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ELECTROLYTIC PRETREATMENT UNIT
GASEOUS EFFLUENT CONDITIONING

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AUGUST 1976

PREPARED UNDER CONTRACT NO. NAS9-14217
BY
UMPQUA RESEARCH COMPANY
MYRTLE CREEK, OREGON
FOR
LYNDON B. JOHNSON SPACE CENTER
NATIONAL AERONAUTICS AND SPACE ADMINISTRATION

UMPQUA
RESEARCH

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1.0 INTRODUCTION AND SUMMARY

The electrolytic pretreatment of urine is an advanced process that eliminates the need for handling and storing the highly corrosive chemicals that are normally used in water reclamation systems. The electrolytic pretreatment process also converts the organic materials in urine to gases (N_2 and O_2) that can be used to replenish those lost to space by leakage, venting, and air lock operations. The electrolytic process is more than a pretreatment, since it decreases the urine solids content by approximately one third, thus reducing the load and eventual solids storage requirements of the urine processing system. The evolved gases from the pretreatment step cannot, however, be returned directly to the atmosphere of a spacecraft without first removing several impurities including hydrogen, chlorine, and certain organic compounds.

The primary goal of this study was to develop a treatment concept that would decrease the impurities in the gas stream that emanates from an electrolysis unit to levels sufficiently low to allow the conditioned gas stream to be safely discharged to a spacecraft atmosphere. This goal was accomplished.

Two methods were experimentally demonstrated that can accomplish the desired cleanup. The bases of the two methods are, respectively: 1) raw urine scrubbing and 2) silica gel sorption.

Sufficient testing was accomplished to demonstrate that the urine used in the raw urine scrubbing process undergoes a significant oxidation in which its TKN and TOC are reduced by approximately 5 to 10%. This should result in the use of less power when scrubber urine is subsequently processed in the electrolysis unit.

It has been demonstrated that spent silica gel used in this application can be readily desorbed and regenerated to its original capacity by applying moderate temperatures at atmospheric pressure. The major impurities identified in the gas stream were Chloroform (1050 ppm max), Methyl Chloride (7000 ppm max), di-Chloro-Methane (500 ppm max), light hydrocarbons (300 ppm max) and di-Chloro-Ethane (100 ppm max). There were a number of other organic impurities in the low ppm range. Most of these materials were identified by GC-mass spectroscopy at JSC.

A subsystem concept comprised of the following sequential unit processes and operations was successfully demonstrated: a) raw urine scrubbing, b) silica gel sorption, c) dilution with cabin air, and d) catalytic oxidation.

All of the unwanted impurities were reduced to acceptable levels before entering the catalytic burner, which served principally to eliminate hydrogen.

2.0 OVERVIEW OF THE STUDY

2.1 Unit processes

The original objectives of this effort were to experimentally investigate two promising unit processes for removing impurities from the gas stream:

a) raw urine scrubbing
b) sorption with used activated carbon

After award of the contract, and in anticipation of possible additional funding, the study was replanned to include two additional unit processes:

c) sorption with silica gel
d) hydrogen separation with a polymer membrane

2.2 Study Approach

Based on these unit processes, four gas conditioning subsystems were defined and a test plan was formulated to work toward the achievement of at least one viable method of gas conditioning in as expeditious a manner as possible, but with emphasis on unit processes c and d. Midway through the study it became evident that additional funding would not become available to support work on unit process d, the polymer-membrane hydrogen-separation process. At this point in time, process c, the silica-gel sorption process, had been successfully demonstrated. With the planned work on the hydrogen separator cancelled, the next step in the Overall Test Plan was to investigate process a, raw urine scrubbing. Since the results were encouraging, a complete subsystem using raw urine scrubbing followed by sorption with silica gel, dilution with cabin air and catalytic oxidation was tested and the concept successfully demonstrated. This test concluded the experimental phase of the study.

2.3 Overall Results

In summary, the study was originally planned to investigate: a) raw urine scrubbing and b) sorption with used activated carbon. Encouraging data were obtained on 1) raw urine scrubbing and c) sorption with silica gel. A viable concept for conditioning the offgas stream from a urine electrolysis unit has been achieved, and the groundwork has been laid for investigating hydrogen separation, which has the potential for substantially decreasing the penalties associated with catalytically burning all of the hydrogen in the gas stream.

3.0 DESCRIPTION OF GAS CONDITIONING METHODS

3.1 Model of the Urine Electrolysis Gas Stream

A model of the gas stream was calculated based on data presented in References 1 and 2 and a mass balance of the electrolytic pretreatment process. The mass balance is presented in Table 3-1 and was based on the following assumptions:

a) Water Use Model (6-man crew)

Urine	9.41	
Urine Flush	2.35	
	11.75 Kg/day	1955.02 g/man-day

b) Urine Solids Input Model (NASA-MDAC 90-Day Test, Reference 3)

Total urine solids: 64.00 g/man-day

c) Composition of Urine Solids Model (Reference 1, Table III)

This table gives the amounts of C, N, O, and H contained in the organic and inorganic salts of typical human urine.

d) Composition of Discharge Gas Model (Reference 2, Figure 28)

This figure presents data on the composition of the discharge gas versus time. The curves were integrated to obtain the percentage values shown on the mass balance in Table 3-1.

The major gases shown in Table 3-1 were taken together with the trace gas analysis presented in Reference 2 to compile Table 3-2, which shows the total amounts of all the gases that had been identified prior to this study. Also shown in Table 3-2 is the calculated time to alert for each of the identified gases. It should be pointed out that Table 3-2 was the best model available of the composition of the gas stream at the start of this study. However, as a result of the GC-mass spec work done during the study, the trace contaminant portion of the model has undergone significant changes. These data are presented in Section 6.

In order to size the gas conditioning components, the amount of gas flow must be known. The average gas flow was calculated as follows:

From Table 3-1, 6.3731 g-moles/man-day × 6 men = 38.24 moles/day

Assume system functions 8 hours per day (maximum sunlight = 14 hours)

38.24 moles/day ÷ 8 hours/day = 4.78 moles/hour

4.78 moles/hour ÷ 60 min/hour = 0.07967 moles/min

0.07967 moles/min × 24 l/mole @ 1 atm-70°F = 1.912 l/min

TABLE 3-1 MASS BALANCE FOR ELECTROLYTIC PRETREATMENT OF URINE

All values in grams per man-day unless otherwise indicated

	g-moles/man-day	%	TOTAL	C	N	O	H	INORGANIC ASH	H_2O
INPUT									
Water (electrolyzed)			53.64			47.68	5.96		
Water (vaporized)			3.19						3.19
Water (hydrated)			1.54						1.54
Water (unchanged)			1832.65						1832.65
Total Water Input			1891.02						
Organic Solids in Water			39.32	11.71	14.03	10.98	2.60		
Inorganic Solids in Water			24.68	.17		3.27	.01	21.23	
Total Solids Input			64.00						
TOTAL INPUT			1955.02	11.88	14.03	61.93	8.57	21.23	1837.38
OUTPUT									
Water			1837.65						
Inorganic Solids in Water			38.82		.04	16.01		21.23	1.54
Gases:									
H_2	4.2850	67.24	8.57				8.57		
O_2	0.4469	7.01	14.30			14.30			
N_2	0.4996	7.84	13.99		13.99				
CO_2	0.9641	15.13	43.42	11.84		31.58			
$(H_2O)_4$	0.1772	2.78	3.19						3.19
CO	0.0003	.00	.08	.04		.04			
Total Gases	6.3731	100.00							
TOTAL OUTPUT			1955.02	11.88	14.03	61.93	8.57	21.23	1837.38

TABLE 3-2 PRELIMINARY MODEL OF THE GASES GENERATED BY ELECTROLYTIC PRETREATMENT OF URINE (SEPTEMBER 1974)

