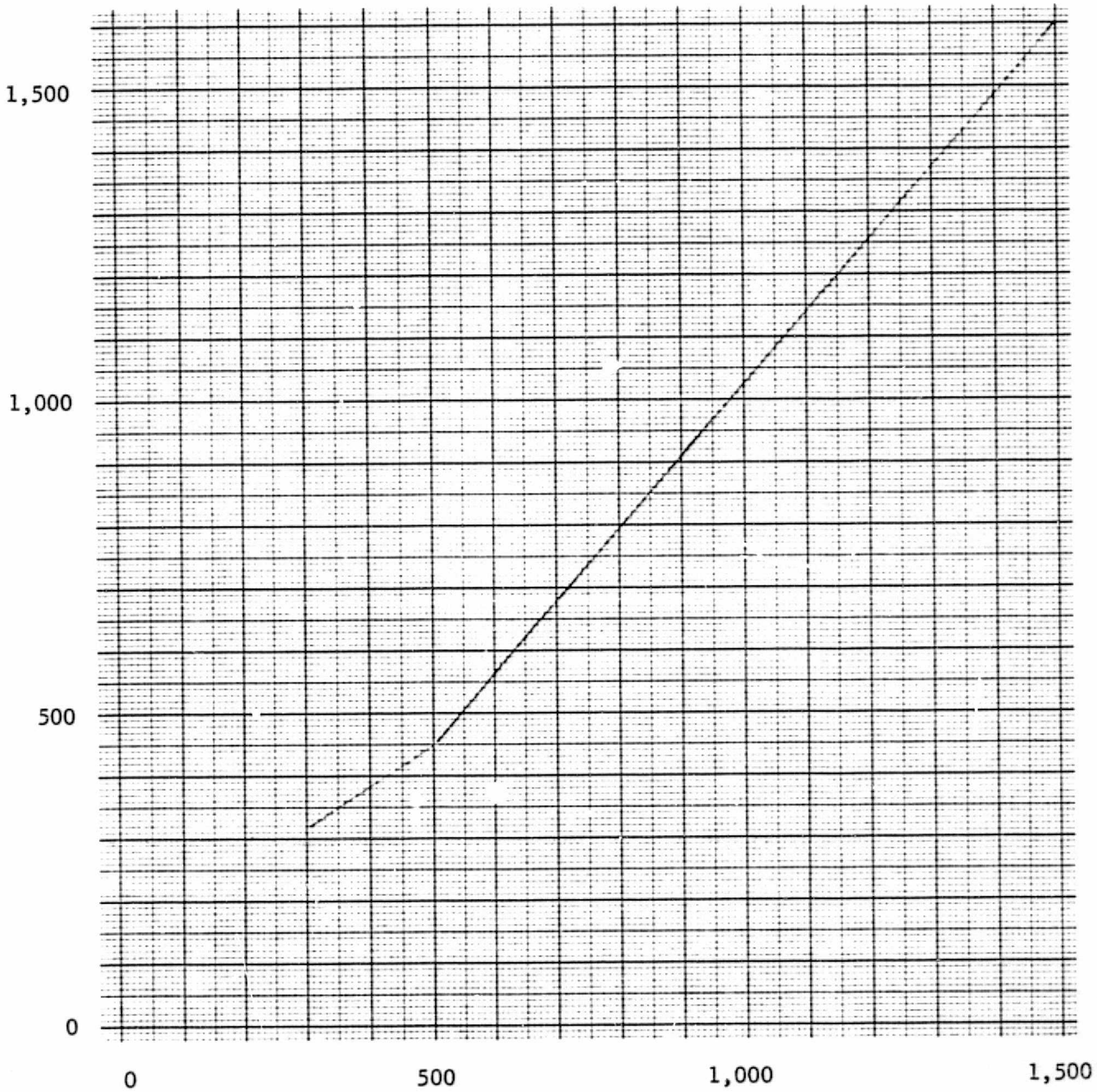
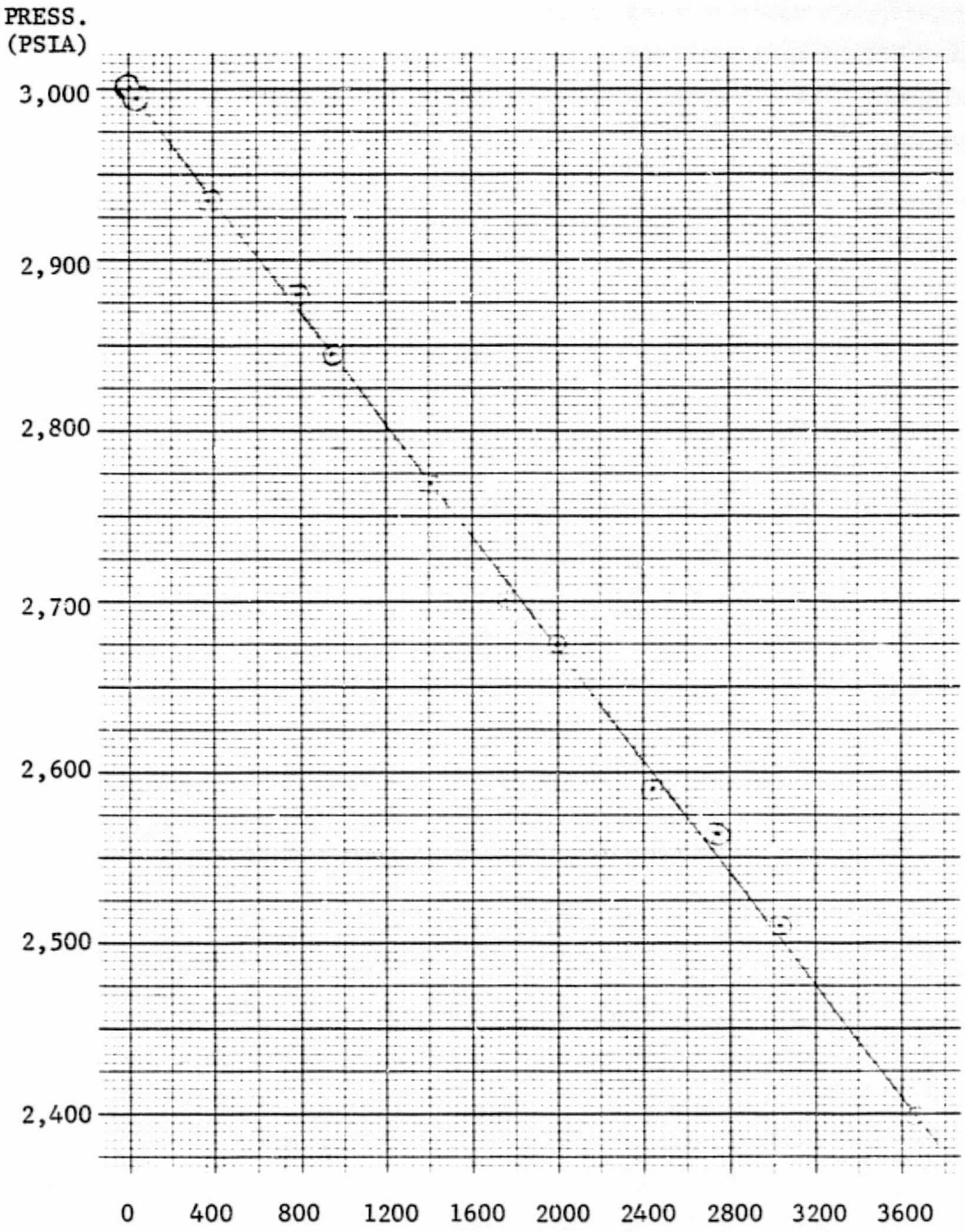


Fig. 7 Reactor Stirring Rate Calibration



LITERS OF OXYGEN AT 21°C AND 760 mmHg

Fig. 8 Oxygen Delivery vs Supply Press. Decrease (6.1 l/psi)

Conditions: Feedrate = 940 ml/hr
Reactor Temperature = 288° C
Pressure = 15,200 kPa
Catalyst: $RuCl_3$
Excess Oxygen Used

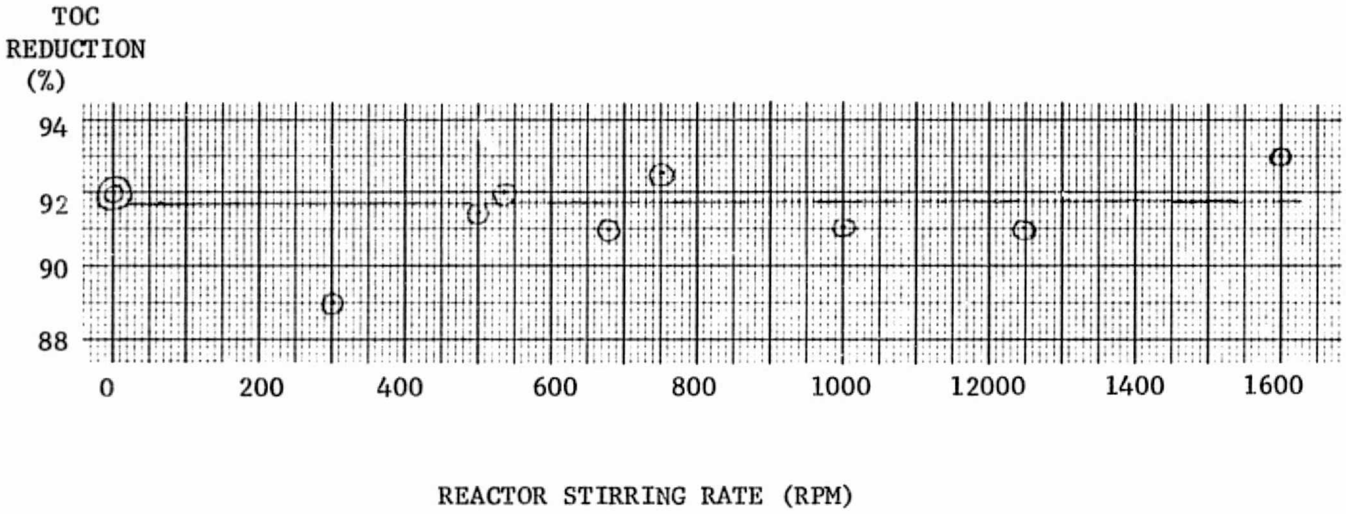


Fig. 9 Effect of Reactor Stirring Rate

Conditions: Reactor Pressure = 15,200 kPa

Temperature = 288°C

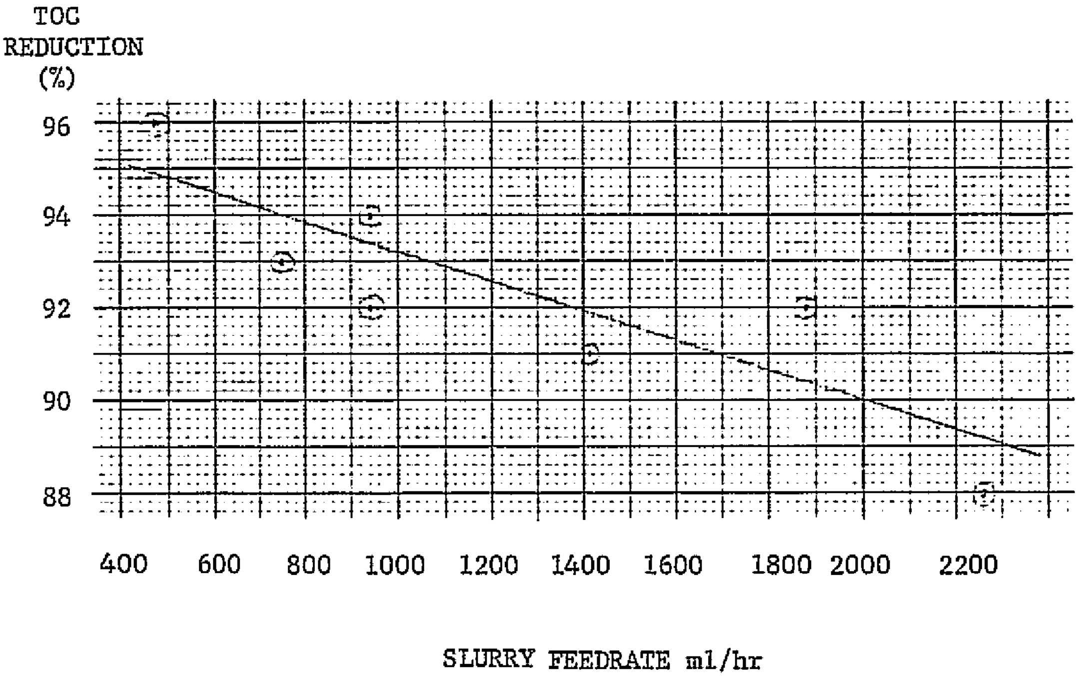


Fig. 10 Effect of Varying Slurry Feedrate

EFFECTS OF VARYING OXYGEN FEEDRATE

Since it is desirable to minimize oxygen consumption, a number of tests were run to determine the effect of reducing oxygen feedrate. The operating conditions for these tests were a slurry feedrate of 470 ml/hr; reactor pressure of 6,480 kPa (940 psi); and reactor temperature of 243°C (470°F). For slurry having a Chemical Oxygen Demand (COD) of 13.533 gm per liter, the above slurry feedrate would theoretically require 6.36 grams of oxygen per hour. For the room temperature and pressure oxygen density of 1.318 gm/l, this amounts to an oxygen flowrate of 80.35 ml/min.

Another method can be used to establish a baseline for oxygen demand. During the material balance test, it was found that the amount of oxygen actually consumed was .01225 gm per gm of slurry (yielding a TOC reduction of 86.7%). At a slurry feedrate of 470 gm/hr, this amounts to 5.756 gm of oxygen per hour, or a flowrate of 72.79 ml/min.

Three methods were used to vary the oxygen flowrate during this series of tests:

- Changing the flow restrictors
- Varying the pressure drop across the restrictors
- Turning the oxygen on and off periodically.

First, two restrictors were used in series, a Lee VDLA-1815313K and an unidentified unit. Since further reduction of flow was needed, the most restrictive unit which Lee makes was purchased and installed; model VDLA 4315220T. This unit was then used in place of the other two, and finally all three were used in series.

The Consolidated Controls Corporation model 1381 x 3900 differential pressure regulator was used to vary the Δ P across these restrictors. During some test runs the oxygen solenoid valve was operated by a timer to supply oxygen intermittently. It was found that if the cycle duration exceeded 16 minutes, TOC reduction deteriorated excessively. The theoretically calculated reactor residence times for the existing operating conditions are: liquid = 1½ hours, gas = 14 minutes.

