ER-171-3-2

DEVELOPMENT OF AN IODINE GENERATOR FOR RECLAIMED WATER PURIFICATION IN MANNED SPACECRAFT APPLICATIONS

FINAL REPORT

by

R. A. Wynveen, J. D. Powell
and F. H. Schubert

August, 1973

Prepared Under Contract No. NAS1-11765

by

LIFE SYSTEMS, INC.
Cleveland, Ohio 44122

for

JOHNSON SPACE CENTER

NATIONAL AERONAUTICS AND SPACE ADMINISTRATION

FOREWORD

The development work described herein was conducted by Life Systems, Inc. during the period June 26, 1972 through August 28, 1973, under NASA Contract NAS1-11765. The Program Manager was Dr. R. A. Wynveen. Technical support was provided as follows.

Personnel	Area(s) of Responsibility
Fred C. Jensen	Iodine valve, accumulator and dispenser design
Glenn A. Little	Ground Support Accessories layout and fabrication
John E. Nemeth	Analytical, Parametric and Endurance Testing
David E. Keck	Control and monitor instrumentation design and fabrication
Manny H. Naft	Mechanical design and alternate iodine generation designs
J. David Powell	Control and monitor instrumentation design
James L. Seago, Ph.D.	Iodine chemistry, electrochemistry and microbiology
John W. Shumar	Product Assurance and materials evaluation
Franz H. Schubert	Mechanical hardware, electrolytic valve and Potable Water System Simulator designs
Boris D. Cahan, Ph.D.	Iodine electrochemistry and ion exchange membrane technology

The program's technicians were R. A. Clark and M. L. Kruszynski. Secretarial work was completed by C. A. Lucas and V. E. Szanati. Dr. Boris D. Cahan is a Senior Research Associate within the Chemistry Department of Case Western Reserve University, Cleveland, Ohio 44106.

The contract's Technical Monitor was O. Karl Houck, NASA Johnson Space Center, Houston, Texas 77058. The program was initially contracted with the NASA Langley Research Center and subsequently transferred to the Johnson Space Center.

TABLE OF CONTENTS

continued-

Table of Contents continued -

continued -

Table of Contents continued -

PAGE

LIST OF FIGURES

LIST OF TABLES

SUMMARY

A four-task program culminated with a successful 30-day test of a prototype Iodine Generating and Dispensing System (IGDS). The IGDS was sized to iodinate the drinking water nominally consumed by six men, 4.5 to 13.6 kg (10 to 30 lb) water per man-day with a ±10 to 20% variation with iodine (I_2) levels of 0.5 to 20 parts per million (ppm). The I_2 treats reclaimed water to prevent or eliminate microorganism contamination. Treatment is maintained with a residual of I_2 within the manned spacecraft water supply. A simplified version of the Chlorogen water disinfection concept, developed by Life Systems for on-site generation of chlorine (Cl_2), was used as a basis for IGDS development.

The IGDS consisted of (1) an I_2 accumulator to store crystalline I_2; (2) an electrochemical I_2 valve to control I_2 flow; (3) a method for dispensing I_2 into water; and (4) nominal controls. The I_2 accumulator had storage volume adequate for 180 days. The I_2 valve consisted of an anion exchange membrane between two noble metal electrodes. The I_2 dispenser consisted of a compartment that passed untreated water over the I_2 generation electrode. The controller allowed regulation of I_2 flow in response to an I_2 concentration to maintain the desired residual I_2 concentration. The IGDS design specification was selected for Space Shuttle Orbiter and Space Station applications. Six operating modes were included to simulate all envisioned arrangements.

Preliminary development was completed with laboratory setups and a breadboard. Ground Support Accessories were provided to check out IGDS materials of construction, obtain design data, simulate the potable water system, and measure I_2 concentration and related aqueous solution parameters.

A mini-Product Assurance Program was included so the impact of manned chamber testing requirements would be included during the initial design activities. Major emphasis was on IGDS materials selection and, based on several exposure tests, Teflon and 316 stainless steel were selected.

An extensive test program was completed. Experiments with cells demonstrated that current densities to 5 mA/cm^2 were possible. Experiments with a breadboard system, operated for 35 days, demonstrated construction material integrity and the combining of I_2 storage, dispensing and valve functions into a single assembly.

Information produced during this development effort identified the IGDS as a contender for potable water microorganism control on future manned spacecraft. It remains to reduce the basic design reported herein to a prototype approaching flight specification. Experiments with the IGDS culminated in a 30-day endurance test. The endurance test was operated at a 5 to 10 ppm I_2 level at room temperature, with a water flow of 27 kg (60 lb) per day and a pressure of 210 kN/m^2 (30 psig). The electrolytic valve operated at 1.6 watt and 6 mA (0.24 mA/cm^2 (0.23 amp/ft^2)).

Potable water contaminated with abundant E. Coliform Group organisms was treated by electrolytically generated I_2 at levels of 5 to 10 ppm. In all instances, the E. coli were eliminated.

Other program activities included (1) development of a mechanical I_2 injection process and (2) a preliminary comparison between the IGDS and the silver ion (Ag^+) water treatment process. The comparison showed the IGDS was lighter, smaller, and avoided the need to replace ion exchange columns. The electrolytic I_2 valve required less than one watt of power.

INTRODUCTION

The potable water supply on future manned spacecraft will use recycled water in the distribution system. Although pasteurization has been recommended as a reliable method for sterilization, other approaches offer distinct advantages in terms of weight, volume, cost and power consumption for maintaining water quality. One such approach was investigated under NASA Contract NAS1-9917,(1) which lead to the development of a laboratory breadboard of an in-situ Cl_2 generating device called the Chlorogen.

Other biocides, Ag^+ and I_2, for instance, have merit and have received attention in the manned space program. Iodine, because of its superior microorganism annihilation potential at low dosages and dose rates, among other advantages, is favored. However, development of suitable devices to dispense and measure I_2 and its concentration in a manned spacecraft application has not occurred except for treating potable water stores in the Lunar Excursion Module of the Apollo and in the Skylab program. The program conducted under Contract NAS1-11765 was, therefore, initiated for the "Development of an Iodine Generator for Reclaimed Water Purification in Manned Spacecraft Applications." Technology produced from the preceding Chlorogen development formed the basis of initial studies for the I_2 generator development program which is described herein.

PROGRAM OBJECTIVES AND STRUCTURE

A program to develop a six-man prototype of an Iodine Generating and Dispensing System (IGDS) was completed by Life Systems, Inc. The ultimate purpose of the IGDS is to treat reclaimed water for microorganism contamination in a manned spacecraft application.

Primary Program Objectives

The primary program objectives were to

1. Convert the Laboratory Breadboard Model of the Chlorine Generating Device(1) into one based upon I_2 as the biocidal agent.
2. Perform experimental research and development with the laboratory breadboard to demonstrate the feasibility of the I_2 generator prior to the development of a six-man prototype of the device.

---

(1) The numbers shown in parentheses refer to references cited at the end of this report.

3. Design and develop (a) the method for storing a 180-day supply of I_2, (b) a mechanism for dispensing the generated I_2 into water, and (c) the instrumentation to allow for automatic I_2 generation in response to a signal reflecting residual I_2 level.

4. Design, develop, fabricate, assemble and functionally test a prototype six-man system on the basis of the analytical and experimental results obtained with the Laboratory Breadboard Subsystem. (The prototype device was specifically designed to iodinate the drinking water nominally consumed by six men (selected in the range 4.5 to 13.6 kg (10 to 30 lb) per man-day) with a ±10 to 20% variation.)

5. Endurance test the prototype subsystem having a goal of 30 days of continuous operation at the nominal design point.

Primary Process Objective

The primary process objective is to treat reclaimed water to prevent microorganism contamination in a zero gravity, manned spacecraft application, by maintaining a preset residual concentration of I_2. This is to be accomplished by integrating the IGDS with a residual I_2 analyzer. The analyzer is to provide a signal to activate the I_2 injection process when I_2 is needed to iodinate reclaimed water or to maintain the pre-established residual level within a potable water supply.

Four Program Tasks

The program was divided into four tasks.

1. Design, develop, fabricate, assemble and functionally check out a breadboard and a six-man prototype of the IGDS.

2. Design, fabricate, assemble and functionally check out the Ground Support Accessories (GSA) needed for the development of components for the laboratory breadboard and the six-man subsystem.

3. Experimental research leading to and including development testing of the nominal six-man IGDS.

4. Program and data management, document preparation and submittal, and contract administration including preparation for and attendance at informal program reviews.

The program's activity was divided such that 54% was devoted to design, development and fabrication of the breadboard and prototype units of the IGDS (Task 1.0); 12% was associated with the GSA for both the breadboard and the six-man system (Task 2.0); 24% was for research and development testing (Task 3.0); and 10% was for data requirements and management of the program to meet cost, schedule and performance objectives (Task 4.0).

IGDS Development (Task 1.0)

The IGDS development involved

1. Analytical studies on replacing the in-situ, biocidal agent generating concept based on Cl_2 chemistry with one based on I_2 chemistry and including
    a. evaluation of the differences between I_2 and Cl_2 chemistry, electrochemistry and biological activity,
    b. evaluation of the materials for applicability to the I_2 system and modification, where required or appropriate, to optimize system/hardware design and development,
    c. preparation of a (i) short summary of the literature defining the organisms that are killed or incapacitated through iodination and chlorination, and the temperature, contact time, solution pH and level of disinfecting agent required,(2) (ii) list of organisms projected for the spacecraft environment, and (iii) cross comparison of iodination and chlorination procedure and concentration required to kill organisms anticipated to be present in a manned space mission,
    d. analysis of approaches to be used for the measurement of I_2 in water,
    e. finalization of the spacecraft water treatment requirements for the selected mission and preparation of a specification for the prototype unit in terms of capacity, capacity range, and ranges in water pressure and temperature, and
    f. preparation of mock-ups of two IGDS concepts and their electronic control module.
2. Modification of the laboratory breadboard of the Cl_2 generating device to enable using it to provide I_2.
3. Design, develop and fabricate the nominal six-man IGDS(a) including an I_2 accumulator, electrolytic I_2 flow control valve (generator), I_2 dispenser, and instrumentation.

Ground Support Accessories (Task 2.0)

The activities devoted to designing and fabricating the GSA included

1. Provision for a potable water system to simulate the end-item application.

(a) A mini-Product Assurance Program was also carried out during the prototype IGDS development (see the Product Assurance section starting on page 54).

2. Provision for a 28 VDC power supply.
3. Provision for an electronic circuit to simulate an I_2 sensor signal corresponding to a 0 to 20 ppm I_2 level (0 to 5 VDC).[a]
4. Establish methods to detect residual I_2.
5. Provisions to allow evaluation of the effect of I_2 on metallic and nonmetallic materials.

These facilities have been called accessories instead of ground support equipment because they would not be required for operation of the prototype system in its end-item application. They are needed to simulate the interface between the IGDS and the projected operating environment and to provide parametric testing facilities.

Research and Development Testing (Task 3.0)

The experimental research leading to and including development testing of the nominal six-man IGDS included

1. A series of experimental investigations carried out on laboratory "set-ups," including a 35-day endurance test on a breadboard, to evaluate application of the Cl_2 generator concept to I_2 generation.
2. Preliminary testing of the nominal six-man IGDS as a system shakedown/debugging procedure.
3. Design Verification Testing (DVT) at 0.5, and 20 ppm and several other concentrations in between.
4. Disassemble, inspect and reassemble for the 30-day endurance test.[b]
5. Continuous 30-day endurance test to establish operational parameters for subsequent automated system operation.
6. Disassemble, inspect and refurbish after the 30-day test.
7. Experiments in which one or more of the E. Coliform Group of bacterial organisms were challenged by the I_2 generated with the breadboard and prototype units.

Program Management (Task 4.0)

The program management activities incorporated to meet NASA's cost, schedule and performance objectives and the data requirements included

---

(a) It was initially planned that the Iodine Colorimeter developed under NAS9-11879 would be provided as Government Furnished Equipment (GFE) to integrate with the IGDS.(3)

(b) No redesign or refurbishment was needed.

1. Monthly meetings with program personnel to discuss contractual cost, schedule and performance objectives versus accomplishments.

2. Four meetings with the contract's Technical Monitor; two at Life Systems' facility and two at the Johnson Space Center (JSC).

3. Preparation and submittal of the following series of data requirements:

    a. an initial Program Plan and its revision to cover the modifications incorporated when the program was transferred to JSC,

    b. Monthly Technical Progress Report Letters, except during the month when other interim type reports were submitted,

    c. two Interim Technical Progress Reports: one on the evaluation of the "Chlorogen" approach to I_2 generation and the other on a comparison between Cl_2 and I_2 for maintaining biocidal conditions in water supplies,

    d. Monthly Financial Management Reports,

    e. one New Technology Report,

    f. a report on additional areas for investigation, and

    g. the Final Report.

Program Additions

After the program was transferred to JSC, several modifications were added, including

1. Incorporation of a standard inspection procedure for use during prototype fabrication and assembly.

2. Provisions for monitoring the nonmetallic materials for applicability to manned testing (later expanded to also include monitoring metallic materials).

3. Provision for the I_2 sensor signal, sample and hold, and synchronization clock since the I_2 sensor projected for use employed sampling on a periodic rather than a continuous basis.

4. Incorporation of two additional challenge experiments in which one or more of the E. Coliform Group of bacterial organisms were incorporated into the water and subsequently challenged with the electrochemically generated I_2.

PREVIOUS RESEARCH AND DEVELOPMENT

Chlorogen

The Chlorogen is an in-situ biocidal agent generator consisting of three series-connected, electrolytic cells. The overall process is illustrated in Figure 1.

One cell serves as a Cl_2 generator and is based on an electrolysis process where Cl_2 is evolved from an aqueous electrolyte made up of sodium chloride (NaCl) and sulfuric acid. Chlorine is produced at the anode and hydrogen (H_2) is produced at the cathode according to the following reactions:

$$\text{Anode:} \quad 2Cl^- = Cl_2 + 2e^- \qquad E_o = -1.36v \qquad (1)$$

$$\text{Cathode:} \quad 2H^+ + 2e^- = H_2 \qquad E_o = 0.00v \qquad (2)$$

$$\text{Overall:} \quad 2Cl^- + 2H^+ = Cl_2 + H_2 \qquad E_o = 1.36v \qquad (3)$$

The Cl_2 is produced at a flow rate proportional to current and at a quantity proportional to the integration of current for the interval of time current is flowing.

The Cl_2 evolved at the anode is in the gas phase since Cl_2 is a gas above 238K (31F). The generated Cl_2 then passes to the second electrolytic cell which is used as an electrolytic valve to control the flow of Cl_2. Chlorine only flows when current flows. Chlorine is consumed at a cathode (via the reverse of reaction (1)) and generated at an anode (via reaction (1)). Starting and stopping of the flow, therefore, results from the opening and closing of an electrical circuit, a process much faster in response than mechanical valves. Because of this feature, the concentration of Cl_2 required in the chlorination process can be carefully regulated. The Cl_2 flow rate is directly proportional to the current flowing because each side of the electrolytic valve contains Cl_2 and the consumption and generation were found to occur with 100% current efficiency.

The by-product H_2 generated in the Cl_2 generation step (reaction (2)) is eliminated immediately by electrochemically reacting it to produce water vapor at the anode of the third electrolytic cell using ambient air as a source of this cell's cathode reactant.

Conversion from Chlorine to Iodine

Figures 2 and 3 are schematics of two versions of the Chlorogen concept, based on its conversion to I_2. They show component locations and their interface with the reclaimed water. As part of the current program, three additional functional components were added to the in-situ biocidal agent generator concept. Additional components are (1) agent storage, (2) a diffuser for dispensing the generated agent into water, and (3) instrumentation for control and monitoring the process.

Figure 2 illustrates the approach in which the I_2 is stored as an acidified iodide (e.g., potassium iodide) and subsequently converted into H_2 and I_2. The

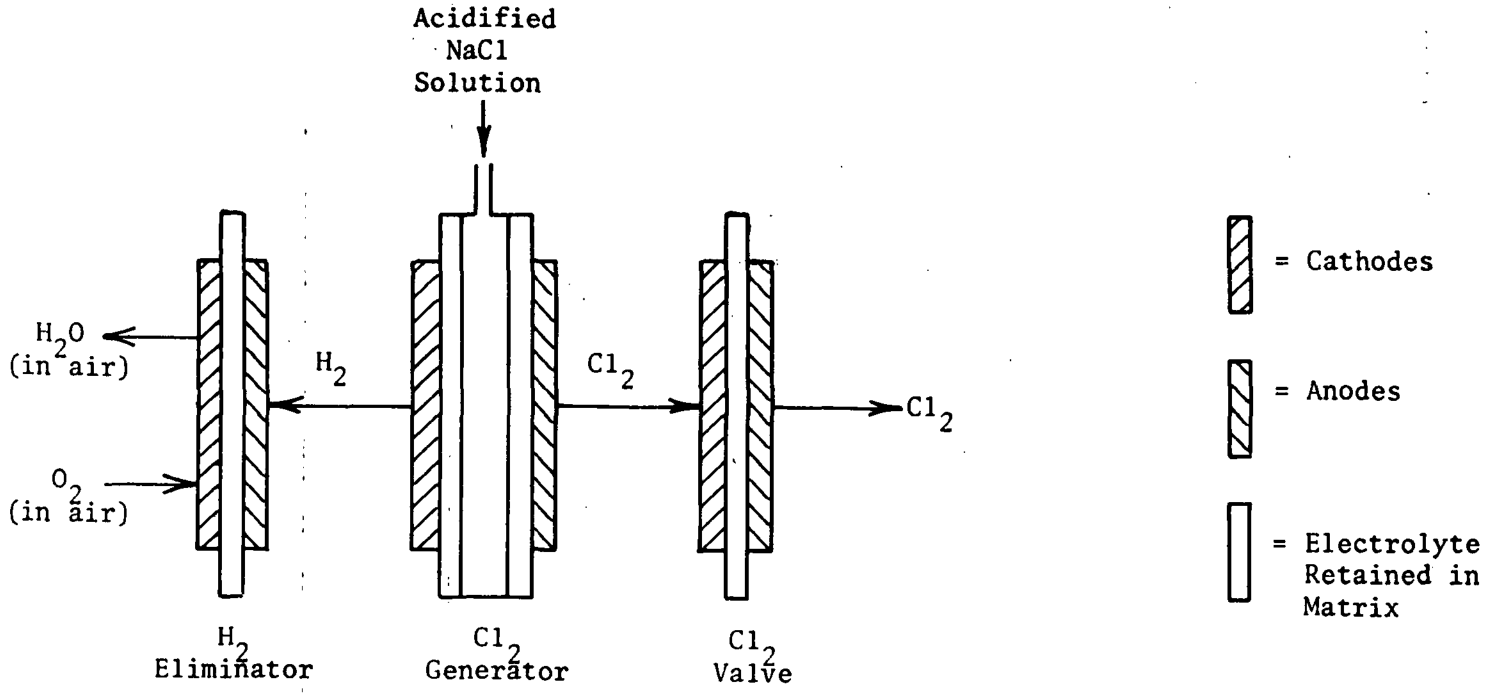


FIGURE 1 CHLOROGEN SYSTEM CONCEPT

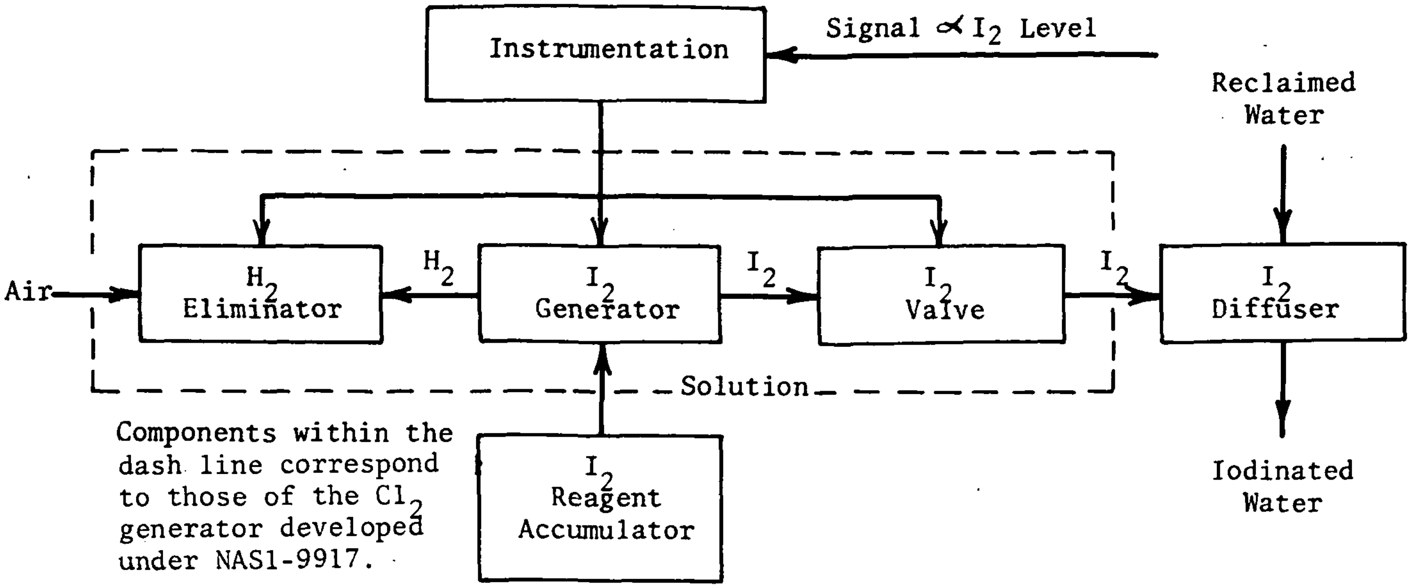


FIGURE 2 SCHEMATIC OF CHLOROGEN-BASED IGDS CONCEPT

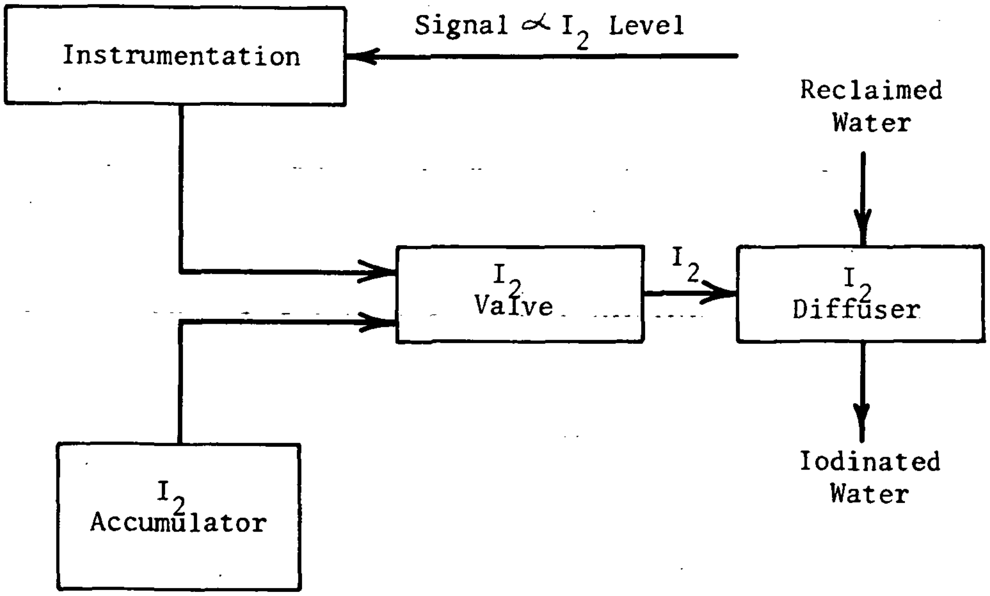


FIGURE 3 SCHEMATIC OF SELECTED IGDS CONCEPT

H_2 formed in the process of forming I_2 from iodide (I^-) is consumed in the H_2 Eliminator. The I_2 dispensing rate is controlled by the electrolytic valve which is an electrochemical cell consisting of two metal screen electrodes separated by an electrolyte. The electrolyte is a potassium iodide soaked anion exchange membrane. The reactions occurring at the electrodes are

Anode:	$2I^- = I_2 + 2e^-$	$E_o = -0.54v$	(4)
Cathode:	$I_2 + 2e^- = 2I^-$	$E_o = +0.54v$	(5)
Overall:	I_2 (solid) = I_2 (aqueous)	$E_o = 0.00v$	(6)

The function of the valve is to accurately dispense I_2 into water.

Figure 3 illustrates a simpler, alternate approach, in which I_2 is stored as a solid in contact with a saturated solution. An electrochemical cell is not used to generate I_2 and, therefore, the I_2 Generator and H_2 Eliminator portions of the on-site, biocidal agent generator are dropped. The I_2 flow rate is still controlled by the electrolytic valve and the accumulator, diffuser, and instrumentation are still needed. A simpler subsystem was not possible with Cl_2 because of the potential hazard of storing a 180-day supply of gaseous Cl_2.

IODINE AS A BIOCIDE

Comparison Between Iodine and Chlorine Characteristics

In changing from Cl_2 to I_2, consideration had to be given to their electrochemical, chemical and biocidal characteristics.

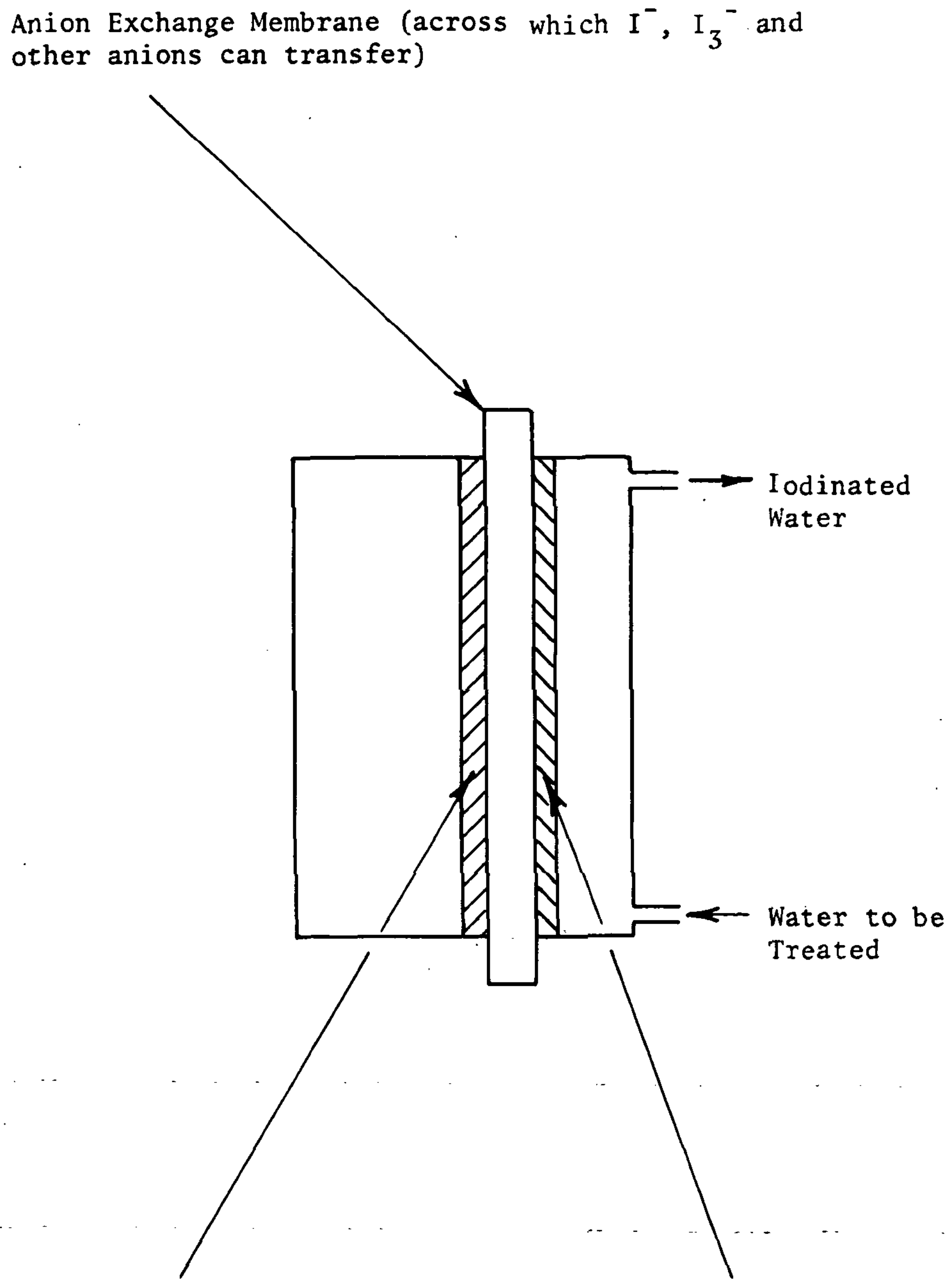
Electrochemical

The literature shows(4) that a smaller potential is required to oxidize I^- to I_2 than to oxidize Cl^- to Cl_2, -0.54 versus -1.36 volt, respectively. Electrical current required for a six-man spacecraft application is less than one amp so power reductions to use I_2 are negligible (less than one watt). The lower oxidizing power of I_2 also means the competitive reaction between Cl_2 evolution and oxygen (O_2) evolution is avoided. (Were it not for the high overvoltage (i.e., voltage greater than oxidation potential) of the competing O_2 evolution reaction, efficient conversion of Cl to Cl_2 would not have been possible.)(1)

Figure 4 depicts the I_2 valve process. Electrical current regulates the electrochemical reaction and the specific anion transferred determines the process efficiency. If 100% of the current flow is via I^-, the current efficiency will be 100%; via I_3^-, the current efficiency will be 300%; and via OH^-, the current efficiency will be 0%. Actual practice indicates a complex combination of all three.

Physical and Chemical

One important difference between I_2 and Cl_2 is that I_2 is a solid at room temperature while Cl_2 is a gas. Thus, I_2 formed through electrolytic generation



Cathode Reactions		Anode Reactions
$I_2 + 2e^- = 2I^-$	Electrochemical	$2I^- = I_2 + 2e^-$
$I_2 + I^- = I_3^-$	Competing	$I_3^- = I_2 + I^-$

FIGURE 4 IODINE VALVE OPERATION

could not be transferred through the gas phase to the electrolytic valve as was possible with Cl_2. This was the important difference that changed the approach to the IGDS from the three-cell concept to solid I_2 storage and on-site flow regulation with one cell, i.e., the electrolytic valve. Other differences between Cl_2 and I_2 chemistry have been reported elsewhere, including standard chemistry texts.(5,6)

Biocidal

A clear distinction between the effectiveness of I_2 and Cl_2 in killing or incapacitating organisms is difficult to make. The principal factors affecting efficient destruction of microorganisms are

a. time of contact,

b. concentration of organisms,

c. concentration of disinfectant,

d. temperature, and

e. solution pH.

A review of the disinfecting literature indicated that both I_2 and Cl_2 can be effective biocidal agents provided the right combination of the above factors exists. It was not an objective of the current program, however, to make a decision or recommendation of the preferred biocidal agent. A report was prepared which did provide a literature summary.(2) The results, however, can be illustrated in Figure 5 by comparing the inactivation of E. Coliform Group microorganisms with I_2 and Cl_2 at low concentrations and pH 6. Both biocidal agents are about equally fast. Other comparisons(7), however, indicate that either I_2 or Cl_2 may have a faster speed of inactivation, depending upon operating conditions or microorganisms.

Figure A-1, Appendix A, illustrates that I_2 and the hypoiodous acid (HOI) formed when I_2 dissolves in water have the broad disinfecting capacity needed for a spacecraft potable water system.

Iodine in Dilute Aqueous Solutions

Evaluation of the effectiveness of any agent for the disinfection of water requires a familiarity with all the chemical reactions that the agent is likely to undergo under actual conditions of use.(8) In the case of I_2, four different substances must be considered. They are elemental I_2, HOI, tri-iodide (I_3^-), and iodate (IO_3^-). The aqueous chemistry of these chemicals are all pH dependent except for I_3^- which has an indirect pH relationship.

Elemental Iodine

The reaction of I_2 with water is

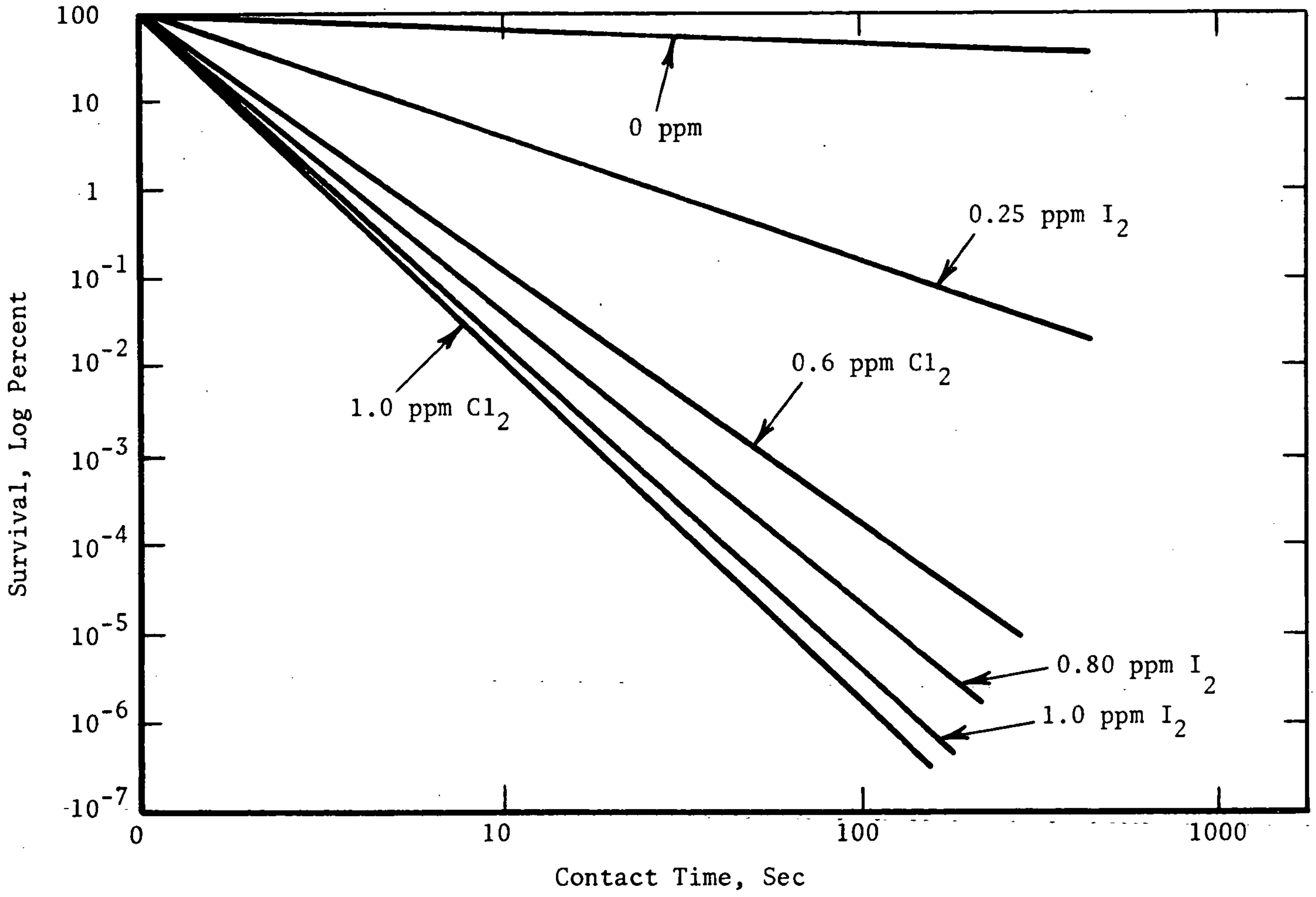


FIGURE 5 INACTIVATION OF E. COLIFORM BY IODINE AND CHLORINE AT pH 6(7)

$$I_2 + H_2O = HIO + H^+ + I^- \qquad (7)$$

with a hydrolysis constant at 298K (77F) of

$$K_h = \frac{(HIO)(H^+)(I^-)}{(I_2)} = 3 \times 10^{-13} \qquad (8)$$

Other I_2 chemistry is summarized in Appendix A, including

1. I_2 solubility in water significantly dependent on temperature (Figure A-2),
2. pH of aqueous solutions of I_2 (Figure A-3),
3. conversion of I_2 concentration from ppm to molarity (Figure A-4), and
4. effect of I_2 and I^- concentration on the reversible oxidation potential for the I_2/I^- couple (Figure A-5).

Hypoiodous Acid

Hypoiodous acid is a primary disinfecting form of I_2. It undergoes ionization to form the hypoiodite ion (IO^-) according to the reaction

$$HOI = H^+ + OI^- \qquad (9)$$

with an ionization constant at 298K (77F) of

$$K_a = \frac{(H^+)(IO^-)}{(HIO)} = 4.5 \times 10^{-13} \qquad (10)$$

Hypoiodous acid is only slightly stronger than pure water as an acid and its ionization is pH dependent

Formation of Tri-iodide Ion

In the presence of added I^-, an aqueous solution of I_2 forms the bactericidally ineffective I_3^- via

$$I_2 + I^- = I_3^- \qquad (11)$$

The equilibrium expression at 298K (77F) for the reaction is

$$K_i = \frac{(I_2)(I^-)}{(I_3^-)} = 1.4 \times 10^{-3} \qquad (12)$$

Because, as shown in Figure 4, the electrochemical reactions involve I^-, the formation of I_3^- becomes a greater factor than in normal I_2 disinfecting processes.

Formation of Iodate Ion

The conversion of HIO to IO_3^- occurs at high pH values (8.0 and over) according to the reaction

$$3HIO + 2OH^- = HIO_3 + 2H_2O + 2I^- \qquad (13)$$

It has been shown that the IO_3^- has no disinfecting ability.(9) Any substantial formation of IO_3^- would, to the extent that it is formed by the consumption of I_2, lower the disinfecting ability of the dose of I_2 added.

Desirable Potable Disinfecting Characteristics

Desirable characteristics of a potable water disinfecting agent include

a. techniques(s) available to monitor its concentration (to ensure effectiveness),

b. no physiological side reactions,

c. applicability to all known organisms,

d. avoidance of corrosiveness,

e. palatability,

f. ease of use,

g. minimum loss of agent/potency on storage, and

h. avoidance of outgassing.

Iodine, in the required dosages, has these characteristics.

Space Shuttle Application

The sterilization procedures for microbiological control considered as applicable for the shuttle water subsystem included pasteurization, addition of Cl_2, bromine, I_2 and Ag^+. Although the pasteurization method was initally selected(10) as the primary water microbiological control methode, I_2 chemical addition was recommended for maintaining bacterial control of the stored backup water. The latter recommendations resulted from the effectiveness of I_2 against a wide spectrum of organisms over a wide pH range with little variation in concentration. In addition, it is compatible for use with all types of water evaporators. More recently Ag^+ has been selected for use aboard the space shuttle vehicle.

Iodine Versus Silver Ion

Results of the IGDS program indicate I_2 has the following advantages over Ag^+:

1. more effective against a broader spectrum of organisms, including viruses, bacteria, fungi (spores), protozoa, and algae, over a wide pH range, with little variation in required concentration,

2. avoids the regular replacement of ion exchange filters used to reduce the Ag^+ concentration prior to consumption of the water,

3. avoids the uncertainty regarding all the conditions (temperature, pressure, pH, flow rate, water line materials, water contaminants, etc.) under which Ag^+ plates out or is reduced to Ag metal,

4. offers the potential for use within the waste water collection cycle of regenerative systems,

5. avoids the difficult sensing of Ag^+ needed to ensure Ag^+ concentration in the water tank and especially, it avoids the sensing needed to ensure that the ion exchange filter has removed excess Ag^+ to the parts per billion range just prior to water consumption,

6. offers greater flexibility in providing an in-flight adjustable dosage level (0.5 to greater than 20 ppm) with the level controllable through the electrolytic valve,

7. provides a simple, regulated process for feeding elemental I_2 into a water supply, and

8. is unaffected by amines which may complex Ag^+.

