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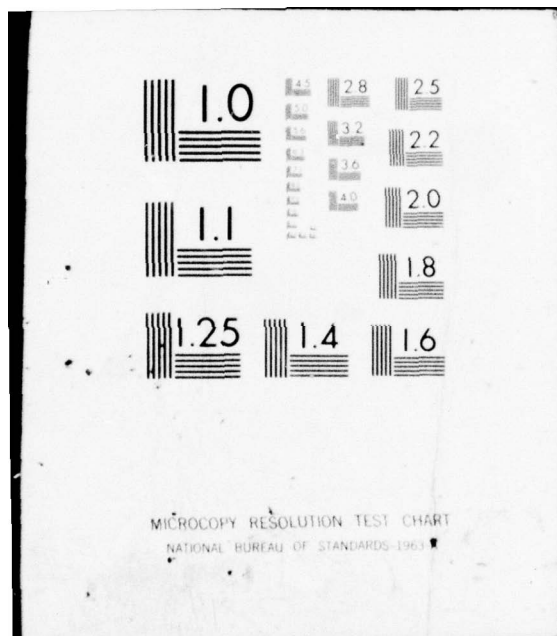
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# RESEARCH AND DEVELOPMENT OF AN OZONE CONTACTOR SYSTEM

ADA031943

## FINAL REPORT

by

G. G. See, P. Y. Yang  
and K. K. Kacholia

June, 1976

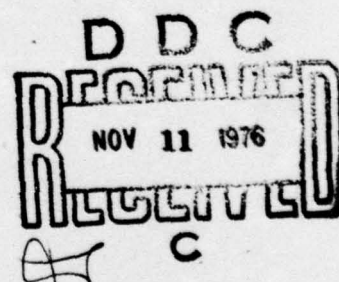
Prepared Under Contract No. DAMD17-76-C-6041

by

*Life Systems, Inc.*  
Cleveland, Ohio 44122

for

**U.S. Army Medical Research  
and Development Command**  
Washington, D.C. 20314



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N, N-diethyl-m-toluamide are some of the organics present in the waste waters. The effectiveness of the ozone oxidation as a function of various operating parameters, including temperatures, pH, ultraviolet light activation, carrier gas flow rate and ozone dosage are discussed. Comparisons are made between the ozone contactor experimental results and those obtained with other reactor types.

Post-treatment of laboratory and composite reverse osmosis permeates with the ozone contactor resulted in a final effluent with total organic carbon and chemical oxidation demand values below the required specifications of 5 mg/l and 10 mg/l, respectively. The residence time required was two hours for composite waste water and four hours for laboratory waste water. Comparing with other reactor types, the Life Systems's Ozone Contactor reduces ethanol to below 5 mg/l total organic carbon with 50% of the power required by the others.

The design and development of a minicomputer-based control and monitor instrumentation for the Ozone Oxidation Unit Process were successfully accomplished. The instrumentation is capable of controlling and monitoring the process parameters detecting component failures, sequencing actuators for mode transitions and reducing operator errors. An electronic Ozone Oxidation Simulator was developed to enable the instrumentation to be tested, debugged and checked out in parallel with the ozone contactor development and testing effort. The instrumentation development was concluded with an estimation of the size of the expected MUST WPE instrumentation in the pilot plant phase.

A Reverse Osmosis Unit Process was designed, fabricated, assembled and checked out. It successfully produced water which synthesizes the MUST waste water influent to the ozone oxidation unit process. A sodium chloride rejection rate of 98% or higher was achieved for a shakedown testing period of over 100 hours of continuous operation.

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Prepared Under Contract No. DAMD17-76-C-6041

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LIFE SYSTEMS, INC.  
Cleveland, Ohio 44122

for

U. S. ARMY MEDICAL RESEARCH AND DEVELOPMENT COMMAND

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FOREWORD

This study was conducted for the U. S. Army Medical Research and Development Command, Washington, DC, under Contract DAMD17-76-C-6041. The Program Manager was Gary G. See. Technical effort was completed by Dr. P. Y. Yang, K. K. Kacholia, T. S. Steenson, F. C. Jensen, G. A. Little, Dr. R. A. Wynveen, C. T. Burger, J. D. Powell and Dr. R. J. Davenport.

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ACRONYMS

COD	Chemical Oxygen Demand
EP	Equalization/Prescreening
FAC	Free Available Chlorine
HC	Hypochlorination
IE	Ion Exchange
LMTOC	Life Systems' Modified Torricelli Ozone Contactor
MUST	Medical Unit, Self-Contained, Transportable
O <sub>3</sub> /UV	Ultraviolet Light Activated Ozone Oxidation
RO	Reverse Osmosis
TOC	Total Organic Carbon
TSA	Test Support Accessories
UF	Ultrafiltration
WPE	Water Processing Element
WWMS	Water and Waste Management Subsystem

## SUMMARY

The development and preliminary characterization of the Life Systems' Modified Torricelli Ozone Contactor system as a post-Reverse Osmosis treatment process for the Water Processing Element in the Medical Unit, Self-Contained, Transportable complex were successfully accomplished. The ozone contactor employs six totally mixed contacting stages with a precontactor stage. The off gases from the six stages are sparged through the precontactor stage for efficient ozone conversion. About 96 to 100% ozone conversion resulted for ozone dosages between 2.8 and 7.9 mg/min/l (0.033 and 0.095 lb/day/gal) of wetted reactor volume.

This report describes the ozone contactor experimental hardware, methodology and results for the various Medical Unit, Self-Contained, Transportable waste waters. Methanol, ethanol, phenol, formaldehyde, acetic acid, acetone, urea, O-toluidine and N,N-diethyl-m-toluamide are some of the organics present in the waste waters. The effectiveness of ozone oxidation as a function of various operating parameters, including temperatures from 303 to 333K (86 to 140F), pH from 7 to 11, ultraviolet light activation, carrier gas flow rate, and ozone dosage are discussed. Comparisons are made between the ozone contactor experimental results and those obtained with other reactor types.

Post-treatment of laboratory and composite reverse osmosis permeates with the ozone contactor resulted in a final effluent with total organic carbon and chemical oxygen demand values below the required specifications of 5 mg/l and 10 mg/l, respectively. The composite waste reverse osmosis permeate was effectively treated in the contactor without elevated temperatures or elevated pH in less than two hours of residence time. The laboratory waste permeate was effectively treated in approximately four hours of residence time. In direct comparison with other reactor types, the Life Systems' Ozone Contactor reduces ethanol to below 5 mg/l total organic carbon with 50% of the power required by the others.

The design and development of a minicomputer-based control and monitor instrumentation for the Ozone Oxidation Unit Process were successfully accomplished. Major instrumentation functions were fabricated, implemented, assembled and checked out. The instrumentation is capable of controlling and monitoring the process parameters, detecting component failures, sequencing actuators for mode transitions and reducing operator errors. An electronic Ozone Oxidation Simulator was developed to enable the instrumentation to be tested, debugged and checked out in parallel with the ozone contactor development and testing effort. Computer control/monitor programs for advanced instrumentation were successfully developed and demonstrated on the Ozone Oxidation Unit Process Simulator.

A study of the size of the expected Medical Unit, Self-Contained, Transportable Water Processing Element instrumentation in the pilot plant phase was conducted. The study included the investigation and estimation of the pilot plant instrumentation cost, maintainability, reliability, volume, power and weight.



The fabrication, assembly, checkout and shakedown testing of a Reverse Osmosis Unit Process were successfully accomplished to produce water which simulates the Medical Unit, Self-Contained, Transportable waste water influent to the Ozone Oxidation Unit Process. Two DuPont B-10 modules were used in the Reverse Osmosis Unit Process. Provisions were made for expansion to include two more B-10 modules. With four B-10 modules, the Reverse Osmosis Unit Process will have a capability of producing at least 15 l/min (4 gpm) permeate from Medical Unit, Self-Contained, Transportable waste water. Over 100 hours of continuous shakedown testing were accumulated on the Unit Process without a shutdown. Permeate flows of 6.6 l/min (1.75 gpm) to 8.3 l/min (2.2 gpm) were achieved with a single B-10 module. A sodium chloride rejection rate of 98% or higher was achieved for the shakedown testing period.

#### INTRODUCTION

The U.S. Army has a requirement to provide a mobile mission-oriented medical treatment system which is designed and equipped to facilitate rapid establishment and disestablishment. This flexibility permits immediate response by medical support units to any tactical, environmental or geographical change. The system will provide a contamination-free and controlled environment in which medical, surgical and other supporting functions can be performed. The mobile medical treatment system is termed the MUST: Medical Unit, Self-Contained, Transportable.<sup>(1)</sup>

Associated with the MUST is a Water and Waste Management Subsystem (WWMS). This subsystem is required to treat and dispose of (without degradation of the environment or danger to personal health) all toxic and contaminated waste materials generated within the functional areas of the MUST medical complex. In addition to the waste treatment and disposal, the WWMS must be capable of producing potable water from a fresh or brackish water source, and reuse water from the MUST medical complex waste water effluents. Waste treatment and the production of reuse and potable water is achieved within the WWMS by a self-contained Water Processing Element (WPE).<sup>(1,2)</sup>

#### MUST WATER PROCESSING ELEMENT

The MUST WPE consists of six unit processes and an integrated control and monitor instrumentation system. The WPE block diagram is shown in Figure 1. The six unit processes within the WPE are: Equalization/Prescreening (EP), Ultrafiltration (UF), Ion Exchange (IE), Reverse Osmosis (RO), Ultraviolet light activated Ozone Oxidation (O<sub>3</sub>/UV) and Hypochlorination (HC).<sup>(3)</sup>

The function of the EP Unit Process is to settle and screen suspended solids and equalize hydraulic loading and concentration variations to result in a more uniform feed to the UF Unit Process.

The function of the UF Unit Process is to separate the suspended and dissolved solutes above a molecular weight of 500 to minimize plugging and fouling of the RO Unit Process downstream of the UF.

(1) References cited in parentheses are listed at the end of this report.

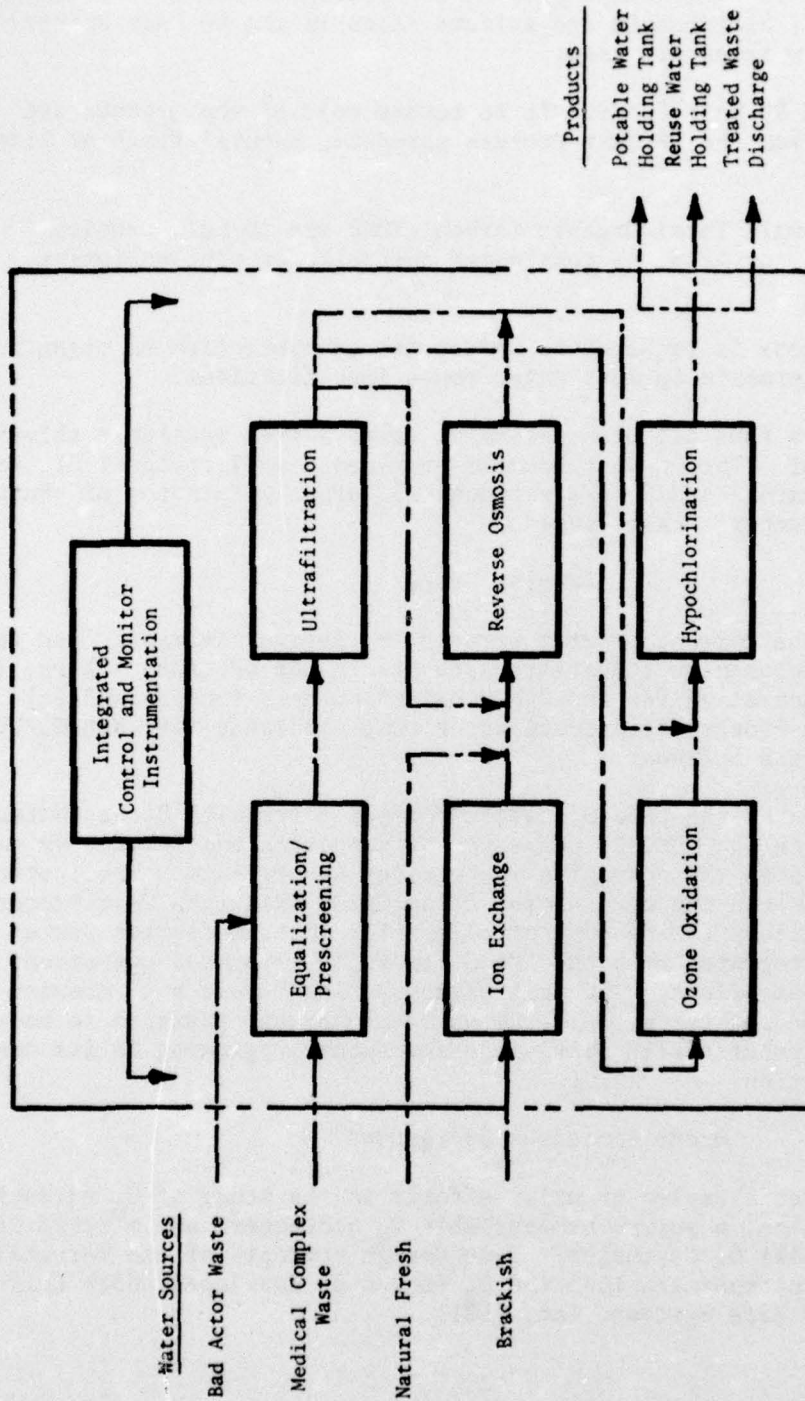


FIGURE 1 WATER PROCESSING ELEMENT BLOCK DIAGRAM



The IE pretreatment is required to prevent the precipitation of calcium and magnesium carbonate, bicarbonate and sulfate salts in the RO Unit Process when hard, brackish water feeds are used.

The function of the RO Unit Process is to remove most of the organic and inorganic solutes from the UF Unit Process permeate, natural fresh or brackish water feeds.

Water containing 5 mg/l Total Organic Carbon (TOC) and 10 mg/l Chemical Oxygen Demand (COD), or less, is considered suitable for nonconsumptive reuse. (2,4)

The O<sub>3</sub>/UV Unit Process is required to reduce the concentration of organic solutes in the RO permeate to meet water reuse specifications.

Army policy requires that all produced water carry a free available chlorine (Cl<sub>2</sub>) (FAC) residual. The HC Unit Process provides 5 mg/l residual Cl<sub>2</sub> for reuse and potable waters and 2 mg/l residual Cl<sub>2</sub> after 20 minutes of contact time for surface discharge waste waters.

#### Program Scope

The objectives of the current program were to (a) design, fabricate and test a breadboard O<sub>3</sub> contactor more compatible with use in the WPE than others, (b) develop the instrumentation for the O<sub>3</sub>/UV Oxidation Unit Process and (c) assemble an RO Unit Process to produce water that simulates MUST WPE O<sub>3</sub>/UV Oxidation Unit Process influent.

The characterization of the Life Systems' Modified Torricelli Ozone Contactor (LMTOC) was made with the MUST RO permeates of composite and laboratory waste waters with emphasis on the composite waste water RO permeate. The instrumentation design emphasized the development of an O<sub>3</sub>/UV Oxidation Unit Process simulator and a minicomputer-based control/monitor instrumentation system capable of being integrated with the LMTOC for fully automated operation. As a part of the program effort, a RO Unit Process using DuPont B-10 modules was designed, fabricated and tested. The RO was designed and packaged to be a semiautomatic unit process with provisions for future upgrading in its control/monitor instrumentation.

#### Ozone Contactor Background

This section includes a review of prior efforts in the study of O<sub>3</sub> oxidation of refractory organics, a survey of available O<sub>3</sub> contactors and a brief discussion of the Torricelli O<sub>3</sub> Contactor. Some design concepts of the Torricelli O<sub>3</sub> Contactor were incorporated into the O<sub>3</sub> contactor developed under this research program by Life Systems, Inc. (LSI).

#### Prior Efforts

Ozone oxidation data from earlier MUST WPE development efforts indicate that



the  $O_3$  oxidation of refractory organics in the MUST waste waters is reaction rate limited.<sup>(5-7)</sup> Power expended in some  $O_3$  contactors in stirring the water to increase the rate of  $O_3$  mass transfer in the aqueous phase is not effective because the oxidation rate is limited by kinetics instead of mass transport. The stirring in these contactors resulted in higher  $O_3$  dissociation rates due to the shearing effect on the  $O_3$  bubbles. Further, the studies indicated that 60 to 75% of the power allocated to the WPE (30 kW) was needed for the  $O_3$ /UV Oxidation Unit Process alone. Because of these results, Life Systems initiated a study of alternative  $O_3$  contactor designs to determine if a more optimal  $O_3$  contactor was available which could oxidize the organics at a lower power expenditure.

#### Available Ozone Contactors

A review of available contactors revealed that a variety of systems have been used or suggested for the  $O_3$ /water contacting process. However, no design investigated met the specific needs of the MUST WPE. The designs<sup>(5,12)</sup> studied were categorized into four main groups and are listed in Table 1.

Based on the review of these contactor types, the sparged column dispersing tower (gas bubbles dispersed in a liquid) contactor appeared to offer the best opportunity for reducing the organics at the lowest possible power expenditure. All the parameters which were shown to affect the reaction rate could easily be controlled and monitored with this type. Effective mass transfer of  $O_3$  could be achieved by selecting stainless steel spargers having the necessary pore diameter to result in bubbles with a diameter of less than 0.25 cm (0.1 in). Near equilibrium quantities of  $O_3$  could be transferred to the aqueous phase by providing sufficient rise height for these bubbles and adequate water residence times. Thus, with this design, the organics could be oxidized at the maximum rate while saving power consumed by stirring in the other contactor designs.<sup>(5,8)</sup>

#### Torricelli Ozone Contactor

Further review of the literature indicated that a specific type of the sparged dispersing tower contactor had been developed by Alfred Torricelli in Europe.<sup>(9)</sup> This contactor was very effective in the disinfection of waste waters with a very high  $O_3$  conversion. This contactor had the added advantage beyond the normal sparged dispersing tower contactor of effectively using all of the residual  $O_3$  in the carrier gas to pretreat the processed water. As such, it acted as an  $O_3$  absorber to eliminate  $O_3$  from being vented with the carrier gas. A schematic of the Torricelli  $O_3$  Contactor is shown in Figure 2.

Major modifications were made to this basic concept to develop the LMTOC. The water inlet and outlet columns were moved down adjacent to the base contactor to meet the 3.5 x 2.0 x 2.1 m<sup>3</sup> (11.50 x 6.50 x 6.75 ft) dimensional constraint of the MUST ward containers. This modification required incorporation of a pump to replace the liquid head. In addition, provisions were made to allow (1) heating and controlling the process water temperature, (2) pH adjustment with monitor and (3) UV light activation. These modifications make the LMTOC a uniquely efficient and compact  $O_3$  contacting system.

TABLE 1 AVAILABLE O<sub>3</sub> CONTACTOR TYPES

- Spray Towers (liquid dispersed in a gas)
- Bubble Plate or Sieve Plate Towers
  - Gas introduced as bubbles of desired size or as bubbles which grow to desired size
  - Massive bubble stream disintegrated in liquid
- Packed Beds
- Dispersing Towers (gas bubbles dispersed in a liquid)
  - Sparged column
  - Sparged column with mechanical mixing
  - Diffusers
  - Positive pressure injection
  - Flooded packed bed

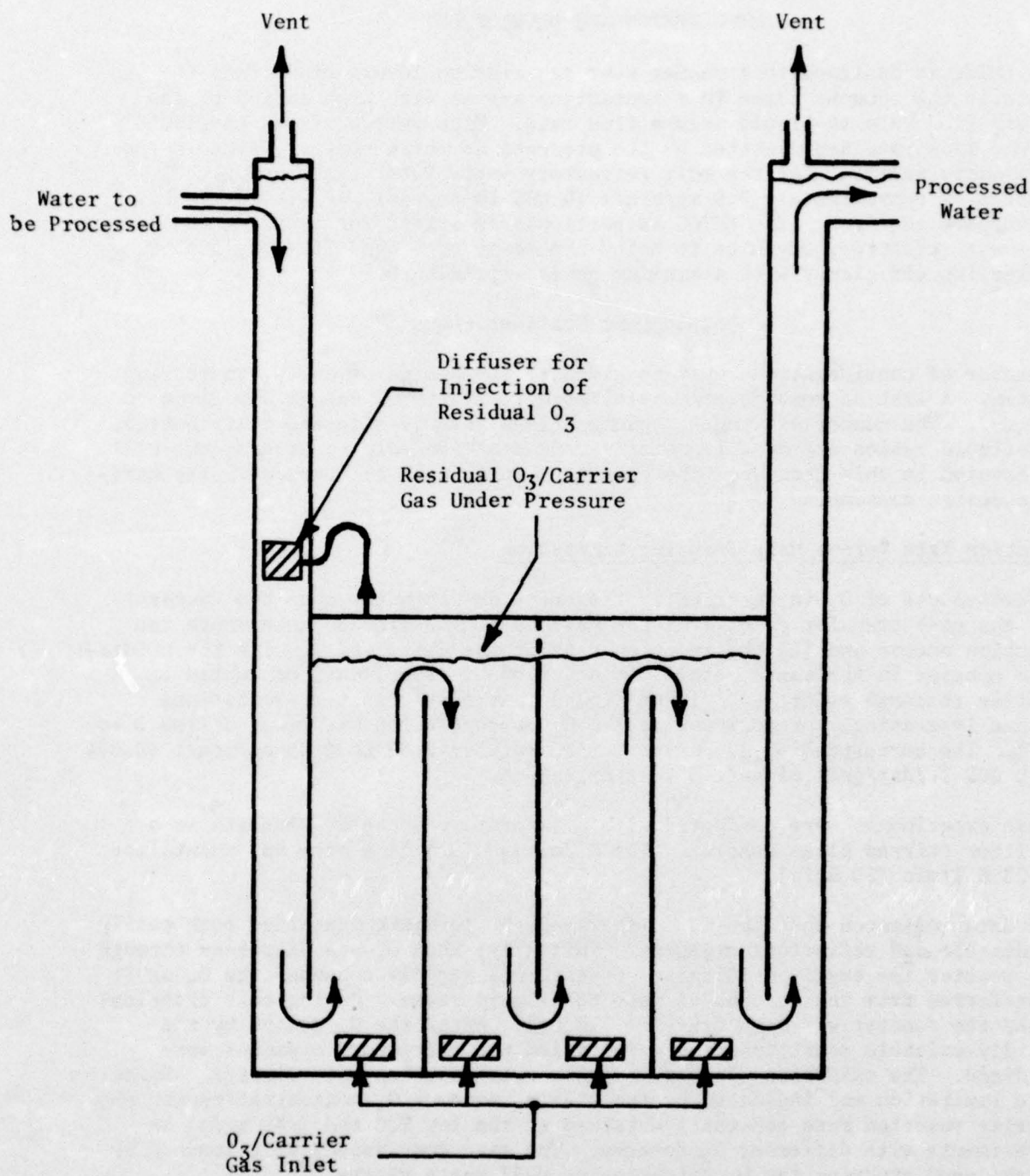


FIGURE 2 TORRICELLI OZONE CONTACTOR SCHEMATIC



## LMTOC DESIGN AND DEVELOPMENT

The LMTOC is designed to transfer near equilibrium levels of  $O_3$  from the gas phase to the aqueous phase in a contacting system with high ratios of gas volume flow rate to liquid volume flow rate. High ratios of gas to liquid volume flow rate are dictated by the presence of refractory organics in the MUST waste waters. For the most refractory waste water (laboratory),  $O_3$  dosages of approximately 7.9 mg/min/l (0.095 lb/day/gal) of wetted reactor volume are required. The LMTOC is particularly suited for reducing the levels of these refractory organics to below the level of 5 mg/l TOC at a high  $O_3$  conversion efficiency with a minimum power expenditure.

### Development Considerations

A number of considerations must be given to the design of any  $O_3$  contacting system. A list of considerations tailored to the LMTOC design are given in Table 2. The number of stages, column height, bubble size and distribution, gas-liquid ratios and mass transfer versus reaction rate considerations will be covered in this section. The remaining items will be covered in the hardware design section.

### Reaction Rate Versus Mass Transfer Limitation

Effective use of  $O_3$  in waste water treatment is dependent upon two factors: (1) the mass transfer of  $O_3$  from the gaseous to the liquid phase where the reaction occurs and (2) the reaction rate of the dissolved  $O_3$  with the oxidizable species in the waste water. In a series of experiments conducted in earlier research efforts,<sup>(5)</sup> it was found that mass transfer limitations became increasingly predominant as the  $O_3$  concentration was reduced from 2 to 0.3%. The corresponding  $O_3$  dosage varied between 2.85 to 0.43 mg/min/l (0.034 to 0.005 lb/day/gal) of wetted reactor volume.

These experiments were conducted with a laboratory waste RO permeate in a 14-liter stirred glass reactor. The  $O_3$ /oxygen ( $O_2$ ) flow rate was maintained at 23.6 l/min (50 scfh).

The data indicated that the MUST laboratory RO permeate contained both easily oxidizable and refractory organics. Initially, when  $O_3$  was dispersed through the reactor the easily oxidizable constituents rapidly consumed the  $O_3$  as it transferred from the  $O_3$  bubbles into the liquid phase. During this transient phase the reactor was mass transfer limited. After the  $O_3$  demand by the rapidly-oxidizable constituents was fulfilled the refractory organics were oxidized. The oxidation of these organics was reaction rate limited. Reaction rate limitation was indicated by the stable, aqueous  $O_3$  concentration and the similar reaction rate constants obtained at the low TOC end (<40 mg/l) in experiments with different  $O_3$  dosages. The same conclusions<sup>(6)</sup> were reached by Sierka when studying the  $O_3$  oxidation of MUST waste waters.

Chian<sup>(7)</sup> and Hewes<sup>(8)</sup> have shown that volatile organics (e.g. ethanol) can be stripped from the MUST waste waters with volume of gas per unit volume of

TABLE 2 LMTOC DESIGN CONSIDERATIONS

- Contactor type
- Number of stages
- Column height
- Bubble size and distribution
- Gas-liquid ratio
- Mass transfer versus reaction rate
- Method of gas dispersion (e.g., gas in liquid)
- Contactor flow compartment configuration
- Catalytic effects on gas/liquid contact
- Co-current/counter-current flow
- Materials of construction
- UV light activation



liquid per minute (VVM) levels higher than one. The significance of the stripping mechanism on the rate of TOC reduction in prior ozonation efforts has yet to be determined.

#### Number of Ozone Oxidation Contactor Stages

In a plug-flow reactor the concentration of reactant decreases progressively as fluid passes through the system. In a totally stirred, mixed-flow reactor, the concentration drops more rapidly to the low effluent value. A plug-flow reactor is more efficient than a totally stirred, mixed-flow reactor for reactions in which the rate is dependent only on the reactant concentrations. (10) The  $O_3$  oxidation rate of the MUST waste water has been shown to be reactant concentration dependent; (6-8) hence, the plug-flow reactor is theoretically a more optimum design.

For the MUST WPE an engineering trade-off study between system complexity and volume minimization indicated that a six-stage reactor was the most practical design. With the six-stage, mixed-flow reactor, the volume can be expected to be 1.3 times greater than the plug-flow reactor design for a 95% organic level reduction.

#### Free Ozone Sparging as an Ozone Transfer Mechanism

As discussed above, the  $O_3$  oxidation of the laboratory RO permeate was shown to be mass transfer limited in the initial stage. However, once the easily oxidizable organics are oxidized, the process becomes reaction rate limited. Under reaction rate limitations a system which can minimize power expenditure for mass transfer of  $O_3$  into the aqueous phase and maximize  $O_3$  conversion is clearly superior. The LMTOC is designed for better than 90%  $O_3$  mass transfer into the aqueous phase without any stirring power expenditure.

Figure 3 shows the LMTOC test stand schematic. The LMTOC is a six-stage, gas-sparged contactor with each stage being a vertical vessel of liquid having a gas disperser at the bottom without stirrers or other moving parts. The gas bubbles flow upward through a co-current or counter-current flow of liquid so that the liquid phase is continuous. The reaction proceeds in the liquid phase with  $O_3$  transferred from the gas phase. For higher  $O_3$  conversion the off gases from the six stages are collected and sparged through the precontactor stage. The configuration of the precontactor stage is similar to the other six stages as described above. Using the mass transfer coefficients for such gas-sparged  $O_3$  reactors obtained by Hill and Spencer, (11) it was found that with bubble diameters of 0.25 cm (0.1 in) nearly 90% of the equilibrium amount of  $O_3$  absorbed into the aqueous phase in 1.8 m (6 ft) of liquid height.

#### UV Light Activation

Utilization of UV light to increase the  $O_3$  oxidation reaction rates has been demonstrated in prior research efforts. (5,8,12) The presence of UV light is believed to have caused the increase in decomposition rate of dissolved  $O_3$  molecules to form free radicals. The reduction rates of TOC showed drastic increases in some cases with UV activation.

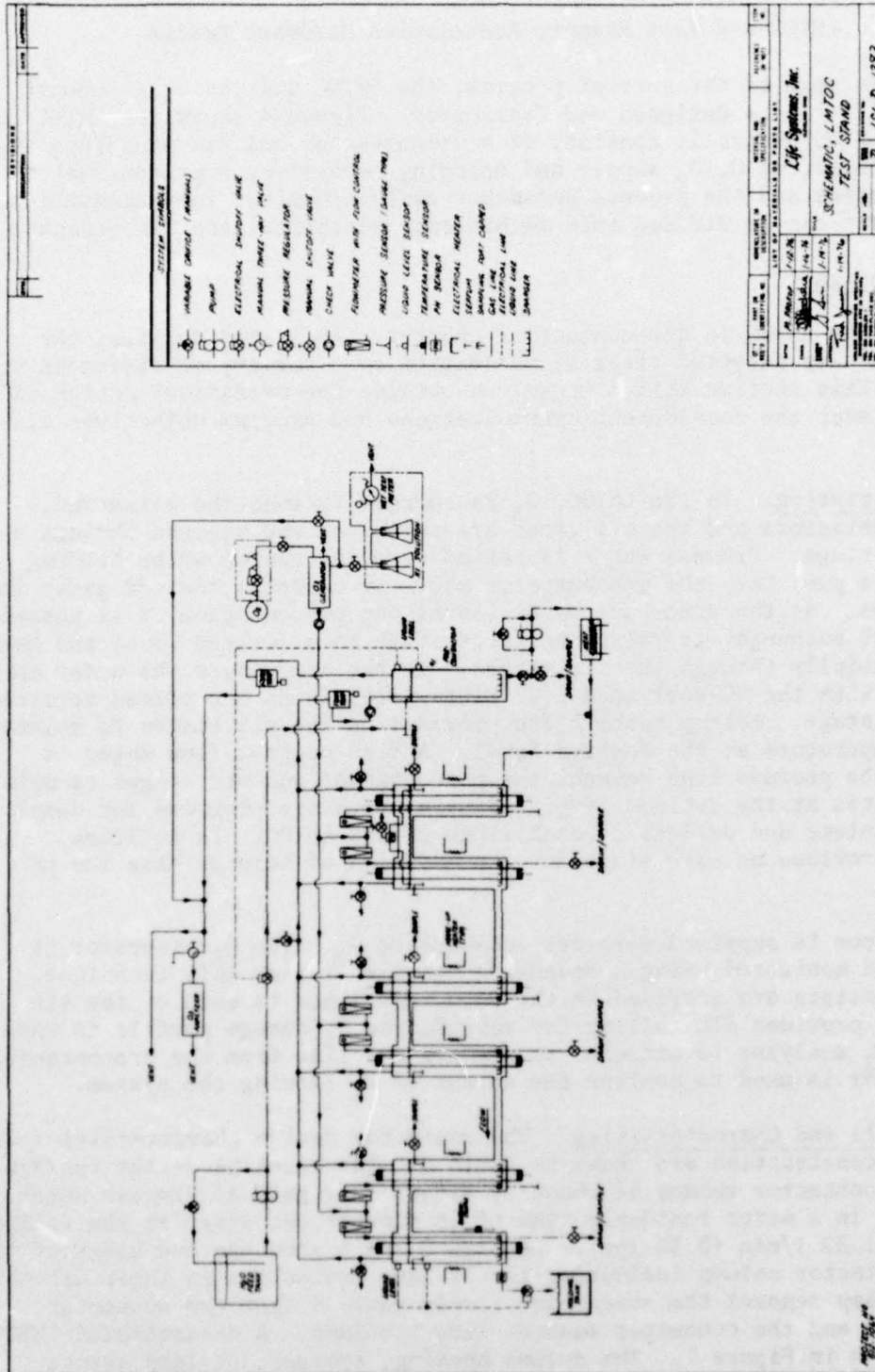


FIGURE 3 LMTOC TEST STAND SCHEMATIC



## LMTOC and Test Support Accessories Hardware Design

As the primary goal of the current program, the LMTOC and its Test Support Accessories (TSA) were designed and fabricated. Figure 4 shows the LMTOC test stand. The LMTOC basically consists of a precontactor and six stainless steel contacting stages, an  $O_3/O_2$  supply and sparging subsystem, a process water control subsystem and the process parameter control/monitor instrumentation. The development can be divided into mechanical, electrical and TSA designs.

### Mechanical Design

The heart of the LMTOC is the contacting chamber. As stated earlier, the sparged column was selected after an evaluation of a variety of different  $O_3$  contactors. This section will discuss and outline the mechanical design of the LMTOC to meet the development considerations and program objectives discussed above.

Hardware Description. In the LMTOC,  $O_3$  is sparged through the six-stage, mixed-flow contactors and the off gases are collected and sparged through a precontactor stage. Process water is passed from the source water holding tank through a pump into the precontactor where it contacts the off gases from the six stages. As the processed water leaves the precontactor it is passed through a heat exchanger to raise the temperature to a desired level and then passes sequentially through the six stages. In the six stages the water comes into contact with the UV-activated  $O_3$ . Ultraviolet lamps are placed vertically through each stage. Makeup heaters are provided on the six stages to maintain the water temperature at the desired level. A flow control flow meter is provided in the process line between the precontactor and six stages to maintain water flow rates at the desired level. Sample ports are provided for sampling at both the inlets and outlets of each stage of the LMTOC. In addition, septums are provided at each stage for the addition of acid or base for pH adjustment.

The  $O_3$  generator is supplied with dry, compressed  $O_2$ . The  $O_3$  generator is calibrated and monitored using a standard potassium iodide (KI) technique. Control flow meters are provided in the  $O_3/O_2$  gas lines to each of the six stages. This provides flexibility for varying the  $O_3$  dosage profile to each column. An  $O_3$  analyzer is attached to the off gas line from the precontactor. The  $O_3$  analyzer is used to monitor the amount of  $O_3$  leaving the system.

LMTOC Materials and Characteristics. The contactor design characteristics and materials of construction are shown in Table 3. The baseline design configuration has a contactor volume of about 35 liters (9.2 gal) of process water which results in a water residence time of 26 minutes per stage at the design flow rate of 1.32 l/min (0.35 gpm). Figures 5 and 6 show the end views of the assembled contactor column indicating the UV lamp connectors in their assembled state. For lamp removal the screws are simply removed from the connector retaining ring and the connector removed with the lamp. A disassembled LMTOC column is shown in Figure 7. The column housing, sparger, UV lamp quartz



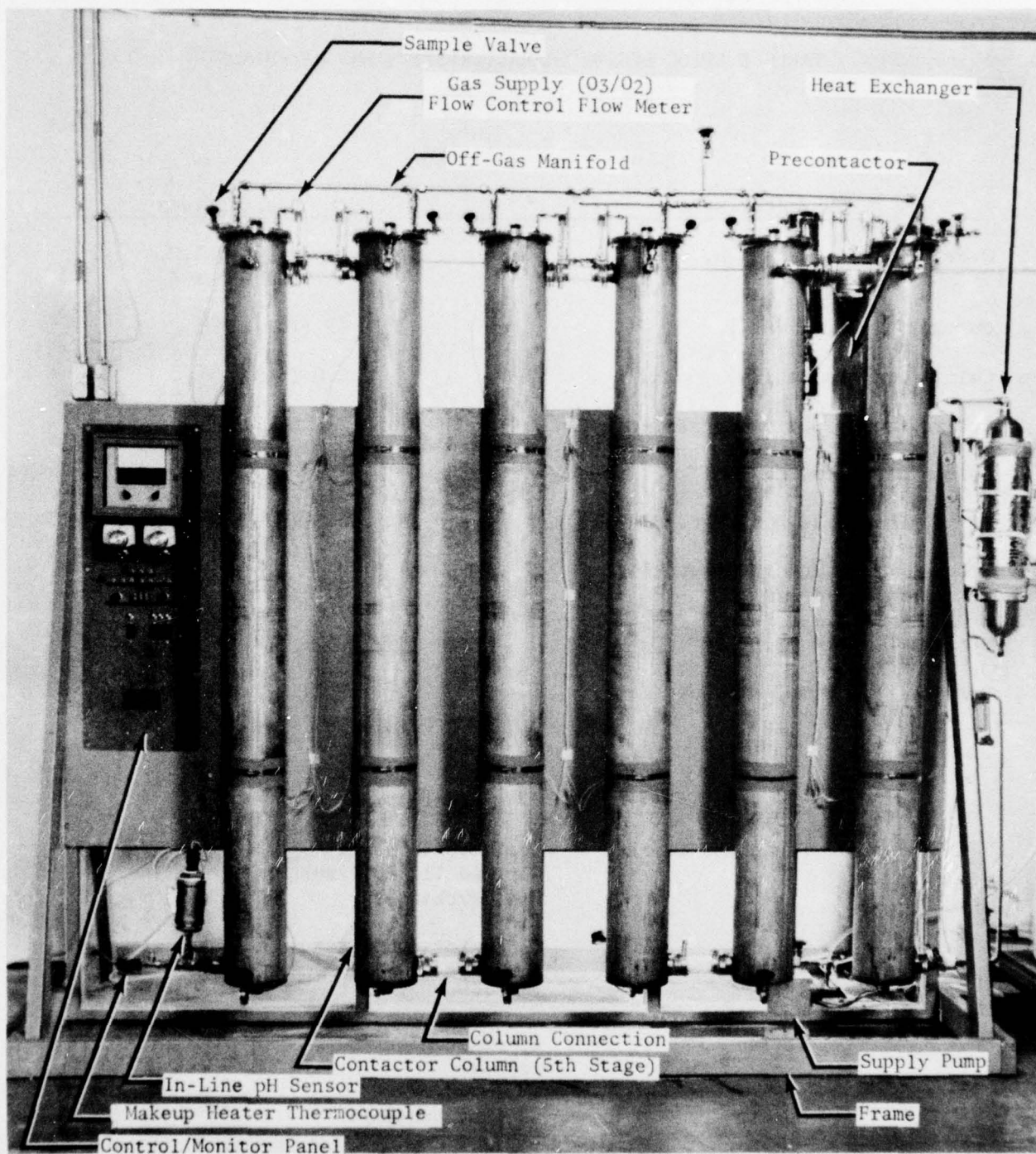


FIGURE 4 ASSEMBLED LMTOC TEST STAND

TABLE 3 LMTOC DESIGN CHARACTERISTICS AND MATERIALS

Characteristic	Descriptions
Overall Size, H x W x D, m (Ft)	2.3 x 2.8 x 1.2 (7.5 x 9.3 x 4)
Column Volume, l (Ft <sup>3</sup> )	35 (1.2)
Column Heights, m (Ft)	2.0 (6.5)
Column Cross-Sectional Area, cm <sup>2</sup> (In <sup>2</sup> )	202.6 (31.4)
Column Diameter, cm (In)	16.8 (6.6)
Off Gas Manifold Diameter, cm (In)	1.3 (0.5)
Process Water Inlet/Outlet Line Diameter, cm (In)	3.5 (1.4)
Sparger Surface Area, cm <sup>2</sup> (In <sup>2</sup> )	95.5 (14.8)
Sparger Pore Size, microns (In)	5 (1.97 x 10 <sup>-4</sup> )

Materials	Description
Stainless Steel	Contactator housing Gas and liquid lines Heat Exchanger Pumps Contactator endplates Spargers Fittings
Teflon	Column connectors Septums
Quartz	UV lamp housings
Glass	Flow control flow meters