	%	Amount[1] Generated mol/man-day	Amount[1] Generated g/man-day	Alert Level mg/m^3	Time to[2] Alert Days
CO_2	15.095	.9641	42.42	7237	6.824
O_2	6.997	.4469	14.30	---	----
N_2	7.882	.4996	13.99	---	----
H_2	67.091	4.285	8.570	800	3.734
H_2O	2.774	.1772	3.190	---	----
Cl_2	.143	.00912	.6475	2	.1236
CO	.05417	.00346	.09688	117	48.31
Total Organics[3] as HCHO	.01675	.00107	.0321	19	23.68
HCl	.001848	.000118	.004307	5	47.54
NH_3	.003758	.000240	.004080	53	490.7
NO_2	.0000293	.00000187	.00008602	3	1395
O_3	.00000210	.000000134	.000006432	0.3	1865
TOTAL	100.0585574	6.38681	83.2550		

Notes:

1 Based on 64 g/man-day of urine solids

2 Assumes 40 m^3/man

3 Total Organics (as HCHO). The organics which had been identified in the effluent gas stream prior to the start of this study are presented in Table 4 of Reference 2. They are as follows:

Major Items	Minor Items
2-2 Dimethyl Butane	Acetone
Dichloromethane	Ethyl Formate
3 Methyl Pentane	Methyl Ethyl Ketone
Formaldehyde	Methyl Alcohol
Propionaldehyde	Isopropyl Alcohol
	Ethyl Alcohol
	Butyl Alcohol
	Secondary Butyl Alcohol

3.2 Selection of Methods

Two of the selected processes (a. raw urine scrubbing and b. sorption with used activated carbon) were identified in the original proposal. Both of these processes were incorporated into subsystems in which the gas stream was diluted with cabin air to 1% H_2 before introduction to a catalytic oxydizer. This reduced the hydrogen level from a maximum of 72% H_2 to ¼ of its explosive limit of 4%. The amount of dilution air required to achieve 1% H_2 is calculated as follows:

$$1.912 \text{ liters/min} \times 71\%/1\% = 135.75 \text{ liters/min}$$

$$135.75 \text{ liters/min} \div 6 \text{ men} = 22.63 \frac{\text{liters/minute}}{\text{man}}$$

After award of the contract, an additional method was identified that involved the separation of hydrogen with a semi-permeable membrane. The hydrogen thus separated would avoid reaction with O_2 in the catalytic oxidizer and thereby save the considerable amount of electrical energy that would otherwise have to be expended to recover hydrogen and oxygen from the product water formed in the oxidation process. A palladium-silver alloy separator was briefly considered but rejected because Pd-Ag will catalytically react O_2 and H_2. These gases approach their stiochiometric ratio near the end of the process.

A Dupont reverse osmosis module made of dacron polyester fibers,Reference 4,can possibly achieve a fairly high percentage of H_2 separation from a stream containing significant quantities of O_2 with little danger of reacting the H_2 and O_2. Although Dupont had curtailed production of these modules, one of their large size units measuring 2.54 cm. diam. x 1.83 m. long was obtained for testing.

The presence of water vapor causes a large reduction in the hydrogen diffusion rate through a polymer membrane. Therefore drying of the gas stream was considered essential and silica gel was selected for this purpose, primarily because of its good capacity for water and its chemical inertness. In addition, it was recognized that many organic compounds are readily adsorbed by silica gel, and it was felt that silica gel might do a respectable job in removing the contaminants in this application.

Thus the following four unit processes were identified for experimental investigation:

a) raw urine scrubbing
b) sorption with used activated carbon
c) sorption with silica gel
d) hydrogen separation with a polymer membrane

These unit processes are discussed in the following paragraphs together with a description of the subsystem into which each was incorporated.

3.3 Method 1, Silica Gel-H_2 Separator- Reactor

This method (see Figure 3-1)uses a Dupont module that contains Dacron polyester hollow-fibers to separate hydrogen from the gas stream. All available information indicated that the materials of construction would be compatible with the different gases shown in Table 3-2. Ammonia attack was one concern, but the low amount in the effluent would probably have little or no effect. Because water vapor greatly decreases hydrogen diffusion, it was recommended that the water vapor level not exceed 0.1%. At a total pressure of 101,325 Pa (14.7 psia), this is equivalent to a water vapor partial pressure of 101.3 Pa (0.0147 psia). The saturation temperature for this partial pressure is approximately 252.8K (-5^{O}F). Approximately 3.4 g/man-day of water vapor would have to be removed to reach 0.1%. This would be accomplished with the small silica gel bed that is shown ahead of the polymer separator. In a real system, two beds could be used and cyclically desorbed, possibly with the same vacuum pump that is used for the polymer separator.

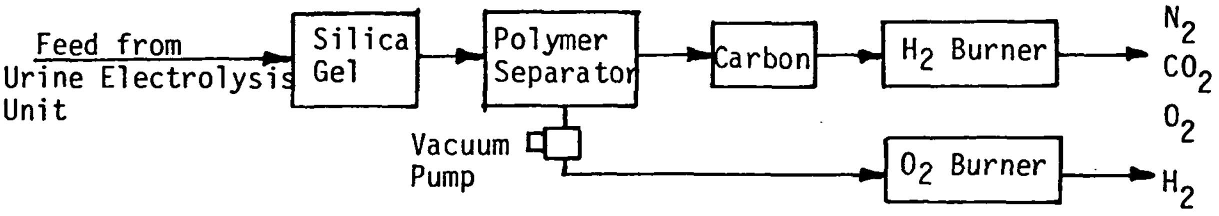
The lowest theoretical level to which the hydrogen on the high pressure side of the hollow fibers can be reduced is to a partial pressure equal to the absolute pressure on the vacuum side. For the high pressure side at 1 atm (14.7 psia) these values would be as follows:

Vacuum Side Pressure, Pa (psia)	Pressure Side Hydrogen, %
6894.8 (1.)	6.8
3447.4 (.5)	3.4
1723.7 (.25)	1.7
689.5 (.1)	.58

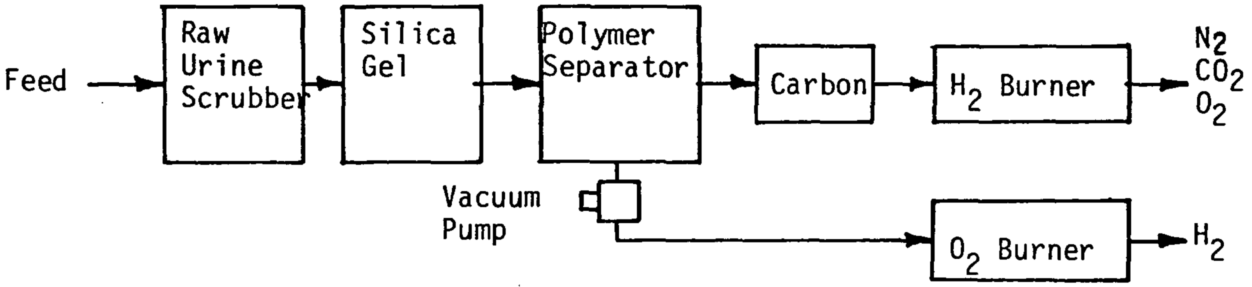
It would be desirable to achieve a hydrogen level comfortably below the explosive limit of 4%. When this is done, it is expected that both the H_2 stream and the main stream can be passed directly, without dilution, to reactors to remove traces of O_2 and H_2 respectively. It was recognized that if the silica gel adsorbed Cl_2 as well as H_2O, then no carbon would be required ahead of the H_2 reactor.