DETERMINATION OF IODINE GENERATION REQUIREMENTS

Both the quantity of water to be treated and the iodination level influence the IGDS requirements.

Water Flow Requirements

The contractual, Shuttle Orbiter, and Space Station specifications all influenced the water flow specification

Contractual Specification

The contract cited that the prototype was to be sized to iodinate the drinking water nominally consumed by six men, selected within the range of 4.5-13.6 kg (10-30 lb) water per man-day with a ±10 to 20% variation. The minimum water flow rate would be six men x 4.5 kg (10 lb) water per day x 1.1 or 29.7 kg (66 lb) per day. The maximum rate would be six men x 13.6 kg (30 lb) water per day x 1.2 or 97.9 kg (216 lb) per day. (Figure A-6 allows rapid conversion of water flows from kg/day (lb/day) to ml/min (in^2/min)).

Shuttle Orbiter Specification

The amount of potable water required by a four-man, shuttle vehicle crew is approximately 18.1 kg (40 lb) per day. This water is used for drinking (7.1 kg (15.6 lb)), food preparation (3.3 kg (7.3 lb)), washing (4.0 kg (8.8 lb)) and urinal flush (3.6 kg (8.0 lb)). The amount of water available from the fuel cells, however, is determined by the power they generate and their efficiency. For a 5.0 kw power output the projected water produced is 46.3 kg (102 lb) per day.

The difference between the water used by the crew and that generated by the fuel cells is available for heat rejection or for other purposes. It is projected, however, that the entire fuel cell-generated water would have to be iodinated, establishing 54.4 kg (102 lb) per day as a design point for the IGDS.(a)

Space Station Specification

Data presented within the Delta Preliminary Design Package of a Space Station Prototype (SSP) Program(12) indicated the range in water flows expected for the six-man system as shown in Table 1. The range found is 22.8 to 77.6 kg (50.3 to 171 lb) per day.

Iodination Level Requirements

The contract called for an ability to operate the IGDS over a range of 0.5 to 20 ppm. Based on taste tests,(b) however, it appeared that water with concentrations of I_2 greater than 5 ppm might not be palatable for continuous, long-term use (e.g., 90 to 180 days). In addition, the literature indicates a level of 5 ppm, or less if longer contact times are used, is adequate to kill organisms contained in potable water (see Figure A-1). It was concluded, therefore, that the quantity eventually needed for water treatment will be less than 20 ppm. In fact, a 5 ppm maximum concentration is recommended because of the previously cited taste and biocidal characteristics.

Selection of Water Flow Design Point

Selection of a water flow, at a 10% excess over the minimum 27.2 kg (60 lb) per day at an iodination level of 20 ppm, results in an IGDS with a capacity that is also compatible with a water flow of 120 kg (264 lb) per day at a 5 ppm level. This latter water flow rate is large enough to satisfy the contractual, shuttle and SSP requirements at the lower iodination level recommended for flight application. Figure 6 presents the I_2 required as a function of I_2 level (ppm) for various water flow rates.

Prototype Iodine Generation Rate

The I_2 generation or feed rate is related to process water flow rate and I_2 concentration as noted above and is determined by the equation

---

(a) It was reported(11) that the fuel cell water production rate would vary from 54.4 to 76.2 kg (120 to 168 lb) per day.

(b) See section titled "Iodinated Water Taste Test," page 70.

TABLE 1 SPACE STATION PROTOTYPE RECLAIMED WATER FLOW RATES

Source of Water	Flow Rate, kg/Day (Lb/Day) Minimum	Normal	Maximum
Urine Recovery	9.3 (20.5)	15.6 (34.5)	33.6 (74.1)
Cabin Temperature and Humidity and Carbon Dioxide Reduction Subsystems	9.7 (21.4)	18.0 (39.7)	31.2 (68.8)
Total	19.0 (41.9)	33.6 (74.2)	64.8 (142.9)
Plus 10% Excess	20.9 (46.1)	37.0 (81.6)	71.3 (157.2)
Plus 20% Excess	22.8 (50.3)	40.4 (89.0)	77.7 (171.4)

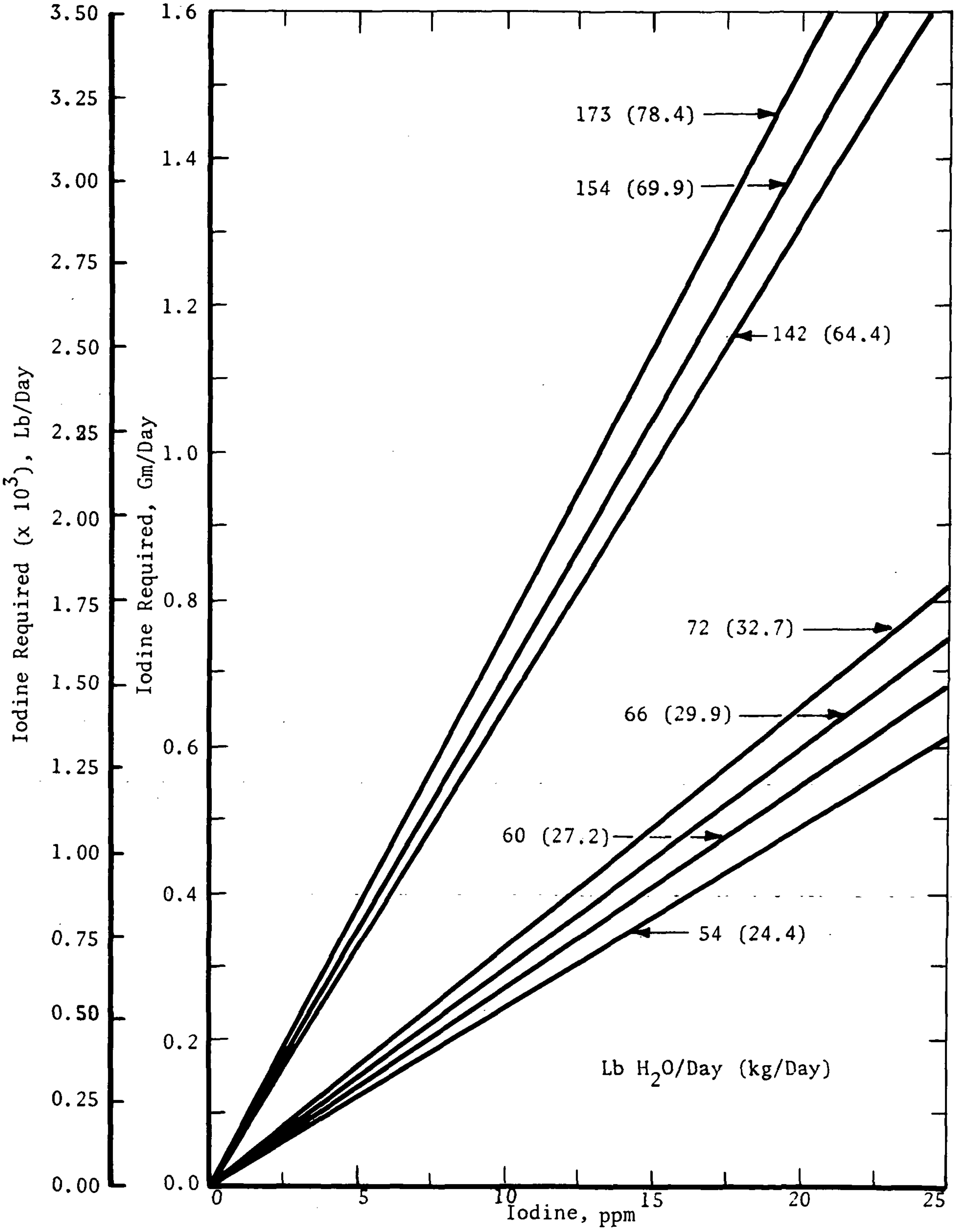


FIGURE 6 IODINE REQUIRED AS A FUNCTION OF FLOW RATE AND CONCENTRATION

$$I_2 \text{ Generation Rate, kg/Day} = (\text{wt. water, kg/man-day})(\text{no. men})(I_2 \text{ conc., ppm})(10^{-6}) \qquad (14)$$

For the current application this becomes

$$\begin{aligned} I_2 \text{ Generation Rate} = (4.5)(6)(20)(10^{-6}) &= 0.54 \times 10^{-3} \text{ kg/day} \qquad (15) \\ &= 1.20 \times 10^{-3} \text{ lb/day} \\ &= 0.54 \text{ gm/day} \\ &= 0.23 \text{ gm/hr} \end{aligned}$$

Another method for expressing the I_2 generation rate is in terms of electrical current required, using Faraday's Law and assuming 100% current efficiency, i.e.,

$$\frac{It}{nF} = \text{moles} \qquad (16)$$

where

I = current, amp

t = time, second

n = number of electrons

F = Faraday's constant, 96,500 amp-sec per equivalent weight of matter

For the prototype application this results in

$$\frac{(0.54 \text{ gm/day})(2 \text{ equiv/mole})(96{,}500)(1000 \text{ mA/amp})}{(254 \text{ gm/mole})(24 \text{ hour/day})(3600 \text{ sec/hr})} = 4.7 \text{ mA} \qquad (17)$$

Figure A-7 presents the I_2 generated as a function of current, assuming 100% current efficiency.

Iodine Storage Capacity

The I_2 that must be stored within the prototype depends upon the process water flow rate, the operating time and the margin added for contingency. For the six-man prototype the quantity of I_2 needed is

$$(0.54 \text{ gm/day})(180 \text{ days})(1.10) = 108 \text{ gm } (0.238 \text{ lb}) \qquad (18)$$

Since I_2 has a density of 4.93 gm/cm^3 (0.178 lb/in^3), the minimum volume of I_2 is 22 cm^3 (1.33 in^3).

Iodine Generating and Dispensing System Design Specification

Table 2 presents the specification used to design the six-man prototype hardware.

TABLE 2 SIX-MAN PROTOTYPE DESIGN SPECIFICATION

Process Water Flow Rate, kg (Lb) per Day		
Nominal	27.2 (60)	
Range (Min.-Max.)	24.4-29.9 (54-66)	
I_2 Generation Rate	Gm/Hr	Lb/Day
Nominal @ 5 ppm Level	0.14	3.0×10^{-4}
Range		
Min. @ 0.5 ppm Level	1.2×10^{-2}	2.7×10^{-5}
Max. @ 20 ppm Level	0.60	1.3×10^{-3}
Set Point	Manually Adjustable	
Water Pressure, kN/m^2 (Psig)		
Nominal	210 (30)	
Range	196-224 (28-32)	
Water Temperature, K (F)		
Nominal	294 (70)	
Range	289-300 (60-80)	
Pressure Drop across Generator, kN/m^2 (Psid)	0.7 (0.1)[a]	
Water Tank Capacity, kg (Lb)	70 (154)	
<u>Water Quality Specification</u>(12)		
Water Circulation Rate, kg (Lb) per Hr[b]	20 (45)	
I_2 Level Sensor Signal Characteristics		
0 to 20 ppm	0 to 5 VDC[c]	
Sample Frequency	TBD[d]	
Control Instrumentation		
Power Source	60 to 400 Hz, 115 VAC, Single Phase	
I_2 Sensor	Simulated	

(a) Design goal with actual level to be less than value cited.
(b) Only applicable to operating modes B and C, see next section of text.
(c) Assumed to be linear although a nominal deviation from linearity is expected at the high I_2 concentration level.
(d) Design will tolerate any sample rate from continuous to 12 samplings per hour.

Prototype Operating Modes

The prototype was designed for a potable water system employing one or more water storage tanks; for example, one tank being filled, one on storage while it is being tested for sterility, and one tank being used. This required that the IGDS be designed to fit each of the six operating modes described below with the help of Figure 7.

Operating Mode A (water line to a tank being filled(a))

In this operating mode, water being reclaimed from various water reclamation subsystems, is accumulated and passes through the IGDS for iodination. The level is established by monitoring the I_2 level downstream of the I_2 valve and using a feedback loop to regulate the I_2 flow rate. The water passes into a storage tank being filled.

Operating Mode B (water line to a tank being filled that contains residual iodinated water from prior use at an unspecified I_2 level)

Operating Mode B is the same as A except the potable water tank being filled contains leftover iodinated water. Since the iodination level of this water would be unknown, it is necessary to recirculate it along with the reclaimed water through the IGDS. It then passes through the I_2 sensor that provides the feedback signal to establish the I_2 injection rate.

Operating Modes C and D (recirculation loop of a tank being used or a tank being used with water being consumed)

These operating modes assume that the IGDS is located in a recirculation loop, including the potable water tank being used. The only difference between Mode C and D is that Mode D assumes water is simultaneously being consumed. This influences water flow rate.

These operating modes are needed when the materials utilized in the potable water tank tend to cause continuous consumption/reaction with the residual I_2. If loss of I_2 from the loop did not occur, these operating modes would not be required.

Operating Modes E and F (recirculation loop of a tank being used with water being added(b) and consumed or a tank being used with water being added only(b))

Operating Modes E and F were not included in the block diagram contained in Figure 7 because they do not reflect operating modes of any projected space missions. These modes are different from Modes C and D in that water is simultaneously being added to the tank being used.

(a) Contract baseline operating mode.

(b) Operating modes added between laboratory breadboard and prototype system testing.

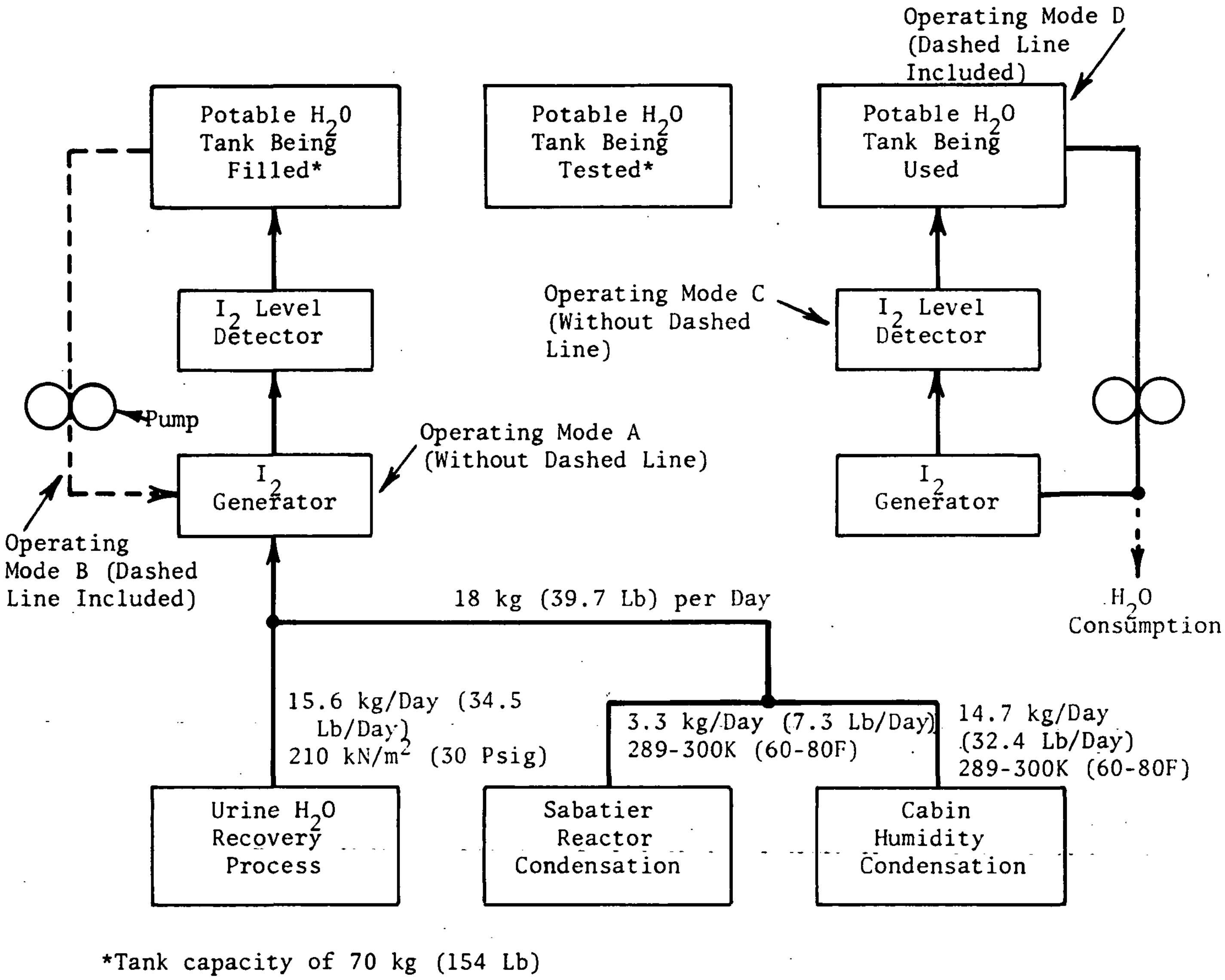


FIGURE 7 BLOCK DIAGRAM OF OPERATING MODES A TO D

IODINE GENERATING AND DISPENSING SYSTEM BREADBOARD

Prior to designing the prototype IGDS, a series of experiments were carried out to verify the concept. The tests were made on the breadboard hardware available(2), as shown in Figure 8, and on various laboratory set-ups. The major purposes were to

a. obtain technical data on the I_2 valve performance,

b. verify the acceptability of dispensing the generated I_2 into the water by flowing the water over the electrode (anode) on which the I_2 was being generated, and

c. verify the applicability of storing the solid I_2 immediately adjacent to the cell's cathode.

The breadboard consisted of an electrolytic I_2 valve, two water reservoirs (one serving as the supply, the other, the iodinated water receiver), a power supply and a water circulation pump. Periodically, and for endurance testing, the water was recirculated instead of a once-through gravity feed operation.

The experimental cells utilized to obtain preliminary data on the I_2 valve were those used on the Chlorogen program and were described previously.(2) They employed electrodes with an active area of 7.62 x 2.54 cm (3.0 x 1.0 in). Figure 9 is a photograph of one of the cells.

The cell consists of an anode, an anion exchange membrane, and a cathode.(a) Adjacent to the anode was a compartment through which the water to be iodinated or reiodinated circulated. Adjacent to the cathode was a compartment where a slurry of water and I_2 crystals were retained. The cell housing was made of plexiglass held together with stainless steel bolts.

The experimental results obtained are presented on page 63 in the section entitled "Laboratory Breadboard Experiments." They indicated

a. an I_2 valve was practical,

b. I_2 could be stored adjacent to the cathode as a slurry,

c. the flowing water leached small amounts of I_2 from the membrane surface, and

d. a build-up of H^+ concentration occurred in the I_2 accumulator compartment.

Regarding Item c, maximum I_2 leaching allowable is a function of water flow rate through the I_2 valve and the desired I_2 level. The larger the flow and the higher the desired I_2 concentration, the smaller the contribution leaching

---

(a) A description of the cell parts is presented beginning on page 27.

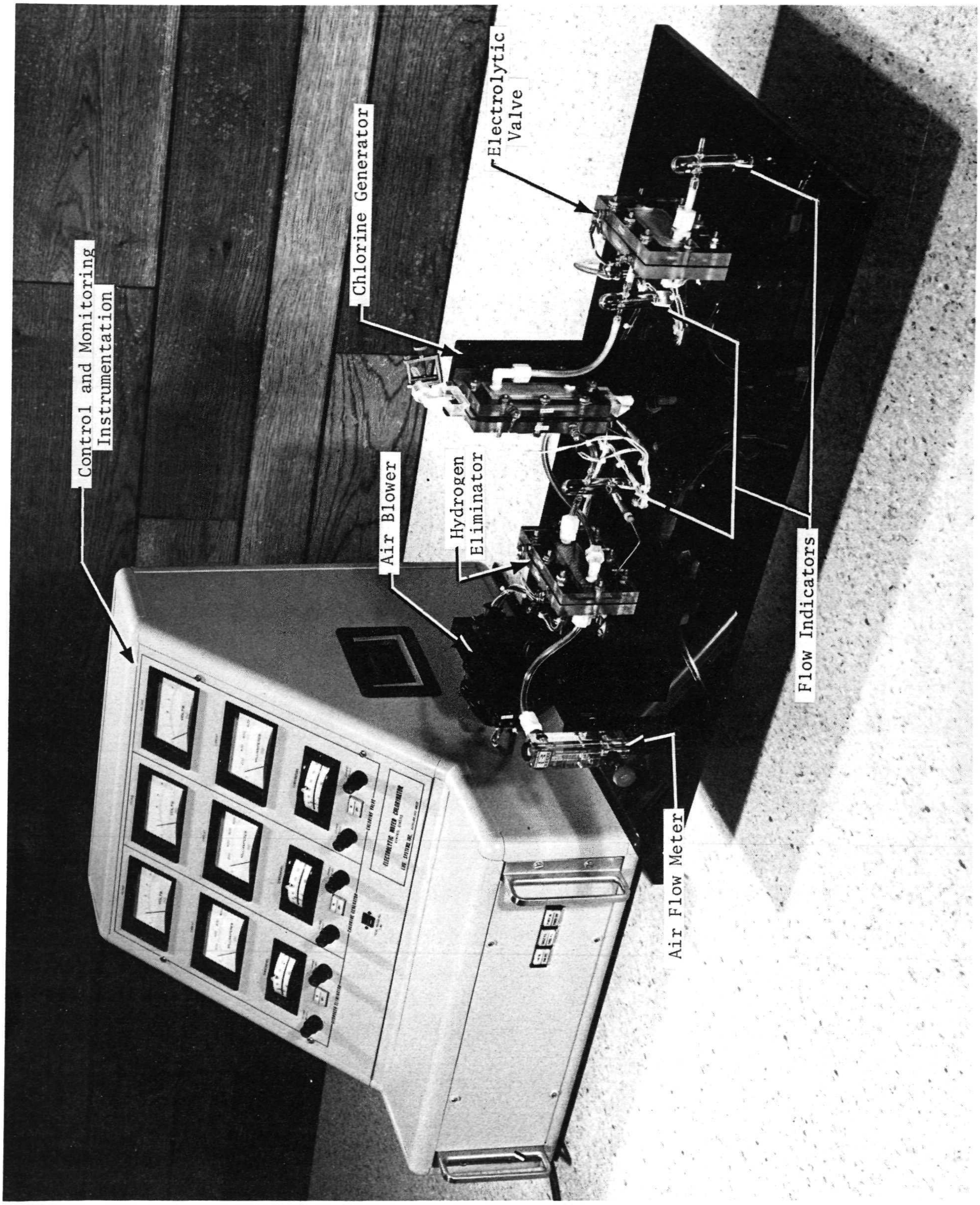


FIGURE 8 LABORATORY CHLOROGEN HARDWARE

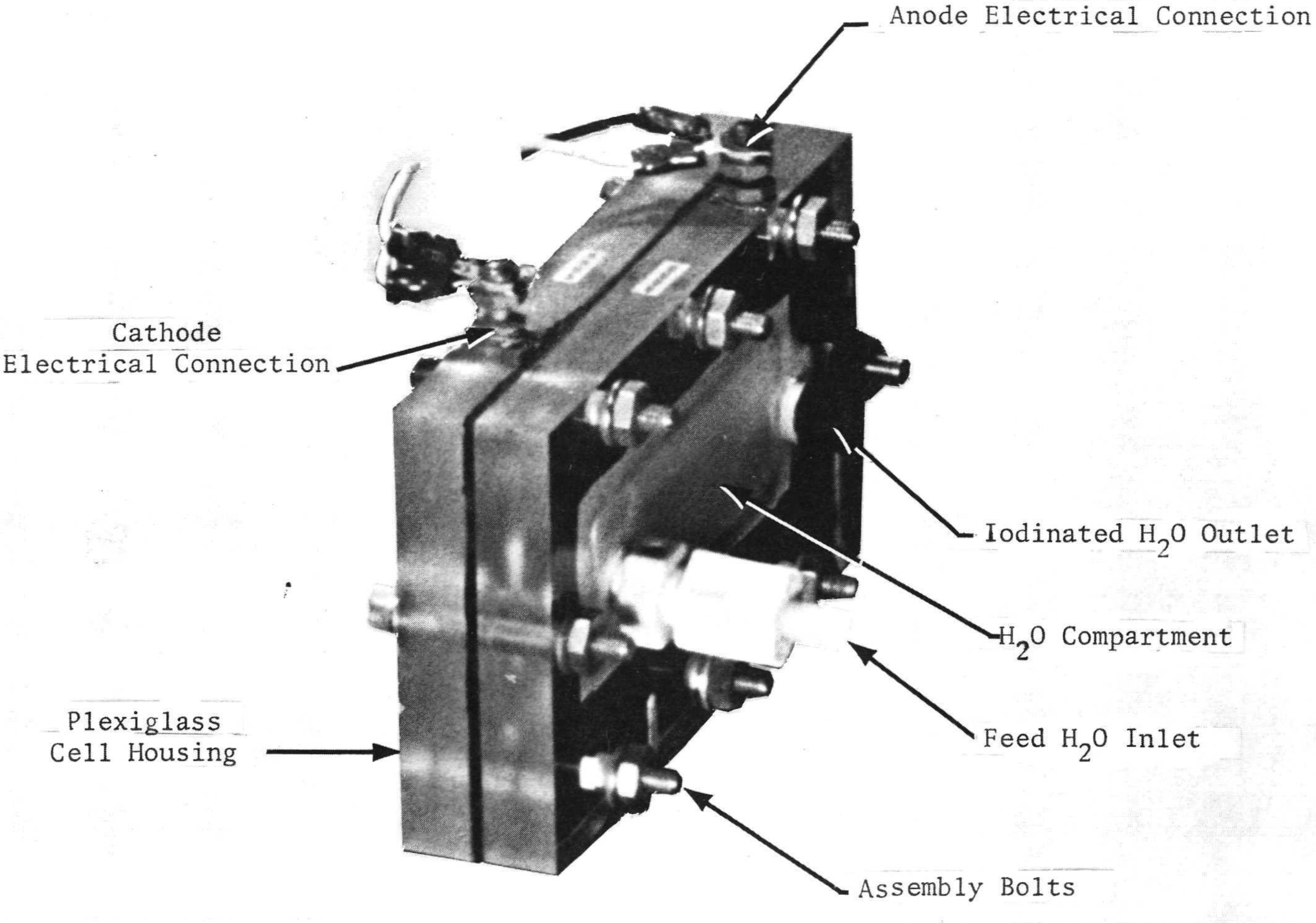


FIGURE 9 EXPERIMENTAL CELL FOR I_2 VALVE CHARACTERIZATIONS

has to the I_2 concentration of the processed water. For the IGDS specification (4.5 kg minus 10% water per man-day and a lower I_2 level of 0.5 ppm) and based on equation (13), the minimum I_2 needed amounts to

$$I_2 \text{ required} = \frac{4.5 \text{ kg}}{1.1} (6)(0.5)(10^{-6}) = 12.4 \times 10^{-6} \text{ kg/day} \qquad (19)$$

or 1.2×10^{-2} gm (2.7×10^{-5} lb) per day. Expressed as electrical current this leach rate amounts to 0.011 mA (per equation (16)).

Regarding Item d, a pH lower than 2 was measured.

IODINE GENERATING AND DISPENSING SYSTEM PROTOTYPE

The IGDS prototype consisted of an I_2 accumulator, the electrolytic I_2 valve, the I_2 dispenser and instrumentation. Figure 10 shows a block diagram of the system integrated into a potable water system and with an I_2 concentration detector.

Iodine Accumulator, Valve and Dispenser

The I_2 accumulator, valve and dispenser functions were combined as shown assembled in Figure 11 and disassembled in Figure 12.

Accumulator

The internal I_2 accumulator volume was approximately 50 cm^3 (3.0 in^3) with dimensions of 9.53 x 2.54 x 2.05 cm (3.75 x 1.0 x 0.81 in). Filled with I_2 at the theoretical density of 4.93 gm/cm^3 (0.178 lb/in^3) provides storage of 244 gm (0.541 lb) of I_2. With 108 gm (0.238 lb) of I_2, it has a storage capacity of 409 days (based on I_2 at 100% density) or 180 days (based on a 56% void volume).

A fiberglass sheet was used to separate the I_2 crystals from directly contacting the electrode. This was not done with all prior cells, but was done in the prototype to determine if the direct contact of solid I_2 against the cathode electrode/membrane surface contributed to I_2 leaching. The change was found to minimize, but not eliminate, the occurrence. A 316 stainless steel screen was used to hold the fiberglass sheet against the plexiglass housing.

Valve

The I_2 valve consisted of an anion exchange membrane between two 70 mesh noble metal screen electrodes.

The primary function of the anion exchange membrane is to immobilize the electrolyte and to prevent the water being iodinated from contacting the solid I_2. While the membrane is conductive to anions (I^-, I_3^-, and OH^-), the cation portion of the electrolyte is immobilized within the membrane. Hydrogen ions,

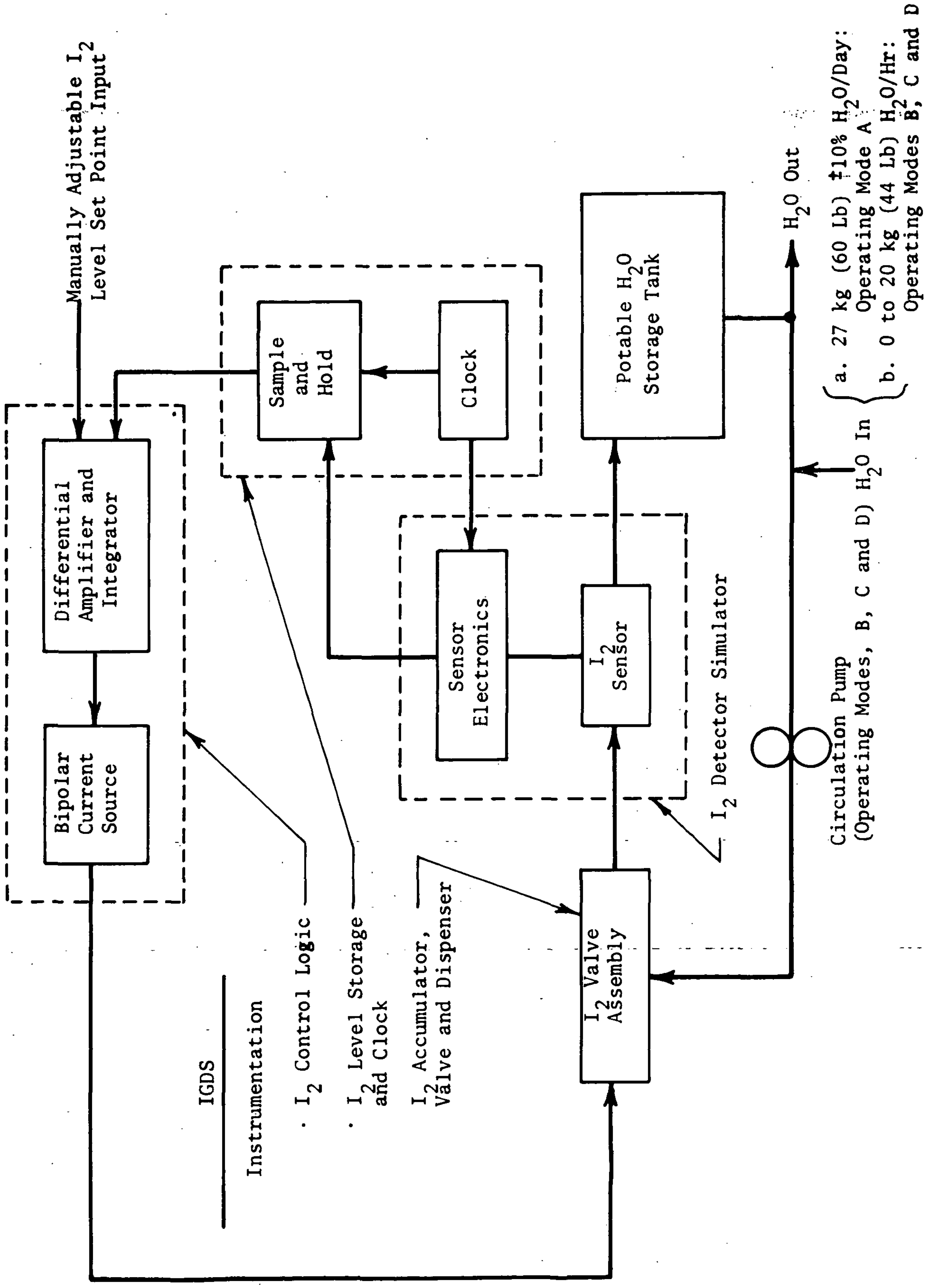


FIGURE 10 IGDS BLOCK DIAGRAM

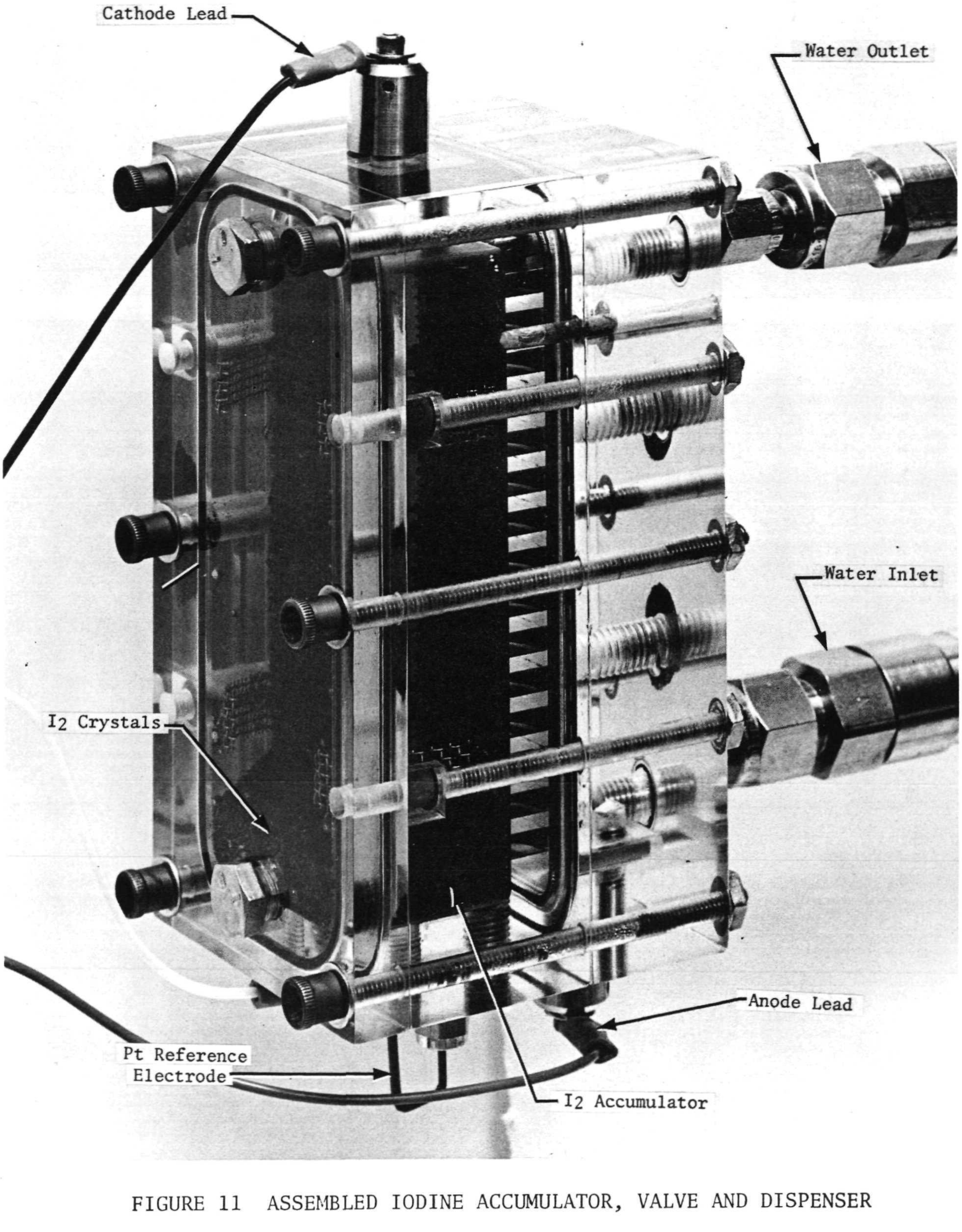


FIGURE 11 ASSEMBLED IODINE ACCUMULATOR, VALVE AND DISPENSER

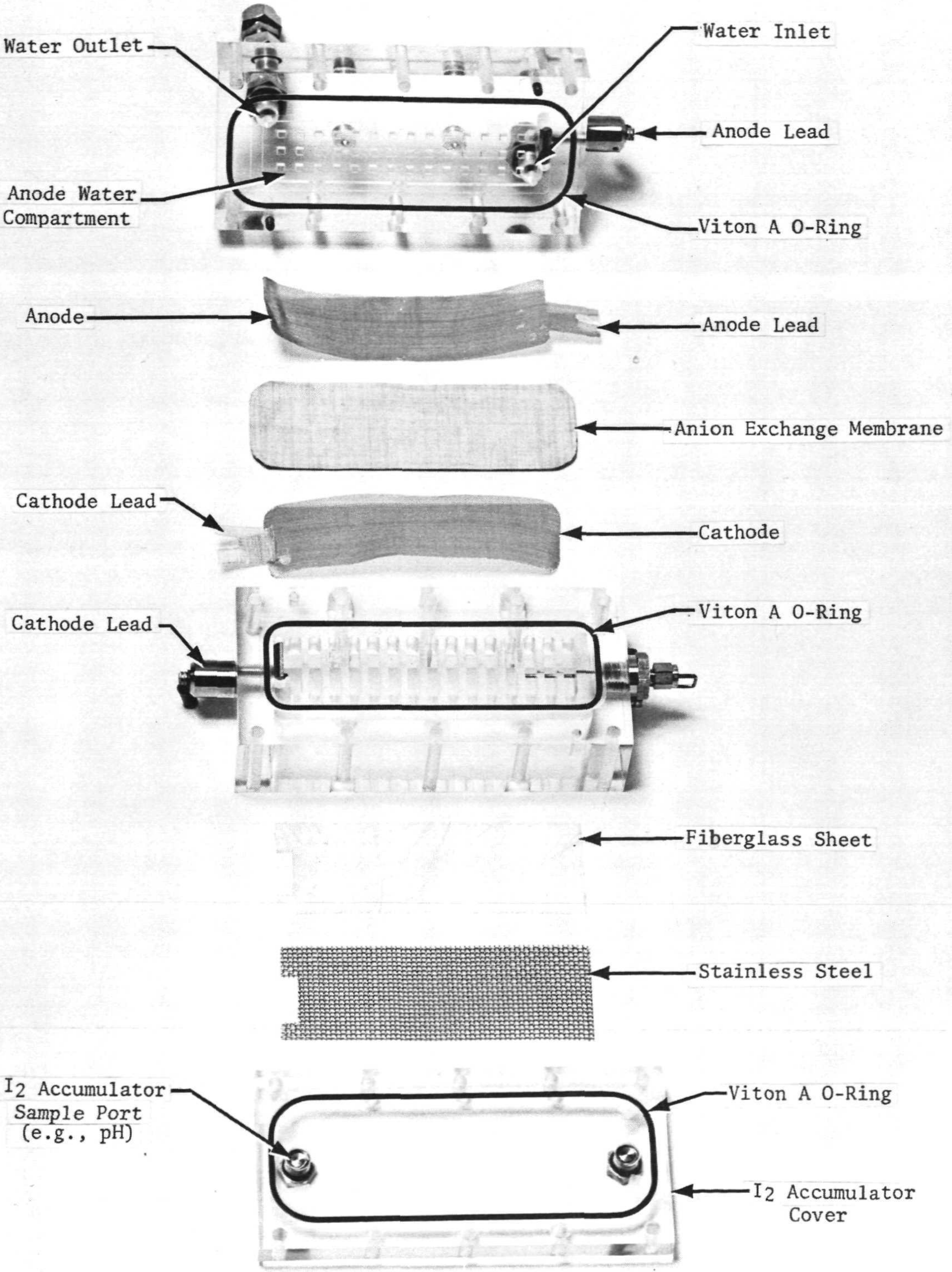


FIGURE 12 DISASSEMBLED IODINE ACCUMULATOR, VALVE AND DISPENSER

water, I_2, HOI, etc. do not pass through the membranes, as a result of the anion exchange mechanism. They may diffuse through, but at an order of magnitude lower rate.