Figure 11 is a graph showing the variation of TOC reduction with oxygen content of effluent gas. Figure 12 shows the effect of oxygen feedrate on TOC reduction. These curves show that the oxygen feed rate can be considerably reduced below the excessive rates which had normally been used. However if a certain TOC reduction is to be achieved, there is a minimum required oxygen feedrate which is several times greater than the theoretical chemical oxygen demand.

of human urine; and 13,420 gm of tap water. These ingredients were individually measured, then mixed using a Waring blender. The resulting slurry consistently had a specific gravity of 1. However, its Total Organic Carbon (TOC mg/l) varied appreciably. The catalyst, ruthenium trichloride hydrate, was automatically injected by a metering pump in the proportion of 0.04329 gm of solid per liter of slurry. Unless otherwise specified, the standard slurry feedrate was 940 ml/hr. An excess of pure oxygen was fed into the reactor for all tests except the series of tests seeking minimum oxygen feedrate.

REACTOR STIRRING RATE TESTS

A series of tests were made to determine the effect of varying the reactor stirring rate. The reactor temperature was 288°C; pressure 15,200 kPa; catalyst was $RuCl_3$ and excess oxygen was supplied. Results are shown in Figure 9 - the stirring rate has no appreciable effect on TOC reduction.

EFFECT OF VARYING REACTOR PRESSURE

Several tests were made at a reactor temperature of 288°C (550°F) and at the standard slurry feedrate of 940 ml/hr. The reactor pressures used were 12,400 kPa (1800 psi) and 15,200 kPa (2200 psi). The results were:

REACTOR PRESS. kPa	TOC (Total Organic Carbon) % Reduction
15,200	92
15,200	94
12,400	86

The poorer performance at lower pressure is consistent with the data plotted graphically on page 102 of the final report for the preceding contract NAS 1-11748.

EFFECT OF VARYING SLURRY FEEDRATE

A series of tests was performed at a reactor temperature of 288°C (550°F) and pressure of 15,200 kPa (2200 psi). The results are plotted on a graph (Fig. 10). It shows that TOC reduction diminishes as the slurry feedrate increases.

Conditions: Slurry Feedrate = 470 gm/hr

Reactor Temperature = 243°C

Pressure = 6,480 kPa

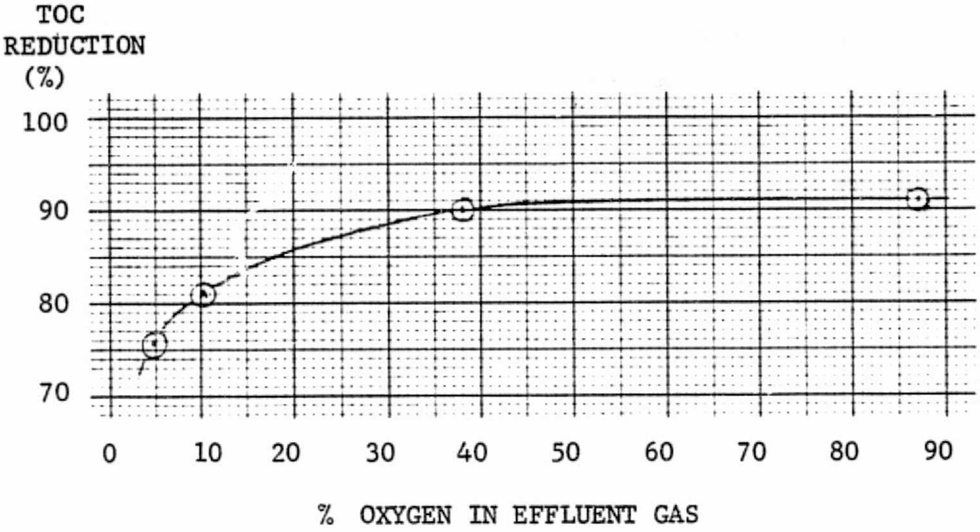


Fig. 11 Variation of TOC Reduction with Oxygen Content of Effluent Gas

Conditions: Slurry Feedrate = 470 gm/hr

Reactor Temp. = 243°C

Pressure = 6,480 kPa

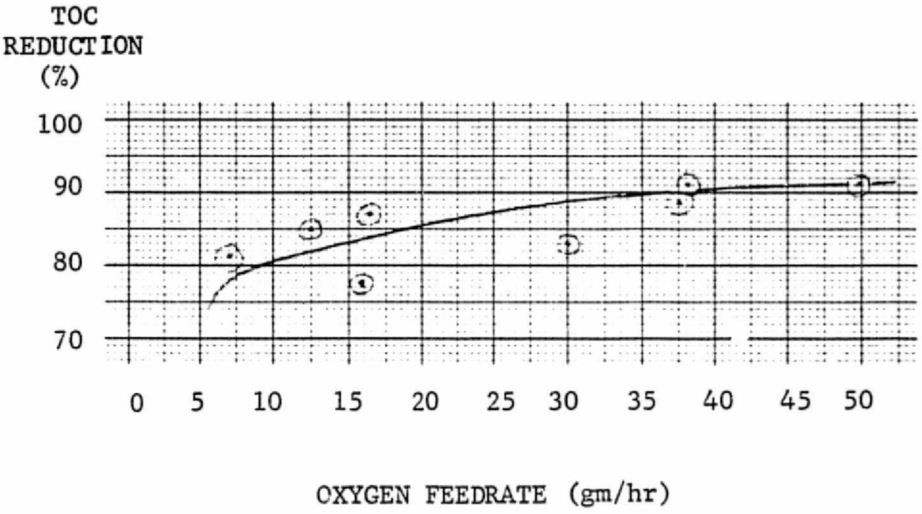


Fig. 12 Effect of Oxygen Feedrate on TOC reduction

POST-TREATMENT OF LIQUID EFFLUENT

In the wet oxidation process, the addition of ruthenium trichloride as a catalyst greatly improves the reduction of TOC, but it has the undesirable side effect of making the liquid very acidic. If a process such as Vapor Compression Distillation is used as a post-treatment following wet oxidation, acidity is very undesirable. The reason for this is that organic acids will be carried over during distillation. Therefore it would be desirable for the wet oxidation effluent to have a pH above 7. The pH could be increased by adding an alkali either before or after wet oxidation. It was reasoned that it would be more desirable to add the alkali before wet oxidation for two reasons: (1) acidity in the wet oxidation reactor and heat exchanger would be reduced, thereby improving the reactor environment, and (2) the addition of an alkali such as NaOH would precipitate out ruthenium in compounds, keeping the catalytic affect, but greatly reducing the etching effect on the reactor walls.

Accordingly, four test runs were made in a batch type wet oxidation reactor. Test A used only sewage, Test B added ruthenium trichloride catalyst which had been titrated with sodium hydroxide, Test C added only ruthenium trichloride to the sewage, and Test D was similar to B except that a much larger amount of sodium hydroxide was added. Results showed that the ruthenium was an equally effective catalyst in B, C and D producing about the same reduction of TOC.

Reactor temperature was 288°C and pressure was 15,200 kPa. Results of these 4 tests are tabulated below:

Test	Final pH	Final TOC mg/1	Ruthenium in end product
A	7.8	122	
B	2.6	29	
C	2.4	28	2 ppm
D	4.2	29	2 ppm

It is believed that because of the presence of carbonic acid in the reactor even large excesses of sodium hydroxide would not completely neutralize the liquid. Therefore it appears preferable to add the alkali after, rather than before wet oxidation.

Next, a series of tests was run for two major purposes. One was to determine whether Vapor Compression Distillation (VCD) is a satisfactory process for purifying the effluent water from the wet oxidation process, or whether an additional step such as charcoal filtration is required. The criteria used to judge water quality were Total Organic Carbon (TOC), electrical conductivity, odor and color. The effluent from the wet oxidation process has objectionable odor and color under all reactor conditions which were used. The electrical conductivity was also too high, due to dissolved salt content. Selection of VCD as the prime post-treatment process enabled simulation in the laboratory by duplicating the distillation at the same conditions that exist inside a VCD machine; pressure 730 mm of mercury below ambient and temperature approximately 32°C. If the wet oxidation effluent liquid is to be vacuum distilled, this opens the possibility of reducing the effluent quality level and brining it up to the required standards by post-treatment. This complements the other major purpose of this test series: to determine the feasibility of operating the reactor at pressures below 6,895 kPa (1000 psi) in order to use available spacecraft oxygen sources. Reactor temperatures must be correspondingly reduced to reduce water vapor pressure, thereby adequate oxygen partial pressure. Incidentally, the weight and power consumption of the wet oxidation system could be reduced as reactor temperature and pressure are lowered. The lower quality of effluent liquid due to reduction of temperature and pressure can be somewhat mitigated by reducing the slurry feedrate, so several runs were made at half the standard feedrate. Table IV gives the conditions and results of this test series. We may make the following observations:

- Increasing pH to a neutral or basic value has a very beneficial effect on TOC. Therefore it should be considered as a pretreatment preceding VCD.
- Vacuum distillation greatly reduces TOC and electrical conductivity, but color and odor remain (except for the product of the 260°C, 15,200 kPa wet oxidation). Therefore, a cleanup step of charcoal filtration is appropriate. This further reduces TOC, as well as eliminating odor and color. By quantitative tests it was determined that approximately 1 gram of charcoal is required per liter of water.
- It was decided that wet oxidation conditions of 6,218 kPa (900 psi), 243°C (470°F) and half of the standard slurry feedrate, or 470 ml/hr would be used for the subsequent material balance test.

REVERSE OSMOSIS

Reverse Osmosis (RO) is a general membrane separation process. As such, it can be viewed as a separator or concentrator. For example: Figure 13 shows an RO water system where water with 2000 mg/l of salt is feed to the system. Eighty percent of the water (at 80% recovery) is forced through the membrane along with perhaps 200 mg/l of salt that "leaks" through. The water and salt solution remaining on the high-pressure side of the membrane is called the concentrate. RO systems can be engineered to adjust the recovery rates and salt passage, but the law of Conservation of Mass which is fundamental to all physics, governs: every pound of water that enters the system must leave the system and every pound of salt that enters the system must leave the system. Proper application of an RO system requires the following precautions. An RO unit should not be run at less than its rated flow; if less liquid is handled the RO unit should be run only intermittently. A minimum size unit has a continuous duty capacity of 100 liters per day. Calcium phosphate is a problem, but can be removed by a resin type of ion exchange water softener which also removes nickel, manganese and ferrous ions. However ferric ions will precipitate out on the membrane, clogging it. Therefore unless ferric ions could be eliminated by filtration or other means, their presence could disquality RO. Polymetrics, Inc. made a computer simulation of what their RO

Table IV Effects of Reactor Temperature, Pressure, and Feedrate; Benefits of Post Treatment

Reactor			Additive Before Vacuum Distil.	pH		Vac. Distil. Batch No.	TOC mg/1			Conductivity mho per cm			Odor		Color	
Temp °C	Pres. kPa	Slurry Feed Rate gm/hr		Before Vac. Distil.	After Vac Distil.		After Wet Ox.	After Vac. Distil.	After Charc. Filter	After Wet Ox.	After Vac Distil.	After Charc. Filter	After Vac Dist.	After Char. Filter	After Vac Dist.	After Charc. Filter
260	15,200	940	None	2.6	3.6	1	327	195		6,800	147					
260	15,200	940	NaOH	8.0	7.1	3	327	21		6,700	17		None			
260	15,200	940	NaOH	10.0	9.0	4	377	21		6,700	20		None			
Pure Water			None	5.4	6.0	5		16		1.5	1.4		None		None	
288	12,400	940	NaOH	8.1	9.1	10	194	9		6,000	134		Slight		None	
260	15,200	940	NaOH	7.0	5.2	2	327	22		6,800	20		None		None	
260	7,100	940	NaOH	7.9	8.9	12	640	78	10	6,000	275	160	Slight	None	Slight	None
243	6,210	470	NaOH	7.6	8.5	14	510	20	20	6,100	206	109	Slight	None	None	None
232	5,020	470	NaOH	7.6	8.8	9	980	150	13	6,500	250	188	Yes	None	Ylw-Green	None
232	4,840	940	NaOH	7.9	9.7	6	1475	140		8,000	1,040		Yes		Ylw-Green	
Tap Water			None			-	11			150						

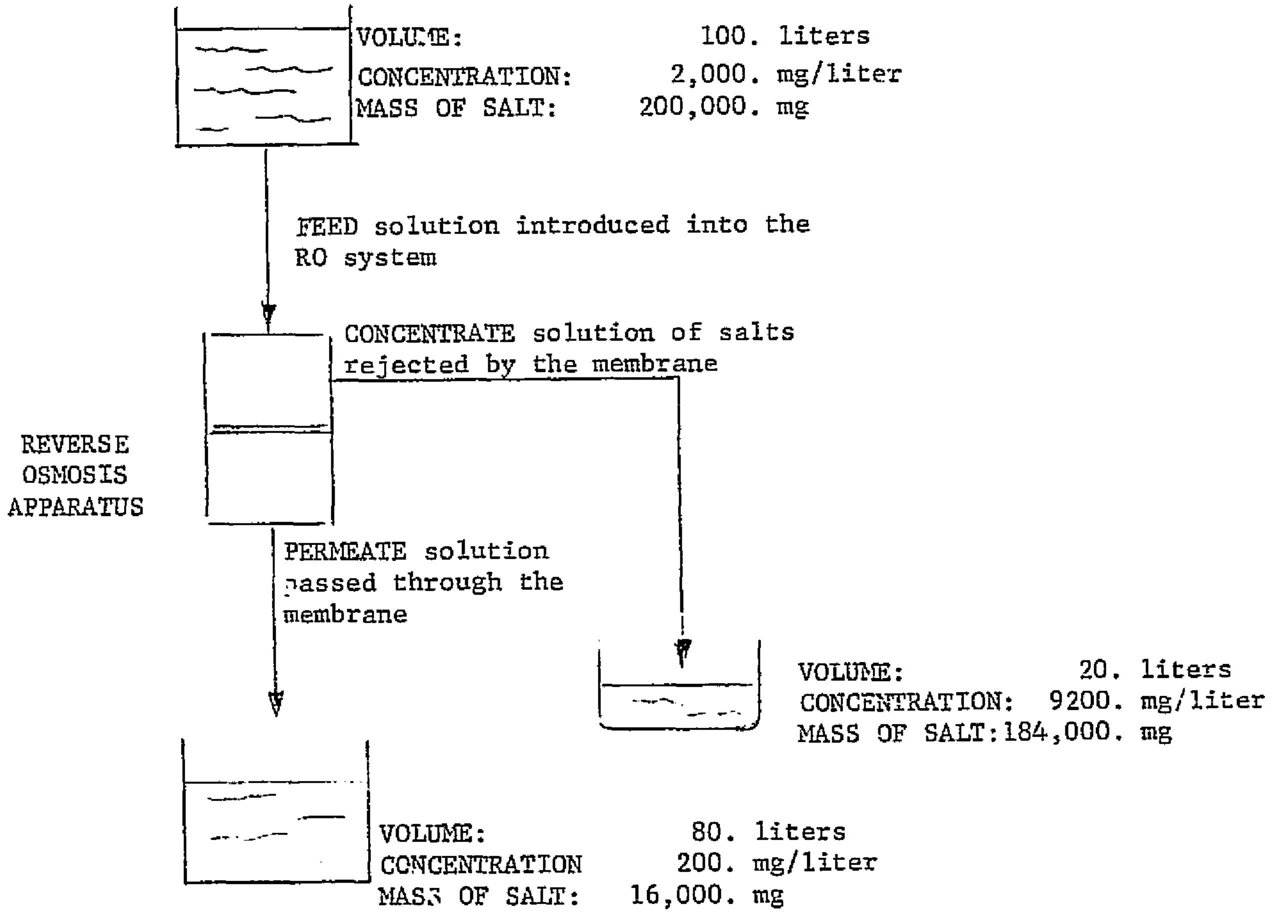


Fig. 13 Reverse Osmosis System as Separator and Concentrator

process would do for our effluent water. They assumed two separate RO stages, the first yielding 90% permeate and the second 50% for an overall yield of 82%. The computer run gave the following results.