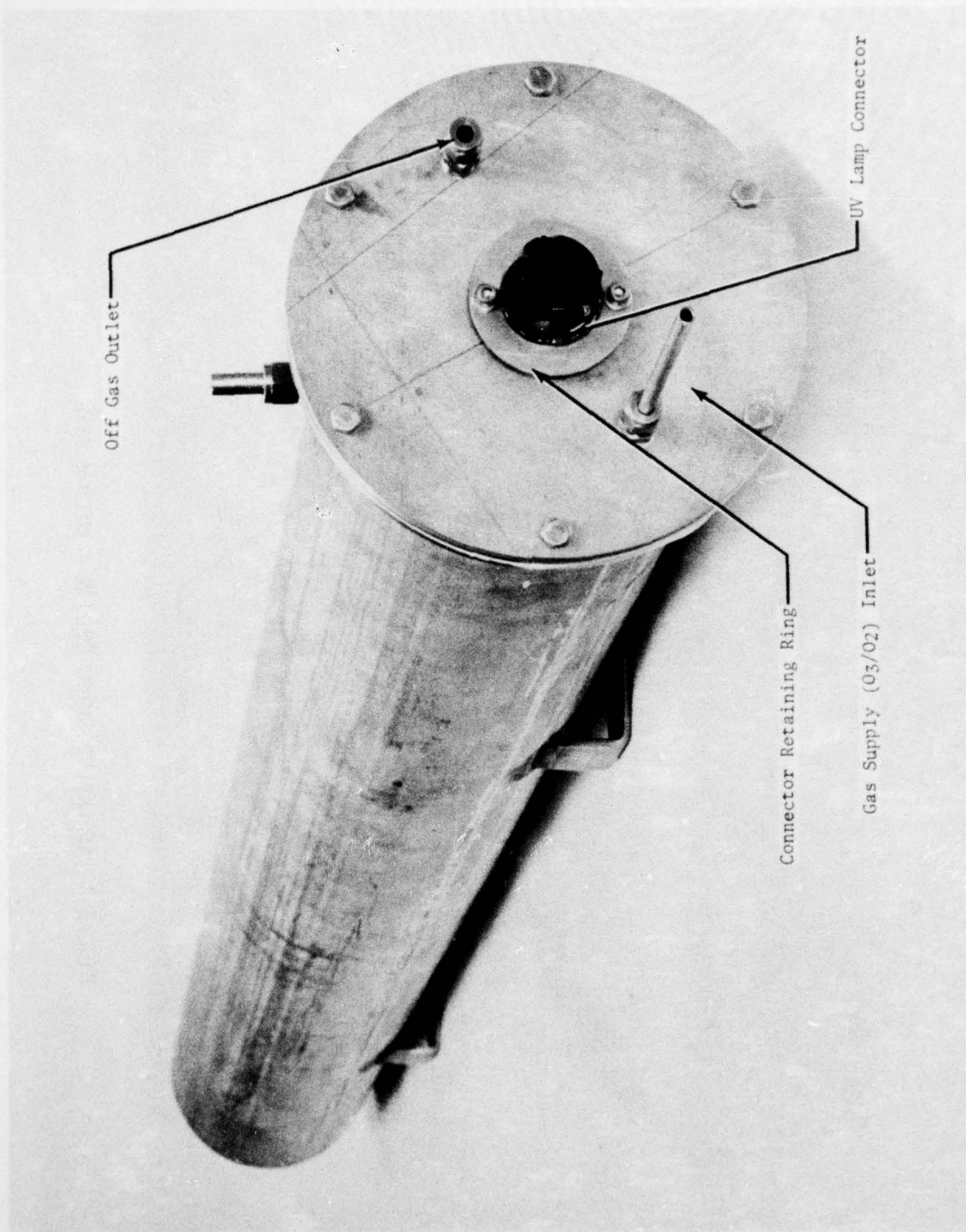


FIGURE 5 ASSEMBLED LMTOC COLUMN - TOP VIEW



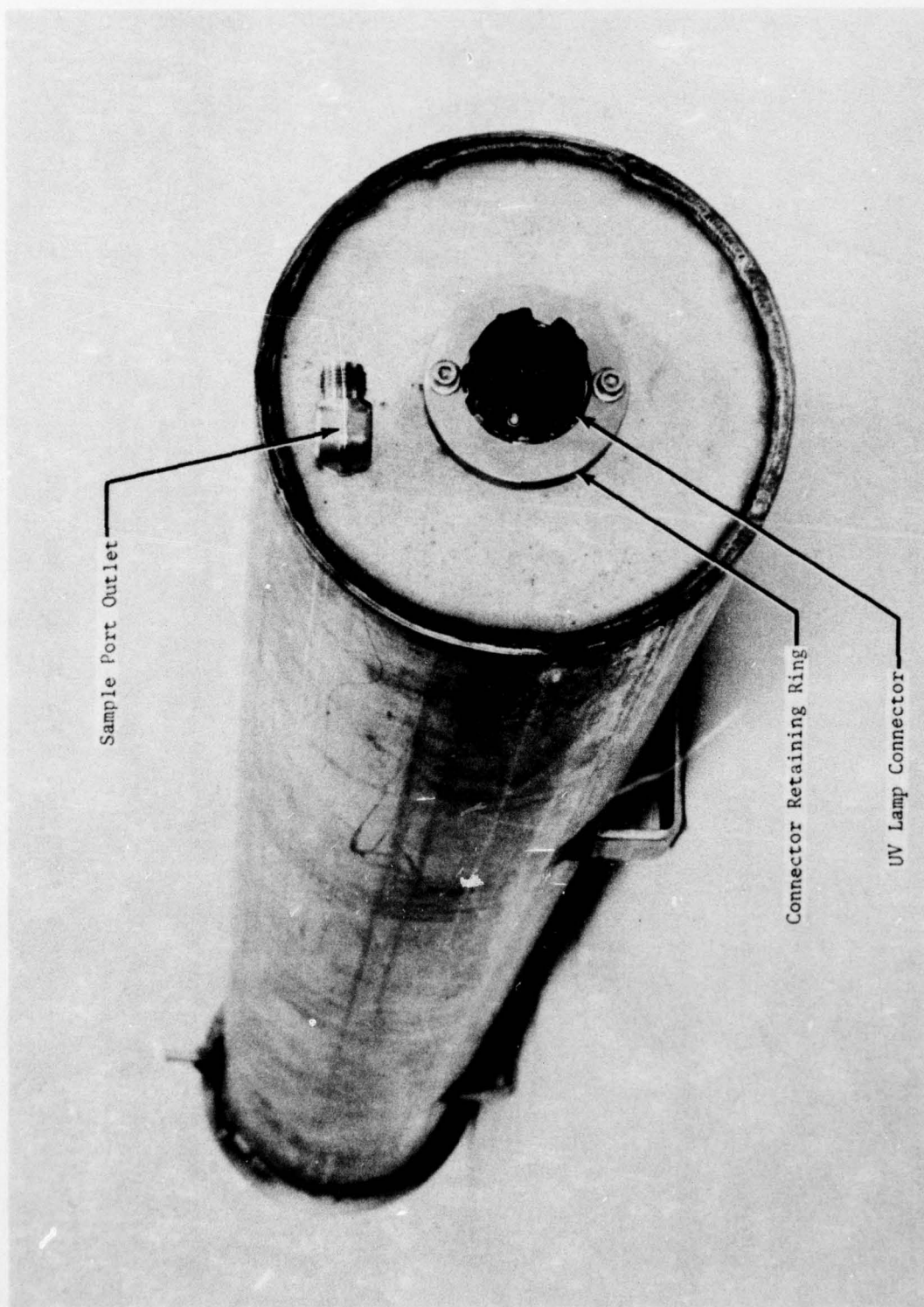


FIGURE 6 ASSEMBLED LMTOC COLUMN - BOTTOM VIEW

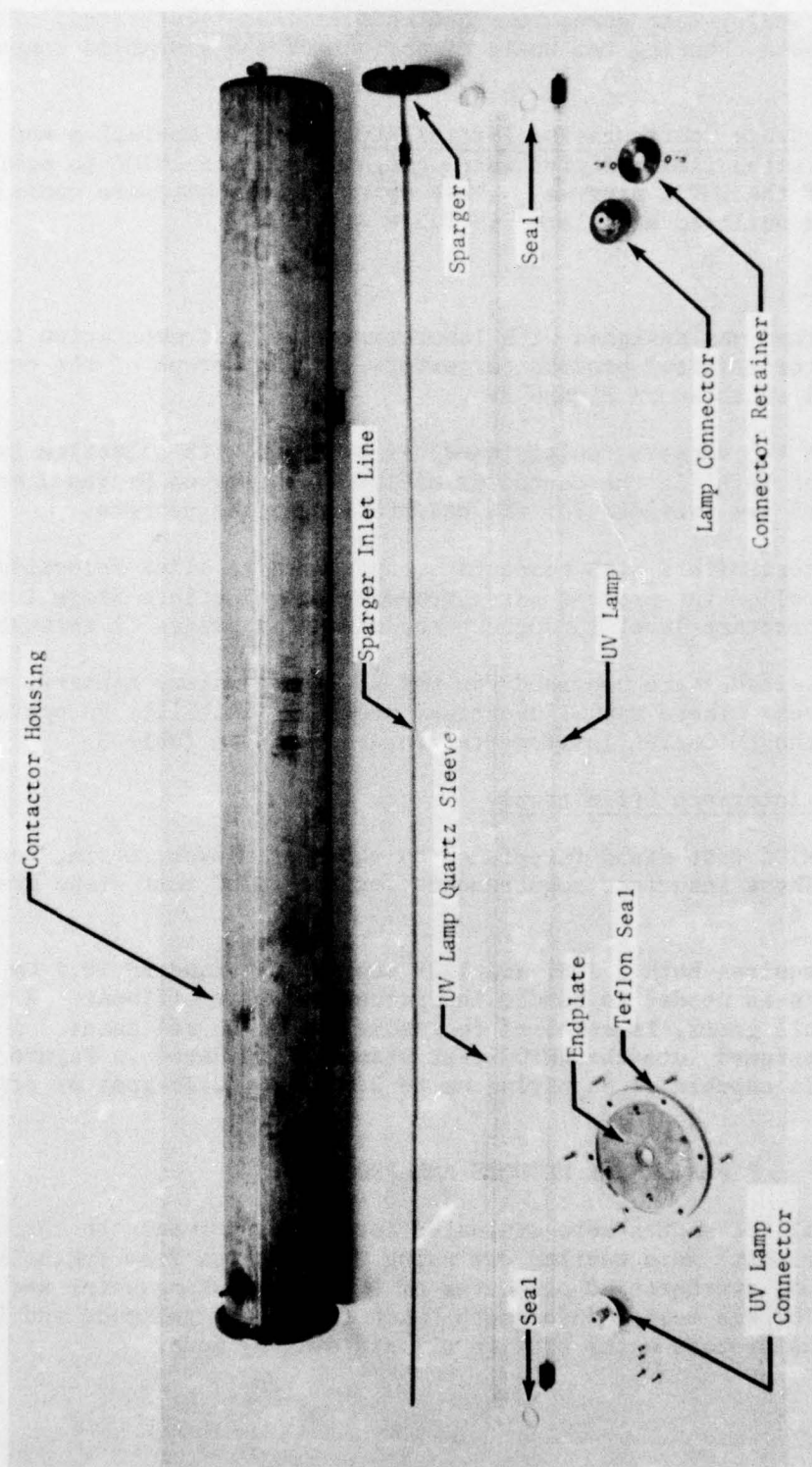


FIGURE 7 DISASSEMBLED LMTOC COLUMN

sleeve, UV lamp, seals, lamp connectors and endplate are illustrated. Figure 8 is a detailed drawing showing the basic dimensions of the assembled contactor column housing.

Operating and Hardware Configuration Flexibility. Certain operating and hardware configuration flexibilities were designed into the LMTOC to achieve the objectives of the LMTOC program. These operating and hardware configuration flexibilities are outlined and listed in Table 4.

#### Electrical Design

The LMTOC test stand was designed with laboratory-type instrumentation to control and monitor critical process parameters. A photograph of the control and monitor panel is shown in Figure 9.

A pH monitor with two sensors (multiplexed) is provided with selection switches to read out the pH level in the contactor effluent or between Stages 1 and 2. Two potentiometers are provided for the calibration of the sensors.

Two temperature controllers with readouts are provided to allow selection, readout and control of the process water temperature going into Stage 1 and to maintain the temperature level throughout the contactor stages (1 through 6).

Manual override switches are provided for the UV lamps, makeup heaters, pumps and solenoid valves. These manual overrides provide flexibility in operation. The features of the LMTOC/TSA instrumentation are shown in Table 5.

#### LMTOC Test Stand Interface Definitions

There are four LMTOC test stand interfaces to consider: power, drain, vent and product water. These interface requirements for the LMTOC test stand are defined in Table 6.

The test stand requires both a 208V and 120V supply. A standard 10.2 cm (4 in) floor drain is needed to handle the processed water effluent. A vent, free of combustible gases, is required to remove the  $O_3/O_2$  off gases. A supply pump is designed into the LMTOC test stand as indicated in Figure 3. The supply pump is capable of supplying up to 2.9 l/min (0.76 gpm) of process water.

### EXPERIMENTAL METHODS AND PROCEDURES

Various analytical techniques were evaluated for the experiments to characterize the LMTOC. Experiments were carried out using RO permeates from synthetic composite RO feeds, synthetic RO permeates of laboratory waste water and ethanol. The LMTOC was tested under both batch (single stage) mode and integrated (continuous process water flow in all six stages) mode.



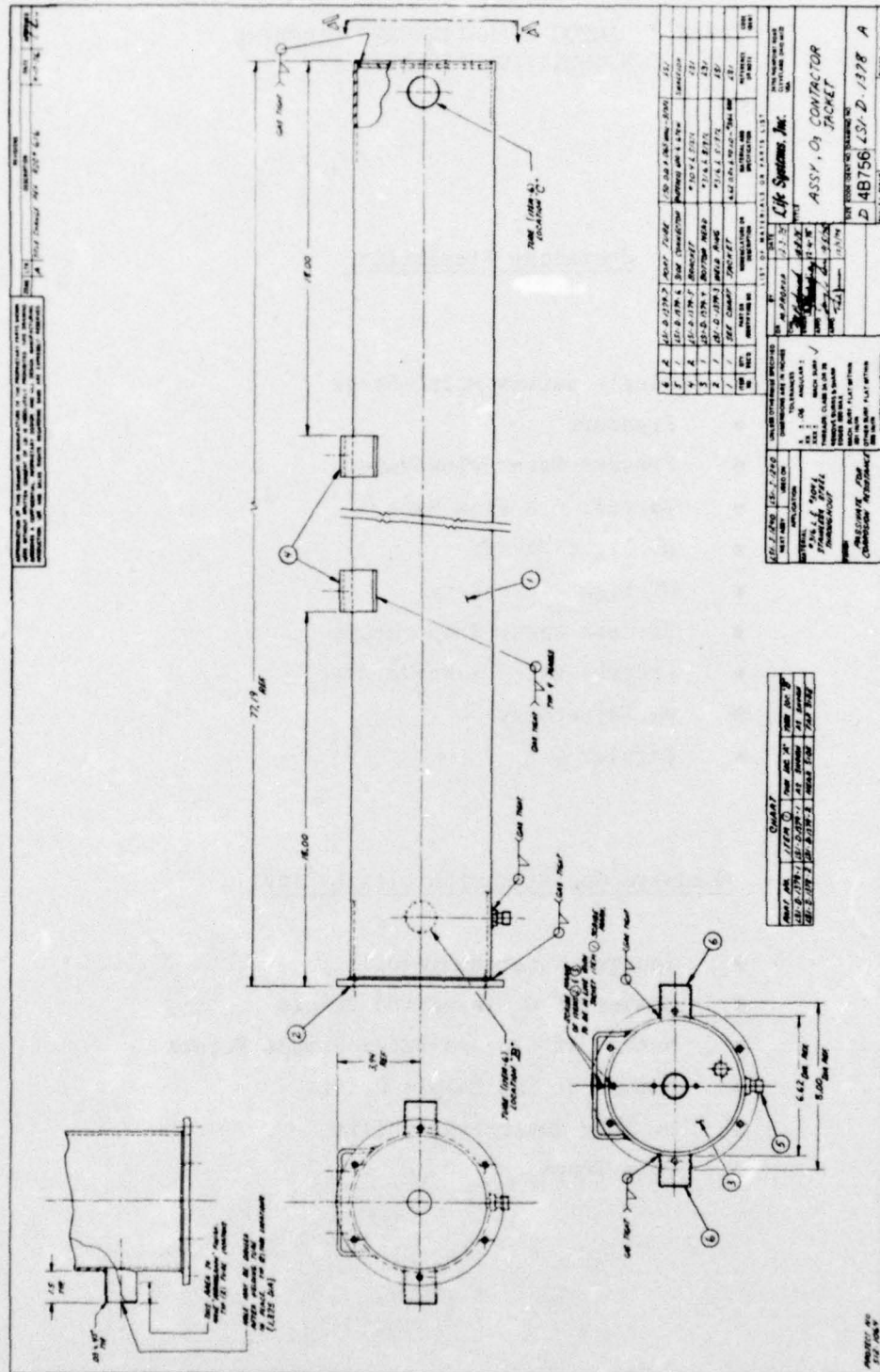


FIGURE 8 ASSEMBLED LATOC COLUMN HOUSING

TABLE 4 LMTOC OPERATING AND HARDWARE  
CONFIGURATION FLEXIBILITY

Operating Flexibility

- Single versus Multi-Stage
- Pressure
- Process Water Flow Rate
- Carrier Gas Flow Rate
- UV Light On/Off
- UV Light Intensity
- Process Water Temperature
- Process Water Make-Up Heat
- pH Adjustment
- Carrier Gas

Hardware Configuration Flexibility

- Sparger Interchangeability
- Number of O<sub>3</sub> Injection Points
- Number of Process Water Sample Points
- Number of Gas Sample Points
- UV Lamp Interchangeability
- Foam Traps



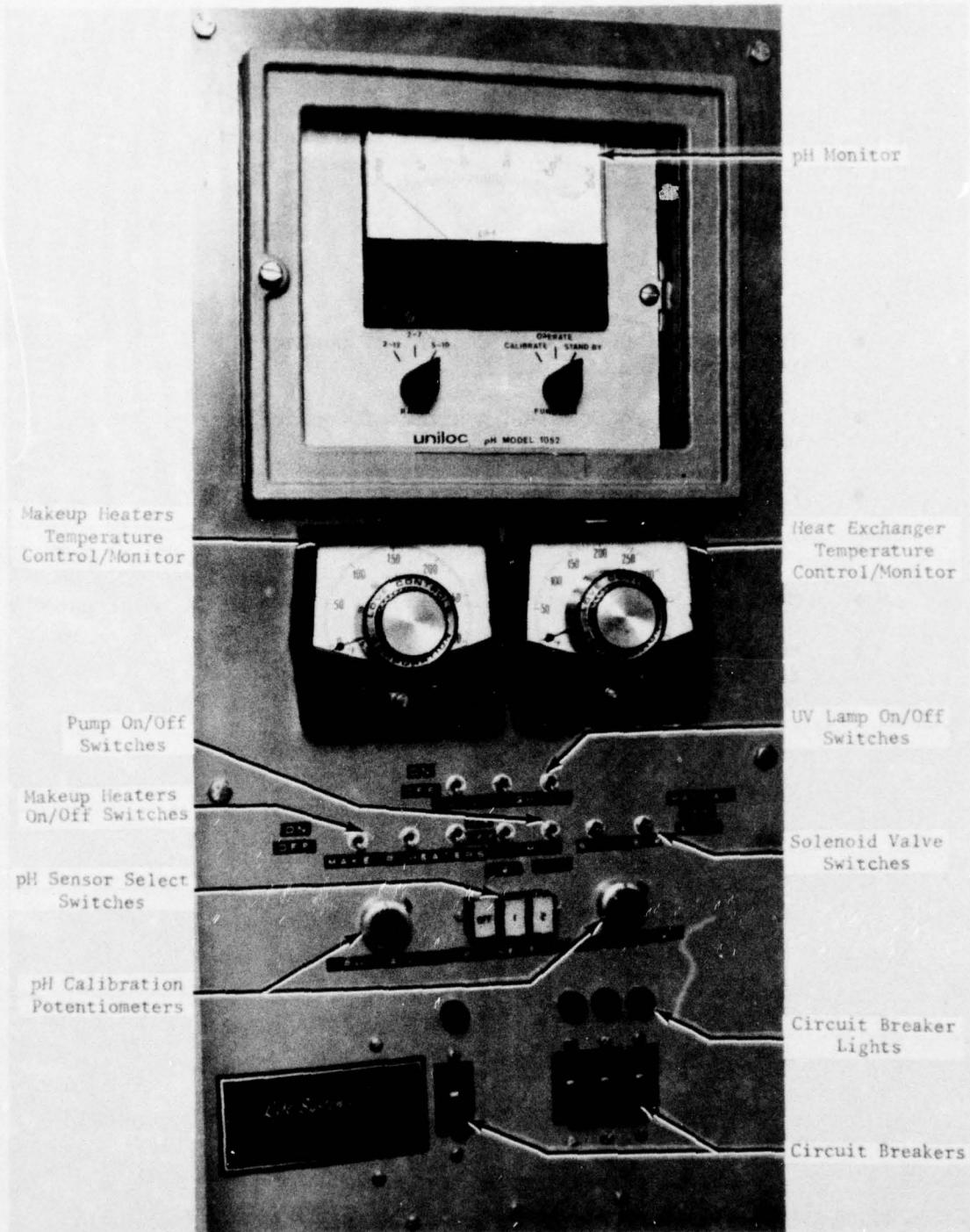


FIGURE 9 LMTOC CONTROL/MONITOR INSTRUMENTATION PANEL

TABLE 5 LMTOC/TSA INSTRUMENTATION FEATURES

- Temperature control and monitor of contactor influent water
- Temperature control and monitor of contactor water to make up heat loss
- Precontactor water level control
- Contactor water level control
- Manual override switches for all actuators
- Manual pH sensor multiplexing to monitor pH at the second stage or in the process water effluent

TABLE 6 LMTOC TEST STAND INTERFACE DEFINITIONS

<u>Interface</u>	<u>Definitions</u>
Power	208V, 3 Phase with Neutral, 60 Hz, 3 kW  120V, Single Phase, 60 Hz, 2 kW
Drain	Standard 10.2 cm (4 In) Floor Drain
Vent <sup>(a)</sup>	Standard 8.9 cm (3½ In) 8.5 x 10 <sup>3</sup> l/Min (300 Cfm) Exhaust Fan
Process Water	Variable <sup>(b)</sup> 1.3 to 2.9 l/Min (0.35 to 0.76 Gpm)

(a) Exhaust line must be free of combustible gases

(b) Function of influent waste water type



#### Methods

The analytical techniques for the various analyses<sup>(13)</sup> used for the LMTOC system characterization are listed in Table 7. On key experiments, head space analysis for volatiles, urea, nitrate and nitrite analyses were conducted by the U.S. Army Medical Research and Development Command (USAMRDC), Fort Detrick, MD. The results are shown in Appendix 1.

#### Waste Water Formulations

Synthetic RO feed constituents for the MUST medical composite waste water are listed in Table 8. The TOC level of the RO feed was found to be 45.5 mg/l. The RO feed was made in an 835 liter (221 gal) batch and concentrated to 20X (90% recovery) to obtain 793 liters (210 gal) of RO permeate (LMTOC MUST composite waste feed). The TOC of the RO permeate varied between 14.2 mg/l and 16.2 mg/l.

A synthetic RO permeate was prepared for the laboratory waste. The constituents for this waste are listed in Table 9. The approximate TOC of the laboratory simulated RO permeate is 105 mg/l. Refractory low molecular weight organics like methanol and acetone account for 99 mg/l (94%) of the TOC.

#### Sample Port Locations

The locations where samples were taken during the experimental efforts are defined in Figure 10. These sample locations are referenced throughout the experimental results discussion.

#### Experimental Program

The experimental program for the characterization and comparison of the LMTOC is summarized in Table 10. The experimental program has centered around the shakedown and checkout testing of the LMTOC, ethanol comparison experiment and the study of five parameters with the MUST composite waste water. The five parameters and ranges studied are outlined in Table 11.

#### LMTOC EXPERIMENTAL RESULTS

During the course of the LMTOC development and testing,  $O_3$  conversion tests, experiments of LMTOC with MUST composite and laboratory waters, experiments with ethanol and experiments with pH, temperature and UV intensity were conducted. These experiments were designed to study the feasibility and characteristics of the LMTOC and the effects of various process parameters in reducing the organic compounds in the process water.

#### Ozone Conversion Tests

A series of five tests were conducted to study the  $O_3$  conversion in the absence of any  $O_3$  demand by organics in the waste water. In these tests the LMTOC was

TABLE 7 ANALYTICAL TECHNIQUES FOR VARIOUS  
ANALYSES USED FOR THE LMTOC

<u>Analyses</u>	<u>Method/Instrument</u>
Nitrate - Nitrite as Nitrogen	EPA Automatic Cadmium Reduction Method
Total Organic Carbon (TOC)	Dorhmann TOC Analyzer
Chemical Oxygen Demand (COD)	EPA Chemical Analysis Protocols
Ambient Ozone	(a) McMillan Chemiluminescent Analyzer (b) Wet KI Technique
Conductivity	(a) Balsbaugh On-Line Analyzer (b) Beckman Conductivity Bridge
pH	(a) Uniloc In-Line pH Sensor (b) Markson Lab pH Analyzer

TABLE 8 SYNTHETIC RO FEED CONSTITUENTS FOR  
MUST MEDICAL COMPOSITE WASTE WATER<sup>(a)</sup>

Compound	Concentration, μl/l	TOC, mg/l
Methanol	29.8	8.8
Acetone	6.3	3.1
Acetic Acid	3.4	1.4
Diethyl Ether	0.6	0.3
N,N-Diethyl-m-toluamide	0.8	0.6
Ethanol	0.5	0.2
Oleic Acid	0.5	0.3
Phenol	1.3 mg/l	1.0
Urea	18 mg/l	3.6
Kodak X-Omat Developer	942	} 26.1
Kodak X-Omat Fixer	942	
Total		45.4

(a) Result of joint discussions between Life Systems, Inc.  
and USAMRDC (3/26/76)



TABLE 9 SYNTHETIC RO PERMEATE CONSTITUENTS  
FOR LABORATORY WASTE WATER (a)

<u>Compound</u>	<u>Concentration, ul/l</u>	<u>TOC, mg/l</u>
Methanol	285.0	84.0
Acetone	30.0	15.0
2-Propanol	1.5	0.72
Diethyl Ether	0.3	0.15
Methyl Ethyl Ketone	0.6	0.33
Formaldehyde	1.5	0.48
Ethanol	1.5	0.63
Phenol	1.2	0.93
o-Toluidine	0.3	0.24
N,N-Diethyl-m-toluamide	0.6 mg/l	0.45
Acetic Acid	3.4 mg/l	1.12
Triton X-100	1.58 mg/l	<u>1.0</u>
	Total	105.05

(a) Result of joint discussions between Life Systems, Inc. and  
USAMRDC (3/26/76)

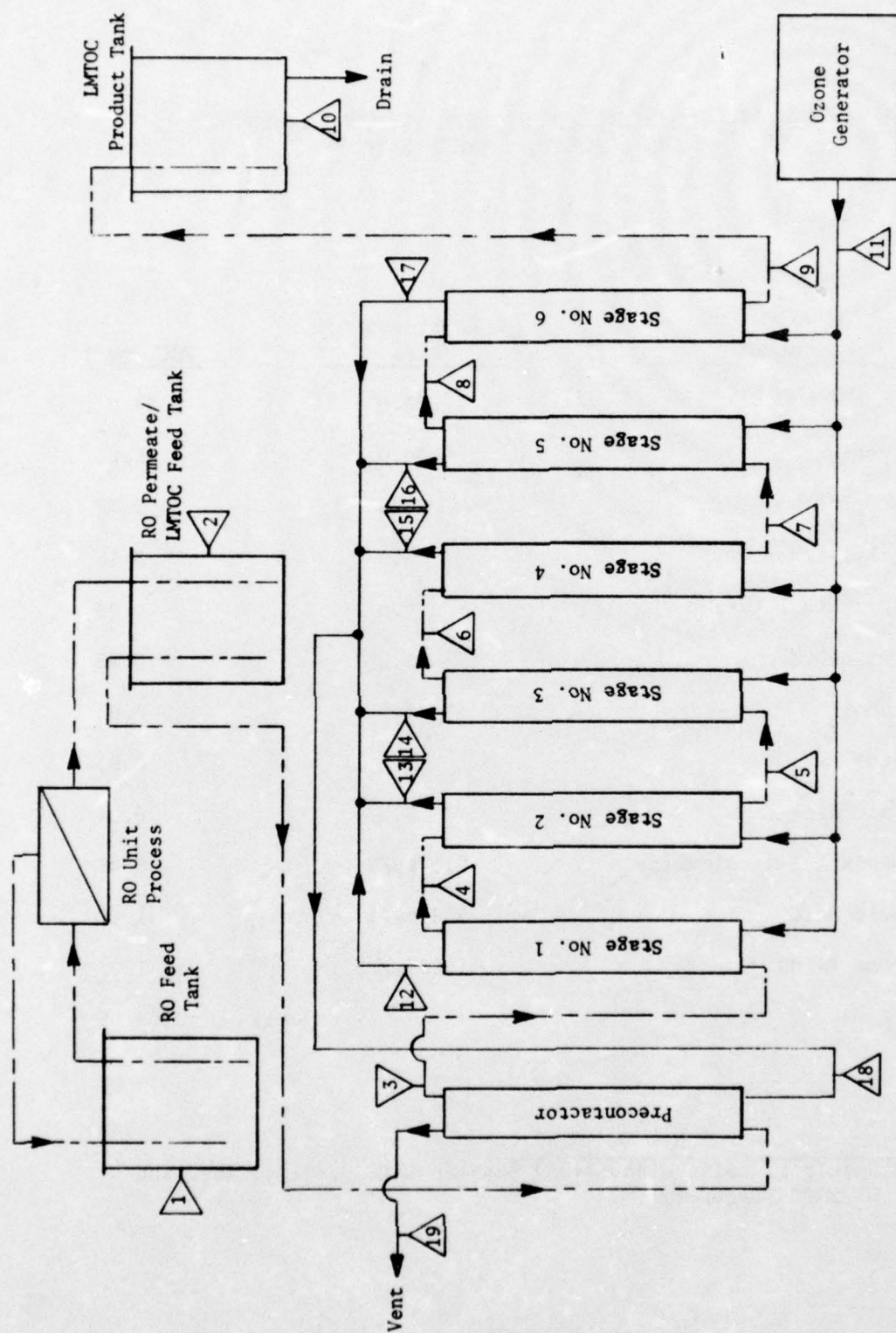


FIGURE 10 RO/LMTOC TEST STAND SAMPLE STATIONS

TABLE 10 LMTOC EXPERIMENTAL SUMMARY

- Checkout (Integrated)
  - Mechanical/Electrical
  - Overnight Sparging
  - Blank  $\Delta$ TOC and  $\Delta$ COD
  - LMTOC Autodecomposition with and without UV
  - Stirred Contactor Autodecomposition with and without UV
- Ethanol Comparison (Batch)
- Laboratory Waste (Batch)
- Composite Feasibility (Integrated)
- Composite pH Effects
  - pH 11 (Batch)
  - pH 7 (Batch)
  - Best (Integrated)
- Composite Temperature Effects
  - 303K (86F) (Batch)
  - 333K (140F) (Batch)
- Complete pH, temperature, power and expendable, trade-off study
- Composite Ozone Dosage
- Composite UV, Last Three (Integrated)
- Composite Gas Flow Rate
- Laboratory, Best of All (Integrated)



TABLE 11 LMTOC EXPERIMENTAL PARAMETERS AND RANGES

Parameter	Range
O <sub>3</sub> Dosage, mg/Min/1 process water (Lb/Day/Gal)	2.8 to 7.9 (0.034 to 0.095)
pH	7 to 11
Temperature, K (F)	303 to 333 (86 to 140)
UV	On/Off, 2, 4, or 6 stages
O <sub>3</sub> /O <sub>2</sub> Gas Flow Rate, l/Min (Scfh)	4.7 (10), batch 28.3 to 37.8 (60 to 80), integrated

run in the continuous mode with well water at 1.3 l/min (0.35 gpm). The results for these tests are shown in Table 12. Ozone conversion is defined as:

$$\text{Percent } O_3 \text{ Conversion} = \frac{O_3 \text{ In} - O_3 \text{ Out}}{O_3 \text{ In}} \times 100$$

An  $O_3$  mass balance equation is shown below:

$$\begin{aligned} \text{Rate } O_3 \text{ In} &= \text{Rate } O_3 \text{ Out} + \text{Rate } O_3 \text{ Dissolved} \\ &+ \text{Rate } O_3 \text{ Dissociated} + \text{Rate } O_3 \text{ Demand} \end{aligned}$$

The rate of  $O_3$  In was obtained from the ozonator calibration curve. The  $O_3$  Out rate was read with the McMillan  $O_3$  Analyzer. The amount of  $O_3$  dissolved, in the absence of UV light, is known to follow Henry's Law.<sup>(14)</sup> No known data exist for Henry's constant in the presence of UV activation. However, at neutral pH and 318K (113F), the solubility of  $O_3$  in aqueous solutions is less than 1%.<sup>(14)</sup> The rate  $O_3$  dissociated and the rate  $O_3$  demand are interdependent.

Due to the highly reactive and unstable nature of  $O_3$ , it is dissociated to a significant extent by the gas spargers. Perrich<sup>(15)</sup> and Chian<sup>(16)</sup> have observed 30 to 80% dissociation of  $O_3$  in fritted gas spargers. At elevated pH's, temperatures and with UV activation, the decomposition of  $O_3$  in the aqueous phase, is expected to be significant.<sup>(14)</sup>

The well water had a background TOC of 2 mg/l. Due to the standard uncertainty ( $\pm 1$  ppm) of the TOC analyzer at low TOC levels, the TOC level of the well water might equal an  $O_3$  demand of less than 1% of the influent  $O_3$ , based on the average oxidation stoichiometry of organic solutes.

The above discussion indicates that most of the  $O_3$  conversion in the well water can be accounted for by  $O_3$  dissociation at the sparger or in the aqueous phase.

The results of Tests 1 and 2, presented in Table 12 and Figure 11, show that without UV light activation, approximately 80% of the  $O_3$  passed through the contactor stages was converted. With UV light activation (Test 3), the  $O_3$  conversion increased to about 90%.

In the precontactor where there was no UV activation in all cases, the  $O_3$  conversion was lower (18%) in Test 1 with lower  $O_3$  dosage (165 mg/min) compared against Test 2 (24% conversion and 165 mg/min  $O_3$  dosage).

In Tests 4 and 5 the entire flow was diverted through Stages 1 and 4, respectively. With UV activation approximately 80%  $O_3$  was converted across these stages. The explanation for these results still remains to be studied. However, there appears to be a correlation between the gas flow rate, the  $O_3$  dosage, the UV light and the percent of  $O_3$  conversion. At higher gas flow rates (e.g., the precontactor and Tests 4 and 5), a lower  $O_3$  conversion was

TABLE 12 EXPERIMENTAL CONDITIONS AND OZONE CONVERSION RESULTS

Test Parameters	Experimental Conditions (a)				
	Test 1	Test 2	Test 3	Test 4 (b)	Test 5 (c)
Ozonator Power, W	165	275	275	275	275
Oxygen Feed Gas Pressure, Psig	17	17	17	17	17
Oxygen Flow Rate, l/Min (Scfh)	9.4 (20)	9.4 (20)	9.4 (20)	6.1 (13)	6.1 (13)
Oxygen Flow Rate Correction Factor	1.353	1.353	1.353	1.353	1.353
Oxygen Flow Rate, l/Min (Scfh)	12.7	12.7	12.7	8.3	8.3
	(27.0)	(27.0)	(27.0)	(17.6)	(17.6)
Ozone Concentration, Wt. %	0.9	1.45	1.45	2.16	2.16
Ozone Dosage, mg/Min	165	265	265	257	257
UV Lights, 70W/Stage	Off	Off	On	On	On

Experimental Results (d)																			
Test No.	PC			Stage 1		Stage 2		Stage 3		Stage 4		Stage 5		Stage 6		Combined			LMTOC Conv %
	Conc Ppm	Conv %	Ppm	Conc	Conv %	Conc	Conv %	Conc	Conv %	Conc	Conv %	Conc	Conv %	Conc	Conv %	Ppm	Conv %		
1	1650	18	1935	79	1935	79	1600	82	2200	76	1100	88	1300	86	2000	78	82		
2	2200	24	3000	79	3000	79	2800	81	3300	77	1900	87	1800	88	2900	80	85		
3	1150	14	1150	92	1250	91	--	--	1500	90	--	--	--	--	1350	91	92		
4	--	--	4450	79	--	--	--	--	--	--	--	--	--	--	--	--	--		
5	--	--	--	--	--	--	--	--	4850	78	--	--	--	--	--	--	--		

(a) Temperatures were between 315 and 328K (108 and 131F)

(b) Entire flow diverted through Stage 1

(c) Entire flow diverted through Stage 4

(d) Conc = Concentration, Conv = Conversion



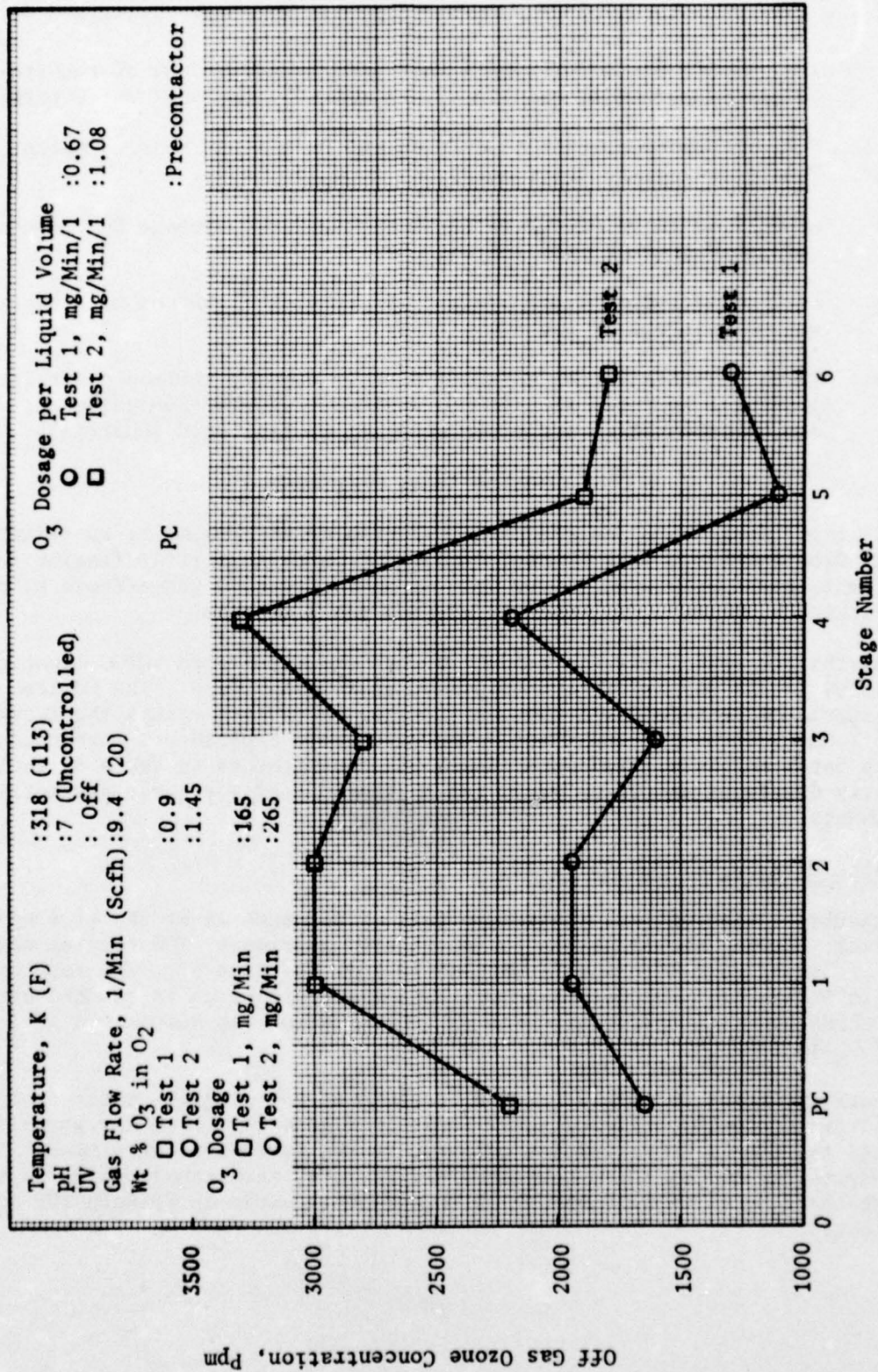


FIGURE 11 OZONE CONVERSION AT VARIOUS STAGES

observed. Higher flow rates result in larger size bubbles and lower gas residence times through the columns. The increase in  $O_3$  conversion with UV activation was expected since UV acts as an  $O_3$  dissociation catalyst.

These results support earlier findings that approximately 200% of the stoichiometric  $O_3$  dosage is needed to oxidize the organics in the MUST waste waters.

A series of  $O_3$  autodecomposition tests were run on the LSI stirred batch reactor. The following are the conclusions from the tests.

- At  $O_2$  flow rates of 4.7 l/min (9.9 scfh), the average  $O_3$  dissociation was 56% without UV and 63% with UV.
- At  $O_2$  flow rates of 1.7 l/min (3.5 scfh), the average  $O_3$  dissociation was 68% without UV and 80% with UV.
- Ozone dissociation as a function of  $O_3$  reactor stirred power is presented in Table 13. At 5.3 kW/1000 l (20 kW/1000 gal), 80% dissociation of  $O_3$  was observed under UV activated conditions.

#### MUST Composite Water Experiments

Several experiments were designed and conducted on the composite waste water product from a RO B-10 Unit Process. In addition to an initial feasibility experiment, parametric experiments were conducted to show the effects of pH, temperature,  $O_3$  dosage, UV light and carrier gas flow rate.

The feasibility experiment was conducted with the integrated LMTOC (continuous water flow in all stages) with a composite waste water feed. The RO feed in this experiment contained all the constituents in Table 8 except the X-ray (Kodak X-Omat) developer and fixer. The parametric experiments were conducted in both batch and integrated modes. All the constituents in Table 8, including the X-ray developer and fixer were used in the RO feed for these parametric experiments.

#### Composite Waste Water Feasibility

The experiment started with all stages full of RO permeate at the 11.6 mg/l TOC level. During the transient period of the experiment, TOC samples were taken at Sample Port 7 (Stage 4) and Sample Port 8 (Stage 5). The results are shown in Figure 12. The data suggest that near equilibrium is reached after about three hours of operation. The  $O_3$  concentration was maintained at 1.8 Wt% of  $O_3$  in feed  $O_2$ .

Six hours after startup the TOC concentrations of the effluent water from all stages were monitored. The data are shown in Figure 13. From the actual TOC readings the 5 mg/l TOC level was achieved in two hours residence time. Since the uncertainty of the TOC measurement was  $\pm 1$  mg/l, the actual residence time to meet the 5 mg/l TOC requirement in the effluent could be between 105 and 245 minutes.