The anion exchange membranes were conditioned prior to incorporation in the cells. This pre-conditioning consisted of soaking the membranes in a stirred solution of 0.6 normal potassium iodide solution for one-half hour. The membranes were then rinsed and again soaked, this time in a 0.1 normal potassium iodide solution for one-half hour. The second step was then repeated. Membranes were always maintained in a wet state.

A reference electrode was included to enable determining at which electrode the polarization occurred. It consisted of a platinum wire immersed in the saturated I_2 solution in the I_2 accumulator adjacent to the cathode.

In the exploratory work the polarization, other than internal resistance, mainly occurred at the anode ($2I^- = I_2 + 2e^-$) with little occurring at the cathode where the slurry existed. In the IGDS more polarization occurred at the cathode because the solid I_2 was retained 0.95 cm (0.38 in) away by the fiberglass sheet.

Dispenser

The I_2 dispenser distributed the water over the valve anode ($2I^- = I_2 + 2e^-$) which was supported on square pegs. The pegs also divided and distributed the water and had 0.25 cm (0.1 inch) sides and were located on 0.64 cm (0.25 inch) centers.

IGDS Instrumentation

The control and monitoring circuits for the IGDS were those necessary for a laboratory/prototype development program but had limited sophistication. They were initially breadboarded and used with the breadboard cell and system testing. For the IGDS they were packaged into the enclosure shown in Figure 13. It contained circuits which

a. monitored the I_2 valve current,

b. monitored the I_2 valve voltages, including anode-to-reference, anode-to-cathode and reference-to-cathode,

c. provided adjustable constant current to the I_2 valve,

d. provided power for the circulation and water feed pumps,(a)

e. monitored the I_2 sensor signal input voltage,

f. provided the temporary storage for I_2 sensor signals, and

---

(a) See section entitled "Potable Water System Simulator," page 45.

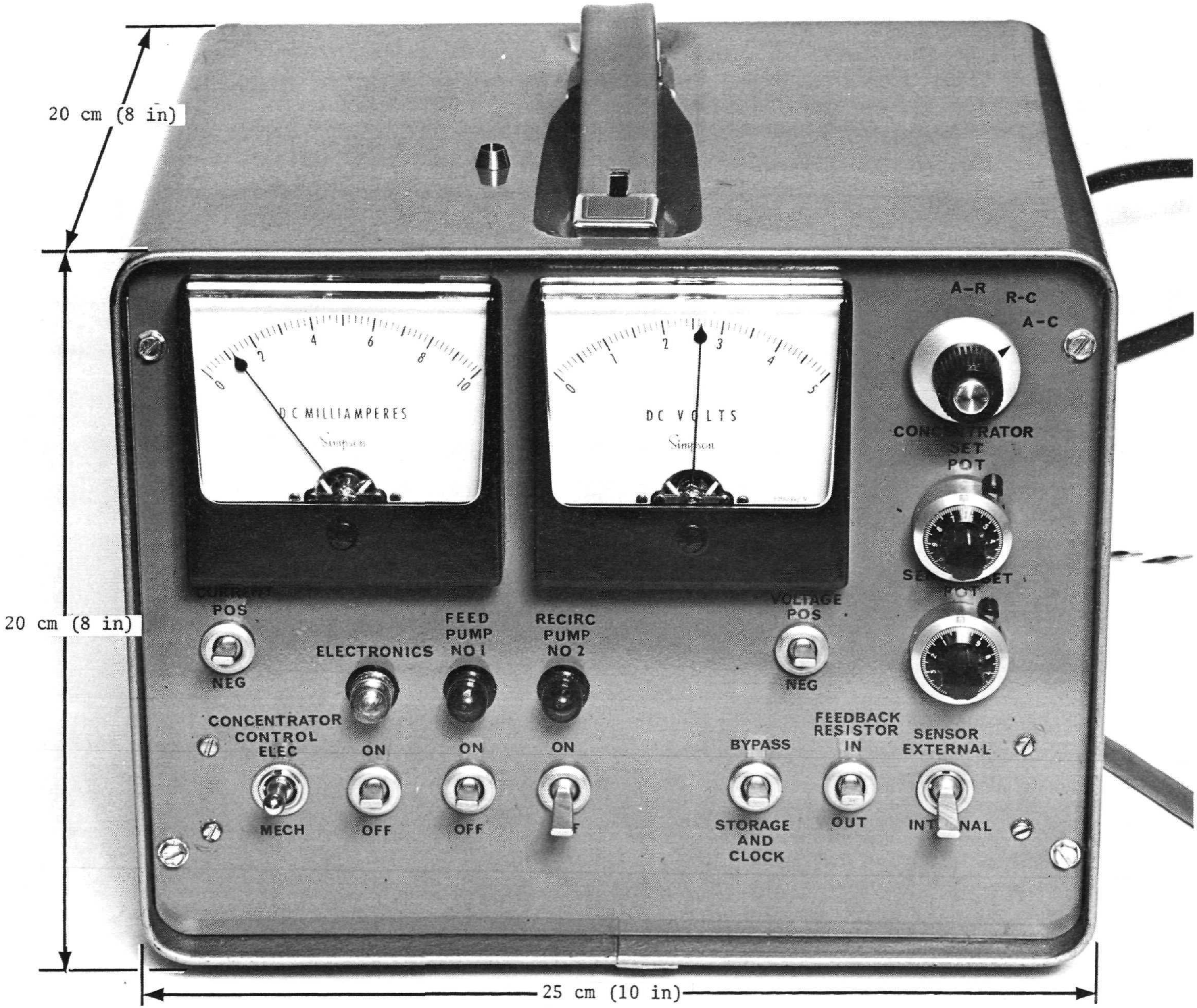


FIGURE 13 CONTROL AND MONITOR INSTRUMENTATION ENCLOSURE

g. provided the I_2 sensor sample activation pulse so that the I_2 level sampling and the internal signal storage systems were synchronized.

The instrumentation has the following features:

1. Closed and open loop control of I_2 concentration.
2. Manually adjustable, I_2 level set point over a 0 to 20 ppm range.
3. Compatability with a 0 to 5 VDC I_2 level signal corresponding to a 0 to 20 ppm I_2 concentration.
4. Sample and hold provisions for periodic I_2 sampling and avoiding sensor noise during sampling.
5. Bipolar current supply, for current reversal, to stop I_2 losses from valve.
6. Constant current source so variations in cell voltage do not affect current flow.
7. Upper voltage limit adjustable to prevent O_2 evolution.
8. Lower voltage limit adjustable to produce appropriate oxidation potential.
9. Ammeter to monitor I_2 valve current.
10. Meter to monitor sensor input voltage, cell anode-to-reference, anode-to-cathode and reference-to-cathode voltages, using a high impedance FET amplifier.
11. All control and monitoring instrumentation in one enclosure.

Control Concepts

Two control concepts were incorporated - integrating and proportional. Each could be operated in a continuously monitoring or periodic monitoring mode.

Integrating, Continuous Monitoring of Iodine Level - A block diagram of the control system with continuous monitoring is shown in Figure 14. A pump circulates liquid to be iodinated through the valve, the sensor and the storage tank. The I_2 level signal from the sensor (E_{FB}) is compared with the I_2 level set point signal established by the adjustable CONCENTRATION SET POT (E_{CS}).[a] Any difference (E) between these two signals (the error) is sent to the integrator. The integrator has an output which is constant only when its input (E) is zero. The integrator output signal is used to control the bipolar current source circuit which, in turn, produces a constant current directly pro-

---

(a) The expressions in capitals refer to the labels used on the instrumentation enclosure's front panel (see Figure 12).

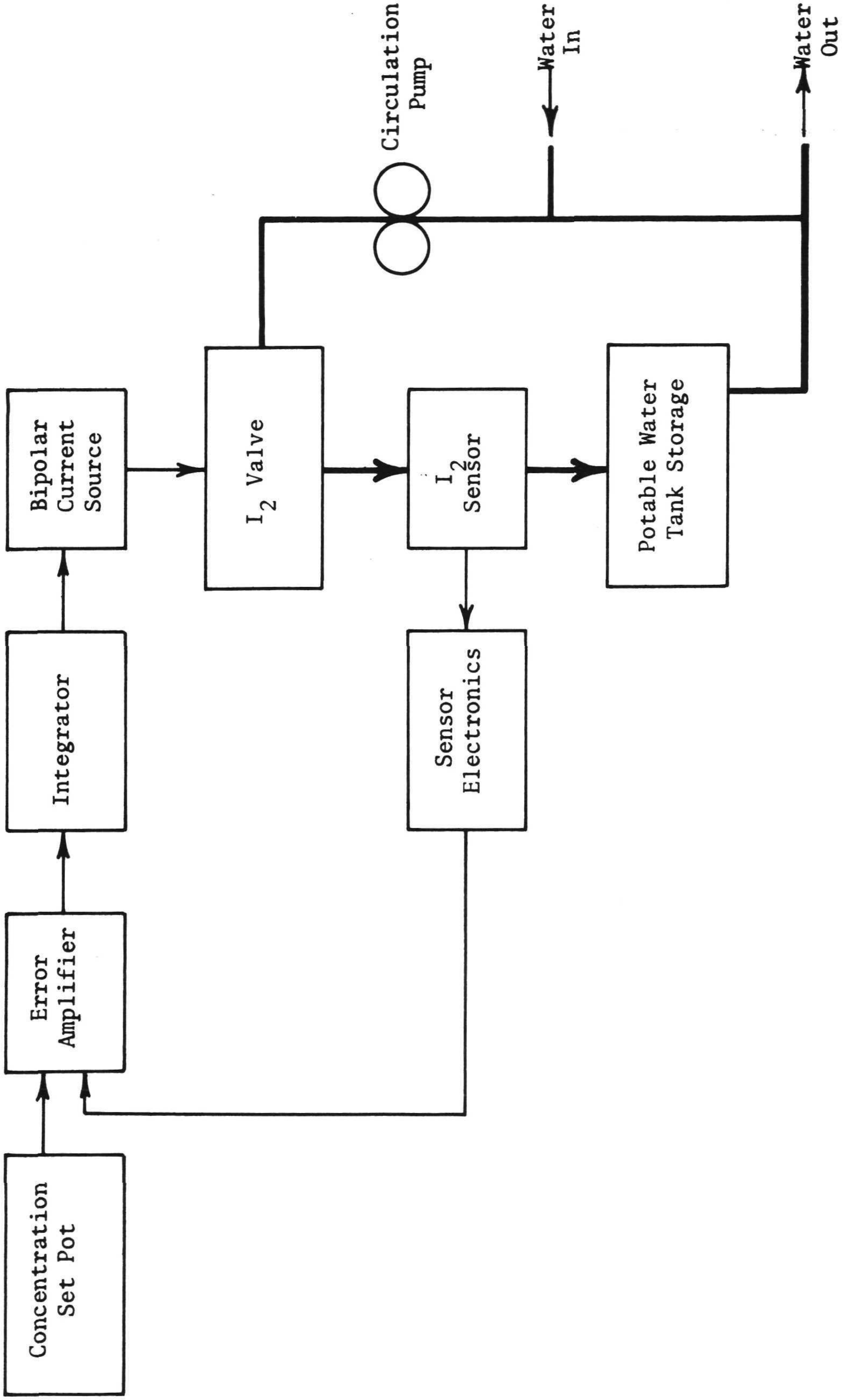


FIGURE 14 IODINE CONTROL SYSTEM BLOCK DIAGRAM (CONTINUOUS MONITORING)

portional to the signal received at its input. The valve generates I_2 and dispenses it into the liquid loop as a function of current flowing through it.

For this system to be in equilibrium, E_{FB} and E_{CS} must be equal which causes the valve to release as much I_2 as is being consumed. If the I_2 level in the circulating loop should decrease, the output from the error amplifier will increase from zero which will cause the output of the integrator to start increasing. This causes a higher current generation and, therefore, a higher I_2 dispensing rate. When the rates of I_2 generation and consumption become equal, the system will reach a new equilibrium and E_{FB} will equal E_{CS} with no output from the error amplifier. However, the output from the integrator and the current source will be at a new value and, in the case illustrated, this value will be higher than the previous one. The system works with zero error, thus the I_2 set point and level will always be identical. The integrator was designed with a very long time constant to compensate for projected system lags.

Integrating, Cyclic Monitoring of Iodine Level - If the sensor is to measure discontinously, a storage system must be inserted between the sensor and the error amplifier, and a clock added as shown in Figure 15. The time constant of the integrator and the rest of the system must be tailored so that relatively small changes in the system operating levels will occur between sample times.

Proportional Controller, Continuous and Cyclic Sampling - If the integrator is replaced with an amplifier, the system will no longer operate with zero error. A difference (i.e., $E_{CS} \neq E_{FB}$) is required because a finite input voltage to the amplifier is needed to provide an output voltage which drives the current source. Figure 16 illustrates system operation.

Assume the control is adjusted to operate along the curve A, B, C, D, E and F of Figure 16 and that the system is operating at point D. If the I_2 level in the circulating loop decreases, the proportional controller will cause the current to move up along the curve from point D and a new equilibrium point will be established when the I_2 generator and consumption rates are again equal, say point C. Note the I_2 concentration is now down to about 8 ppm while the current is up to 10 mA. The error increased to accomplish this. (For purposes of illustration the change was made large, 10 to 8 ppm. In actual practice this difference would be kept very small.) With the proportional type controller, a change in the I_2 concentration will vary the current in the cell until I_2 generation and consumption are equal. In the integrating system there would be no net change in I_2 concentration to accomplish the same results. Therefore, the curve shown by the vertical dotted line through D in Figure 16, represents the integrating system operation.

The actual error at any operating point and the error change in response to changes in I_2 level, depends upon the gain of the amplifier and current source characteristics. Thus, the slope of line B-E (Figure 16) can be varied through an adjustment in amplifier gain as shown in Figure 17. In addition, both the maximum and minimum current levels can be changed by adjustment of circuit components.

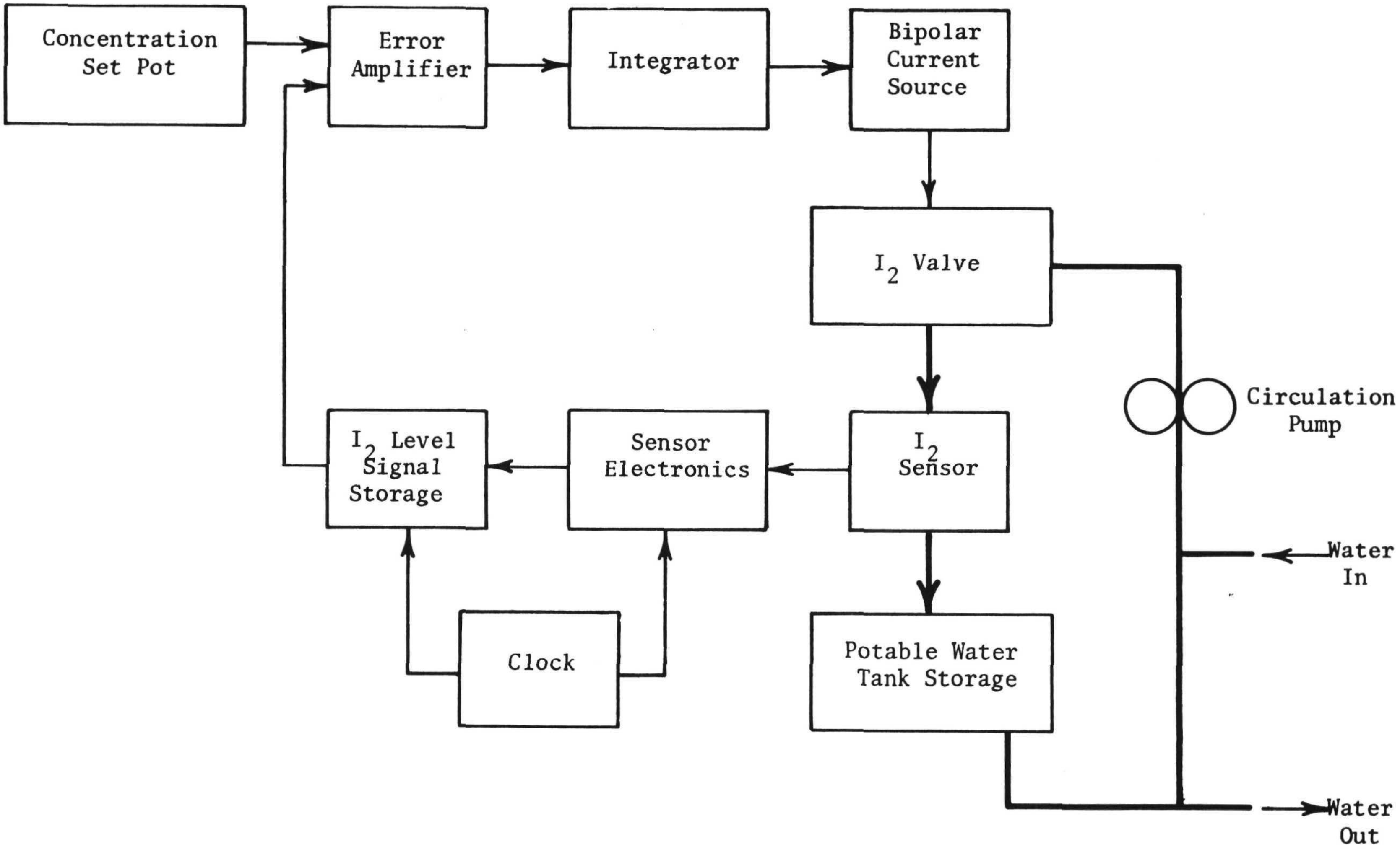


FIGURE 15 IODINE CONTROL SYSTEM BLOCK DIAGRAM (PERIODIC SAMPLING)

"Page missing from available version"

page 37

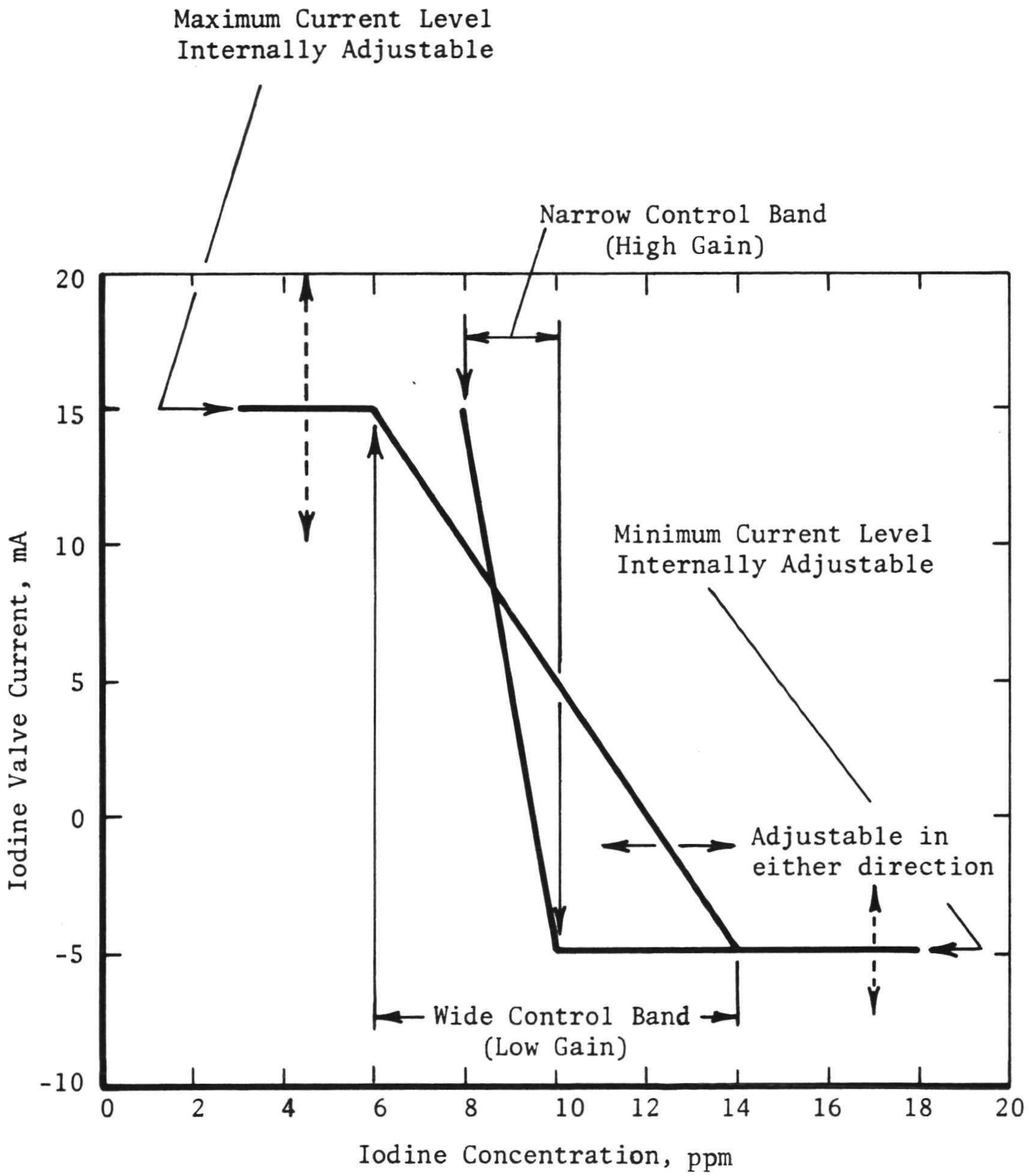


FIGURE 17 PROPORTIONAL CONTROLLER OPERATING FLEXIBILITY

Both modes of operation were incorporated in the controller. The FEEDBACK RESISTOR switch, when in the IN position, converts the integrator to an amplifier. Since the control can be used in either operating mode, it will be possible to study each in subsequent development.

Circuit Construction

The control and monitoring system electronics for the IGDS were constructed on two assemblies:

1. The I_2 valve control logic including bipolar current source, and
2. The I_2 control storage and clock.

An I_2 control sensor simulator(a) and a mechanical I_2 injection control were also added to the enclosure but are not part of the IGDS.(b) These four units were assembled on vector boards and tested independent of each other and used when needed during the breadboard testing. Following this, the four vector board assemblies were installed in a cabinet as shown in Figure 18, along with performance indicators and controls (see Figure 12). Then, the complete package was debugged and tested with the four electronic subassemblies connected together. Once the subassembly was functional, it became the prototype instrumentation.

Control Logic and Bipolar Current Source - A photograph of the I_2 control logic and bipolar current source is shown in Figure 19.

Control Logic. The control logic circuit contains the error detecting and amplifying circuit, as well as the integrator/amplifier. This circuit produces an internal voltage which is proportional to the difference between the I_2 level set point (CONCENTRATION SET POT on the front panel) and the concentration as measured (simulated) by the I_2 sensor. This error signal is fed to a circuit which, as described previously, can be used in either an integrating or an amplifying (proportional) mode with a long lag time. In either mode, the output from this circuit is sent to the bipolar current source.

Bipolar Current Source. The bipolar current source provides the electrical current required by the I_2 cell. It is a controlled, constant current source which produces a current (independent of cell voltage) whose magnitude and direction are controlled by the signal from the control logic. The magnitude of the maximum current in either direction is internally adjustable. Circuits are also incorporated to limit the maximum voltage which can be applied to the cell in either direction by internal component adjustment.

Storage and Clock - Figure 20 is a photograph of the Storage and Clock.

(a) Discussed in the section entitled "Ground Support Accessories," page 45.

(b) A mechanical I_2 injection concept was carried along in parallel until the electrolytic valve concept successfully passed the 30-day test with the breadboard hardware.

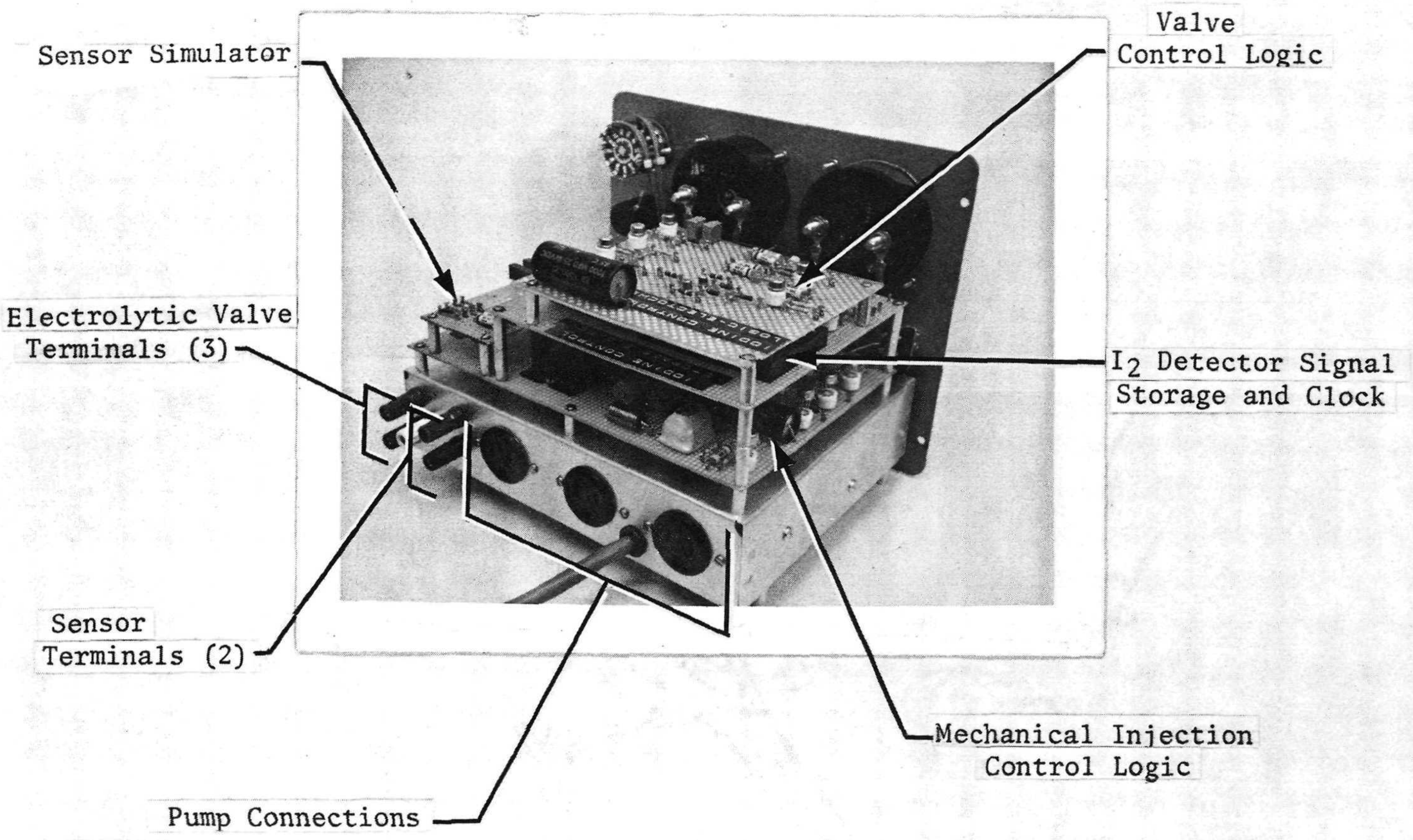


FIGURE 18 ELECTRONIC CONSTRUCTION AND MOUNTING

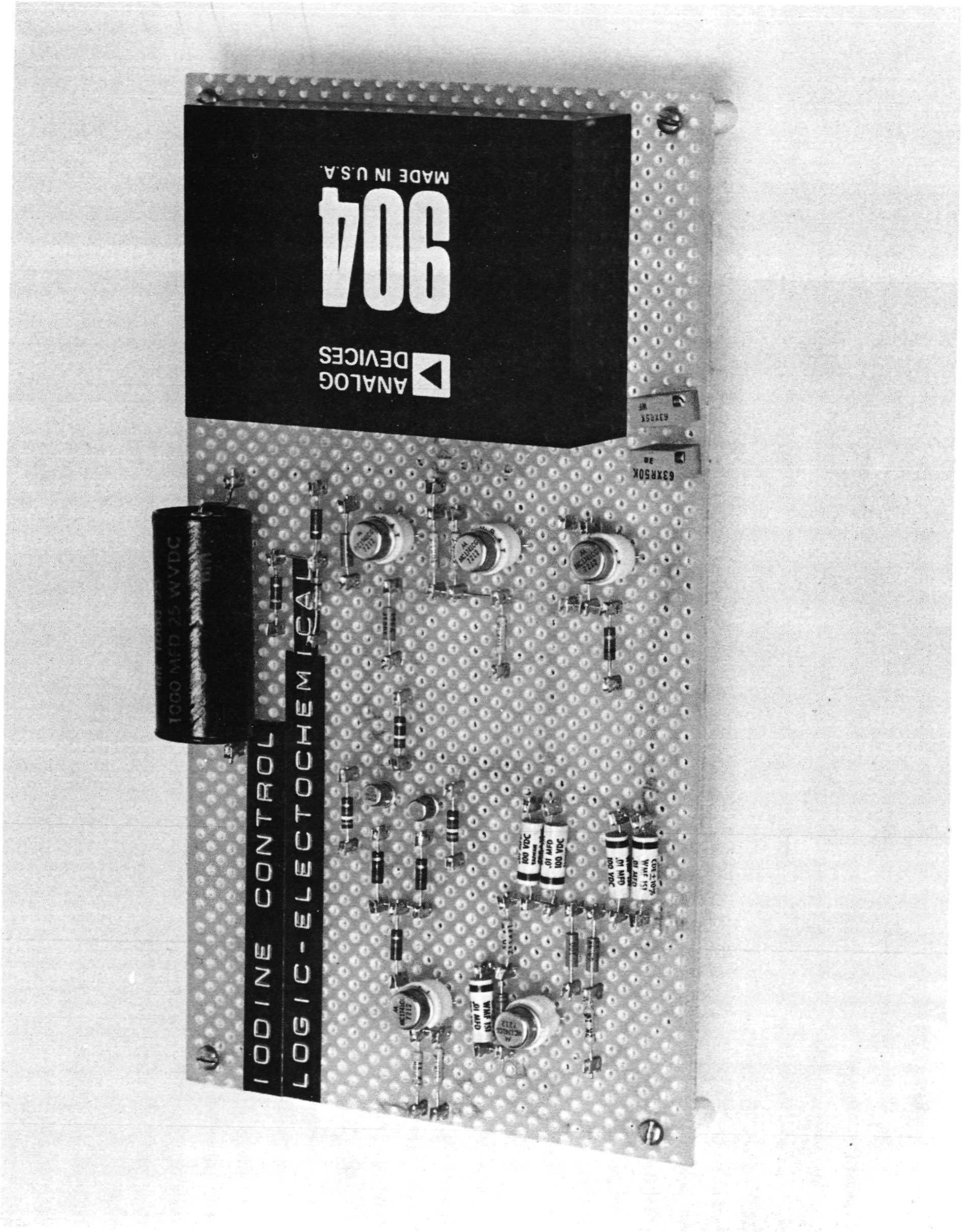


FIGURE 19 IODINE CONTROL LOGIC AND BIPOLAR CURRENT SOURCE

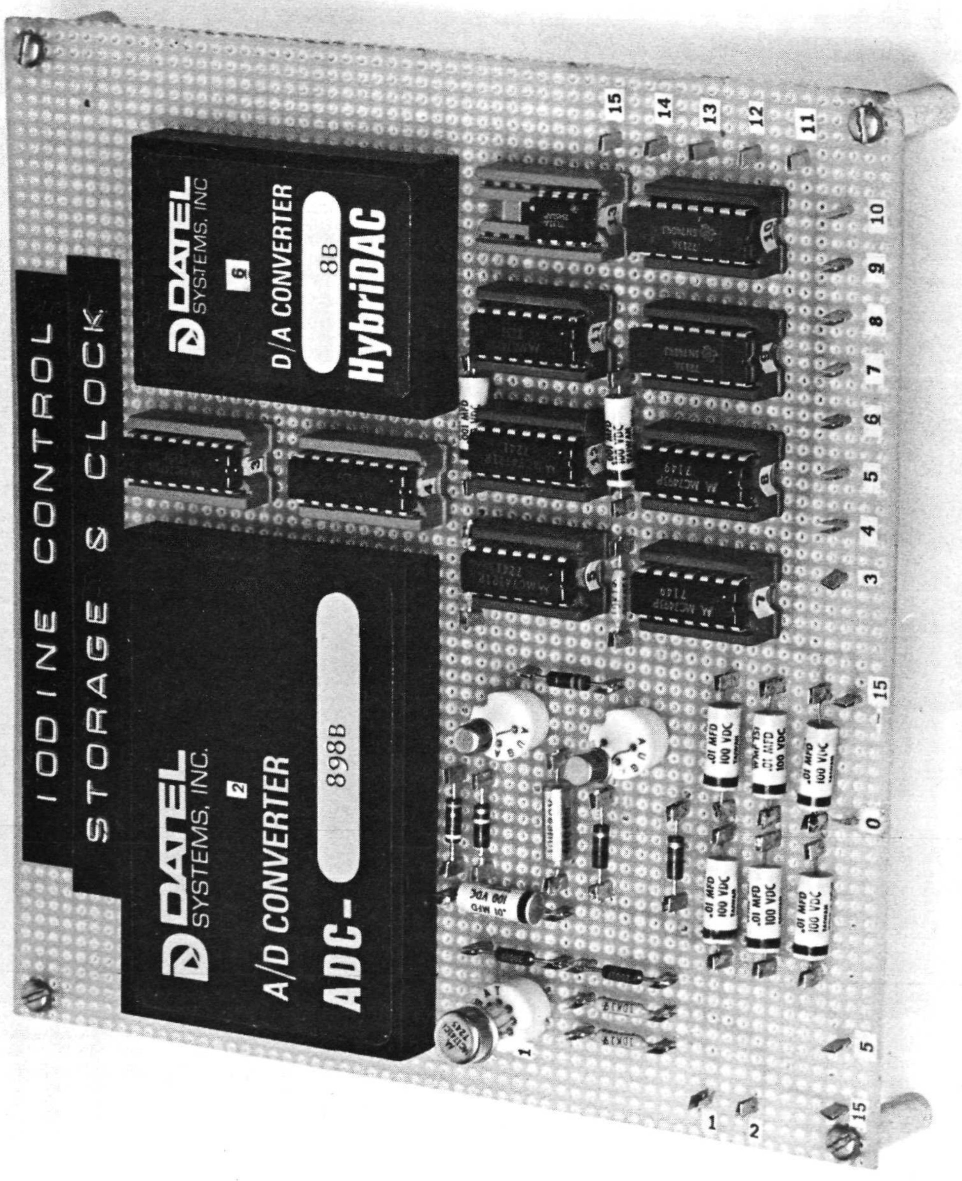


FIGURE 20 IODINE CONTROL STORAGE AND CLOCK

Storage (Iodine Sensor). The I_2 sensor, being developed by NASA, measures I_2 concentration at discrete time intervals of one minute or longer.(3) An accurate measurement of I_2 concentration will only be available after each measurement is made. The sensor signal storage circuit was, therefore, developed to accept an I_2 level signal and store it until a new sensor signal is obtained.

Figure 21 is a block diagram of this storage system. When a signal from the I_2 system is to be stored, a clock signal will cause the I_2 sensor signal to be converted to an eight-bit digital number that is then stored in eight bistable devices. Output from the storage is converted from digital to analog which is used in the I_2 control system. The stored signal is assumed to be the latest I_2 concentration and will be maintained until a new clock signal is received, at which time the system will store the updated I_2 sensor signal. By storing the digital form of the I_2 sensor signal, the length of time which the signal is stored can be extended indefinitely. Conventional analog storage techniques have a very limited storage time.

System Clock. The system clock, consisting of a free-running unijunction oscillator feeding an eight-bit binary divider provides the means for synchronizing the operation of the I_2 sensor with the storage circuit. Outputs from the eight-bit divider are connected to two decoding arrays. One array provides a signal to the I_2 sensor to initiate a reading. The other decoding array provides a signal to initiate storage. These two clock output signals are set to occur at different times. The I_2 sensor is automatically instructed to measure. After a time interval during which the measurement is taken and signals are allowed to stabilize, the sensor storage system is automatically instructed to store the stabilized I_2 level signal.

As assembled, the clock produced a sample signal to the I_2 sensor, followed by a storage pulse to the storage circuits every 32 seconds. The time between the store and sample pulses can be varied in one second increments and the cycle time can be varied in one second increments up to a maximum of 256 seconds, by retuning the decoding array circuits.

Control Adjustment Summary

Operation of the IGDS is automatic. No control adjustments are needed. Two adjustments are provided, however, for system operating flexibility, the CONCENTRATION SET POT and the I_2 SENSOR SET POT. Once the instrumentation is integrated with an I_2 sensor, only the CONCENTRATION SET POT will be needed.

Because this was a development program, several factory adjustments and wiring modifications were provided which can change the performance characteristics of the IGDS. These include

a. system clock timing (set at 32-second intervals),

b. maximum and minimum current of the bipolar current source (set at +10 and -5 mA, respectively),

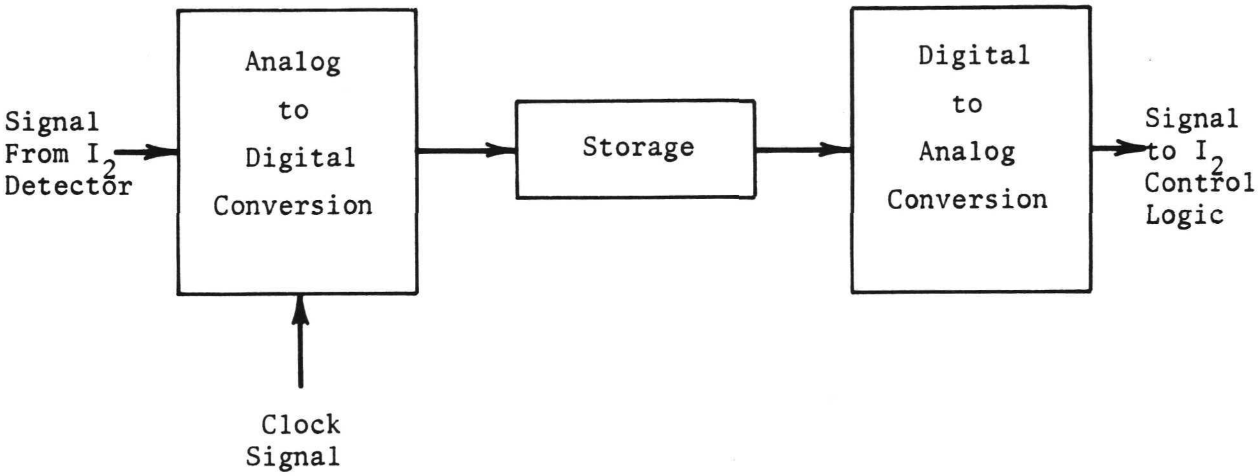


FIGURE 21 SENSOR STORAGE BLOCK DIAGRAM

c. the maximum voltage (positive and negative direction) of the bipolar current source (set at +8 and -1 volt, respectively), and

d. the gain and time constant of the integrating amplifier and control logic.