Substance	Wet Ox Effluent (mg/l)	RO Product (ppm)	RO Concentrate (ppm)
Na	866	26	7,800
K	926	27	5,500
$S^{=}$	0.27	0.27	-
SO_4	451	2.3	3,800
Cr	2.34	.012	20.
Cl^-	1,679	48	10,000
Mo	7.95	.04	60
Acetate	774	8.2	5,900
Phosphate P	71	6.2	1,300
Total Carbon	514	257	-
NO_3(N)	87.5	5.7	360
Total Dissolved Solids	5,045	120	35,000

ELECTRODIALYSIS

Electrodialysis is a process in which ions are transferred from one solution through a membrane into another solution under the driving force of an electrical potential. The membranes contain ion exchange groups and have either a positive or negative fixed electrical charge. Positively charged anion membranes will allow anions to pass but will repel cations. Negatively charged cation membranes will allow cations to pass but will repel anions. When anion and cation membranes are arranged alternately in a 'stack' and voltage is applied across them, they will act to concentrate the ionic species in one solution that is in contact with them while diluting another.

Ion exchange membranes are relatively impermeable to non-ionic species. However, some transfer of non-ionic species and water will occur. The amount of water transfer depends on the amount of current passed through the membrane and the "tightness" of the membrane. The transfer of water from the diluting to the concentrating stream limits the maximum concentration attainable. It also accounts in part for the volume loss in the stream that is being demineralized.

Electrodialysis stacks consist of anion and cation membranes separated by plastic spacers. These spacers are specifically designed and manifolded to provide uniform flow distribution within the stack and across the surface of the membranes. A stack section that defines adjacent concentrating and diluting compartments is referred to as a "cell pair". It consists of one anion membrane, one cation membrane, and two spacers.

The degree to which a solution is demineralized is proportional to the electrical current flowing through the stack. The current is limited by the electrical resistance of the stack components and by the maximum voltage which can be applied before over-heating or polarization occurs. Therefore, for solutions requiring a large amount of salt transfer or a high degree of demineralization, it becomes necessary to pass the solution through a number of stages to obtain the desired product. This can be conveniently accomplished by a batch operation: a given amount of solution is recirculated through the stack until the average ion concentration is reduced to the required level.

LMSC purchased the services of Ionics, Inc. of Watertown, Mass. to treat 14 gallons of liquid effluent from the wet oxidation system by electrodialysis. The objective was to reduce the electric conductivity of the feed solution to 200 micromhos per cm or less while obtaining the highest possible yield.

Three experimental runs were made. The first two were essentially identical except that one was carried out at a voltage of one volt per cell pair while the other was made at four volts per cell pair. The results indicated that there is little incentive to operate at high voltage as an adequate process rate is achieved at low voltage. The third run was made to determine the processing rate under steady state conditions in the waste stream.

The results from Run #3 indicated that a standard Ionics' Stackpack with a 20 cell pair stack will process 34.07 liters of wet oxidation effluent in approximately 4-½ hours. The product yield is 95% with a total power consumption (including pumping) of 4.52 watt-hours per liter of feed solution.

A study was made to determine the size of electrodialysis equipment required to treat 34.07 liters per day of wet oxidation effluent.

The apparatus used in the study was an Ionics' Stackpack electrodialysis unit set up for batch operation. The stack consisted of 10 cell pairs using Ionics 103QZL-219 anion membranes and 61CZL-183 cation membranes. These membranes are of intermediate "tightness" and resistivity. The electrodes used were a platinized columbium anode and a stainless steel cathode. The effective membrane area of the stack was 0.25 square meters.

Three separate runs were made, each using 10 liters of wet oxidation effluent as the feed. This solution was recirculated through the diluting compartments of the membrane stack. Progress of the runs was followed by periodically measuring the conductivity of the feed solution. Temperature, pH and flowrate of the stream were also recorded along with the DC current. The product yield was determined by observing the level change in the feed batch tank. The batches were stopped when a conductivity of less than 200 micromho/cm was achieved. Samples at the start and finish of the batches were collected.

Seven liters of wet oxidation effluent were used as the waste (or concentrating) stream during Run #1. The conductivity of this solution increased as it picked up ions during the course of the batch. The concentrated solution obtained at the end of the run was used for the waste stream at the start of Run #2. The concentrated solution obtained from the end of Run #2 was used for the waste stream in Run #3.

A third stream was circulated through the two electrode compartment of the stack during the runs. The solution in this stream was made up to be 0.1 N sodium sulfate.

A summary of the results for the three runs is presented in Table V.

During Run #1 one volt per cell pair was applied to the stack. The average temperature during the batch was 30°C. From a starting conductivity of 6928 micromhos per cm, the desired endpoint of 200 micromhos per c m was reached in 105 minutes. Product yield was 96.8%. The calculated processing rate for the run is 24.61 liters per hour per square meter of membrane area. The stack power consumption was 2.85 watt-hours per liter of feed solution.

Run #2 was essentially identical to Run #1 except the applied voltage was four volts per cell pair. The batch reached a conductivity of 200 micromhos per cm in 45 minutes. Product yield was 96.7%. The calculated processing rate is 57.45 1 per hour m^2 with a stack power consumption of 11.25 watt-hours per liter of feed solution.

During Run #2 the concentration of the waste stream leveled off at an electric conductivity of approximately 65,000 micromhos per cm. This is essentially the concentration of the water transfer through the membranes and represents

Table V SUMMARY OF RESULTS - ELECTRODIALYSIS

Batch Volume = 2.64 gallons = 9.99 liters
Membrane Area = 2.5 ft^2 = 2322 square cm

Run No.	Average Operating Temperature C°	Volts per Cell Pair	Conductivity mmho/cm at 21.1°C		Batch Time to Reach 200 mmho/cm mins	Processing Rate (l/hr - m^2)	Yield	Power* Consumption watt-hr/liter	Electrical meq
			Starting	Final					
1	30	1.0	6928	192	105	24.61	96.8	2.85	787
2	30.6	4.0	6043	148	45	57.41	96.7	11.25	966
3	26.7	1.0	7019	200	150	17.24	95.0	3.46	959

*Stack only, does not include pumping power steady state condition. Run #3 was conducted to determine the processing rate at steady state. The applied voltage during the run was one volt per cell pair. The average temperature was 26.7°C. A conductivity of 200 micromhos per cm was reached in 150 minutes with a product yield of 95%. The calculated processing rate for this run is 17.24 liters per hour per square meter with a stack power consumption of 3.46 watt-hrs per liter of feed solution.

Although the processing rate achieved in Run #2 at four volts per cell pair is more than three times the processing rate achieved in Run #3 it should be noted that power consumption is also more than three times greater. Therefore, it is probably more feasible to operate at the low voltage condition. However, a trade-off between power consumption and added weight of membrane area should be made.

The pumping power used by the Stackpack during Run #3 was estimated to be approximately 1.06 watt-hours per liter assuming a 62% combined efficiency for pump and motor.

MATERIAL BALANCE TEST

This test was run to determine by quantitative analysis, a balance of all influents versus all effluents with the wet oxidation system running at stable operating conditions.

Operating Conditions:

A reactor pressure of 6,210 kPa (900 psi) was used since stored spacecraft oxygen is presumed to be available at this pressure. The corresponding reactor temperature selected was 243°C (470°F) and the slurry feed rate was 470 ml/hr. There was no stirring in the reactor. Duration of the test was 77.27 hours, after one day of operation to reach equilibrium conditions. Feedrates of oxygen and catalyst were appreciably above the minimums.

Test Procedures:

Figure 14 is a schematic diagram of the test system. The oxygen input was determined by measuring the reduction in pressure of the two high pressure oxygen tanks using gage P1. A preceding section of this report on "Calibration" gives the relationship between the decrease in tank pressure and oxygen delivered. The rate of oxygen delivery was constant since the regulator maintained a constant differential pressure across the restrictor orifice. No refilling of the high pressure oxygen tanks was required during the test.

The slurry hold tank was filled to a level near the top of the sight glass when the test began. The quantities of feces, urine and water comprising the slurry were weighed for each batch of slurry added. At the end of the test, the amount of slurry required to refill the hold tank to its initial level was weighed and the total was recorded.

Initially, one dose of ruthenium trichloride catalyst was added directly into the slurry hold tank, but when it appeared toward the end of the first batch of slurry that the characteristic dark color due to the presence of catalyst was diminishing, the catalyst metering pump was turned on. The amount of catalyst solution delivered by this metering pump over a given time period was known, therefore total catalyst injection could be calculated.

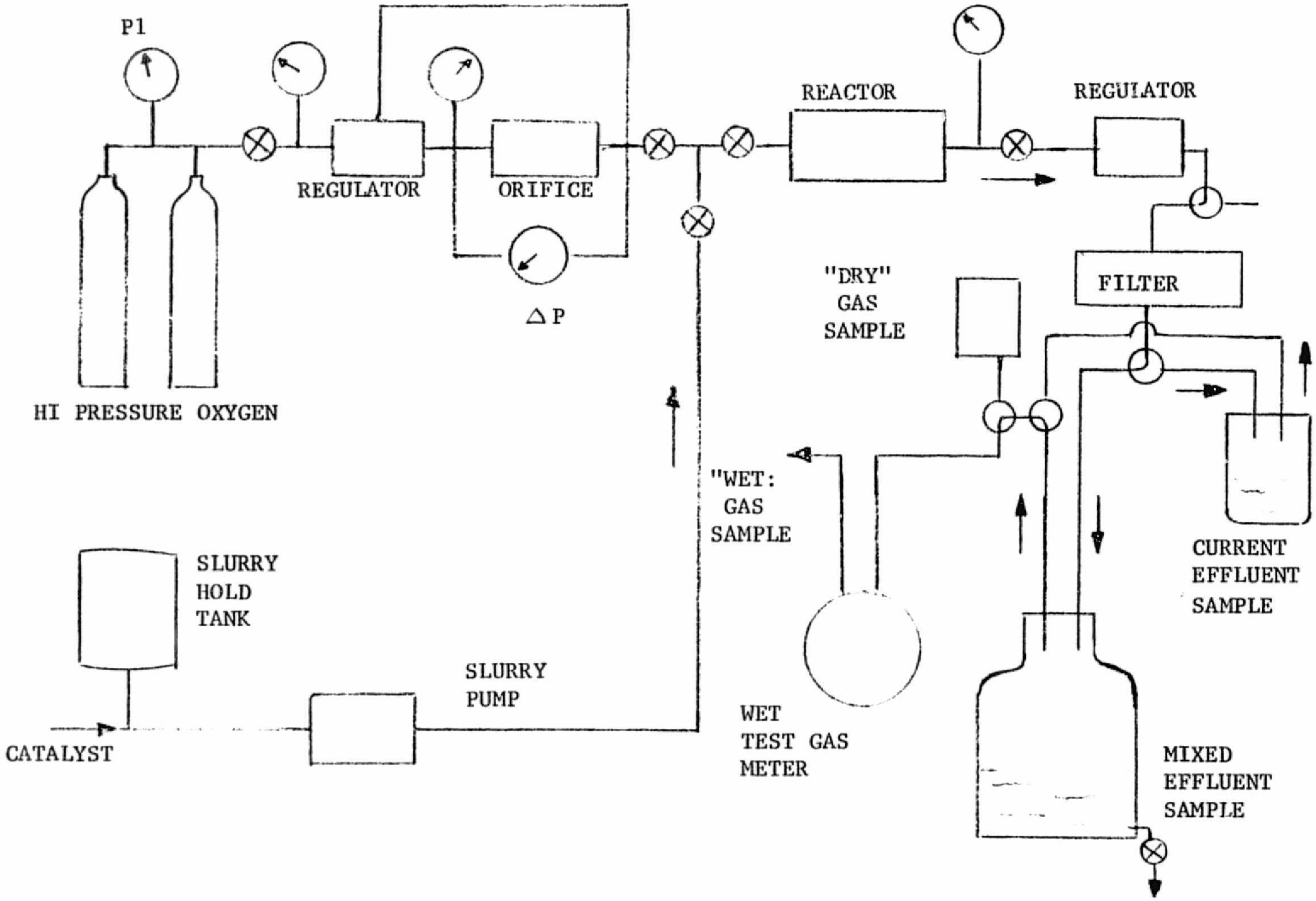


FIGURE 14 SCHEMATIC FOR MATERIAL BALANCE TEST

All effluents leaving the reactor and heat exchanger passed through a 5 micron cloth filter element. A new filter element was weighed and installed before the test. After completion of the test, it was dried and re-weighed. It was found to have collected 0.5 gm of solid ash. After filtration, the effluent was routed to either of two containers which were used to collect the liquid and pass the gas on. The smaller one was used to obtain current samples; the larger one to collect effluent liquid at all other times. All of the effluent liquid was weighed. Cumulative effluent gas was measured by means of a wet test meter. If "dry gas" samples of current effluent were collected for analysis before passing through the wet test meter, the duration of such interruptions was recorded, so that the volume of gas sample could be calculated by rate x time. "Dry gas" samples were taken ahead of the wet test meter to avoid dissolving water soluble gases, although the water probably became saturated with such gases early in the test. Gas samples were taken daily. Two liquid effluent samples were taken daily and mixed together. Two raw slurry samples were taken daily and mixed together.