TABLE 13 O<sub>3</sub> DISSOCIATION AS A FUNCTION OF  
O<sub>3</sub> REACTOR STIRRER POWER (SPEED)

Variable Transformer Setting	Stirrer Power, W	Stirrer Power, kW/1000 Gal	UV Activation, Residual O <sub>3</sub> Conc., Ppm		Percent O <sub>3</sub> Dissociation UV Activation
			O <sub>3</sub> Analyzer	Actual	
10, 20 <sup>(a)</sup>	0.0	0.0	>5000 <sup>(b)</sup>	5000	>62.4
30	24.0	8.63	4300	4225	68.2
40	38.0	13.66	3350	3300	75.2
50	2.5	19.87	2700	2700	79.7
60	69.0	24.80	2050	2175	83.7
70	87.5	31.50	1900	2060	84.5
80	108.0	38.80	1750	1950	85.3
90	130.5	46.90	1550	1825	86.3
100	157.5	56.60	1400	1750	86.8

● Generator Conditions

Power, W	30
Pressure, kN/m <sup>2</sup> (Psig)	100 (15)
O <sub>2</sub> Flow, l/Min (Scfh)	13.5 (3.57)
O <sub>3</sub> Conc in Feed, %	1.33
O <sub>3</sub> Dosage, mg O <sub>3</sub> /Min	29

(a) At these settings no stirrer motion was observed.

(b) The O<sub>3</sub> Analyzer reading fell outside the analyzer detection range.



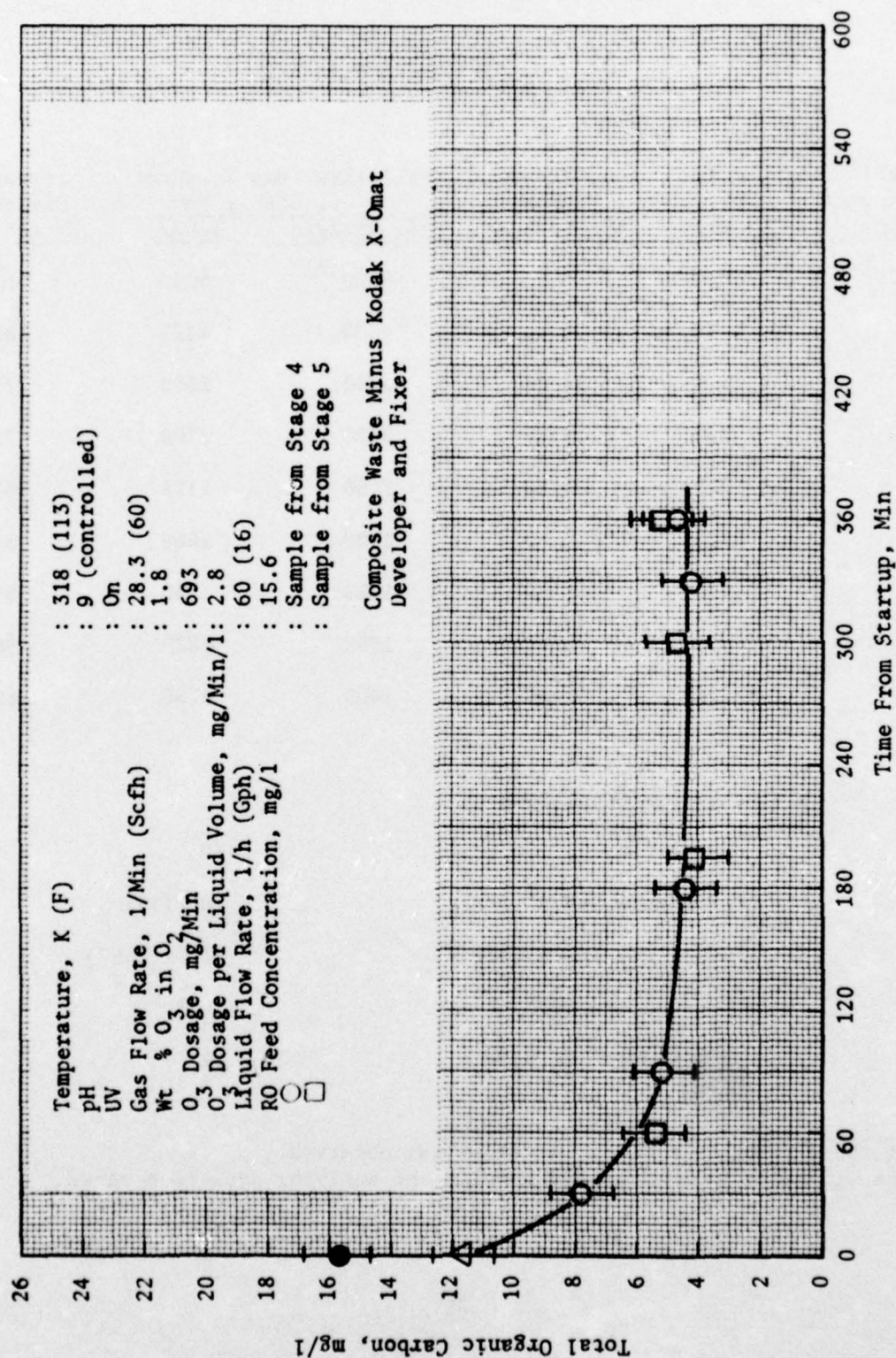


FIGURE 12 INTEGRATED COMPOSITE WASTE TRANSIENT TOC PROFILE

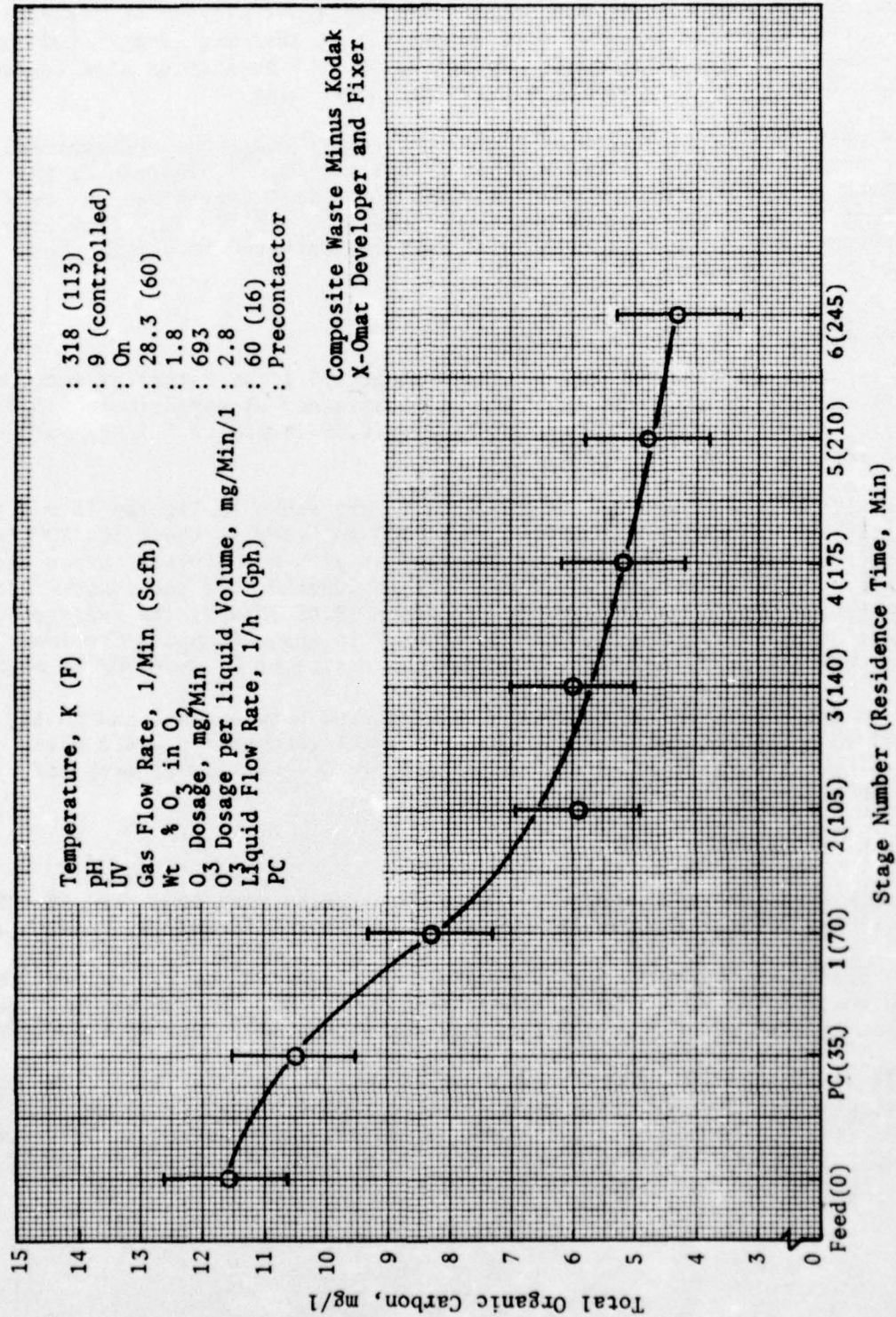


FIGURE 13 INTEGRATED COMPOSITE WASTE FEASIBILITY TEST



After the samples for the TOC profile were taken, the flow rate was reduced to 0.378 l/min (6 gph) to yield a 90 minute residence time through each stage. The TOC of the precontactor and Stage 5 effluents are plotted in Figure 14. The TOC components waste water data suggests that less than 5 mg/l TOC can be achieved in a single, mixed-flow reactor without UV activation at a temperature of 303K (86F), a pH of 9 and 90 minutes residence time.

In the past, little oxidation of highly refractory compounds (ethanol and acetic acid) has been observed without UV activation.<sup>(8)</sup> Therefore, the reduction in TOC in the precontactor under these test conditions may be a result of organic stripping at the 28.3 l/min (60 scfh) ( $O_3/O_2$ ) flow rate in the precontactor.<sup>(16)</sup> Further studies must be conducted to verify these results and inferences.

#### Effect of pH

Batch experiments were run with a single stage (35 liter batch) at a temperature of 318K (113F). The  $O_3$  concentration was maintained at approximately 3.3 Wt% in the  $O_2$  feed. An  $O_3$  dosage of 205 mg/min (0.65 lb/day or 5.9 mg/min/l of wetted reactor volume) was maintained.

The results of the pH studies at pH 7 and 11 are shown in Figures 15 and 16, respectively. Within the accuracy of the TOC analyzer at these low TOC levels no difference in TOC reduction was observed at either pH level. Since the decomposition of  $O_3$  is more rapid at pH 11, a lower  $O_3$  off gas concentration was observed. For an  $O_3$  dosage of 205 mg/min (0.65 lb/day) the average  $O_3$  effluent at pH 11 was 1250 ppm, while at pH 7 it was 2000 ppm. The lower  $O_3$  off gas concentration indicates a higher conversion of  $O_3$  under pH 11 conditions.

Since no difference in TOC reduction was observed between pH 7 and pH 11, pH control is not necessary with the LMTOC design for composite waste water processing. Figure 17 shows the comparison of TOC profiles at each stage under pH 7 and pH 11 conditions.

#### Effect of Temperature

Two batch experiments on composite waste RO permeate were conducted at 303 and 333K (86 and 140F). The results are shown in Figures 18 and 19, respectively.

The  $O_3$  dosage was maintained at 205 mg/min (0.65 lb/day, or 5.9 mg/min/l of wetted reactor volume and an  $O_3$  concentration of 3.3%. The pH was initially adjusted to a value of 9 in the feed and was uncontrolled during the experiment.

At 333K (140F) the TOC reduction was not as fast as at 303K (86F). The  $O_3$  concentration in the off gases under identical conditions was lower (1200 ppm) at 333K (140F). Consequently, a higher  $O_3$  demand was observed at 303K (140F). The TOC was reduced to below 5 mg/l after 90 minutes of operation at 333K (140F) compared to 50 minutes at 303K (86F).



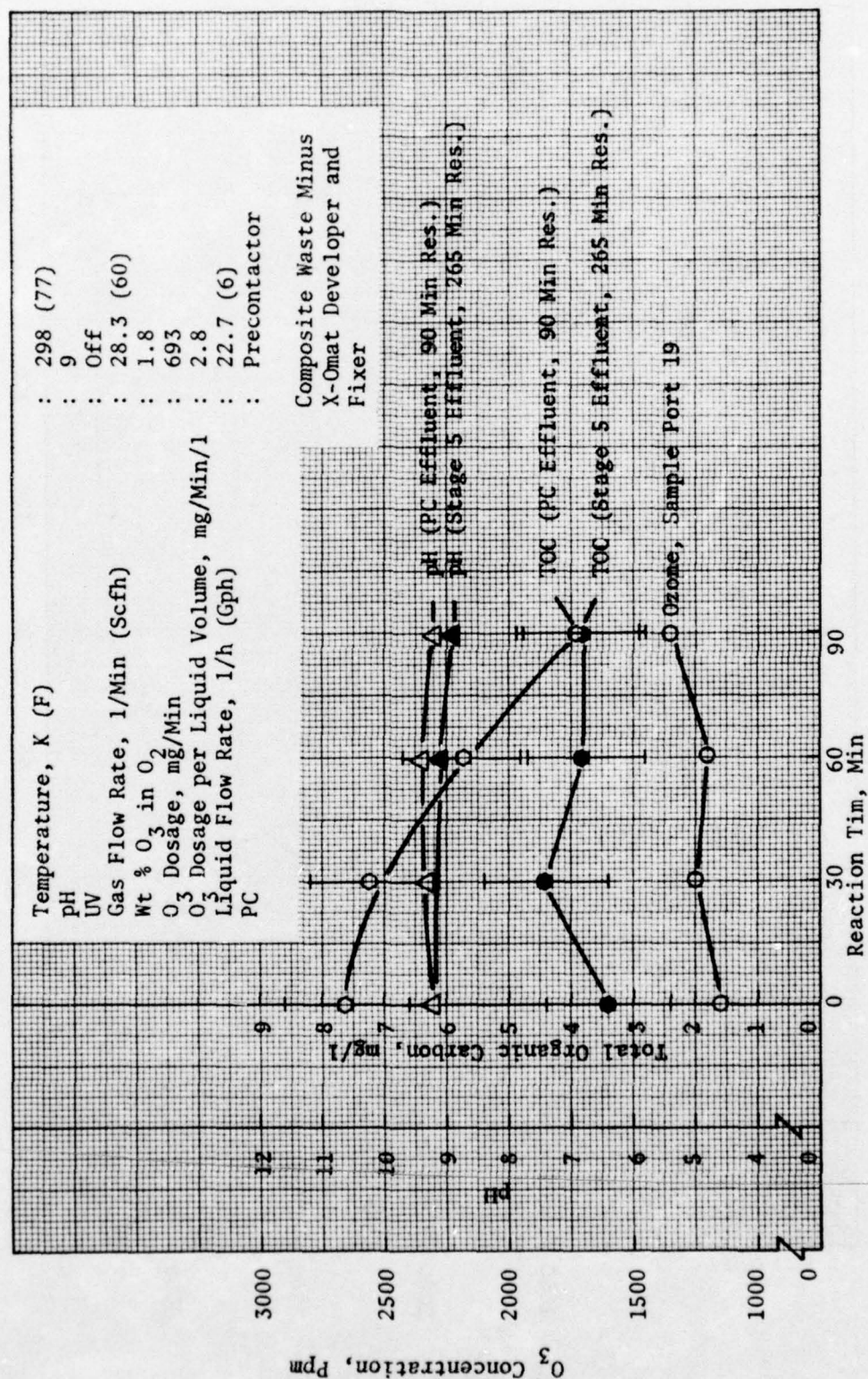


FIGURE 14 INTEGRATED COMPOSITE WASTE 6 GPH FLOW STUDY

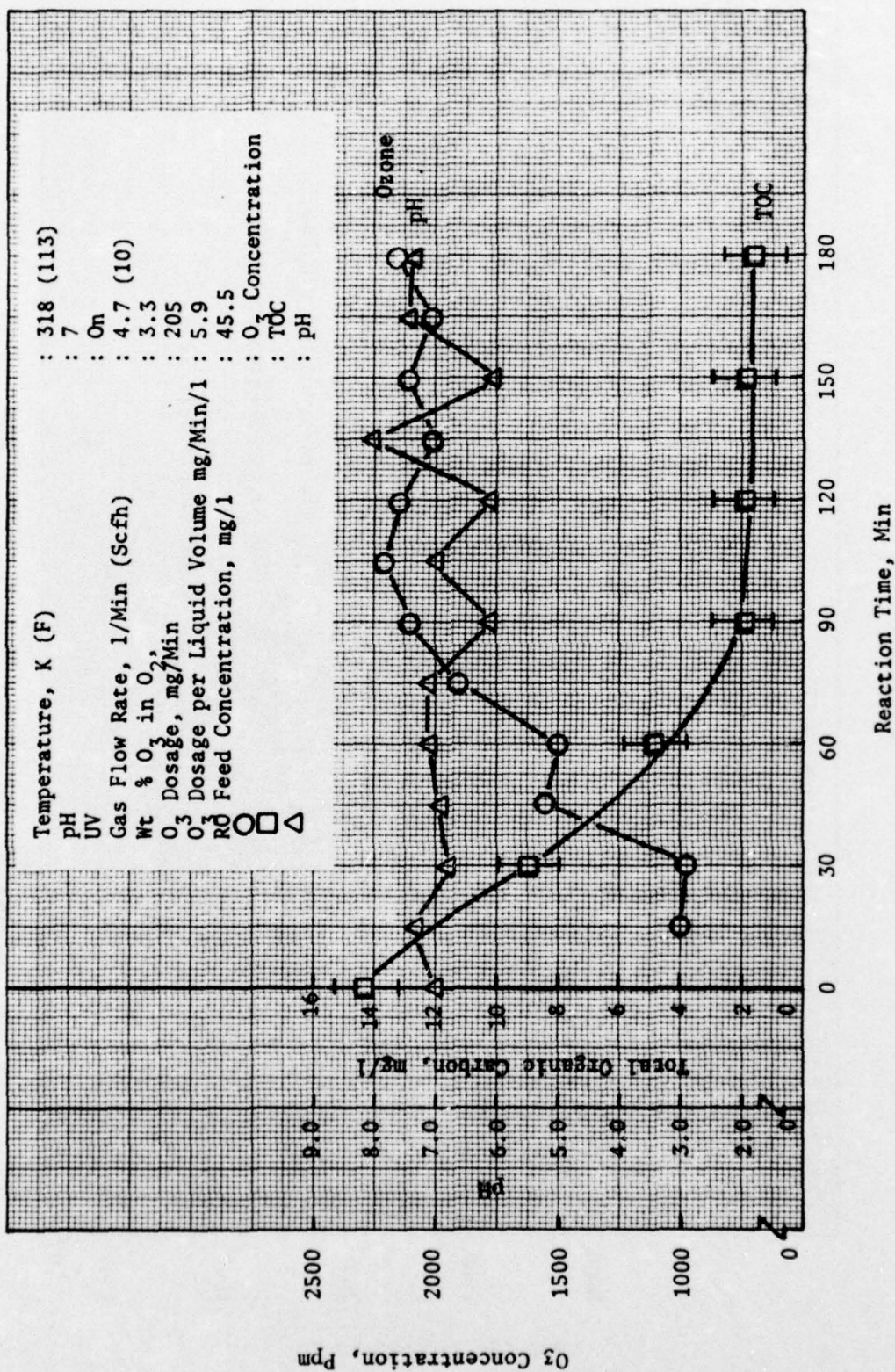


FIGURE 15 BATCH OXIDATION OF COMPOSITE WASTE, pH EFFECT (7)



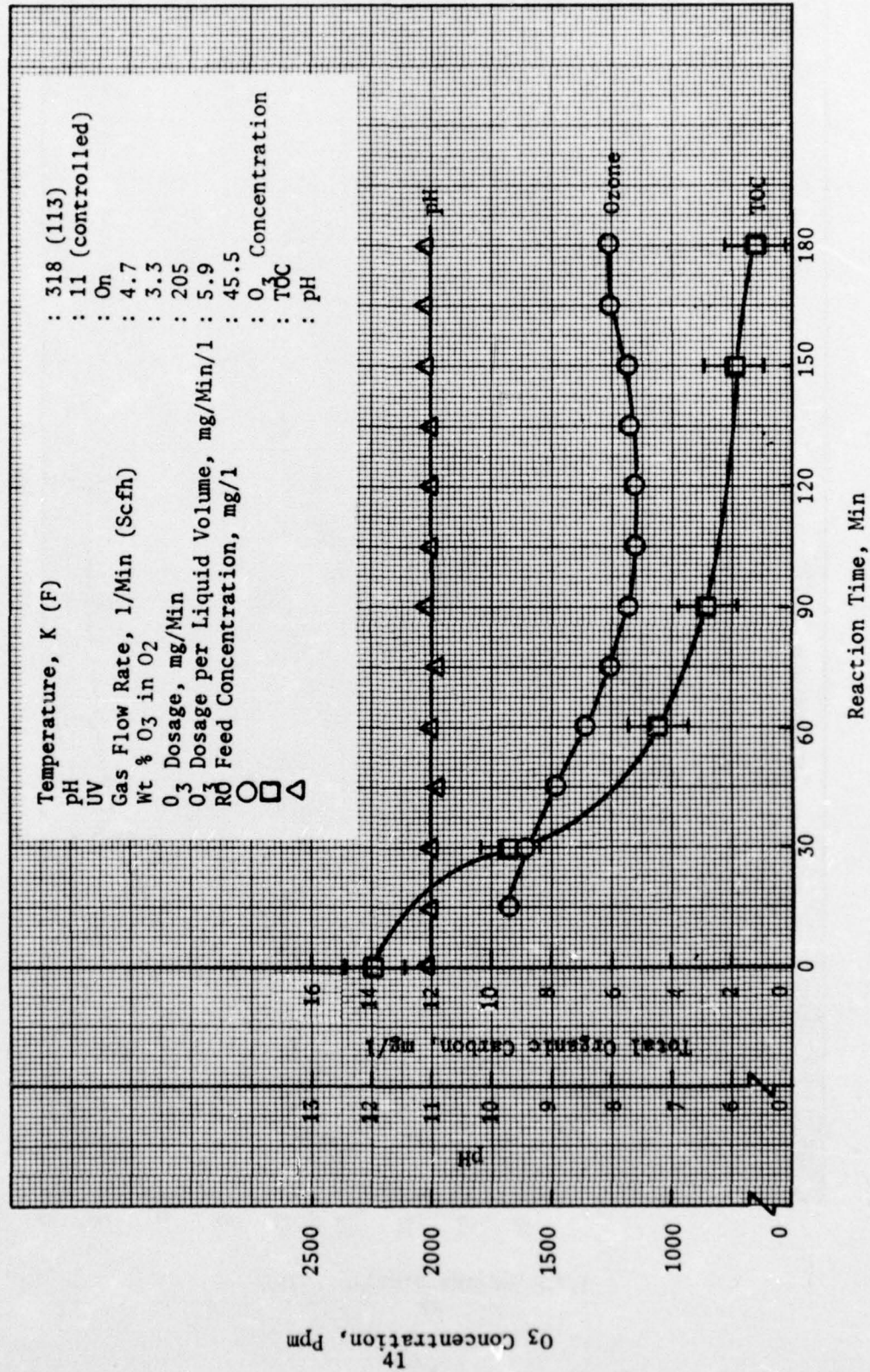


FIGURE 16 BATCH OXIDATION OF COMPOSITE WASTE, pH EFFECT (11)



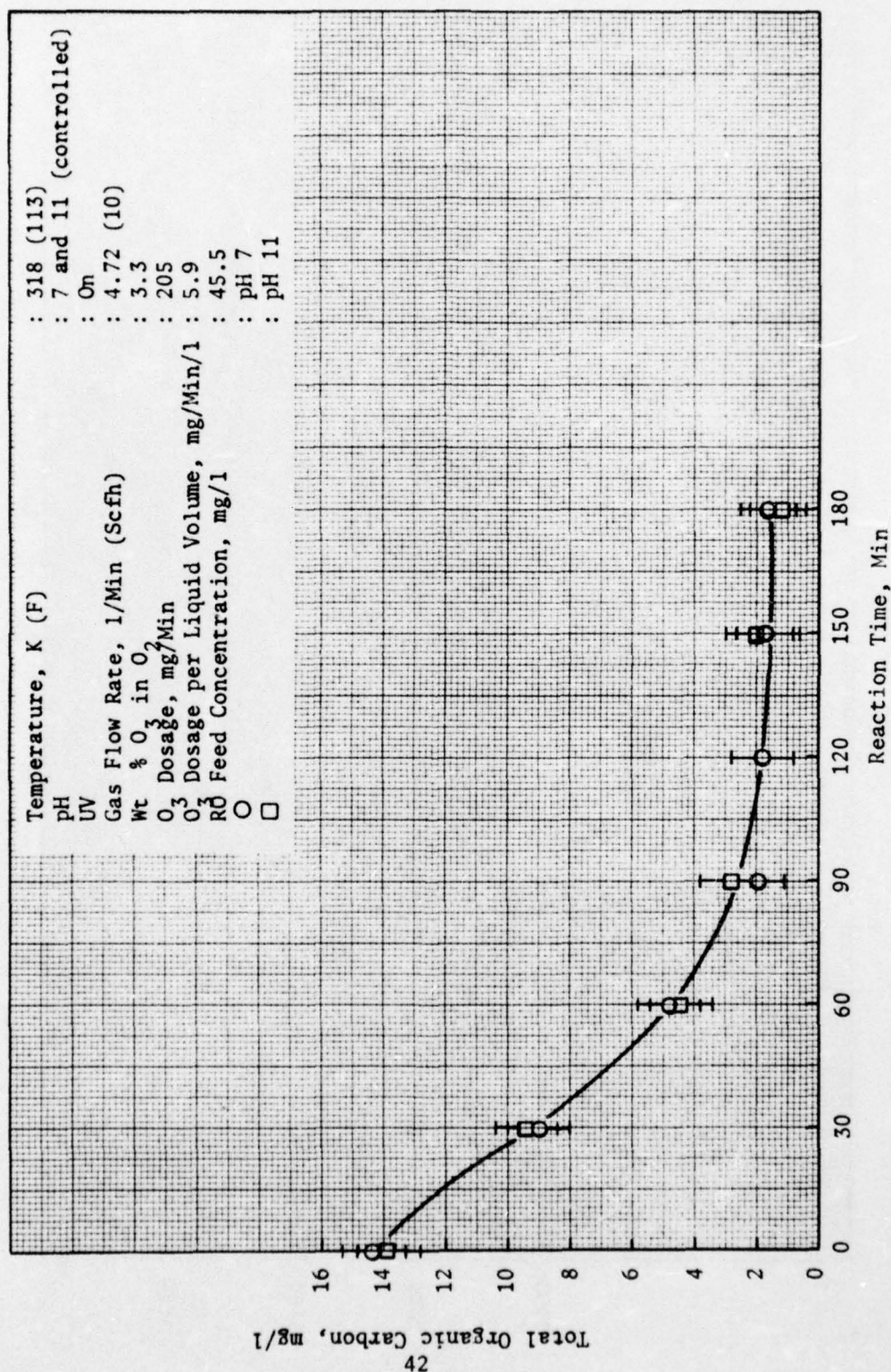


FIGURE 17 BATCH OXIDATION OF COMPOSITE WASTE, pH EFFECT

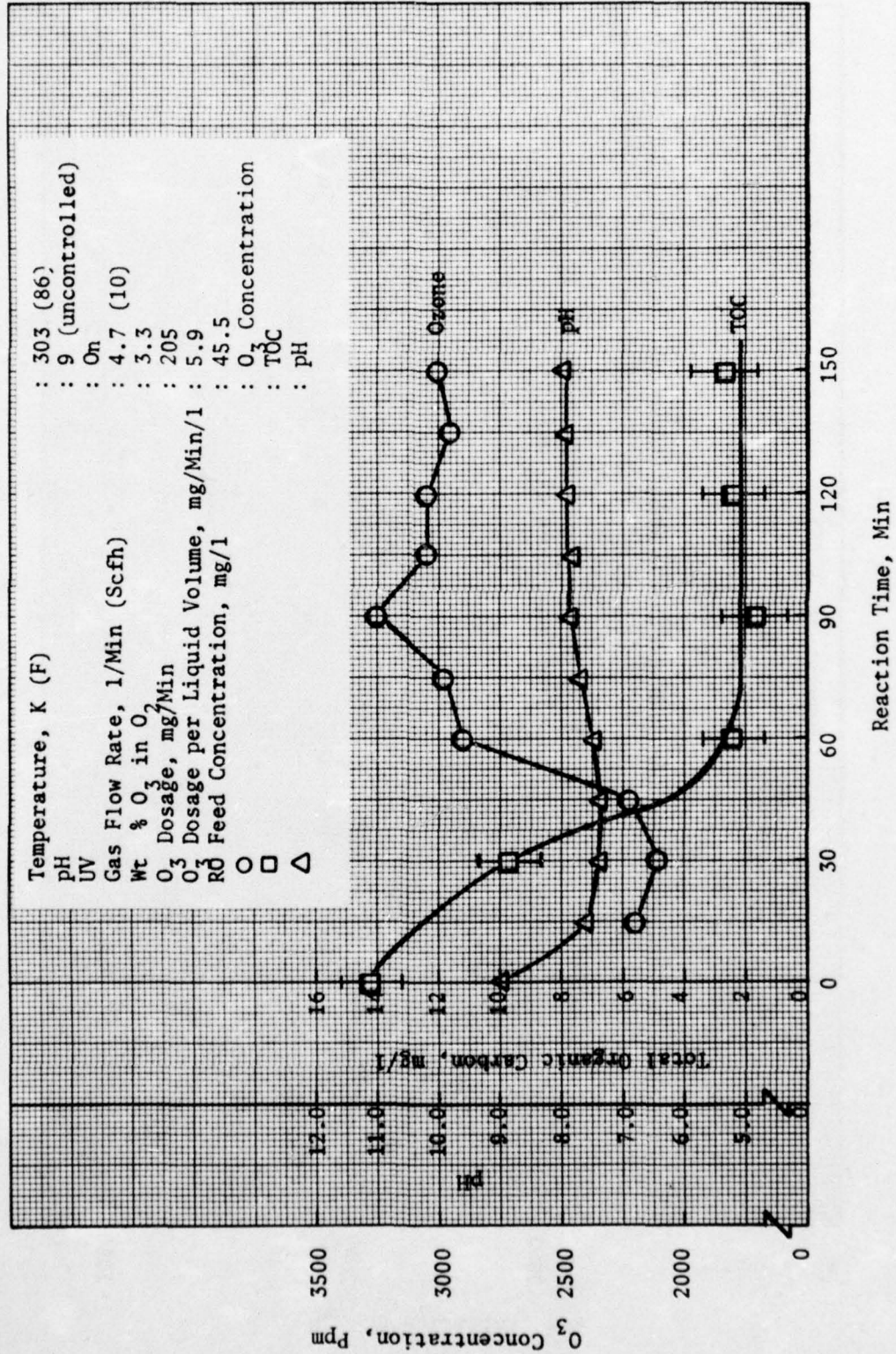


FIGURE 18 BATCH OXIDATION OF COMPOSITE WASTE, TEMPERATURE EFFECT (303K)



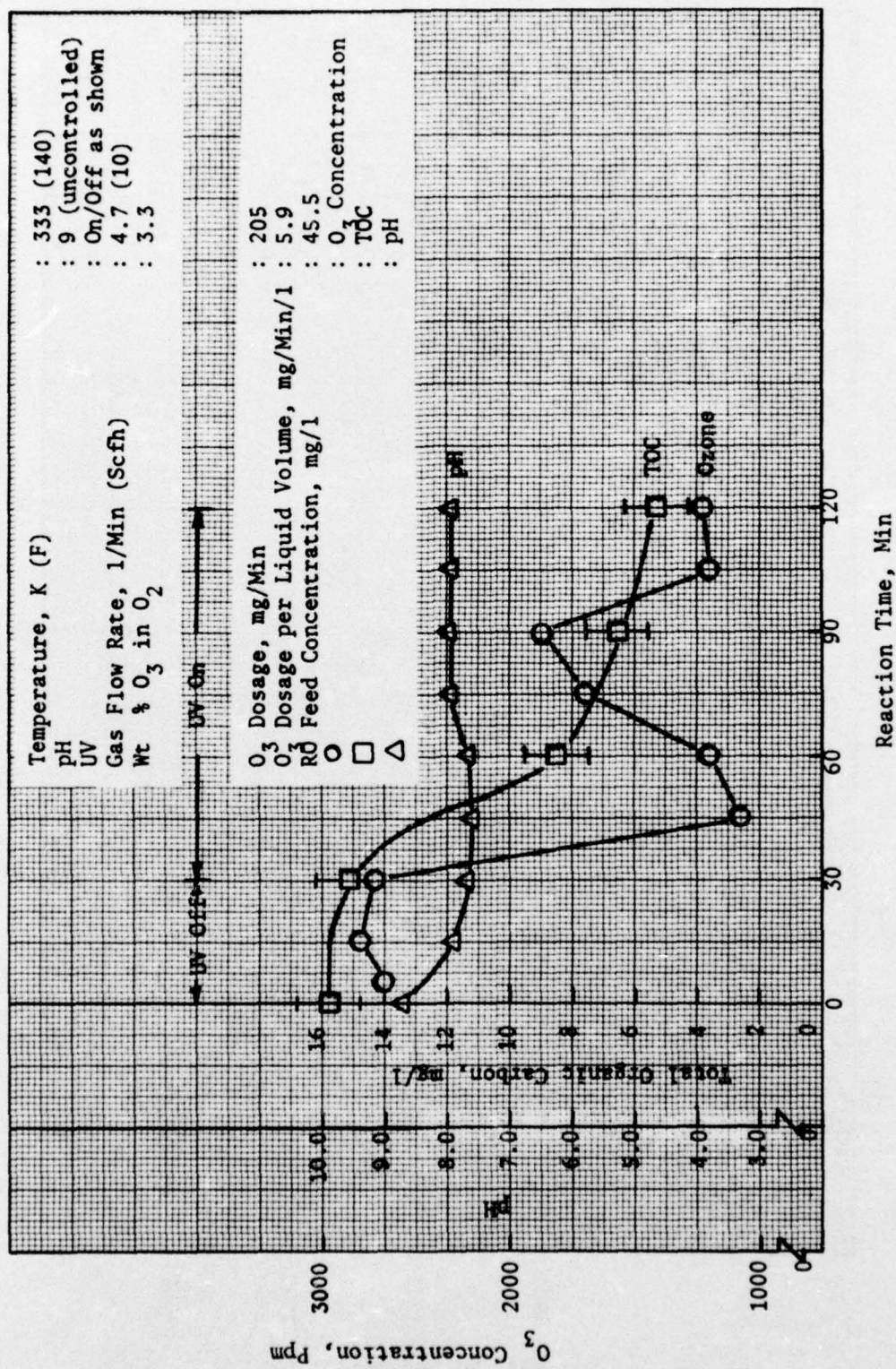


FIGURE 19 BATCH OXIDATION OF COMPOSITE WASTE, TEMPERATURE EFFECT (333K)



These temperature experiments indicate that 303K (86F), the expected temperature of the RO permeate, is adequate for the  $O_3$  oxidation of the MUST composite waste with the LMTOC. Therefore, these conditions should result in minimum power and system complexity.

During the 333K (140F) experiment the UV lamps were turned off for the first 30 minutes of the experiment. Only a small reduction in TOC concentration was observed during this period. This emphasizes the importance of UV activation in reducing the organics in the MUST composite waste at 333K (140F).

#### Effect of Ozone Dosage

A composite waste integrated experiment was conducted at a 664 mg/min (2.7 mg/min/l of wetted reactor volume dosage. The  $O_3$  concentration was maintained at 1.76% in the  $O_2$  feed. The conditions of the experiment and the results are shown in Figure 20. The steady-state effluent pH and  $O_3$  concentrations at the six stages are also plotted in this figure.

Under 664 mg/min (2.7 mg/min/l of wetted reactor volume)  $O_3$  dosage conditions the TOC of the composite waste water was reduced to below 5 mg/l in approximately a 70-minute residence time (at the end of Stage 1). These results compared well with the batch test results at near the same pH and temperature conditions shown in Figure 18.

The average  $O_3$  concentration in the off gases was observed at approximately 1500 ppm. The average  $O_3$  concentration in the effluent from the precontactor was 600 ppm. This constituted a 97%  $O_3$  conversion at an  $O_3$  dosage of 2.7 mg/min/l of wetted reactor volume.

The effluent from Stage 3 (see Figure 20) is well below the TOC specifications of 5 mg/l. Hence, the  $O_3$  feed to the last three stages is unnecessary. In general, the results indicate that the composite waste water can be easily reduced to below 5 mg/l TOC in a one-hour residence time at an  $O_3$  dosage of 2.7 mg/min/l of wetted reactor volume without pH and temperature control. A two-hour residence time (three stages) will give a safety factor to ensure the WPE effluent water TOC is below 5 mg/l.

Figure 21 shows the results of another experiment at a lower  $O_3$  dosage of 534 mg/min (2.2 mg/min/l of wetted reactor volume), 1.42 Wt%  $O_3$  in  $O_2$  and a slightly higher temperature of 308K (95F). The residence time to reduce TOC to below 5 mg/l was much longer than the previous one shown in Figure 20. In this experiment, 158 minutes residence time was required to meet the 5 mg/l TOC level compared to the previous 90 minutes. However, the limiting factor to meet the water quality specifications was the time required to reduce COD. As shown in the figure, about 245 minutes residence time was needed to reduce the COD to below 10 mg/l at the 534 mg/min  $O_3$  dosage.

The parametric testing on the LMTOC indicates that for the MUST composite waste water RO permeate, the most effective parameter levels are an  $O_3$  dosage

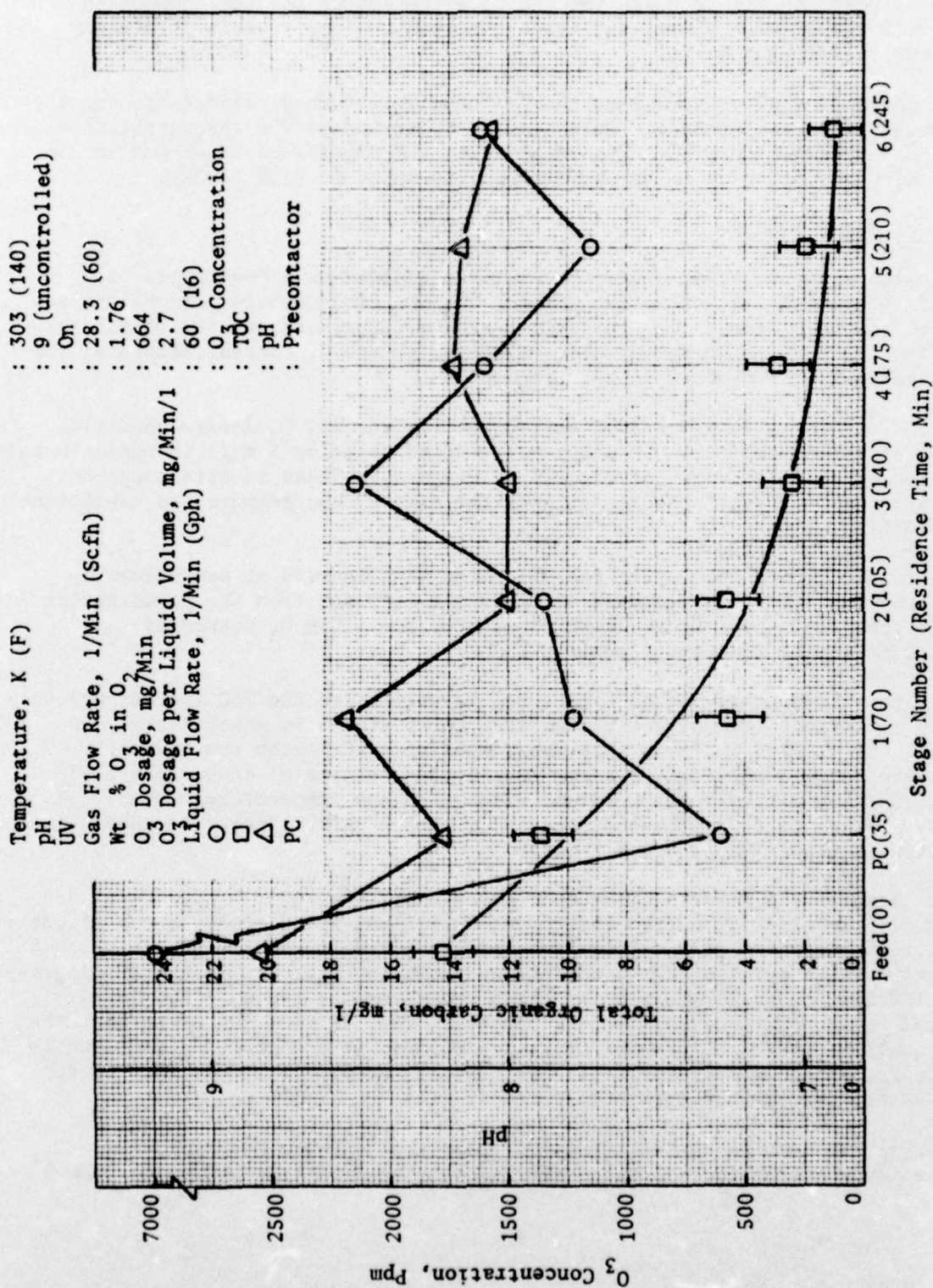


FIGURE 20 INTEGRATED COMPOSITE WASTE TEST AT BEST CONDITIONS



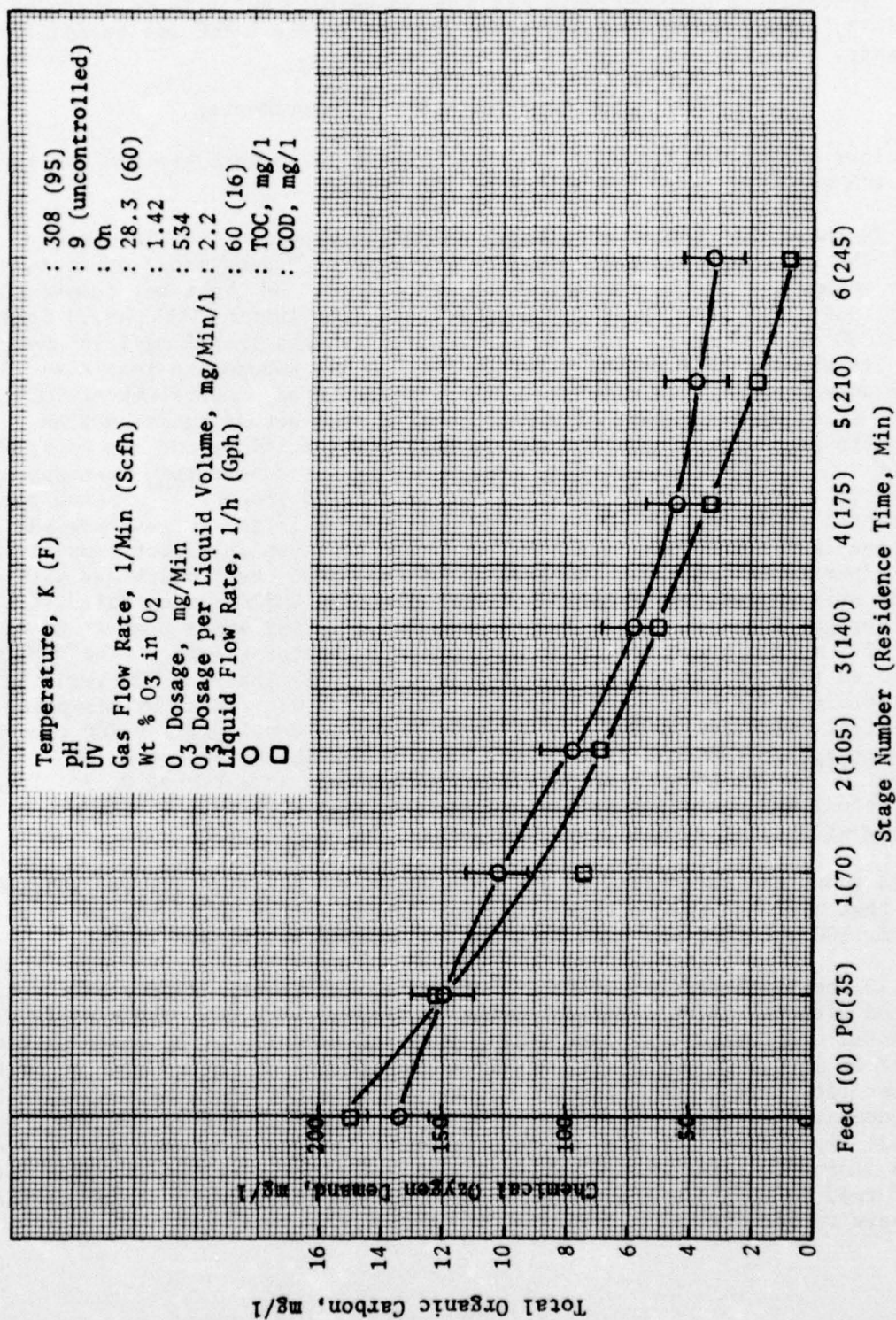


FIGURE 21 INTEGRATED OXIDATION OF COMPOSITE WASTE, STAGE TOC PROFILE



of 2.7 mg/min/l of wetted reactor volume, 303K (86F) feed temperature and pH 9 feed. Typically, the RO permeate has a pH value of 9 and a temperature of 303K (86F). Thus, no pH or temperature control in the LMTOC are needed for this waste.