Mechanical Controller

The control system for mechanical I_2 injection was designed and the circuits built (see Figure 22), tested and installed in the instrumentation package. A variable speed pump, which can be used to mechanically dispense I_2 to the circulating loop is operated by this system. The pump can be operated by setting the CONCENTRATION CONTROL switch to the MECHANICAL position and plugging the I_2 dispensing pump into the proper receptacle on the back of the package. In this mode of operation the only modification is that the bipolar current source is replaced with an adjustable, variable voltage supply that varies pump speed.

The control logic, system clock, I_2 sensor signal storage and sensor simulator circuits all operate as previously described when integrated with the mechanical controller.

GROUND SUPPORT ACCESSORIES

Ground Support Accessories were needed to (a) check out cell materials of construction, (b) obtain exploratory data on cell operation, (c) simulate a potable water system, and (d) measure I_2 concentration and aqueous solution parameters.

Material Testing Setups

The procedure and setups used to test proposed IGDS materials of construction are discussed in the Material Testing section on page 56.

Experimental Cells

The experimental cells and laboratory breadboard were discussed in the section, Iodine Generating and Dispensing System Breadboard on page 24 and illustrated in Figures 8 and 9.

Potable Water System Simulator

The Potable Water System Simulator (PWSS) was used to simulate the interface between the IGDS and the projected operating environment. Figure 23 presents its schematic. Table 3 identifies the PWSS components and indicates which are needed as a function of the six operating modes described on page 22. In addition to providing a source of water to be iodinated, it also contained provisions for monitoring flows, pressures and temperatures. It had taps for obtaining water samples. Figures 24 and 25 are photographs of the front and back of the PWSS. Major components of the PWSS were the water storage tank and water recirculation pump.

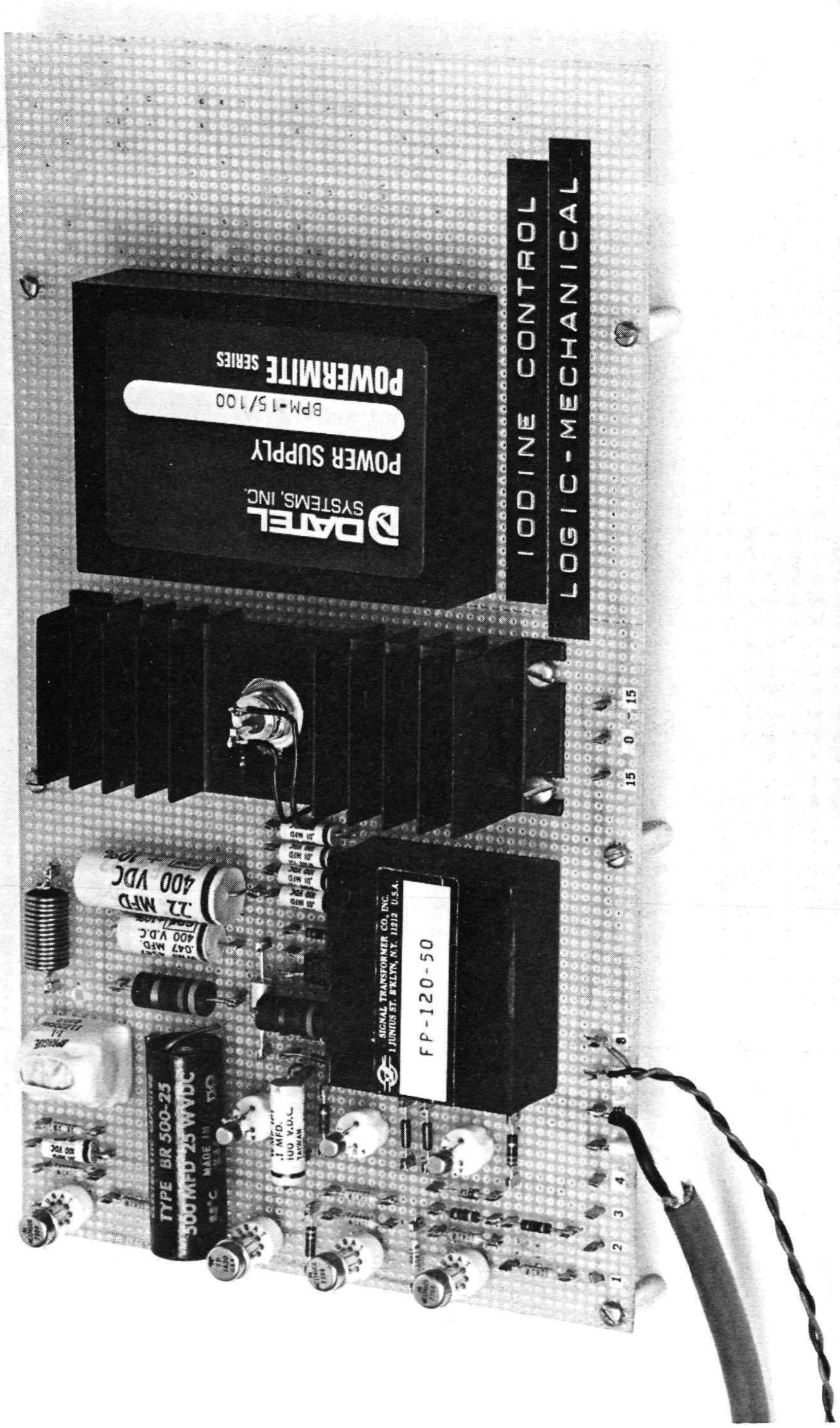


FIGURE 22 IODINE CONTROL LOGIC FOR MECHANICAL INJECTION

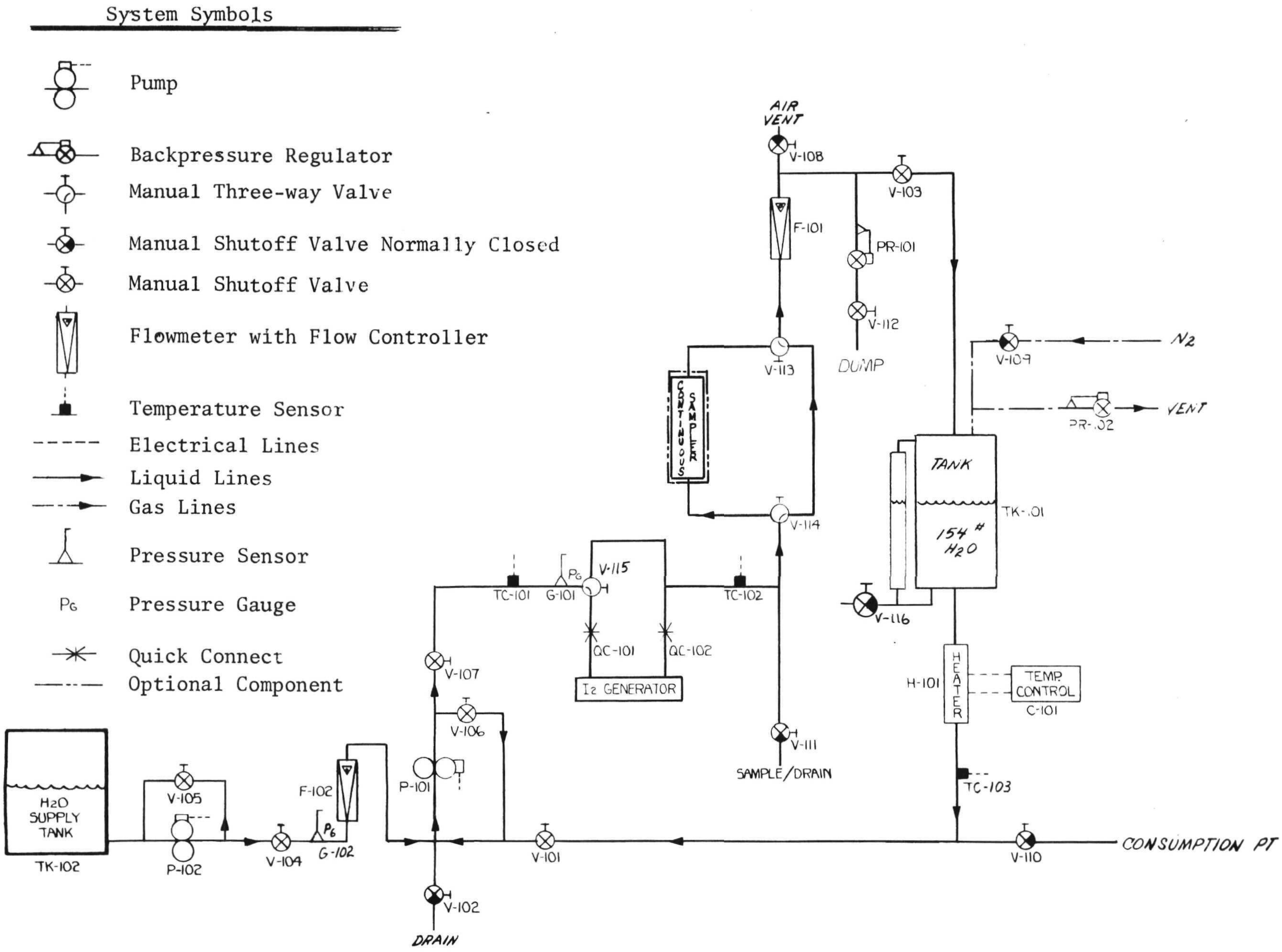


FIGURE 23 POTABLE WATER SYSTEM SIMULATOR

TABLE 3 POTABLE WATER SYSTEM SIMULATOR PARTS LIST

LSI Part No.	Part Description	Mode of Operation[(a)] A	B	C	D	E	F
TK-101	Water loop tank to simulate spacecraft water storage	x	x	x	x	x	x
TK-102	Source water supply tank to fill and refill loop	x	x			x	x
H-101	Heater to heat water temperature			x	x	x	x
P-101	Pump to recirculate water within the loop	x	x	x	x	x	x
P-102	Pump to deliver H_20 from supply tank to loop	x	x			x	x
C-101	Controller to regulate H-101 and maintain water temperature			x	x	x	x
F-101	Flowmeter controlling flow during recirculating modes			x	x	x	x
F-102	Flowmeter to control and monitor flow of H_20 from TK-102	x	x			x	x
PR-101	Backpressure regulator to maintain loop H_20 pressure	x					
PR-102	Backpressure regulator to maintain loop H_20 pressure	x	x	x	x	x	x
G-101	Measure H_20 pressure upstream of IAVD	x	x	x	x	x	x
G-102	Measure H_20 pressure downstream of IAVD	x	x			x	x
QC-101	Allow rapid installation of IAVD	x	x	x	x	x	x
QC-102	Allow rapid installation of IAVD	x	x	x	x	x	x
LSI-100	I_2 Accumulator, Valve, and Dispenser (IAVD)	x	x	x	x	x	x
TC-101	Thermocouple measure H_20 temperature upstream of IAVD	x	x	x	x	x	x
TC-102	Thermocouple measure H_20 temperature downstream of IAVD	x	x	x	x	x	x
TC-103	Thermocouple measure H_20 temperature after the heater			x	x	x	x
V-101	Valve controlling flow between loop H_20 tank and pump			x	x	x	x
V-102	Valve enabling loop water to be drained out	x	x	x	x	x	x
V-103	Valve	x	x	x	x	x	x
V-104	Valve controlling flow from source water tank	x	x			x	x
V-105	Pump by-pass flow control valve	x	x			x	x
V-106	Loop recirculation pump by-pass flow control valve	x	x	x	x	x	x
V-107	Recirculation control and pump backpressure control valve	x	x	x	x	x	x
V-108	Air vent valve for draining water loop	x	x	x	x	x	x
V-109	Nitrogen flow control valve	x	x	x	x	x	x
V-110	Water consumption flow control valve				x	x	
V-111	Water sample control valve	x	x	x	x	x	x
V-112	Water drain valve	x					
V-113	Valve allowing continuous I_2 level sampling	x	x	x	x	x	x
V-114	Valve allowing continuous I_2 level sampling	x	x	x	x	x	x
V-115	Valve allowing H_20 flow to by-pass the IAVD	x	x	x	x	x	x
V-116	Water loop tank drain valve	x	x	x	x	x	x

(a) See page 22 for definition.

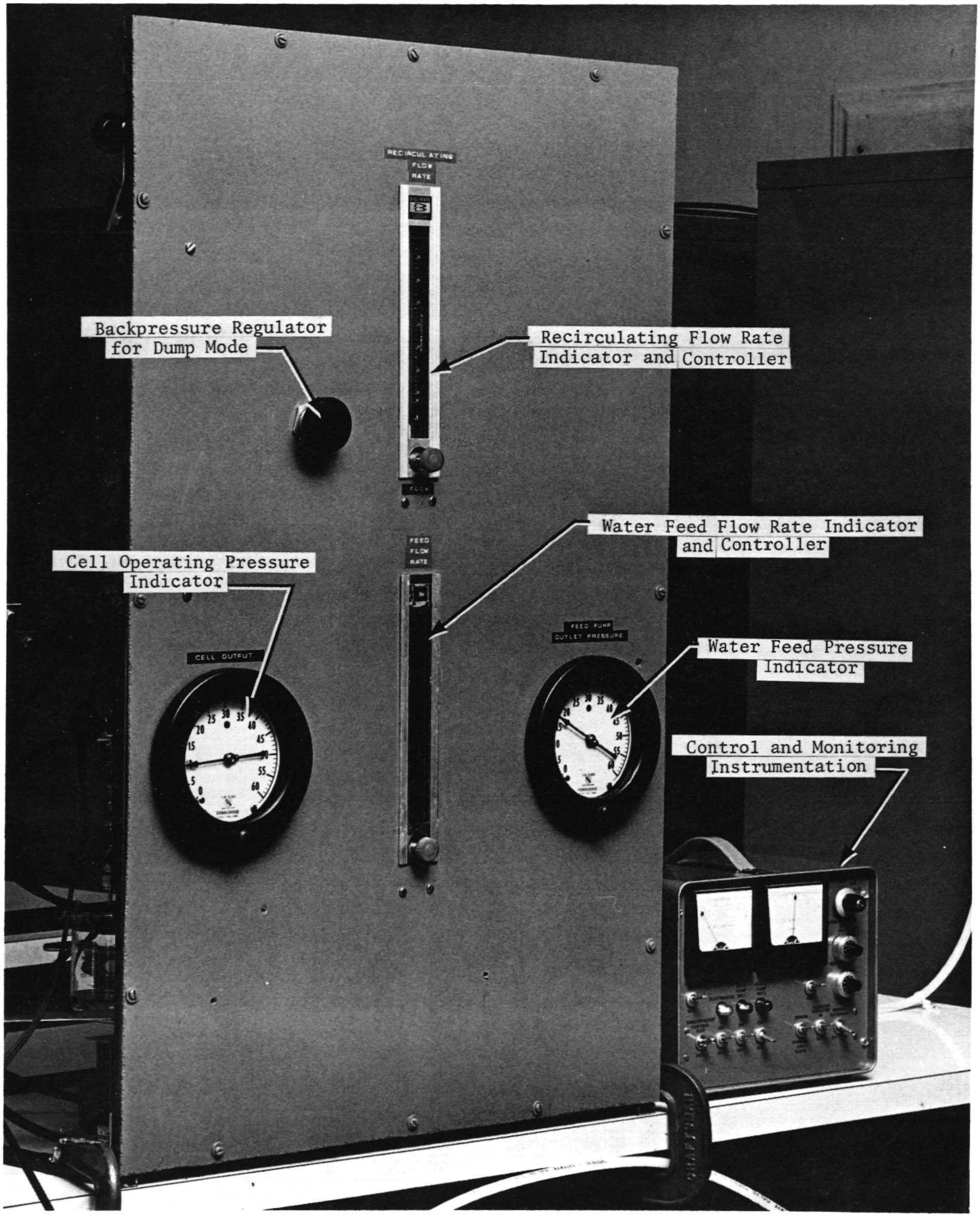


FIGURE 24 POTABLE WATER SYSTEM SIMULATOR, FRONT VIEW

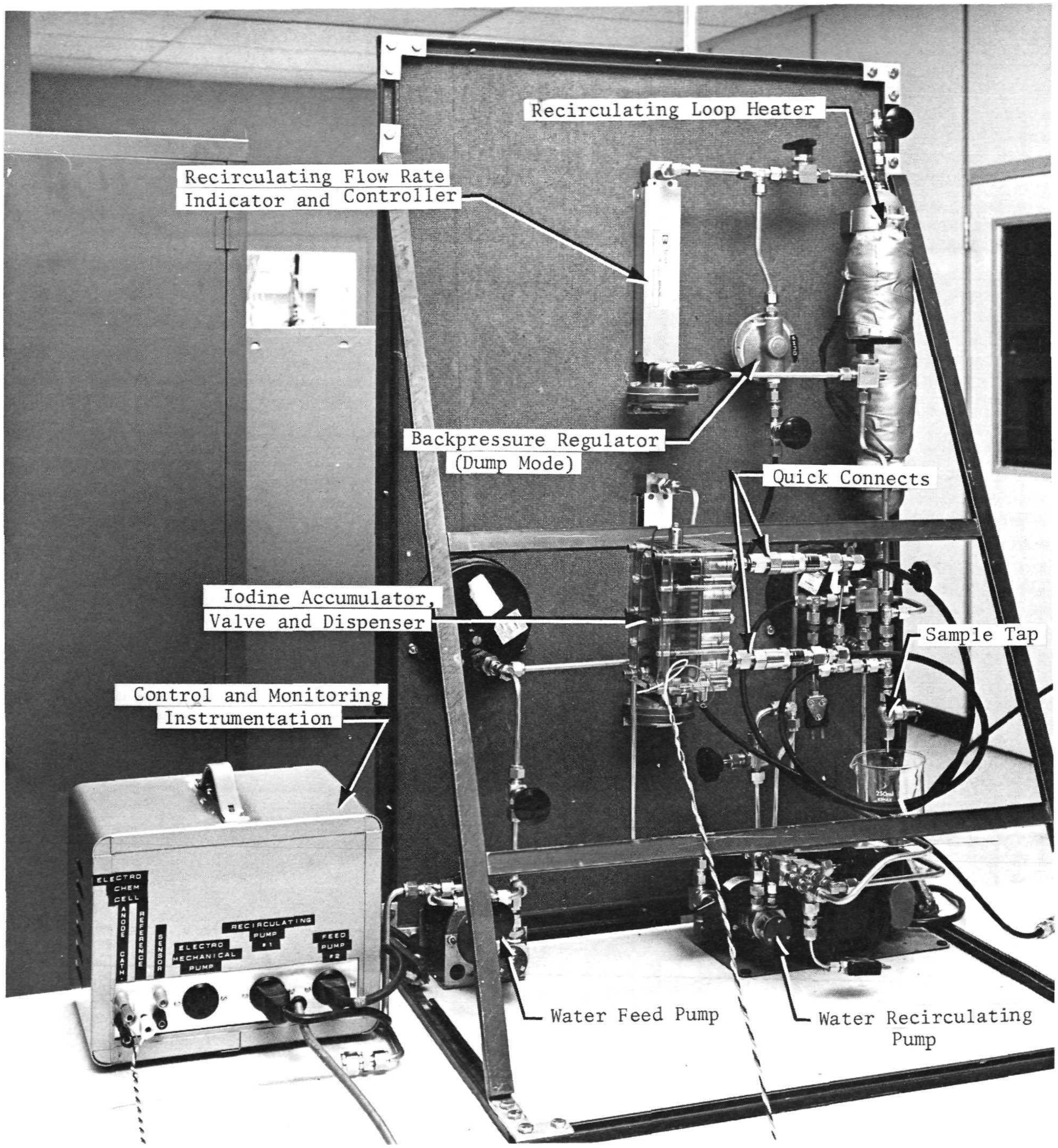


FIGURE 25 POTABLE WATER SYSTEM SIMULATOR, REAR VIEW

Storage, Fill and Water In Use Tank Simulator

Figure 26 is a schematic of the tank design that was fabricated. It has a usable water volume of 79 liters (2.8 ft^3), which is sufficient to hold 69.9 kg (154 lb) of water with 13% ullage gas volume. The tank was made from 316 stainless steel. It employed an external glass tube for reading the liquid level in one-liter intervals as calibrated by emptying the tank into a graduated 12 liter (0.42 ft^3) container (see Figure A-7).

Water Recirculation Pump

A gear pump was used to circulate water within the PWSS. It has a capacity of 3.8 liter/min (1 gal/min) at 210 kN/m^2 (30 psig). It is capable of operating up to 2100 kN/m^2 (300 psig) at 366K (200F). A magnetic coupling is used to eliminate possible leakage.

Iodine Detector Simulator

A circuit was constructed to simulate the I_2 sensor signal (see Figure 27) because the Iodine Colorimeter was not available.(3) This allowed the system to be operated in an open loop mode so system operating parameters could be measured. The simulator was installed in the instrumentation enclosure (see Figure 18). The only parameters which could not be determined using the detector simulator approach are the closed loop control stability and gain requirements. (To determine these requires using the actual detector with the feedback loop closed around the system.)

Packaged Monitor Instrumentation and Accessory Controls

Figures 13 and 18 present photographs of the packaged IGDS controller. The controller also contains the PWSS pump controls, parameter monitor instrumentation, the mechanical feed controller, and power converters.

Analytical Considerations

During the course of the program, measurements were made of the concentration of I_2, I^- and H^+ in the various waters tested. Provisions for measuring I^- concentration and pH were made, using an in-line plastic block to retain a calomel, a glass electrode and I^--specific electrode. Because the fiber liquid junction of a standard calomel electrode is difficult to maintain in a flowing solution, valves were installed with a bypass arrangement. Care was taken to insure that the solution had stabilized, through the formation of a stable liquid junction, before a meter reading was taken.

Measurement of Iodine Concentration

Two methods were used to measure the level of I_2 within the water: colorimetric and thiosulfate titration. An evaluation was made of the interval between taking a sample and its analysis. The purpose was to insure that samples taken on Saturdays, but not analyzed until the following Monday morning,

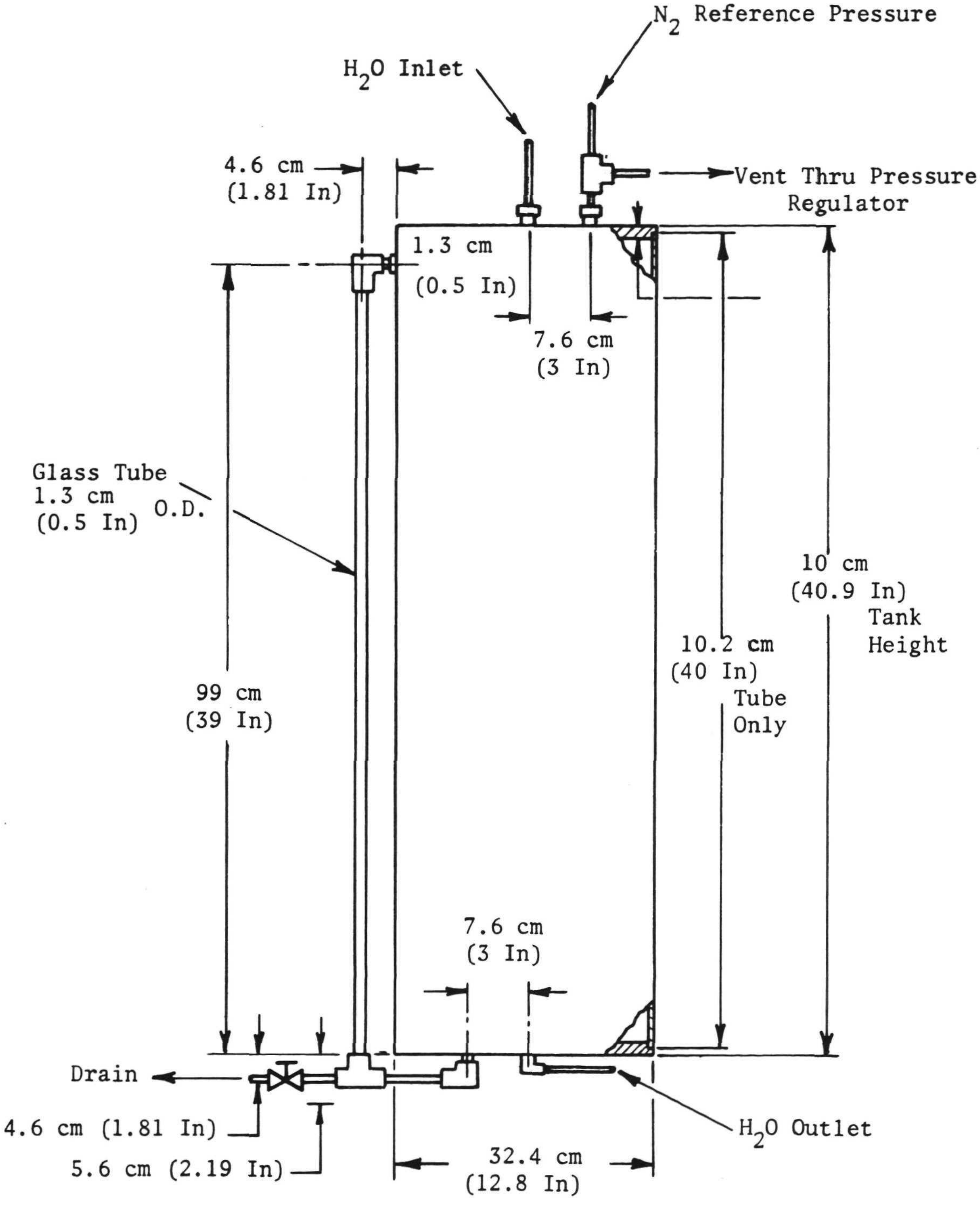


FIGURE 26 POTABLE WATER SYSTEM SIMULATOR WATER TANK

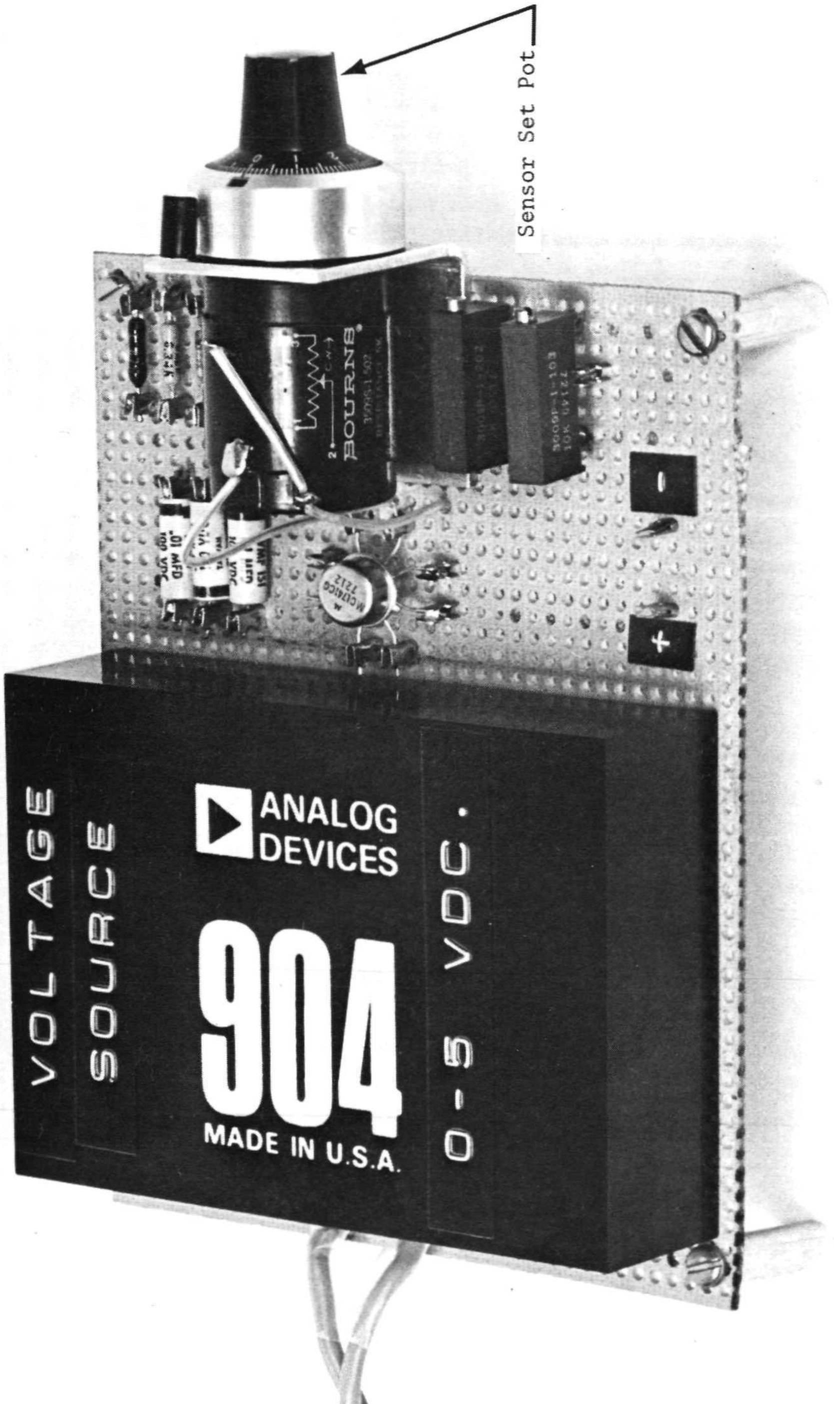


FIGURE 27 IODINE SENSOR SIMULATOR

would not have a low I_2 level because of loss of I_2 from the solution. The results indicated[(a)] that analyses of I_2 solutions, within the first 40 hours, gave almost identical readings. As the storage time increased to beyond 50 hours, however, the I_2 level measured decreased sufficiently to reflect a lower level than that considered to be within experimental accuracy.

Colorimetric Method - Spectrophotometric measurements of I_2 concentration for the potable water were made with a Fisher Electrophotometer II, Model 81. The colorimetric procedure offers the advantages of speed, simplicity, sensitivity and lower cost over the amperometric method.(8,14)

The procedure followed was essentially an adaptation of the procedure by Black & Wittal.(15) The colorimetric method was most suited for low concentrations of I_2. Figure A-10 presents the calibration curve obtained over the concentration range 0.5 to 2.0 ppm, using an optical path of 23 mm. Figure A-11 presents the calibration curve obtained over the concentration range 5 to 25 ppm, using an optical path of 50 mm.

Thiosulfate - Levels of I_2 concentration above 2 ppm were determined using the standard thiosulfate process.(16)

Hydrogen Ion Concentration

The concentration of the H^+ (pH) of solutions were determined with a pH meter. It was calibrated periodically as illustrated in Figure A-12.

Iodide Ion Concentration

Two methods were used to determine the concentration of I^-.

Specific Ion Electrodes - A specific ion, I^- electrode was used, employing a pH meter to measure the electrode voltage. The electrode was calibrated as illustrated in Figure A-13.

Oxidation to Iodine - Since I^- is colorless and cannot be measured photometrically, it had to be oxidized using an "Oxone" solution. A solution containing both I_2 and I^- was first photometrically analyzed for I_2. Then "Oxone" was added to oxidize the I^- to I_2 and the solution again photometrically analyzed. The difference between the readings was taken as the value of the I^- concentration.

PRODUCT ASSURANCE

A mini-Product Assurance Program was included so the impact of manned chamber testing requirements would be included during the initial design activities. The functions included were quality control, maintainability, safety and nonmetallic material control. Quality control was added to ensure reproducibility of cell design and configuration during subsequent developmental activities. Maintainability was included to ensure that the subsystem would

---

(a) See Figure A-9.

have a design and configuration that could be operated and maintained by personnel not associated with its development. Safety was included in anticipation of manned chamber requirements. Nonmetallic materials control was included in preparation for the nonmetallic material and outgassing specification(17) required of equipment to be operated within manned spacecraft.

The Product Assurance Program was carried out during the prototype IGDS development.

Quality Control

The Quality Control activities performed during the fabrication and assembly of the prototype IGDS consisted of

1. performance and documentation of receiving inspections on all vendor supplied parts,
2. maintaining a record of all rejected parts and authorized rework,
3. insuring that assembly techniques specified in the design drawings were complied with, and
4. configuration control on all design drawings.

This minimum activity insured that no defective components or parts were incorporated into the IGDS and that design drawings correctly reflected the progression of the design from the initial concepts through the final engineering drawings.

Maintainability

The design was evaluated for maintainability. Only nominal considerations were needed, however, since the application configuration remained unspecified. The prototype's I_2 accumulator was configured so that rapid filling with solid I_2 crystals was possible. The prototype interfaced with the PWSS using quick connect fasteners.

Design analyses performed indicated no maintainability problems are anticipated for a flight unit; e.g., no ion exchange cartridges need replacing, the I_2 valve is a static device, etc.

Safety

The IGDS is inherently safe. A Safety Hazard Analysis, however, was used to verify that no system or system component characteristics would be dangerous to personnel or equipment. The disinfecting concept is essentially a static device that operates at room temperature. It does not employ high voltages, hot temperatures, rotating components, etc.

Iodine Generating and Dispensing System Materials Program

The candidate materials control program conducted during the design and fabrication of the IGDS included

1. surveying the literature for materials compatibility to I_2 and HOI[a],
2. performing compatibility tests above to verify acceptability in the environment projected for the IGDS,
3. maintaining a record of all nonmetallic materials used, and
4. guiding the design so that only those materials or configurations that meet the requirements of D-NA-022(17) are used.

Survey Results

The literature and vendor survey revealed that polypropylene, cast acrylic (Plexiglas), Teflon, Viton, Fluorel, ethylene propylene, epoxy resins, polysulfone, polyamides and nylons were candidate nonmetallic materials.(18-22) Table 4 summarizes the results. The metallic materials: 316, 316L and 321 stainless steel were selected as candidates. Under a separate contract,(7) it was found that of six nonmetallic materials studied (silastic, polyisoprene, butyl rubber, Nordel, Viton and polyurethane), silastic was the most acceptable because of minimum I_2 sorption, I_2 conversion, or I_2 substitution reaction. Butyl rubber, Nordel and Viton were unacceptable because of the taste and odor they contribute to water when interacting with I_2. Polyisoprene and polyurethane were unacceptable because of continuous I_2 sorption.

Material Testing

To verify their acceptability, several of the materials listed above were subjected to compatibility tests. Two tests were performed: one in which the samples were placed in a saturated solution of I_2 (250 to 300 ppm) at room temperature, and an accelerated reaction test in which the samples were placed in a solution saturated with I_2 (1100 ppm) at 338K (149F), an arbitrarily selected elevated temperature.

Room Temperature Test - A sample piece of the proposed material was placed in a glass bottle with a ground-glass top containing a saturated I_2 solution. Before exposing the material to the I_2 solution, each piece was cleaned with a soap solution, dried at 339K (150F), weighed, and visually checked with the naked eye and a 14X power microscope. Where appropriate, materials were also dimensionally measured using a micrometer.

---

[a] In addition, chemical compatibility data was gathered for Cl_2 and hypochlorous acid so that both the I_2 and Cl_2 generator approaches to water purification could be compared from a materials point of view.

TABLE 4 IODINE AND CHLORINE MATERIAL COMPATIBILITIES(a,b)

	Iodine			Chlorine	
	Solid	10% Solution	500 Ppm Solution	Gas	Satd. Water
Polyvinylchloride	U	S	F(20)	U	S
Penton	S	S	--	U	U
Polypropylene	U	S	S(20)	U	S
Polyethylene	S	--	--	S	S
Fiberglass Reinforced Epoxy	U	S	S(20)	--	S
Viton	S	S	--	--	S
Teflon	S(19)	--	S(20)	--	S(19)
Cast Acrylic (Plexiglas)	--	--	U(20)	--	--
Polysulfone	--	S	S	--	F
Fluorel	--	S	S	--	S
Fluorocarbons	S(18)	--	--	S(18)	--

(a) All data was obtained from Ref. 16 except when noted.
(b) Key:
S = Satisfactory   U = Unsatisfactory
F = Fair   -- = No data available

Test solutions were prepared by placing I_2 crystals in deionized water until the solution was saturated. Only the saturated water (without any solid I_2 crystals) was placed in the glass bottles. Every two weeks the solution was standardized to determine I_2 lost, then appropriately brought back to the initial concentration.

Table 5 presents the room temperature soak test results observed with the I_2 saturated, aqueous solution. Teflon, polypropylene, polyvinylchloride and Plexiglas were found to be acceptable nonmetallic materials. Only Teflon failed to take on the brown, I_2 stain or to continuously consume (remove) I_2 from the saturated solution. The acceptable metallic materials were 316 stainless steel and platinum.

High Temperature Test - Sample pieces of the proposed materials were placed in a single glass flask containing deionized water and I_2 crystals. The flask was heated to 338K (149F), resulting in an I_2 saturated solution of ≈1100 ppm. (This is about four times the saturation level of water at 298K (77F)). The test was continued for approximately three and a half months. Care was taken to insure that solid I_2 crystals were always present within the water.

Table 6 presents the 338K (149F) accelerated soak test results observed with the I_2 saturated, aqueous solution. Teflon, polypropylene and polyvinylchloride were found acceptable. Only Teflon failed to take on the brown, I_2 stain. Platinum was an acceptable metallic material. The 316 stainless steel had marginal acceptability at the elevated temperature and was not acceptable when exposed to I_2 vapors. The latter results were similar to those reported by Kinman for 304 stainless steel.(21)

Ninety- and 180-Day Membrane Exposure Tests

An experiment was completed to investigate the long-term effects of I_2 and water upon the prototype anion exchange membrane. Since the intended usage for the IGDS is long duration missions, it was of interest to determine if membrane degradation occurred on prolonged exposure to the aqueous I_2 slurry.

Procedure - Membranes 7.9 x 10.8 and 0.04 cm (3-1/8 x 4-1/4 and 0.017 in) thick were conditioned in the normal manner and placed in a sealed container with approximately 100 gm (0.22 lb) of I_2. Sufficient water was included to insure the membranes were completely immersed. The container was sealed and placed in storage at room temperature.

At the end of 90 and 180 days, the membranes were removed and examined for defects. No evidence of membrane deterioration was detected. After both examinations, the membranes remained flexible with no cracks or evidence of ion exchange material flaking off the matrix support grid. All membranes were returned to the exposure container except one which was assembled in an experimental cell. This membrane successfully passed the 21 kN/m^2 (3 psi) pressure differential test. The membrane's electrochemical performance was then tested. In all cases it gave results similar to that observed on membranes not exposed.