3.4 Method 2, Raw Urine Scrubber-Silica Gel-H_2 Separator-Reactor

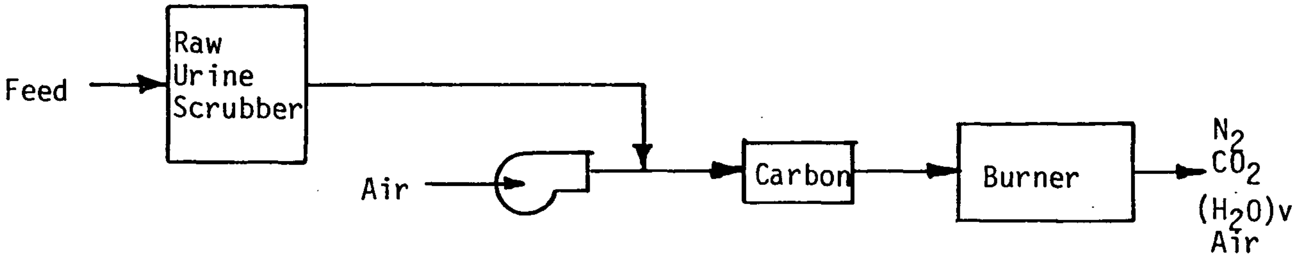
This method (see Figure 3-1)was added at the suggestion of the contract monitor and is a variation of Method 1. A raw urine scrubber is placed ahead of the other units to take advantage of the oxidizing capacity of the gas stream



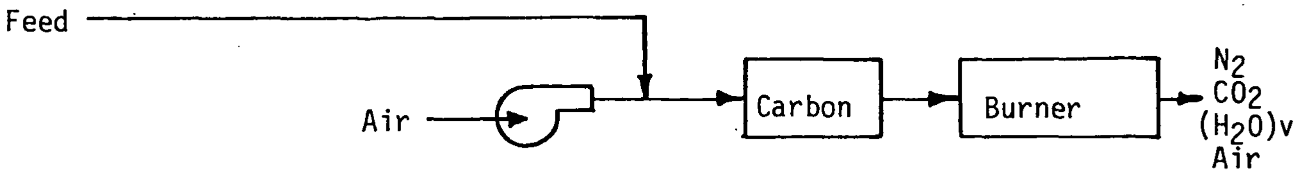
Method 1



Method 2



Method 3



Method 4

Figure 3-1, Urine Electrolysis Gas Conditioning Methods and reduce the load on the silica gel bed.

3.5 Method #3, Raw Urine Scrubber-Dilution-Reactor

This method (see Figure 3-1) consists of processing the gas stream in four steps as follows:

(1) scrubbing with raw urine to remove Cl_2,
(2) mixing with cabin air to reduce the hydrogen concentration below the explosive mixture range (less than 4% by volume),
(3) sorption by activated carbon to remove residual Cl_2 and other trace impurities including organic gases, HCl and NO_2,
(4) reacting the hydrogen, NH_3, and CO with oxygen in a catalytic bed.

In this method the raw urine itself is used to scrub the chlorine from the effluent gas. Thus no expendable material is required. In addition, some oxidation of urine organics occurs, thereby reducing the amount of oxidation needed in the electrolytic pretreatment process. Also, the raw urine is disinfected by the chlorine. The primary chemical reactions that apply to scrubbing chlorine with raw urine are described in the following paragraphs.

Each mole of chlorine gas rapidly combines with the water in urine to form a mole of hydrochloric acid and a mole of hypochlorous acid as follows:

$$Cl_2 + H_2O \rightarrow HCl + HOCl \qquad (1)$$

All of the oxidizing capability of Cl_2 is embodied in the HOCl, which has been observed to react with urea as follows:

$$3HOCl + H_2NCONH_2 \rightarrow CO_2 + N_2 + 3HCl + 2H_2O \qquad (2)$$

Also, any ammonium ion present would react with HOCl:

$$3HOCl + 2NH_4OH \rightarrow N_2 + 3HCl + 5H_2O \qquad (3)$$

The raw urine would tend to become acid as the HCl component increases. This can be prevented, if desired, by thermal decomposition of urea to form ammonium ions:

$$H_2NCONH_2 + 3H_2O \xrightarrow{T>340^0K} 2NH_4OH + CO_2 \qquad (4)$$

The ammonium ions would then buffer the HCl:

$$NH_4OH + HCl \rightarrow NH_4Cl + H_2O \qquad (5)$$

The overall reaction for either urea or ammonium oxidation and buffering of the HCl with NH_4^+ from thermally decomposed urea is:

$$4H_2NCONH_2 + 4H_2O + 3Cl_2 \xrightarrow{T>340^0K} 6NH_4Cl + 4CO_2 + N_2 \qquad (6)$$

The components required to accomplish the liquid scrubbing of chlorine are shown in Figure 3-2. They include: (1) a bubble generator that produces micron size bubbles by liquid shear forces; (2) a contactor scrubber where the

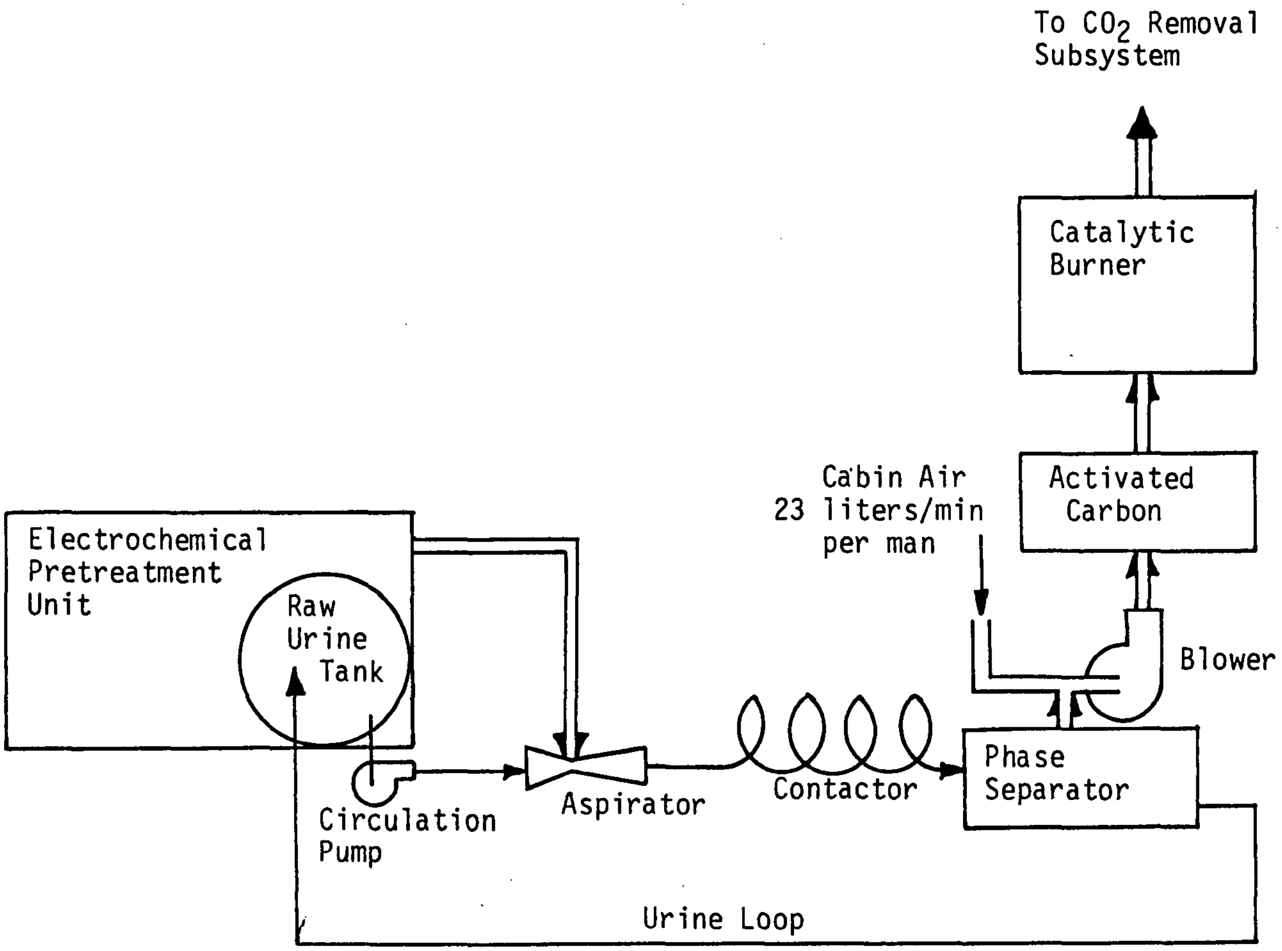


Figure 3-2, Method 3, Raw Urine Scrubber-Dilution-Reactor chlorine reacts with the water, etc. (equations (1-6); (3) a liquid-gas separator; (4) a raw urine storage tank; (5) a circulation pump; (6) a blower; (7) an activated carbon bed and (8) a catalytic hydrogen burner.