Methods Used for Chemical Analyses

A variety of substances were required to be quantitatively determined, and to accomplish these analyses, various different analytical techniques were used:

Substances	Analytical Methods
Total Carbon (TC) and TOC	Dohrmann Envirotech Organic Analyzer
Chem. Oxy. Demand (COD) and Total Kjeldahl Nitrogen (TKN)	Standard EPA waste water method
Effluent liquid - metals	Spectrophotometer (Atomic absorption)
Effluent liquid - acetete	Gas chromatograph
Effluent liquid - others	EPA std. waste water analysis methods
Effluent ash - C, H, N	Elemental analysis
Effluent ash - others	Emission spectrograph
Raw influent slurry and effluent liquid - Freeze vacuum dried solid residue - C, H, N, O, S, P	Perkin-Elmer Elemental Analyzer
Effluent gas	Methods are tabulated with the results, below.

The solid material in the influent slurry and the effluent liquid were dealt with in several ways. Some of the influent slurry was filtered, leaving a solid residue, which did not include soluble salts. Another portion of the influent slurry sample was freeze vacuum dried at temperatures below 0°C and pressures of about 755 mmHg below ambient; the solid-residue from this operation did include soluble salts.

Similarly, one portion of the liquid effluent sample was freeze vacuum dried, but another portion was heated to evaporate volatile materials, leaving a solid residue.

RESULTS OF MATERIAL BALANCE TEST

Water vs. Salts vs. Solids

Raw Slurry

Residue filtered out (soluble salts not retained)	6.155	gm/1
Residue from freeze vacuum drying (includes salts)	15.4	gm/1
Difference (soluble salts)	9.24	gm/1
Total soluble salts	335.7	gm
Water probably accounts for nearly all of the remaining	984.6	gm/1
If so, water in 36,333 gm of slurry	35,773.5	gm

Liquid Effluent

Residue from freeze vacuum drying	5.62	gm/1
Residue from hot evaporation at sea level pressure	5.04	gm/1
The small difference may be due to more volatile substances being driven off by heat. Water probably accounts for nearly all of the remaining	994.4	gm/1
If so, water in 36,328 gm of effluent liquid	36,124.5	gm

WATER BALANCE

IN		OUT	
Raw Slurry	35,773.5 gm	Liquid effluent	36,124.5 gm
Water dissolving catalyst	10	Water vapor in effluent gas	54
	35,783.5 gm		36,178.5 gm

The 395 gm difference is probably due to water produced in the reactor.

ANALYSES OF SOLID RESIDUES

	Solid Residue gm/l	C gm	H* gm	N gm	O* gm	S gm	P gm
Raw Slurry	15.4	158.05	29.8	28.1	140.2	0.363	.0945
Effluent Liquid	5.62	2.11	1.21	5.54	29.8	2.99	0.178

*Does not include elemental H and O contributed by water.

OVERALL QUANTITIES

Slurry Input:	Feces	843.8 gm
	Urine	12,840.1
	Water	22,649.1
	Slurry	36,333.0 gm
Liquid effluent		36,328.0 gm
Water vapor in effluent gas		54.0 gm
Catalyst - water		10.4 gm
Catalyst - $RuCl_3$ anhydride		2.7 gm
Oxygen input (at 21°C and 760 mmHg)		2,910.0 liters
Weight of O_2 input		3,835.0 gm
Vol. of effluent gas (at 21°C and 760 mmHg)		2,932.0 liters
Ash remaining on exit filter		0.5 gm
Heater power		255 watts

Comparison of influent raw slurry with effluent liquid:

	TKN mg/l	pH	Filterable Residue gm/l	Evaporated Residue gm/l	COD mg/l	TOC mg/l	% TOC Reduction
Raw Slurry	2177	8.9	6.15	-	13,553	4062	-
Effluent Liquid	17.5	2.5	0	5.04	1,261	405	90.1

TOTAL MASS BALANCE

INFLUENT		EFFLUENT	
O_2 gas	3,835 gm	Ash	0.5 gm
Slurry	36,333	Liquid	36,328
Catalyst Solution:		Effluent gas	4,030
Water	10.4		
$RuCl_3$ anhydride	2.7		
Total	40,181.	Total	40,358

UNDISSOLVED OXYGEN USED

Pure O_2 gas in	3,835 gm
Pure O_2 gas out	3,390.1
Pure O_2 gas used	445 gm

ASH ON FILTER

% by weight: C = 7.92, H = 1.32, N = 1.7

Less than 0.1% each of Al, Fe, Si, Ti, Cu, Mg.

Total amount of ash = 0.5 gm

ANALYSIS OF EFFLUENT LIQUID (mg/l)

SO_4	451.	Ca	12.5
SO_3	ND	Cr	2.34
S ion	0.27	Ni	8.57
TKN	17.5	Mn	.00725
N in NH_3	ND	Mo	7.95
N in NO_3	87.5	Na	866.
Cl ion	1679	K	926.
TC	514.	Fe	0.132
Acetate	775.	Ru	1.24
P in PO_4	71.2		

ANALYSIS OF ABOVE DATA FOR C, H, N, O, S, P

Substance	Mol. Wt.	gm. of Substance in 36.33 l	gm. of Element #1	gm. of Element #2	gm. of Element #3
SO_4	96	16.384	S = 5.4613	O = 10.92	
SO_3		N.D.			
S ion		.00981			
TKN		.6357			
N in NH_3		N.D.			
N in NO_3		-	N = 3.179		
O_3 in NO_3	62	-		O = 10.9	
TC		18.67			
Acetate $C_2H_3O_2$	59	28.154	C = 11.46	H = 1.436	O = 15.259
P in PO_4		-	P = 2.586		
O_4 in PO_4	95			O = 5.34	
Organic S		N.D.			

ELEMENT TOTALS FROM ABOVE

C	18.67 gm
H	1.436
N	3.815
O	42.417
S	5.47
P	2.586

EFFLUENT GAS

There were 2,932 liters of effluent gas at 21°C and 760 mm Hg. Methods and results of the analyses were:

Gas	mg/m^3	Total Mass	Analytical Method
O_2	115.6 (10)^4	3,389.4 gm	Gas Chromatograph (GC)
N_2	1.81 (10)^4	53.07 gm	GC
N_2O	3.06 (10)^4	89.9 gm	Infra Red
NO_2	0.896	2.63 mg	Chemo-luminescence
NO	0.0501	.1469 mg	Chemo-luminescence
NH_3	0.302	.8855 mg	Hypochlorite/phenol (colorimeter)
CO_2	15.1 (10)^4	442.73 gm	GC
CO	355.	1040.9 mg	GC
CH_4	882.	2586 mg	GC
SO_2	0.022	.0645 mg	Para Rosaniline (colorimetric)
H_2S	Insignif.	-	Iodiometric
Total hydrocarbons	765*		G.C.
H_2O		54 gm	Saturation assumed
TOTAL		4023 gm	

*Note that CH_4 alone is greater than Total Hydrocarbons. This discrepancy is attributed to the interference of N_2O in the GC.

Table VI is a tabulation of total influents and total effluents for the elements which are of greatest interest.

Table VI BALANCE OF SELECTED ELEMENTS

Element	INFLUENTS (grams)				EFFLUENTS (grams)					
	In H_2O	Gas	Slurry (July 27 & 28 Avg.)	Total	In H_2O (Liquid and Vapor)	Gas	Liquid		Ash	Total
							By Elemental Analysis	By Compound Analysis		
C	-	-	158.05	158.05	-	123.16	18.7***	(18.67)	0.04	141.9
H	3,975.9	-	29.8*	4,005.7	4,019.8	0.65*	1.21*	(1.44)*	0.007	4,021.7
N	-	-	79****	79	-	110.4	5.55	(3.81)	0.008	115.9
O	31,807.6	3,835	140.2*	35,782.8	32,158.7	3,745.7**	29.8*	(42.2)*	-	35,934.1
S	-	-	0.363	0.36	-	Insig.	2.99	(5.47)	Indeterm.	2.99
P	-	-	0.0945	0.09	-	-	0.178	(2.59)	"	0.18
Ru	-	-	1.312	1.31	-	-	0.045	-	Very Tiny	0.05
Total Water	35,783.5				36,178.5					

*Not including H and O in water

**3390.1 gm of pure O_2 and 355.6 gm in gaseous compounds

***By Total Carbon Analysis - Dohrmann Envirotech

****By Total Kjeldahl Nitrogen analysis of Oct. 4 sample

NITROUS OXIDE IN EFFLUENT GAS

Since N_2O was discovered in the effluent gas (during the material balance test) at a concentration of 25.3 grams per cubic meter, a means of suppressing its formation was needed. $RuCl_3$ is the catalyst which has been used to reduce TOC and to suppress ammonia. In order to find a process which would produce neither ammonia or N_2O, a series of tests were run in one of our batch test reactors because this was much more rapid and efficient than using the continuous flow reactor. These tests were run at 243°C and 6210 kPa unless otherwise indicated. The first two tests were run with $RuCl_3$ catalyst. The first test was performed on urea; the second on ammonia. The first test resulted in 14.4 gm/m^3 of N_2O; the second produced 12.6. This suggested that a $RuCl_3$ catalyst alone would result in too high an oxidation state of nitrogen. Subsequent $RuCl_3$ runs (7 and 8) performed on sewage also produced excessive quantities of N_2O. (Test #7 was run at initial conditions of 149°C and 2413 kPa for 2 hours, then at 243°C and 6210 kPa).

The above tests verified the magnitude of the N_2O problem. As a solution, it was decided to try a two stage approach. The first stage would employ copper sulfate as a catalyst at a reactor temperature of 243°C, while the second stage would use $RuCl_3$ at a lower temperature. Results are given in Table VII. We can make the following observations:

- Nitrous oxide was completely suppressed, except for the end of run #3. That may have been an erroneous value, but in any case the second stage need not have a residence time as long as 90 minutes (note that there was no N_2O at 30 minutes).
- Concentrations of NO_2, NO and NH_3 were all below MAC.
- TOC removal exceeded 91%.
- TKN removal exceeded 93% (runs 5 and 9).

The next step planned was to demonstrate this two stage process in the continuous flow system. To do this, an Inconel reactor (left from an earlier contract) was plumbed in between the first pass of the heat exchanger and the Hastelloy reactor. Unfortunately the Inconel reactor had no internal

Table VII BATCH REACTOR DATA FOR N_2O SUPPRESSION

Test No.	Stage	Time (min)	Temp (^oF)	Press (psi)	pH	(mg/l)		(mg/m^3)				
						TKN	TOC	N_2O	NO_2	NO	$N_2\%$	NH_3
3	1	0	470	900	7.1	62	195					
	1	30			5.5		19					
	1	90			2.9		15					
	2	30	300	450				N.D.	0.06	.012	2.12	0.42
	2	90			2.6	14	15	144	0.053	.011	1.14	0.68
4	1	0	470	900	8.0	103	325					
	1	30			7.2		37					
	1	90			2.9		26					
	2	30	250	400		21		N.D.	0.10	.018	1.7	N.D.
	2	90			2.7	14	28	N.D.	0.09	.018	1.1	N.D.
5	1	0	470	900	8.0	103	325					
	1	30			6.5		25					
	1	90			2.9		23					
	2	30	350	450	2.7	7	25	N.D.	0.066	.016	2.16	N.D.
	2	90			2.7		23	N.D.	0.062	.013	1.1	N.D.
9	1	0	470	900	8.9	479	927					
	1	15					78					
	1	30			2.0	14	83					
	1	60					77					
	2	30	300	450	1.9	28	81	N.D.	0.34	.15	3.8	0.3
	2	60					83	N.D.	0.30	.10	4.0	0.3

NOTE - Catalyst for Stage 1: $CuSO_4$

Stage 2: $RuCl_3$

baffles for plugged-flow, and in addition had a stagnant appendage (for a magnetically coupled stirrer) at the inlet end. Figure 15 schematically shows the components which were added to the system. All of these were carefully flushed out with water for long periods of time, in both directions. The $CuSO_4$ pump was disassembled and cleaned. The Inconel reactor had dual electrical heaters which were controlled by a thermocouple, controller and relay. After leaving the Inconel reactor, the fluid passed through a cooling coil, and then a high pressure Milton Roy metering pump was used to inject $RuCl_3$ catalyst before entering the Hastelloy reactor.

The added Inconel reactor had a volume of 1285 ml, plus 155 ml of coupler volume (which was a stagnant appendage). Liquid slurry occupied half of the volume, and gas the other half. At a cold liquid flowrate of 470 gm/hr (hot 578 gm/hr) liquid residence time in this reactor was 1.11 hours. The Hastelloy reactor, having a liquid volume of 875 ml, had a liquid residence time of 1.51 hours. Operating the reactors at 6550 kPa with the Inconel reactor at $243^\circ C$ and the Hastelloy reactor at $149^\circ C$, the 3650 TOC of the raw slurry was reduced to an effluent TOC of 600, but there was 2% of N_2O present in the effluent gas. It was believed that this N_2O was formed due to residual $RuCl_3$ in the first pass of the heat exchanger. Accordingly, the system was replumbed to eliminate the first pass through the heat exchanger (even though it had previously been flushed out with 10% nitric acid for 30 minutes, there may still have been residual Ruthenium which might have oxidized NH_3 to N_2O at the elevated temperature of the heat exchanger). Consequently, it was decided to bypass the incoming heat exchanger pass. The system was run again. This time raw slurry TOC = 3750, interstage TOC = 895 and effluent liquid TOC = 865. But unfortunately the effluent gas still had 1½ to 2% N_2O. It is believed that because $RuCl_3$ had been put into the slurry hold tank during the proceding material balance test, there was enough Ruthenium still in this tank and plumbing (despite subsequent water flushing) to have an active effect (i.e. - to promote N_2O formation in the Inconel reactor). Therefore, no further testing was done and the system was returned to its original single reactor configuration.