#### MUST Laboratory Waste Water Experiments

Experiments with synthetic MUST laboratory waste water were carried out in both batch and integrated operations of the LMTOC.

Figure 22 shows the results of a batch oxidation experiment of laboratory waste. The  $O_3$  dosage was maintained at 277 mg/min (7.9 mg/min/l of wetted reactor volume). The  $O_3$  concentration was 4.4 Wt%, UV light on, temperature at 308K (95F), and feed pH at 9, uncontrolled. The initial TOC was 94 mg/l and the COD was 380 mg/l. The TOC was reduced to less than 5 mg/l in approximately 215 minutes of reaction time and the COD was reduced to less than 10 mg/l in approximately 230 minutes. Since the expected uncertainty of TOC readings is  $\pm 1$  mg/l and that of COD is  $\pm 3$  mg/l, the actual reaction time required to reach the 5 mg/l TOC and 10 mg/l COD specifications may be in the range of 225 to 235 minutes. This reaction time was longer than what was observed in a previous study with a 14-liter stirred reactor.<sup>(5)</sup> Since the experimental conditions were considerably different in the current program from those in the previous research, care must be taken in direct comparison of the experimental results. Comparison of the LMTOC test conditions with the previous stirred reactor research<sup>(5)</sup> shows that the LMTOC was operated at a much lower gas flow rate (4.72 l/min versus 23.6 l/min) and a much lower  $O_3$  dosage (7.9 mg/min versus 64.55 mg/min per unit reactor volume). The VVM in the stirred reactor experiment was high enough (2.35) that the TOC reduction result obtained was probably a combination of physical removal by stripping and chemical oxidation by  $O_3$ . The kinetic equation developed for TOC removal under such a high VVM condition cannot be used for the design of an  $O_3$  contactor.<sup>(16)</sup> The LMTOC experiments were conducted at a VVM of 0.13<sup>3</sup> (closer to the actual WPE operating level). Chian has indicated that little or no stripping will occur at the lower VVM level.<sup>(16,17)</sup>

Table 14 shows the comparison of the LMTOC with the 14-liter stirred reactor.<sup>(5)</sup> Notice that although the reaction time was longer in the LMTOC the ratio of  $O_3$  dosage/mg TOC oxidized was much less in the LMTOC (19.2 versus 78.60).

Figure 23 shows the results of three integrated laboratory waste experiments conducted with the LMTOC. The following conditions were kept the same in all three cases: temperature at 308K (95F), pH 9 (uncontrolled),  $O_3$  concentration 2 Wt% of  $O_2$  in  $O_2$ ,  $O_3$  dosage 768 mg/min (3.1 mg/min/l of wetted reactor volume) and water flow rate of 26.5 l/hour. In the first experiment the feed gas was maintained at the typical 28.3 l/min (60 scfh) flow rate and the UV lamps were on in all six stages. In the second experiment the gas flow rate was increased to 37.8 l/min (80 scfh) with UV on. In the last experiment the gas flow rate was returned to 28.3 l/min but the UV lamps in the first three stages of the LMTOC were turned off.

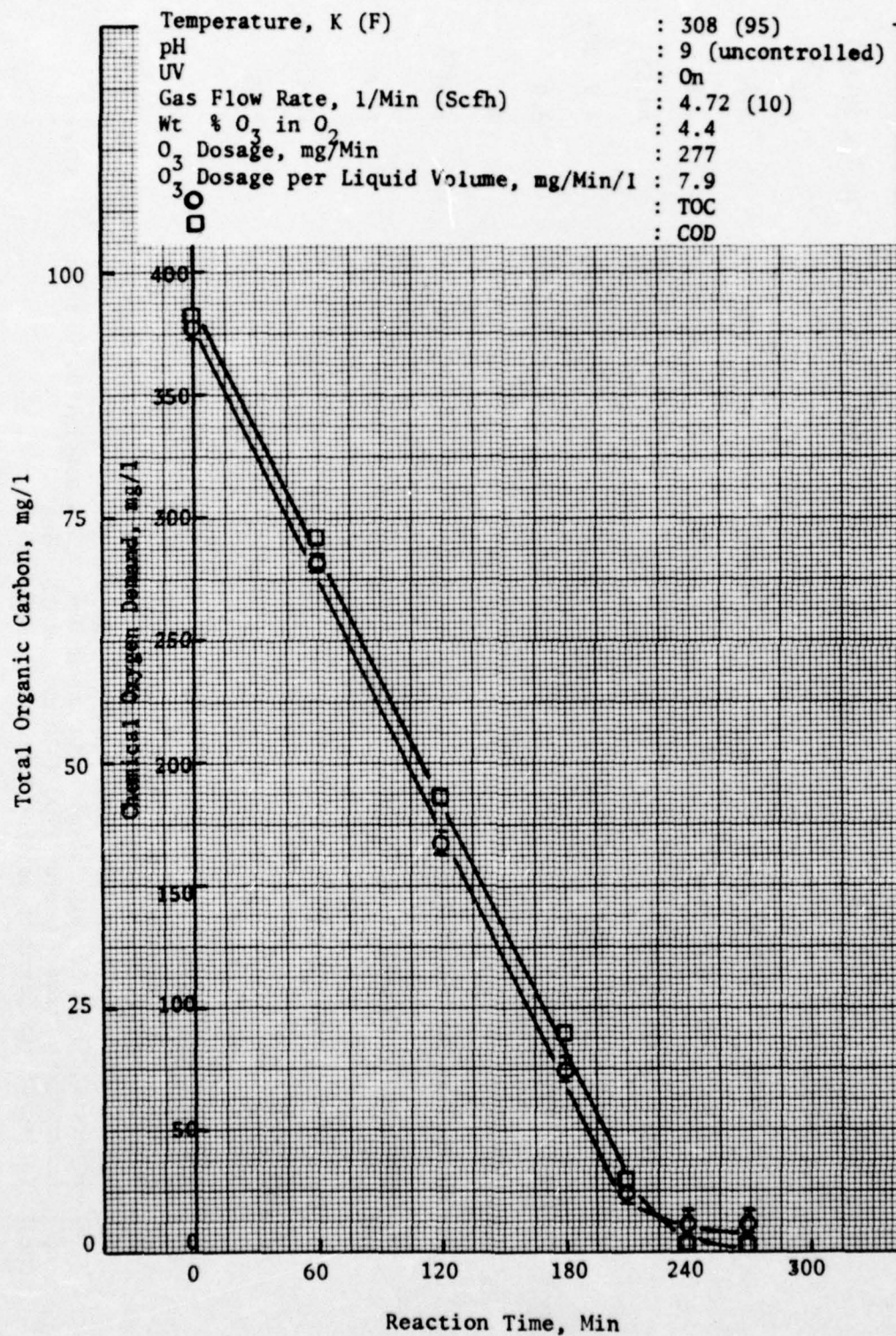


FIGURE 22 BATCH OXIDATION OF LABORATORY WASTE



TABLE 14 COMPARISON OF BATCH O<sub>3</sub> OXIDATION OF LABORATORY RO PERMEATES

Parameter	Life Systems LMTOC	Stirred Reactor
Reactor Size	35 liters <sup>(a)</sup>	10 <sup>(b)</sup>
Dimensions (ID x H), cm, In	15.7 x 213 (6.2 x 84)	25.4 x 38 (10 x 15)
Initial TOC, mg/l	94 <sup>(c)</sup>	120 <sup>(d)</sup>
Total UV Power, W/l Water	2.02	3.50
Effective UV Power, W/l Water <sup>(e)</sup>	1.52	3.50
O <sub>3</sub> Dosage/l Water, mg/Min/l	7.9	64.55
Stirring Power, kW/m <sup>3</sup> (Hp/1000 Gal)	--	3.9 (20)
O <sub>3</sub> Concentration, Wt %	4.4	2.0
Reaction Time for T <sub>1/2</sub> , Min	108	70
Residence Time for TOC <5 Ppm, Min	215	140 <sup>(f)</sup>
O <sub>3</sub> Dosage/mg TOC Oxidized, mg O <sub>3</sub> / mg Oxidized	19.2	78.60
O <sub>2</sub> Flow Rate, l/Min (Scfh)	4.7 (10)	23.6 (50)
VVM <sup>(g)</sup>	0.13	2.35

(a) Single stage (Batch)

(b) A 14 liter New Brunswick stirred reactor

(c) Synthetic RO permeate at 308K (95F) and pH 9 (initial)

(d) Actual RO permeate at 303K (86F) and pH 7 (initial)

(e) Effective UV power only 75% of input power due to 25% absorption in UV quartz sleeve

(f) Extrapolated from TOC plot (TOC = 20 mg/l at 120 Min)

(g) Volume of gas per unit volume of liquid per minute



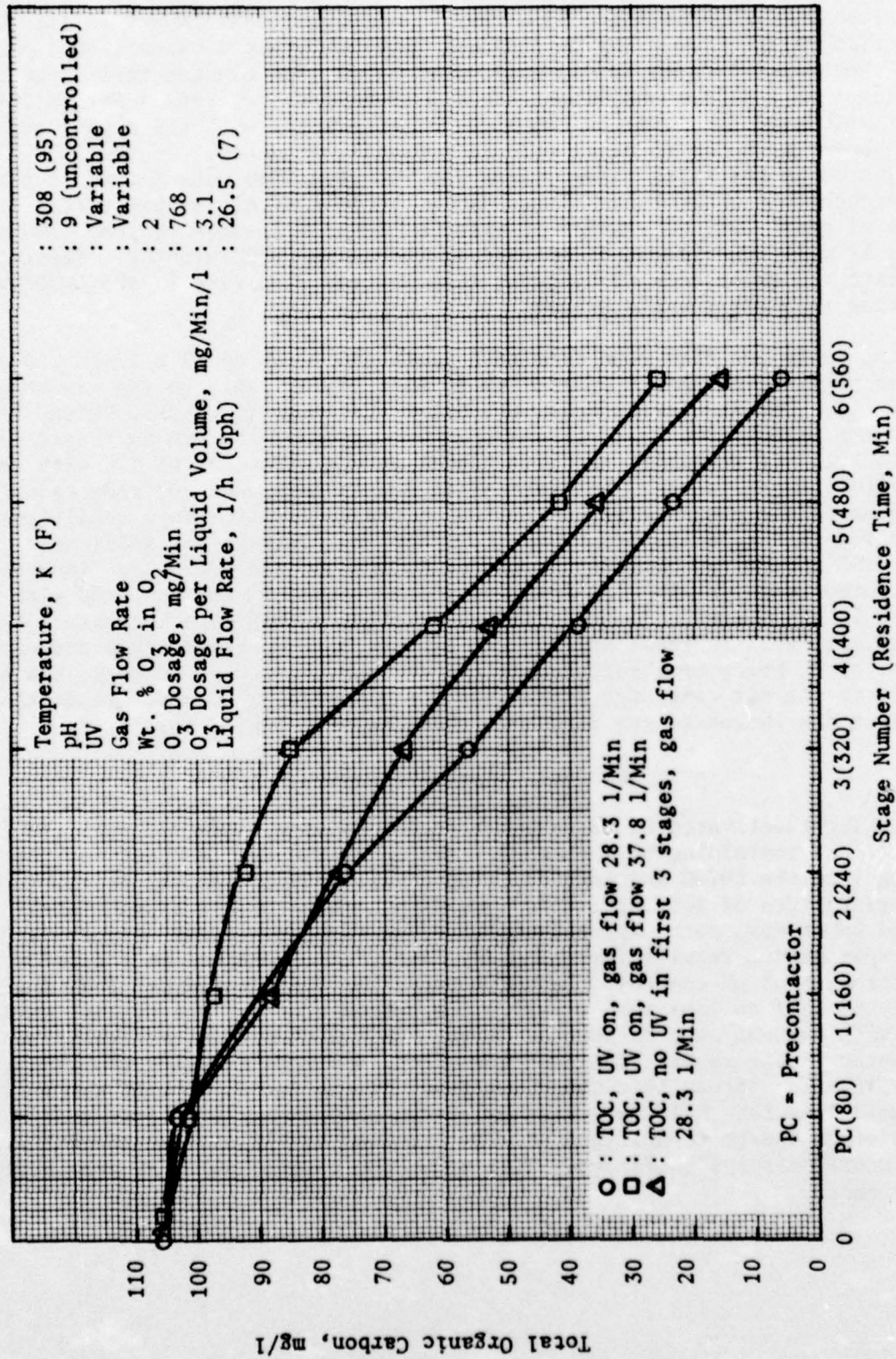


FIGURE 23 INTEGRATED LABORATORY WASTE, STAGE TOC PROFILE

The results indicate that with UV activation in all six stages and a gas flow rate of 28.3 l/min, the LMTOC has the best TOC reduction rate in the three experiments. The higher gas flow rate has a significant impact on the TOC reduction rate. Processing an influent laboratory waste water with 106 mg/l TOC, the effluent water TOC from the last stage (560 minutes residence time) reached 6 mg/l in the experiment of 28.3 l/min gas flow rate. For an influent water with same TOC level of 106 mg/l the experiment with the higher gas flow rate showed a TOC level of 26 mg/l in the effluent water. In the experiment with no UV in the first three stages the TOC reduction rate showed little difference in the first two stages and a significant difference at the third stage of the LMTOC. The effluent water of Stage 3 (residence time 320 minutes) had a 67 mg/l TOC without UV instead of the 56 mg/l TOC with UV. These findings indicate the importance of UV activation and gas flow rate in the LMTOC when treating laboratory waste waters.

The increased gas flow rate from 28.3 l/min (60 scfh) to 37.8 l/min (80 scfh) had no significant impact on the VVM (0.13 to 0.18) ratio in the six stages of the LMTOC. The VVM levels were well below the level (1.0) that Chian showed stripping effects would occur. In the precontactor, the VVM increased from 0.8 to 1.1. If stripping had some effect on the reduction of TOC with the laboratory waste it would be expected to see an increased TOC reduction in the effluent of the precontactor under the increased gas flow rate conditions. As indicated in Figure 23, this does occur but it is almost insignificant. The lower TOC reductions observed in the six stages at the increased flow rate are attributed to larger bubble diameters and decreased  $O_3$  contact time with the liquid in the stages. In other words, as the gas flow rate increases, the bubble size will increase and the gas contact time decrease, thus reducing the amount of  $O_3$  being transferred into the aqueous phase. In summary, the stripping effect at the six contactor stages was not expected or observed since the VVM level at the increased gas flow rate remained very low (0.18).

#### Ethanol Comparison Experiment

The UV light-activated  $O_3$  oxidation batch tests were conducted with simulated waste water containing only ethanol. The objective was to check out the performance of the LMTOC and compare it with experimental results available with different types of reactors.<sup>(8,18)</sup> Conditions similar but appropriately scaled up or down were employed in the first of these tests. Figure 24 presents the experimental results for the  $O_3$ /UV oxidation of ethanol in the LMTOC reactor without pH control. In three hours the TOC was reduced from approximately 58 mg/l to less than 5 mg/l. Comparison plots of the ethanol oxidation data with the LSI stirred reactor, LMTOC and two other reactor types are presented in Figure 25. The test parameters and results are summarized in Table 15. To achieve less than 5 mg/l TOC in the final effluent the LMTOC consumed less than half the total energy to reduce each milligram of TOC. The ratio of  $O_3$  dosage to milligram of TOC oxidized was 12.8:1 for the LMTOC. This represents a significantly higher  $O_3$  conversion (50%) than previously experienced.



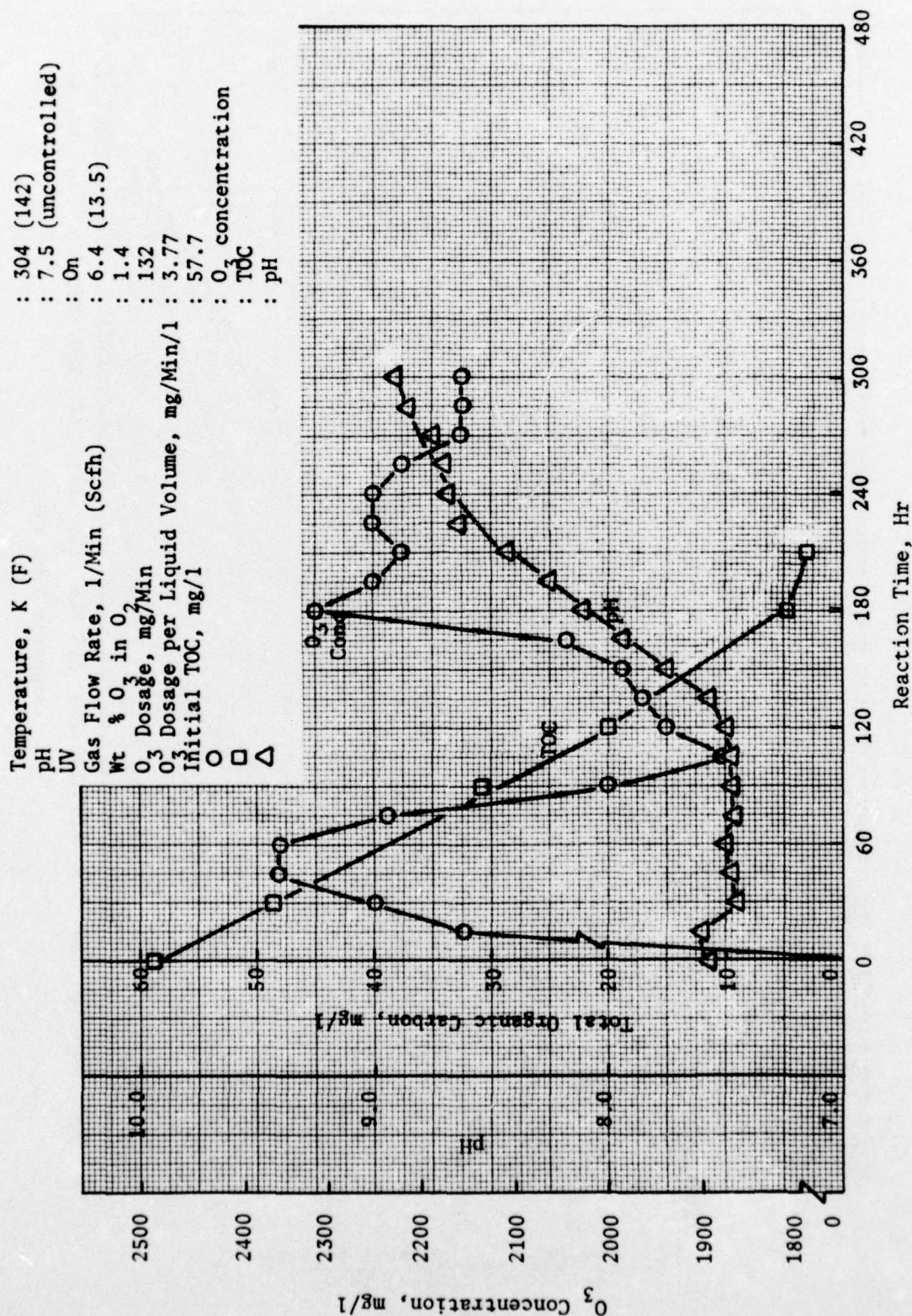


FIGURE 24 BATCH O<sub>3</sub>/UV OXIDATION OF ETHANOL



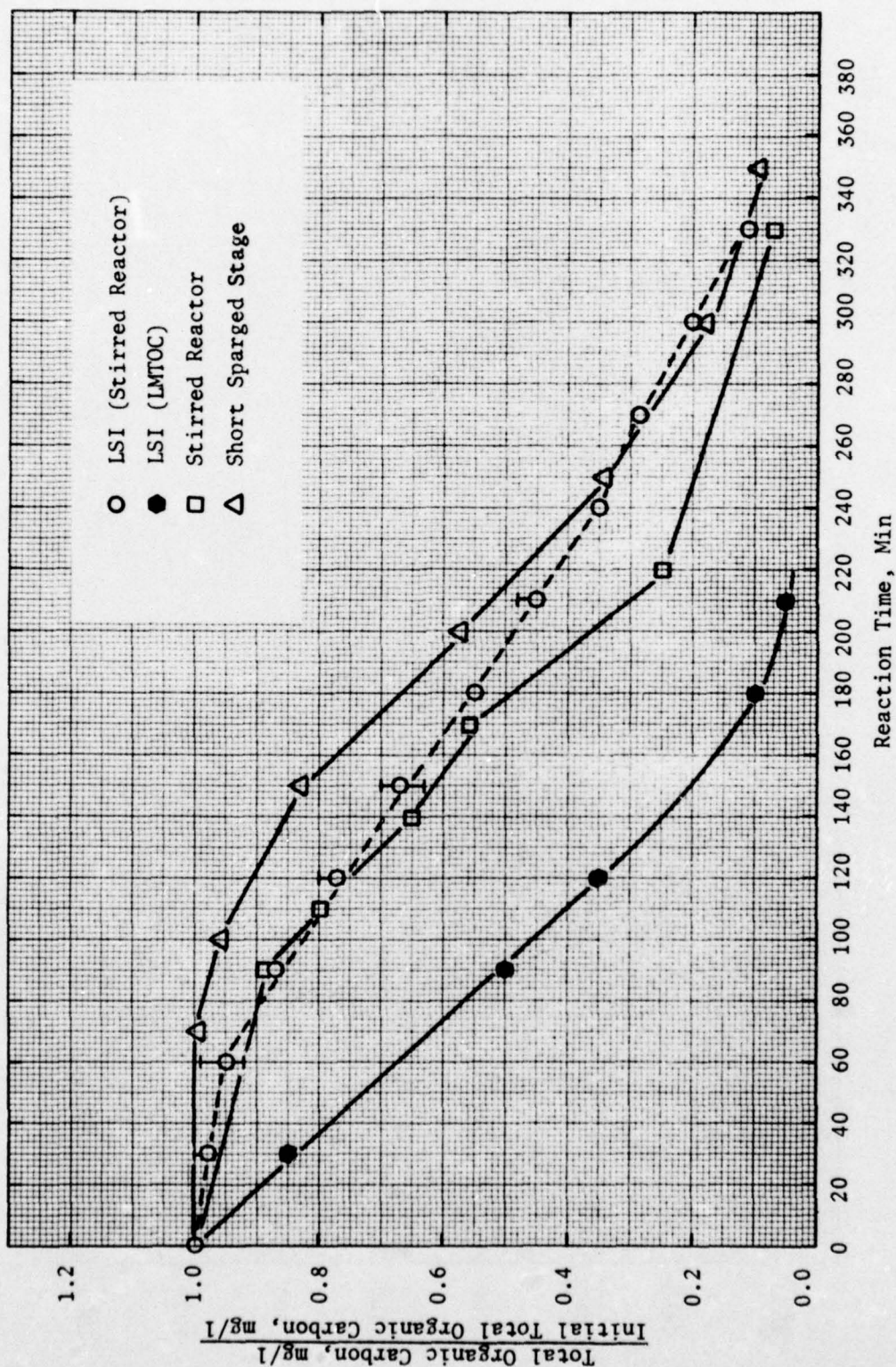


FIGURE 25 COMPARISON OF  $O_3/UV$  OXIDATION OF ETHANOL BY VARIOUS REACTOR TYPES

TABLE 15 COMPARISON OF BATCH TEST RESULTS OF ETHANOL OXIDATION

Parameter	LMTOC	Short Sparged Column (18)	Stirred Reactor (8)
Reactor Volume, l	35 (a)	12	10
Reactor Size, ID x H, cm (Ft)	15.2 x 213 (0.5 x 7)	15.2 x 91.4 (0.5 x 3)	30.5 x 30.5 (1 x 1)
Initial TOC, mg/l	57.7	65	67
Total UV Power, W/l Water	2.02	3.58	1.50
Effective UV Power, W/l Water (b)	1.52	2.69	1.50
O <sub>3</sub> Dosage/l Water, mg/Min/l	3.77	3.95	4.50
Stirring Power, kW/m <sup>3</sup> (Hp/1000 Gal)	--	--	20
O <sub>3</sub> Concentration, Wt%	1.4	2.6	3.0
Reaction Time for T <sub>1/2</sub> , Min	92	214	175
Residence Time for TOC < 5 Ppm, Min	180	345	320
O <sub>3</sub> Dosage/mg TOC Oxidized, mg O <sub>3</sub> /mg TOC Oxidized	12.8	22.0	22.8
O <sub>2</sub> Flow Rate, l/Min (Scfh)	6.4 (13.50)	1.3 (2.75)	1.0 (2.20)
Water Temperature, K (F)	304 (88)	303 (86)	303 (86)
Total Energy/mg TOC Oxidized, W-h/mg TOC Oxidized	0.398	0.828	0.97

- (a) Single stage (Batch)  
 (b) Effective UV power only 75% of input power due to 25% absorption in UV quartz sleeve  
 (c) Total Energy = Stirring Energy + UV Energy + O<sub>3</sub> Generation Energy  
 (d) O<sub>3</sub> Generation Energy = 10 kW Hr/Lb O<sub>3</sub>



A second LMTOC ethanol batch experiment was conducted at a lower initial TOC concentration. Except for the initial TOC all the test conditions were kept very close to the previous ones. The TOC was reduced to 5 mg/l in approximately two hours. The results are shown in Figure 26. In comparing Figure 26 with Figure 24 one can see very little difference in the pH and  $O_3$  concentration curves. However, the TOC versus time curves are significantly different. In the experiment with higher initial TOC at 60 mg/l, about 120 minutes reaction time was required to reduce the TOC to 20 mg/l and only 60 minutes from 20 mg/l to 5 mg/l. In the second experiment a reaction time of 120 minutes was needed to reduce the initial TOC of 20 mg/l to 5 mg/l. This comparison illustrates the drastic difference in the results of the batch  $O_3$  oxidation experiments with different initial TOC concentrations.

#### UV Intensity Experiments

The UV light intensity measurements were made as a function of distance from the light source in synthetic laboratory RO permeate waste waters. The test apparatus is shown in Figure 27.

A Blak-Ray shortwave UV meter (Model J-225) was used in the experiment. Ranges on the UV meter J-225 are 0 to 2400  $\mu W/cm^2$  (A scale) and 2000 to 12,000  $\mu W/cm^2$  (B scale). The meter is designed for measuring energy from wavelength 230 to 270 nm (peak sensitivity about 250 nm).

Figure 28 shows the curve of UV intensity versus distance from the lamp. The results indicate that a 95% reduction of UV intensity was observed in about 13 cm (5 in) of synthetic laboratory waste water RO permeate. This suggests that for effective utilization of UV the process water must be kept very close to the lamp.

Actual  $O_3$  oxidation experiments will have to be conducted at variable lamp spacings and/or lamp intensities to determine the UV spacing and intensity effects on TOC reduction.

#### Post-Experimental Analysis of the LMTOC Components

Post-experimental analyses were conducted on the LMTOC at two different times in the course of the experimental activities. The first analysis was conducted after the completion of four experiments (the  $O_3$  autodecomposition experiments, ethanol batch experiment, laboratory integrated waste experiment and a laboratory batch waste experiment) and the second analysis after completion of the entire experimental program (18 additional composite and laboratory waste water experiments). The initial four experiments were conducted using raw well water as the base for the synthetic wastes. The well water contained an approximate hardness of 340 ppm. The remaining experiments were conducted with well water which was first processed by the RO unit process (DuPont B-10 membranes).

The post-experimental analysis after the initial four experiments was conducted when one stage (No. 5) developed a leak at the UV lamp quartz and contactor



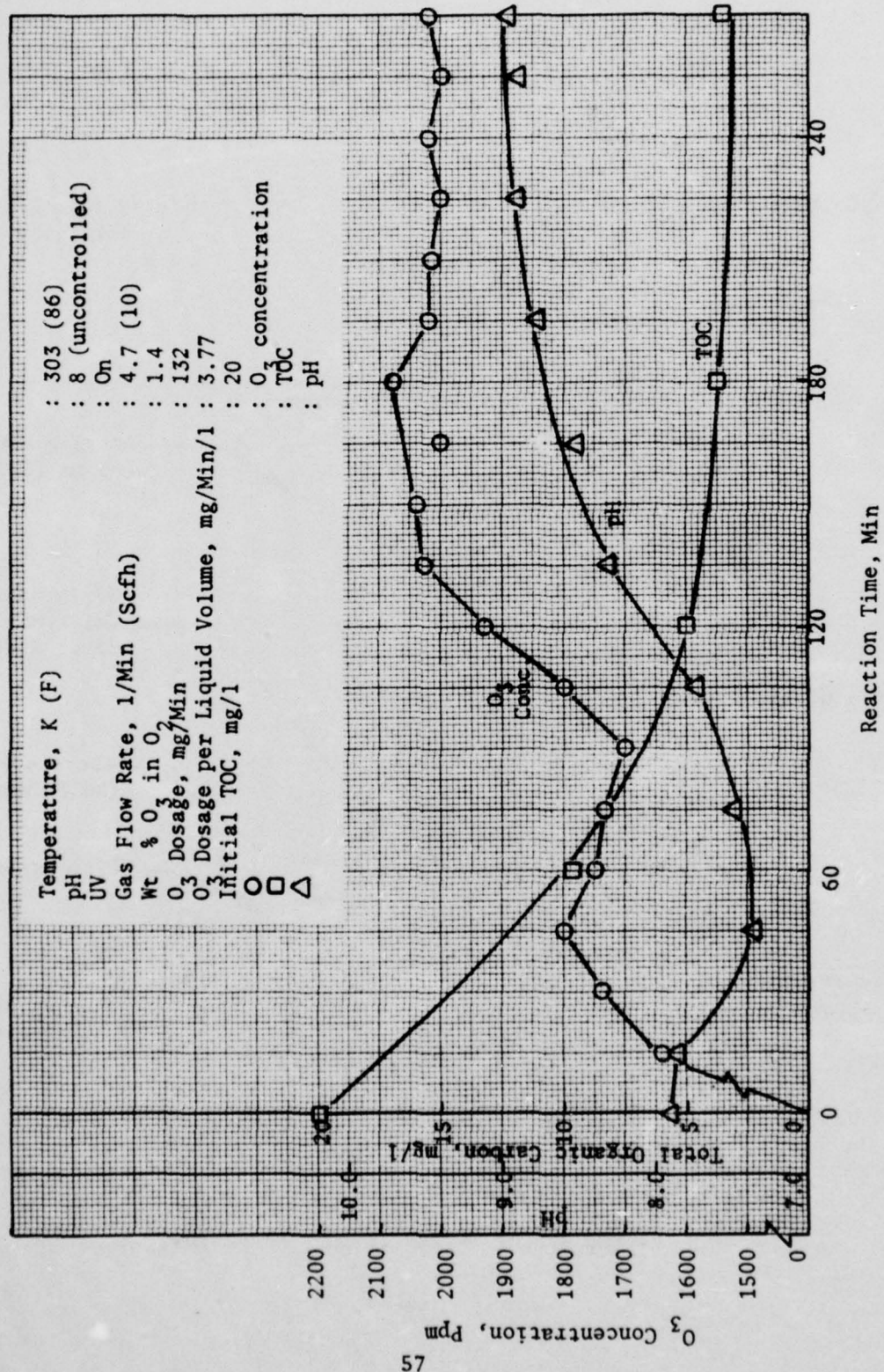


FIGURE 26 BATCH O<sub>3</sub>/UV OXIDATION OF ETHANOL

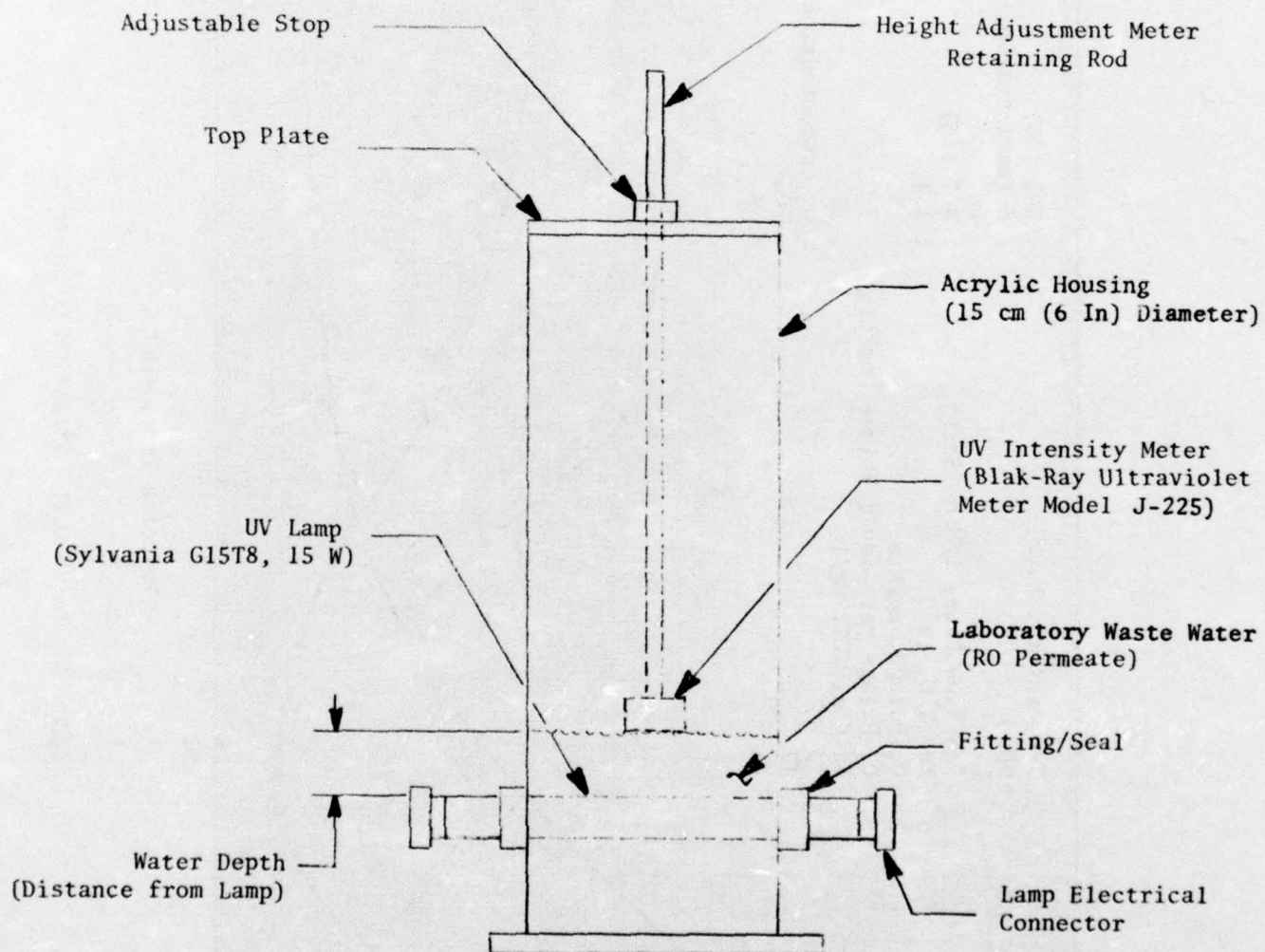


FIGURE 27 UV INTENSITY TEST APPARATUS



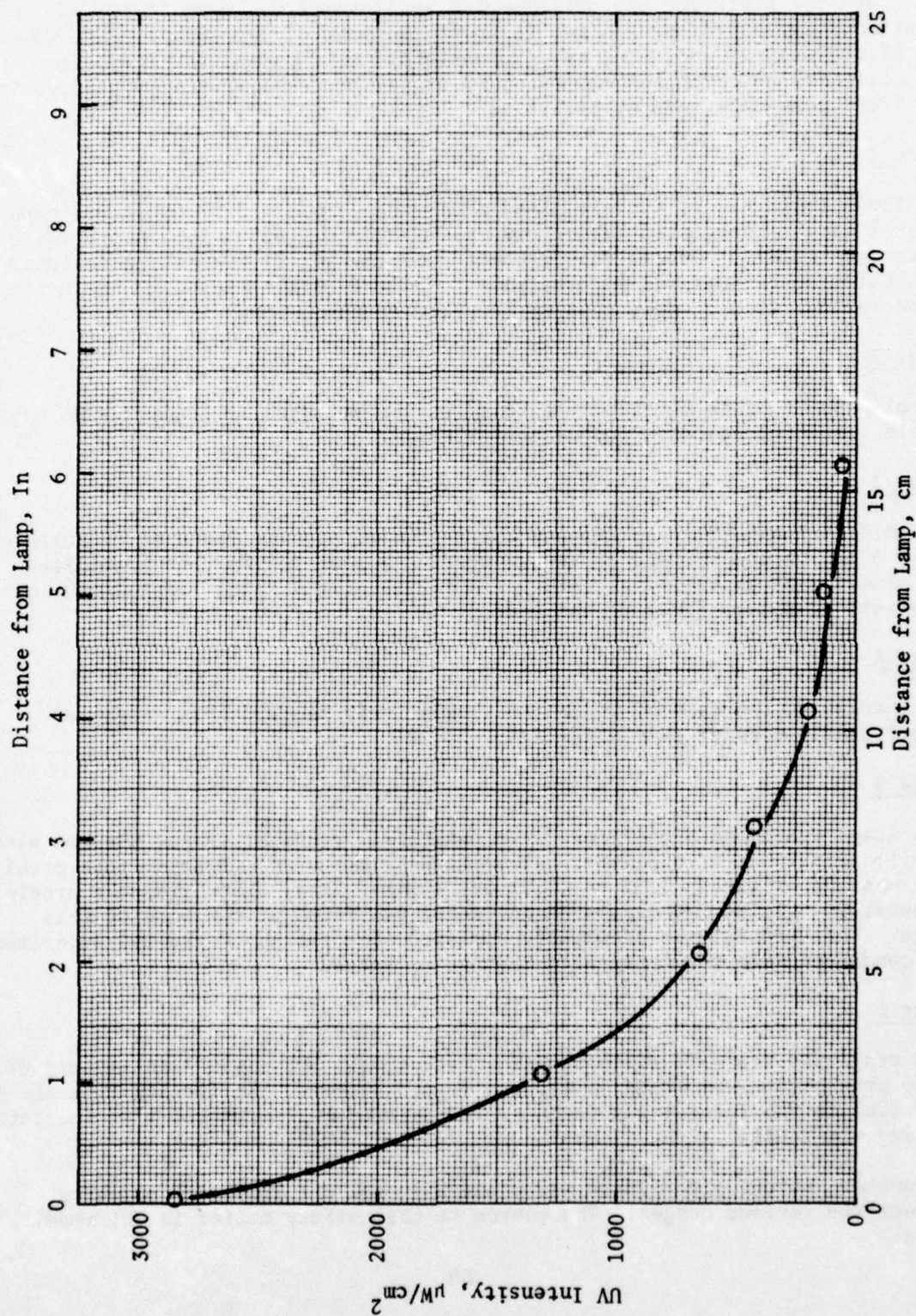


FIGURE 28 UV INTENSITY VERSUS DISTANCE FROM LAMP



housing seal at the base of the contactor stage. While correcting the leak in the stage the contactor was disassembled and inspected. Upon inspection a gelatinous white precipitate was found at the base of the column. Subsequently, all of the stages, including the precontactor, were opened for inspection. The following observations according to stage were made at the completion of the first post-experimental analysis:

#### Stage 1

The first stage was relatively clean after the completion of  $O_3$  autodecomposition, ethanol batch, integrated laboratory waste water and batch laboratory waste water experiments. Due to the frequent draining and filling which followed in each batch test there was no precipitate and only a slight amount of rusting found in this stage. Most rusting occurred at the welds.