3.6 Method #4, Dilution-Activated Carbon-Reactor

This method (see Figure 3-1) involves the removal of chlorine in a bed of activated carbon. A diluting air stream is mixed with the hydrogen containing stream in order to obtain a non-explosive mixture before it is introduced to the carbon bed. The carbon used in this application could be obtained from other life support systems such as the urine phase separator and space cabin toxin control system which use beds of activated carbon to remove trace quantities of organic compounds from the air. These beds have a relatively low capacity for the hydrocarbons at the low concentration levels at which they must operate and consequently must be periodically replaced. These beds, however, should still have a rather large capacity for chlorine. By reusing these beds to scrub the chlorine essentially no expendable materials would be required. The exhaust from the reused bed would then be passed through a fresh carbon bed to remove trace compounds that might poison the catalytic burner (the first bed being saturated with organics), and finally to the hydrogen burner. The exhaust from the H_2 burner in both systems has a high CO_2 concentration and therefore would logically be directed to the inlet of the cabin's CO_2 scrubber.

The first carbon bed might possibly be regenerated or reactivated by the action of the gaseous chlorine. An experiment described on p. 14 of Reference 2 indicated successful reactivation of carbon by a chlorinated liquid stream. A gas stream should have much better transport dynamics and would probably not cause the severe deterioration of carbon that was observed in the liquid experiment.

3.7 Tradeoff Comparisons

A rudimentary tradeoff analysis of the four methods shown in Figure 1 was performed and the results are summarized in Table 3-3. On the basis of total equivalent weight, the order of preference of the methods is:

Method	Total Equivalent Weight, Kg
1	136
2	173
4	232
3	268

TABLE 3-3 TRADEOFF ANALYSIS OF URINE ELECTROLYSIS GAS CONDITIONING METHODS

Method	Item	Elec. Power	Elec. Power Equiv. Weight (0.16 Kg/w)	Thermal Rejec.	Thermal Rejec. Equiv. Weight (0.11 Kg/w)	Installed Weight	Spares/ Expend. Weight	Total Equiv. Weight	TOTAL, Kg
		watts	Kg	watts	Kg	Kg	Kg	Kg	
1	pressure drop	50	8.0					8.0	
	vacuum pump	90	14.4	90	9.9	9.6	2.7	36.6	
	silica gel	4	.6	6	0.7	9.1	2.7	13.1	
	H_2 separator					22.7	6.8	29.5	
	Carbon					4.6	1.4	6.0	
	reactors			53	8.5	9.1	2.7	20.3	
	penalty for later having to electr. all reacted O_2	78	12.5	25	2.8	7.3	2.2	24.8	138.3
			(same as #1 except for an added urine scrubber)						
2	raw urine scrubber	90	14.4	90	9.9	9.1	2.7	36.1	174.4
3	raw urine scrubber		(see above)					36.1	
	carbon					13.6	4.1	17.7	
	dilution fan	100	16.0	100	11.0	4.6	1.4	33.0	
	reactor			254	27.9	18.2	5.4	51.5	
	penalty for later having to electr. all reacted H_2	411	65.8	157	17.3	34.6	10.4	128.1	266.4
4	dilution fan		(see above)					33.0	
	carbon					13.6	4.1	17.7	
	reactor		(see above)					51.5	
	penalty for later having to electr. all reacted H_2		(see above)					128.1	230.3

The main reason that Methods 3 and 4 are so much heavier than Methods 1 and 2 is because of the large power and thermal penalties associated with reacting all of the H_2 with O_2 from the cabin. The water formed must later be electrolyzed to recover oxygen.

These trade-off numbers underscore the desirability of being able to separate hydrogen from the gas stream.

4.0 TEST PLAN

Using the results of the trade-off analysis as a guide in assigning priorities, an optimum test program was developed. The logic flow diagram for the test program is presented in Figure 4-1.

The manner in which the test program was actually carried out is shown in Figure 4-2. Midway through the study, after steps 1,2 and 3 had been accomplished, it was learned that the additional anticipated funding for the accomplishment of step 4 was unavailable. Therefore the study was advanced to step 7. Step 8A was added because the raw urine scrubber removed most, but not all, of the contaminates. The configuration of step 8A (Raw urine scrubbing followed by silica gel) resembled the first part of Method #2 (see Figure 3-1). Following the successfull testing of this configuration it was tested in conjuction with the dilution and reactor processes shown in Method #3, but without the activated carbon. This setup was designated as Modified Method #3 and is shown schematically in Figure 4-3. The weight penalty for this configuration is calculated by replacing the carbon penalties in Method #3 with the silica gel penalties in Method #1. The weight penalty summary for Modified Method #3 is presented in Table 4-1.

TABLE 4-1 WEIGHT PENALTIES FOR MODIFIED METHOD #3

	Elec Power	Elec Power Equiv. wt kg/w= 0.16	Therm Rejec. kg/w= 0.11	Therm Rejec. Equiv. wt	Instal-led wt	Spares/ Expend wt	Total Equiv wt	Total
Item	watts	kg	watts	watts	kg	kg	kg	kg
raw urine scrubber	90	14.4	90	9.9	9.1	2.7	36.1	
silica gel	4	.6	6	.7	9.1	2.7	13.1	
dilution fan	100	16.0	100	11.0	4.6	1.4	33.0	
reactor			254	27.9	18.2	5.4	51.5	
electrolysis of reacted H_2O	411	65.8	157	17.3	34.6	10.4	128.1	261.8