TABLE 5 RESULTS OF SATURATED IODINE SOAK TEST (ROOM TEMPERATURE)

Material	Form	Initial	Final	Changes		Comments/Observations	Recommendations
		Weight, Gm		Weight, Gm	%		
Nonmetallics							
Polypropylene	Screen	0.0596	0.0598	+0.0002	+ 0.33	Clear plastic sample became a brown shade and continusoulsy removed I_2 from solution	Acceptable structurally but consumes I_2
Polyvinylchloride	Sheet	5.0118	5.0234	+0.0116	+ 0.2	Sample discolored and lost 1% of initial length	Acceptable
Cast Acrylic (Plexiglas)	Sheet	2.0864	2.0795	-0.0069	- 0.3	Sample became a uniform brown color although initial consumption of I_2 stopped	Acceptable
Vinyl	Tubing	1.2860	1.3683	+0.0823	+ 6.4	Sample became a deep brown and opaque and continuously removed I_2 from solution	Structurally acceptable
Teflon	Sheet	1.0987	1.0986	-0.0001	$\sim$0	Sample became pink shade	Completely acceptable
	Tape	0.0570	0.0576	+0.006	+10.5	No I_2 adsorbed, tape retained adhesive bond	Acceptable (at lower temperatures/concentrations)
	Tubing	0.9616	0.9695	+0.0079	+ 0.82	Slight coloration observed	Acceptable
Viton	Sheet	0.4661	0.6780$^{(a)}$	+0.2119	+45.4	Sample continuously consumes I_2 and a slight surface roughness developed	Unacceptable because of consumption
Fluorel	Sheet	0.3213	0.5387$^{(a)}$	+0.2174	+ 6.77	Sample continuously consumes I_2 and surface roughness developed	Unacceptable because of consumption
Ethylene Propylene	Sheet	0.3727	0.6180$^{(a)}$	+0.2453	+65.8	Sample continuously consumes I_2 and surface roughness developed	Unacceptable because of consumption
Polypropylene (Glass Filled)	Sheet	-	-	-	-	Sample became brown shade but remained smooth	Acceptable
Metallics							
Aluminum 6061	Sheet	5.0615	5.0367	-0.0248	- 0.4	Sample pitted and light brown corrosion product observed	Unacceptable
Stainless Steel 316	Sheet	2.4628	2.4624	-0.0004	$\sim$0	No change	Acceptable
Gold Plated Nickel	Screen	0.1778	0.1677	-0.0101	- 5.7	Gold plate deteriorated and sample became brittle	Unacceptable
Platinum	Screen	0.0471	0.0470	-0.0001	- 0.2	No change	Acceptable

---

(a) Sample weight after five days storage in the atmosphere while continuous volatilization of I_2 observed.

TABLE 6 RESULTS OF ACCELERATED IODINE SOAK TEST, 338K (149F)

Material	Form	Comments	Recommendations
Nonmetallics			
Polypropylene	Screen	Sample became brown but remained smooth, transparent, and flexible	Acceptable
Polyvinylchloride	Sheet	Sample discolored and was slightly distorted	Acceptable
Teflon	Sheet	Sample was unaffected	Acceptable
	Tape	Teflon was unaffected but adhesiveness was lost	Acceptable as seal material
Viton	Sheet	Sample lost flexibility	Unacceptable
Fluorel	Sheet	Sample lost flexibility	Unacceptable
Ethylene Propylene	Sheet	Sample lost flexibility	Unacceptable
Metallics			
Aluminum 6061	Sheet	Sample became pitted	Unacceptable
Stainless Steel 316	Sheet	Sample became slightly pitted(a)	Unacceptable
Gold Plated Nickel	Screen	Gold plate deteriorated and nickel disintegrated	Unacceptable
Platinum	Screen	Sample was unaffected	Acceptable

(a) A second sample held over the I_2 saturated solution; i.e., exposed to humidified I_2 vapor, showed surface reaction within three days.

Membrane exposure to an aqueous I_2 slurry does not affect the physical, ion exchange, or electrochemical properties of the membrane. No problems are anticipated due to membrane degradation. The test membranes have been returned to the aqueous I_2 slurry environment to generate further data on long-term exposure.

Material Compatibility with Flammability and Outgassing Specification

An evaluation was made of the nonmetallic and metallic materials' acceptability to the NASA flammability and outgassing specification for manned flight vehicles.(17) The subsystem drawings were used as control documents. Efforts were made to maximize the number of acceptable materials. However, in the event an IGDS required material that would not have been acceptable or if data on its flammability and outgassing did not exist, a specimen of the material would have been sent to JSC for evaluation. The original material, however, was to be used unless an acceptable alternate material was known and readily available.

The following IGDS materials were found to meet the requirements of the specification: Teflon, Viton A, anion exchange membrane, and 316 and 316L stainless steel. The anion exchange membrane is acceptable by virtue of its projected in-flight use configuration, where it will be immersed in an aqueous solution and retained in a vented container.

The IGDS cell housing was fabricated of transparent Plexiglass, because of a desire to visually observe cell operation. A flight system will have the housing constructed of 316 stainless steel, since Plexiglass does not meet the specification. If the system is to be exposed to elevated temperatures (e.g., 338K (149F), the steel shall be Teflon-coated to avoid the slight pitting observed.

Metallic and Nonmetallic Materials in the Potable Water System

An evaluation was made of the metallic and nonmetallic materials that come in contact with iodinated water in the PWSS. The results of this analysis are summarized in Table 7. Each component in the system(a) was evaluated. Table 8 indicates the location of the materials.

TEST RESULTS

The following experiments were completed during the program:

1. laboratory breadboard to establish design data,
2. hardware material compatibility with the environment,
3. supplementary cell characterization,
4. IGDS design verficiation and endurance testing, and

(a) Reflected by Drawing LSI-D-605, Rev. A.

TABLE 7 PWSS MATERIALS CONTACTING IODINATED WATER

Metallic Materials	Nonmetallic Materials
316 Stainless Steel	Teflon
304 Stainless Steel	Viton A
302 Stainless Steel	Kel-F
Platinum	Ceramic
	Lucite
	Ion Exchange Membrane
	Iodine Crystals

TABLE 8 MATERIALS WETTED BY IODINATED WATER IN THE PWSS

Part Identification Number	Part Description	Material(s) Exposed to Water
V-101, -102 and -103	Two-Way Ball Valve	316 Stainless Steel, Teflon
V-104 thru V-112 and V-116	Manual Two-Way Valve	316 Stainless Steel, Teflon
V-113, -114 and -115	Manual Three-Way Valve	316 Stainless Steel, Teflon
QC-101 and -102	Quick Connects	316 and 302 Stainless Steel, Viton A
G-101 and -102	Pressure Gauge	316 Stainless Steel
PR-101 and -102	Backpressure Regulator	304 Stainless Steel, Teflon
P-101 and -102	Gear Pump	316 Stainless Steel, Teflon, Ceramic
F-101 and -102	Flowmeter	316 Stainless Steel, Teflon, Kel-F, Borosilicate Glass
TC-101, -102 and -103	Sheathed Thermocouple	316 Stainless Steel
TK-101	Water Loop Tank	316 Stainless Steel
H-101	Tank Heater	316 Stainless Steel
LSI-D-611	IGDS	316 Stainless Steel, Plexiglas, Platinum, Ion Exchange Membrane, Fiberglass, I_2 Crystals, Viton A, Teflon
- -	Tubing and Fittings	316 Stainless Steel

5. bacterial challenge.

Except for material studies which were discussed previously (page 56), results of these experiments are presented below.

Laboratory Breadboard Experiments

Various tests were carried out to verify the electrochemical concept. These included determining the effect of current on electrolyte I_2 valve voltage, internal cell resistance, etc. Also, a 35-day test was performed using a laboratory breadboard cell and the projected IGDS anion exchange membrane. These experiments formed the basis of the IGDS design.

Primary purposes of the breadboard tests were to determine membrane and material performances. In addition, it was desired to obtain other design data on time effects, limiting current densities that could be drawn, variations in I_2 generation current efficiency, and to evaluate the effect of varying the process water flow rate.

Typical measurements and results of the 35-day test are shown in Figure 28. Presented are the operating current, the cell's terminal voltage, anode-to-reference voltage, calculated current efficiency, and the pH of the anode solution and the saturated I_2 solution in the compartment adjacent to the cathode. The cell was generally operated in a constant current mode. The data indicated that operation from 5 mA to 20 mA was possible. During the first eight days of the test, terminal voltage was relatively unstable, indicating changes that were evidently occurring which influenced cell internal resistance. The terminal voltage later became more dependent upon cell current but gradually declined to about 1 volt at a 3 mA current level. Anode voltage was less sensitive to current changes and did not appear to appreciably decline as the test progressed. It may be inferred from this data that the anode maintained its electrochemical stability throughout the test. Current efficiency, determined by the equation

$$\frac{I_2 \text{ measured} - I_2 \text{ calculated}}{I_2 \text{ calculated}} \; 100 = \text{Current Efficiency} \qquad (20)$$

increased throughout the test and exceeded 100% at one period. This characteristic is attributed to I_2 leaching and/or I_3^- transfer across the membrane. The pH of the iodinated solution declines more rapidly at the higher current levels. It is expected that transfer of OH^- from the cathode compartment instead of the generated I^- is responsible for this fall in pH. The abrupt rise in pH in the cathode compartment on the 21st day resulted from draining and refilling the compartment with fresh I_2 saturated water. The specific changes in pH with time have little quantitative significance since they relate to the quantity of liquid involved which was randomly varied.

Although the 35-day test was mainly completed to establish the lack of membrane deterioration or electrode corrosion, it also was able to identify the buildup of H^+ in the cathode compartment and the I_2 leaching possibility.

FIGURE 28 RESULTS OF THE 35-DAY CHARACTERIZATION TEST

Solutions to the Acid Build-up Problem

Various possible solutions to the build-up of H^+ in the I_2 accumulator exist, including

1. operating at 100% current efficiency by
    a. lowering the current density or
    b. increasing the area of electrode-membrane contact at the anode.
2. Utilizing acid-resistant materials and employing a larger I_2 accumulator volume for greater H^+ tolerance,
3. neutralizing H^+ by adding alkali to the I_2 accumulator, and
4. consuming H^+ by an electrochemical reaction that needs H^+ to operate.

Techniques 1 and 2 are feasible and straightforward. Technique 2 was followed in the IGDS.

Addition of alkaline electrolyte (e.g., KOH) directly to the I_2 reservoir or in a reservoir separated from the I_2 reservoir by an anion exchange membrane, does not work because of the reaction of I_2 with alkaline media.

$$3I_2 + 6KOH = KIO_3 + 5KI + 3H_2O \tag{21}$$

Only the addition of a controller based on monitoring the increase in H^+, together with controlled, but limited addition of alkali, will avoid the acid build-up and reaction (21) above. Technique 3, therefore was not considered practical because of the complexity it adds.

Technique 4 was eliminated because of its complication and the development needed.

The Leaching Problem

Analysis of these preliminary test results indicated that low current densities were desired to maximize current efficiency and minimize the evolution of O_2 caused by the transfer of OH^- instead of I^-. Since this increased the H^+ and I_3^- concentration, the amount of dissolved I_2 increased (tied up as I_3^-). This indicated the membrane would become more susceptible to I_2 leaching on the anode side which, on checking with low water flow rates, was then observed.

Initial tests for membrane leaching were made using very large water flow rates of 500 to 570 kg (1100 to 1260 lb) per minute.(a) These large rates were initially expected to be the maximum recirculation rates that would be needed

---

(a) 350 to 400 ml/min (21.4 to 24.4 in^3/min).

in the end-item application. Initial tests were, therefore, for the assumed "worse case" condition. After the water flow rate specification was finalized (see section entitled "Water Flow Requirements," page 16) at 30 kg (66 lb) per day (21 ml/min), the occurrence of leaching became apparent. Supplementary experiments were then carried out to quantify the leaching and the effect of water flow, temperature and time.

Supplementary Experiments To Quantify Leaching

Between the time the initial valve and breadboard experiments were completed and the IGDS was fabricated, a series of supplementary experiments were made. Their purpose was to quantify experimental findings observed during testing of I_2 valves. The supplementary experiments included

1. measurement of the effect of water flow rate on I_2 leach rate,
2. measurement of the effect of temperature (286, 296 and 349K (55, 73 and 104F, respectively)) on I_2 leach rate (without current flowing), and
3. measurement of the effect of time on I_2 leach rate.

Leach Rate versus Flow Rate

Figure 29 quantifies the effect water flow rate has on the leach rate. The constant leach level at the high water flow rate explains why leaching was not observed during the experimental work carried out prior to freezing the IGDS design. The I_2 level resulting from leaching at the high flow rates used, yielded an I_2 level below the detection limit.

Leach Rate versus Temperature

Experimenting with single cells indicated that the I_2 leach rate varied with water temperature. An experiment, therefore, was undertaken to quantify this effect. Results, shown in Figure 30, indicate the leach rate increases with increasing temperature with a rapid rise occurring at the more elevated temperatures.

Leach Rate versus Time

Figure 31 quantifies the effect time has on the leach rate at a constant water flow rate of 19 ml/min (1.1 in^3/min). Results of two different tests are indicated. Each test was carried out for nine days. The data indicated with circles were obtained on a cell through which no current has ever passed. The data indicated with triangles were obtained on a valve that had been operating under continuous load but was put on to open circuit and, after 30 minutes, the leach rate determined.

The results indicated an average leach rate of approximately 0.08 gm (1.8×10^{-4} lb) I_2 per day at a temperature of 296 ±5K (73 ±9F).

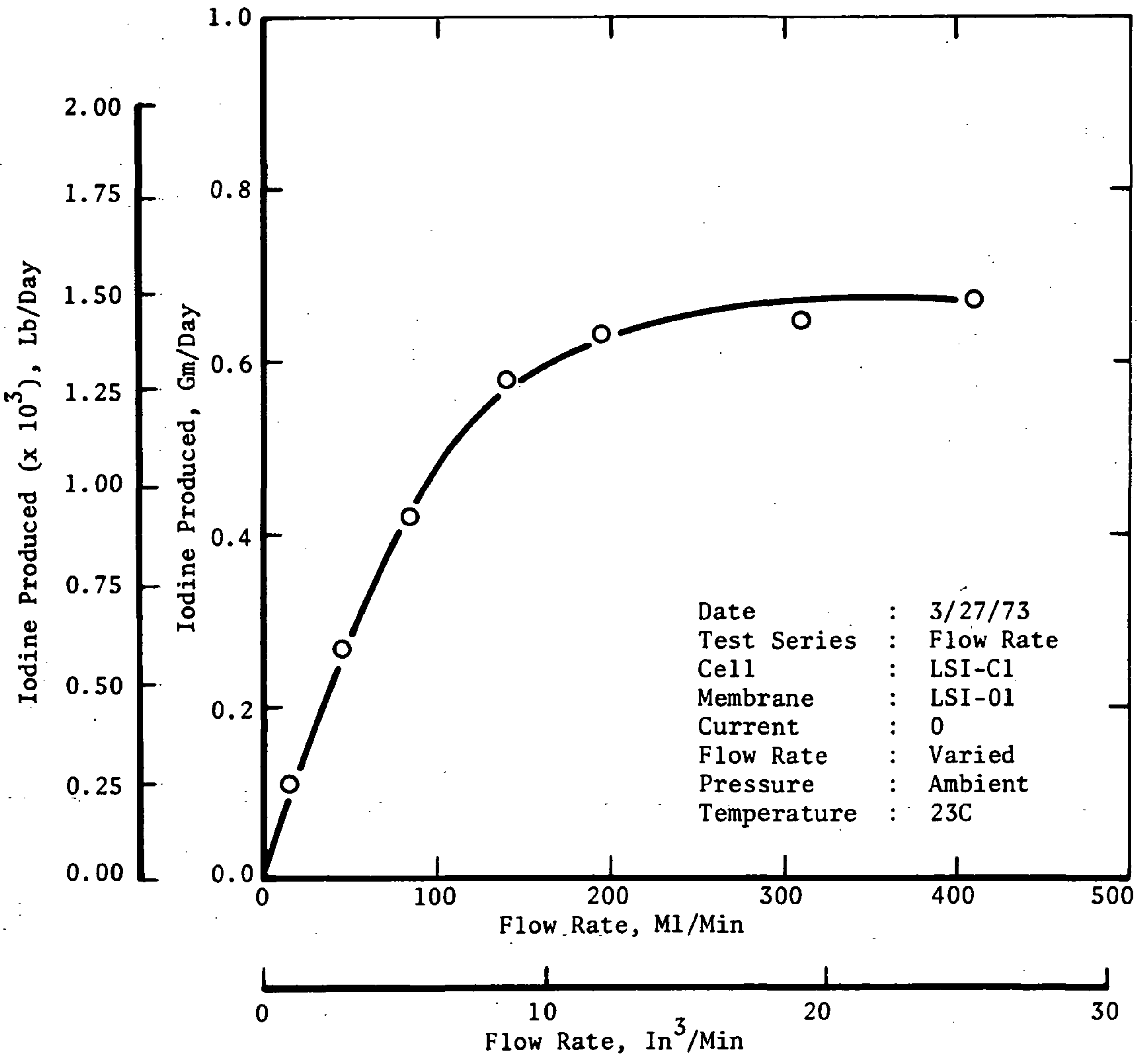


FIGURE 29 LEACH RATE AS A FUNCTION OF WATER FLOW

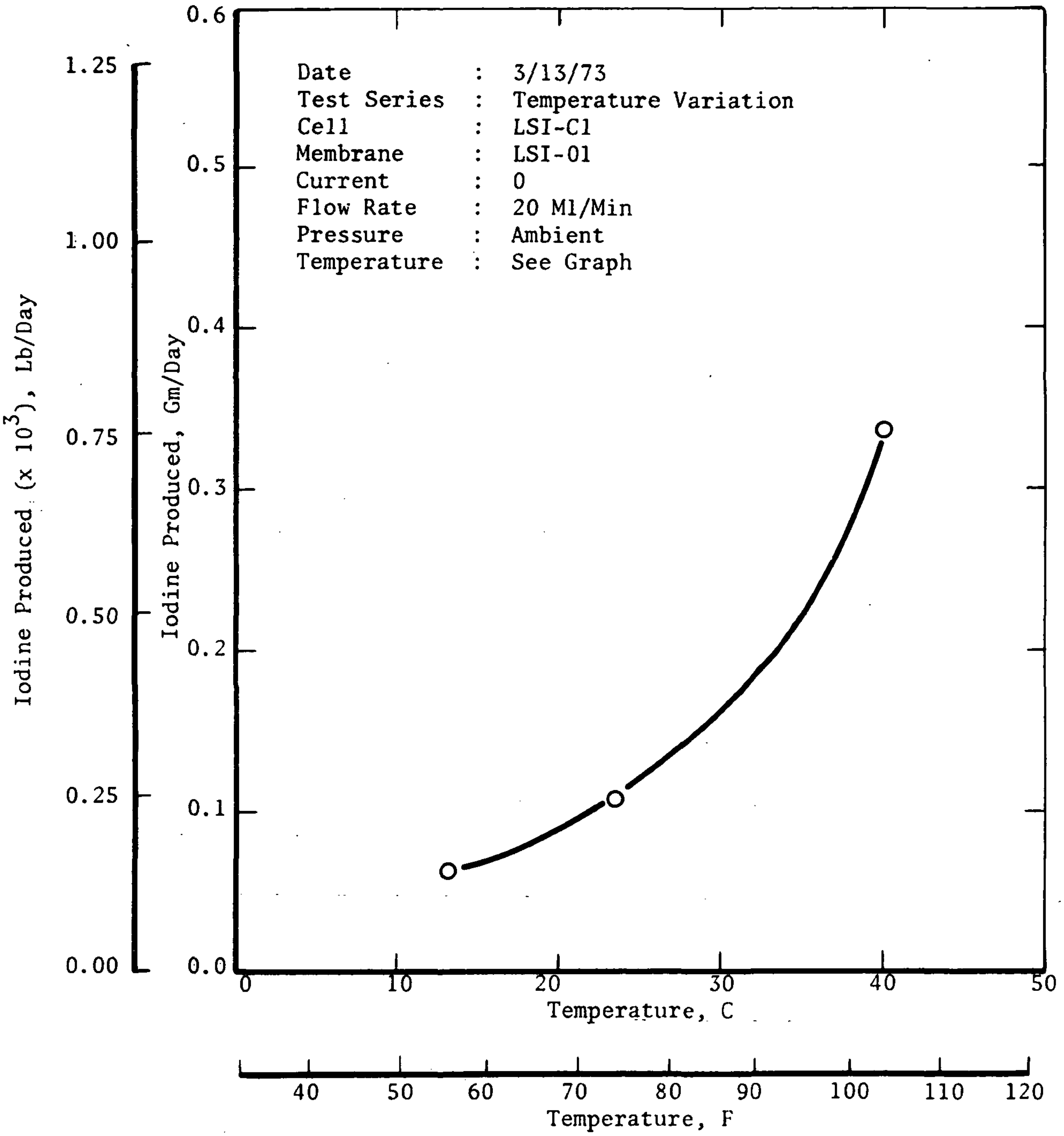


FIGURE 30 LEACH RATE AS A FUNCTION OF TEMPERATURE

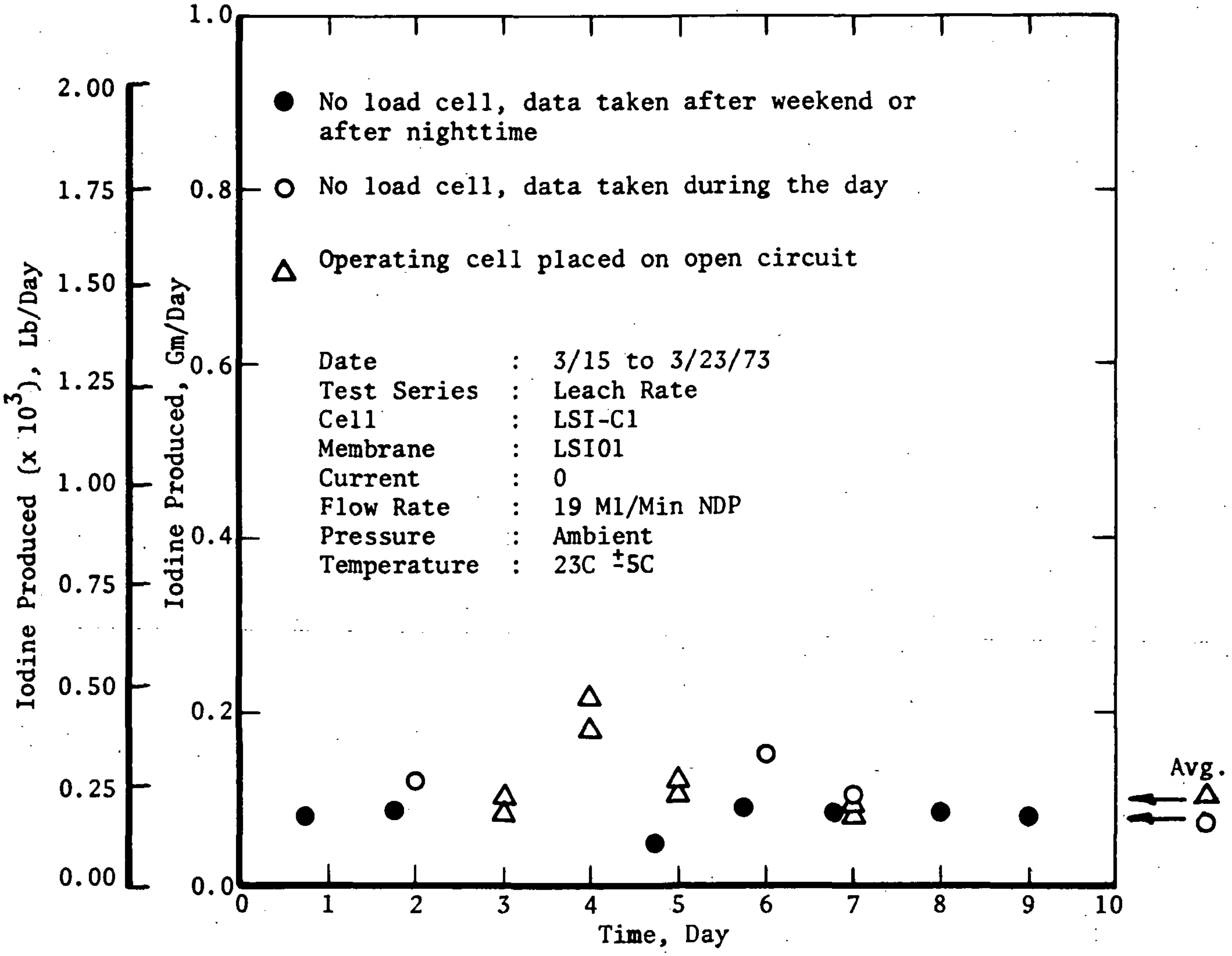


FIGURE 31 LEACH RATE AS A FUNCTION OF TIME

Minimizing Leach Rate

Operation and construction factors can minimize the I_2 leach rate.

The operation factors that minimize leaching are

1. slower water circulation rates that minimize flushing I_2 from the anode membrane surface (decreasing the diffusion driving force),
2. lower water temperature,
3. greater operating current densities (decreasing the concentration of I_2 at the cathode surface and increasing the concentration of I_2 at the anode surface), and
4. minimized concentration of H^+ in the I_2 accumulator (high H^+ concentrations yield high dissolved I_2 as I_3^-).

The construction factors that minimize leaching are

1. smaller membrane/electrode areas,
2. greater separation between the solid I_2 and the membrane, and
3. avoidance of incorporation of I^- during the membrane conditioning process.

While not yet completely eliminated, the observed leaching appears to be manageable and possibly can be eliminated totally.

Material Experiments

The material tests completed were reviewed in the IGDS Materials Program seciton (page 56). This included material stability to saturated I_2 solutions at room temperature and at 338K (149F). The membranes were characterized after 90 and 180 days.

Iodinated Water Taste Experiments

Water samples, varying in I_2 level from 0.5 to 20 ppm, were tasted to determine their palatability. This test indicated that flight systems would not utilize residual I_2 concentrations of 20 ppm. Even a 10 ppm specification seemed unlikely. Taste at these levels is very "antiseptic." While tolerable for a short term, the taste would become objectionable under continued, long-term use. It was concluded, therefore, that the system specification would eventually assume a lower I_2 level, probably 5 ppm.

IGDS Experiments

After the IGDS was fabricated, inspected and assembled, the following series of tests were completed:

1. Shakedown,
2. Calibration,
3. Design Verification Test (DVT), and
4. Endurance.

The unit was disassembled after the DVT and Endurance Tests and no change or refurbishment was needed between these tests.

Shakedown Test

Upon assembly of the IGDS and its integration into the PWSS, various tests were used to shake down the system. These included

1. The system was operated and all valves and connections, including flowmeters and pressure regulators, were checked for leakage.
2. A 103 kN/m^2 (15 psid) pressure differential was applied across the flowmeter controller. (By design a differential of 207 kN/m^2 (30 psid) is needed for control.)
3. The system was pressurized to 207 kN/m^2 (30 psia) and found leak-free.

The system was calibrated after all mechanical, electrochemical and electronic components, including the valve, were found to be working properly.

Calibration Test

The calibration tests performed included

1. calibration of the PWSS water loop recirculation and water feed flowmeters (R-6-15A and R-2-15-A, respectively) see Figures A-14 and A-15, and
2. calibration of the PWSS water supply tank sight tube (see Figure A-8).

Since the electronic meters were calibrated at the manufacturer (±3% full-scale deflection error), no additional calibrations were made.

Design Verification Test

After the shakedown and calibration tests were completed the IGDS was characterized. This included the following five experiments:

1. A current density span was made to determine high and low limits of I_2 production and to compare performance with the breadboard valves (Figure 32). The higher voltage above 1 mA/cm^2 with the

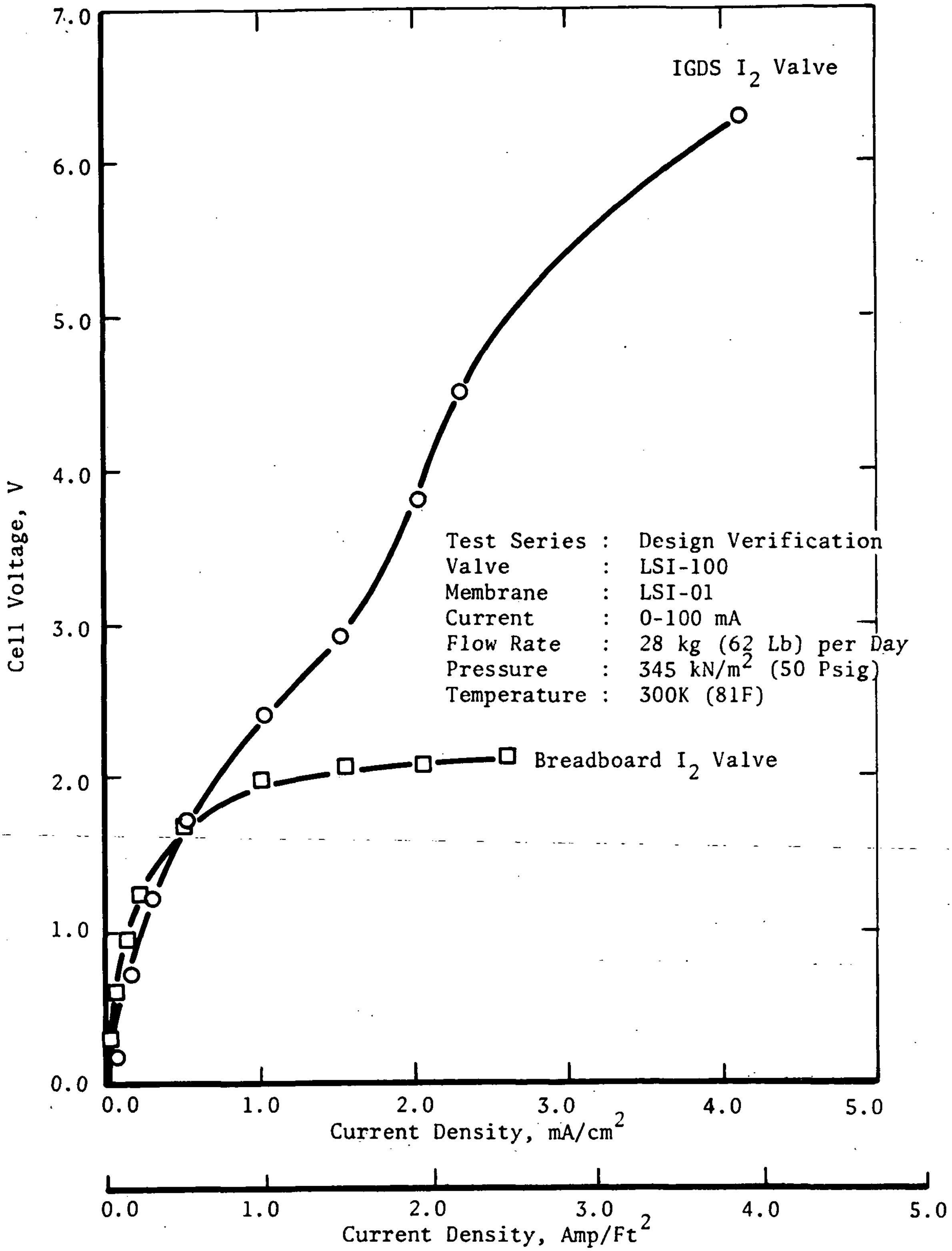


FIGURE 32. CURRENT-VOLTAGE CHARACTERIZATION: IGDS AND BREADBOARD VALVES

IGDS probably resulted from increased cathode polarization because of the greater distance separating the solid I_2 from the cathode when the fiberglass I_2 retaining sheet was used.

2. A leach rate test (Figure 33) versus time (21 days) was made which indicated the phenomena began after six days and leveled out shortly thereafter. The cause of the three scattered data points is unknown.

3. An efficiency versus current density span (Figure 34) was made, indicating a current density of 1.5 to >4.0 mA/cm^2 was applicable although a lower (0current density (0.23 mA/cm^2 (0.24 ASF)) was used as baseline to minimize loss of efficiency by OH^- transfer.

4. Total I_2 and I^- production versus current density runs (Figure 35) were made to characterize IGDS capacity.

5. Cell voltage plus anode and cathode versus reference voltage tests as a function of current density (Figure 36) was made for IGDS power level characterization. The reason for the abrupt rise in the cathode-to-reference voltage is not known.

The water sample to be analyzed was taken at the dump valve, downstream of the flowmeter for all the parametric DVT tests presented. If the potable water side of the IGDS was depressurized (sample taken), water saturated with I_2 passed across the membrane as the water pressure in the I_2 accumulator equilibrated with the (now lower pressure) potable water side of the IGDS.

Endurance Test

Following completion of the Shakedown, Calibration and Design Verification Tests, the I_2 accumulator, valve and dispenser assembly was disassembled, checked for integrity and reassembled when all components were found normal. A 30-day endurance test at nominal conditions was then completed. Performance data obtained from this test are presented in Figures 37 to 44.

Operating Conditions

During the test the parameters cited in Table 9 were held essentially constant, although some variation occurred during the test as noted.

In Figure 37, citing the water flow rate variation, the difference between the solid data points and the open circles is that the latter were calculated based upon total water accumulated (generally over seven hours or a weekend) divided by the time passed. The solid circles were flow measurements generally taken over a 5 to 15 minute period. The flow was maintained near 19.5 ml/min (28 kg (62 lb) per day).

In Figure 38, citing water loop pressure versus test time, the operating pressure is shown to be approximately constant at the designed 210 kN/m^2 (30 psi), once a check at 238 kN/m^2 (34 psi) was demonstrated.