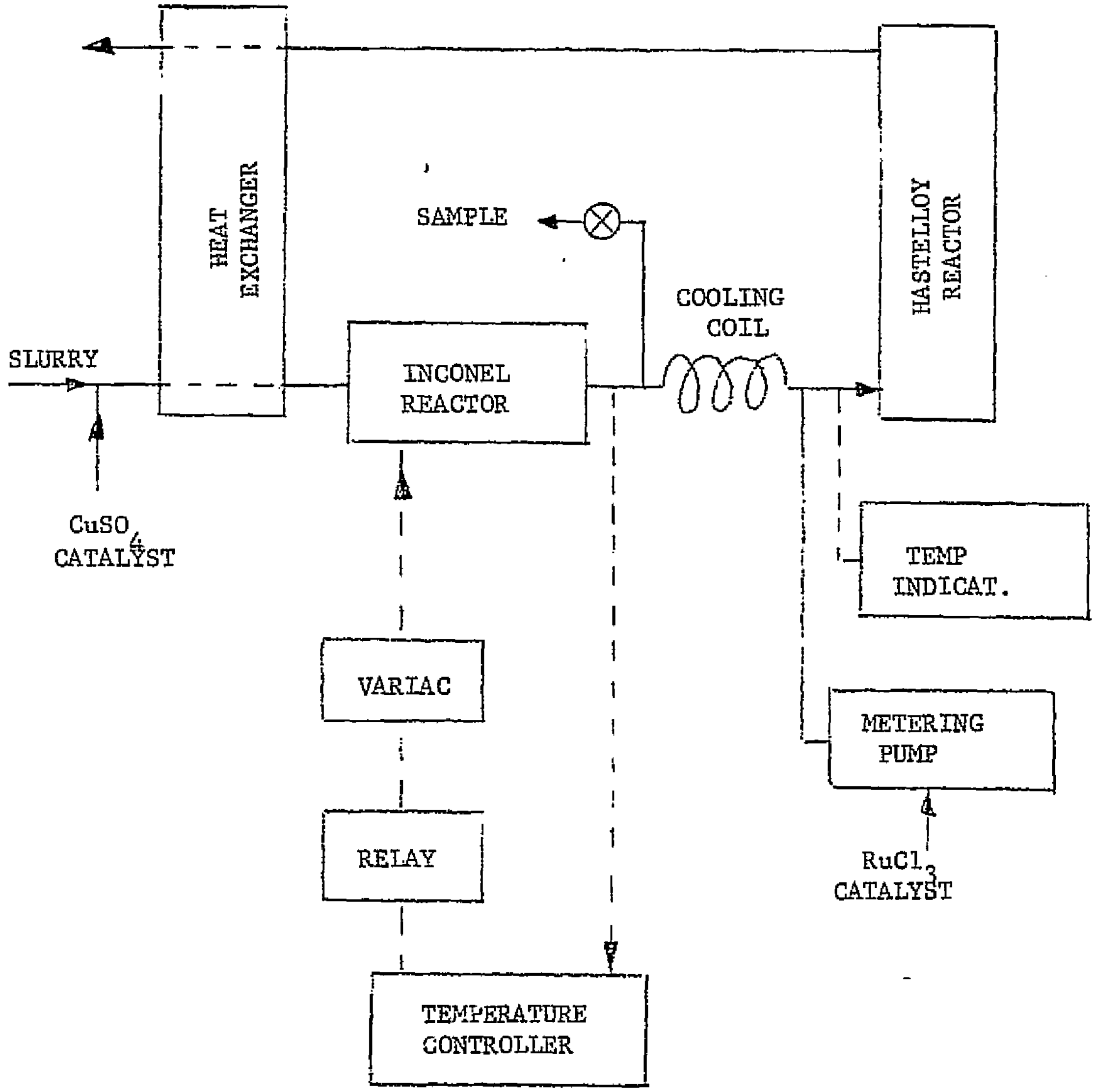


Figure 15 ADDITION OF FIRST STAGE REACTOR & AUXILIARIES TO WET OXIDATION SYSTEM

PROCESSING OF TRASH MATERIALS

Since the modified Urschel shredder which was used in the previous contract was unsuccessful, a new method was sought. A power disc sander was modified to evaluate dry abrasion as a technique for grinding up dry trash. A hollow tube was mounted perpendicular to the abrasive disc to serve as a feed tube. This tube was fitted with a ramrod. A vacuum cleaner was used to collect the ground-up trash. Various degrees of success were achieved with the dry trash constituents:

- Cloth required heavy feed pressure, and caused abrasive particles to become unglued from the disc. Considerable heat was generated, and a fire hazard existed.
- Aluminized Mylar melted and loaded the abrasive disc. Globs of Mylar formed. Required feed pressure was low.
- Polyurethane required low feed pressure, became a powder, and did not damage the abrasive disc.
- Towelettes were difficult because they were damp. High feed pressure was required.
- Teflon melted, then congealed into lumps.
- Q-Tips (wooden) - quite satisfactory.
- Dry paper towels - reduced easily, but appeared to present a fire hazard.
- Polystyrene spoons - handled easily, but instead of abrading, melted and formed rice-kernel sized particles.

Dry abrasion was not deemed sufficiently promising to warrant further investigation.

A commercial garbage shredder (restaurant grade of sink drain disposal - Waste King Model EHD) was used to do the shredding of the trash materials. Initial tests of dry trash constituents showed that cloth was the most difficult of all the materials to shred. It was necessary to precut cloth into pieces not larger than 15 cm square to prevent overloading of the cutter assembly, or failure to enter into the perimeter of the cutter assembly. Large pieces of waste material tend to revolve with the rotor and resist being flung outward by centrifugal action. A laboratory test loop was assembled, with the Waste King shredder, a vertical hold tank having a 22° half angle exit cone at its bottom, and a Vanton pump connected in series. When cloth and aluminized Mylar sheet were shredded simultaneously, the Vanton pump stopped pumping after four minutes. Its inlet was clogged by the slurry. Apparently the clogged particles of Mylar provided nuclei around which the cloth fibers could collect thus forming a conglomerate which bridged the 3/4 NPT pump inlet.

The following waste model was formulated as being typical of the trash mixture aboard manned spacecraft.

TYPE OF WASTE	grams per day	
Urine	8,652	
Feces	498	
Flush Water	10,102	
Subtotal		19,252
Moist Food (dog food)	1,042	
Aluminized Mylar film	272	
Polyethyelen film	1,132	
Polystyrene spoons	498	
Paper towels	724	
Towelettes	226	
Gauze	36	
Q-Tips (wooden)	9	
Mylar	45	
Teflon film	45	
Subtotal		3,009
Disinfectant		45
Wash water		3,850
Total		26,156

Table VIII Trash Particle Size - Waste King Disposal Unit

Material	Cycle	Average (mm)	Largest (mm)	Smallest (mm)
Mylar film (aluminized)	1	8.9 x 8.9	15.2 x 12.7	.51
	2	3.8 x 3.8	6.3 x 3.8	<.51
	3	3.8 x 5.1	7.6 x 3	↓
	4	2.5 x 3.8	7.6 x 7.6	
	5	3 x 3	10.2 x 3.8	
	10	5.1 x 1.5	10.2 x 7.6	
	20	3 x 3	10.2 x 10.2	
Polyethylene film	1	5.1 x 3.8	6.3 x 8.9	.25 x .76
	2	3.3 x 3.3	6.3 x 2.5	.51
	3	2 x 3.3	5.1 x 3.8	<.51
	4	2.8 x 2.5	5.1 x 2.5	↓
	5	2.8 x 1.8	4.5 x 3.8	
	10	2.3 x 1.5	3.3 x 2.5	
	20	1.8 x 1.5	3.3 x 1.8	
Polystyrene (spoons)	1	2.8 x 3	7.1 x 3.3	.25
	2	3.3 x 2.3	7.1 x 3.8	↓
	3	3.3 x 2	5.1 x 2.3	
	4	2.3 x 2	4.8 x 3.5	
	5	2.3 x 2	6.9 x 1.8	
	10	2.3 x 1.5	5.1 x 2.5	
	20	2 x 1.5	3 x 1.5	
Paper towels	1 20	It is virtually impossible to distinguish between 1st and 20th cycle samples. They all become fibers.		
Towelettes	1	5.1 x 5.1	16.5 x 5.6	<.51
	2	5.1 x 3.8	12.7 x 5.1	↓
	3	4.3 x 2.5	7.6 x 3.8	
	4	5.1 x 3.8	8.9 x 7.6	
	5	3 x 3	7.6 x 3.3	
	10	3 x 3.8	7.8 x 5.1	
	20	3.3 x 2.5	5.8 x 3	
Gauze	1	—	25. thread	< .25
	2		25. thread	↓
	3		None	
	20		None	
Q-Tip (wooden)	1	10.2 x 1.8	19 x 2.3	.51
	2	5.1 x 2.3	9 x 1.8	↓
	3	3.8 x 2	8 x 1.8	
	4	6.3 x 1.2	8.9 x 1.8	
	5	8 x 1.	6.3 x 2	
	10	5.1 x 1.5	4.3 x 2	
	20	2.9 x 1.5	5.8 x 1.3	

Table VIII Trash Particle Size - Waste King Disposal Unit (Continued)

Material	Cycle	Average (mm)	Largest (mm)	Smallest (mm)
Teflon sheet	1	5.6 x 3.6	18 x 7.6	.76 x .5
	2	3.8 x 3.8	10.2 x 2.8	1 x 2.5
	3	6.3 x 2.5	10.2 x 3.8	.51
	4	3.8 x 3.5	6.3 x 3	↓
	5	3.8 x 3.8	7.6 x 3.6	
	10	3.6 x 3.6	7.1 x 2.5	
	20	3.6 x 2.5	5.1 x 2.5	

A shredder fineness test was conducted. Each dry constituent of the above waste model was separately recycled through the shredder. A sample was extracted after each cycle and examined for particle size. The goal was to achieve 1.6 mm particles in as few cycles as possible. In examining the samples, the largest and smallest particles observed were measured, as well as what appeared to be most representative of the average particle. Table VIII lists the results. It can be seen that the goal of 1.6 mm maximum size was not always achieved even after 20 cycles. However this size goal had been selected arbitrarily. In some instances, particle sizes increased with successive cycles. This was due to selecting samples which were not representative, or to human error in measurement. In general, all materials were easily shredded. The mylar film tended to roll into a ball, and when unrolled gave the dimensions reported. Polyurethane film reduces very easily, and could almost be shredded without water. Polystyrene spoons formed hard, sharp particles which caused problems in the Vanton pump downstream. The hard polystyrene chunks, as well as wood from Q-Tips became entrapped and caused rupture of the elastomeric stator of the Vanton pump. Paper towel is not a shredder problem except that it absorbs the budgeted wash water. Towelettes are easy to shred, but the soap which they contain foams up which could cause difficulties during liquid/gas phase separation. Gauze is the most difficult material to shred. The problem is overcome by using a slow feed rate. Particle size could not be determined because pulling gauze apart caused formation of an endless strand. Teflon tends to break into small hard particles.

A shredder minimum water test was run. Several things became apparent:

- o The Waste King shredder works well, with little or no clogging.
- o The Vanton slurry pump clogs easily, and will not pump mixtures of high viscosity.
- o Particles which are ligher than water float in the reservoir, and since the exit is at the bottom, they do not recycle when gravity is present.

For the next series a screen filter was inserted between the Waste King shredder and the reservoir until all trash had been shredded twice. The screen filter was then removed and the second cycle trash was recirculated, adding water as necessary until the entire batch was processed and judged pumpable. Results are:

Run No.	Water Volume (liters)			Time (min)
	First Cycle	Final	Goal	
1	30.3	-	3.85	152
2	15.1	53	3.85	112
3	7.6	56.8	3.85	88

Run 1 was conducted without a filter screen and encountered several stoppages to unclog the circulating pump. Some of the trash recycled an unknown known of times while others cycled only once. Due to spillage and the decision to change the test setup to insert a screen filter, the water volume was never recorded.

Runs 2 and 3 were conducted using a filter screen for the first two cycles. Note that the water required for the first shredding cycle of run 3 is about twice the desired value. After removal of the screen, additional water had to be added to complete the recycling process of the entire batch. There is some consistency in the final amount of water consumed. This reflects the fact that the recirculating pump was the limiting component in the subsystem. Even with a more suitable pump, there seems little chance that the final goal of 3.85 liters can be achieved, because of water retention by the shredded materials. It is estimated that 14 grams of water would be needed per gram of dry trash, as an average. This is about 15 times as much as the trash model. An attempt was made to measure viscosity of the trash slurry, with the result being above $2(10)^6$ centipoise.

In order to use a water ratio which would not exceed the maximum projected amount available on a spacecraft, a slurry consisting of 5.5 times (by weight) as much water as mixed ground dry trash was prepared. This represents all of the available urine and flush water in addition to the wash water. All trash particles were screened so that none exceeded 0.8 mm. The slurry was placed in a tank having a smooth, gradual exit section consisting of a cone with a 22° half angle. Gas pressure was applied at the top of the tank and the discharge ball valve was opened. The result was that most of the free water ran out while nearly all of the trash remained in the tank. This disappointing result raised the question of whether smaller trash particles would result in a pumpable slurry.

Contacts were made with fluid mechanics experts in various educational institutions in an attempt to evaluate the slurry pumping situation. Flow of mixtures of solids and liquids (i.e., slurries) in conduits differs from flow of homogeneous liquids in several important ways. With liquids, the complete range of velocities is possible, and the nature of the flow (laminar, transitional, or turbulent) is defined by the physical properties of the fluid and system. With slurries, two additional distinct flow regimes and several more physical properties are superimposed on the liquid system. The two regimes of slurry flow involve:

- Homogeneous Slurries: Here, the solid particles are homogeneously distributed in the liquid media, and the slurries are characterized by high solids concentrations, and fine particle sizes. Such slurries often exhibit non-Newtonian rheology (i.e., the effective viscosity is not constant, but varies with the applied rate of shearing strain).
- Heterogeneous Slurries: Here, concentration gradients exist along the vertical axis of a horizontal conduit even at high flowrate, i.e., the fluid phase and the solid phase retain their separate identities. Heterogeneous slurries tend to be of lower solids concentration and have larger particle sizes than homogeneous slurries. In space, the vertical concentration gradients would disappear, but the problem of earth development remains.

Many slurries are of mixed character; the finer particle-size fractions join with the liquid media to form a homogeneous vehicle, while the coarser sizes act heterogeneously. Some authors have defined the boundaries between slurry types based on the top particle size (largest 5%) and specific gravity of the solids. For example: with 1.0 specific gravity solids, a heterogeneous slurry would be composed of particle 2.4 mm or larger. A homogeneous slurry would be composed of particles .56 mm or smaller. Slurries composed of particles between these bounds would be classified as compound. For higher solids specific gravities, the particle size boundaries are smaller.

A quantity of trash having the correct proportions of the dry trash model was prepared. The first step was dry shredding in the Waste King disposal unit at a rate of 1.3 kg/hr, consuming electricity at the rate of 220 watt hour per Kg to produce particles of approximately 3 mm. (This compares with 1987 watt hr. per Kg of dry trash during wet grinding.) The dry trash was then divided into 3 batches, each of which was processed through a Mikropul pulverizer under the following conditions:

Batch	(Mix by Weight)	Rate, gm Of Dry Trash Per Hour	Work,* Watt Hr. Per gm of Dry Trash
1	All Trash	453	1.647
2	Half Trash Half Water	566	1.318
3	16% Trash	906	0.8233

*These are estimates. A 1 hp motor was used on the pulverizer, but the actual energy consumption was not observed. The Mikropul operator did attempt to maximize the processing rate, but it was not reported if overload was the governing processing rate criteraion.

A screen classification of batch number 1 is as follows:

Screen Opening (mm)	Amt. Remaining on Screen (% by wt)
1.651	1.42
0.833	11.37
0.416	6.95
0.295	19.59
0.175	28.28
0	32.38

Classification was complicated by the phenomenon of cloth and paper fibers adhering to each other and forming a mat after passing through a screen. Lengthy manual agitation of the mat would break it up and allow fibers to descend to the next screen where a new mat would form. Thus, the fines tend to be composed more of the friable materials, and the coarses of fibrous materials.