#### Stage 2

A small amount of precipitate was found on the spargers of Stage 2. No appreciable oxidation was observed at the welds.

#### Stage 3

A significant amount of gelatinous white precipitate was found at the base of Stage 3 and on the surface of the sparger. At some points this precipitate was almost 0.32 cm (0.12 in) thick. There was also a significant amount of other sticky matter found at the base.

#### Stage 4

The sparger at the base of Stage 4 was relatively clean. This stage was significantly cleaner than Stage 3.

#### Stage 5

This stage again had significant precipitation. The sparger was covered with a white gelatinous precipitate. At various points on the sparger this precipitate was approximately 0.32 cm (0.12 in) thick. Also, white granular precipitate of possibly calcium and magnesium carbonate was found at the base of this stage. This precipitate presumably appeared when the pH controlled experiment was conducted with the integrated laboratory waste.

#### Stage 6

This stage was again found to be relatively clean. No significant amount of white precipitate was found on the spargers. However, the fitting from the  $O_3$  feed line to the sparger was found to be corroded. It appeared that the tube fitting was faulty.

A brownish, sticky precipitate was present in all the Teflon tubes which connect the various stages. The source of this sticky matter is not readily known.

The following observations were made at the completion of the final post-experimental analysis.

The first stage was in a considerably different condition from what was observed in the previous inspection. A considerable amount of oxidation had taken place at the waste water exit port and at the bracket welds. The ethylene propylene O-ring at the upper end of the quartz tube was found to have undergone oxidation. Products of this oxidation were present as a brown film on the surface of the quartz sleeve. (The O-rings were later replaced by Teflon ones.) A slight amount of white precipitate was found at the base of the column. This precipitate was not found on the sparger.

Stages 2 to 4 were similar to what were observed before with the exceptions that oxidation of bracket weld points was observed and the ethylene propylene O-rings at the upper end of the quartz tube were found in the same condition as the one in Stage 1. Again, a brown film was found on the surface of each quartz sleeve.

Stages 5 and 6 were not opened because the O-rings at the ends of the quartz sleeves were already made of Teflon.

The white gelatinous precipitate found in the stages is probably due to the high pH water experiments (pH 9) conducted with the synthetic waste waters made up from the raw well water with a high hardness. After the first post-experimental analysis findings and the subsequent preprocessing of the well water with the RO unit process, the amount of gelatinous precipitate was reduced significantly. Small quantities of precipitate were found in the first stage when the final post-experimental analysis was conducted. However, ten of the 18 experiments conducted after the first post-experimental analysis inspections were batch experiments conducted in the first stage alone. Two of these experiments were at pH 11 where precipitation is enhanced. It is not anticipated that precipitation will be a problem in the final design since brackish and natural fresh waters will be passed directly through IE, RO and HC, bypassing the O<sub>3</sub>/UV Unit Process and reuse water will be treated by RO prior to O<sub>3</sub> oxidation and maintained at a pH 9 or below. However, the formation of precipitation should be watched for in the pilot plant studies of the MUST WPE.

#### LMTOC Experimental Problems

Several problems were encountered during the LMTOC testing period and were resolved in the course of the program activities.

Sample contamination and TOC analyzer stability problems were encountered during the experiments. Sample contamination was later avoided by using glass sample bottles and caps with Teflon liners. Procedures and consistency in the TOC analyzer operation must be strictly adhered to in order to ensure accurate and consistent TOC results.



The water pressure drop and gas pressure drop in the contactor and the gas line created a problem in water overflows in the earlier phase of the experiments. This problem was solved by modifications of the contactor inlet/outlet lines. Water seals also presented another problem at one time.

If water gets into the gas lines and the system is operated at a relatively low gas flow rate (4.7 l/min (10 scfh)), there is a possibility that the process water might backflow through the check valve to the generator. Since the  $O_3$  generator is normally operating at a flow rate of 28.3 l/min (60 scfh), this problem does not exist in normal operation. However, during shutdown of the LMTOC test stand the problem of water flowing into the gas line was encountered. This problem can be avoided by opening an off gas manifold valve before shutdown to depressurize the contactor before the gas flow is shut off.

The  $O_3$  analyzer also requires a dry gas feed for proper operation. Moisture in the contactor off gas must be removed before the off gas is fed into the analyzer.

#### INSTRUMENTATION

In parallel to the LMTOC development, the instrumentation for the  $O_3$ /UV Unit Process was designed and major functions of the design were selected for fabrication and checkout on a minicomputer controlled  $O_3$ /UV Unit Process Simulator. Because the instrumentation in the WPE is expected to be an integrated system which controls and monitors all six unit processes of the MUST WPE, certain instrumentation design decisions, such as the instrumentation approach, its architecture, and the operator/system interface design, have to be made at the overall WPE system level rather than the unit process level. The rest of the instrumentation functions, including individual control loops, mode control, transition control and fault detection and isolation analysis, will be discussed at the  $O_3$ /UV Unit Process level.

#### Background

Instrumentation is used to control and monitor a process. Effective instrumentation results in the minimization of operator man-hours, operator skill level, operator error, system failures, downtime, maintenance, and the maximization of system and personnel safety. The functions of instrumentation are shown in Figure 29. (19)

In general, instrumentation can be divided into control and monitor functions. (20) Control functions include operating mode control, mode transition, maintenance of set points, implementation of set point modifications and automatic optimization of set points. Monitor functions are defined as Fault Detection, Isolation and performance Analysis (FDIA). There is an overlap between the control and monitor functions which includes instrumentation functions necessary to achieve personnel safety, system safety (e.g., automatic shutdown), system component fault correction and elimination of operator errors.



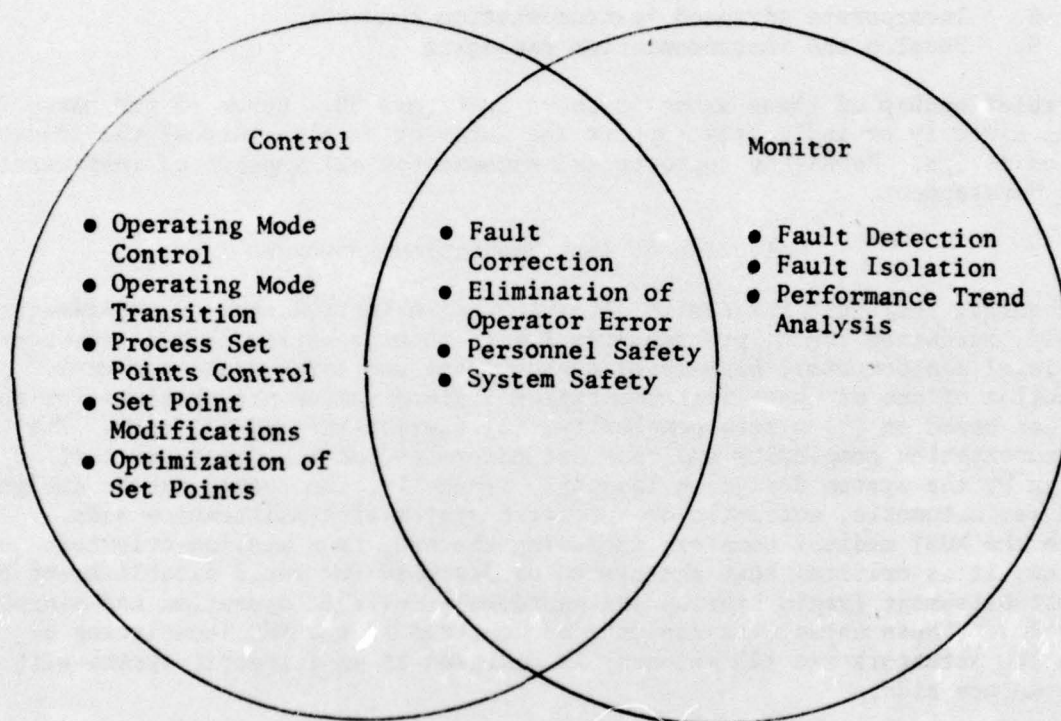


FIGURE 29 FUNCTIONS OF INSTRUMENTATION

### Instrumentation Development

Before an instrumentation design can be initiated, it is important to understand and define the development areas which must be addressed. Nine instrumentation development areas have been identified.<sup>(3)</sup> These areas include:

1. Evaluate unit process operation and parametric performance requirements
2. Evaluate the integrated unit processes
3. Develop operator/system interface
4. Develop system maintenance aids
5. Develop instrumentation's interior architecture
6. Incorporate developer's knowledge of system operation
7. Develop the TSA control interface
8. Incorporate advanced instrumentation concepts
9. Develop the instrumentation packaging

The relationship of these areas is shown in Figure 30. Seven of the nine areas directly or indirectly support the interior architecture of the instrumentation design. Packaging supports and encompasses all aspects of instrumentation development.

### Selection of Instrumentation Approach

In general, there are six design alternatives in instrumentation implementation; namely, hardwired logic, programmable logic, microprocessor or microcomputer, low-level minicomputer, high-level minicomputer and large-scale computer. The selection of one of these instrumentation implementation alternatives for the WPE was based on (1) system complexity, (2) flexibility and (3) cost. The instrumentation complexity and cost are determined at the first level of design by the system design philosophy. Typically, the system can be designed as a semiautomatic, automatic or automatic system with maintenance aids. Since the MUST medical complex, including the WPE, is a mission-oriented system, it is critical that the system be designed for rapid establishment and disestablishment (rapid startup and shutdown), reliable operation and minimum downtime. These objectives can only be achieved if the WPE (consisting of over 100 actuators and 100 sensors) is designed as an automatic system with maintenance aids.

Instrumentation complexity and cost is further determined at the second level of design by the number of parameters monitored and controlled, the number of operating modes, the number of allowable mode transitions, the level of fault detection, fault isolation, fault prediction and fault correction, and the parameter controllability. In order to quantify the instrumentation complexity, the number of small-scale integrated circuit packages needed for hard-wired implementation was used as a complexity index.

### System Complexity

The current industrial instrumentation implementation practice as a function of complexity is shown in Figure 31.<sup>(21,22)</sup> Random logic implementation is used whenever the complexity is low enough. As the complexity increases,

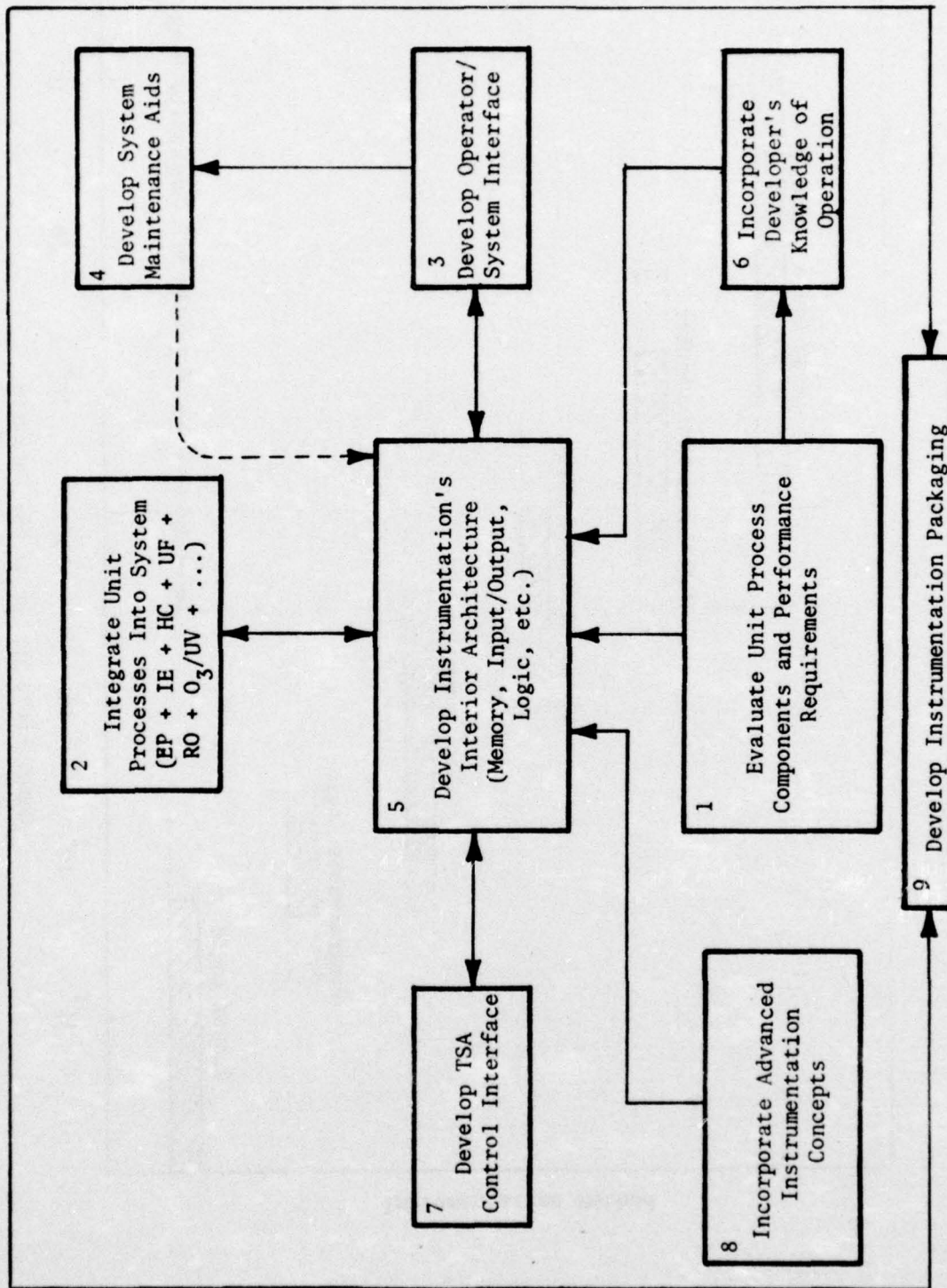


FIGURE 30 INSTRUMENTATION DEVELOPMENT AREAS



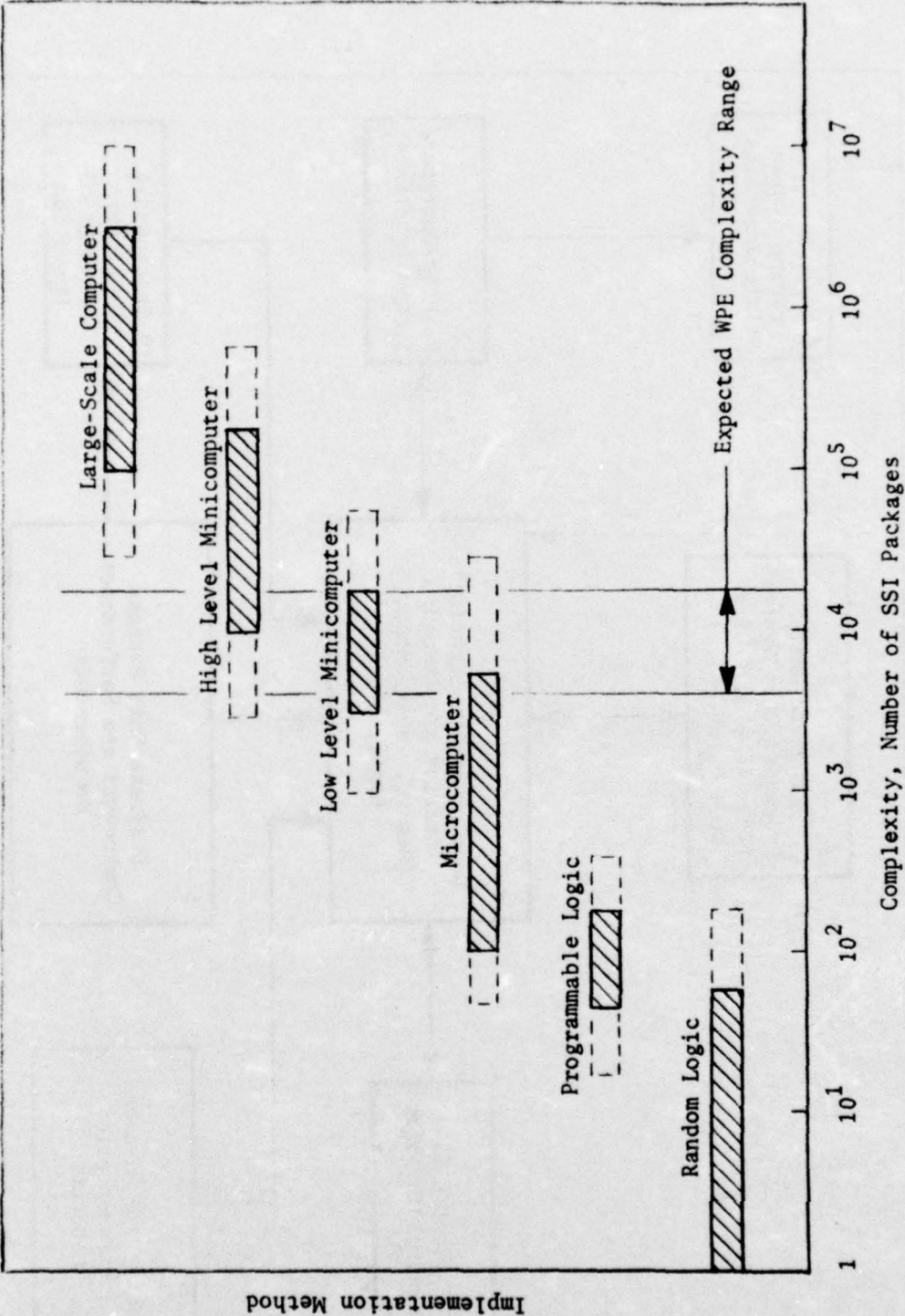


FIGURE 31 INDUSTRIAL INSTRUMENTATION IMPLEMENTATION PRACTICE

programmable logic and microprocessors are selected. Minicomputers and sometimes large-scale computers are used in cases where the complexities are too high to be implemented with microcomputers. For the WPE instrumentation design with advanced maintenance aids, the complexity is high enough that a low-level minicomputer is needed.

#### Flexibility

Flexibility is an inherited characteristic in a computer-controlled system. Control and monitor set points can be changed easily in such a system. The number of operating modes can be changed. Operating mode transitions can be altered, timing sequences changed, and scaling factors updated with little hardware modification. Since the WPE is at an early stage of development, a number of system and unit process changes can be anticipated. To handle these changes in the most cost effective manner, an instrumentation design with maximum flexibility for change is desired. This flexibility can only be achieved with a computer design.

#### Cost

Two types of cost must be considered in instrumentation design; namely, hardware cost and development cost. These costs are difficult to establish on an exact basis since they are functions of production quantity and the organization developing the instrumentation. In spite of these factors, certain cost quantifications can be made to develop an understanding of the hardware and development cost trade-offs.

Hardware Cost. Hardware cost per implemented function as it is related to instrumentation complexity and approach is shown in Figure 32. The costs per function are based on a production quantity of 1 to 25 WPEs/year.

Large-scale or small computers have a high initial cost compared to random logic. Thus, the hardware cost per function is typically very high. Since the computer hardware cost is fixed, the cost per increasing number of functions decreases. This trend continues until the capacity of the computer is exceeded. Then the cost per function increases.

Random logic has the advantage of initial low and variable hardware cost. However, the hardware cost per implemented function starts to increase almost immediately. This increasing cost trend continues with increased instrumentation complexity.

The hardware cost per function indicates that for the WPE instrumentation design with maintenance aids, the low-level minicomputer has the lowest hardware cost.

Development Cost. The development cost is mostly software programming time in a computer-based system and is mostly logic design and circuit design time in a random logic or programmable logic implemented system. Development cost per

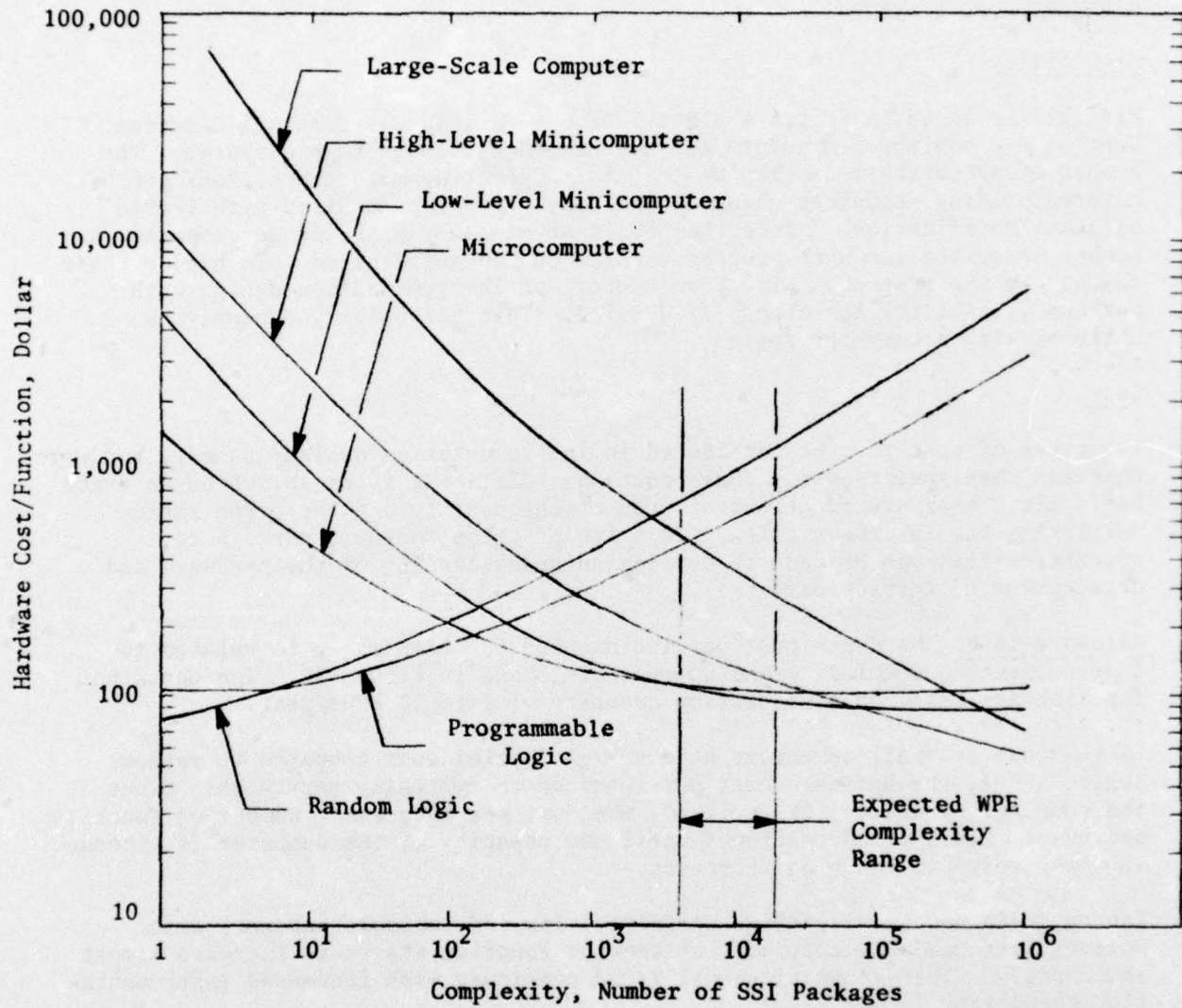


FIGURE 32 ESTIMATED INSTRUMENTATION  
HARDWARE COST VERSUS COMPLEXITY



implemented function versus instrumentation complexity is shown in Figure 33. The logic and circuit design cost per function increases with increasing complexity because of the more complicated electronic interfaces among different functional modules. Development cost per function in a computer-based system decreases as complexity increases. This is because the cost of system software and the number of subroutines can be shared by more functions. This decreasing cost stops when the system complexity exceeds the computer capability. An increasing development cost per function begins as soon as this limit is reached. In general, program development cost per function is higher for a microcomputer than a minicomputer because of better software support in the minicomputer industry. Similarly, program development cost for a minicomputer system is higher than a larger-scale computer system because the latter has even better software support from the computer manufacturer.

Computers have the advantage that in production the primary electronic hardware is the printed circuit (PC) boards of the computer. The basic minicomputer is produced in high quantities and used throughout the industry; hence, a well-debugged hardware design with low component infant mortality can be expected.

#### Instrumentation Architecture

Once the minicomputer instrumentation approach has been selected the architecture or functional areas of the design can be established. Instrumentation architecture is typically influenced by:

1. Speed of controlled process
2. Performance goals
3. System safety requirements
4. Fault tolerance requirements
5. Reliability
6. Size goals
7. Number of sensors and actuators involved
8. Characteristics of the controlled parameters
9. Modularity
10. Expendability
11. Cost

Three possible WPE instrumentation configurations are shown in Figures 34, 35, and 36. The single processor (uniprocessor) system employs a dedicated minicomputer for the control and monitor of the WPE system. This configuration has the advantage of simplicity, small size and low cost. The dual processor configuration may be designed so the second processor is an identical replication of the first for a true redundancy, or so the second processor is a simple shutdown processor to perform a safe shutdown operation. The dual processor has the advantage of high reliability and fail-safe shutdown capability. The multiprocessor configuration employs three or more computers in the design. There are a number of different types of multiprocessor designs. For example, each computer can be assigned for a specific function and all computers are

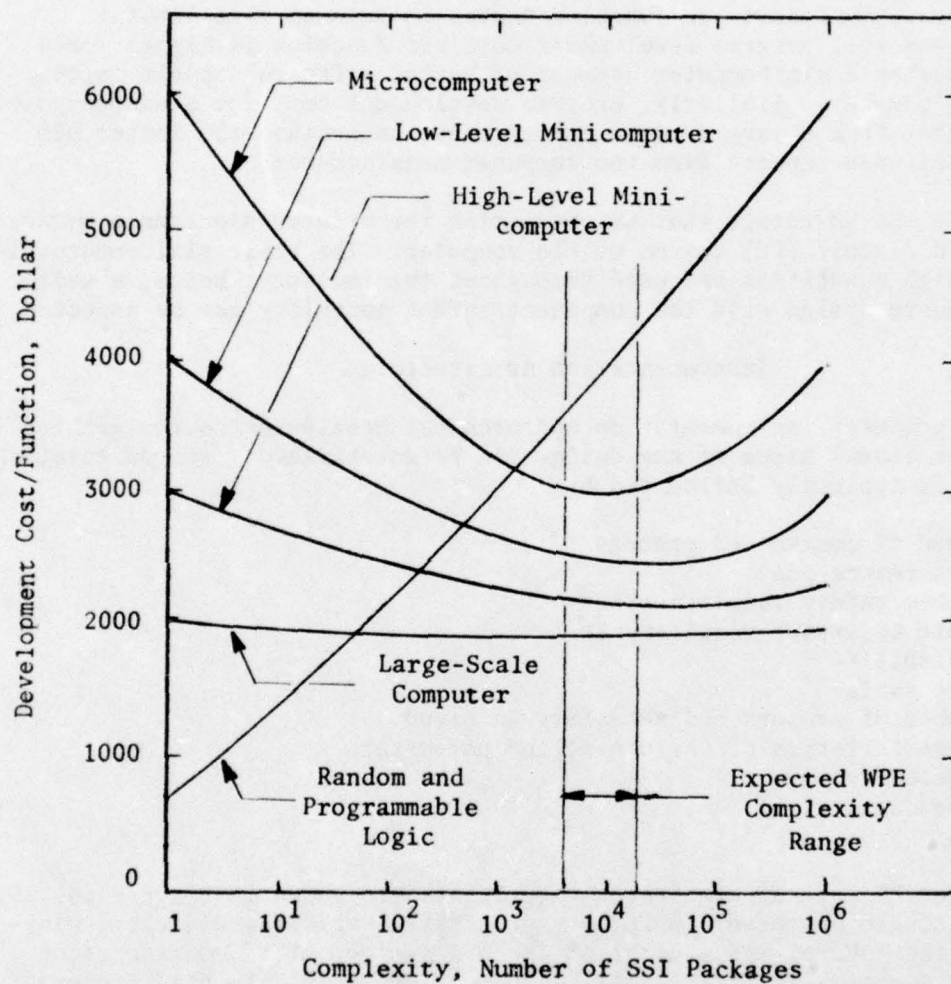


FIGURE 33 ESTIMATED INSTRUMENTATION DEVELOPMENT COST VERSUS COMPLEXITY

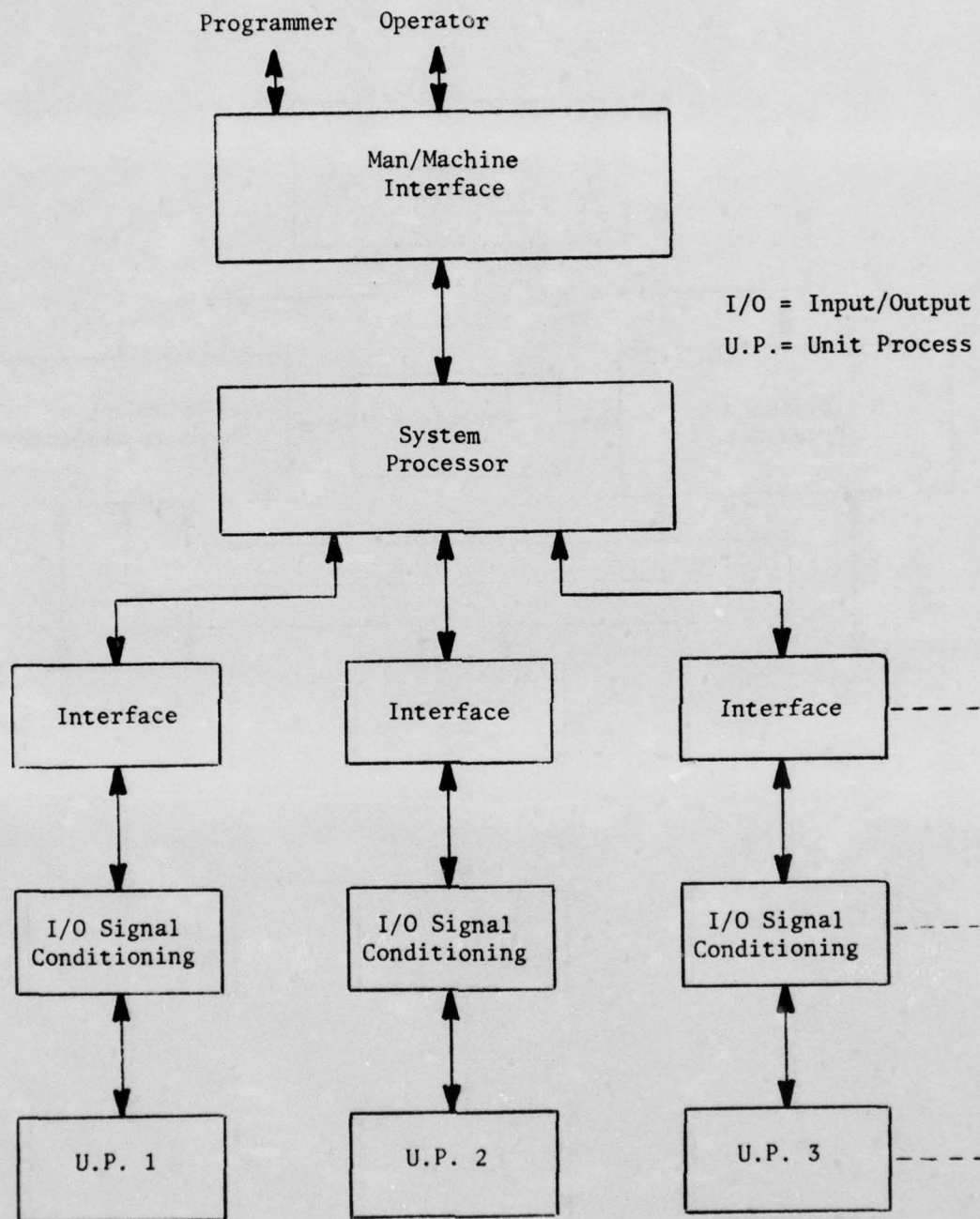


FIGURE 34 UNIPROCESSOR CONFIGURATION



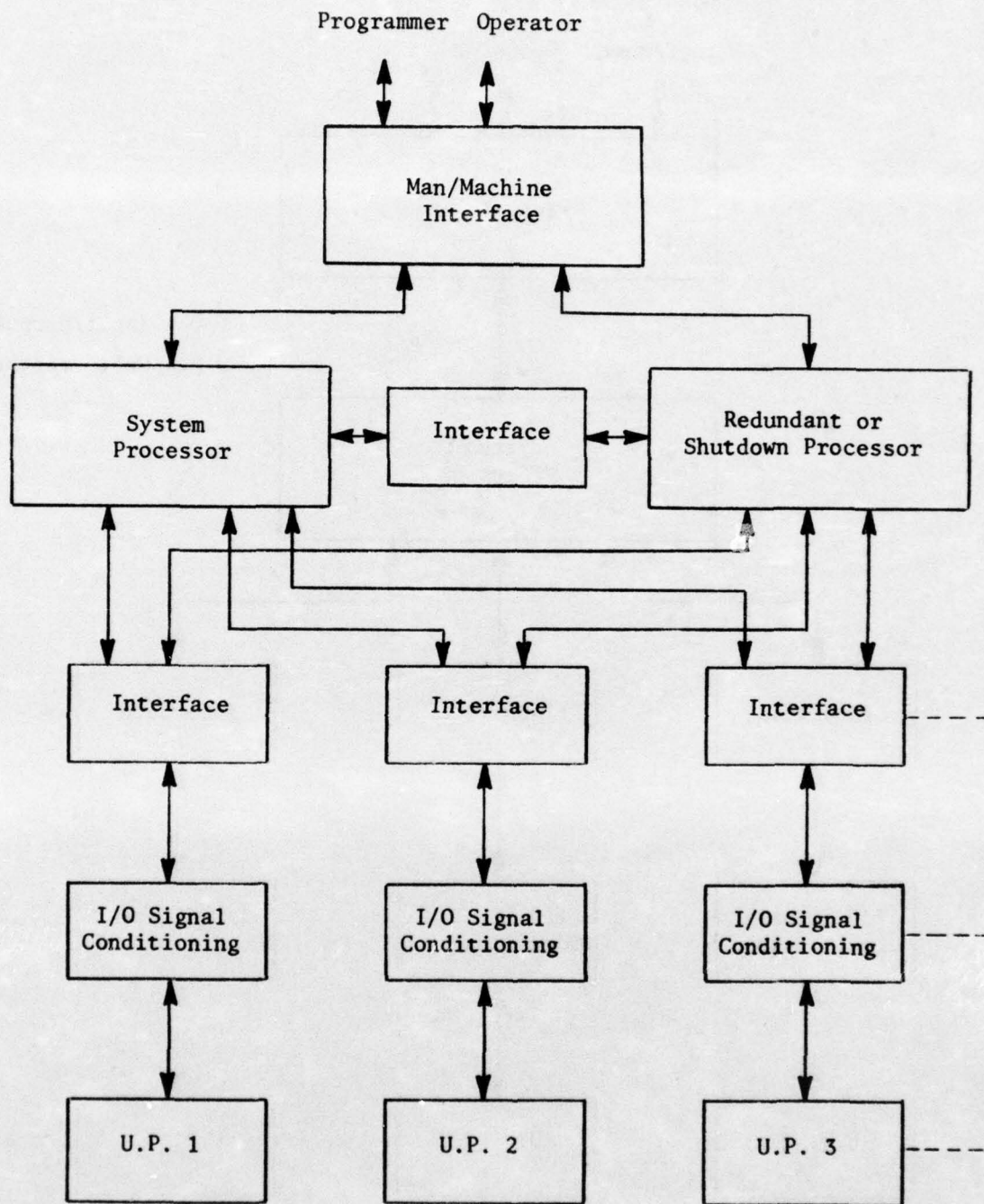


FIGURE 35 DUAL PROCESSOR CONFIGURATION

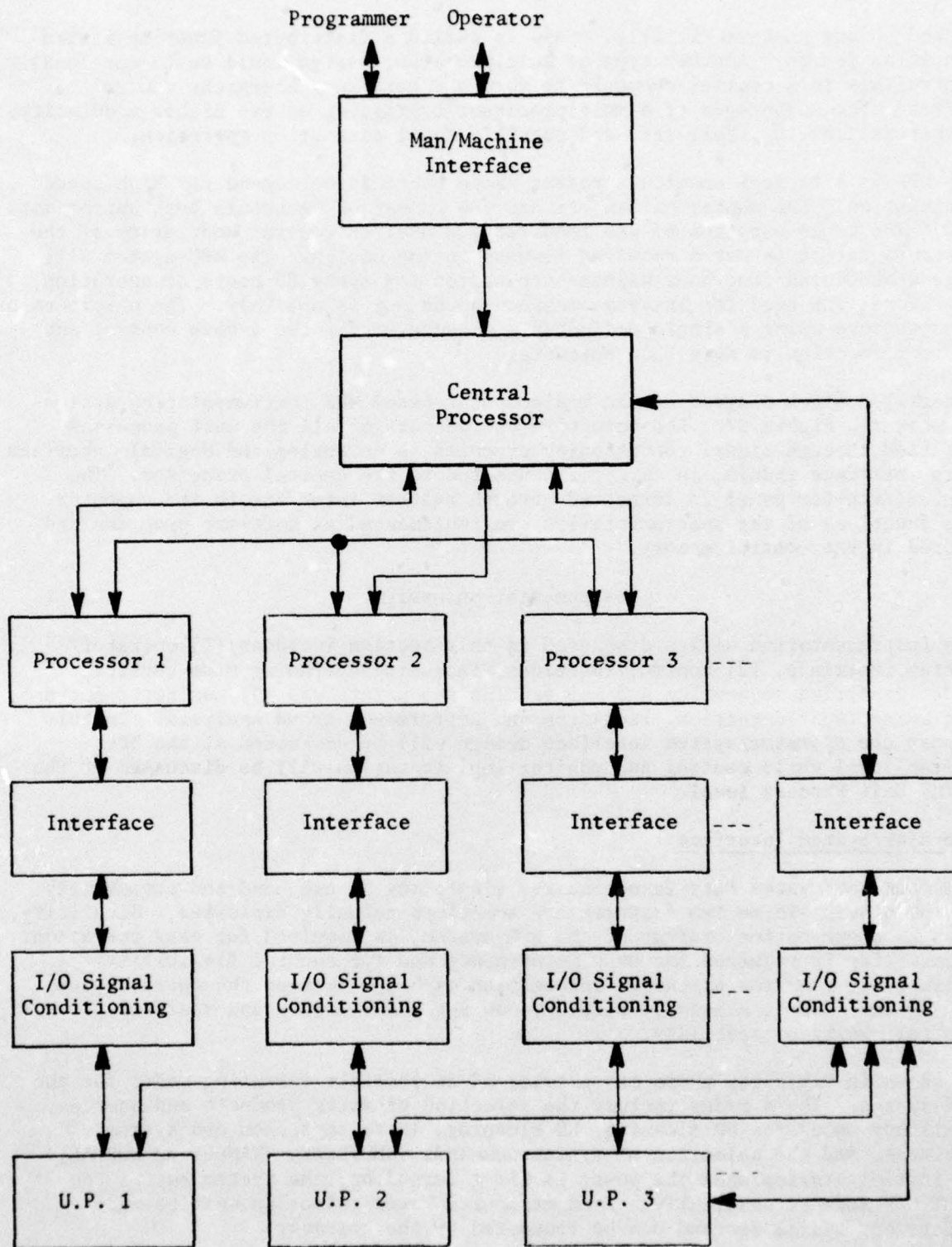


FIGURE 36 MULTIPROCESSOR CONFIGURATION

linked to one another directly. This is called a distributed function instrumentation design. Another type of multiprocessor design would be to tie local controllers to a central computer to form a supervisory hierarchy control system. The advantages of a multiprocessor configuration are higher modularity, higher reliability, fail-safe and possibly fault correction operation.

The WPE is a typical chemical process where there is no demand for high-speed computation. The number of sensors and the number of actuators both approximate 100. The implementation of all required and desired control modularity of the instrumentation is not a required feature in the design. The WPE system will have a scheduled four-hour maintenance period for every 20 hours of operation; therefore, the need for instrumentation redundancy is unlikely. The uniprocessor architecture using a single dedicated minicomputer for the entire control and monitor function is more than adequate.