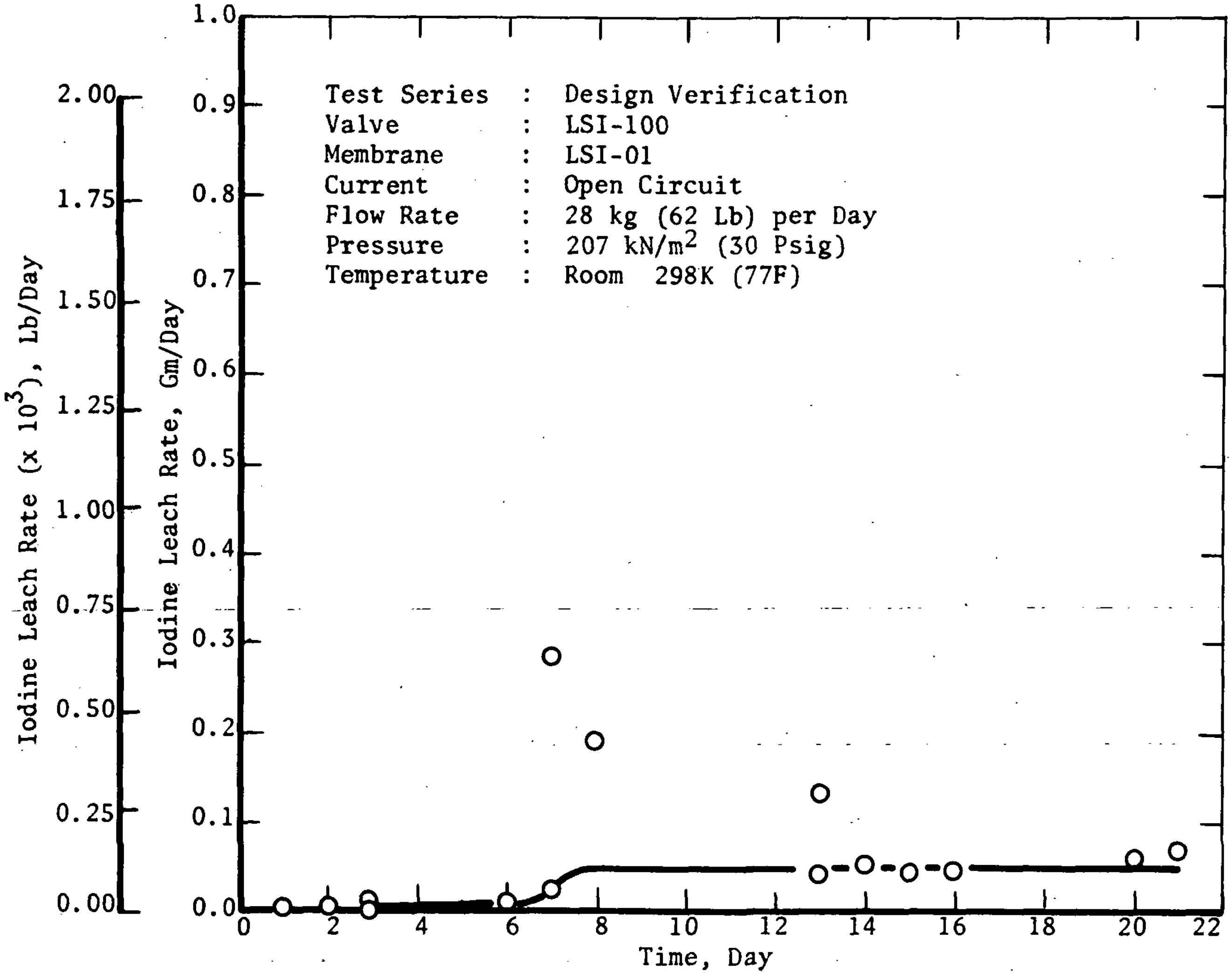


FIGURE 33 LEACH RATE OF IGDS AS A FUNCTION OF TIME

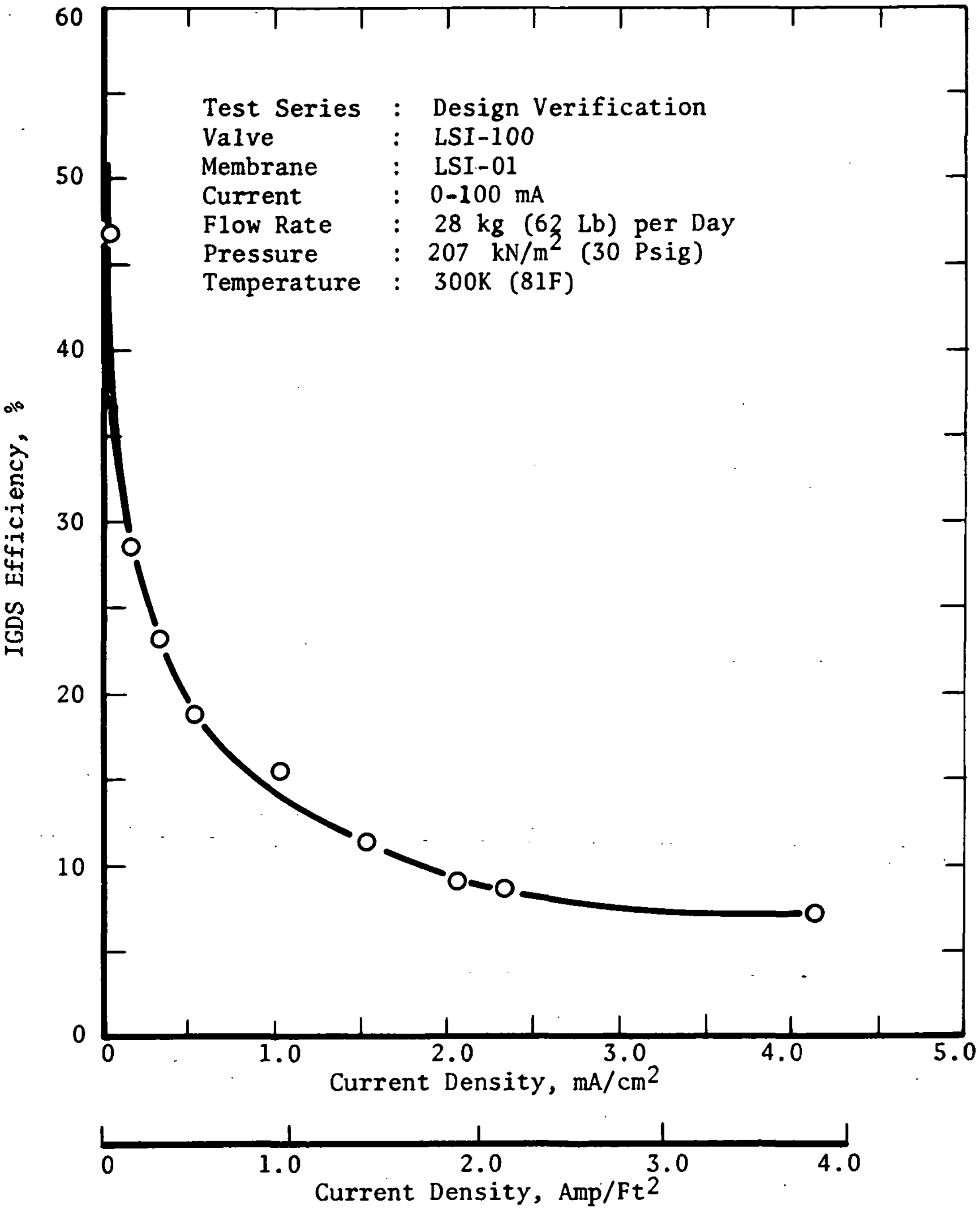


FIGURE 34 EFFECT OF CURRENT DENSITY ON EFFICIENCY

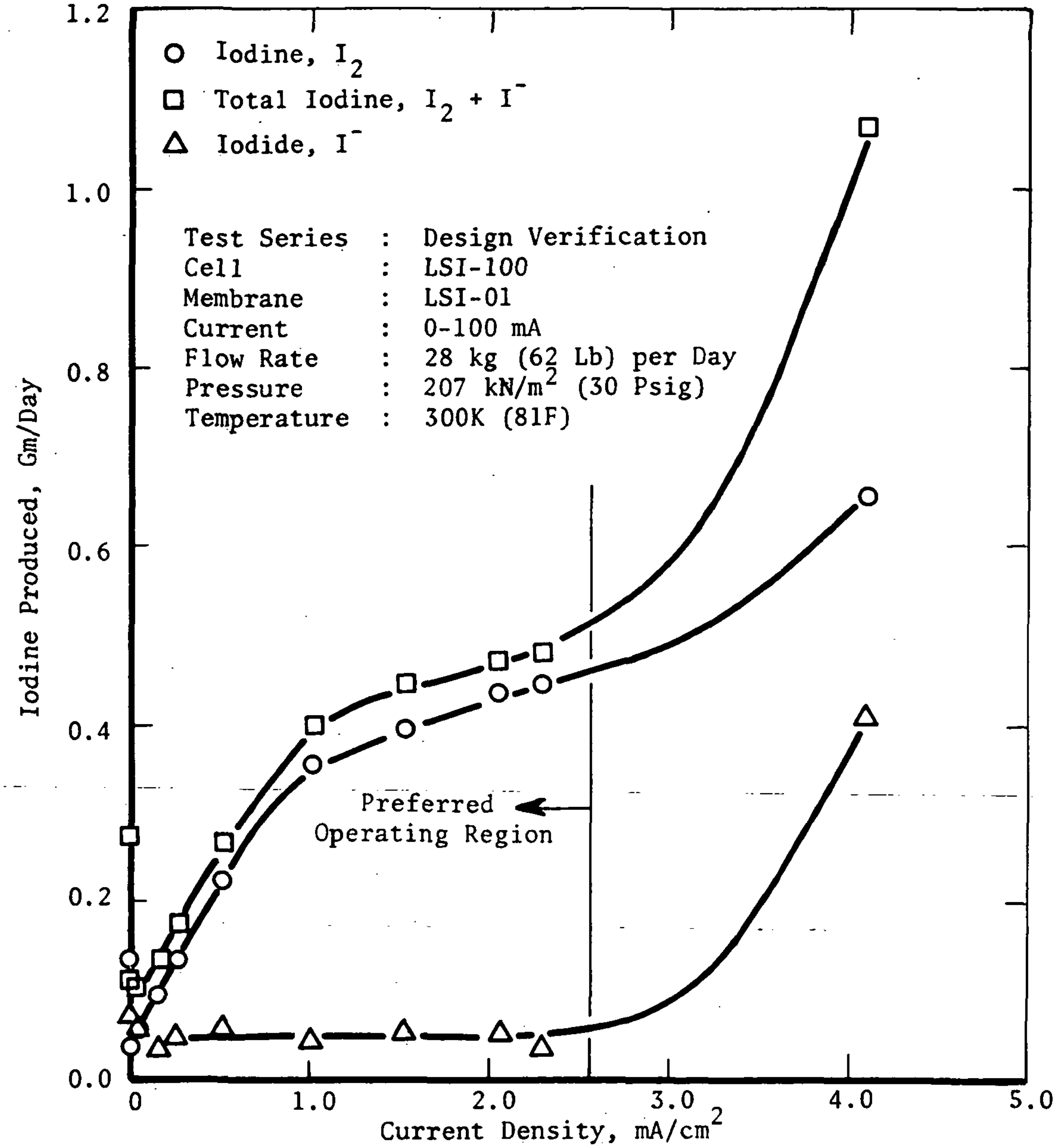
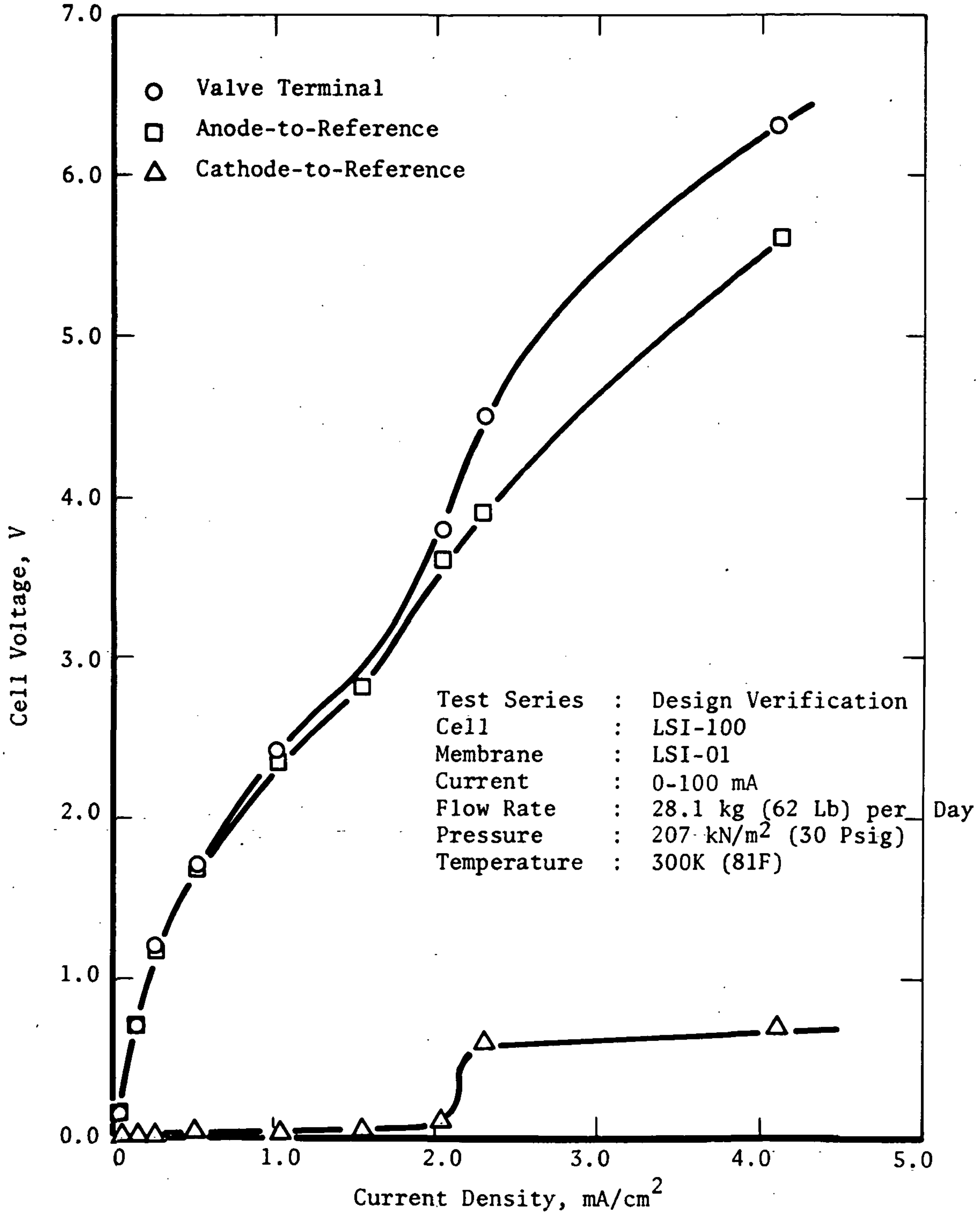


FIGURE 35 TOTAL IODINE GENERATED VERSUS CURRENT DENSITY



Test Series : Design Verification
Cell : LSI-100
Membrane : LSI-01
Current : 0-100 mA
Flow Rate : 28.1 kg (62 Lb) per Day
Pressure : 207 kN/m^2 (30 Psig)
Temperature : 300K (81F)

FIGURE 36 CELL VOLTAGE VERSUS CURRENT DENSITY

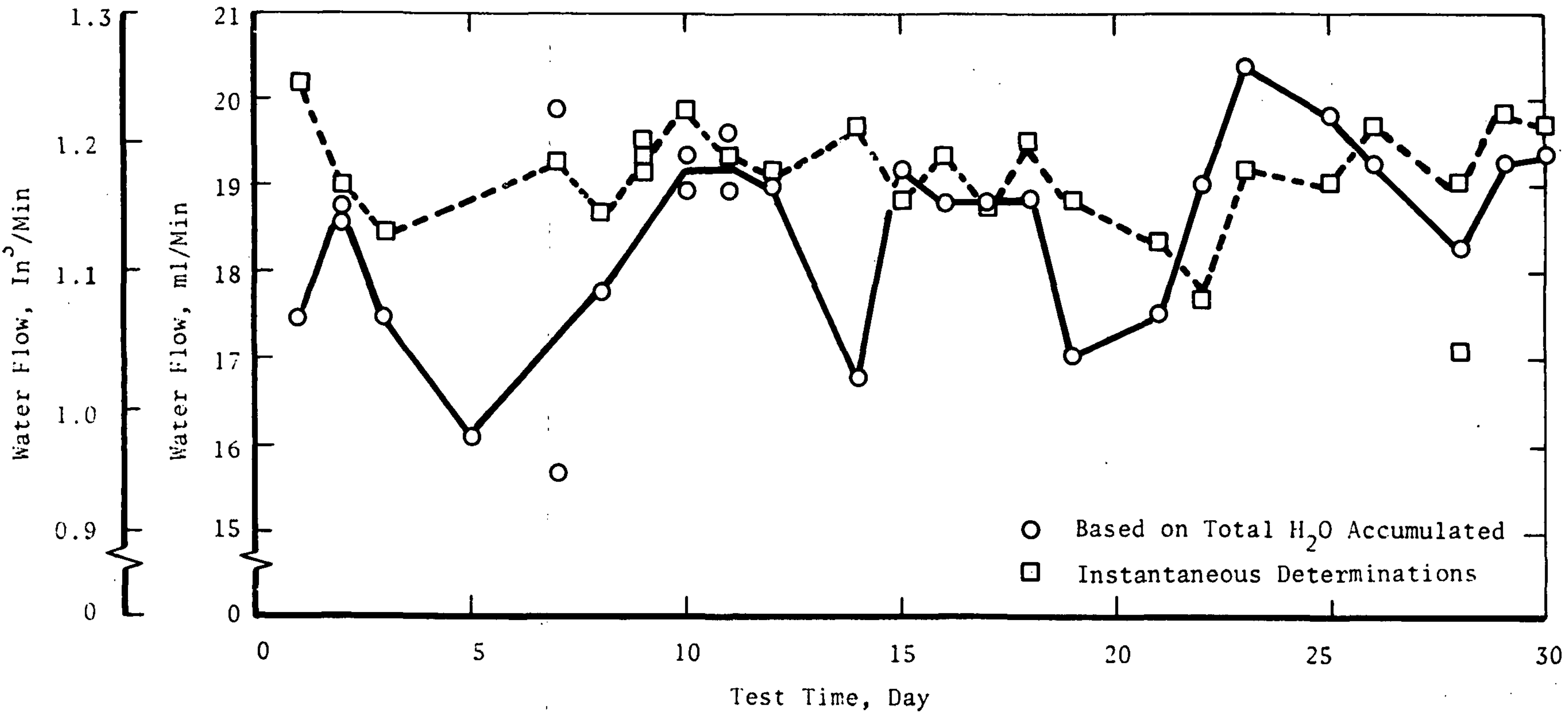


FIGURE 37 WATER FLOW RATE VARIATIONS

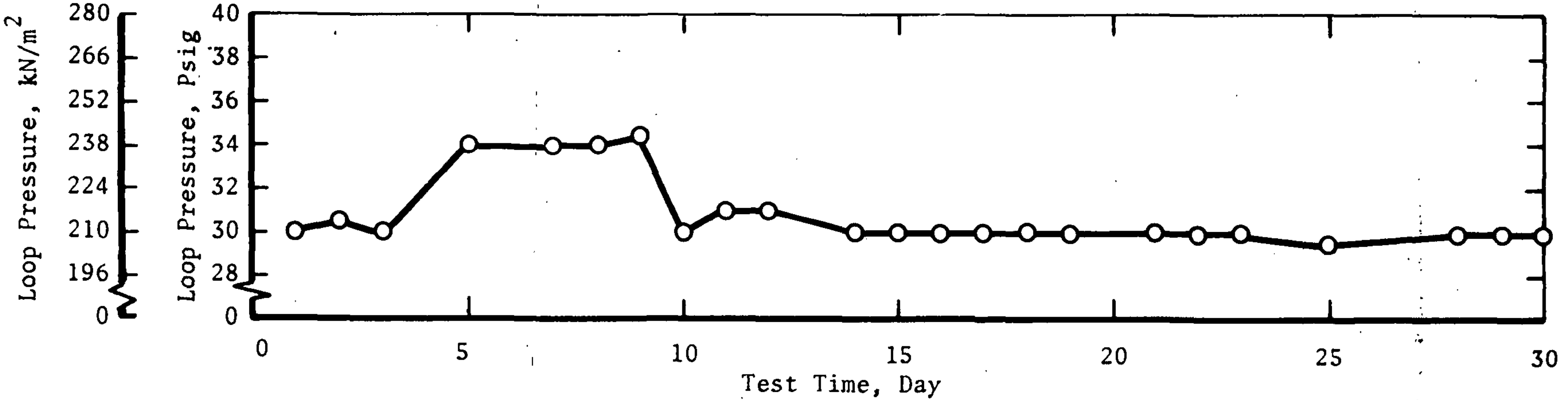


FIGURE 38 PWSS LOOP WATER PRESSURE VARIATIONS

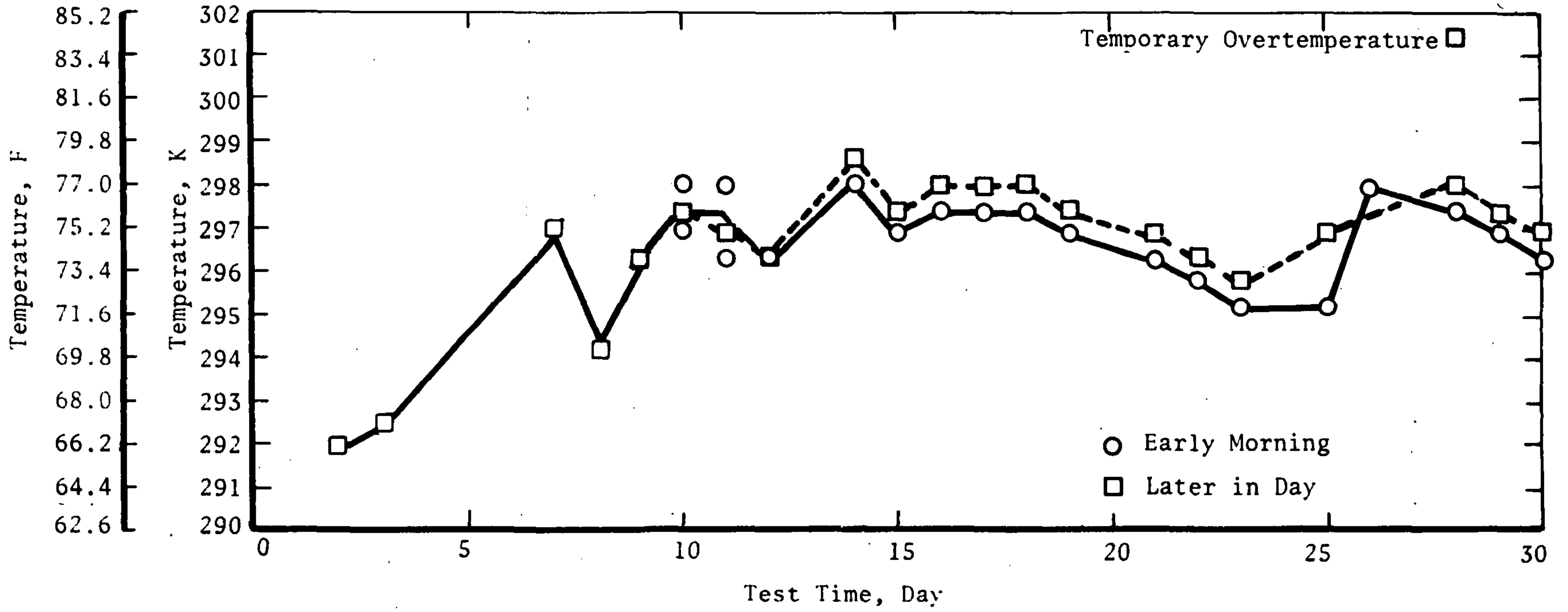


FIGURE 39 WATER TEMPERATURE VARIATIONS

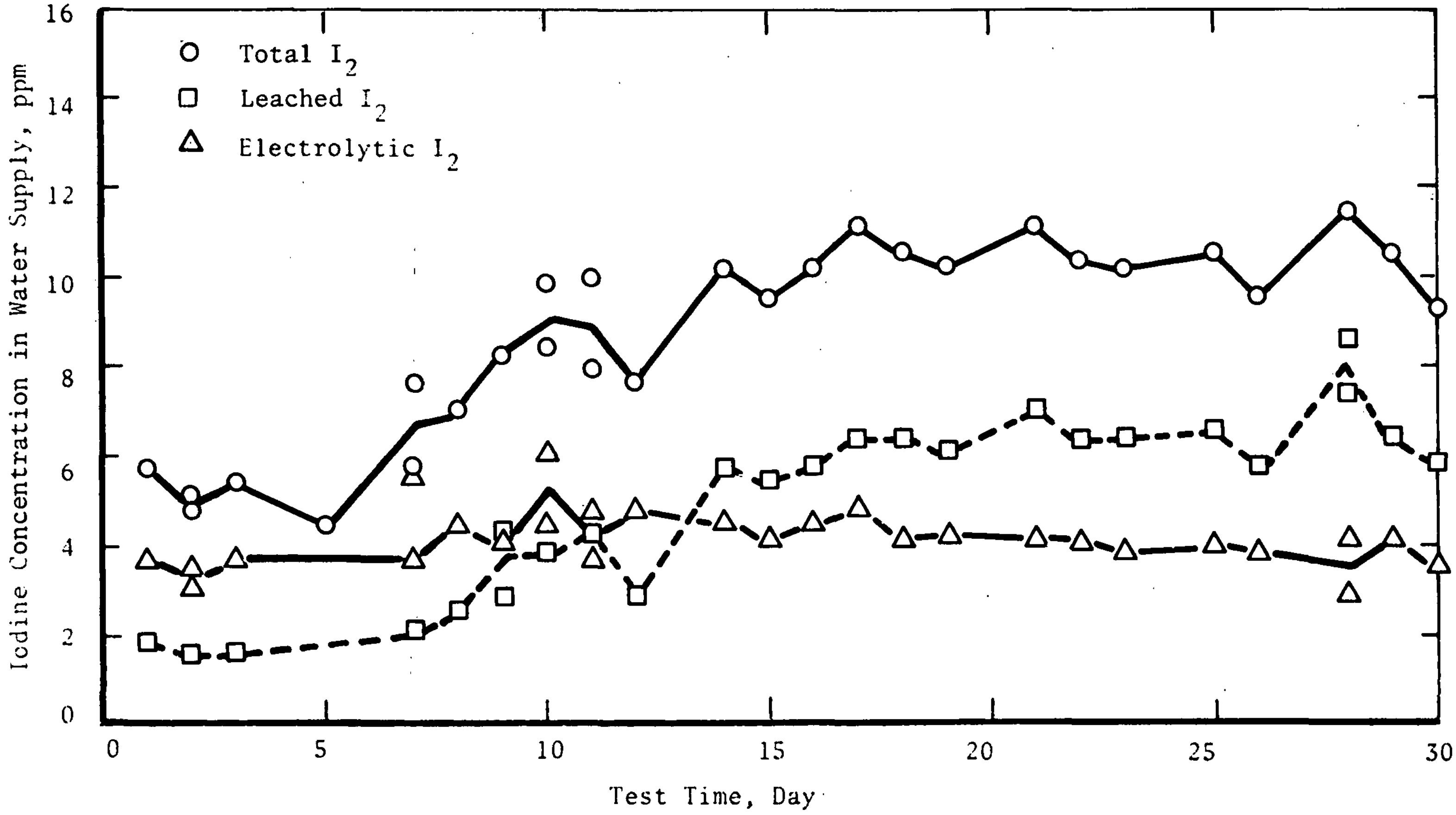


FIGURE 40 IODINE PRODUCTION VARIATIONS

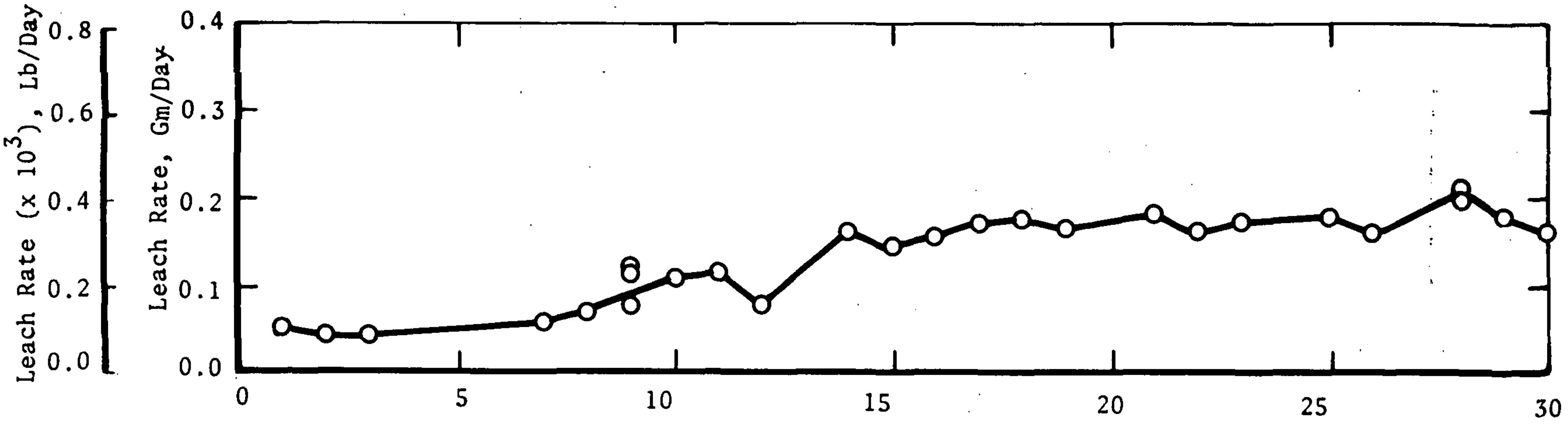


FIGURE 41 LEACH RATE VARIATIONS

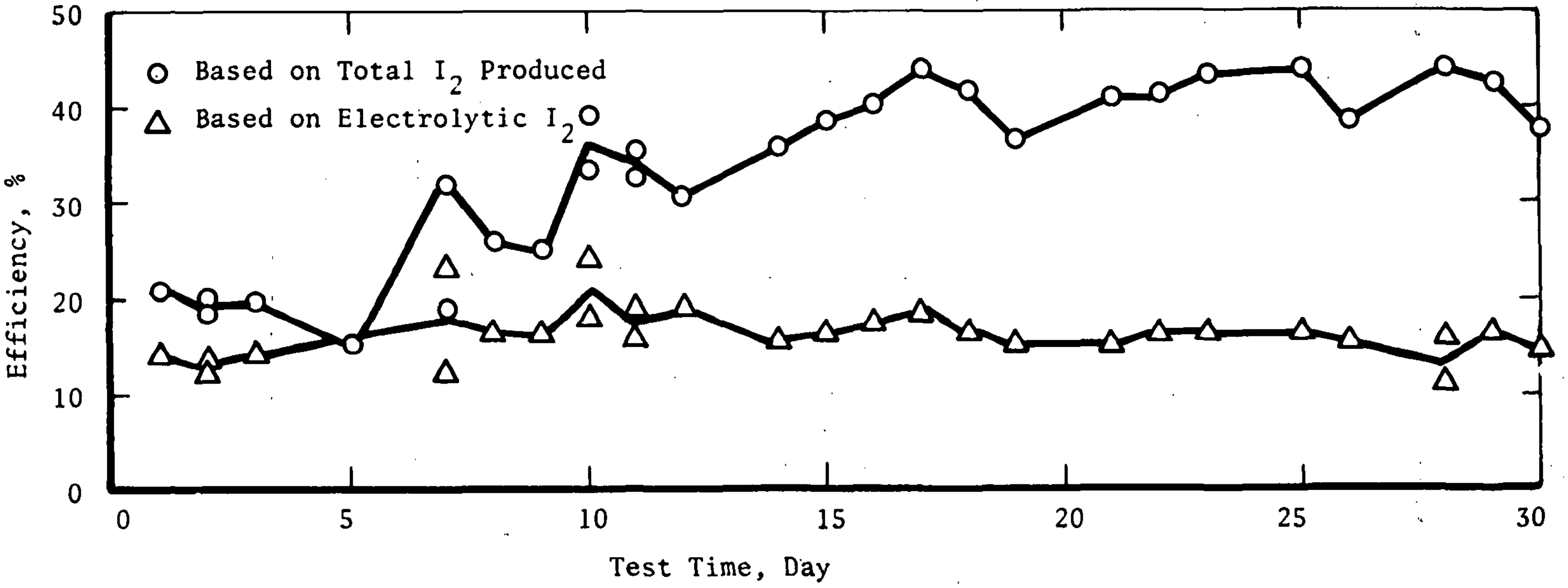


FIGURE 42 EFFICIENCY VARIATIONS

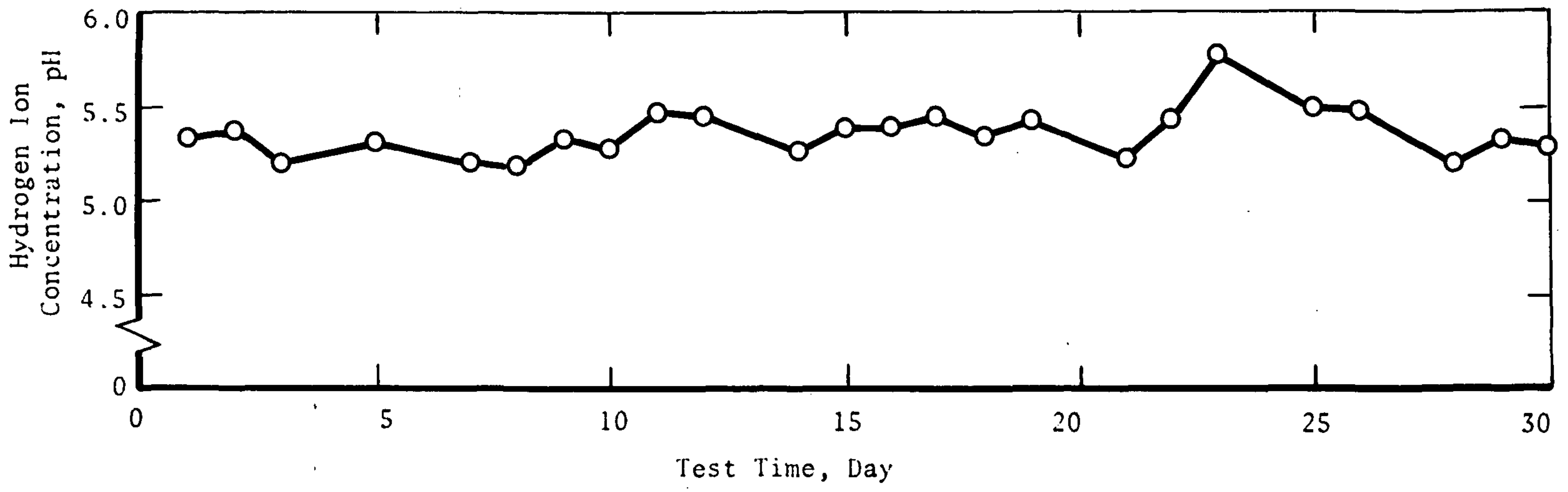


FIGURE 43 pH VARIATIONS IN IODINATED WATER

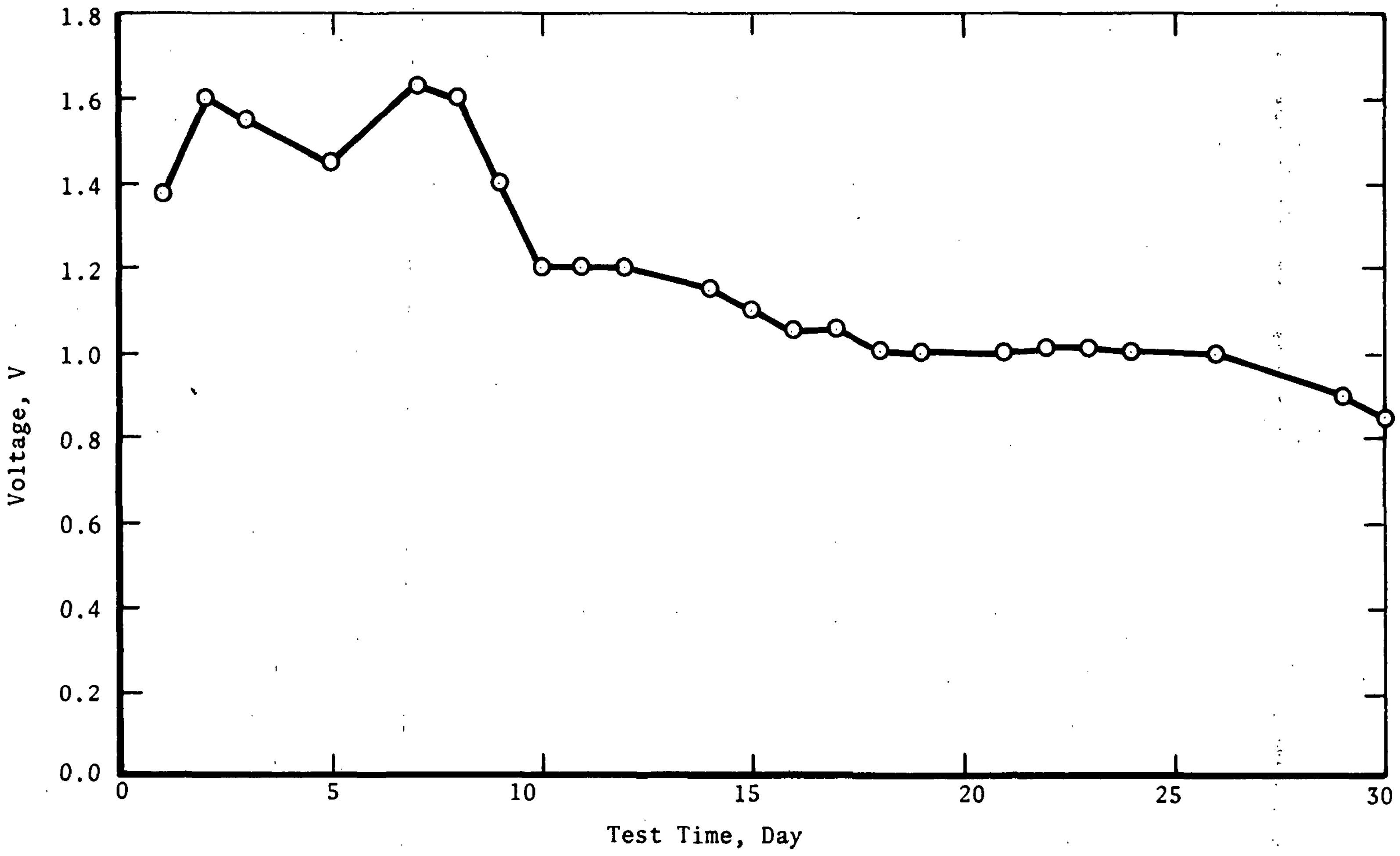


FIGURE 44 VALVE VOLTAGE VARIATIONS

TABLE 9 ENDURANCE TEST PARAMETERS

Parameter	Nominal Value	Variation Over 30-Day Test	See Also
Water Flow Rate,			
ml/Min (In^3/Min)	19 (1.2)	20.5-11.5 (1.2-0.7)	Figure 36
kg/Day (Lb/Day)	27 (60)	29.4-5.2 (65-36)	
Water Feed Pressure, kN/m^2 (Psig)	315 (45)	Constant	Not Plotted
Water Pressure at the Valve, kN/m^2 (Psig)	210 (30)	203-277 (29-39.5)	Figure 37
Valve Current, mA	6[a]	Constant	Not Plotted
Temperature, K (F)	Room	292-300 (66-80)	Figure 38

---

[a] Except when a leach rate was measured with no current flowing (valve on open circuit).

In Figure 39, citing the temperature variation, the difference between the solid data points and the open circles is that the latter was measured first thing in the morning and the latter after the system had equilibrated to room temperature later in the day. Prior to the ninth day, only the one reading was taken later in the day. No data point was taken after the system had equilibrated on the 26th day. Thus, the crossover noted does not have quantitative significance.

Iodine Dispensed - The I_2 dispensed was a result of that generated by the valve and that leached by the water. As indicated in Figure 40, the I_2 produced varied from 5 ppm initially to 11 ppm, with the I_2 generated electrochemically contributing about a constant 4 ppm and the remainder resulting from leaching. (Methods to minimize leaching were discussed on page 70.)

Leach Rate - The DVT (Figure 33) indicated an I_2 leach rate of 0.05 gm/day (1.8 ppm at 28 kg (62 lb) water flow per day). After disassembly and reassembly the leach rate increased to an initial 0.15 gm/day, 0.2 gm/day after ten days, and leveled off at 0.3 gm/day after 17 days as shown in Figure 41. It is uncertain if the increase was caused by the reassembly or by the acid build-up and plexiglass attack cited in the "Post-Test Inspection," below. Insufficient controls were used to maintain such stable operation that the variations cited could be quantitatively explained.

Iodine Generation Efficiency - The efficiency of the electrolytic portion of the I_2 dispersion remained relatively constant at 15% (see Figure 42) and tracked the I_2 produced electrolytically (see Figure 40). When the leached I_2 was included, the efficiency increased from 20% initially to 40 to 45% from the fifteenth day on. Insufficient controls were used to maintain such stable operation that the variations could be quantitatively explained.

Iodinated Water pH - The concentration of the H^+ (pH) of the iodinated water remained relatively constant within the band 5.2 to 5.5 pH units (see Figure 43).

Valve Voltage - The valve's voltage started initially at about 1.6 volts and ended at about one-half this level, at a constant current of 6 mA (0.24 mA/cm^2 (0.23 amp/ft^2)) as shown in Figure 44. Cathode- and anode-to-reference voltage were measured but not plotted. The cathode-to-reference remained essentially constant at 0.01 to 0.025 volt. The anode-to-reference tracked the terminal voltage almost identically except for the cathode-to-reference contribution.

Post-Test Inspection

After the 30-day endurance test, the IGDS was removed from the test stand and the cathode fluid drained into a container. Its pH was measured and found to be 1.05.

The anode cavity was flushed with distilled water and the cover from the cathode cavity was removed. It was observed that no solid I_2 was left, although the solution was very dark, indicating adequate availability of I_2. The bottom of the cell had approximately a 0.16 to 0.32 cm (1/16 to 1/8 in) thick layer of sludge. The stainless steel screen and screen supports were in excellent condition, being only slightly discolored by the sludge near the bottom.

The cathode compartment lid was stained. It was basically black near the bottom of the cell, changing to a light brown near the top. The Helicoil insert located in the bottom part of the cathode compartment lid, was partially corroded away. The Viton A O-ring used to seal the cathode compartment lid appeared to have a slight set, but showed no signs of deterioration.

The bottom of the Plexiglass cathode cavity appeared to be attacked by the acidified catholyte. A sludgy deposit was formed, consisting partially of the Plexiglass itself. Discoloration of the cathode compartment again was blackish at the bottom of the cell and dark cinnamon or amber at the top.

The platinum reference electrode was clean and the Teflon covering was slightly tanned near the part that passed through the cathode compartment wall.

The Plexiglass plug, inserted with methylene chloride at the bottom of the cell during fabrication, appeared to be swollen into the cavity about 0.16 cm (1/16 in). This plug had been inserted to secure the lead from the reference electrode. Plug material that had swollen into the cathode compartment was soft and readily removable.

The cathode electrode looked very clean, as did the metallic stainless steel part used to secure the electrode to the side of the compartment.

The fiberglass separator was discolored, but structurally intact. In separating the valve's electrode-membrane-electrode sandwich, the membrane remained attached to the anode and had two areas where a brownish, sludge-like deposit was attached to it. This was opposite the two bottom slots of the cell and appears to have been a transfer of the material noted above. The membrane appeared slightly wrinkled, with the greatest wrinkling, about a 0.08 to 0.10 cm (0.03 to 0.04 in) depression, toward the bottom part of the cell, but showed no other visible degradation. Sludge-like deposits were easily removed from the membrane.

All parts were cleaned with sodium thiosulfate, rinsed with distilled water, and stored.

Sludge formation and Plexiglass staining can be accounted for by the H^+ build-up. As the acid concentration increased, it attacked the Plexiglas allowing I_2 staining. As the H^+ concentration increased, the amount of I_2 dissolved in solution increased via

$$I_2 + I^- = I_3^- \tag{22}$$

with the I^- being formed to balance the H^+ buildup. The H^+ concentration increased when OH^- were removed from the catholyte as part of the current carrying process due to the equilibrium

$$H_2O = H^+ + OH^- \qquad (23)$$

The variation in color (black at bottom to amber at top) resulted from the gradual dissolution of solid I_2 and its diffusion upward with an increase in I_2 concentration and staining tendency following this diffusion process.

Bacterial Challenge Experiments

Three experiments were completed in which one or more of the E. Coliform Group of bacterial organisms were incorporated into the potable water and subsequently challenged by I_2. One experiment was conducted during the development leading up to prototype system design. The other two were conducted at the beginning and completion of the IGDSs 30-day endurance test. The results are summarized in Table 10 and show that E. coli cannot survive in the iodinated water with I_2 concentrations of 4.8 ppm or greater

OTHER PROGRAM ACTIVITIES

In addition to the activities discussed in the preceding sections, various other experimental and analytical evaluations were made. They are reviewed below.

Mechanical Iodine Injection

At the beginning of the program it was not known whether an electrochemical I_2 feed system could be developed within the program scope. As a backup, therefore, a mechanical I_2 injection system was designed and its control system fabricated and tested. This activity lead to the following conclusions:

1. The mechanical injection approach is feasible.
2. The variable voltage approach to pump control is applicable.
3. The technology needed is available to fabricate such an approach to I_2 injection.