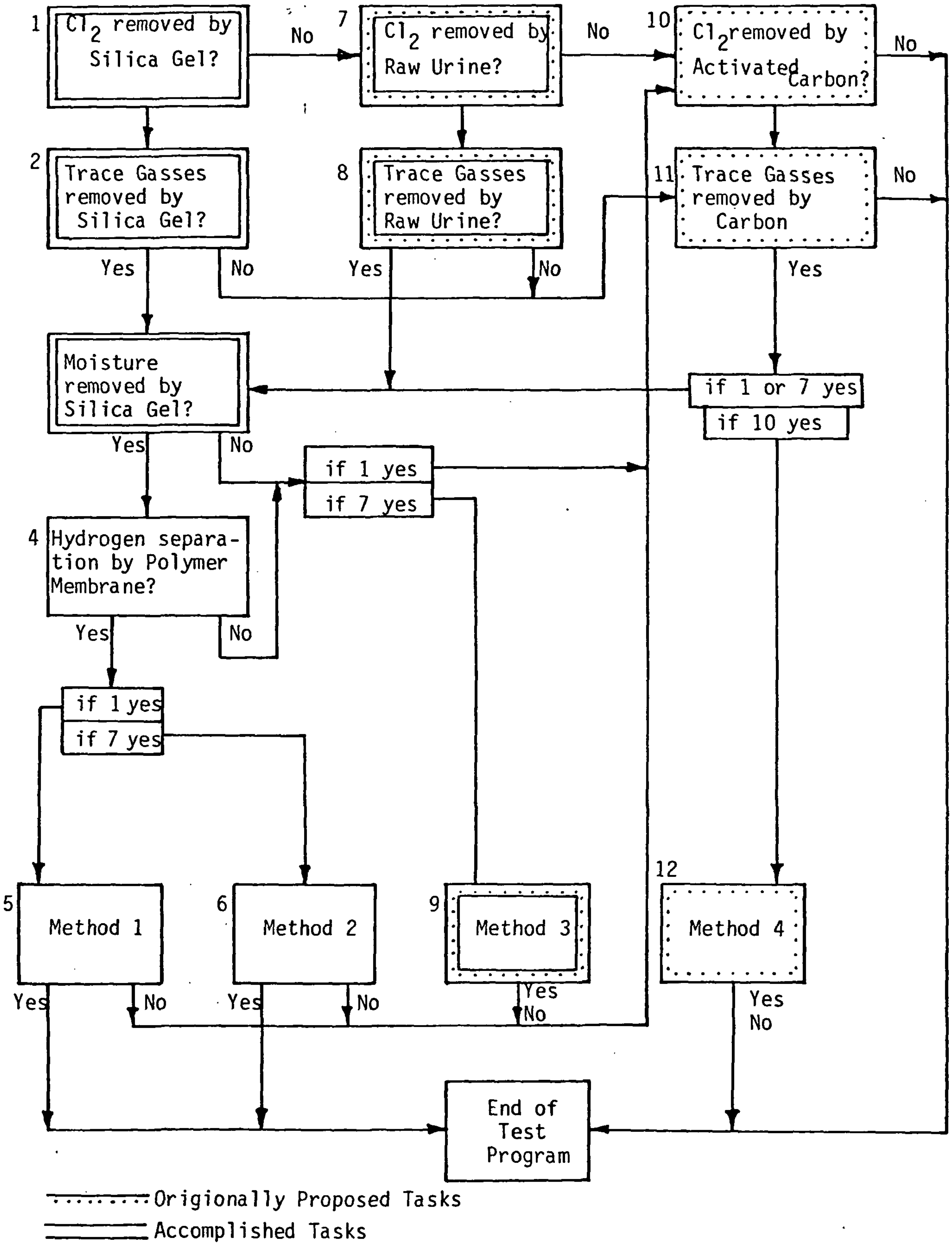


Figure 4-1, Overall Test Plan

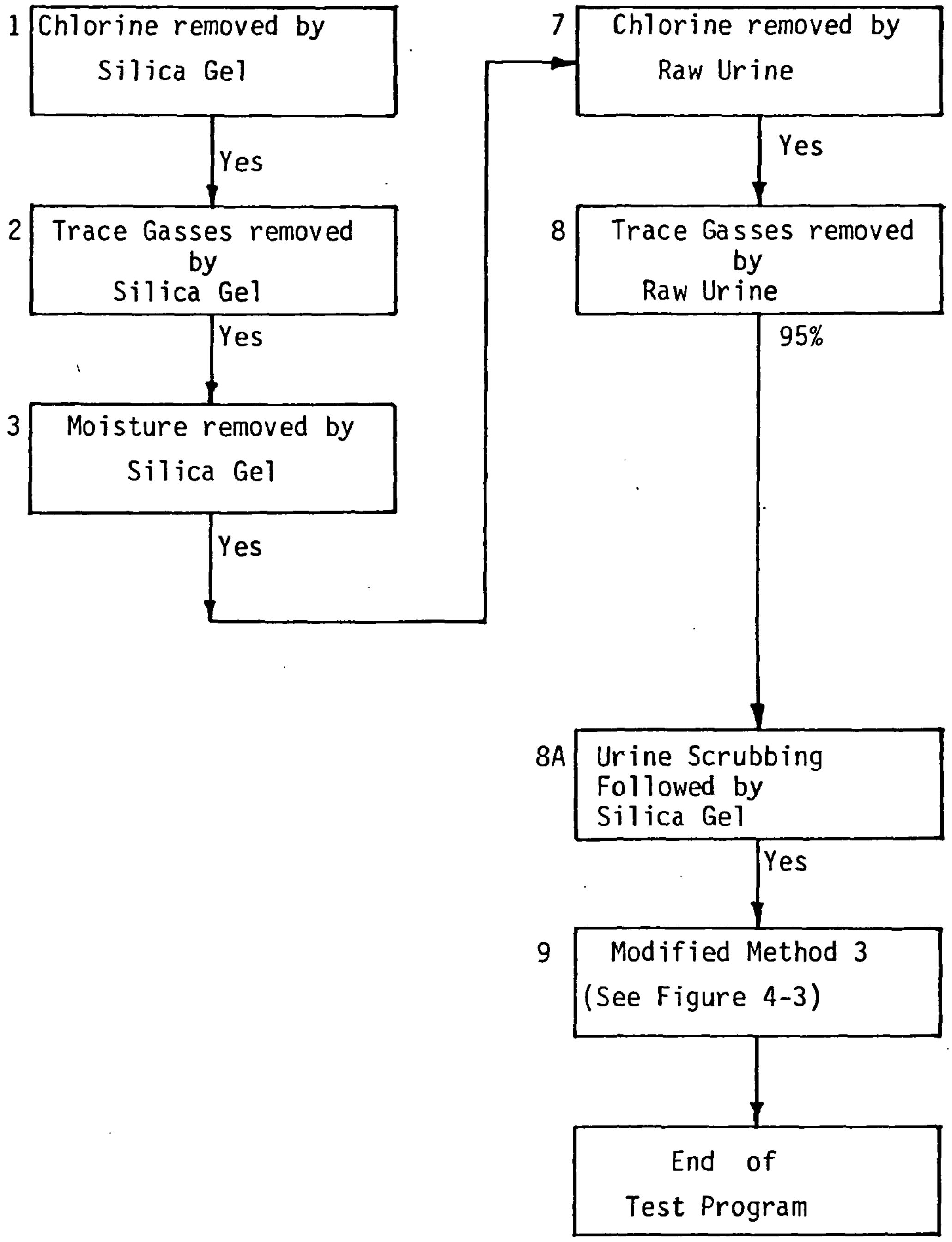


Figure 4-2, Tests Performed

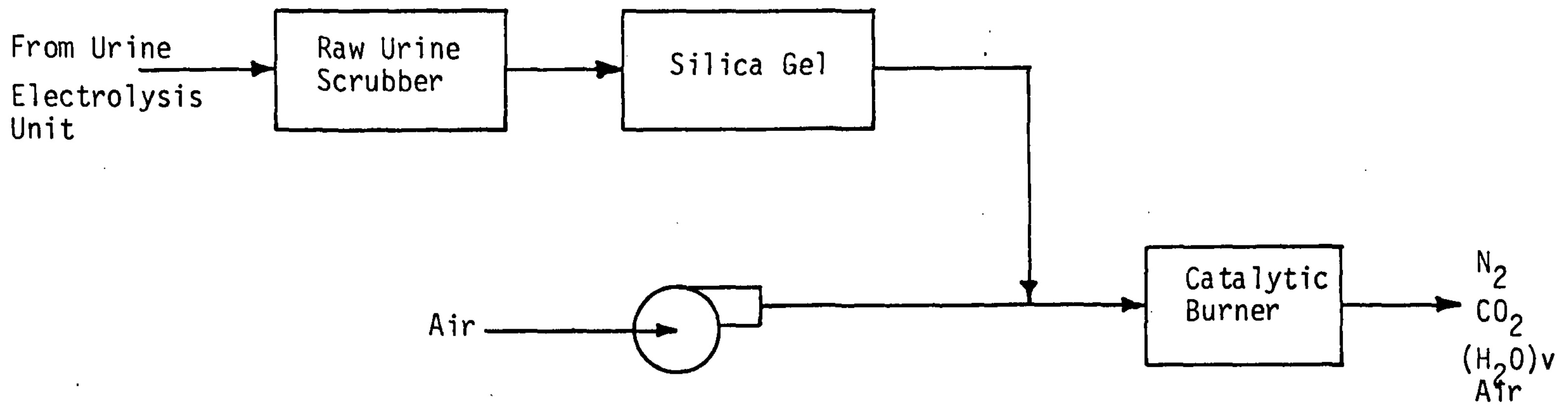


Figure 4-3, Modified Method 3

5.0 LABORATORY EQUIPMENT

5.1 GFE Electrolysis Unit

Some of the key components in the test program were obtained from the GFE Electrolysis Unit. This unit was constructed under the program described in Reference 2 and was furnished to URC by NASA to use in whole or in part for these studies.