Rough estimates of specific gravity, made by floatation of waste constituents in water, are as follows:

Constituent	Specific Gravity	Remarks
Mylar (Al)	$\simeq 1.0$	Almost Stable
Polyethylene	< 1.0	Slightly Bouyant
Polystyrene	> 1.0	Readily sinks
Q-Tips (wood)	< 1.0	
Gauze	> 1.0	Sinks when wet
Teflon	> 1.0	Heaviest of all
Paper	< 1.0	Will settle out when wet
Towelette	< 1.0	Unopened
Polyester	> 1.0	Readily sinks

The overall specific gravity was adjudged near 1.0. The denser materials are in the minority in the waste model and are in the fines of the pulverized particles, thus by the definition of slurry mentioned above, the pulverized material would form a compound slurry.

All the literature and experts agree that no matter what type of slurry is involved, the flow should be turbulent. While design of a system for operation below the transition critical velocity is acceptable for truly homogeneous slurries, no turbulent forces exist to suspend even trace amounts of heterogeneous particles. The system as currently configured operates at a Reynold's number of 2.2 x 10^{-5}, clearly in the laminar flow region.

Viscosity measurements were conducted on a carefully constructed batch of trash with all particles 1.7 to 2.4 mm. This test was performed prior to receipt of the Mikropul pulverizer output. The first step of the test was to determine the amount of water required to saturate the dry trash. This was done by thoroughly wetting the solids, then decanting after standing overnight. This mixture represents the volume of solids which enter into various slurry computations. Viscosities were measured using a Brookfield RTV rotating concentric cylinder viscosimeter. This unit operates by measuring the torsion exerted on a stirring rod immersed in the sample. Some doubts exist as to the validity of measurements achieved. The small diameter of the stirring rod turning in a fixed location could possibly create a "pool" of water in the mixture with resultant lower than actual readings. Varying the water content and adding an emulsifying agent (liquid hand soap) resulted in the following viscosity measurements.

Data Point	Sample	Volume Fraction (Vol. Solids/Total Vol.)	Emulsifier ($\mathrm{gm\ soap/cm^3}$)	Viscosity (centipoise)
1	1	1.0	0	∞
2	1	0.5	0	50,000
3	1	0.33	0	3,750
4	1	0.33	0.01	1,867
5	1	0.33	0.02	3,800
6	2	0.50	0	29,500
7	2	0.50	0.015	5,967
8	2	0.50	0.03	4,033
9	2	0.40	0.024	2,350
10	2	0.33	0.02	2,300

The trends are fairly obvious, although no explanation exists for data point 5 reversing the trend.

It is interesting to note that the decanted trash has a specific gravity of 0.84, a weight fraction of 0.18, and a volume fraction of 1.0. Using this data a relationship between volume fraction (ϕ) and weight fraction (WF) was derived.

$$\frac{\text{DRY TRASH}}{\text{LIQUID}} = \text{WF} = \frac{1}{\frac{6.6138}{\phi} - 1.0582}$$

Applying this to the waste model yields the following:

Mixture	WF	ϕ
Decanted trash	0.18	1.00
Trash model	0.51	2.19
Total waste model	0.17	0.95

The model weight and volume fractions are extremely dry and judging from the limited viscosity data taken would have prohibitively high viscosities.

In an attempt to determine what the present system can reasonably handle, a slurry sample was compounded using pulverized material from the Mikropul pulverizer. Water and emulsifier were added to produce $\emptyset = 0.524$ and 0.03 g soap/cm^3. This slurry was inserted into the slurry tank mounted directly above slurry pump intake valve #10 and, with 20 psig psig pressure, was injected into the pump. The pump then discharged through a simulated heat exchanger. About 2.5 cm from the end of stroke, the pump stopped due to compaction of solids within. All the liquid was pressed out. The discharge end of the pump cylinder has a 45^o half cone angle.

In order to determine whether individual trash components would behave in the same manner as the mixed trash slurry, a number of tests were run. The following materials were dry ground (once thru) the Waste King disposal unit:

Polyethylene sheet
Paper towel
Aluminized mylar film

Polystyrene spoons were also processed but it was necessary to add water and to put them thru the grinder twice. Each of these materials was separately mixed with an equal volume of water, and each slurry batch was placed in a tank having a 22^o half cone angle exit funnel. The top closure was clamped down, 30 psig was applied in the top of the tank, and its discharge valve was opened while the system injector piston pump was on its intake stroke. The result was the same in all cases; the trash remained in the converging outlet funnel of the tank while most of the water passed out of the tank.

Summary of Results of Trash Processing Investigation

- o Difficulties and problems which became apparent during abrasive grinding of trash caused this technique to be evaluated as unpromising, and consequently it was dropped.

- A Waste King commercial garbage disposal unit was found to be capable of shredding some trash model constitutents dry (paper towels, polyethylene sheet and aluminized Mylar film). However others require water for shredding.
- To reduce particle size still further, the shredded material can be pulverized in a commercial device such as that manufactured by the Mikropul Company. Again, some constituents can be pulverized while dry, but others require 6 to 15 times as much water as the amount specified in the total waste model. Energy consumption of the pulverizer was high:

		Watt Hr/kg
DRY (Some trash components cannot be dry ground)		
Waste King disposal unit for shredding		220
Mikropul pulverizer (if required)		1647
WET	Water gm/gm	
Waste King	15	1987
Mikropul (if required)	6	823

- The problems of transporting trash slurries were not solved. Turbulent flow is needed for effective transport, but our system's Reynolds number is about 10^6 below the lower limit. Low flow velocities cause solids to settle, packing of solids on any discontinuity, and squeezing of water through packed solids. Pulverizing the particle size to 0.178 mm did not help transport. All of the constituent materials produced transport problems.

There is a basic incompatibility between the low slurry viscosity and high flowrate which are needed for good transport, and the high viscosity and extremely low feedrate of this wet oxidation system. Because of this basic incompatibility and the corollary difficulties encountered in our laboratory tests, it was decided at meetings held at NASA-JSC in Houston on 4 November '75 and at LMSC in Sunnyvale on 19 November '75 that further efforts to process trash would be discontinued under this contract.

OXYGEN GENERATION

Consideration was given to the electrochemical generation of an oxygen source for the wet oxidation process. Four approaches were examined: electrolysis of the feed slurry, in-situ electrolysis in the reactor, electrolysis of the brine separated from the effluent with recycle to the feed stream and high pressure electrolysis of water. In the first three sources above a zero-gravity method of separating out the cathodically-produced hydrogen gas would be required. This would be the most difficult to implement in the case of in-situ electrolysis in the high-pressure reactor.

The electrolysis of feed slurry poses the potential problems of electrode fouling and buildup of solids on the hydrogen separator membrane. These effects would lead to increased IR losses and electrode overvoltages with resultant higher heat rejection and power requirements.

In-situ electrolysis in the reactor does not appear to be feasible, not only because of the impracticality of separating the hydrogen, but also because of the electrode area requirements (Note: a six-man urine pretreatment system alone would require 160 in^2 of total electrode area.) Stray currents due to the imposed electric field in the presence of the metal reactor walls would also be difficult to avoid.

Of the first three sources the best candidate appears to be the electrolysis of the brine separated from the effluent with recycle. This approach has the advantages of higher electrolyte conductivity, absence of suspended solids, and low-pressure hydrogen separation. It suffers, however, in that it would result in a gradual buildup of chloride ion concentration that would pose a corrosion problem in the reactor.

In summary, and for the various reasons given above, it does not appear that electrochemical generation of oxygen from the liquid being processed in the wet oxidation system is worthy of further pursuit. Water electrolysis to obtain oxygen should require considerably less than 2 volts per cell to be practical;

however, urine electrolysis work done at LMSC indicates that more than 5 volts per cell is required in urine because of the much lower conductivity. This high cell voltage much more than offsets any saving in power that the low pressure oxygen source (NaOCl liquid) affords.

The last approach of electrolyzing water to provide oxygen appears to be the most practical overall. This contract was not tasked to analyze this approach, but another technology development contract was funded to investigate this problem. The original effort was done on NAS 9-13430, "Solid Polymer Electrolyte Water Electrolysis System Development" by General Electric and is reported in CR 147501. The specification for that study included only the high pressure end of the range of pressure being considered for the wet oxidation process. The results of that study are reported here.

HIGH PRESSURE OXYGEN GENERATION

System Analysis

Design analyses were made for the electrolytic generation of oxygen at up to 17.24 MN/m^2 (2500 psia) with the basic Solid Polymer Electrolyte (SPE) cell and module configuration. Requirements for the oxygen generator would be that it provide oxygen at a temperature between 283 and 343K (50-140°F) and at a constant rate of 2.95 kg/day (6.5 lb/day) into a reactor in which pressure normally varies between 14.5 and 15.9 MN/m^2 (2100-2300 psia). The water utilized by the oxygen generator would be supplied at ambient pressure. The high pressure oxygen output control valve would be part of the waste process system. Two separate electrical signal pulses of 5 VDC from the waste process system would be utilized to start and stop the oxygen generator.

Electrolysis cell operating performance at high pressure was derived from development testing of SPE electrolysis cells for oxygen generation at 20.79 MN/m^2 (3015 psia) and conforming to requirements for a Navy oxygen generating plant under Contract N00024-72-C-5557, Project Serial No. SF0433-104, Task 16670 under Naval Ship Systems Command, Department of the Navy. At a mean cell operating condition of 393K (120°F), 17.24 MN/m^2 (2500 psia) and at a current density of 242 mA/cm^2, it was determined that 12 cells of 214.2 cm^2 area would provide the required oxygen rate of 2.957 kg/day (6.52 lb/day). Process water circulation rate would be 26.1 kg/hr (57.5 lb hr) for an electrolysis module water inlet temperature of 311K (100°F) and a H_2/H_2O outlet temperature of 333K (140°F).

This was the operating temperature and the current density demonstrated during most of the life tests on submarine cells operating in the cathode water feed mode. Higher operating temperatures were not found advantageous since higher parasitic losses due to gas diffusion through SPE cells at high pressures offset the reduction in cell operating voltage. Predicted input power to the power conditioner (90% efficiency) and electrolysis module would be 1425 watts ~~whereas~~, heat rejection from both components would be 672 watts. Constant input current would be 50.9 amps at a supply voltage of 28 VDC.

The electrolysis module design for 17.24 MN/m^2 (2500 psia) would be similar in concept to the 2.86 MN/m^2 (415 psia) design of an advanced system under development for NASA. That is, the stack of 12 cells would be contained within a dome pressurized with nitrogen to reduce cell gasket differential pressures to about 690 kN/m^2 (100 psid).

Functional components are similar to those of the six-man advanced system except that differential rather than absolute back pressure regulators would be employed for high pressure control and means of system pressurization and depressurization. The process water loop operating conditions of lower temperature and higher pressure have allowed for elimination of the regenerative heat exchanger. This requires operation of the phase separator/pump at about 333K (140°F) and the deionizer at about 311K (100°F) which is considered feasible for both components.

A hand-loading base pressure regulator is used for manually raising and lowering system operating pressure. O_2 and H_2 differential back pressure regulators and the module dome pressure regulator are referenced to regulated base pressure. It is proposed that the O_2 generator system would be started at low pressure and be self-pressurized by electrolysis at increasing power levels as base pressure is adjusted upward to design operating conditions at the following regulated values:

N_2 Base Regulated Pressure: $P_{N_2\ \mathrm{BASE}} = 0\text{-}16.5\ \mathrm{MN/m^2}$ (0-2400 psia)
(Manually adjusted)

Hydrogen Regulated Back Pressure: $P_{H_2} = P_{N_2\ \mathrm{BASE}} + 345\ \mathrm{kN/m^2}$ (50 psid)

Oxygen Regulated Back Pressure: $P_{O_2} = P_{N_2\ \mathrm{BASE}} + 690\ \mathrm{kN/m^2}$ (100 psid)

N_2 Dome Regulated Pressure: $P_{N_2\ \mathrm{DOME}} = P_{N_2\ \mathrm{BASE}} + 1135\ \mathrm{kN/m^2}$ (150 psid)

Relief valves are provided for overpressure protection and would backup Failure Detection and Isolation instrumentation with Emergency Shutdown controls. Several manual valves are shown for shut-off and venting capability to completely depressurize the system.

A water temperature regulator controls process water delivered to the deionizer and to the H_2 side of the module at a temperature of 311K (100°F). Two-phase H_2/H_2O flow leaves the module at a temperature of 333K (140°F) and is delivered to the phase separator. Because of the high pressure, only a small amount of water vapor is discharged with the hydrogen at this temperature. Water discharged from the phase separator is cooled by the heat exchanger with some by-pass directly to the temperature regulator. A water accumulator, as in the advanced six-man WES, has the dual function of providing logic for make-up water addition and a water storage capacity for load changes. A piston-type make-up pump delivers feed water from ambient supply pressure to the accumulator at a pressure of about 16.9 $\mathrm{MN/m^2}$ (2450 psia). Coolant supplied to the system removes waste heat from the heat exchanger and power conditioner.

A trade-off study would be required to determine the most feasible means of meeting power, weight, start/stop requirements of a waste process system. Because of a high rate of O_2 and H_2 gas diffusion at high pressure or so-called "fuel celling", gas pressures would drop suddenly if load was removed from the electrolysis module. Either a standby current must be maintained such that a small net O_2 and H_2 gas production is discharged through the back pressure regulators, or as the pressures fall in the electrolysis module, nitrogen would be admitted through check valves to hold system pressure at regulated N_2 base pressure. For short, frequent down times of the waste process reaction, maintaining a standby load on the O_2 generator sounds feasible whereas for long, infrequent down times, the nitrogen back fill would conserve power with little addition weight for N_2 usage.

It was determined that a standby current of 16 amps would be required to overcome estimated iffusion losses at a module temperature and pressure of 316K 110°F) and 17.2 MN/m^2 (2500 psia). An input power of 386 watts and system heat rejection of 375 watts would be required to maintain this condition. Some O_2 valve control would be necessary to dump or by-pass the small amount of oxygen discharged from the O_2 generator.

Complete removal of current from the electrolysis module would result in nitrogen at 16.5 MN/m^2 (2400 psia) replacing essentially all oxygen and the equivalent stoichiometric amount of hydrogen which are gradually combined to form water by diffusion at the electrodes in the SPE cells. It was calculated that a total quantity of 4.54 kg (10 lb) of nitrogen would be used for 100 system shutdowns. This nitrogen would necessarily be purged overboard with the generated O_2 and H_2 for a sufficient period of time after restarting to eliminate this diluent from oxygen delivered to the waste process reactor. Assuming three complete system changes which include N_2 pressurization of the module dome, the total nitrogen requirement would be 7.5 kg (16.5 lb). The gas cylinder volume requirement for initial storage at 41.3 NM/m^2 (6000 psia) and use at 16.5 MN/m^2 (2400 psia) would be 26.4 liters (0.932 ft^3) equivalent to a sphere having an inside diameter of 36.9 cm (14.5 inch).