A detailed block diagram of the minicomputer-based WPE instrumentation design is shown in Figure 37. The actuators and sensors of all the unit processes are tied through signal conditioning circuits to an analog and digital interface. This interface module, in turn, is connected to the central processor. The control/monitor panel is connected through another interface to the computer. The functions of the instrumentation are implemented as software programs and stored in the control memory.

#### Instrumentation Design

The instrumentation design discussed in this section includes (1) operator/system interface, (2) control functions, including operating mode control, mode transition sequencing and maintaining set points and (3) monitor functions, including fault detection, isolation and performance trend analysis. In this report the operator/system interface design will be discussed at the WPE system level while control and monitor implementation will be discussed at the O<sub>3</sub>/UV Unit Process level.

##### Operator/System Interface

The operator/system interface requires simplicity on one hand and versatility on the other. These two features are sometimes mutually exclusive. Simplicity, such as a one-button startup of the WPE system, is required for easy operation. Versatility is required for easy maintenance and for control flexibility. Versatility requires extensive information exchange between the operator and the system such as a message display, new set point inputs and operator control override capability.

As shown in Table 16, there are a total of 26 possible operating modes for the WPE system. These modes include the selection of water products and sources, auxiliary modes for UF cleaning, RO cleaning, IE regeneration and system drainage, and the selection of system commands (SHUTDOWN, STANDBY or NORMAL). At initial startup when the power is first turned on, the system enters the SHUTDOWN mode automatically. Product/source water selection must be made before any system command can be requested by the operator.



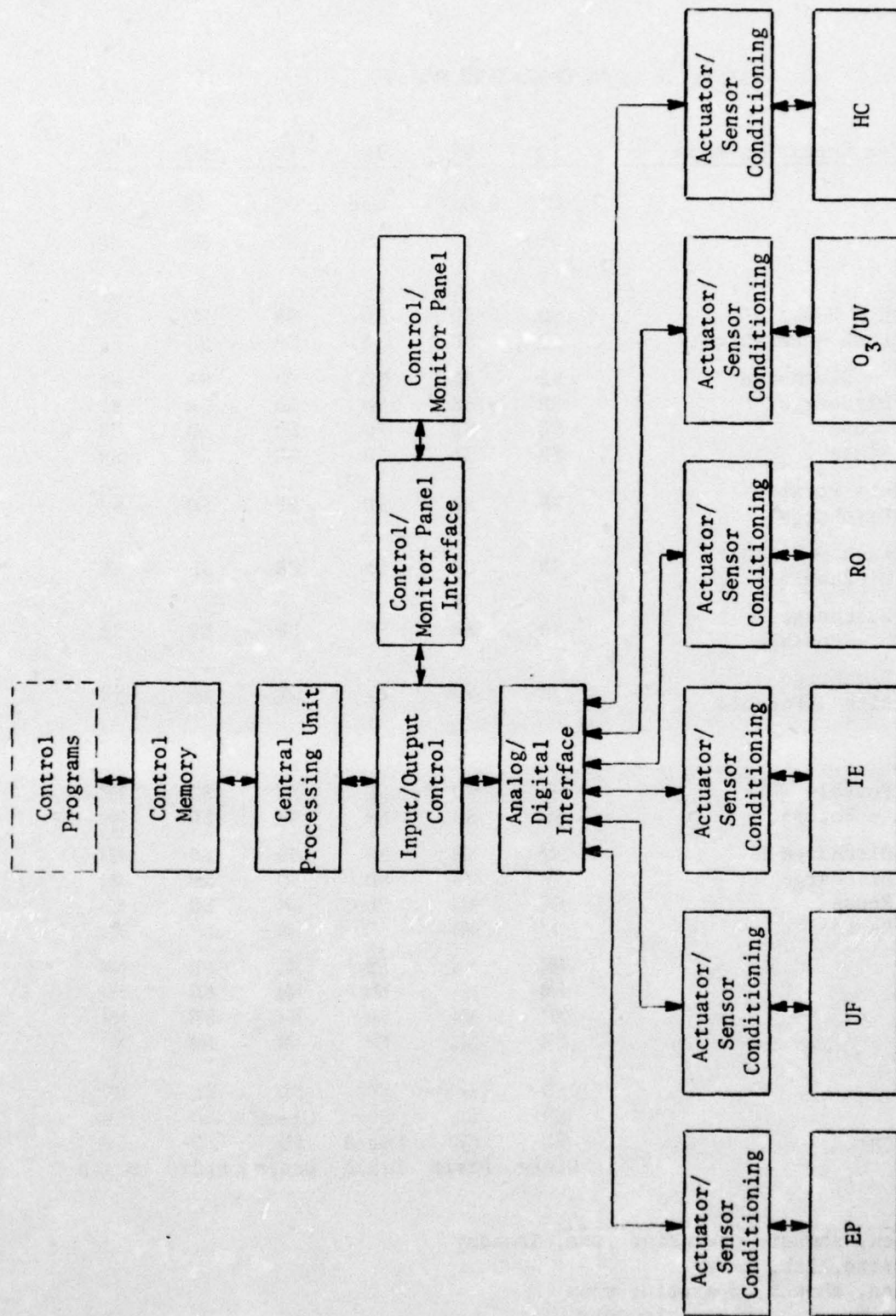


FIGURE 37 WPE INSTRUMENTATION

TABLE 16 WPE OPERATING MODES

WPE System Operating Mode	EQ	UF	IE	RO	OO	HC
Power Off	Off	Off	Off	Off	Off	Off
Shutdown (SD)	SD	SD	SD	SD	SD	SD
Standby (SB)						
(F) Fresh → Potable	SD	SD	SD	SB	SD	SB
(K) Brackish → Potable	SD	SD	SB	SB	SD	SB
A <sup>(a)</sup> → Discharge	SB	SB	SD	SD	SD	SB
B → Discharge	SB	SB	SD	SD	SB	SB
C → Reuse	SB	SB	SD	SB	SD	SB
D → Reuse	SB	SB	SD	SB	SB	SB
F+A { Fresh → Potable A → Discharge	SB	SB	SD	SB	SD	SB
K+A { Brackish → Potable A → Discharge	SB	SB	SB	SB	SD	SB
B+F { B → Discharge Fresh → Potable	SB	SB	SD	SB	SB	SB
B+K { B → Discharge Brackish → Potable	SB	SB	SB	SB	SB	SB
Normal (NM)						
Fresh → Potable	SD	SD	SD	NM	SD	NM
Brackish → Potable	SD	SD	NM	NM	SD	NM
A → Discharge	NM	NM	SD	SD	SD	NM
B → Discharge	NM	NM	SD	SD	NM	NM
C → Reuse	NM	NM	SD	NM	SD	NM
D → Reuse	NM	NM	SD	NM	NM	NM
F+A	NM	NM	SD	NM	SD	NM
K+A	NM	NM	NM	NM	SD	NM
B+F	NM	NM	SD	NM	NM	NM
B+K	NM	NM	NM	NM	NM	NM
UF Clean	SD	Clean	SD	SD	SD	SD
RO Clean	SD	SD	SD	Clean	SD	SD
IE Regeneration	SD	SD	Regen	SD	SD	SD
System Drain	Drain	Drain	Drain	Drain	Drain	Drain

- (a) A = kitchen, shower, operating room, laundry  
 B = composite, lab, x-ray  
 C = kitchen, shower, operating room  
 D = composite, lab, x-ray, laundry



There are four system modes and seven allowable system mode transitions for the WPE as shown in Figure 38. These four modes are POWER OFF, SHUTDOWN, STANDBY and NORMAL. POWER OFF and NORMAL modes are self-explanatory. In the STANDBY mode there is no water production. However, actuators and sensors which require warmup time or startup time are activated. For example, water temperatures, water levels and air dryer refrigerant loop temperatures must be maintained which require related unit process heaters, pumps and compressors to be activated. In the SHUTDOWN mode the instrumentation and sensors are on with the valves in shutdown positions and the actuators off.

Figure 39 shows the WPE control/monitor panel design. The control panel is designed to eliminate possible operator errors. If mode transitions which are not allowable are requested, the system will send out warning messages to the monitor panel and take proper actions. For example, if the transition from the SHUTDOWN mode to the NORMAL mode is requested the system will inform the operator that direct transition is not allowed and a STANDBY request is automatically generated. When the steady-state STANDBY mode is reached the system automatically implements the transition from the STANDBY to NORMAL. If a change of source or product water is requested when the WPE is in NORMAL or STANDBY mode the request will be rejected and an error message displayed on the monitor panel. Change of the product/source water selection and requests for auxiliary modes are allowed only in the system SHUTDOWN mode.

Multiple-colored lights are used to indicate steady-state and mode transitions. Amber indicates a mode transition is in progress and that the system has acknowledged the request for a new operating mode. A green light indicates that the system is currently in the requested mode. A number of manual override switches are provided in a recessed panel behind the water source description panel. These manual overrides include the primary actuators and the ones needed for system maintenance.

The monitor panel includes a system status summary, a monitor message display, a monitor command keyboard and timers required for scheduled maintenance. The system status summary has four lights: NORMAL, CAUTION, WARNING and ALARM. Except for the NORMAL status there will be messages displayed on the gas discharged dot matrix display panel indicating the cause of the CAUTION, WARNING or ALARM. Through the monitor command keyboard the operator can examine and modify a control or monitor set point. On-line display of parametric data can also be requested.

Figure 40 is a photograph of the WPE control/monitor panel. The monitor message and command functions were implemented on a CRT/keyboard terminal not shown in the photograph. Table 17 summarizes all components of the control/monitor panel and their functions.

#### Ozone Oxidation Unit Process Control Instrumentation

The objective of the O<sub>3</sub> Oxidation Unit Process is to oxidize the organic compounds in the process water to a level below 5 mg/l TOC and 10 mg/l COD.



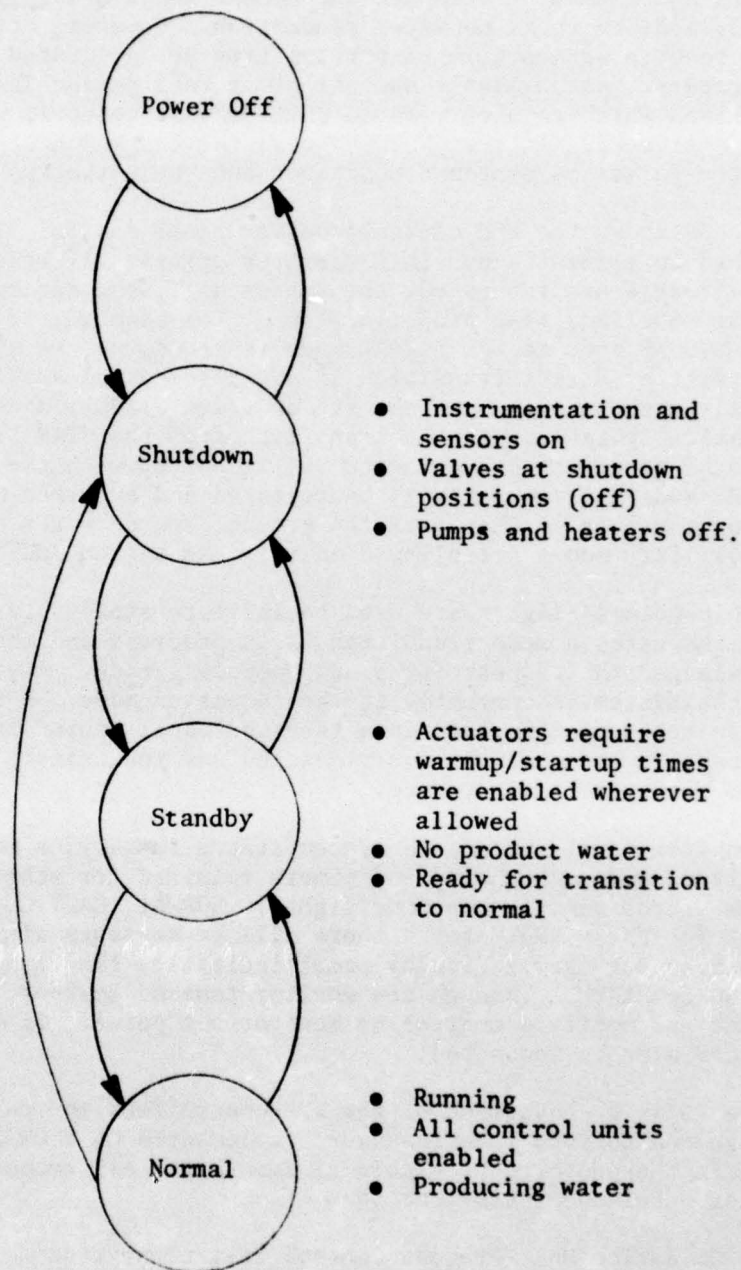
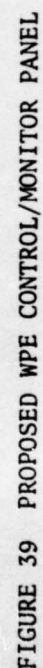


FIGURE 38 OZONE OXIDATION UNIT PROCESS  
MODE TRANSITION DIAGRAM



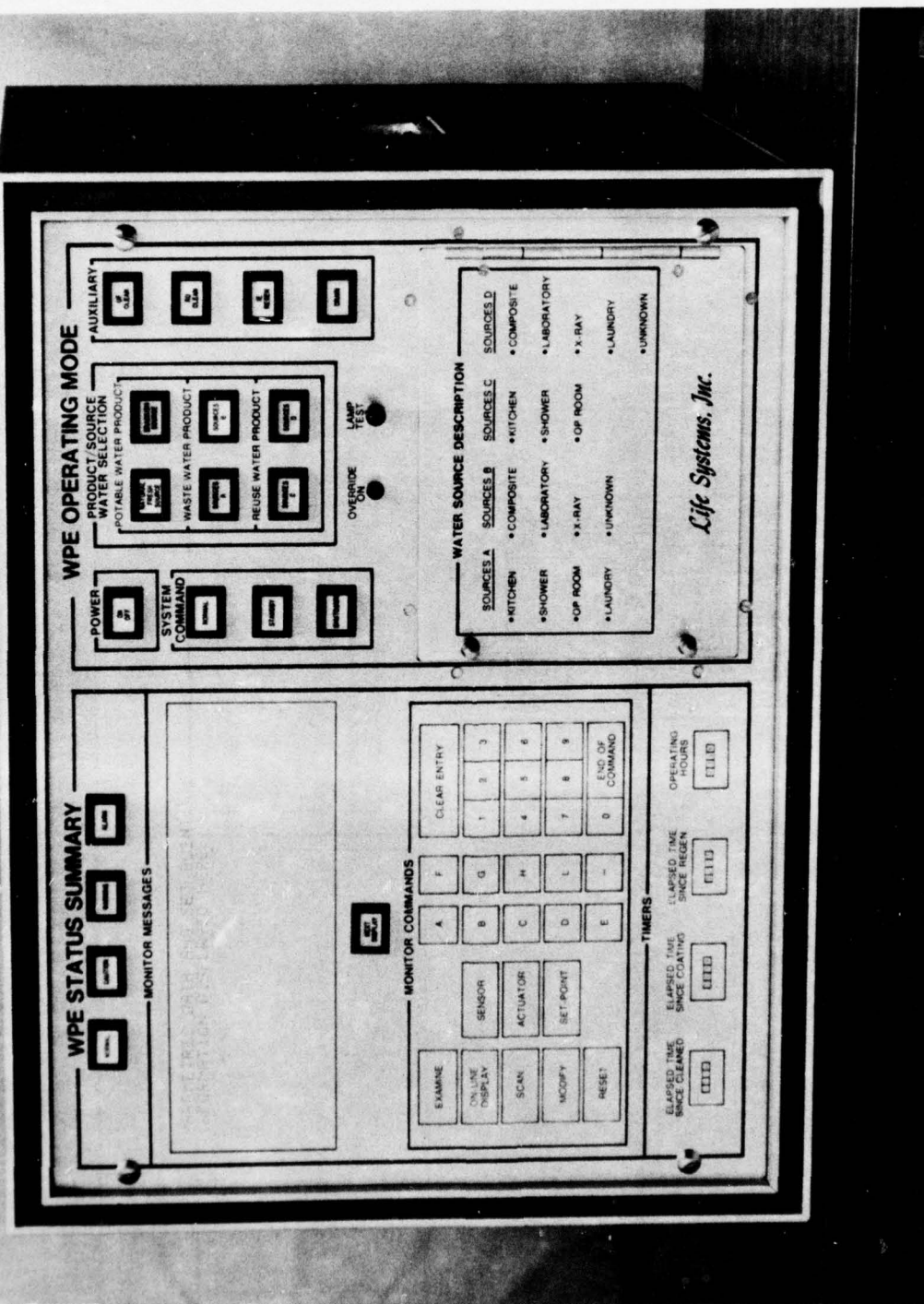


FIGURE 40 CONTROL/MONITOR PANEL



TABLE 17 WPE CONTROL/MONITOR PANEL COMPONENTS

Control Panel

Component	Function
System Command	Pushbutton switches for mode transition request. Light displays indicate current mode (in green) or transition in process (in amber).
<ul style="list-style-type: none"> <li>● On/Off</li> <li>● Shutdown</li> <li>● Standby</li> <li>● Normal</li> </ul>	<ul style="list-style-type: none"> <li>Power on/off request/indicator.</li> <li>Shutdown mode request/indicator.</li> <li>Standby mode request/indicator.</li> <li>Normal mode request/indicator.</li> </ul>
Product/Source Water Selection	Pushbutton switches activated in shutdown mode for product and source water selection. Green lights indicate current selection which may be cancelled by a second push or by selecting another mode. Validity is automatically checked.
<ul style="list-style-type: none"> <li>● Natural Fresh Source</li> <li>● Brackish Source</li> <li>● Source A</li> <li>● Source B</li> <li>● Source C</li> <li>● Source D</li> </ul>	<ul style="list-style-type: none"> <li>Produce potable water from fresh source.</li> <li>Produce potable water from brackish source.</li> <li>Treat source A for waste discharge.</li> <li>Treat source B for waste discharge.</li> <li>Reuse source C for non-consumptive purposes.</li> <li>Reuse source D for non-consumptive purposes.</li> </ul>
Auxiliary	Activated in shutdown mode for auxiliary maintenance modes.
<ul style="list-style-type: none"> <li>● UF Clean</li> <li>● RO Clean</li> </ul>	<ul style="list-style-type: none"> <li>UF module cleaning.</li> <li>RO module cleaning.</li> </ul>

continued-

Table 17 - continued

Component	Function
Auxiliary - continued	
• IE Regeneration	IE module regeneration.
• System Drain	Drain all water tanks.
Lamp Test	Lamp test pushbutton.
Manual Overrides	Switches and potentiometers on recessed panel for override.
• Override On	Indicator lit if any override switch is on.
• Switches and Potentiometers	For manual override.

Monitor Panel

Component	Function
WPE Status Summary	Summary of system status as indicated by the four lights.
• Normal	System normal.
• Caution	Cause of this status is explained on the monitor message panel.
• Warning	Cause of this status is explained on the monitor message panel.
• Alarm	Cause of this status is explained on the monitor message panel.
Monitor Messages	Display panel for system/operator communication. Total of 256 characters.

continued-

Table 17 - continued

Component	Function
Monitor Messages - continued	
• Next Display	Light on indicates there are more than 256 characters to output. Push the switch to update the display to the next 256 characters.
Monitor Commands	Operators command to monitor for setpoint modifications within allowable ranges and for display of data.
• Examine	Examine a parameter or setpoint.
• On-line Display	Display up to 16 parameters and update them every minute.
• Scan	One button scans and displays data of key parameters.
• Reset	Clear panel display and previous commands.
• Other Switches	For selection of sensor, actuator and setpoint numbers.
Timers	Monitors elapsed times for scheduled maintenance.
• Elapsed Time Since Cleaned	For UF clean.
• Elapsed Time Since Coating	For RO clean.
• Elapsed Time Since Regeneration	For IE regeneration.
• Operating Hours	



The ultimate controlled process variable is, therefore, the water TOC and COD concentrations. To achieve the optimum  $O_3$  and water organic reaction rate a number of process parameters have to be controlled within limited ranges. The controlled process parameters and ranges selected for allowable changes are listed in Table 18. Each parameter is controlled and maintained in the desired range by a software control program. A description of the control programs selected for the  $O_3$  Oxidation Unit Process is given in Table 19.

A recessed manual override panel is provided under the Water Source Description panel. Actuators with high power consumption (heaters,  $O_3$  generator, pumps, etc.) and actuators related to maintenance (drain valves, filter select, etc.) are provided with manual overrides.

#### Ozone Oxidation Unit Process Monitor Instrumentation

This section discusses the aspect of monitor instrumentation which is used for component fault detection and isolation. Fault detection refers to the function of detecting a component failure or failures from the observations of system symptoms. Fault isolation is the diagnostic function which analyzes the detected symptoms and isolates the cause to a specific fault or to a limited number of faults. Figure 41 shows the relationships among probable faults and their corresponding symptoms. In general, faults can be detected as long as sensors for detecting the symptoms are available. On the other hand, fault isolation or diagnostics present a rather complicated problem mainly because faults and symptoms are not on a 1:1 correspondence. There have been 130 faults and 27 symptoms identified for the LMTOC. A detailed study of the fault detection in the  $O_3$  Oxidation Unit Process is given in Appendix 2.

A flow chart describing the fault diagnostic and isolation steps for the  $O_3$  Oxidation Unit Process high TOC/COD symptom is given in Figure 42. The diagnostics begin with a checking of the  $O_3$  flow rate and the  $O_3$  generator feed gas pressure to isolate the fault to a few most probable causes. If both the  $O_3$  flow rate and the feed gas pressure are low, either the air compressor or the desiccant dryer select has failed. If the symptoms are TOC/COD high,  $O_3$  flow rate low and  $O_3$  generator feed gas pressure normal, then the most probable cause will be the solenoid valves which control the  $O_3/O_2$  to the contactor or precontactor.

The fault isolation level is a dependent variable of the number of monitor sensors. The cost/performance ratio is the key factor in determining where to stop in fault isolation level. It is felt that if failures can be isolated down to three or less most probable causes in the WPE, trouble-shooting can then be done within the allowable system downtime.

#### Ozone Oxidation Unit Process Simulation

Major instrumentation functions for the LMTOC were selected, fabricated and assembled. An electronic LMTOC simulator was developed to enable the checkout and debugging of the instrumentation functions. Figure 43 shows the simulation

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TABLE 18 OZONE OXIDATION UNIT PROCESS  
CONTROL AND MONITOR PARAMETERS

Controlled Parameters<sup>(a)</sup>

Parameter	Normal Set Point	Allowable Range
TOC, Effluent, mg/l	4.5	0 to 20
pH, Precontactor	9	2 to 12
pH, Contactor	9	2 to 12
Temperature, Water, K (F)	316K (110)	294 to 339 (70 to 150)
Temperature, Contactor, K (F)	316K (110)	294 to 339 (70 to 150)
Dew Point, Air Supply, K (F)	233K (-40)	210 to 239 (-80 to -30)

Monitored Parameters

Parameter	High Alarm Set Point	Low Alarm Set Point
Temperature, O <sub>3</sub> Gen, K (F)	305 (90)	--
Temperature After Cooler, K (F)	300 (80)	--
Temperature, Refrig, Dryer, K (F)	283 (50)	--
Pressure O <sub>3</sub> Gen., kN/m <sup>2</sup> (Psig)	--	69.9 (10)
Flow O <sub>3</sub> Gen., l/Min (Scfh)	--	472 (1000)
Flow, Product Water, l/Min (Gpm)	--	11.4 (3.0) <sup>(b)</sup>

(a) All controller parameters are also monitored for performance trend analysis

(b) Root mean square value

TABLE 19 OZONE OXIDATION UNIT PROCESS CONTROL PROGRAMS

Parameter	Description
TOC/COD	Proportional control of $O_3$ dosage to process water utilizing both feedback and feed forward water TOC/COD signals. The goal is to maintain effluent TOC/COD less than required set points. Only feed forward control is activated during dry start up since a feedback signal will not be available. If effluent TOC/COD doesn't meet the specifications, water will be recycled back to the contactor.
Process Water Temperature	Proportional control to maintain temperature at set point. A hot water heat exchanger with variable orifice diverter valve is the actuator.
Contactor Water Temperature	On/off control to make up the heat loss.
Precontactor Water pH	On/off control of acid and base additives to process water to maintain pH at 9.
Contactor Water pH	Same as above; maintain pH at 9 during entire Ozone Oxidation Process.
Precontactor Water Level	In reuse mode, high level sensor will stop influent water from previous unit processes. In discharge mode, high level sensor will open the effluent solenoid valve and output water intermittently.
Contactor Water Level	In reuse mode, high level sensor opens effluent valve and low level sensor closes it. Effluent water is intermittent.
Air Temperature	Open loop control with high temperature shutdown monitor. Air passes through a cooler and a refrigerant loop. Air temperature should always be 40F or lower.
Air Dew Point	Dew point feedback signal controls selection of desiccant. When not selected, the desiccant is regenerated by a heater.

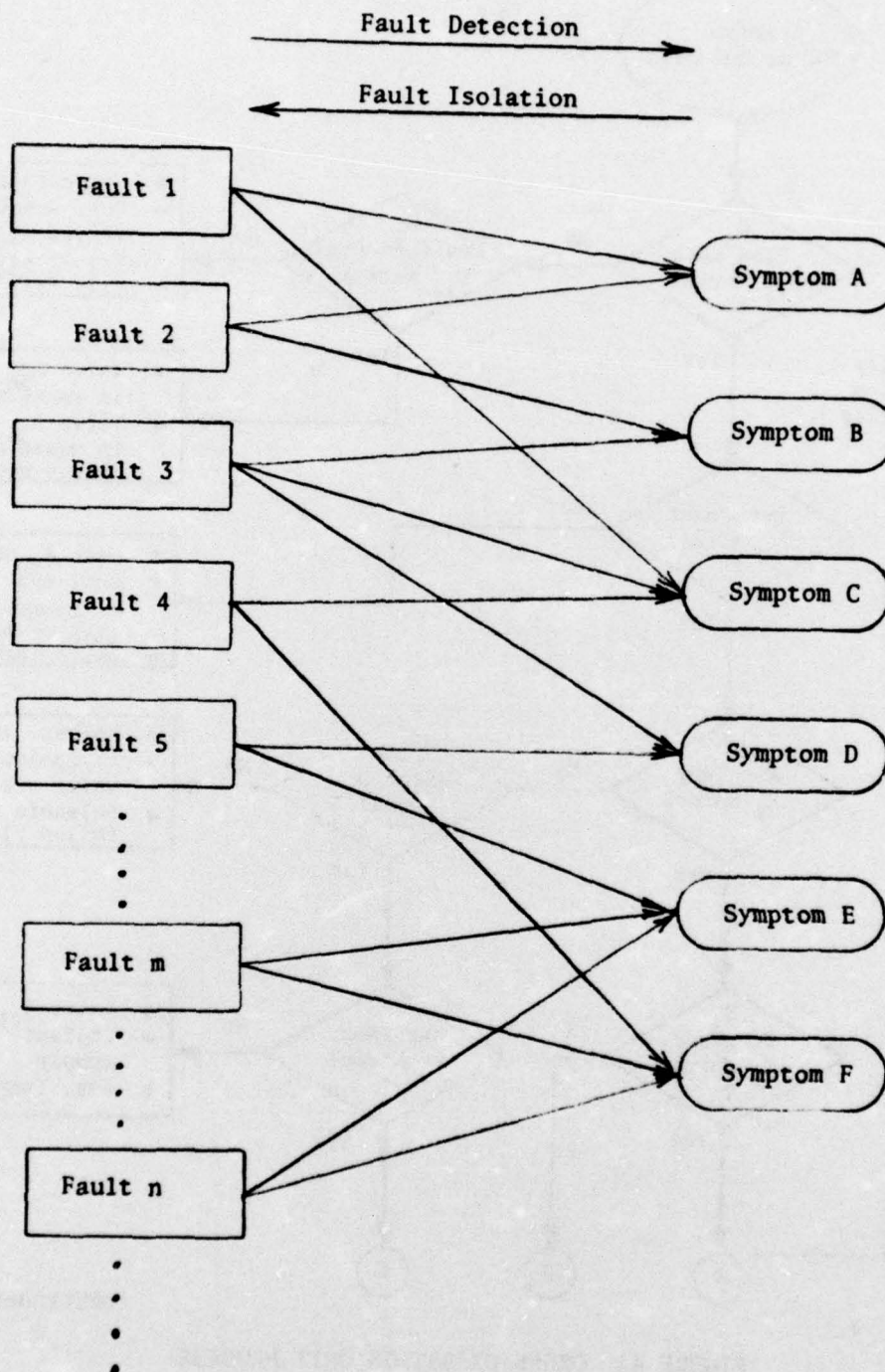


FIGURE 41 FAULT DETECTION AND ISOLATION RELATIONSHIPS



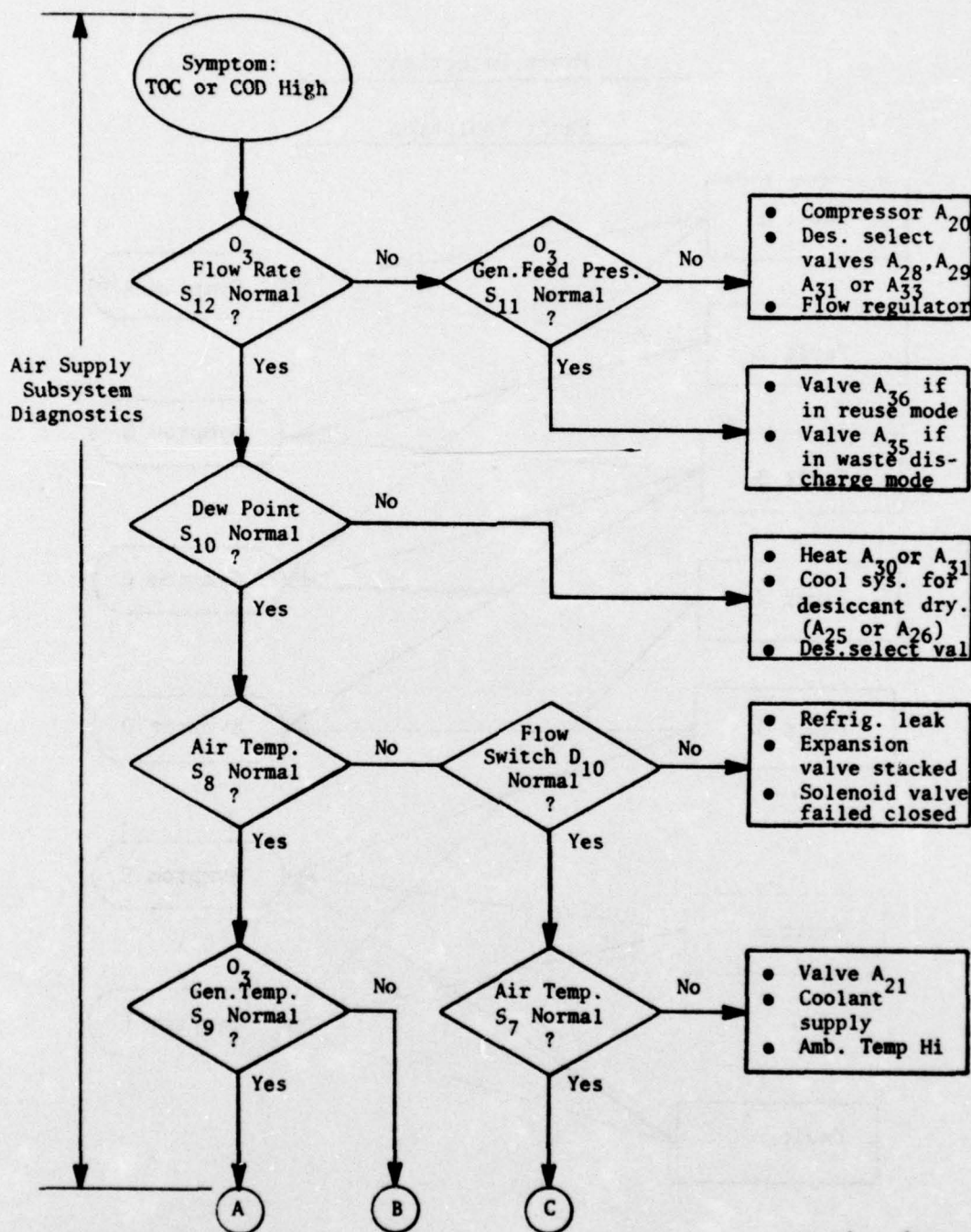
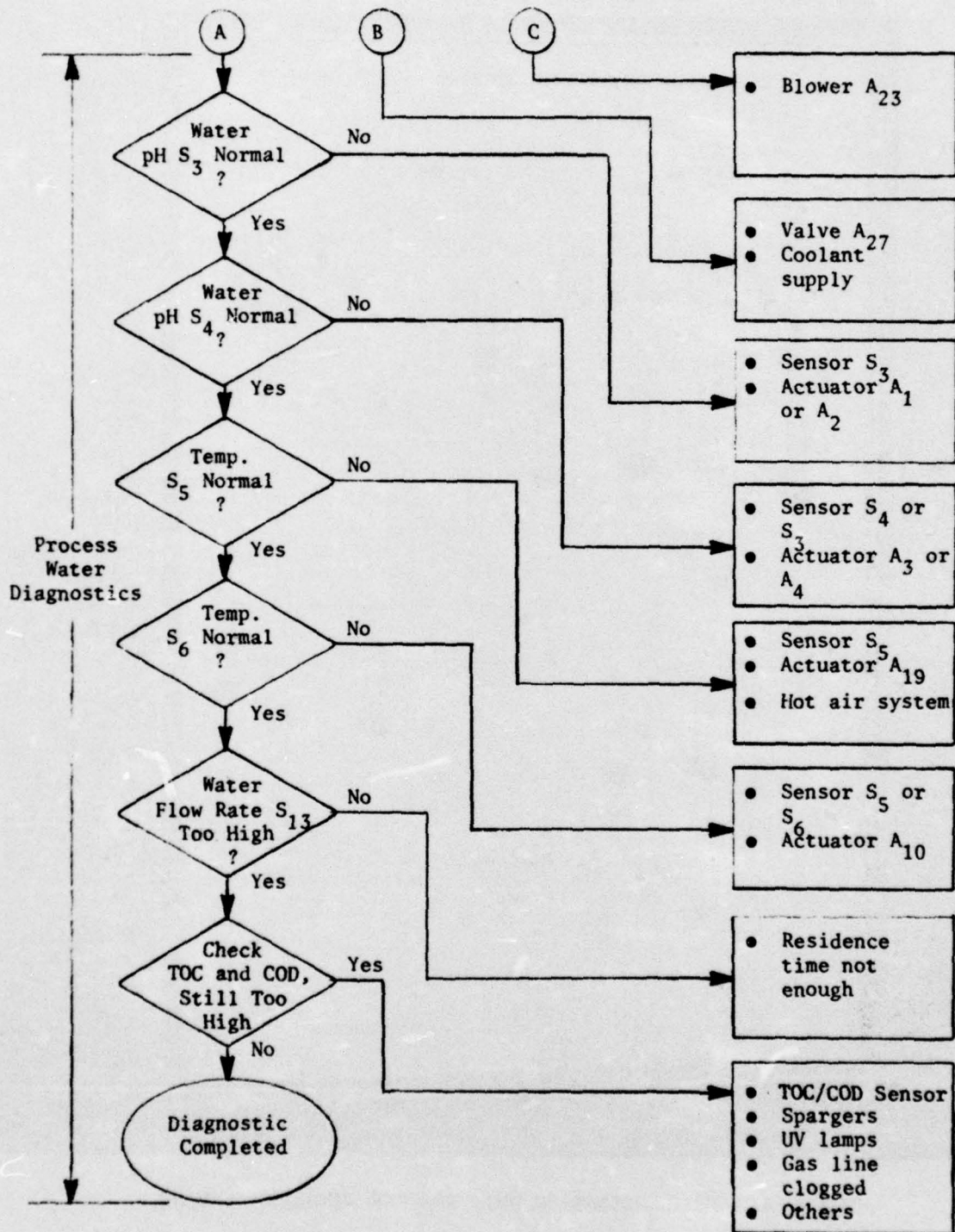


FIGURE 42 OZONE OXIDATION UNIT PROCESS  
DIAGNOSTICS EXAMPLE - TOC OR COD HIGH

Figure 42 - continued



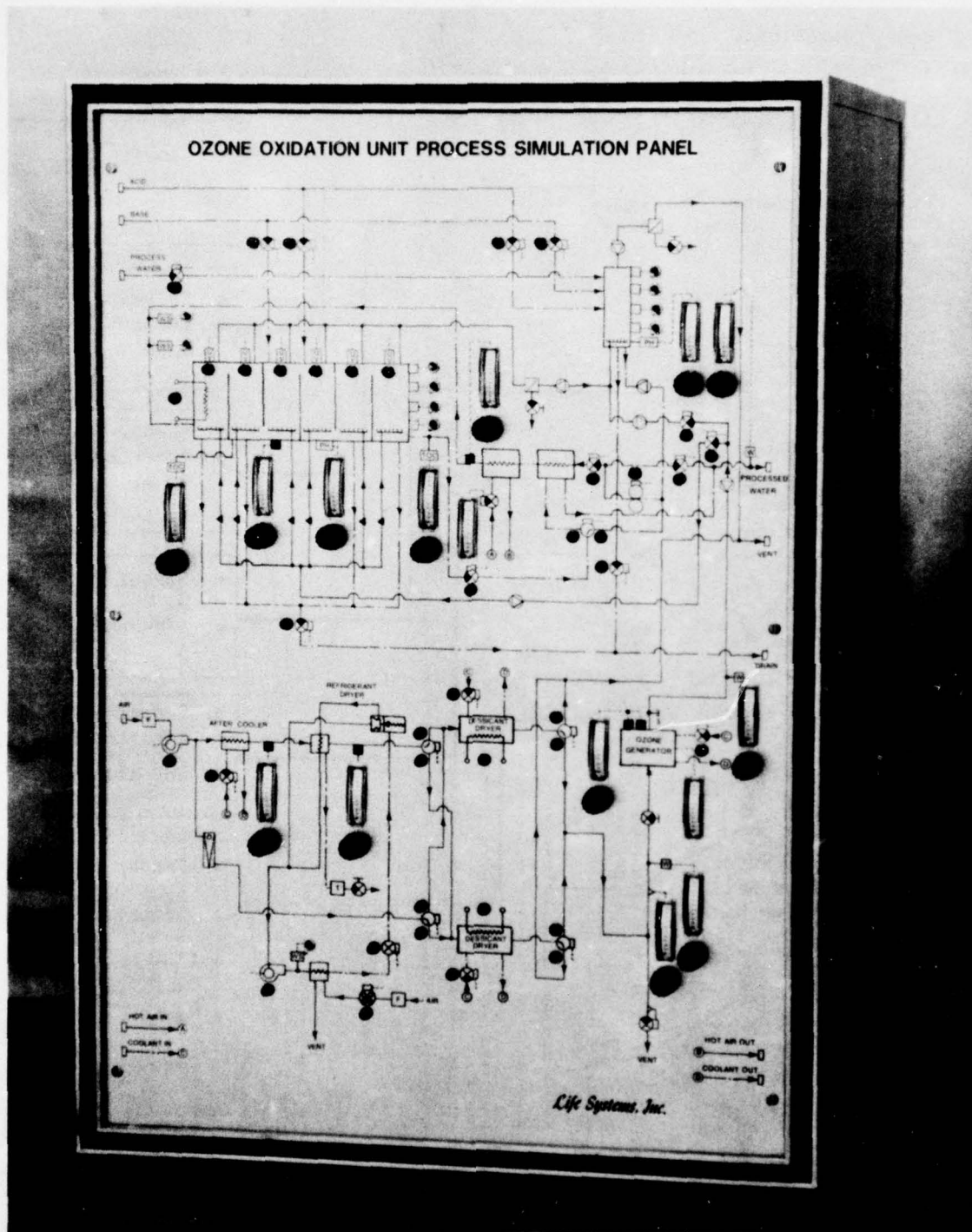


FIGURE 43 OZONE OXIDATION UNIT PROCESS SIMULATION PANEL



panel. The analog sensors (linear type) are simulated by a potentiometer with panel meter readouts; the digital sensors (on-off type) are simulated by toggle switches; the analog actuators such as  $O_3$  generator and heaters are simulated by panel meters; and the digital actuators by light-emitting diode (LED) indicators.

Table 20 shows the summary of  $O_3$  Oxidation Unit Process control and monitor programs implemented on a LSI-2<sup>3</sup> minicomputer. Table 21 presents a detailed description of the programs. These programs include a control/monitor panel service routine, an operating mode and mode transition control module input and output modules, individual control modules for pH, temperature, water level, TOC,  $O_3$  dosage and desiccant dryer control, a fault detection and trend analysis module, a message output module and miscellaneous tables and utility programs.

The instrumentation features incorporated in the simulation are operating mode control, mode transition control, operator/system interface simplicity, elimination of operator error, fault detection and trend analysis, system and personnel safety, direct digital control of various process parameters and flexibility.

#### WPE Pilot Plant Instrumentation Size

In the course of the LMTOC instrumentation development activities the MUST WPE instrumentation size was estimated. The study of WPE instrumentation size was necessary because it had an impact on the LMTOC instrumentation and vice versa. The size study included cost, maintainability, reliability, volume, power and weight estimates. As prerequisites the WPE instrumentation performance goals, its system protection requirements, the level of maintenance aids, design approach, required flexibility and instrumentation features were evaluated.

The expected WPE development will include a pilot plant, prototype, and pre-production models before full production. In the pilot plant phase, the instrumentation can be characterized as a fully automatic system with maintenance aids, extensive data acquisition capability, flexibility in design to allow testing of various control schemes and simple operator/system communication. The electronic hardware will be mostly off-the-shelf with only a basic packaging effort. In the prototype phase the data acquisition and testing flexibility features can be minimized and the electronics will be repackaged with custom-made components to reduce the size. In the pre-production phase, military specification electronics will be used and semiconductor memory be incorporated into the instrumentation. This evolution of WPE instrumentation is summarized in Table 22.