Initial inspection of the unit after arrival at URC revealed that only the electrolysis loop was complete with a full complement of components. The electrodialysis loop had many missing components including the cell and bladder tank.

In order to prepare the unit for checkout tests, the urine electrolysis loop and the raw urine storage tank, which contained urine from previous use, were flushed out with tap water (specific conductivity = 155 micromhos per centimeter). Next the electrolysate and raw urine tanks were filled with tap water and the system was activated. (Relay K21 was removed to prevent an automatic transfer to the electrodialysis loop.) Automatic transfer from the raw urine tank to the electrolysate tank occurred and processing started. The system was then shut off and allowed to stand idle for five days. Both the electrolysate tank and the raw urine tank lost a substantial part of their air pressure. The tanks were repressurized with air. The raw urine tank was charged with salt water (NaCl = 20 g/l). The normal automatic transfer occurred and processing started. The electrolysis cell conditions were set at 18V and 16 amps. The circulation rate was too low, so the Fluorocarbon pump was adjusted to its maximum flow setting, after which the system was run for three hours. During this time the automatic gas vent in the electrolysis loop functioned properly. The system was then shut down and drained. The electrolyzed salt water had a chlorine odor. After draining, the system was flushed with tap water.

It was recommended that further tests of the unit, using raw urine, not be undertaken because the three-hour salt water test demonstrated that the electrolysis part of the unit was in good working condition. It was felt that there was nothing more to be gained by running with urine. It was also recommended that the unit not be used in-situ, but instead a bench-top electrolysis loop be built from unit parts and components. This recommendation was based on the following reasons: a) the in-situ unit would be difficult to completely flush out between tests, b) the air leaks in the bladder tanks would be a problem, and c) a bench setup would have much greater flexibility.

5.2 Instruments

The following instruments were used to collect the data during the test program.

5.2.1 Flow Meters

Liquid flows which were essentially constant throughout a test were measured by stopwatch and graduated cylinder. Gas flows were measured with glass rotameters containing glass balls. These meters, Gilmont models F2000, through F2300 cover the range from 0-90 ml/min to 200-12,500 ml/min and are calibrated for air. Flows for other gässes were calculated using the manufacturers instructions and measured gas compositions.

5.2.2 Gas Analysis

Fixed gasses were measured on a Matheson Model 8430 gas chromatograph. A molecular sieve column was used for oxygen, nitrogen and hydrogen, and a Poropak Q column for carbon dioxide and water vapor. Helium carrier was used for most testing , however it enabled hydrogen to be reliably measured only from 25% to 100%. For lower levels of hydrogen, nitrogen carrier was used.

Trace hydrocarbons were measured on a Perkin Elmer Model 3920 gas chromatograph using flame ionization detectors. A didecylphthalate column (non polar) and a Carbowax 1540 (polar) column were used for the identification of some of the compounds after they were originally identified by GC/MS at NASA-JSC.

Samples submitted to NASA-JSC were analyzed using gas chromatography-mass spectrometry for the qualitative analyses and gas chromatography for the quantitative analyses. The GC-MS system consists of a Hewlett Packard 7620 gas chromatograph with a flame ionization detector connected to a Nuclide 12-90-G mass spectrometer. The same Hewlett Packard gas chromatograph was used for the quantitative analyses.

Initially the samples were analyzed on a gas chromatographic column to determine which components were separated by that particular column. Next a gas chromatograph-mass spectrometric analysis was made taking spectra of the separated components for identification. The samples were analyzed again on the same column using only the gas chromatograph for quantitation of the identified components. The quantitation is accomplished by analyzing a standard gas blend of known concentration on the same column and comparing the response to the sample response. Five different columns were used in an attempt to identify as many components as possible. All of the components were separated on an OPN-Porasil C column or a chromosorb 102 column except chloroethylene, chloroacetylene, ketene, and acetylene. These components were separated on a chromosorb 104 column.

The column descriptions are as follows:

OPN-Porasil C	80/100 mesh packed in a 1/8" x 7' stainless steel column
Chromosorb 102	80/100 mesh packed in a 1/8" x 6' stainless steel column
Chromosorb 104	80/100 mesh packed in a 1/8" x 7' stainless steel column

Most of the components of low concentration are reported as less than 5 ppm because standards were not available in the NASA-JSC laboratory for these components.

5.3 Chlorine Measurements

The chlorine levels in the various gas streams required a method that covered the range from less than a part per million to the percent levels. This was accomplished with two techniques, continuous absorption in an indicating liquid, and grab samples with a syringe.

The low level tests were accomplished by passing the gas stream through a fritted glass tube immersed in an orthotolidine solution. The resulting color was then measured spectrophotometrically at 490 mμ vs standards prepared from known chlorine solutions. This method is sensitive to less than 1 microgram of chlorine, and the minimum detection level in the gas stream is solely dependent on the amount of gas sampled.

The higher levels were measured by injecting 0.25 to 20 ml samples of the sample gas (via a glass syringe) into a 60 or 100 ml serum bottle fitted with a rubber septum and containing 20 ml orthotolidine solution. The bottle was then shaken thoroughly and the color of the solution measured.

At intermediate chlorine levels, both the above methods were used with good agreement.

6.0 TEST PROGRAM

The test program was conducted in logical steps, as dictated by the Overall Test Plan and its modifications. A summary of the nine experimental runs, the basic goal of each test, and the results, is presented in Table 6-1. Runs 1 through 6 were conducted to verify the basic gas generation data developed in earlier studies, Reference 2, especially chlorine generation characteristics, and the ability of silica gel to dry the stream and hopefully remove chlorine and trace contaminants. Run 6 was conducted to investigate the performance of a silica gel bed with L/D characteristics different from previous runs, and provide extensive data on the generation of trace gasses by the electrolysis process. Runs 7, 8 and 9 demonstrated the concept of using raw urine to scrub chlorine from the gas stream, starting with a feasibility test in Run 7 and culminating with an integrated system in run 9 which include the combustion of hydrogen, and regeneration of a previously used silica gel bed. This final test configuration contained essentially all the unit processes necessary for an operational flight system.

6.1 Gas Stream Characterization and Silica Gel Evaluation

Runs 1 through 6 were conducted to provide complete data on the gasses produced during the electrolysis of urine, and the behavior of silica gel as a dessicant and gas sorber.