Component Design

Because cell design would be identical to and the stack assembly of 12 cells would closely approximate the advanced module configuration, only the enclosure plate and domed enclosure would require redesign for higher pressure. Conditions of maximum operating pressure of 17.9 MN/m^2 (2600 psia), a proof pressure (1.5 X) of 26.9 MN/m^2 (3900 psia), and a burst pressure (2.5 X) 44.8 MN/m^2 (6500 psia) were applied for module design stress analysis. The following modifications would apply to the module design. The bottom enclosure plate would consist of 7075S-T6 aluminum, 5.08 cm (2.00 inch) thick utilizing type 316 stainless steel inserts for fluid porting and corrosion resistance. The lower module operating temperature permits the use of this design approach and use of this high strength aluminum to reduce weight. The elliptical dome of the module is made of 2024-T6 aluminum with a wall thickness of 1.42 cm (0.56 inch) and having a flange thickness of 3.8 cm (1.50 inch). The diameter of the flange and enclosure plate have been increased from 33 to 37.4 cm to accomodate these larger dimensions and larger flange bolts. Twenty-four flange bolts 9/16-18 thread are specified by MS9738 (17-4 pH), precipitation hardened stainless steel. The estimated weight of this 12-cell 17.24 MN/m^2 (2500 psia) module design, is 41 kg (90.5 lb).

A water accumulator design which incorporates an external sensing rod is not feasible for much higher pressures because of imbalance forces. A modified design would include an internal stainless steel piston with encapsulated magnets and without a piston rod. Reed-type position switches might be contained in sealed tubes inserted in the end plates of accumulator. Water capacity is modest, 164 cc (10 $in.^3$), because of elimination of the volume of the regenerative heat exchanger in the water process loop and the high operating pressure. Cylinder size would be 7.62 cm (3.00 inch) O.D. x 20.6 cm (8.12 inch) long. Net additional weight over the accumulator of the advanced system is 1.4 kg (3.1 lb) to a total weight of 7.13 kg (15.74 lb).

The phase separator/pump would not change in concept, but would increase in size and weight to provide for a heavier housing to accomodate the higher internal pressure. Estimated weight would be 9.1 kg (20 lb.)

Design of a nitrogen bottle for minimum weight considered an ARDEFORM sphere made from cryogenically formed 301 stainless steel shell with external glass filament reinforcing. As fabricated, the steel inner shell is under compression and zpproaches a zero stress condition at the design operating pressure of 41.3 MN/m^2 (15,000 psia). The external fiber structure is of glass filament wound construction with resin cured at 422 K (300°F). Demonstrated tensile strength at room temperature is 2275 MN/m^2 (330,000 psi). Design configuration would be a stainless steel shell of 36.9 cm (14.5 inch) inside diameter and wall of 3.19 mm (0.125 inch) surrounded by a fiberglas shell with a wall thickness of 4.19 mm (0.165 inch). This configuration would weigh 19.5 kg (42.9 lb) and hold 12.5 kg (27.5 lb) of nitrogen having a standard volume of 13.4 std. liters (380 SCF).

Gas regulators for differential control at high pressure are commercially available as special equipment for the conditions cited. Contact was made with Tescom Corporation, Minneapolis, Minn., who provided information on the units listed in the following table.

Because of the higher process water flow rate and higher operating pressure than the advanced system, the deionizer would utilize a larger 1000 ml capacity sampling cylinder rated for 24.1 MN/m^2 (3500 psia) operation. Container size would be 8.9 cm (3.5 inch) outside diameter by 29.2 cm (11.5 inch) long, weighing 5.56 kg (12.25 lb).

Pressure rating of the dual heat transfer coil heat exchanger, P/N 3101-6, 4-8-6X, Parker Hannifin Corp., is 18.6 MN/m^2 (2700 psia). Similarly, most standard commercial tube fittings, hand valves, check valves, etc., have pressure ratings greater than 20.7 MN/m^2 (3000 psia) and would be suitable for service in this system.

Major components of a water electrolysis system which are weight sensitive to operating pressure are listed below. A comparison of component weight is provided between that of the advanced system at nominal 2.860 MN/m^2 (415 psia) and that of the high pressure O_2 generation system at nominal 17.2 MN/m^2 (2500 psia)

Regulator Name	Tescom P/N	Weight kg	Max Inlet Pressure	Outlet Pressure MN/m^2 (psia)	Max Reference Pressure	Positive Bias Differential kN/m^2 (psid)
N_2 Base Pressure Hand Loader	26-1024-34	2.5	41.4(6000)	.1- 17.2(15-2500)	-	-
N_2 Dome Pressure Reduced W1 Ref.	Similar to 26-1000	2.5	41.4(6000)	Max 17.6(2550)	16.5(2400)	103(150
O_2 Back Pressure Regulator W1 Ref.	Similar to 26-1700	2.3	17.2(2500)	15.8(2300)	16.5(2400)	69(100)
H_2 Back Pressure Regulator W1 Ref.	Similar to 26-1700	2.3	16.9(2450)	15.8(2300)	16.5(2400)	34.5(50)

Component Name	6.8 Kgm/day (15 lb/day) 2748 kN/m^2 (400 psig)	2.95 Kgm/day (6.5 lb/day) High Pressure O_2 Gen.
	Weight kg (lb)	
Electrolysis Module	32.9 (72.5)	41.1 (90.5)
Water Accumulator	5.7 (12.7)	7.1 (15.7)
Phase Separator	7.2 (15.8)	9.1 (20.0)
Deionizer	1.5 (3.3)	5.6 (12.3)
N_2 Gas Bottle (Empty)	Not Included	19.5 (42.9)
N_2 Gas Bottle (Full)	-	32.0 (70.4)
N_2 Base Pressure Regulator	2.5 (5.5)	2.5 (5.5)
N_2 Dome Pressure Regulator	2.5 (5.5)	2.5 (5.5)
O_2 Back Pressure Regulator	0.7 (1.5)*	2.3 (5.0)
H_2 Back Pressure Regulator	0.7 (1.5)*	2.3 (5.0)

*Absolute Back Pressure Regulators designed to specification.

It should be noted that the component weights listed, excepting the N_2 gas bottle, are for commercial off-the-shelf hardware or for preprototype designs without a critical regard for weight and would not be representative of flight-weight designs.

SAFETY STATEMENT

The NASA wet oxidation system has been reviewed for safety and has been found to be adequate. A Hazards Analysis was completed on the system.

The Hazards Analysis identified the reactor, heat exchanger, reactor bleed-down valve, and cooling coil as critical components (i.e., components, the failure of which, would constitute a serious hazard). Any failure of a relief valve, burst disc, or bleed-down valve to open could cause a critical failure only if it was accompanied by a second failure in which the system pressure was significantly increased.

Safety devices built into the system include three switches with total system shut-down capabilities. The reactor pressure switch and the reactor temperature switch sense out-of-tolerance conditions in the reactor and heat exchanger and shut down the system. The filter pressure switch will sense a high differential pressure across the two filters and shut down the system. These three switches act as back-ups to one another since a change in pressure/temperature in one subsystem will result in a change in the other.

In addition, there are bleed-down valves in the slurry, oxygen, and reactor subsystems, burst discs in the reactor and filter subsystems, and relief valves in the slurry, hydraulic, and catalyst subsystems to prevent excessive pressure build-up. The hydraulic subsystem has a pressure switch which will shut off the slurry feed. The loader, slurry, oxygen, and reactor subsystems can be isolated from the rest of the system by isolation valves. The system can be monitored by pressure gages provided on all subsystems, except the loader subsystem and thermocouples located in the slurry and reactor subsystems. The entire system is boxed in a partial enclosure. These multi-redundant automatic and manual safety devices sufficiently diminish hazards associated with a potential rupture occurring as a result of over pressurization.

PRODUCT ASSURANCE

Efforts in the Quality and Reliability disciplines continued from the previous contract (NAS 1-11748) and were concentrated on changes to the previous system.

In the quality area effort consisted of the review of purchase documentation for inclusion of appropriate quality requirements, receiving inspection for part identification and damage and a monitoring by quality engineering of the assembly/test progress.

Elimination of the trash processor module resulted in a reduced effort in the areas of safety and reliability. The component changes and improvements (single hydraulic piston/cylinder in lieu of two bladders, improved bearing location/design, heat exchanger re-design, elimination of unnecessary filters, improved regulator, etc.) described elsewhere in this report were judged to be improvements in the overall system reliability and safety.

CONCLUSIONS

These conclusions are based on analytical studies and laboratory testing performed under this contract.

- The wet oxidation system is capable of automatically processing slurry of human wastes and water, with safety and reliability. However post treatment of the liquid effluent is needed to remove color, odor, or further reduce TOC, depending on whether the reactor was operated at relatively high or low temperatures and pressures.
- Reactor pressure can be reduced below 6,895 kPa (1000 psi) to enable use of existing oxygen sources aboard spacecraft. Size and weight can accordingly be reduced.
- Reactor stirring is unnecessary. Stirring motor, magnetic coupler, stirrer, internal bearings and bearing cooling apparatus can be eliminated. Influent and effluent lines could be located on the reactor's longitudinal centerline (one at each end) instead of entering radially. This has fabricating and strength advantages, as well as permitting a large diameter entry tube.
- A material balance test quantitatively defined the influents and effluents of the wet oxidation system while processing sewage. An undesirable effluent gas was nitrous oxide.
- In a series of batch reactor tests, it was found that nitrous oxide could be suppressed by using a two stage reaction. The first stage employs copper sulfate as a catalyst and the second employs ruthenium trichloride. However when a second reactor was added to the continuous flow wet oxidation system to use this two stage scheme, N_2O could not be suppressed. This was probably due to the presence of residual $RuCl_3$ in the slurry hold tank and plumbing (despite water flushing) which promoted N_2O formation in the first stage reactor.
- Analytical studies and laboratory tests were performed in an attempt to process a typical spacecraft trash model. Shredding of most materials was reasonably successful, but consumed large amounts of electrical energy. We were not able to pump slurries of water and small trash particles thru conduits.
- A study was made of several alternative schemes to generate oxygen by electrolysis from the liquid being processed. None of the alternatives was deemed sufficiently promising to warrant further work.

RECOMMENDATIONS FOR ADDITIONAL AREAS OF INVESTIGATION

Safe, automatic and reliable operation of the wet oxidation system has been proven using sewage. Operation of the system with reduced temperature, pressure and oxygen feedrate has been demonstrated. As a consequence of lower operating temperature and pressure, lighter weight components could be built, especially a lighter reactor. Since no stirring is required in the reactor, all stirring components could be deleted and the inlet and outlet ports of a new reactor could be located axially in-line. An axial reactor influent line would also be highly desirable to admit a thick slurry containing trash.

Although we were able to shred dry trash into small particles, we were unable to pump the resulting slurry. A development program to solve this pumping problem would involve a considerable effort. But a much more formidible task is finding a way to inject the trash into a high pressure reactor (or heat exchanger). Some thought has been devoted to these problems, and several different approaches are possible.

If the trash is to be transported as a water slurry, high flow velocities and adequate liquid are essential. All of our testing lacked high flow velocities. In a continuous wet oxidation system, injecting the slurry into the high pressure heat exchanger and reactor must necessarily be done at a slow rate. The first two of the following systems attempt to reconcile these inconsistent requirements.

See Figure . Concept A has 2 loops. The left hand prepares a slurry using wet shredding. The slurry circulates in this loop at a turbulent velocity until the appropriate valves are actuated to send the slurry into the right hand loop. The right hand loop is also equipped with a pump to keep the slurry flowing turbulently. When the reservoir in this loop has filled, the bypass tube is closed off and the slurry is routed into the injection pump via the hollow stem of the piston.

Concept B (see Fig. 17) has an identical left hand loop to that of Concept A. The right hand loop is similar too except that instead of being introduced directly into the injection pump, the high speed stream of slurry enters a pre-injection cylinder thru an entry port. Some of the water passes out of this cylinder thru a screen at the exit port. When the piston moves, it first covers the inlet port, after which water is squeezed out of the slurry and runs out of the exit port. The remaining trash solids and a minimal amount of water are then forced into the injection pump cylinder.

Concept C (see Fig. 18) embodies a completely different approach in which the ground trash remains dry. The ground dry trash is compressed in a loading cylinder which is equipped with a valve to vent most of the entrapped air. A large diameter ball valve is then opened and the loading piston forces the trash into the injection cylinder. After the loading valve is closed, the injection piston begins its stroke and a vent valve is opened to permit most of the remaining trapped air to escape. This vent valve is then closed, the large diameter ball valve leading to the heat exchanger is opened, and the injection piston completes its stroke. It is essential that the ball valve, the heat exchanger and the reactor have internal diameter at least as large as the injection cylinder to avoid clogging.