Mechanical Approach Design

Two different designs were considered applicable. Schematics of these are shown in Figures 45 and 46. The former is the preferred concept and employs periodic pumping of water through a saturated I_2 solution until the I_2 level meets specification. Figure 46 shows a mechanical concept based upon using micro-pistons to cyclically inject saturated I_2 solution into the water. Figure 47 illustrates the flow of saturated I_2 solution needed to raise the I_2 level in water flowing at various rates.

The lower the desired I_2 level and the slower the water flow rate, the more difficult it is for a mechanical system to establish a consistent I_2 level because of the difficulty in reliabily pumping a fluid at low flow rates (e.g., 5 ml/min (0.3 in^3/min)).

TABLE 10 RESULTS OF THE BACTERIAL CHALLENGE EXPERIMENTS

Experiment Number	Iodine Valve	Initial E. Coli. Level (a)	Iodine Level, Ppm	Final E. Coli. Level (a)
1	Exp. Cell	2.0×10^5	5.4	0
2	IGDS	3.8×10^5	10.2	0
3	IGDS	1.8×10^5	4.8	0

(a) In organisms per 100 ml.

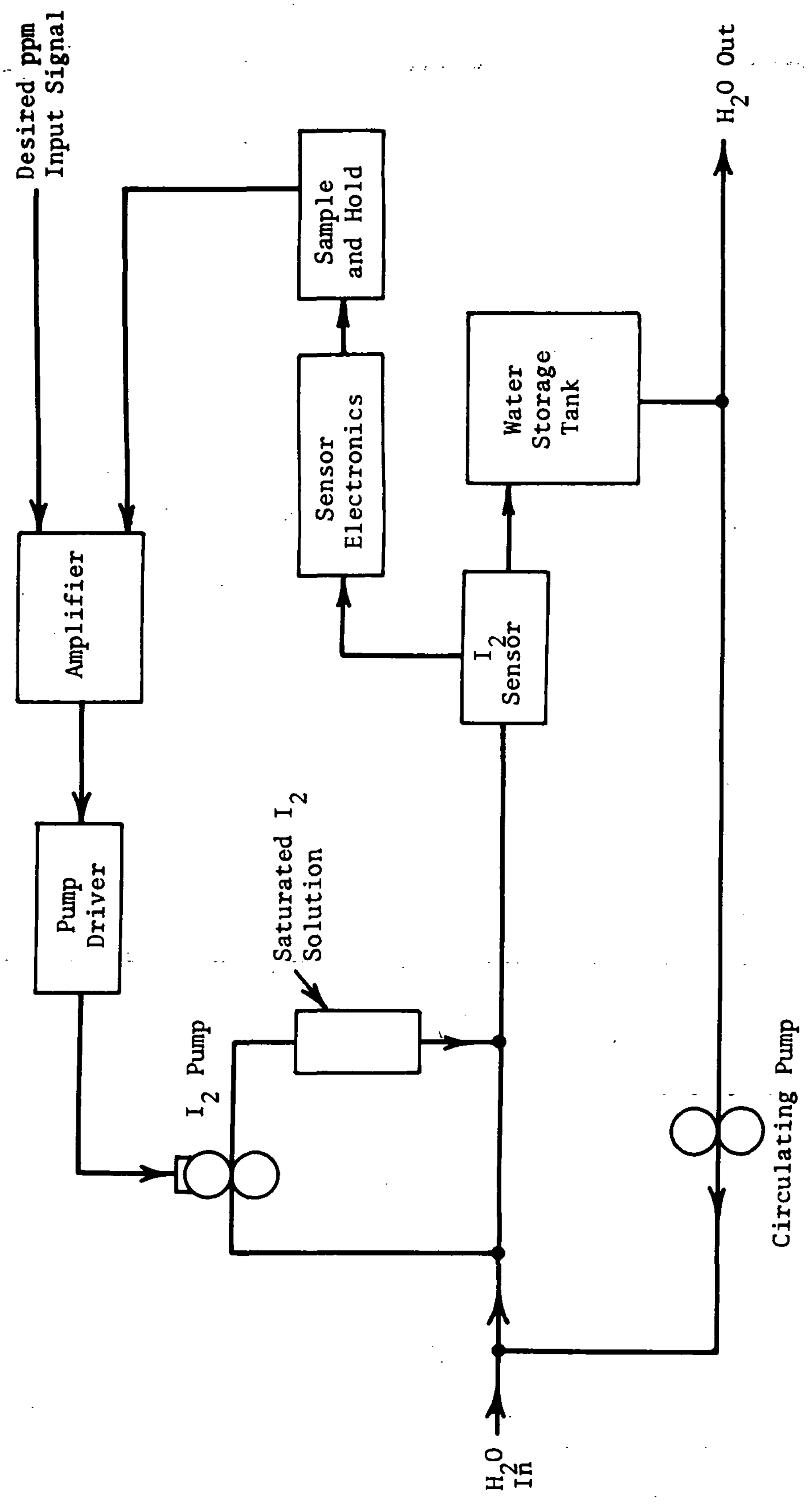


FIGURE 45 MECHANICAL IGDS

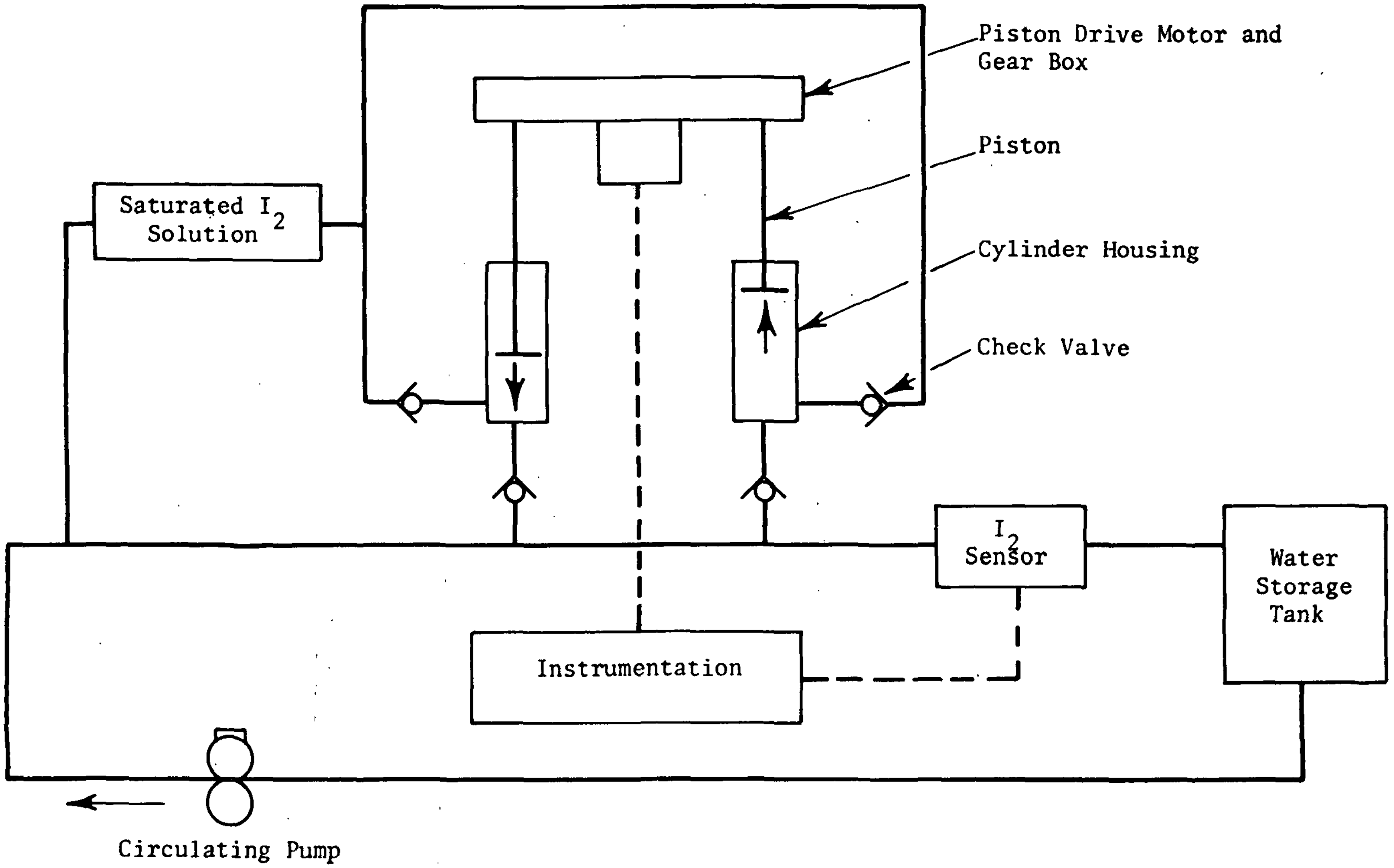


FIGURE 46 POSSIBLE MECHANICAL INJECTION SYSTEM

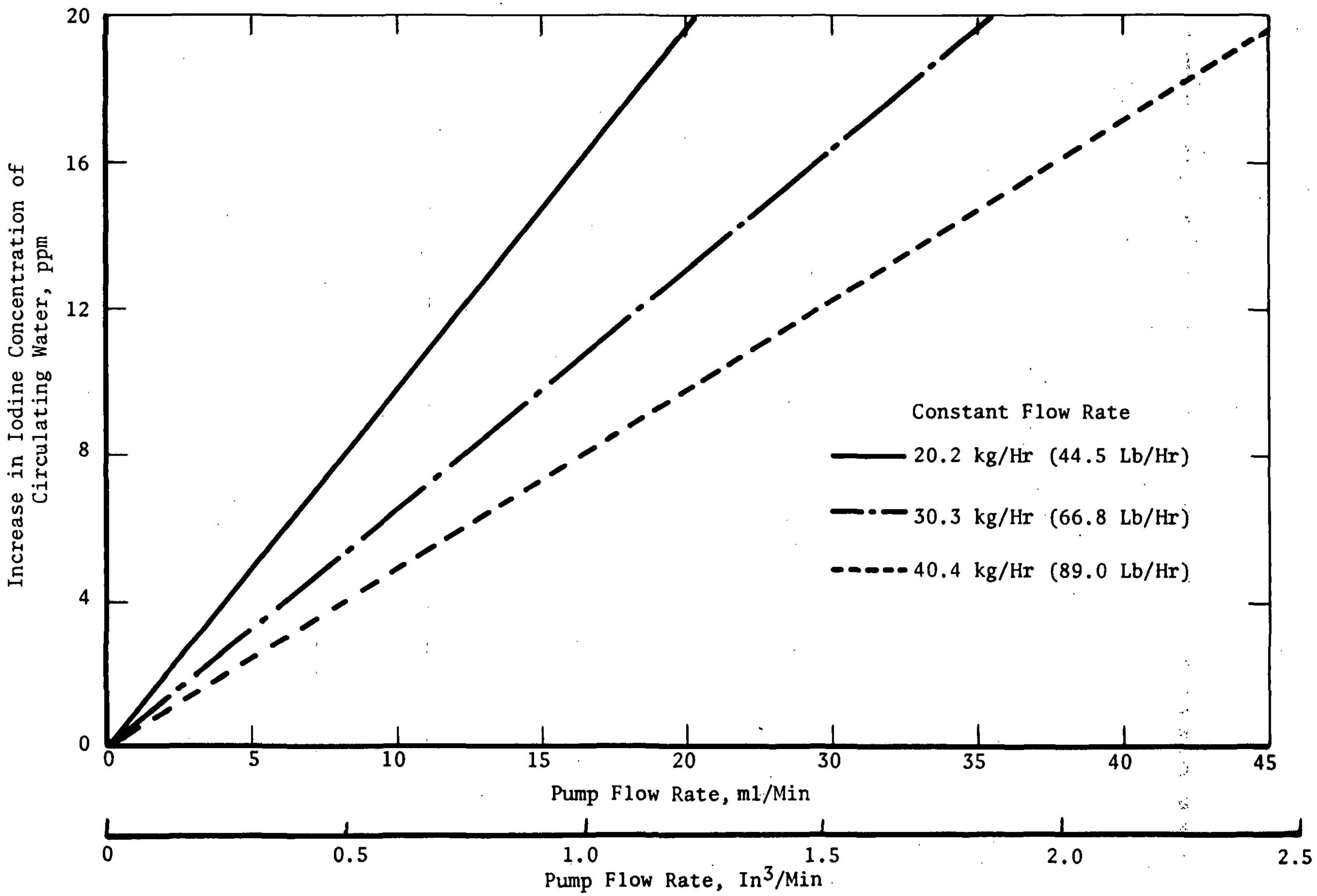


FIGURE 47 SATURATED IODINE SOLUTION FLOW RATES

Advantages and Disadvantages

A limited comparison was made of the advantages and disadvantages of the mechanical and electrochemical methods.

Electrochemical Approach - The electrochemical approach is attractive because it

a. allows more accurate control of I_2 injection,

b. has a lower power requirement,

c. has no moving parts,

d. is simple to scale-up, and

e. offers the potential of de-iodinating water treated with excess I_2 to provide an overkill.

The disadvantage was the initial uncertain level of development risk.

Mechanical Approach - The mechanical approach was attractive because

a. it was able to iodinate at fast rates (but had difficulty meeting I_2 feed regulation at low feed rates), and

b. its technology is more established, reflecting less development risk.

The disadvantages were

a. it has a larger equivalent weight (larger system component and power) and

b. it requires continuous or periodic operation of a pump.

Mechanical Approach Work Terminated

Effort directed toward the mechanical was terminated when the electrochemical approach offered the more attractive solution to the I_2 feed requirement. This occurred after test data indicated the process was feasible, and a 35-day endurance capability was demonstrated with breadboard hardware.

Preliminary Comparison of Potable Water Disinfecting System

A preliminary system evaluation was made between the electrochemical I_2 system, the mechanically fed I_2 system, and an Ag^+ system for an application requiring 33 kg (72 lb) water per day at 20 ppm I_2 for 180 days and 33 kg (72 (lb) water per day at 2 ppm Ag^+ for 180 days. Results are summarized in Table 11. Details are presented in Table 12. The I_2 approach has a lower weight because it avoids the need for ion exchange columns required to remove excess Ag^+.

TABLE 11 BIOCIDAL AGENT COMPARISON

Approach	Weight, kg (Lb)	Volume, Liter (Ft^3)	Power, Watt
Electrolytic I_2	3.0 (6.7)	1.8 (0.06)	8
Mechanical I_2	5.1 (11.3)	2.9 (0.10)	67
Ag^+	23.9 (52.8)	8.4 (0.30)	0

TABLE 12 COMPARISON OF POTABLE WATER BIOCIDAL AGENT SUBSYSTEMS[a]

Component	Electrochemical I_2 Injection			Mechanical I_2 Injection			AgCl Injection[b]		
	Weight kg (Lb)	Volume cm^3 (In^3)	Power, Watt	Weight kg (Lb)	Volume cm^3 (In^3)	Power, Watt	Weight kg (Lb)	Volume cm^3 (In^3)	Power, Watt
Power Supply and Electronics	1.02 (2.24)	440 (27)	7	1.02 (2.24)	440 (27)	7	-	-	-
Cases	0.37 (0.82)	-	-	0.68 (1.50)	-	-	-	-	-
Valve	1.37 (3.01)	1,330 (81)	1	-	-	-	-	-	-
Iodine	0.28 (0.61)	-	-	0.28 (0.61)	-	-	-	-	-
Injector	-	-	-	0.08 (0.18)	30 (2)	-	-	-	-
Pump	-	-	-	2.38 (5.24)	1,750 (107)	60	-	-	-
Pump Drive Amplifier	-	-	-	0.26 (0.57)	440 (27)	-	-	-	-
Saturator	-	-	-	0.44 (0.97)	250 (15)	-	-	-	-
AgCl Columns	-	-	-	-	-	-	3.17 (7.00)	2,290 (140)	-
Ion Exchange Column[c]	-	-	-	-	-	-	3.99 (8.81)	1,020 (62)	-
(Five Sets of Spares)	-	-	-	-	-	-	16.76 (37.00)	5,080 (310)	-
	3.04 (6.68)	1,770 (108)	8	5.14 (11.31)	2,910 (178)	67	23.92 (52.81)	8,390 (512)	0

(a) Without the biocidal agent level detector and making engineering estimates of projected component weight, volume and power.
(b) See Ref. 23.
(c) 30-Day replacement.

CONCLUSIONS

A program to develop a prototype Iodine Generating and Dispensing System (IGDS) was successfully completed, including functional testing of the breadboard and six-man prototype systems.

The program was initiated on the basis of using a previously developed chlorine (Cl_2) generating concept. Conversion of the on-site Cl_2 generating concept to an iodine- (I_2) based device was successful. Two electrolytic components, the hydrogen (H_2) eliminator and disinfecting agent generator, were eliminated.

The electrochemical I_2 valve developed during the program proved to be an acceptable method of dispensing and controlling I_2 in simulated spacecraft potable water stores. Iodine produced by the generator eliminated tracer E. coli bacteria from simulated water stores.

Exploration of I_2 chemistry for the intended end application of controlling microbial growth in spacecraft potable water stores showed, among other things, that I_2 concentrations of 5 ppm or less will eliminate most microorganisms and not adversely affect water "taste."

A water flow rate of 120 kg (264 lb) per day can be iodinated to 5 ppm by the prototype generator that measures 9.53 x 2.54 x 2.05 cm (3.75 x 1.0 x 0.81 in) and contains, in addition to electrochemical components, enough I_2 for 180 days. Iodination is attained with less than 10 watts power being consumed by the prototype whose design is compatible with the 78 kg (171 lb) and 46.3 kg (102 lb) per day anticipated water flow rates of a Space Station and Space Shuttle, and the possible water system operating modes for these spacecraft.

Other studies performed during the program indicated, for a variety of reasons, that I_2 is a preferred biocide, in particular, for a Shuttle Orbiter application. However, additional development is needed to avoid I_2 leaching from the generator and to enhance generator performance. Other work is necessary to integrate an improved I_2 generator with an I_2 level detector/monitor since the experiments performed in this program were with a simulated detector. Analysis of the development necessary indicates that a flight qualified electrochemical I_2 generator can be readied for Space Shuttle utilization.

REFERENCES

1. Schubert, F. H. and Wynveen, R. A., "Development of a Laboratory Breadboard Model of a Chlorine Generating Device to Chlorinate Reclaimed Water on a Spacecraft," Final Report, NASA CR-111854, December, 1970.
2. "A Comparison Between Chlorine and Iodine to Maintain Biocidal Conditions In Water Supplies," Life Systems, Inc., ER-171-7, November 10, 1972.
3. "Iodine Colorimeter," Beckman Instruments, Final Report, NAS9-11879, July, 1971.
4. Latimer, W. M., "The Oxidation States of the Elements and Their Potentials in Aqueous Solutions," Second Ed., Prentice-Hall, Inc., New York, 1952.
5. "Evaluation of Chlorogen Approach to Iodine Generation," Life Systems, Inc., ER-171-6, September 8, 1972.
6. Moeller, T., "Inorganic Chemistry," John Wiley & Sons, Inc., New York, 1954.
7. "Corrosion Control and Disinfection Studies in Spacecraft Water Systems," Engineering Science, Inc., NAS9-9431, March, 1972.
8. Black, A. P., Kinman, R. N., Thomas, Jr., W. C., Freund, G., and Bird, E. D., "Use of Iodine for Disinfection," J. AWWA, November, 1965.
9. Chang, Shih L., "The Use of Active Iodine as a Water Disinfectant," J. Am. Pharm. Assoc., 48, 417, 1958.
10. "Space Shuttle Environmental Control/Life Support Systems," Hamilton Standard, NASA CR-1981, May, 1972.
11. "Space Shuttle ETC/LSS System Definition," AiResearch Manufacturing Company, NAS9-11592, May 20, 1972.
12. "Water and Waste Management Group," Delta Preliminary Design Package, Space Station Prototype (SSP) Document No. A66, Hamilton Standard, June, 1971.
13. "Potable Water Specification," NASA Johnson Space Center, SD-W-002, May 16, 1970.
14. Olivieri, V. P., Donovan, T. K. and Kawata, K., "Inactivation of Virus in Sewage," J. San. Engr. Div., 97, ASCE, 661, October, 1971.
15. Black, A. P. and Wittal, G. P., "New Methods for the Colometric Determination of Iodine Residuals, Part 1 - Iodine, Iodide and Iodate," J. AWWa, 59, pgs 471-490, April, 1967.
16. "Standard Methods for the Examination of Water and Wastewater," American Public Health Assoc., Inc., 13 Ed., 1971.

17. "Procedures and Requirements for the Flammability and Offgassing Evaluation of Manned Spacecraft Nonmetallic Materials," D-NA-0002, NASA Manned Spacecraft Center, July, 1968.

18. "The Encyclopedia of Plastics," Plastic Piping Systems, Inc., Newark, New Jersey, 1972.

19. "Seal Compound Manual," Parker Seal Co., Culver City, California, 1971.

20. "Valcourse Chemical Compatibility Chart," Valcor Engineering Corp., Kenilworth, New Jersey, 1967.

21. Private communication, J. W. Shumar, Life Systems, Inc. and R. N. Kinman, College of Civil Engineering, University of Cincinnati, August, 1972.

22. "Modern Plastics Encyclopedia," No. A, 49, McGraw-Hill Inc., New York, 1972.

23. "Water and Waste Management Group," Delta Preliminary Design Package SSP Document Nos. 71, 114 and 142, Hamilton Standard, Appendix 1, pgs 102-103, June, 1971.

APPENDIX A - REFERENCE DATA

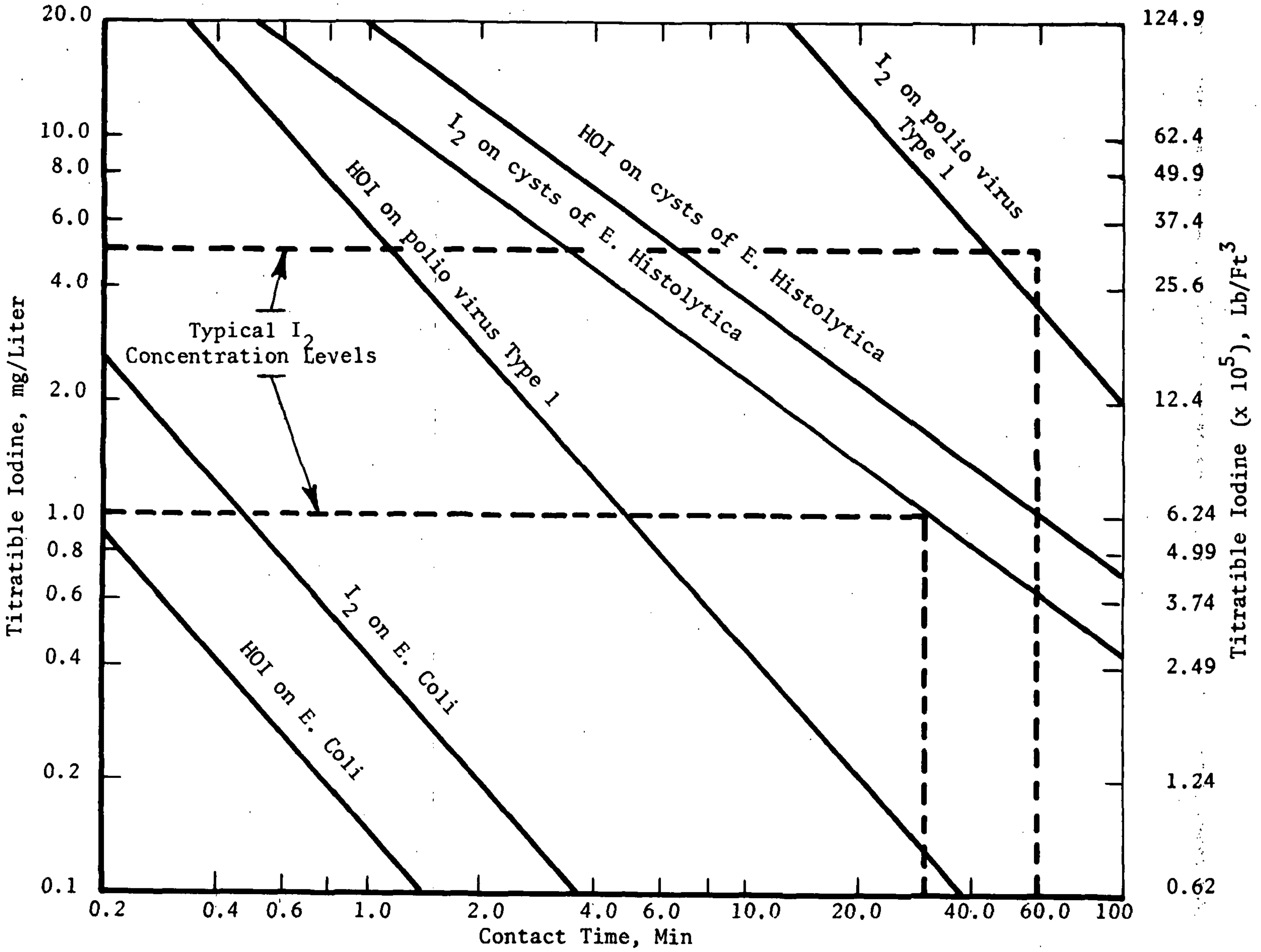


FIGURE A-1 TIME VERSUS CONCENTRATION RELATIONSHIP IN THE DESTRUCTION (99.9%) OF CYSTS, VIRUS AND BACTERIA BY I_2 AND HOI AT 291K (64F)

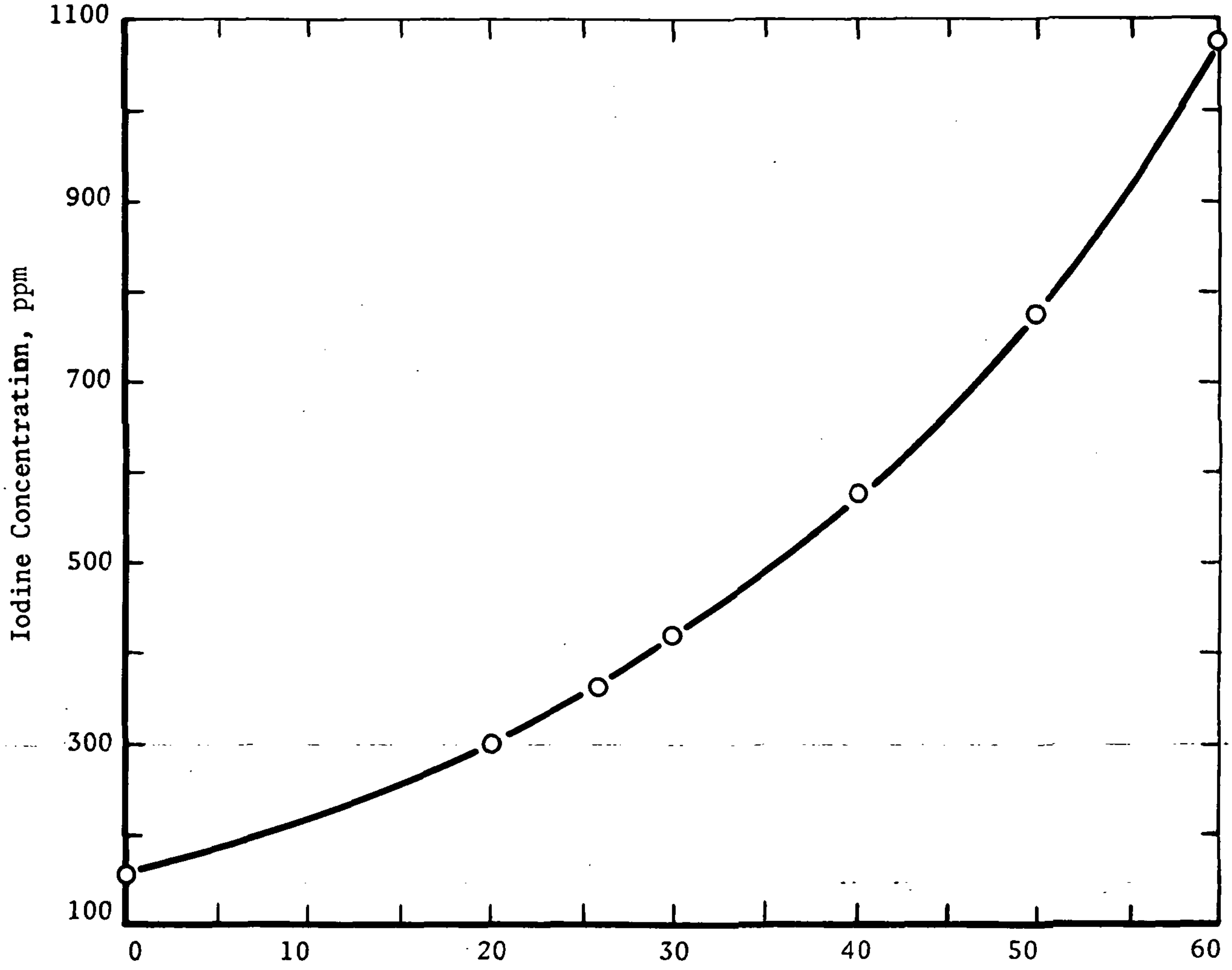


FIGURE A-2 IODINE SOLUBILITY IN WATER[a]

---

[a] Black, A. P., Kinman, R. N., Thomas, Jr., W. C., Freund, Gerhard and Bird, E. D., "Use of Iodine for Disinfection," J. AWWA, 57, pg. 1403, Nov., 1965.