6.1.1 Basic Test Apparatus and Procudure

The basic test set up used during Runs 1 through 6 is shown in Figure 6-1. Prior to the test program, urine was pooled from male lab personnel and immediately chilled to 275K. After 30 liters had been collected, it was pooled and thoroughly mixed. This urine pool was held at 275K and used as the source of all urine during the 9 Runs. Two liters of raw urine, (approximately 1 man day charge) together with five drops of Dow Corning Anitfoam FG 10 were introduced into the 3.78 liter (1 gallon) plastic bottle fitted with a bottom outlet. The urine was pumped from the bottom via a Fluorocarbon Model Sp1000 Teflon diaphragm pump, through a glass condenser to a plastic chamber which housed pH probes connected to a Beckman H-5 meter. The flow then went to the GFE Electrolytic cell, described in Reference 2, which was operated at 10 amps DC (15-18 volts) for all runs. The urine leaving the cell, mixed with the evolved gasses, was then returned to the bottle. The separated gasses were fed to another glass condensor to remove some moisture which collected in a trap. This trap also served as a sample point for gas samples described as Silica Gel Feed. Flow was monitored via an all glass rotameter, and then fed to the Silica

TABLE 6-1 TESTING SUMMARY

Run Number	Test Goal	Test Configuration	Data Presented	
1	Procedure Verification, Silica Gel Evaluation	Fig 6-1	Fixed Gasses Liquid Parameters	Fig. 6-2a Fig. 6-2b
2	Repeat Run 1, First Regeneration of Silica Gel	"	" "	"
3	Repeat Runs 1,2; Second Silica Gel Regeneration	"	" "	"
4	Effect of Maintaining pH above 7 to Minimize Chlorine Evolution	"	Cl_2 Evolution Curve	Fig. 6-3
5	Repeat Runs 1,2,3, Third Silica Gel Regeneration, Basic Hydrocarbon Testing	"	GC/MS Analyses	Table 6-2
6	Repeat Runs 1,2,3,5 with longer Silica Gel Bed, Intensive Hydrocarbon Analyses	"	Silica Gel Chlorine Loadings Chlorine Breakthrough Curve Silica Gel Bed after Run Gas Chromatograms Hydrocarbon Generation Profile GC/MS Data Silica Gel Bed Loadings	Table 6-3 Fig. 6-4 Fig. 6-5 Fig. 6-6 Fig. 6-7 Table 6-3 Table 6-4
7	First Attempt at Urine Scrubbing	Fig 6-8	Chlorine Absorption Chlorine Curves Effect on Urine	Table 6-5 Fig. 6-9 Table 6-6
8	Urine Scrubbing Followed by Silica Gel	"	"	"
9	Integrated System Test	Fig 6-10	" Hydrogen dilution GC/MS Data	" Fig. 6-11 Table 6-7

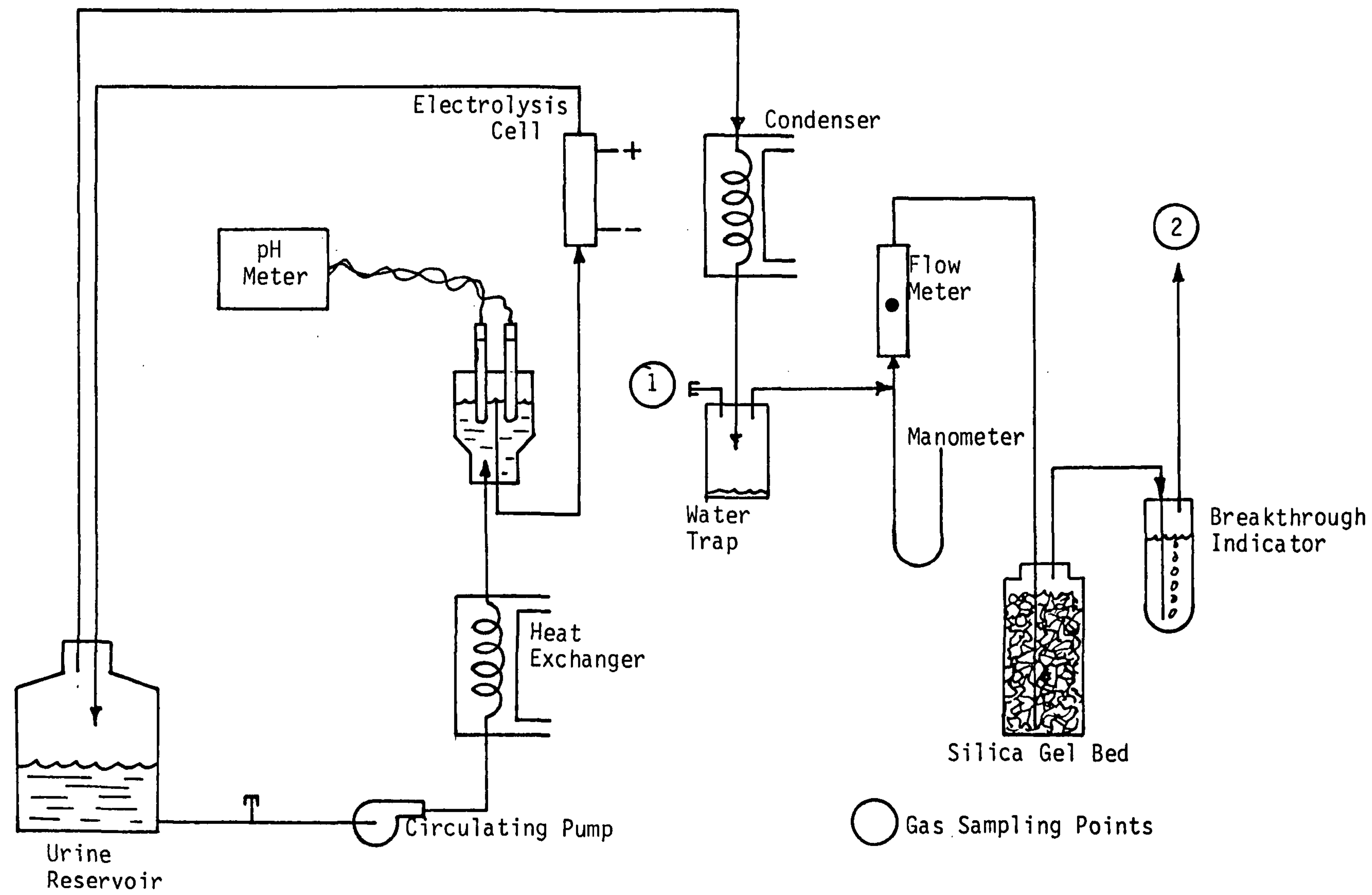


Figure 6-1, Urine Electrolysis Test Schematic (Runs 1-6)

Gel bed as shown in the schematic. Chlorine breakthrough was indicated by an orthotolidine containing bubbler at the exit.

A test was initiated after filling the 1 gallon bottle with urine, charging and connecting the Silica Gel bed, and purging the system with helium. The circulating pump was allowed to attain equilibrium flow, and the test was initiated by energizing the electrolytic cell. Baker Catalog No. 3401 indicating Silica Gel, 6-16 mesh was used throughout the test program.

6.1.1 Test Results Runs 1 through 6

The basic composition of the gas stream resulting from the electrolysis of urine is shown in Figure 6-2a. These data are a composite gathered during Runs 1,2,3,5 and 6. Run 4 was not included since the basic electrolysis process was being modified by pH adjustment. The liquid parameters are shown in Figure 6-2b. The data in these two figures are essentially identical to those presented in Reference 2, however this is the first reporting of the chlorine evolution curve. The chlorine curve actually includes data from Runs 1,2,3,5,6, and 7 which were all the runs in which raw urine was electrolyzed. The quantity of chlorine generated during this process is in the neighborhood of 1.5 grams (for 2 liters of urine). The reported flow rate represents the rotameter readings corrected for density based on the gas analyses. The reading of a rotameter is directly proportional to the $\sqrt{\text{density}}$, thus a stream with changing amounts of hydrogen and carbon dioxide in addition to oxygen and nitrogen is very difficult to measure accurately, however a comparison with later data based on hydrogen dilutions shows the reported flows to be within 10-15%.

The coincidence of the pH dip with the chlorine peak lead to Run 4 in which an attempt to maintain the pH of the urine was made. Solutions of NaOH were injected at the liquid sample port at the points indicated on Figure 6-3. Although the pH got out of hand briefly, the effect of controlling the pH on the amount of chlorine evolved is dramatically evident. The total amount of NaOH added was less than 4 grams. At that point in the test program, this amount of expendable material appeared to merit consideration as a viable approach to minimizing the chlorine removal problem. Although the fixed gas and liquid parameter data from Run 4 were not included in preparing Figure 6-2a and 6-2b, the results fell within the scatter of the data, and no significant changes attributable to controlling the pH were evident.