Because the WPE is mission oriented, both maintainability and reliability are very critical. Maintainability is defined as a characteristic of design which is expressed as the probability that an item will be retained in or restored to a specific condition within a certain period of time. Maintainability can be defined by the mean system downtime, which in turn includes mean service

TABLE 20 SUMMARY OF O<sub>3</sub> OXIDATION UNIT  
PROCESS CONTROL PROGRAMS

<u>Program</u>	<u>Size (Words)</u>
Control/Monitor Panel Service Routine (CMSRV)	258
Operating Mode and Model Transition Control (OPCON)	679
Input (ADIN)	58
Output (OUTIN)	45
Control Modules (CNT)	461
Fault Detection and Trend Analysis (FTDT)	257
FTDT Monitor Set Point Table	96
Message Output (MSG)	61
Message Vectors and Buffer	1,056
Control Set Point Table and I/O Buffers	48
Other Utility Programs and Tables	<u>256</u>
	TOTAL 3,275

TABLE 21 DESCRIPTION OF O<sub>3</sub>/UV UNIT PROCESS  
CONTROL/MONITOR SOFTWARE

Control/Monitor Panel Service Routine (CMSRV)

- Read Pushbutton Commands from the Front Panel
- Verify Command Validity
- Allow Product/Source Selections and Auxiliary Mode Selections in SHUTDOWN Mode Only
- Allow UF Clean, RO Clean, and IE Regeneration Modes Concurrently
- Allow One of the Potable Water Product Selections Running Concurrently with One of the Wastewater Treatment Selections
- Verify System Mode Transitions
- Generate Intermode Transitions Whenever Necessary

Operating Mode and Transition Control (OPCON)

- Select Unit Processes for Current Product/Source Mode (e.g., Select Ozone Oxidation Unit Process Only in Discharge B Source Mode or Reuse D Source Mode)
- Implement Steady State Operating Mode Control: Power Off, Shutdown, Standby, and Normal
- Implement Mode Transition Sequences: (1) Shutdown to Standby Transition, (2) Standby to Normal Transition, (3) Normal to Standby Transition, (4) Normal to Shutdown Transition, (5) Standby to Shutdown Transition, (6) Shutdown to Power Off Transition, (7) Power Up Transition (Power Off to Shutdown Mode)

Input and Output Modules (ADIN and ADOUT)

- Set Up Automatic Input Instructions at Interrupt Locations and Digital Data to Input Buffer (INBUF)
- Read in Analog
- Separate Digital Data from Analog Data and Store all Sensor Data in Sensor Table (SENTBL)
- Get Process Output Commands which include Digital and Analog Actuator Set Points



Table 21 - continued

- Manipulate and Store the Output Commands in Predetermined Format at the Output Buffer
- Set Up Automatic Output Instructions at the Interrupt Locations

Control Modules (CNT)

- Pre-Contactor pH Control (PPHC)
- Contactor pH Control (CPHC)
- Water Temperature Control (WTC)
- Contactor Temperature Control (CTC)
- Pre-Contactor Water Level Control (PWLC)
- Contactor Water Level Control (CWLC)
- Desiccant Dryer Control (DDC)
- TOC/COD Control (TOC)
- TOC/COD Control Feed Forward Control (TFF)

Fault Detection and Trend Analysis

- Get Current Sensor Data
- Scan the Monitor Set Point Table, Check High and Low Set Points for Performance Trend
- Get Summary of Faults and Update System Status
- Output Message to the Display Panel whenever a Fault is Detected
- Request System Shutdown if ALARM Situation is Detected
- Provide Flexibility for Enable/Disable All or Portion of the Fault Detection and Trend Analysis Functions
- Provide Flexibility for Set Point Changes

TABLE 22 EVOLUTION OF MUST WPE INSTRUMENTATION

Pilot Plant

- Fully Automatic Operation with Maintenance Aids
- Extensive Data Acquisition Capability
- Flexibility in Design to Allow Easy Changes of Control Schemes During Test Period
- Use Core Memory
- Use off-the-shelf Electronics, Basic Packaging Effort Only

Prototype

- Fully Automatic Operation with Maintenance Aids
- Operator/System Communication through a Control/Monitor Panel Only
- Use Core Memory
- Custom-made Electronics to Reduce Size
- Repackaged Electronics

Pre-Production

- Semiconductor Memory
- Military Specification Electronics

time for scheduled maintenance, mean fault isolation time, adjustment-calibration time, cleanup time, fault correction time, checkout time, inspection time, turn-around time for scheduled/unscheduled or preventive/corrective maintenance, and required tools and skill level for maintenance. Among these, the fault isolation and correction time and the required skill level are most critical for the WPE. Therefore, the instrumentation should incorporate self diagnostics and fault prevention features.

Reliability is usually measured by mean-time-between-failures (MTBF). With integrated circuit electronics, differences in instrumentation reliability are largely a function of the number of PC board interconnections. The industry trend has been in the direction of using more and more large-scale integration components and/or micro- and minicomputers to improve reliability. Such an approach would certainly have the best performance/cost ratio in achieving the maintainability and reliability goals in the WPE instrumentation.

The estimated volume of the pilot plant WPE control/monitor instrumentation is about 53 x 53 x 66 cm (21 x 21 x 26 in) or 0.19 m<sup>3</sup> (6.6 ft<sup>3</sup>). The memory size will be about 8K words, the weight will be about 90 kg (200 lb) and the electrical power consumption about 750W. These estimates do not include the sensors or actuators nor the TSA data acquisition unit. It does, however, include the capability for communication with a TSA computer which could be designed for data acquisition, TSA control/monitor, program modifications and conversational mode operator/system communication.

#### REVERSE OSMOSIS UNIT PROCESS

The RO Unit Process is needed in the MUST WPE to remove organic and inorganic solutes from the UF Unit Process permeate and brackish water feeds.

The mechanical and electrical hardware design, fabrication, checkout and shakedown testing of a RO Unit Process employing DuPont B-10 RO Modules were successfully accomplished in this program. The objectives were to (1) develop a semiautomatic RO Unit Process to produce RO permeate waters for the LMTOC testing and (2) make provisions for upgrading the RO to an automatic system capable of being integrated into the MUST WPE.

#### Hardware Design and Development

The RO Unit Process hardware design and development includes mechanical design, electrical design and unit process interface definitions. The RO Unit Process is shown in Figure 44.

The RO tank has level sensors and controls for maintaining levels. It is provided with a low level and a high level shutoff for its effluent and influent, respectively. A positive displacement piston pump provides the necessary pressure (5500 kN/m<sup>2</sup> (800 psig)) for the B-10 RO modules. The influent to the RO modules flows through parallel in-line 5μ and 1μ basket-type filters. Pressure drops across the filter and modules are monitored for predicting



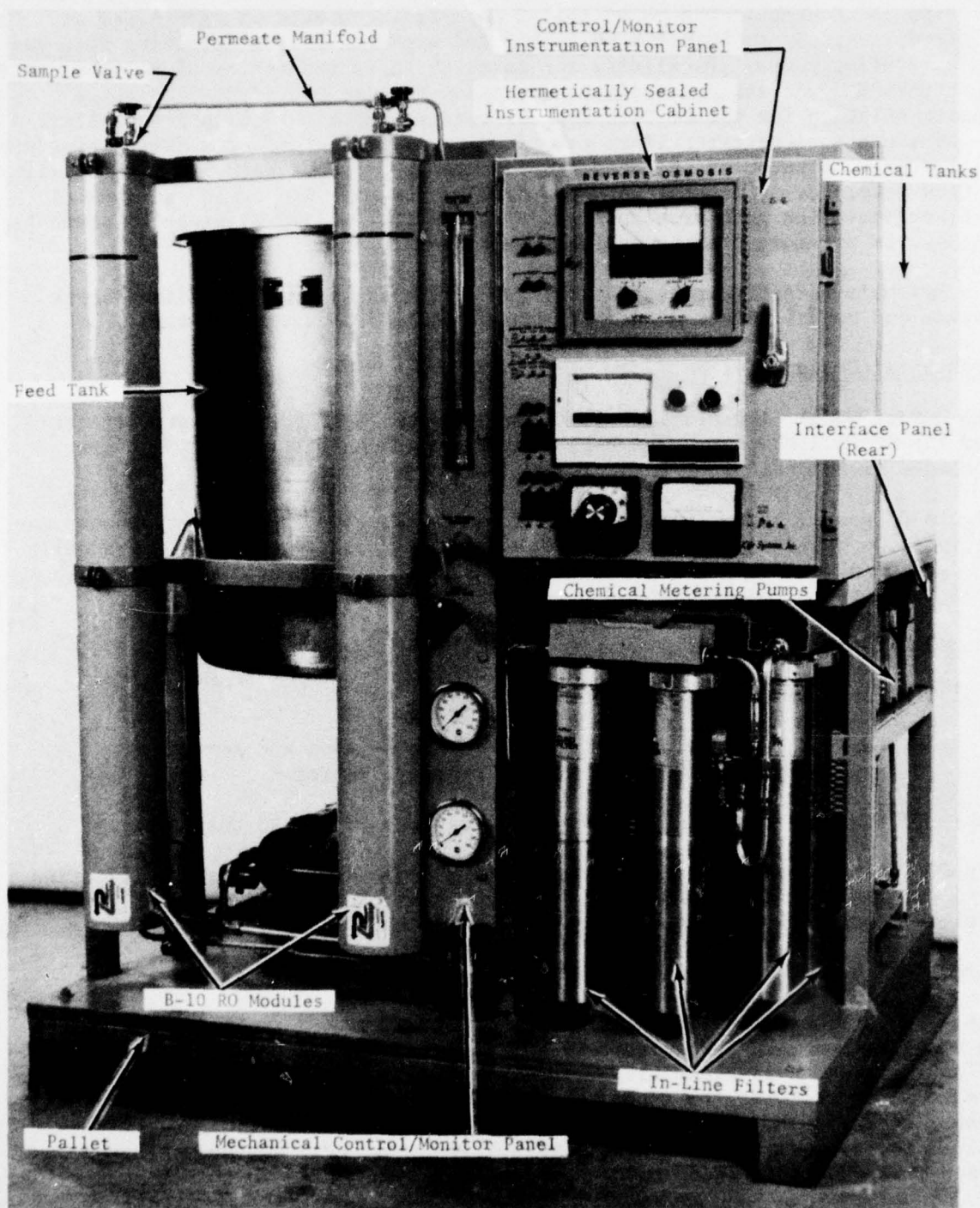


FIGURE 44 LIFE SYSTEMS' RO UNIT PROCESS

routine maintenance. The pH of the RO feed tank contents is maintained at desired levels by an immersed pH sensor and a pH controller operating acid and base metering pumps. Facilities for metering known quantities of polyphosphate into the RO feed tank for scaling prevention is also provided. The basic instrumentation for the RO Unit Process consists of a pH monitor/controller, a temperature monitor/controller, a RO feed pressure indicator, a filter pressure differential transducer, a conductivity meter to monitor permeate water quality, a flow meter for monitoring permeate flow, a low pressure switch to prevent the recirculation pump from running dry and a high pressure switch to prevent the system pressure from exceeding a safe limit.

The system's mechanical controls consist of a backpressure regulator, check valves and manual valves needed for proper and safe system operation.

#### Mechanical Design

The DuPont Model 6440-015 10.2 cm (4 in) diameter B-10 modules were selected for the RO Unit Process. The B-10 module specifications and drawings are included in Appendix 3 of this report.

Figure 45 shows the schematic of the RO Unit Process. The RO Unit Process was designed to operate in a batch, semicontinuous or continuous mode, with daily production of at least 8,000 liters (2,100 gallons) permeate water when two B-10 modules are employed in series.

Mechanical design considerations for the RO Unit Process include:

1. safe system operation
2. minimum power, weight and volume
3. easy access to controls, monitors, interfaces and servicing
4. system pressure regulation and pressure relief

Table 23 presents the primary mechanical features of the RO Unit Process.

The RO Unit Process specification is given in Appendix 4. This specification outlines the operating conditions, physical characteristics, material characteristics, electrical characteristics and interfaces.

#### Electrical Design

The RO Unit Process instrumentation was designed to be a semiautomatic system to meet the functional requirements for system operation, performance, safety, reliability and maintainability with minimal cost. Automatic control was employed in controlling the water pH, water level, pumps and water temperature. Manual controls and/or overrides of pumps, chemical tank (acid/base/polyphosphate) valves and process stream solenoid valves are provided. An automatic shutdown feature is incorporated to protect the system. The shutdown conditions include:

1. feed tank water level high or low alarm
2. conductivity over-range alarm

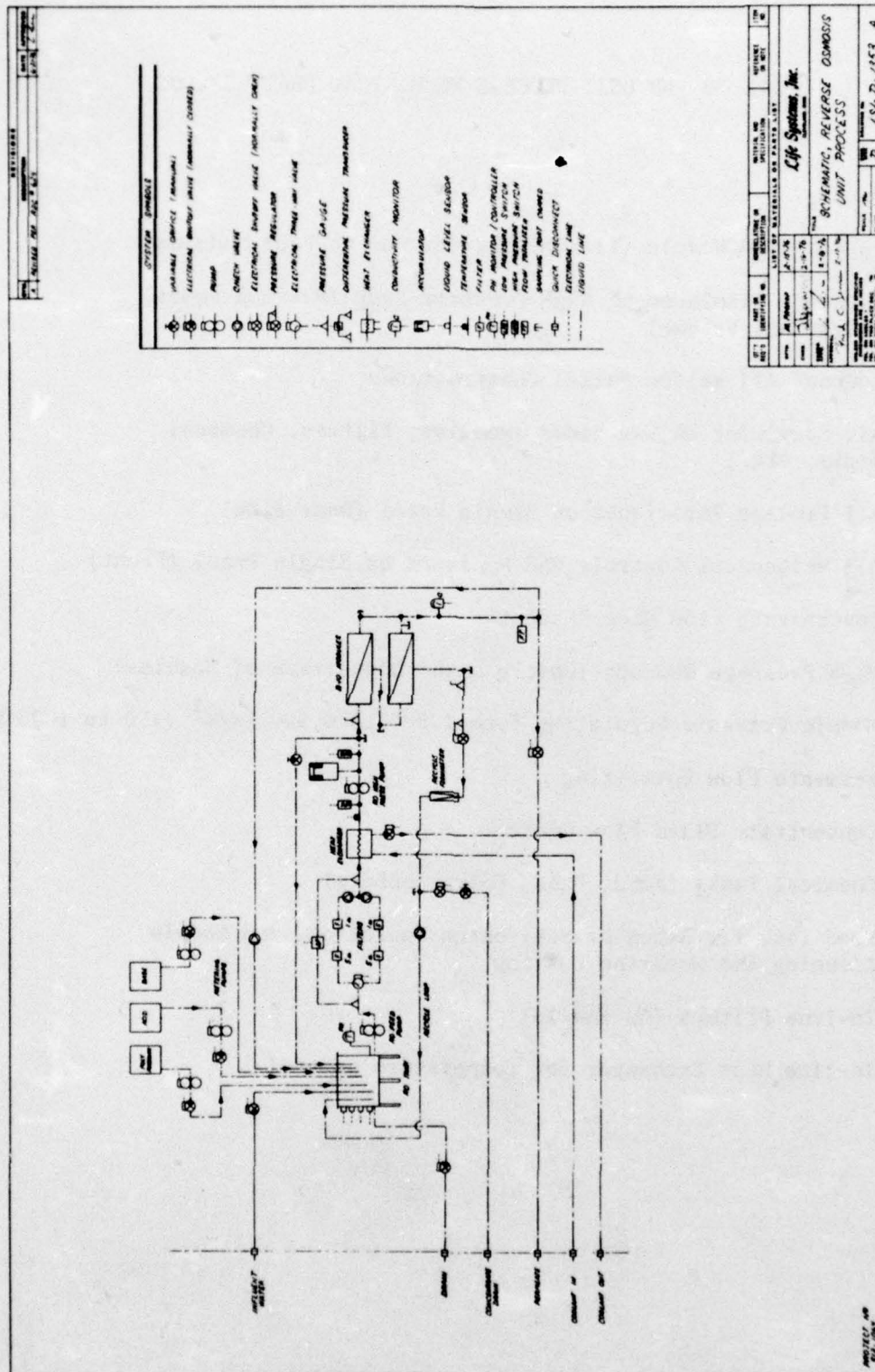


FIGURE 45 REVERSE OSMOSIS UNIT PROCESS SCHEMATIC



TABLE 23 RO UNIT PROCESS MECHANICAL FEATURE LIST

- DuPont B-10 Module (Flexibility for One to Four Modules)
- Positive Displacement High Pressure Pump (Minimum Power, Weight and Volume)
- Compact All Welded Pallet Constructions
- All Servicing on Two Sides (Modules, Filters, Chemical Tanks, etc.)
- All Process Interfaces on Single Panel (Rear Side)
- All Mechanical Controls and Monitors on Single Panel (Front)
- Concentrate Flow Rate Readout
- High Pressure Readout (Upstream and Downstream of Modules)
- Module Pressure Regulation from 1,000 to 6,900 kN/m<sup>2</sup> (150 to 1000 Psi)
- Permeate Flow Totalizing
- Concentrate Bleed Flow Control
- Chemical Tanks (Acid, Base, Polyphosphate)
- Feed Tank for Batch or Semi-batch Operation, and Module Cleaning and Membrane Coating
- In-line Filters (5μ and 1μ)
- In-line Heat Exchanger for Temperature Control

3. low pressure alarm
4. high pressure alarm

Electrical monitor readouts are provided for feed tank liquid level, pH, temperature, filter bank pressure differential, high/low pressure, and conductivity.

Table 24 summarizes the RO Unit Process electrical instrumentation features. Figure 46 is a photograph of the RO mechanical and electrical monitor/control panels.

#### Reverse Osmosis Test Stand Interfaces Definitions

Figure 47 shows the block diagram of the RO unit process interfaces. The definitions of the interfaces are listed in Table 25.

The interfaces primarily include influent water, product water, drain, brine bleed, chemical additives, electric power, coolant supply and maintenance supplies. The chemical additives include the chemicals necessary to produce synthetic waste water for running the experiments, the acid and base solutions for pH control and the polyphosphate for prevention of calcium sulfate ( $\text{CaSO}_4$ ) scaling. The maintenance supplies are filters, RO module cleaning and coating solutions (PT-A, PT-B), lubricants, etc.

#### Reverse Osmosis Experimental Results

A series of experiments were conducted to test the operational characteristics of the RO Unit Process. These experiments included:

1. Checkout tests of mechanical and electrical components.
2. Shakedown testing which included experiments of RO productivity, sodium chloride (NaCl) rejection rate and temperature effects on permeate flow rate.
3. Measurement of noise level and electrical power consumption.

#### Checkout Tests

Tests were conducted to ensure that all components of the RO Unit Process met the operational requirements. These tests included the pump capacity tests, high pressure line checkout, sensor calibration and tests with artificial alarm conditions. All components checked out as designed. During the checkout tests it was determined that a positive pressure is required at the inlet to the positive displacement high pressure pump. The manufacturer of the high pressure pump specified that it can operate without cavitation with an inlet pressure as low as  $-47.2 \text{ kN/m}^2$  (-8 psig). With a negative inlet pressure, tests revealed that pump cavitation occurs and results in excessive pressure fluctuations ( $517.1 \text{ kN/m}^2$  ( $\pm 75$  psig)).

TABLE 24 RO UNIT PROCESS ELECTRICAL FEATURE LIST

- All Controls and Monitors on Single Panel (Front)
- Automatic High Pressure Shutdown and Readout
- Automatic Low Pressure Shutdown and Readout
- Automatic Permeate High Conductivity Shutdown and Readout
- Automatic pH Control (Acid and Base) and Readout
- Filter  $\Delta P$  Readout
- Water Level Control
- High and Low Water Level Shutdown and Readout
- Automatic Process Water Temperature Control and Readout
- Polyphosphate Metering
- Manual Overrides on All Pumps and Valves
- Motorized Filter Bank Selection
- All Electronics Hermetically Sealed in Cabinet
- All Wiring Contained in Frame or Conduit



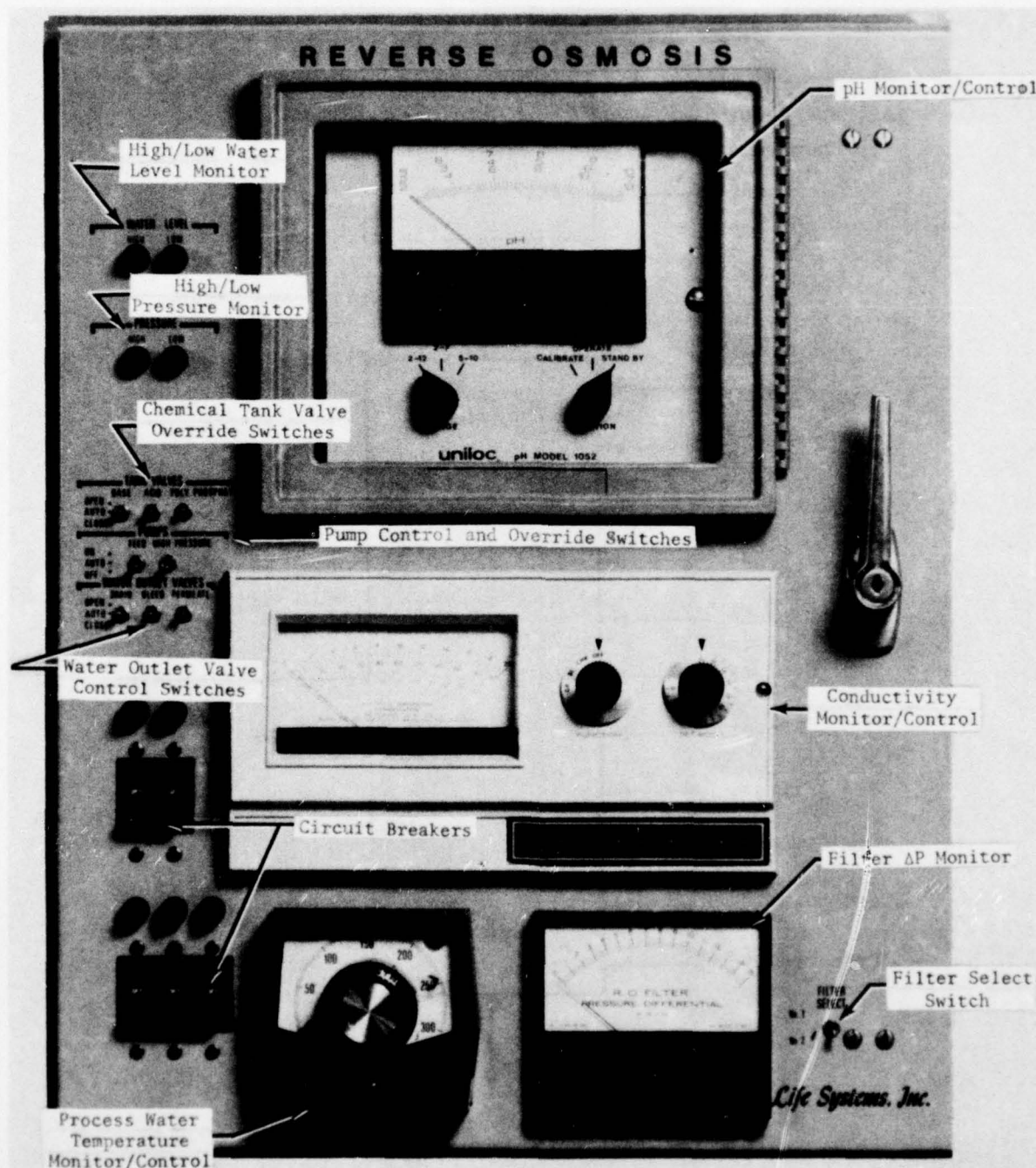


FIGURE 46 RO UNIT PROCESS INSTRUMENTATION PANEL

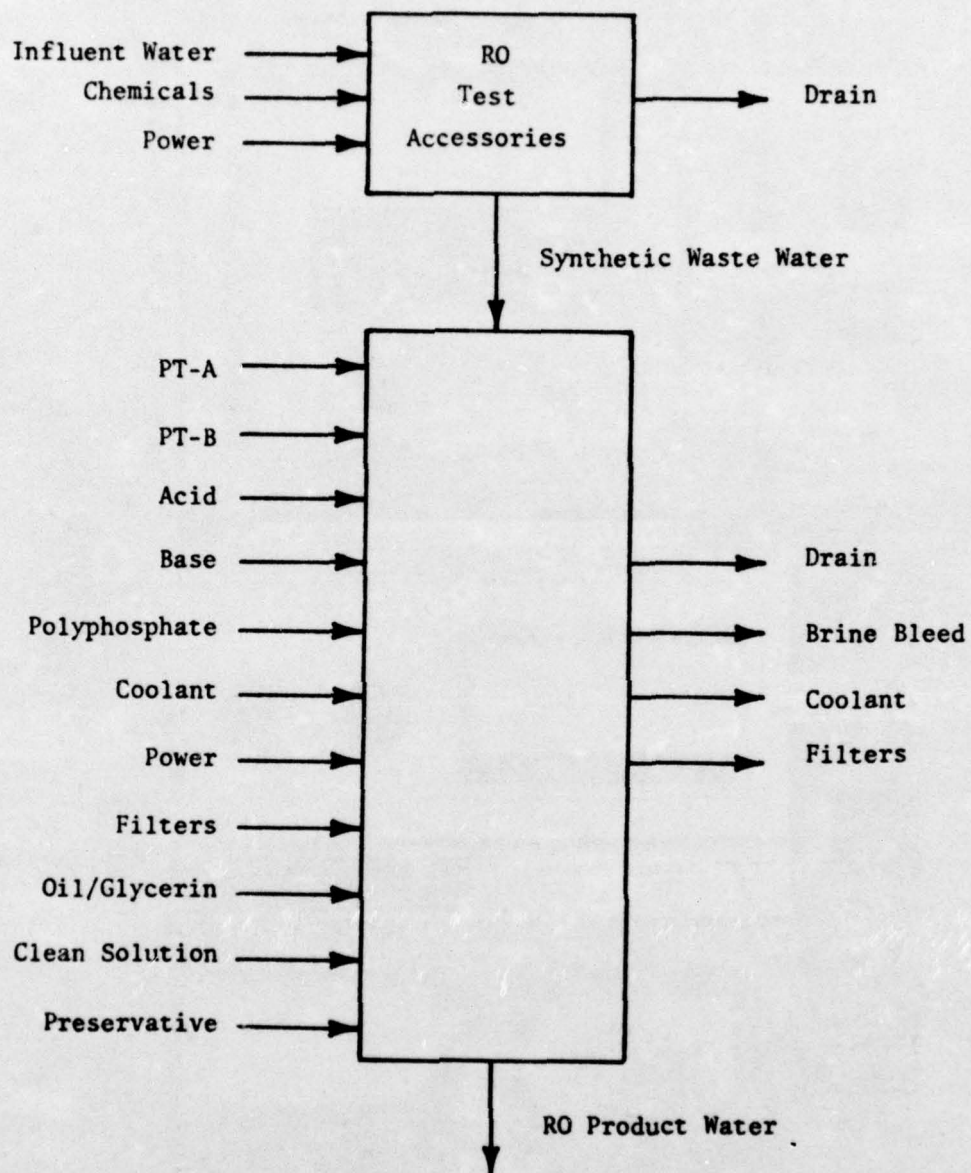


FIGURE 47 RO INTERFACE BLOCK DIAGRAM

TABLE 25 RO UNIT PROCESS INTERFACE DEFINITIONS

Interface	Definitions
PT-A <sup>(a)</sup>	80 Ppm Polyvinyl Methyl Ether
PT-B <sup>(b)</sup>	80 Ppm Tannic Acid and 1% Citric Acid
Acid	2N $H_2SO_4$
Base	2N NaOH
Polyphosphate	Sodium Hexametaphosphate, $(NaPO_3)_6$
Coolant	Water, 23 l/Min @ 286K (6 Gpm @ 55F)
Power	208V, 3 Phase With Neutral, 60 Hz, 7 kW
Filters	Cartridge Micro-WYNDII D-PPTY and D-PPTB
Oil <sup>(b)</sup>	Standard, Non-Detergent
Preservative <sup>(c)</sup>	Technical Grade Glycerin and Formaldehyde
Cleaning Solutions, Organic Fouling	NAOH/ $N_2O$ <sup>(d)</sup>
Cleaning Solutions, Inorganic Fouling	0.25% Biz and 1% Citric Acid
Drain	Standard 10 cm (4 In) Floor Drain
Brine Bleed	Variable <sup>(e)</sup> 0.038 to 1.5 l/m (0.1 to 0.4 Gpm)
Product Water	Variable <sup>(e)</sup> 3.79 to 13.25 l/m (1 to 3.5 Gpm)

(a) DuPont trade name (Post Treatment)

(b) Used for high-pressure pump (1¼ quarts/500 operating hours)

(c) Used for B-10 module preservation (17.5% Wt glycerin and 1.5% Wt)

(d) (pH of solution should be maintained between 10 and 11, but should not exceed 11)

(e) Function of the number of RO modules on line



#### Shakedown Testing

Figure 48 shows the results of a continuous RO productivity and its NaCl rejection test for a single B-10 module for more than one hundred hours of operation. The permeate flow was maintained between 6.6 l/min (1.75 gpm) and 8.3 l/min (2.2 gpm) and the NaCl rejection rate was 98% or higher over the entire testing period.

Figure 49 shows the effect of temperature on the RO permeate flow rate. An increase in the permeate flow rate from 9.5 l/min (2.5 gpm) to 13.6 l/min (3.6 gpm) was observed when the temperature of feed water increased from 294 to 310K (70 to 98F). The permeate flow rate data was corrected for the change in water viscosity. The results correlate with the theory that the increase in permeate flow rate was attributed to the change in process water viscosity.

#### Noise and Power Measurement

The RO Unit Process sound level was measured in a 9.5 x 10.5 (31 x 34 ft) room with hard walls and a 6 m (20 ft) high ceiling. The sound levels of the unit process were measured from two locations about 1.8 m (6 ft) from the high pressure pump (the primary noise source) as shown in Figure 50. The readings of location A and B were 87 dbA and 86 dbA, respectively, when the RO Unit Process was in normal operation. Because the RO Unit Process was located very close to two hard walls when the readings were taken, the actual sound level without the reflections would be lower in a "soft" room. However, the sound levels measured are probably representative of the levels in a MUST ward container without any noise suppression. Since these measurements exceed the allowable level (85 dbA) some noise suppression will most likely be needed for the WPE pilot plant and future developments.

The RO electrical power consumption of each major electrical component was measured and the results are shown in Table 26. The total RO Unit Process power consumption under normal operation is approximately 6 kW.

The problems identified in the testing period included:

1. High pressure pump flexible line fatigue.
2. Pressure gauge oscillations due to high pressure pump feed pressure fluctuations.
3. Cavitation at high pressure pump inlet.
4. Leakage and plugging of the flow totalizer.
5. Electrical relay failure.

These problems were resolved during the course of the program activities.

Modifications of the RO Unit Process were made according to the results of the checkout and shakedown testing.

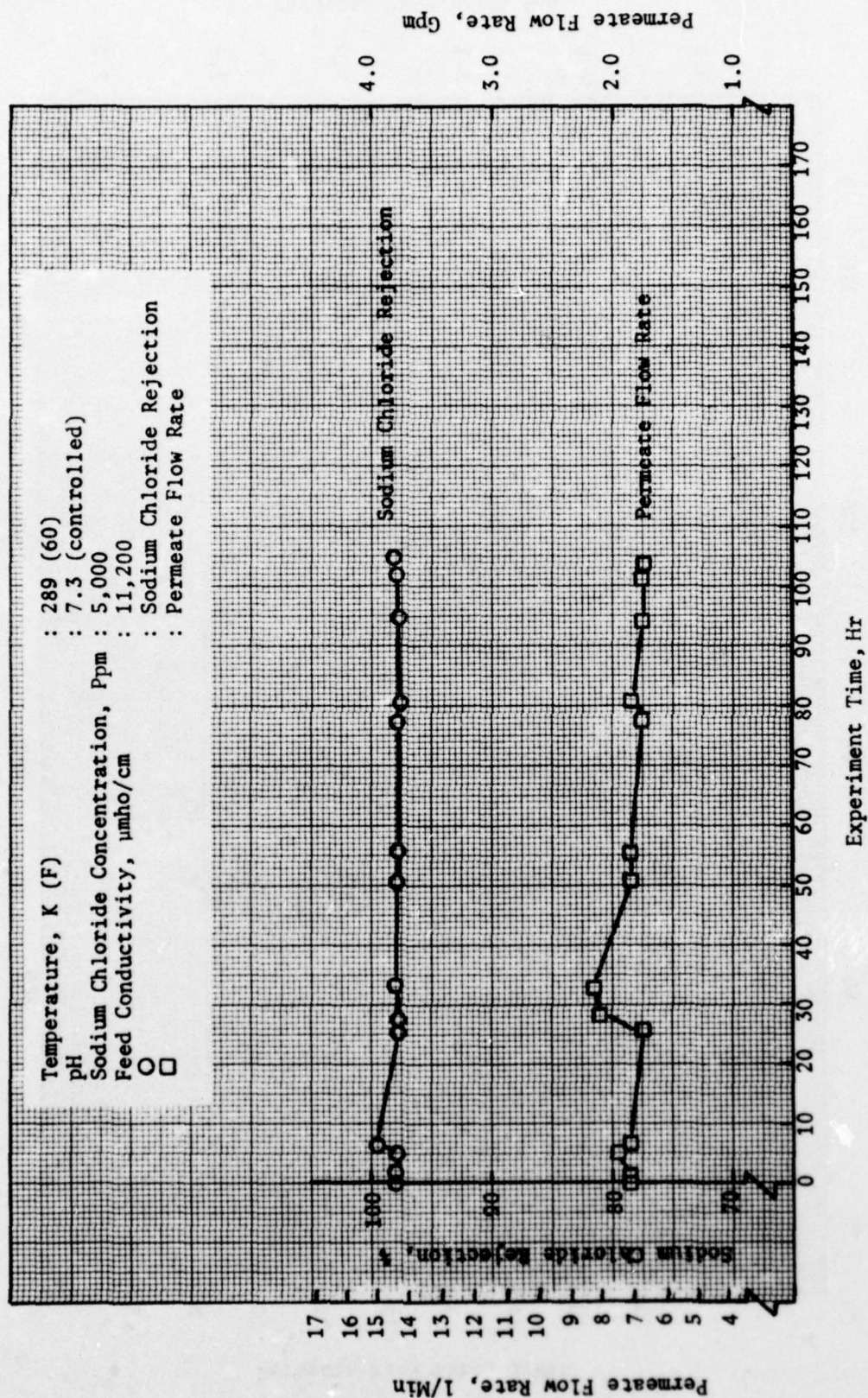


FIGURE 48 RO UNIT PROCESS SHAKEDOWN TEST RESULTS



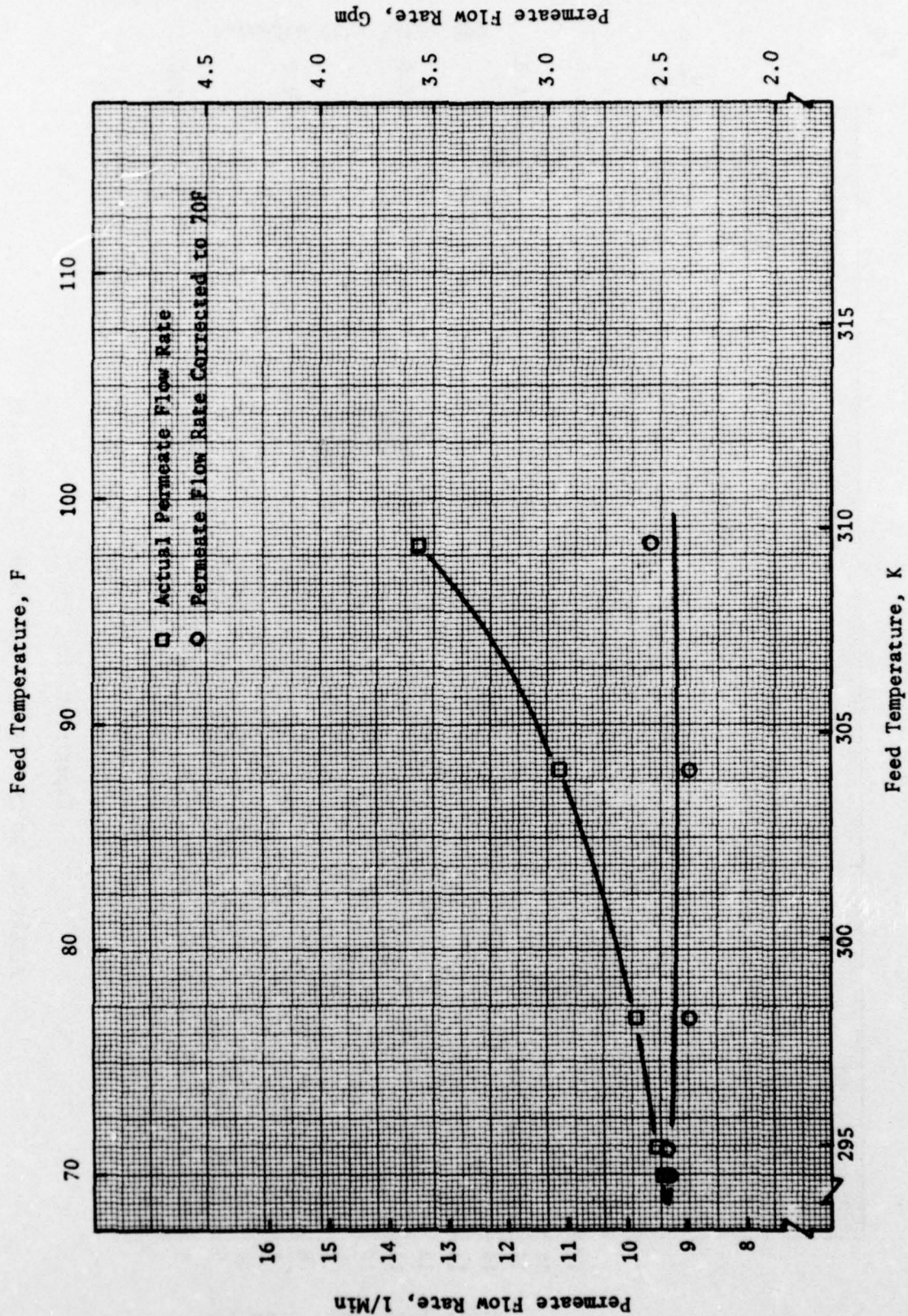


FIGURE 49 RO TEMPERATURE EFFECT ON PERMEATE FLOW RATE



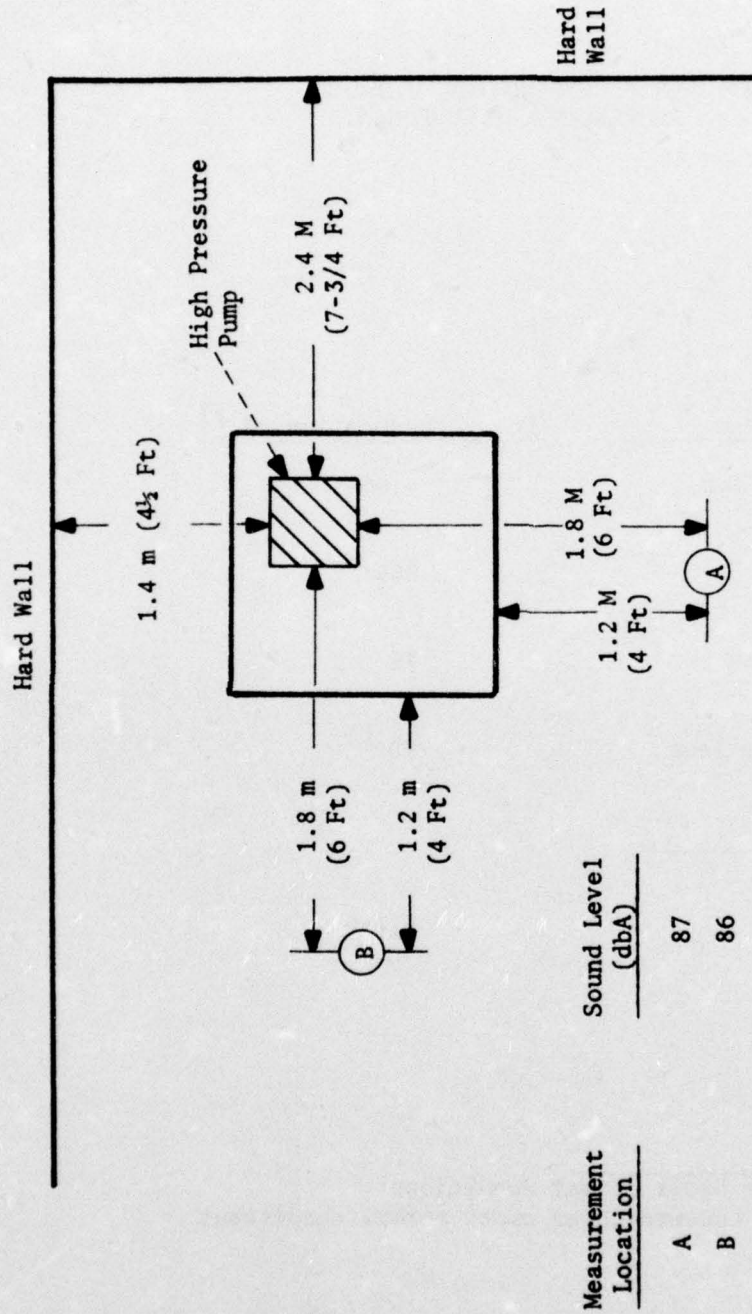


FIGURE 50 SOUND LEVEL MEASUREMENT OF THE RO UNIT PROCESS

TABLE 26 POWER CONSUMPTION OF MAJOR  
RO ELECTRICAL COMPONENTS

<u>Component</u>	<u>Power Consumption, W<sup>(a)</sup></u>
High Pressure Pump	4,800
Feed Pump	585
Instrumentation	75
Solenoid Valve (each)	20 <sup>(b)</sup>
<hr/>	
Total	5,460

(a) Operating under normal conditions

(b) Does not consume power under normal conditions

## CONCLUSIONS

The primary goal of this research was to develop an LMTOC system to be a post-RO treatment process for the MUST WPE. A secondary goal was to study and design the instrumentation which would be required for controlling and monitoring the LMTOC under fully automated operation. In the course of developing the LMTOC it was necessary to design and fabricate an RO Unit Process to produce RO permeate for the testing of the LMTOC. These goals were successfully achieved and the following conclusions are drawn from this research:

1. The LMTOC has succeeded in reducing the organic solute concentrations in the RO laboratory and composite waste water permeate to meet the required water quality specifications of less than 5 mg/l TOC and 10 mg/l COD.
2. The COD is the limiting factor in meeting the water quality specifications. In the experiments conducted, a longer residence time was required to reach the 10 mg/l COD specification than the 5 mg/l TOC specification. This implies that COD sensing instead of TOC sensing should be used in the feedforward/feedback loops to control the  $O_3$  generator. A practical on-line water quality sensor for the automatic control of LMTOC is still not available today. The control/monitor algorithms, however, would remain nearly the same regardless of the choice in the water quality sensors.
3. In the UV activated LMTOC no pH or temperature control is needed for treating the composite waste RO permeate. A typical composite RO permeate, at pH 9 and temperature 303K (86F), can be readily and directly reduced to below 5 mg/l TOC at an  $O_3$  dosage of 1.04 mg/min/l of process water. The 5 mg/l TOC specification was met in the third of the six LMTOC stages in less than two hours of residence time.
4. High  $O_3$  conversion was observed with the LMTOC. Under typical operating conditions of the LMTOC the  $O_3$  conversion rate is between 96 and 100%.
5. In treating MUST laboratory waste RO permeate a much longer residence time is required compared to treating composite RO permeate. To meet the TOC and COD specifications, approximately 3-3/4 hours are required at an  $O_3$  dosage of 7.9 mg/min/l (0.095 lb/day/gal) of wetted contactor volume.
6. In the ethanol oxidation experiment with the LMTOC, the TOC can be reduced from 58 mg/l to less than 5 mg/l in approximately three hours at an  $O_3$  dosage of 3.77 mg/min/l (0.045 lb/day/gal) of wetted contactor volume. This represents a significantly lower power consumption than previously experienced. In direct comparison with two other  $O_3$  reactors the LMTOC consumes 50% or less total energy.