Concept D (Fig. 19) requires 2 reactors since it is a batch process rather than continuous flow. This permits the trash to be introduced into each reactor at ambient pressure, with very low injection pressure. Large diameter ball valves are used to admit the slurry, so there is no flow impediment. These same valves are used to discharge the effluent. For zero g operation, a piston is needed in each reactor to discharge the effluent. The piston is actuated by gas pressure from the left end. After the first reactor has been emptied of effluent, the left end is vented so that a new charge of slurry

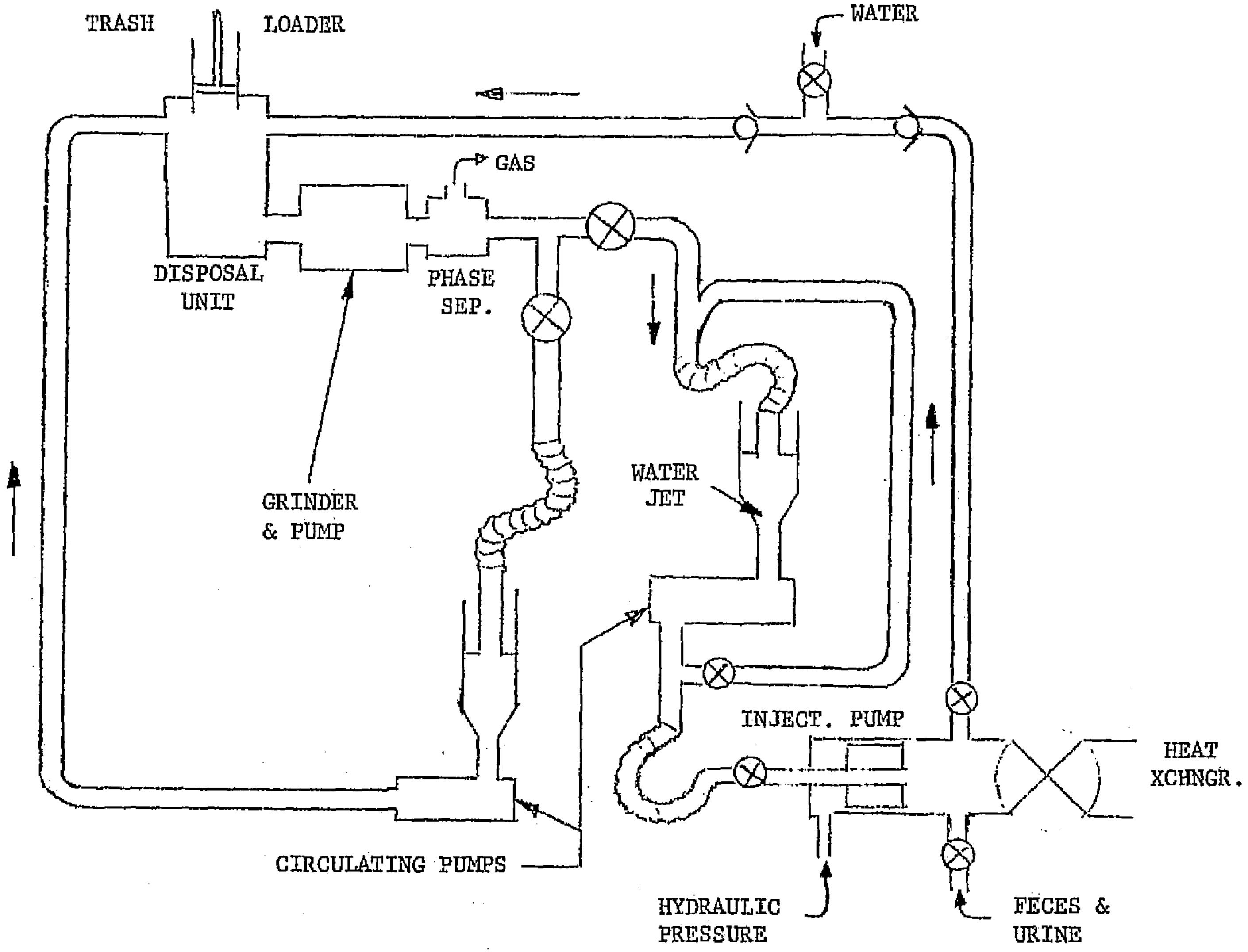


Fig. 16 TRASH LOADING CONCEPT A

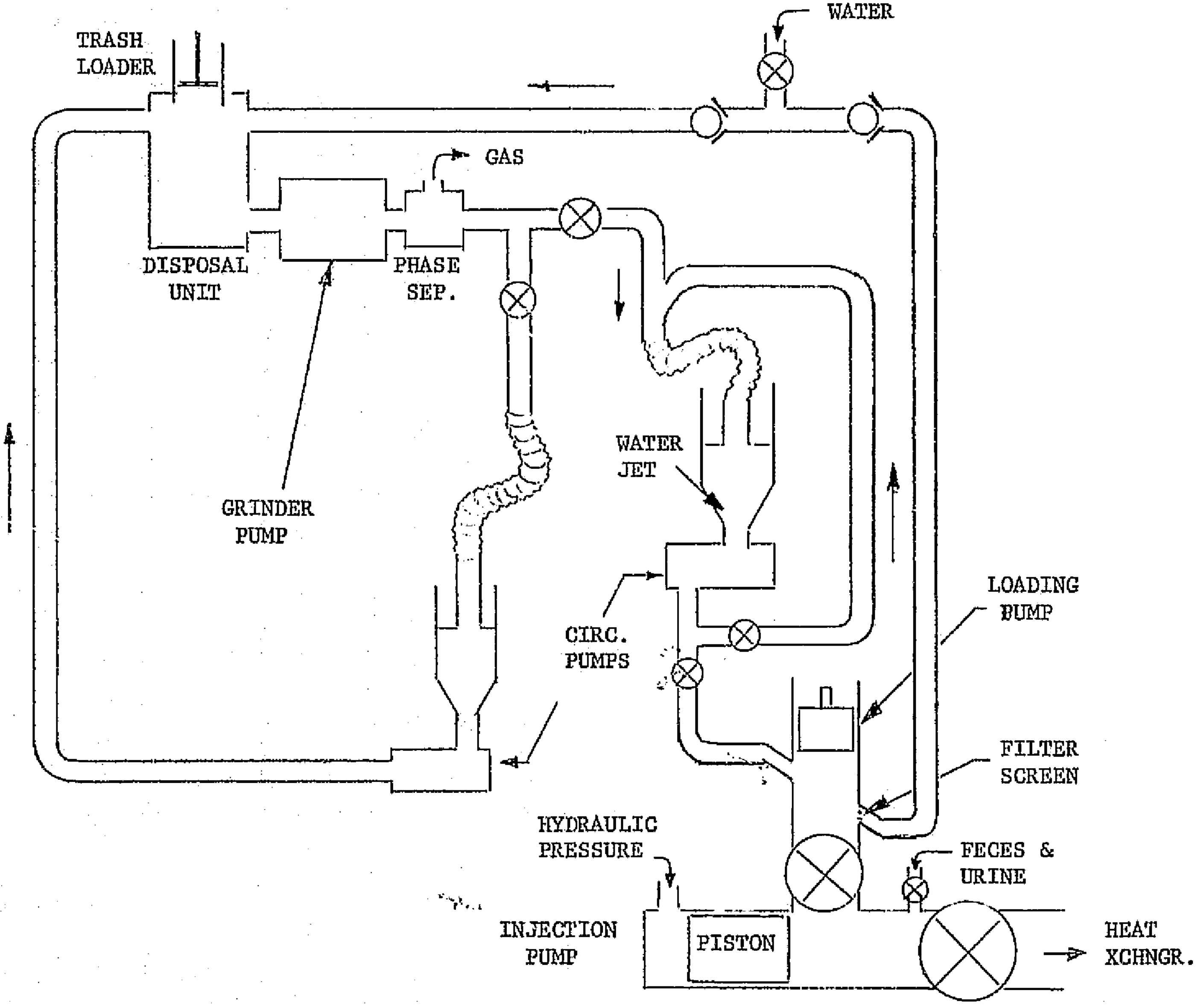


Fig. 17 TRASH LOADING CONCEPT B

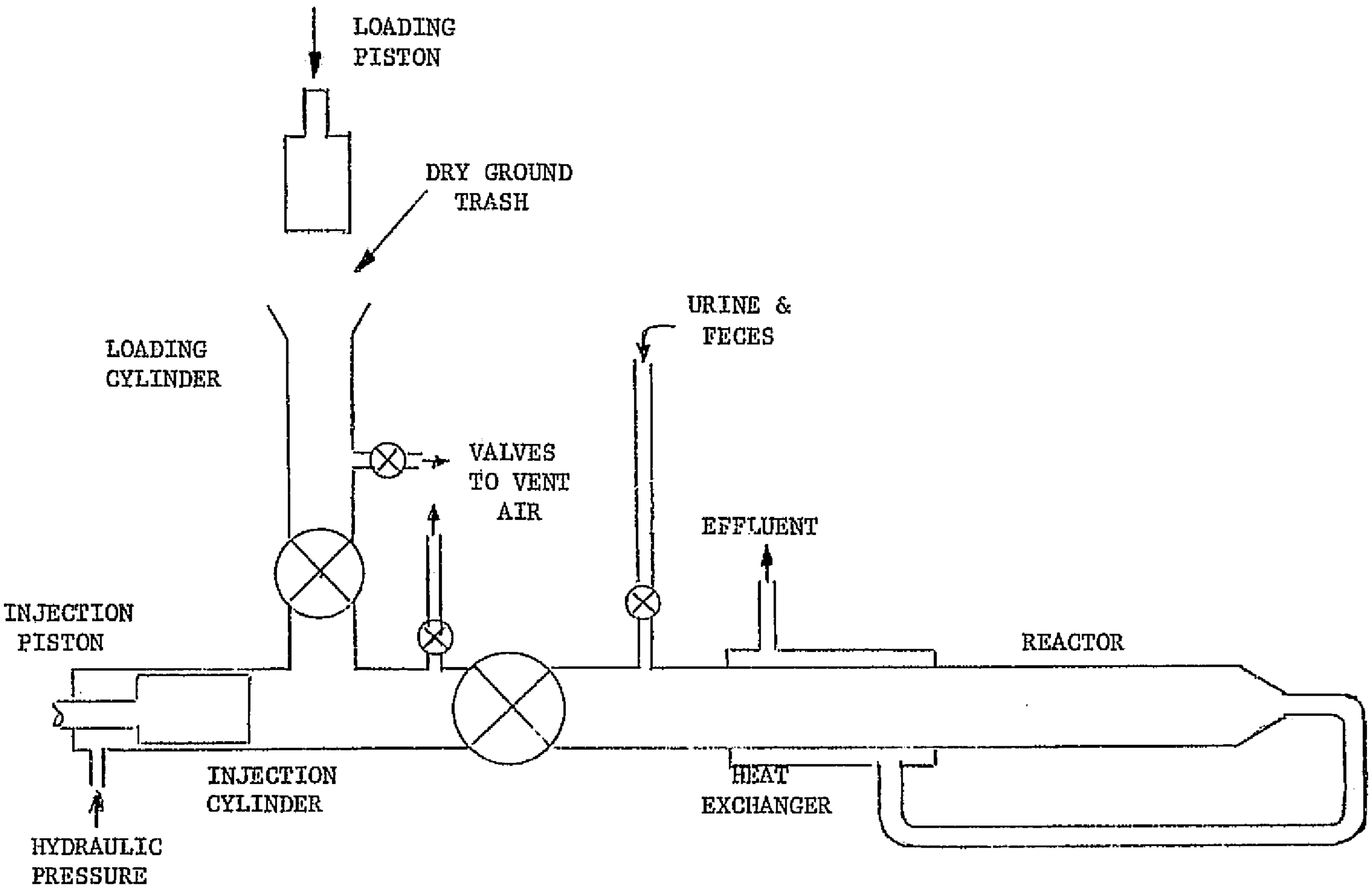


Fig. 18 Trash Loading Concept C

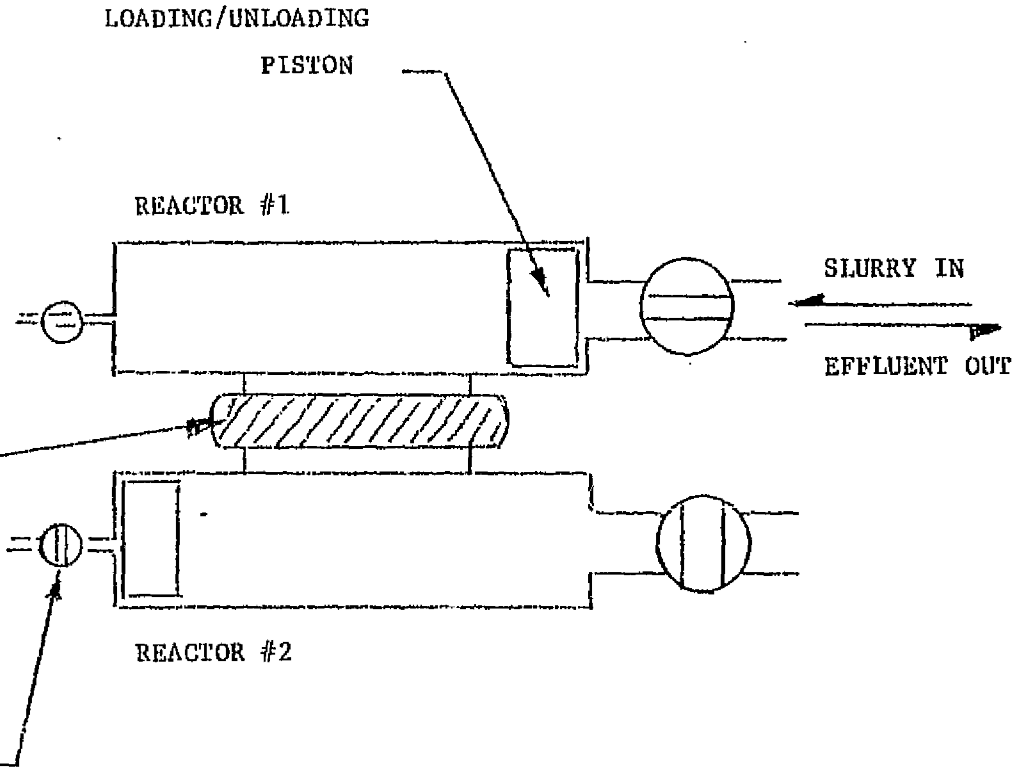


Figure 19 TRASH LOADING CONCEPT D

can be injected from the right end. Then, to prevent the piston from leaving the left end of the reactor during wet oxidation it is magnetically latched into position. To minimize the energy used in reheating each reactor during each cycle, a sliding thermal conductor is used to transfer heat from one reactor to another. When both reactors have reached approximately the same temperature, the slider is removed and the reactor containing the new batch of raw slurry is electrically heated.

In addition to eliminating the problem of high pressure slurry injection, this concept has the advantage of reducing the oxygen storage pressure requirement.

All of these trash loading schemes share the following shortcomings:

- Weight of the equipment
- Electrical energy consumed in grinding and feeding the trash
- Reasonably complex systems with implied penalty in reliability level
- New, untried methods which would require a sizable development program
- Performance in zero g cannot be predicted with assurance.

In addition, Concepts A and B require large amounts of water (although this water could be recycled).

REFERENCES

1. "Design, Fabrication and Testing of a Wet Oxidation Waste Processing System" - Final Report for Contract NAS 1-11748 by LMSC Bioengineering for NASA Johnson Space Center dated May 15, 1975.

2. Report No. NASA CR-112151, "Design and Development of a Prototype Wet Oxidation System for the Reclamation of Water and the Disposition of Waste Residues Onboard Space Vehicles", Final Report for Contract NAS 1-9183 by LMSC Biotechnology for NASA Langley Research Center dated May 26, 1972.

ABSTRACT

This report describes further development and testing of the automatic wet oxidation system which was built under a previous contract, NAS 1-11748. This system is a laboratory pre prototype of an apparatus suitable for use aboard manned spacecraft to convert sewage into sterile liquid, gas and a tiny residue of ash. Additional processing is required to make the liquid effluent suitable for drinking and wash water. The problems of processing trash materials were not solved. Effects of varying the operating conditions were investigated, including reactor pressure and temperature, sewage feedrate and oxygen feedrate. Reactor stirring was found to be unnecessary. The benefits of several effluent post-treatments were evaluated. A material balance test was performed during a 3 day run of the wet oxidation apparatus and quantitative analyses made of the influents and effluents.