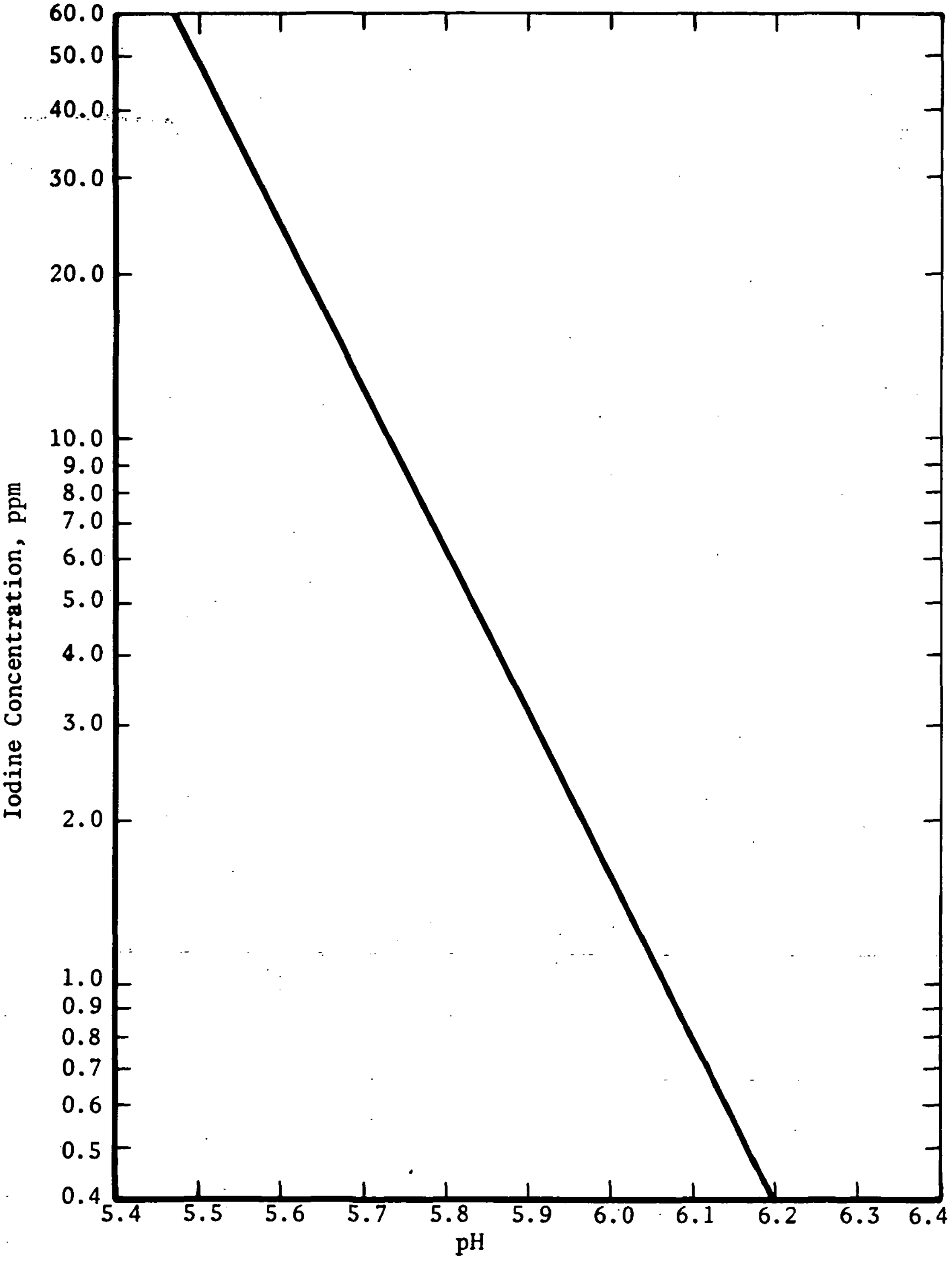


FIGURE A-3 pH OF AQUEOUS SOLUTIONS OF IODINE

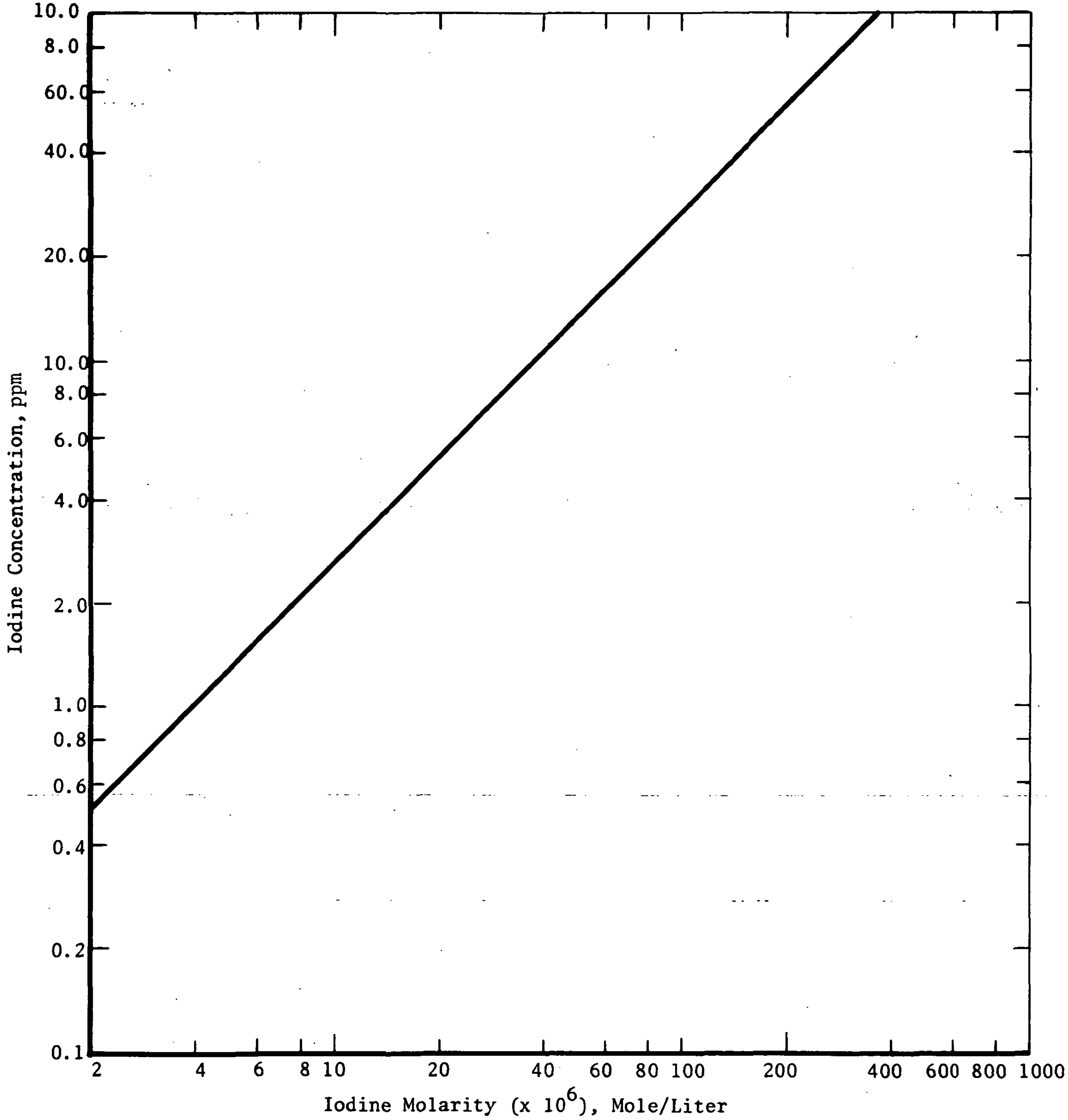


FIGURE A-4 CONVERSION OF IODINE CONCENTRATION FROM PPM TO MOLARITY

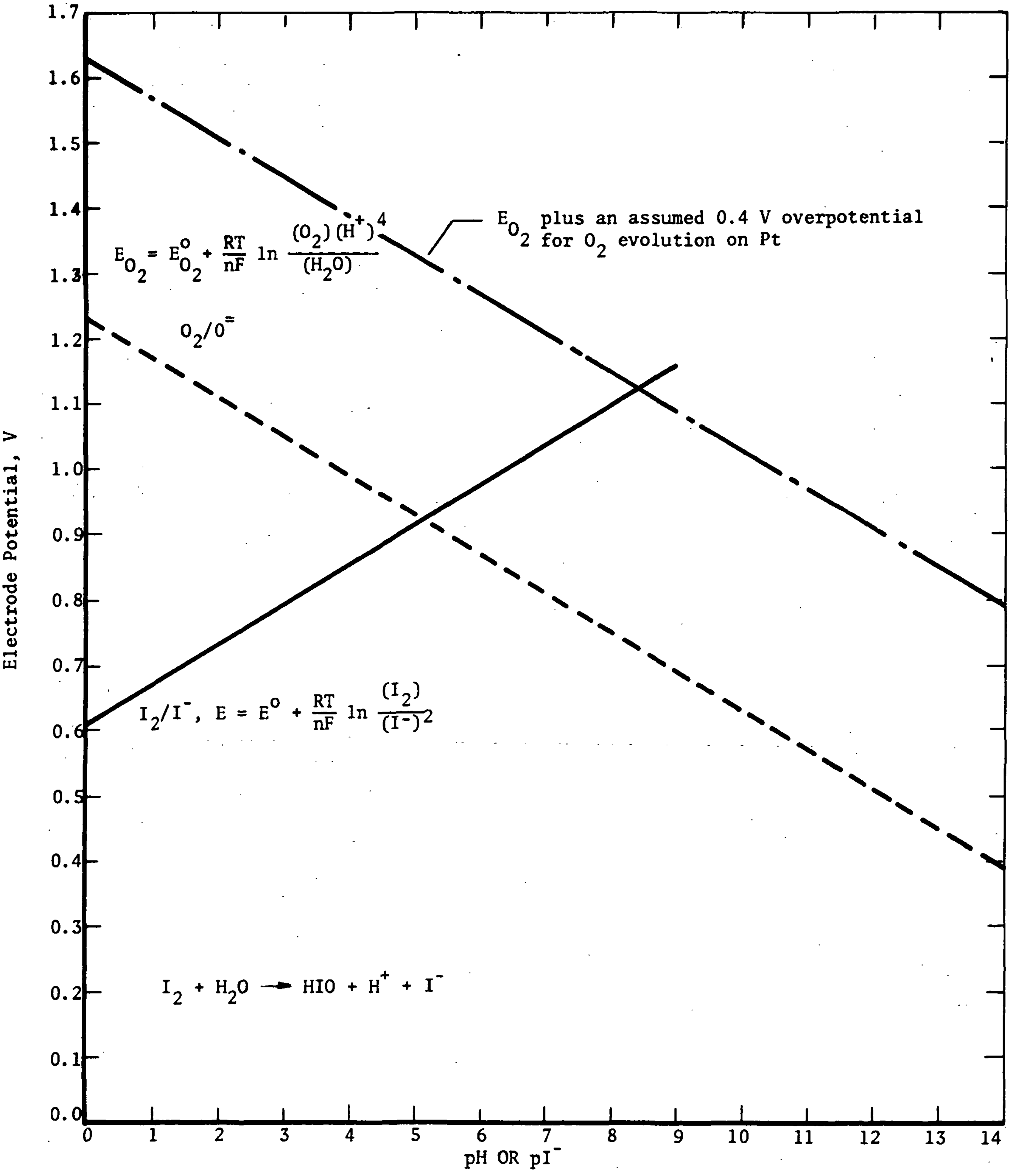


FIGURE A-5 ELECTRODE POTENTIAL AS A FUNCTION OF pH AND pI$^-$

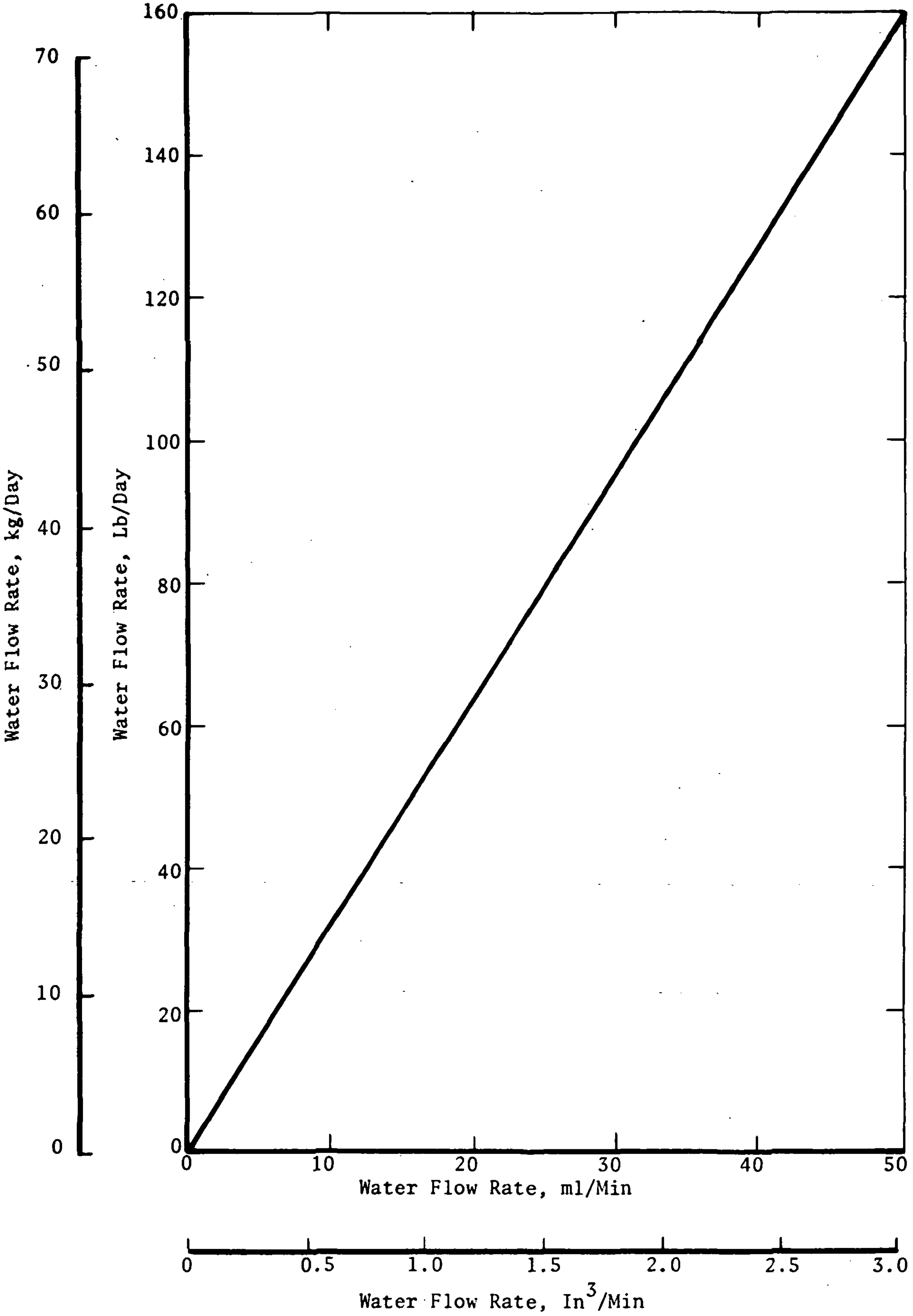


FIGURE A-6 CONVERSION FROM WEIGHT TO VOLUME FLOW

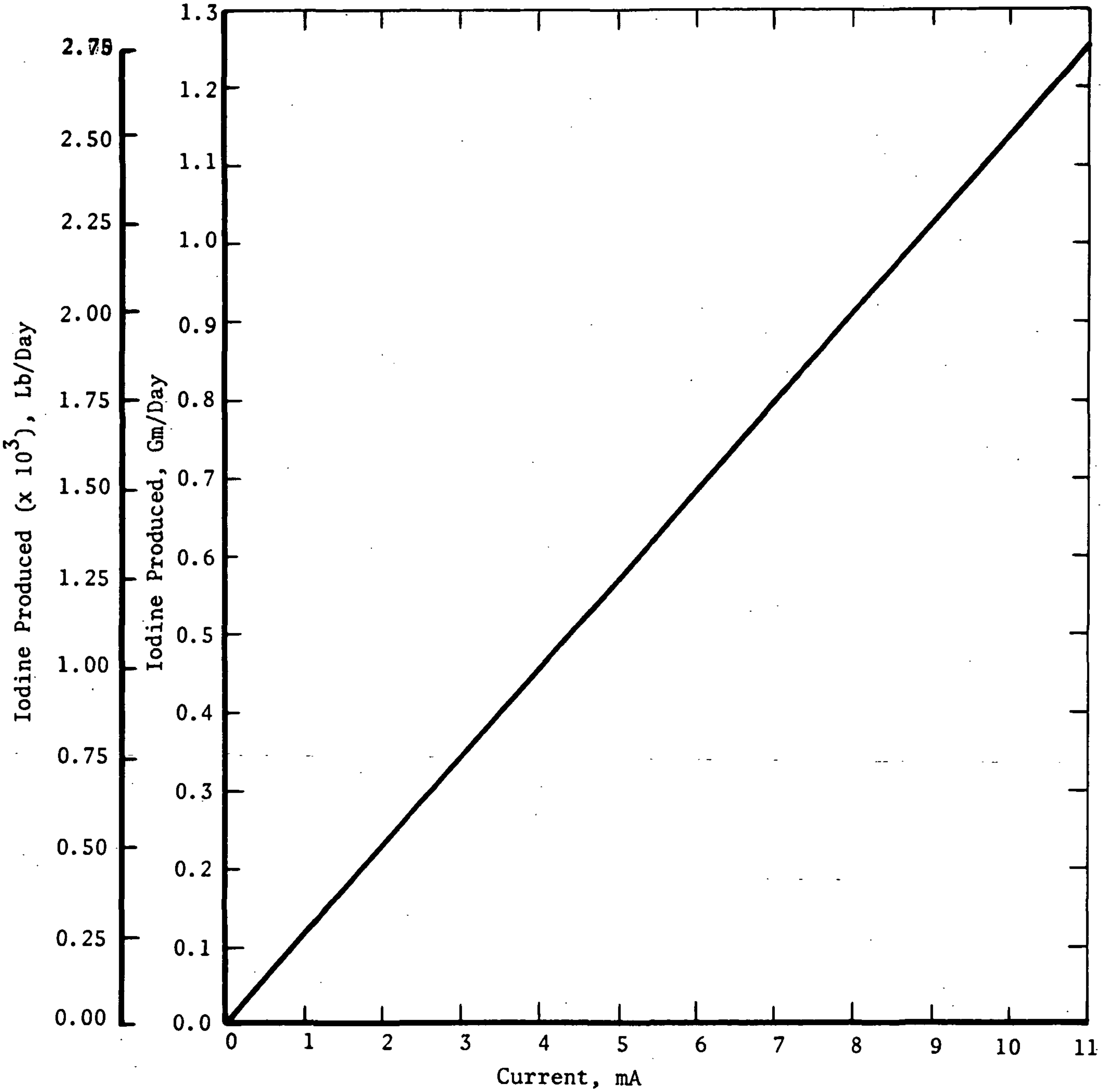


FIGURE A-7 THEORETICAL IODINE GENERATION RATE

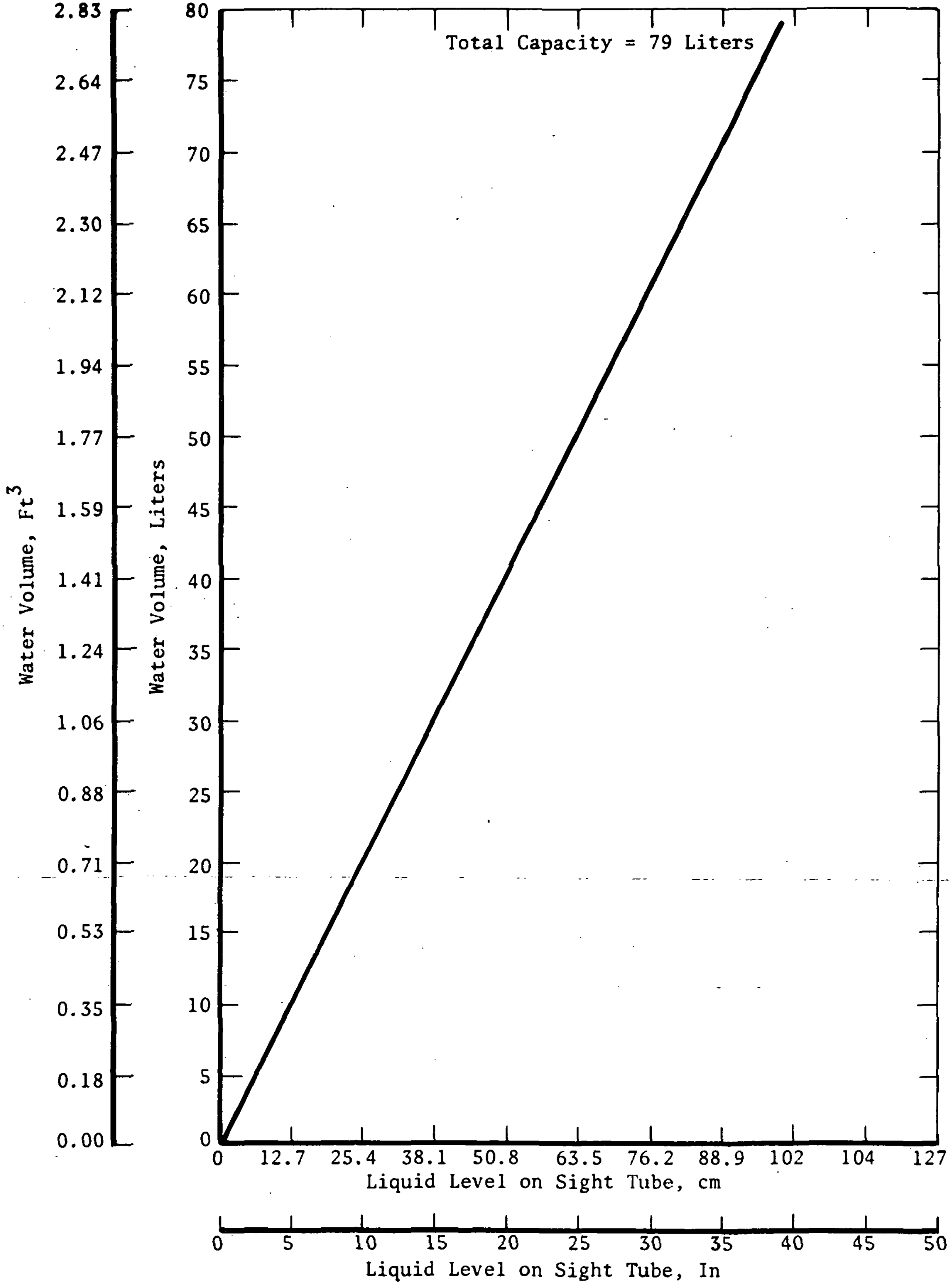


FIGURE A- 8 CALIBRATION OF WATER STORAGE TANK

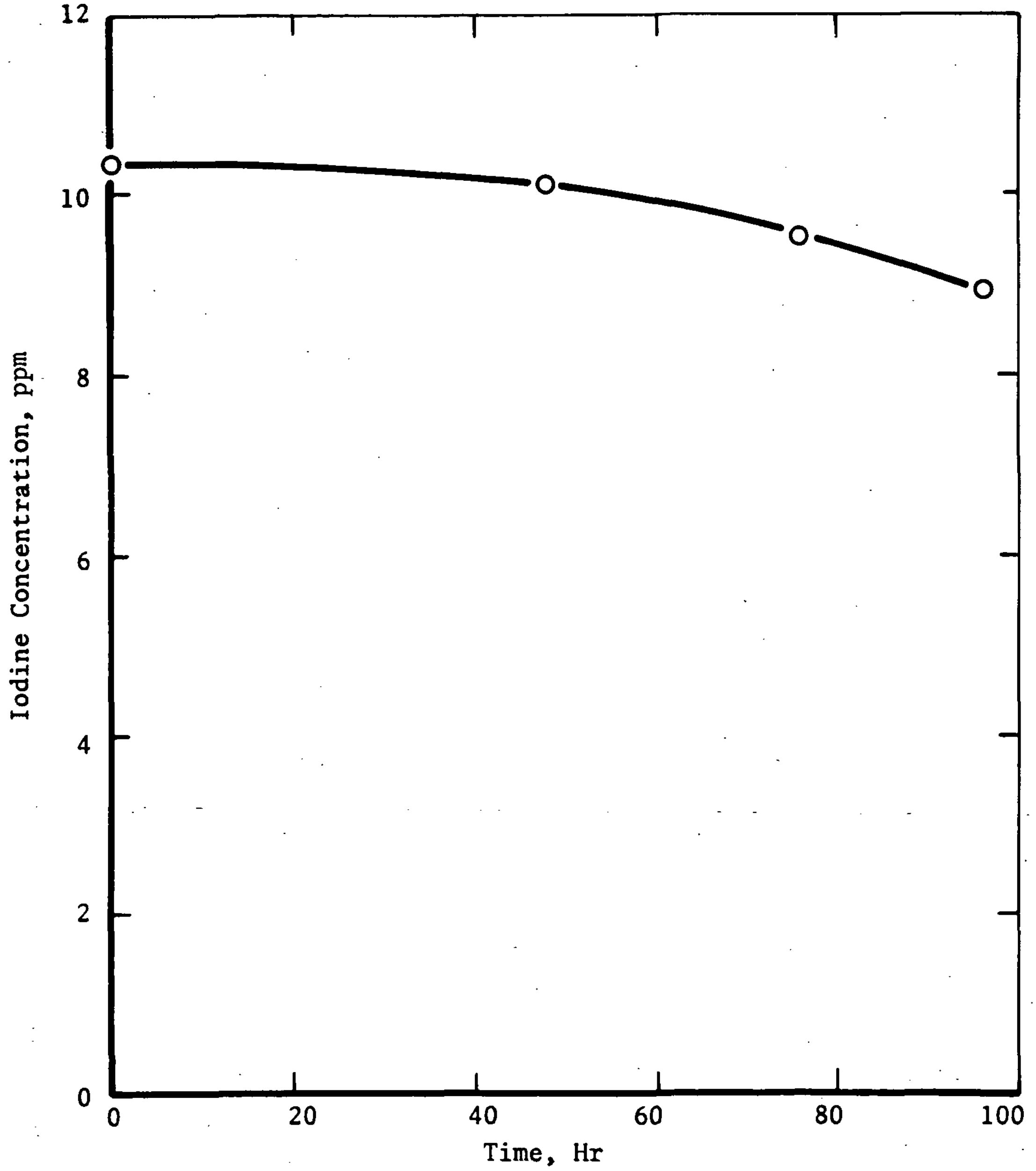


FIGURE A-9 EFFECT OF STORAGE TIME ON IODINE CONCENTRATION

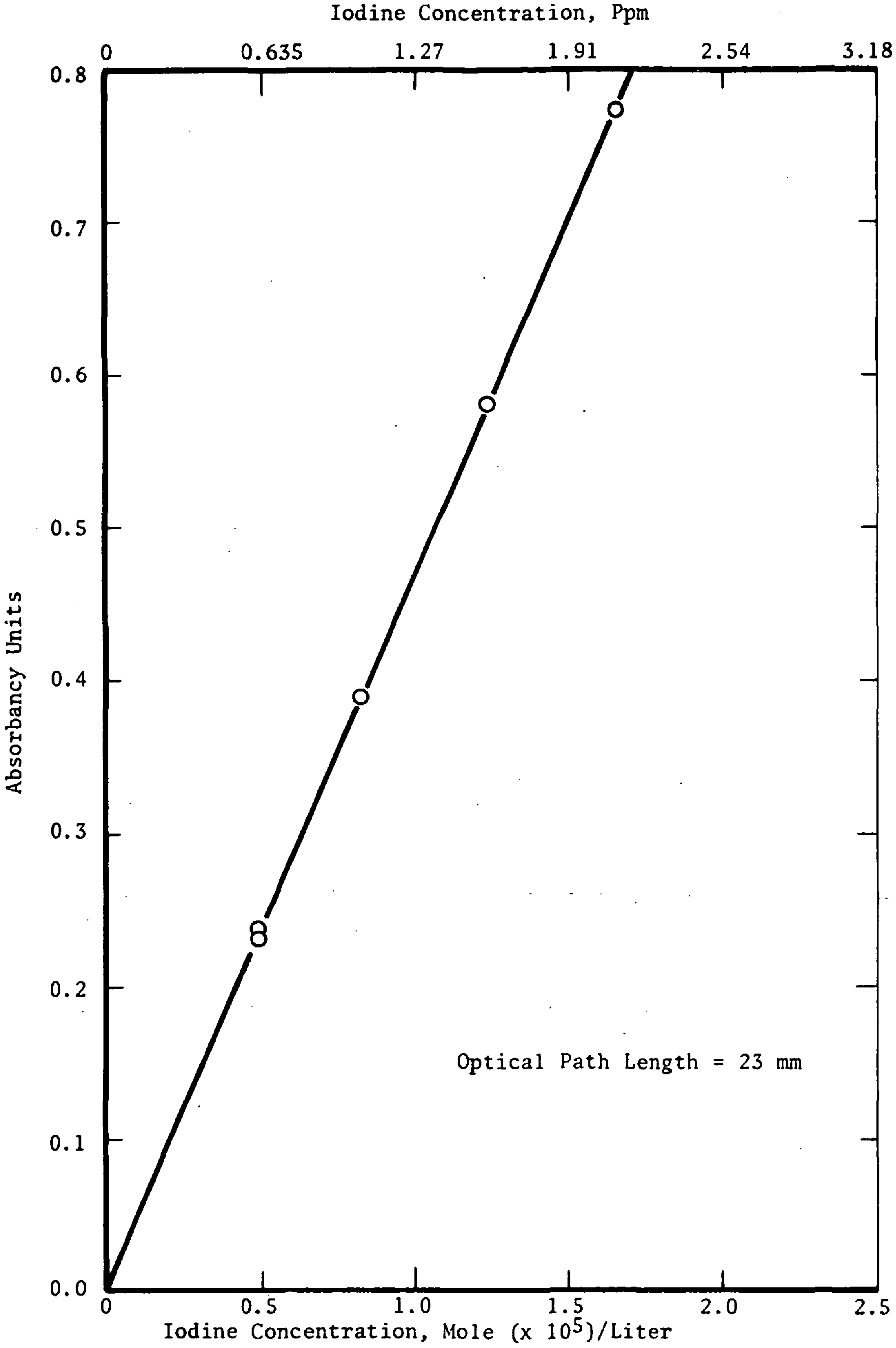


FIGURE A-10 ABSORBANCY VERSUS AQUEOUS IODINE CONCENTRATION

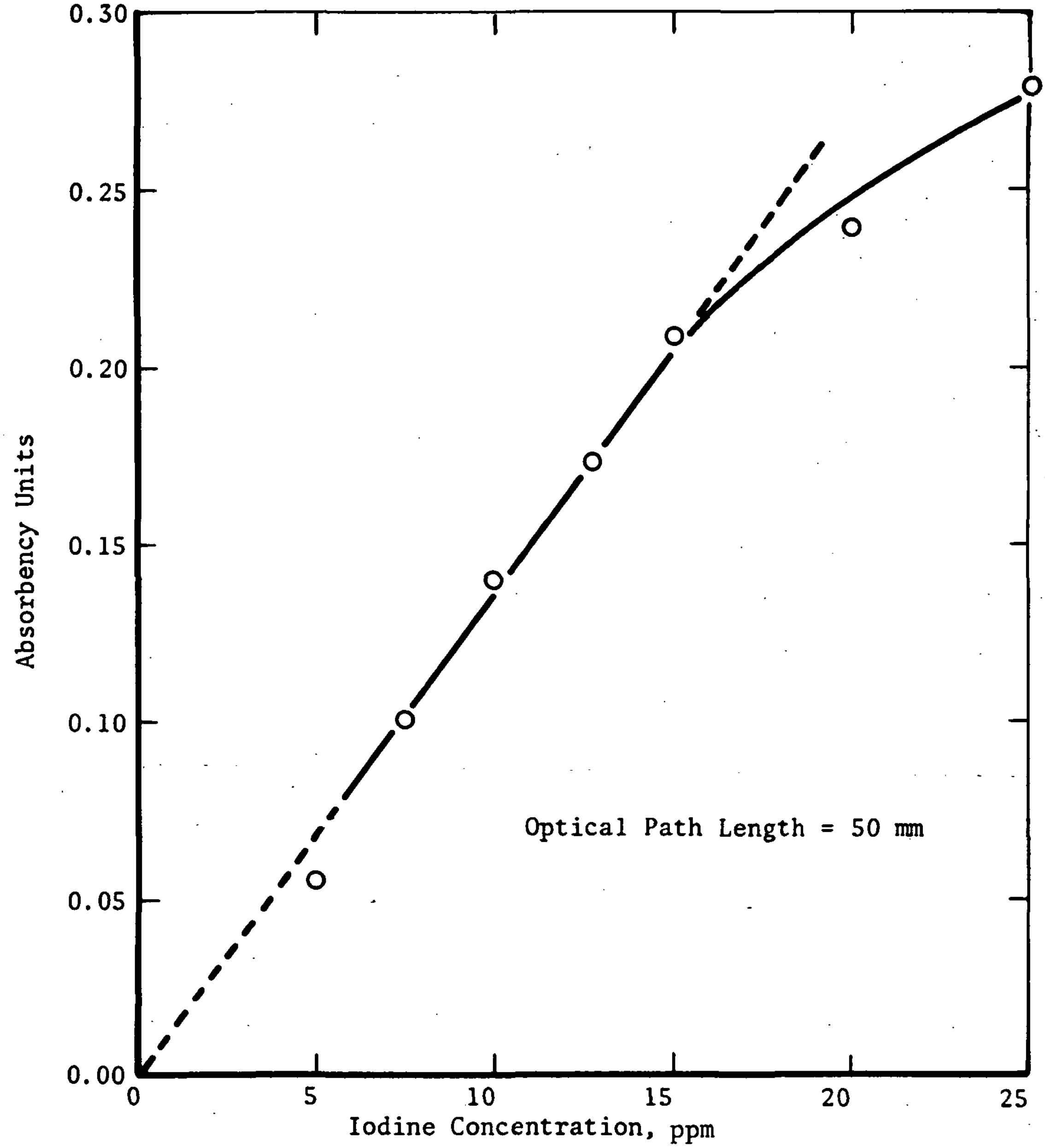


FIGURE A-11 ABSORBANCY VERSUS AQUEOUS IODINE CONCENTRATION

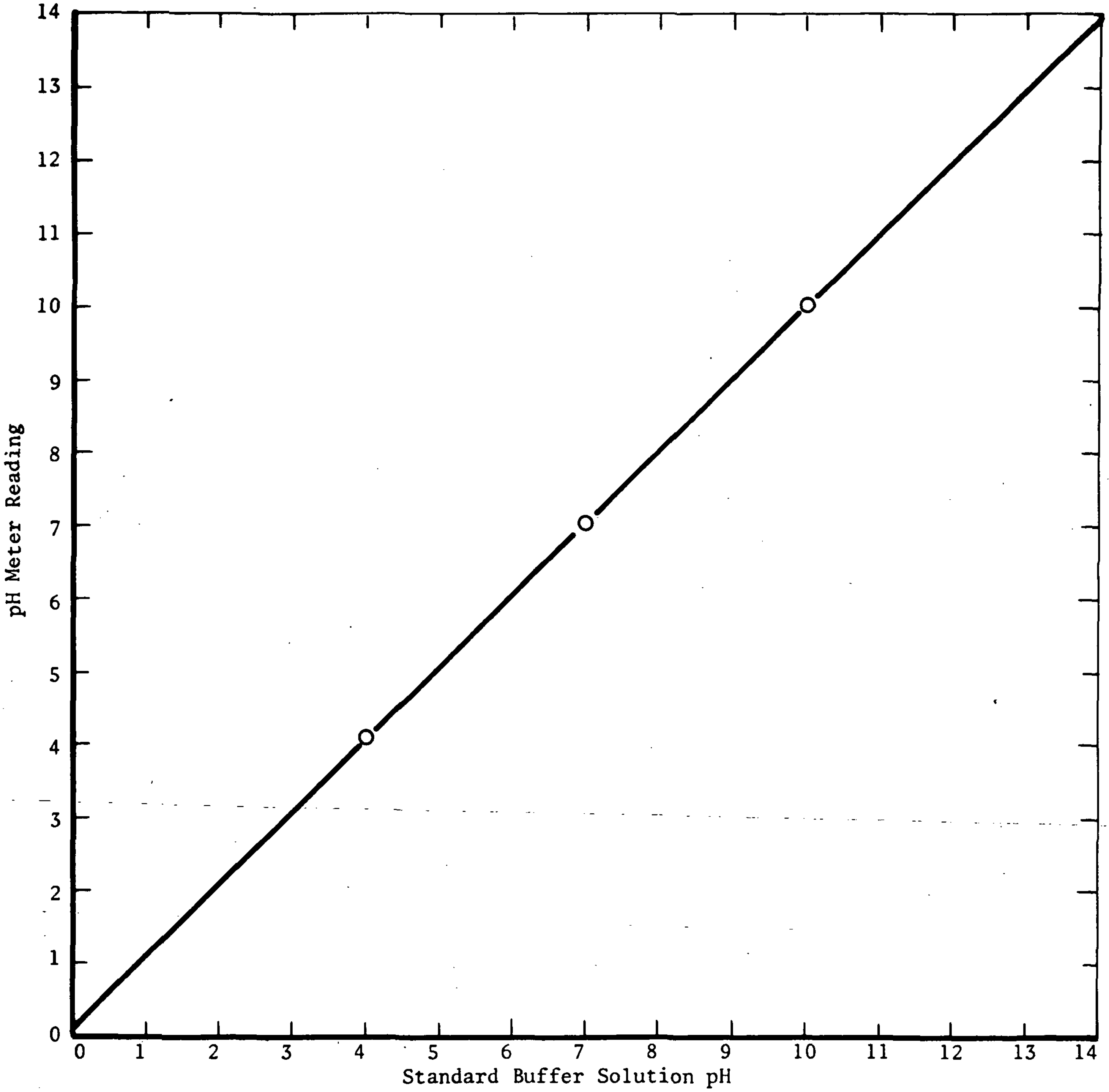


FIGURE A-12 CALIBRATION OF NO. 11 CORNING GLASS ELECTRODE USING A GGF CALOMEL

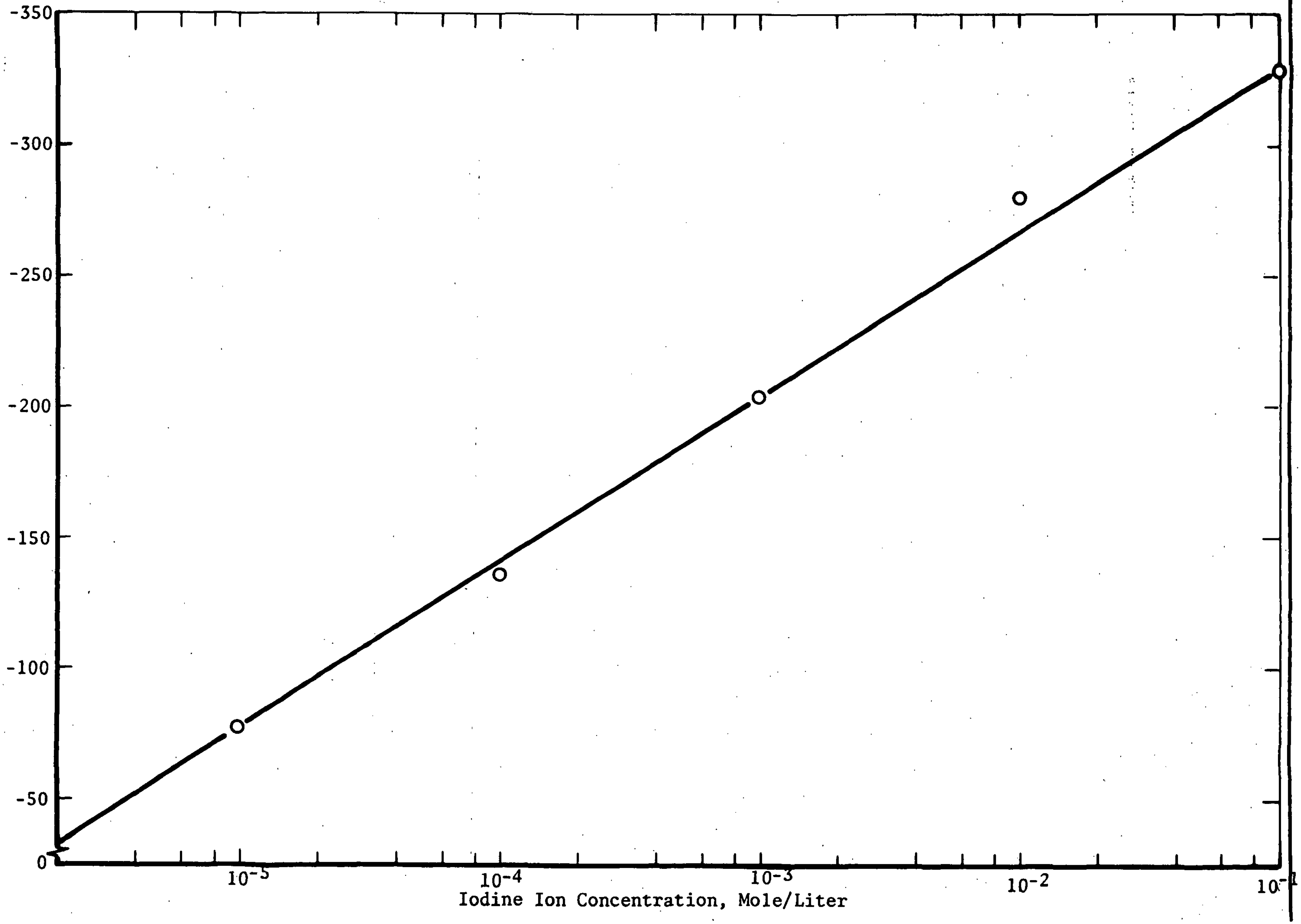


FIGURE A-13 IODIDE ION CALIBRATION CURVE

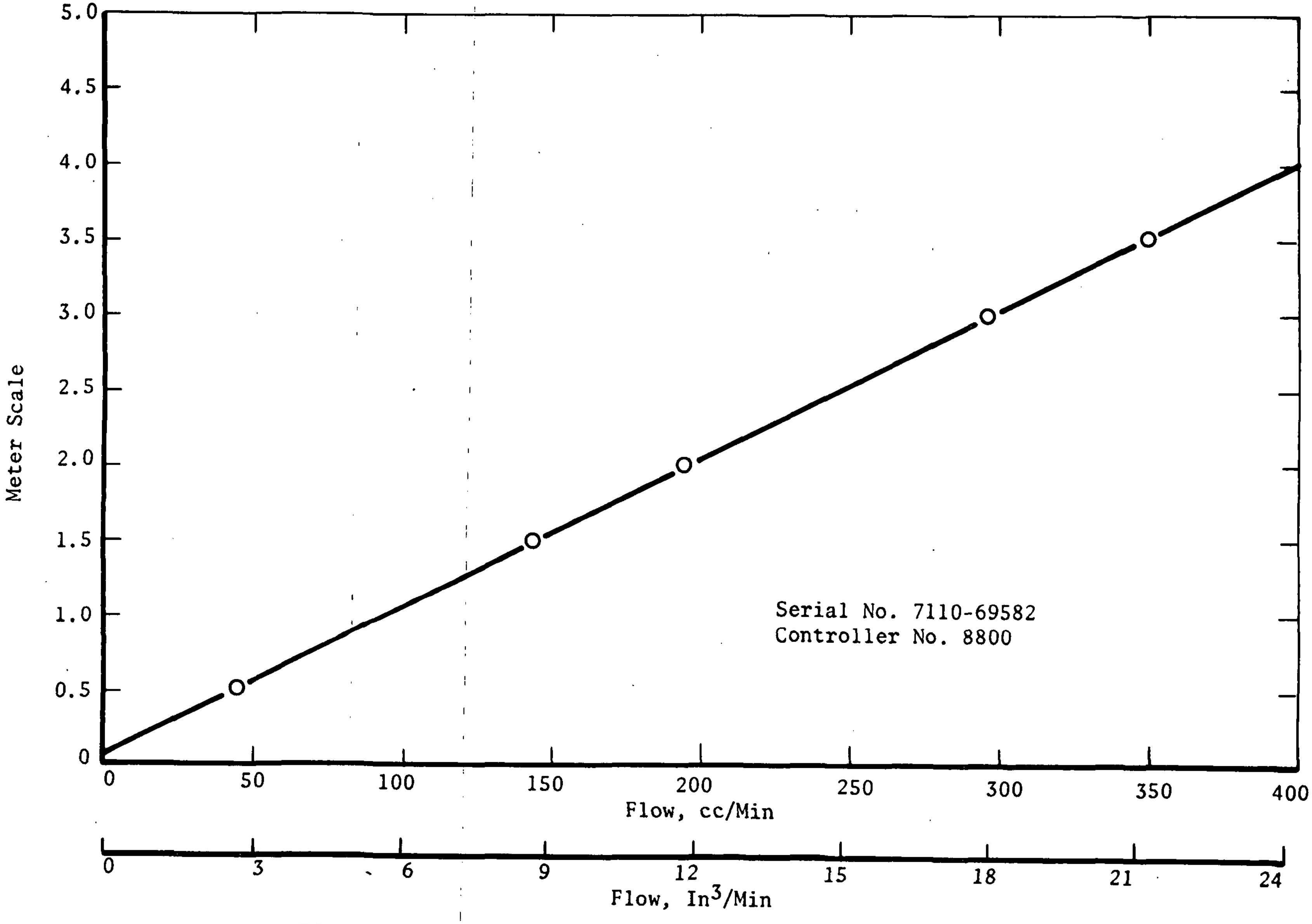


FIGURE A-14 CALIBRATION OF R-6-15-A FLOWMETER TUBE

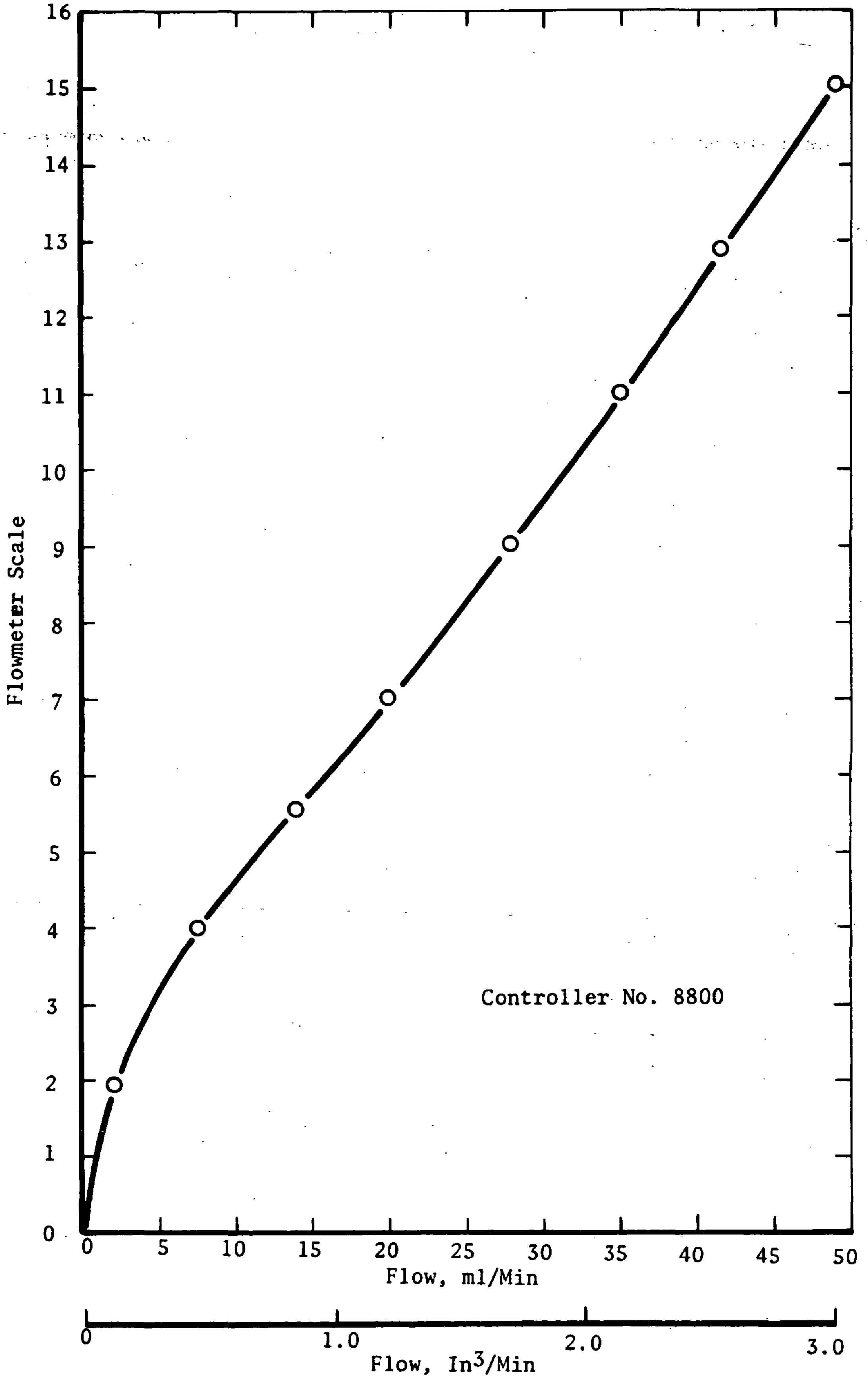


FIGURE A-15 CALIBRATION OF R-2-15-A FLOWMETER