7. The UV intensity in the process water reduces rapidly as the distance from the lamps increases. Test results showed that the UV intensity reduced 95% in a distance of 13 cm (15 in) in synthetic MUST laboratory waste water. For effective utilization of UV the water must be kept very close to the lamps.
8. Precipitation of salts assumed to be calcium and magnesium sulfates and carbonates was observed in the LMTOC during the post-experimental inspection. However, in the WPE this problem could be eliminated by use of sodium polyphosphate or the IE Unit Process.
9. The oxidation of the LMTOC contactor welds indicate that heat treatment is needed. In future development stainless steel with lower carbon (e.g., SS 316L) should be investigated.
10. The feasibility of advanced control and monitor instrumentation for the  $O_3$  Oxidation Unit Process was demonstrated in the minicomputer-based instrumentation. Considering the maintainability, reliability, cost, weight, volume and power consumption, a minicomputer or microcomputer based instrumentation will have the best cost/performance ratio in controlling and monitoring the  $O_3$  Oxidation Unit Process as well as the WPE for fully automated operation.
11. The RO Unit Process B-10 module maintained a NaCl rejection rate of 98% and a permeate flow between 6.6 l/min and 8.3 l/min during a 100 hour continuous shakedown test.
12. A flexible line fatigue problem was encountered in the shakedown testing of the RO Unit Process. Further investigation of this problem is needed in future programs.
13. The power consumption of the RO unit process is approximately 6 kW. The sound level is close to the 85 dbA maximum level recommended by the MUST WPE specification. Therefore, noise suppression is likely to be needed for the WPE pilot plant and future development.

#### RECOMMENDATIONS

The experimental data of this program showed promise for the LMTOC in meeting the water quality criteria for the MUST WPE. The feasibility of advanced control and monitor instrumentation for the automation of the  $O_3$  Oxidation Unit Process has been demonstrated by the minicomputer controlled simulation panel developed in this program. In summary, this research program has dealt with essential issues in engineering technology relating to reduction of organic compounds by  $O_3$ /UV oxidation. Future studies relating to or growing out of this research should include technical improvement in the LMTOC test stand, additional experiments for further optimization of the unit process and additional work to completely implement and demonstrate the advanced instrumentation capabilities for the  $O_3$  Oxidation Unit Process as well as the WPE.

The following items are recommended by Life Systems, Inc. for further study:

1. Extend the LMTOC testing to increase the data base for studying the effects of operating parameters on different MUST waste waters (e.g., pH, temperature,  $O_3$  dosage, UV light intensity and  $O_2$  flow rate on the reduction of TOC and COD). Not all parametric effects have been studied on the MUST laboratory waste organic solute concentration reduction. No studies have been conducted on waste waters other than MUST composite and laboratory waste water.
2. Modify the LMTOC pre-contactor to introduce a supplemented compressed air supply into the pre-contactor for the studies of physical removal of organics by gas stripping. The stripping effect is believed to take place when volume of gas per unit volume of liquid per minute is high enough (e.g., >1). Stripping may be an effective means of reducing the organic solute concentrations as a pretreatment step to the UV/ $O_3$  Unit Process.
3. Establish best sparger material identified as one able to provide the small bubble size for good  $O_3$  mass transfer and avoid  $O_3$  auto-decomposition. Sparger materials to be studied include epoxy-coated fiberglass and sintered polyethylene. Different sparger materials will result in a better  $O_3$  utilization and lower power requirement for the  $O_3$ /UV Unit Process.
4. Establish the  $O_3$ -in-water concentration as a function of column height, including studies to establish the oxidizing specie ( $O_3$ ) as a function of waste water and not another oxidant. Carry out an  $O_3$  mass balance to arrive at a complete  $O_3$  utilization picture to determine areas where  $O_3$  is not efficiently used.
5. Establish UV light requirements as a function of distance between lights, frequency of wave length relative to contaminants adsorption frequency and type of contaminant.
6. Perform experiments to study the effect of organic solute concentration reduction with the addition of catalysts. Catalysts may increase organic solute concentration reduction rates on the initial stages of the LMTOC.
7. Carry out endurance testing of the LMTOC to determine the areas where maintenance will be needed to maintain efficient operation.
8. Complete the implementation of the advanced control and monitor instrumentation, including on-line set point modifications, conversion of data into engineering units and fault diagnostics, including dynamic performance trend analysis, fault detection, fault isolation, fault correction instructions and fault tolerance.
9. Include system maintenance data into the operator/system display to simplify training.



10. Establish the operator/system keyboard interface for communicating with the system's control/monitor instrumentation.
11. Implement the next step in the development sequence to convert the current instrumentation into a custom-packaged design based on microprocessor technology.
12. Expand the testing of the RO Unit Process to gain extended operating experience and identify problems with system components exposed to long periods of operation.

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APPENDIX 1

HEAD SPACE, NITRATE, NITRITE, UREA,  
CALCULATED TOC AND MEASURED TOC RESULTS

TABLE A1-1 HEAD SPACE, NITRATE, NITRITE, UREA, CALCULATED TOC AND MEASURED TOC RESULTS

Experiment (a) and Sample Port Number	CH <sub>3</sub> OH (Methanol) as TOC, mg/l		C <sub>3</sub> H <sub>6</sub> O (Acetone) as TOC, mg/l		Urea as TOC, mg/l		Nitrate + Nitrite as N		Total Calculated TOC, mg/l		TOC Measured mg/l	
A - 02	10.3		5.9		2.8		-		19.0		11.6	
A - 03	6.6		5.0		2.7		-		14.3		10.5	
A - 04	3.6		3.0		2.7		-		9.3		8.3	
A - 05	1.6		1.9		2.7		-		6.2		6.1	
A - 06	1.4		1.1		2.8		-		5.3		6.0	
A - 07	0.5		0.7		2.8		-		4.0		5.7	
A - 08	<0.3		0.4		2.8		-		<3.5		4.8	
A - 09	<0.3		0.2		2.7		-		<3.2		4.3	
B - 01	8.2		6.5		2.7		-		17.4		15.0	
B - 02	4.0		4.2		2.8		-		11.0		9.4	
B - 03	1.7		2.4		3.0		-		7.1		4.4	
B - 04	1.2		1.8		2.8		-		5.8		2.8	
B - 05	0.7		0.8		2.7		-		4.2		2.6	
B - 06	<0.3		0.5		2.7		-		<3.5		1.9	
B - 07	<0.3		0.3		2.7		-		<3.3		1.2	
C - 01	8.9		5.3		2.7		-		16.9		14.4	
C - 02	2.5		3.3		2.7		-		8.5		9.0	
C - 03	1.2		1.5		2.8		-		5.5		4.9	
C - 04	0.2		<0.005		2.5		-		2.7		1.9	
C - 05	<0.3		<0.005		2.3		-		<2.6		1.5	
C - 07	<0.3		<0.005		1.7		-		<2.0		1.4	
D - 01	9.7		6.2		2.7		-		18.6		14.2	
D - 02	-		-		2.7		-		-		9.5	
D - 03	0.2		0.1		2.7		-		3.0		2.4	
D - 04	0.5		0.1		2.7		-		3.2		1.6	
D - 05	<0.3		<0.005		2.7		-		<3.0		2.4	
D - 06	<0.3		<0.005		2.5		-		<2.8		2.6	
D - 07	<0.3		<0.005		2.6		-		<2.9		2.4	

continued -

(a) See Definition of Experiments in Table A1-2.



Table Al-1 - continued

Experiment (a) and Sample Port Number	CH <sub>3</sub> OH (Methanol) as TOC, mg/l	C <sub>3</sub> H <sub>6</sub> O (Acetone) as TOC, mg/l	Urea as TOC, mg/l	Nitrate + Nitrite as N	Total Calculated TOC, mg/l	TOC Measured mg/l
E - 01	9.8	4.9	2.8	-	17.5	15.8
E - 02	3.0	3.5	2.8	-	9.3	15.2
E - 03	0.8	1.4	2.8	-	5.0	8.5
E - 04	<0.3	<0.2	2.8	-	<3.2	6.6
E - 05	<0.3	<0.005	3.4	-	<3.7	5.3
E - 06	<0.3	<0.005	3.0	-	<3.3	4.8
F - 02	7.2	6.1	2.7	<0.05	16.0	14.3
F - 03	4.7	4.8	2.5	<0.10	12.0	10.9
F - 04	1.8	2.9	2.6	<0.22	7.3	4.6
F - 05	0.7	1.2	2.7	<0.30	4.6	4.5
F - 06	0.5	0.1	2.8	<0.72	2.9	2.3
F - 07	<0.1	0.01	2.9	<1.20	<3.0	2.8
F - 08	<0.1	0.005	3.8	<1.90(b)	<3.9	1.9
F - 09	<0.1	<0.001	3.3	<2.50(b)	3.4	0.9

(a) See Definition of Experiments in Table Al-2.

(b) No Nitrite.

TABLE A1-2 DEFINITION OF EXPERIMENTS (a)

Date	Exper. No.	Description	Temp., K (F)	pH	O <sub>3</sub> Conc., %	O <sub>3</sub> Dosage, mg/Min (Lb/Day)
3/26/76	A	Integrated Composite Feasibility Test	318 (113)	9 <sup>(b)</sup>	1.8	693 (2.20)
3/30/76	B	Batch Composite	318 (113)	11	3.3	205 (0.65)
3/30/76	C	Batch Composite	318 (113)	7	3.3	205 (0.65)
3/31/76	D	Batch Composite	303 (86)	8	3.3	205 (0.65)
4/1/76	E	Batch Composite	333 (140)	8	3.3	205 (0.65)
4/5/76	F	Integrated Composite Feasibility Test	303 (86)	9 <sup>(b)</sup>	1.76	664 (2.11)

(a) UV lamps were on in all experiments. Gas flow rates were maintained at 28.3 l/Min (60 Scfh) for integrated tests and at 4.7 l/Min (10 Scfh) for batch tests. Process water flow rates were 1 l/Min (16 Gph) in the two integrated tests A and F.

(b) Feed pH value, uncontrolled in the experiment.

APPENDIX 2

LMTOC FAULT DETECTION  
AND ISOLATION ANALYSIS



## INTRODUCTION

The purpose of a Fault Detection and Isolation Analysis (FDIA) is to establish the required sensors in the Ozone ( $O_3$ ) Unit Process to allow complete and rapid fault detection and isolation to the Line Replaceable Unit (LRU). This report covers the study of  $O_3$  Oxidation Unit Process component failure symptoms, interface failure symptoms and performance trend analysis. The component failures include sensor failures, actuator failures and mechanical failures such as spargers, flow regulators, etc. The interface failures include the mechanical interfaces and process stream interactions between the  $O_3$  Oxidation Unit Process and the other five unit processes of the Water Processing Element (WPE) system. For example, shortage of influent water from a previous unit processes or shortage of a hot air supply or coolant supply to the heat exchangers are typical interface failures.

The relationship between failures and symptoms is shown in Figure A2-1. It is unusual for a 1:1 correspondence relationship to exist between a failure and a symptom. In other words, a failure typically results in a number of symptoms, and a certain symptom can be the result of a number of failures. A thorough study of all possible component failures and interface failures and their associated symptoms is needed for the FDIA. As shown in Figure 1, fault detection is the process of detecting the existence of a failure or failures in the unit process by sensing the presence of their associated symptoms. This process is considered easier than visa versa, namely, fault isolation.

The symptoms of  $O_3$  Oxidation Unit Process component failures are listed in Table A2-1. In this study, only single failure cases are considered. Multiple failure cases are unlikely to happen and, therefore, are excluded in the study.

Unit process failure symptoms can be caused by unit process interface failures. Therefore, these interface failures must be considered in any unit process FDIA. Possible interface failures are shown in Table A2-2.

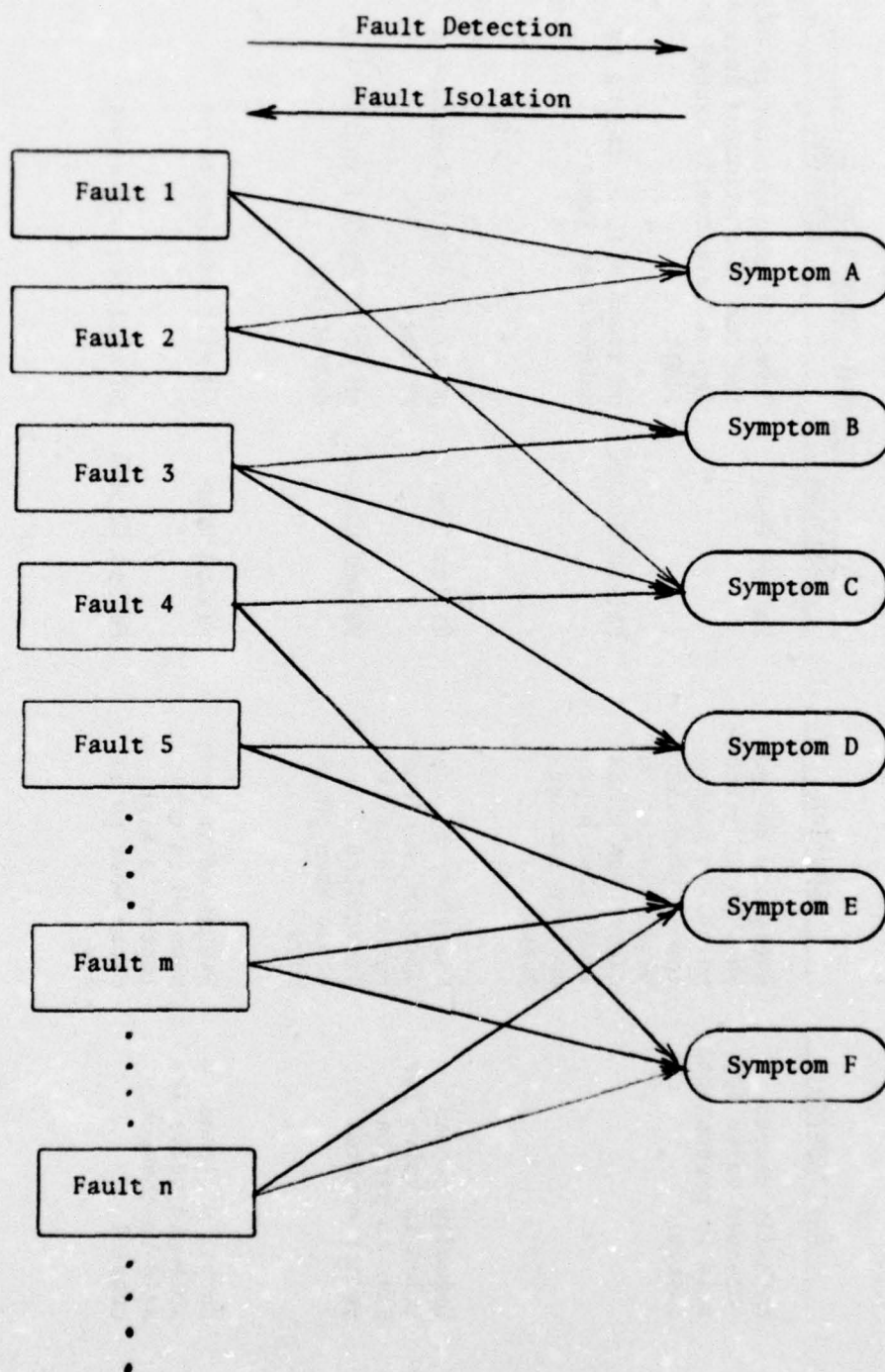


FIGURE A2-1 FAULT DETECTION AND ISOLATION RELATIONSHIPS

TABLE A2-1 SYMPTOMS OF OZONE OXIDATION UNIT PROCESS COMPONENT FAILURES

Part 1 Unit Process Actuators

Code	Description	Function	Fault Detection	
			Failure	Symptom
A <sub>1</sub>	Normally closed solenoid valve for acid to precontactor control	Energized to open when pH in precontactor is higher than a prescribed high setpoint and close when pH is lower than a prescribed low setpoint	Failed Open	Trend of precontactor pH will indicate a continuous decreasing and eventually exceed low limit
			Failed Closed	pH trend will be rising to exceed high limit
A <sub>2</sub>	Normally closed solenoid valve for base to precontactor control	Energized to open when pH in precontactor is below a prescribed setpoint; close when pH is high	Failed Open	pH trend should indicate a rising
			Failed Closed	pH trend should indicate a decreasing
A <sub>3</sub>	Normally closed solenoid valve for acid to contactor control	Energized to open when pH in contactor is high; close when pH is low	Failed Open	pH will be decreasing
			Failed Closed	pH will be increasing

continued-



Table A2-1 - continued

Code	Description	Function	Fault Detection	
			Failure	Symptom
A <sub>4</sub>	Normally closed solenoid valve for base to contactor control	Energized to open when pH in contactor is low; close when	Failed Open Failed Closed	pH will be rising pH will be decreasing
A <sub>5</sub>	Ozone contactor feed pump, centrifugal type	Running at normal mode when precontactor has water	Failed Off  Failed On	Failed to run; contactor influent flow low; flow switch D <sub>10</sub> can detect Failed to stop; will not detect, but not critical
A <sub>6</sub>	Normally closed solenoid valve to control contactor influent water	Energized to open in NORMAL REUSE mode; closed in STANDBY, SHUTDOWN or NORMAL WASTE DISCHARGE mode for contactor bypass	Failed Open  Failed Closed	Flow switch D <sub>10</sub> can detect; also contactor water level high alarm (D <sub>8</sub> ) will be triggered in WASTE DISCHARGE mode Flow switch D <sub>10</sub> can detect
A <sub>7</sub>	Normally closed solenoid valve to control water route for waste discharge	Energized to open in NORMAL WASTE DISCHARGE mode; closed otherwise	Failed Open Failed Closed	Flow switch will detect There will be no waste discharge product; product flow sensor can detect

continued-

Table A2-1 - continued

Code	Description	Function	Fault Detection	
			Failure	Symptom
A <sub>8</sub>	Normally closed solenoid valve to control contactor effluent water	Energized when water reaches D <sub>7</sub> ; deenergized when water drops to D <sub>6</sub>	Failed Open	Water level will trigger D <sub>5</sub>
			Failed Closed	Water level will trigger D <sub>8</sub>
A <sub>9</sub>	Motor-driven, 3-way valve	Switch to recycle position when TOC high alarm is tripped	Failed Open	If failed at recycle position, Ozon Unit Process product flow should give a warning indication
			Failed Closed	If failed at normal product position, can be detected by flow monitor
A <sub>10</sub>	Contactor make-up heater	On/off temperature control when contactor has water	Failed On	Temperature sensor (S <sub>6</sub> ) should see a rising trend
			Failed Off	S <sub>6</sub> should see a decreasing trend
A <sub>11</sub> A <sub>12</sub> A <sub>13</sub> A <sub>14</sub> A <sub>15</sub> A <sub>16</sub>	UV lamp, Stage 1 UV lamp, Stage 2 UV lamp, Stage 3 UV lamp, Stage 4 UV lamp, Stage 5 UV lamp, Stage 6	On when in NORMAL REUSE mode	Failed Off	Ozone generation power required will be abnormally high
			Failed On	Not critical, will not be detected

continued-

Table A2-1 - continued

Code	Description	Function	Fault Detection	
			Failure	Symptom
A <sub>17</sub>	Normally closed solenoid valve for precontactor drain	On (open) in DRAIN mode; off (closed) otherwise	Failed Open	Precontactor water level low, alarm will be tripped; also product flow sensor should indicate a low production period
A <sub>18</sub>	Normally closed solenoid valve for contactor drain	On (open) in DRAIN mode; off (closed) otherwise	Failed Closed	Level sensors can detect
			Failed Open	Contactor water level low, alarm will be tripped
			Failed Closed	Water level sensors can detect
A <sub>19</sub>	Servo-driven diverter valve to control hot air flow rate	Proportionally controlled by temperature sensor, S <sub>5</sub> , when activated in NORMAL REUSE mode	Failed Open	If diverter valve failed to follow command, make-up temperature sensor, S <sub>5</sub> , should see an abnormally high or low temperature; S <sub>6</sub> should also see an abnormally high or low temperature
A <sub>20</sub>	Air compressor	Running in STANDBY or NORMAL mode	Failed Off	Ozone generator feed gas pressure will be low (S <sub>11</sub> ) and ozone outlet gas flow rate (S <sub>12</sub> ) will be low

continued-



Table A2-1 - continued

Code	Description	Function	Fault Detection	
			Failure	Symptom
A <sub>20</sub>	continued		Failed On	Not critical; can be detected by S <sub>11</sub> and S <sub>12</sub>
A <sub>21</sub>	Solenoid valve for after-cooler heat exchange control	Energized to open when air dryer is running in STANDBY or NORMAL mode	Failed Closed	Temperature monitor (S <sub>8</sub> ) will detect
			Failed Open	Not critical; only waste energy in heat exchanging
A <sub>22</sub>	Refrigerant line compressor	Turned on in NORMAL or STANDBY mode	Failed Off	Both temperature (S <sub>9</sub> ) and flow switch (D <sub>9</sub> ) will detect
			Failed On	Not critical; waste electrical energy (flow switch D <sub>9</sub> can also detect)
A <sub>23</sub>	Blower for refrigerant loop	Turned on in NORMAL or STANDBY mode	Failed Off	Temperature monitor (S <sub>9</sub> ) will detect
			Failed On	Not critical; D <sub>10</sub> will detect
A <sub>25</sub>	Normally closed solenoid valve to control coolant to desiccant I	Cooling desiccant when in use	Failed Open	Desiccant will not regenerate; can be detected by dew point
			Failed Closed	Desiccant will not absorb moisture; detected by dew point

continued-

Table A2-1 - continued

Code	Description	Function	Fault Detection	
			Failure	Symptom
A <sub>26</sub>	Normally closed solenoid valve to control coolant to desiccant I	Cooling desiccant when in use	Failed Open	Desiccant will not regenerate; can be detected by dew point
			Failed Closed	Desiccant will not absorb moisture; detected by dew point
A <sub>27</sub>	Normally closed solenoid valve to control coolant to ozone generator	Cooling ozone generator	Failed Open	Will not be detected
			Failed Closed	Ozone generator over-heated temperature sensor will cause shutdown
A <sub>28</sub>	Motor-driven, 3-way valve for desiccant dryer select for process air	Select desiccant dryer alternately when dew point of process air reaches a prescribed set point	Failed Off	If failed to switch, dew point of process air will reach warning or alarm limit
A <sub>29</sub>	Motor-driven, 3-way valve for desiccant dryer select for regeneration	Select desiccant dryer alternately when dew point of process air reaches a prescribed set point	Failed Off	If failed to switch, desiccant dryer will not regenerate and eventually dew point of process air will be high

continued -

Table A2-1 - continued

Code	Description	Function	Fault Detection	
			Failure	Symptom
A <sub>30</sub>	Desiccant dryer regeneration heater I	Turned on when desiccant is being regenerated	Failed On	Desiccant will not regenerate, and dew point will be high
			Failed Off	Process air will not be dried, and dew point will be high
A <sub>31</sub>	Motor-driven, 3-way valve for controlling air to vent or selection I	Switch to vent when heater is on; to ozone generator when	Failed Open	If failed to switch to vent, dew point of ozone generator feed air will be high
			Failed Closed	If failed to switch to ozone generation, ozone generator feed air pressure will be low
A <sub>32</sub>	Desiccant dryer regeneration heater II	Switch to vent when heater is on; to ozone generator when heater is off	Failed At Position A	If failed to switch to vent, dew point of ozone generator feed air will be high
			Failed At Position B	If failed to switch to ozone generation, ozone generator feed air pressure will be low
A <sub>33</sub>	Motor-driven, 3-way valve for controlling air to vent or to ozone generator selection II	Switch to vent when heater is on; to ozone generator when heater is off	Failed At Position A	If failed to switch to vent, dew point of ozone generator feed air will be high
			Failed At Position B	If failed to switch to ozone generation, ozone generator feed air pressure will be low

continued-



Table A2-1 - continued

Code	Description	Function	Fault Detection	
			Failure	Symptom
A <sub>34</sub>	Ozone generator electrodes power	Controlled by TOC/COD sensors when activated	Failed High	Not critical; waste more power (possibly a wattage feedback or an ozone analyzer)
			Failed Low	TOC trend should trigger CAUTION, etc.
A <sub>35</sub>	Normally closed solenoid valve for ozone to precon-	On when in NORMAL WASTE DISCHARGE mode; off (closed)	Failed Open	Not initial in NORMAL WASTE DISCHARGE mode; TOC will be rising in NORMAL REUSE mode
			Failed Closed	Flow sensor, S <sub>12</sub> , should detect <sup>(a)</sup>
A <sub>36</sub>	Normally closed solenoid valve for ozone to contactor control	On when in NORMAL REUSE mode; off (closed) otherwise	Failed Open	Not initial in NORMAL REUSE mode; will lose efficiency in NORMAL WASTE DISCHARGE mode and will go undetected
			Failed Closed	Flow sensor, S <sub>12</sub> , should detect <sup>(a)</sup>

<sup>(a)</sup> If both A<sub>35</sub> and A<sub>36</sub> are closed, pressure relief valve opens to prevent system overpressure.

continued-

Table A2-1 - continued

Code	Description	Function	Fault Detection	
			Failure	Symptom
Part 2 Unit Process Sensors				
D <sub>1</sub>	Precontactor water level sensor, bottom	Monitor water level low alarm	Failed on (Wet)	Will not be detected
			Failed Off (Dry)	Will be detected by false alarm and by D <sub>2</sub> if D <sub>2</sub> indicates it's wet
D <sub>2</sub>	Precontactor water level sensor, 2nd from bottom	Control normally closed valve A <sub>0</sub> in DISCHARGE mode, start influent in REUSE mode	Failed On (Wet)	Will be detected by D <sub>1</sub> (shutdown)
			Failed Off (Dry)	Will be detected by D <sub>3</sub>
D <sub>3</sub>	Precontactor water level sensor, 2nd from top	Control open valve A <sub>0</sub> in DISCHARGE mode; stop influent in REUSE mode	Failed On (Wet)	Will be detected by D <sub>2</sub>
			Failed Off (Dry)	Will be detected by D <sub>4</sub> (shutdown)
D <sub>4</sub>	Precontactor water level sensor, top	Monitor water level high alarm	Failed On (Wet)	Will be detected by false or by D <sub>3</sub>
			Failed Off (Dry)	Will not be detected

A2-12

continued-

continued-

Table A2-1 - continued

Code	Description	Function	Fault Detection	
			Failure	Symptom
D <sub>5</sub>	Contact water level sensor, bottom	Contact water level control/monitor similar to above	Failed On (Wet)	Will not be detected
			Failed Off (Dry)	Will be detected by false alarm and by D <sub>2</sub> if D <sub>2</sub> indicates it's wet
D <sub>6</sub>	Contact water level sensor, 2nd from bottom	Contact water level control/monitor similar to above	Failed On (Wet)	Will be detected by D <sub>1</sub> (shutdown)
			Failed Off (Dry)	Will be detected by D <sub>3</sub>
D <sub>7</sub>	Contact water level sensor, 3rd from bottom	Contact water level control/monitor similar to above	Failed On (Wet)	Will be detected by D <sub>2</sub>
			Failed Off (Dry)	Will be detected by D <sub>4</sub> (shutdown)
D <sub>8</sub>	Contact water level sensor, top	Contact water level control/monitor similar to above	Failed On (Wet)	Will be detected by false alarm or by D <sub>3</sub>
			Failed Off (Dry)	Will not be detected

continued-



Table A2-1 - continued

Code	Description	Function	Fault Detection	
			Failure	Symptom
D <sub>9</sub>	Refrigerant loop flow switch	Monitor leakage of or malfunction of refrigerant loop, switch is on when flow rate is high	Failed On Failed Off	Will not be detected Will be detected by false alarm
D <sub>10</sub>	Contactor influent flow switch I	Set to trip (on) if <3 Gpm to monitor pump and valves	Failed On Failed Off	False alarm Will be detected in DISCHARGE mode
D <sub>11</sub>	Contactor influent flow switch II	Set to trip (on) if >0.5 Gpm to monitor pump and valves	Failed On Failed Off	False alarm False alarm
S <sub>1</sub>	TOC or COD analyzer at stage 2 of contactor	Provide feed forward signal for ozone generation control; needed for initial startup and for early warning of high TOC water	Failed Open	Will result in the loss of feed forward function which might cause recycling of product water (frequent recycling of water is the symptom)
S <sub>2</sub>	TOC or COD analyzer at contactor outlet	Provide feedback control of TOC ozone generation	Failed High Failed Low	False alarm Will not be detected (serious, redundancy required)

continued-

Table A2-1 - continued

Code	Description	Function	Fault Detection	
			Failure	Symptom
S <sub>3</sub>	Precontactor pH sensor	pH control	Failed within range	Once control of pH is initiated, pH should stay in a narrow range (e.g., 7 to 11); if sensor failed, water pH in contactor will sooner or later trigger high or low alarm limit
			Failed out-of-range	If sensor fails and stays out of range for a prescribed period of time, it will be detected
S <sub>4</sub>	Contacting pH sensor	pH control	Failed out-of-range	False alarm
			Failed within range	TOC will be rising
S <sub>5</sub>	Temperature sensor to control heat exchanger A <sub>19</sub>	Control process water temperature	Failed out-of-range	False alarm
			Failed within range	Temperature S <sub>6</sub> will see an abnormally high or low temperature

continued-

Table A2-1 - continued

Code	Description	Function	Fault Detection	
			Failure	Symptom
S <sub>6</sub>	Temperature sensor in contactor	Make-up control of process water in contactor	Failed out-of-range	Detected by False alarm
			Failed within range	Temperature S <sub>6</sub> will see an abnormally high or low temperature
S <sub>7</sub>	Temperature sensor of ozone generator cooling system	Monitor	Failed High	Will be detected by shutdown
			Failed Low	Will not be detected
S <sub>8</sub>	Temperature sensor	Monitor after compressor cooler temperature	Failed High	Will be detected by dew point sensor
			Failed Low	Will not be detected
S <sub>9</sub>	Temperature sensor	Monitor after refrigerant loop temperature sensor	Failed High	Will be detected by dew point sensor
			Failed Low	Will not be detected
S <sub>10</sub>	Dew point sensor	Control desiccant dryer selection	Failed High	There will be a false alarm resulting in a switch between the two desiccant dryers; no damage to the system should occur

continued-



Table A2-1 - continued

Code	Description	Function	Fault Detection	
			Failure	Symptom
S <sub>10</sub>	Dew point sensor - continued		Failed Low	Ozone generator will be down-graded gradually. Recommendation: back up the DP-controlled desiccant with a timing control; in other words, alternated when time $\geq$ TBD hours or when DP $\geq$ TBD C, whichever occurs first
S <sub>11</sub>	Pressure sensor	Monitor ozone generator feed gas pressure	Failed High	False alarm (warning)
			Failed Low	Will be detected by flow
S <sub>12</sub>	Gas flow sensor	Monitor ozone generator outlet gas flow	Failed out-of-range	False alarm
			Failed within range	Will not be detected
S <sub>13</sub>	Liquid flow sensor	Monitor ozone unit process product water	Failed out-of-range	False Alarm
			Failed within range	Will not be detected

continued-

Table A2-1 - continued

Code	Description	Function	Fault Detection	
			Failure	Symptom
M <sub>1</sub>	Spargers	Spurge ozone into process water	Clogged	TOC high
M <sub>2</sub>	Flow regulator	Regulate ozone feed gas pressure	Failed High	Will not be detected
			Failed Low	Feed gas pressure low (detected by pressure sensor)

TABLE A2-2 SYMPTOMS OF OZONE OXIDATION  
UNIT PROCESS INTERFACE FAILURES

<u>Description of Interface</u>	<u>Type of Failure</u>	<u>Symptom</u>
Influent water supply to Ozone Unit Process	Shortage	Precontactor and contactor water level low
Hot air supply to process water heat exchanger	Supply shortage or temperature low	Temperature of process water low and even- tually TOC high
Coolant supply to post- compressor cooler	Supply shortage or temperature high	Air temperature high; dew point high
Coolant supply to desiccant dryers	Supply shortage or temperature high	Dew point high
Coolant supply to ozone generator	Supply shortage or temperature high	Ozone generator over temperature shutdown



APPENDIX 3

RO B-10 MODULE SPECIFICATIONS

DUPONT PERMASEP PERMEATOR  
MODEL NO. 6440-015  
B-10 PERMEATORS  
PRODUCT SPECIFICATIONS

Membrane Type, cm (In)	B-10, 10.2 (4) diameter
Membrane Configuration	Hollow Fiber
Shell Dimensions, cm (In)	14.0 OD x 11.7 ID x 119.4 Long (5.5 x 4.625 x 47)
Shell Material	Filament-wound Fiberglass Epoxy
End Plates	Fiberglass epoxy
Snap Rings	15-4 PH-MO Stainless Steel
Connections, cm (In)	Feed and Permeate 1.3 (0.5) female, NPT Concentrate, 1 (.375) female, NPT
Permeator Weight, filled with water, kg (Lb)	227 (50)
Initial Product Water Capacity <sup>(a)</sup> , l/day (Gpd)	5700 (1,500)
Salt Passage <sup>(b)</sup>	1.5% <sup>(a)</sup>
Rated Operating Pressure, $\text{kN/m}^2$ (Psig)	5500 (800)
Temperature Range, K (F)	273-303 (32-86)
pH Range <sup>(c)</sup> , continuous exposure	5-9
Conversion Range <sup>(c)</sup>	10-50% (for soluble salts)
Operating Position	Horizontal or vertical
Permeate Back Pressure <sup>(c)</sup> , $\text{kN/m}^2$ (Psig)	345 (50 max)

(a) Based on operation with a feed of 30,000 ppm NaCl at  $5500 \text{ kN/m}^2$ , 298K (800 Psi, 77F) and 30% conversion. For operation at other conditions consult Permasep Products.

(b) Dependent on water analysis and conversion.

(c) For operation outside this range, consult Permasep Products.

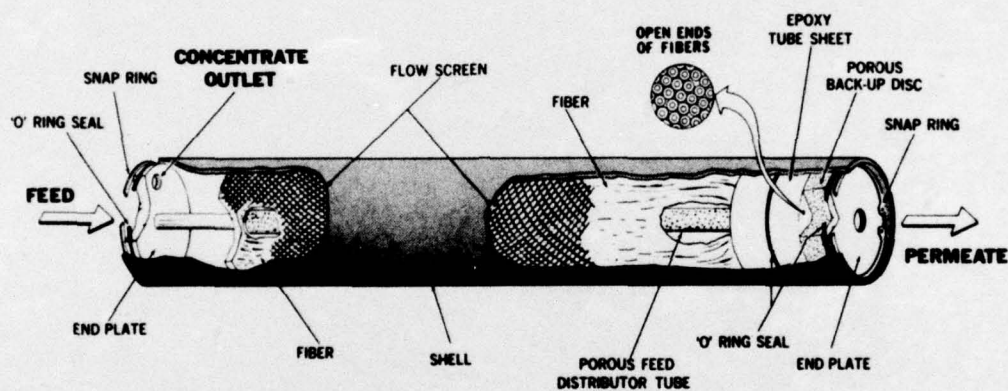


FIGURE A3-1 CUTAWAY DRAWING OF PERMASEP PERMEATOR<sup>(a)</sup>

(a) From DuPont Permasep Technical Bulletin 125. Permasep is a registered trademark of DuPont.



APPENDIX 4

RO UNIT PROCESS SPECIFICATIONS

<b>Life Systems, Inc.</b> CLEVELAND, OHIO 44122	<b>SPECIFICATION</b>	NO.	REVISION LTR.
		PAGE 1 OF 2	DATE
TITLE RO UNIT PROCESS SPECIFICATIONS			

RO MODULES

One to four DuPont 10 cm (4 In) B-10 hollow fine-fiber permeator

NOMINAL OPERATING CONDITIONS

1. Full-size permeator product rate: 3.8 l/Min (1 Gpm)
2. Total membrane requirement for 16,000 l/Day or 16 m<sup>3</sup>/Day (4200 Gpd)  
MUST system: three and one-half modules in a series arrangement
3. Mode of Operation: continuous
4. B-10 module inlet pressure: 5500 kN/m<sup>2</sup> (800 Psig)
5. B-10 module inlet temperature: 302 K (85F)
6. Feed recovery: 90%

PHYSICAL CHARACTERISTICS

<b>Weight</b>	
Basic System Dry, Kg (Lb)	680 (1500)
Spares, Kg (Lb)	136 (300)
Total, Kg (Lb)	820 (1800)
<b>Volume</b>	
Basic System, m <sup>3</sup> (Ft <sup>3</sup> )	2.5 (90)
Spares, m <sup>3</sup> (Ft <sup>3</sup> )	0.4 (15)
Total, m <sup>3</sup> (Ft <sup>3</sup> )	2.9 (105)
Basic Dimensions (LxWxH), m (Ft)	1.2 x 1.4 x 1.5 (4 x 4.5 x 5)

MATERIAL CHARACTERISTICS

Nonmetallic	Nylon, polypropylene, teflon, fiberglass, epoxy
Metallic	316 SS, 304 SS and other compatible ferrous and nonferrous alloys

ELECTRICAL CHARACTERISTICS

Supply Voltage, VAC	(i) 208 (ii) 110
Line Frequency, Hz	60

<b><i>Life Systems, Inc.</i></b> CLEVELAND, OHIO 44122	<b>SPECIFICATION</b>	NO.  PAGE 2 OF 2	REVISION LTR.  DATE										
TITLE RO UNIT PROCESS SPECIFICATIONS													
<div data-bbox="292 403 449 434" data-label="Section-Header"> <u>INTERFACES</u> </div> <div data-bbox="289 462 449 495" data-label="Section-Header"> <u>Mechanical</u> </div> <table data-bbox="362 520 1180 642"> <tr> <td>RO Feed Tank Drain, cm (In)</td> <td>0.6 (1/4) tube</td> </tr> <tr> <td>RO Concentrate Drain, cm (In)</td> <td>1.0 (3/8) tube</td> </tr> <tr> <td>RO Permeate, cm (In)</td> <td>1.0 (3/8) tube</td> </tr> <tr> <td>RO Makeup Water, cm (In)</td> <td>1.3 (1/2) tube</td> </tr> </table> <div data-bbox="290 665 449 701" data-label="Section-Header"> <u>Electrical</u> </div> <table data-bbox="362 726 1208 787"> <tr> <td>Connector</td> <td>Amphenol MS Type (MIL-C-5015D)</td> </tr> </table> <div data-bbox="290 814 466 846" data-label="Section-Header"> <u>Environment</u> </div> <p data-bbox="365 871 683 905">Laboratory Atmosphere</p>				RO Feed Tank Drain, cm (In)	0.6 (1/4) tube	RO Concentrate Drain, cm (In)	1.0 (3/8) tube	RO Permeate, cm (In)	1.0 (3/8) tube	RO Makeup Water, cm (In)	1.3 (1/2) tube	Connector	Amphenol MS Type (MIL-C-5015D)
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RO Permeate, cm (In)	1.0 (3/8) tube												
RO Makeup Water, cm (In)	1.3 (1/2) tube												
Connector	Amphenol MS Type (MIL-C-5015D)												



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