A breakthrough curve for chlorine with silica gel is shown in Figure 6-4. Although the log scale distorts the essentially Gaussian shapes of the peaks, its use provides a better idea of the very low actual levels of the fronts and

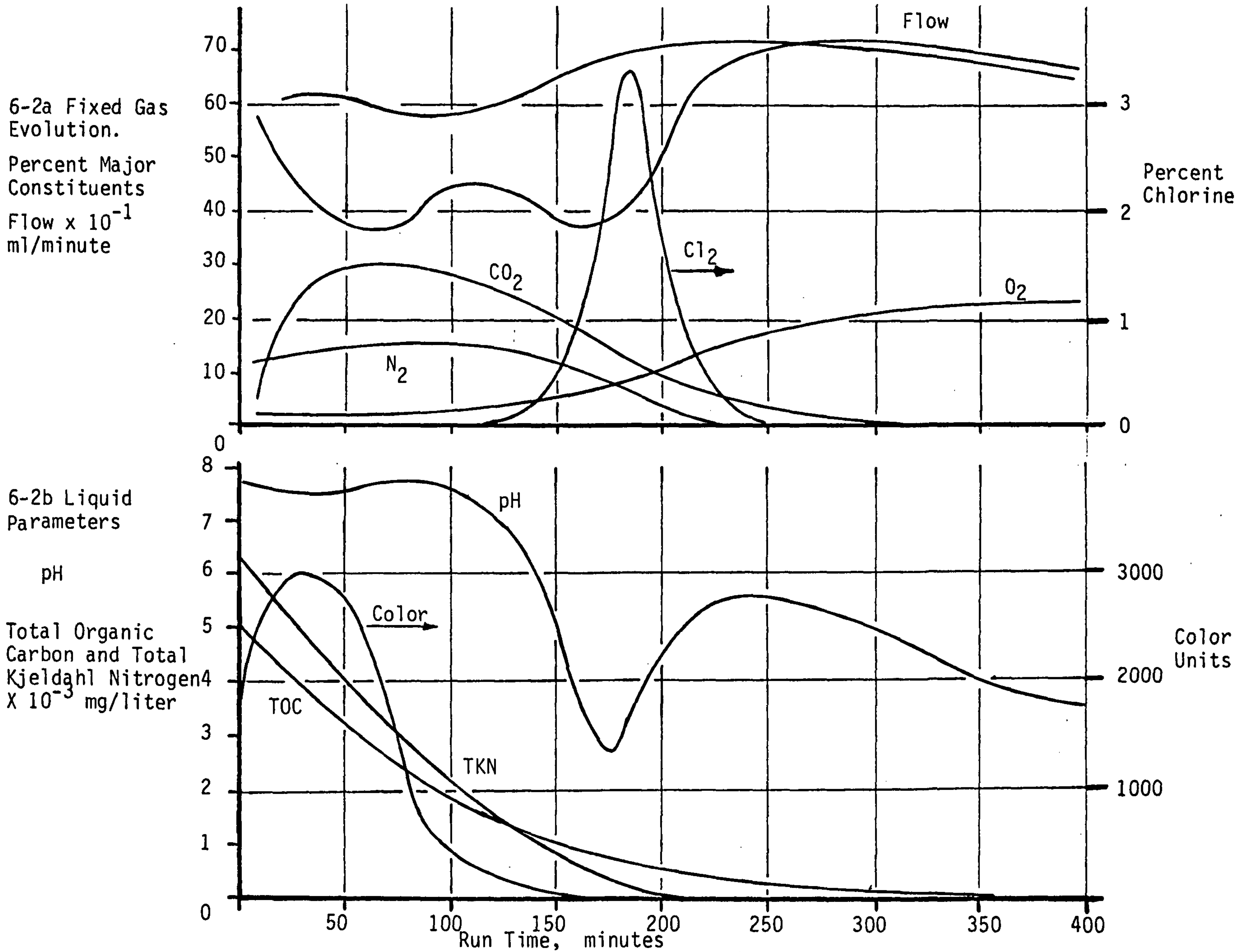


Figure 6-2, Accumulated Data From Runs 1,2,3,5,6.

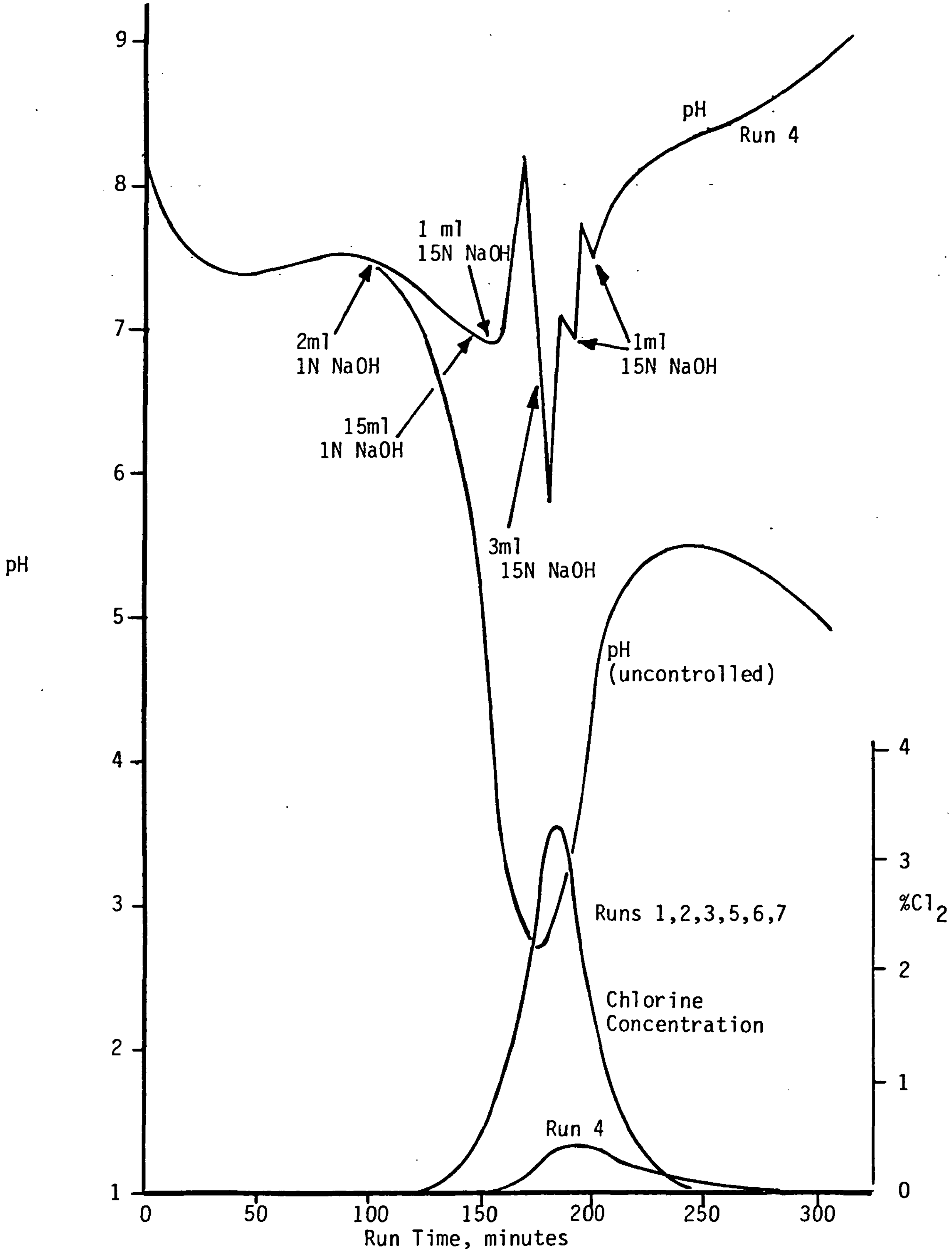


Figure 6-3, Chlorine Evolution With pH Controlled (Run 4)

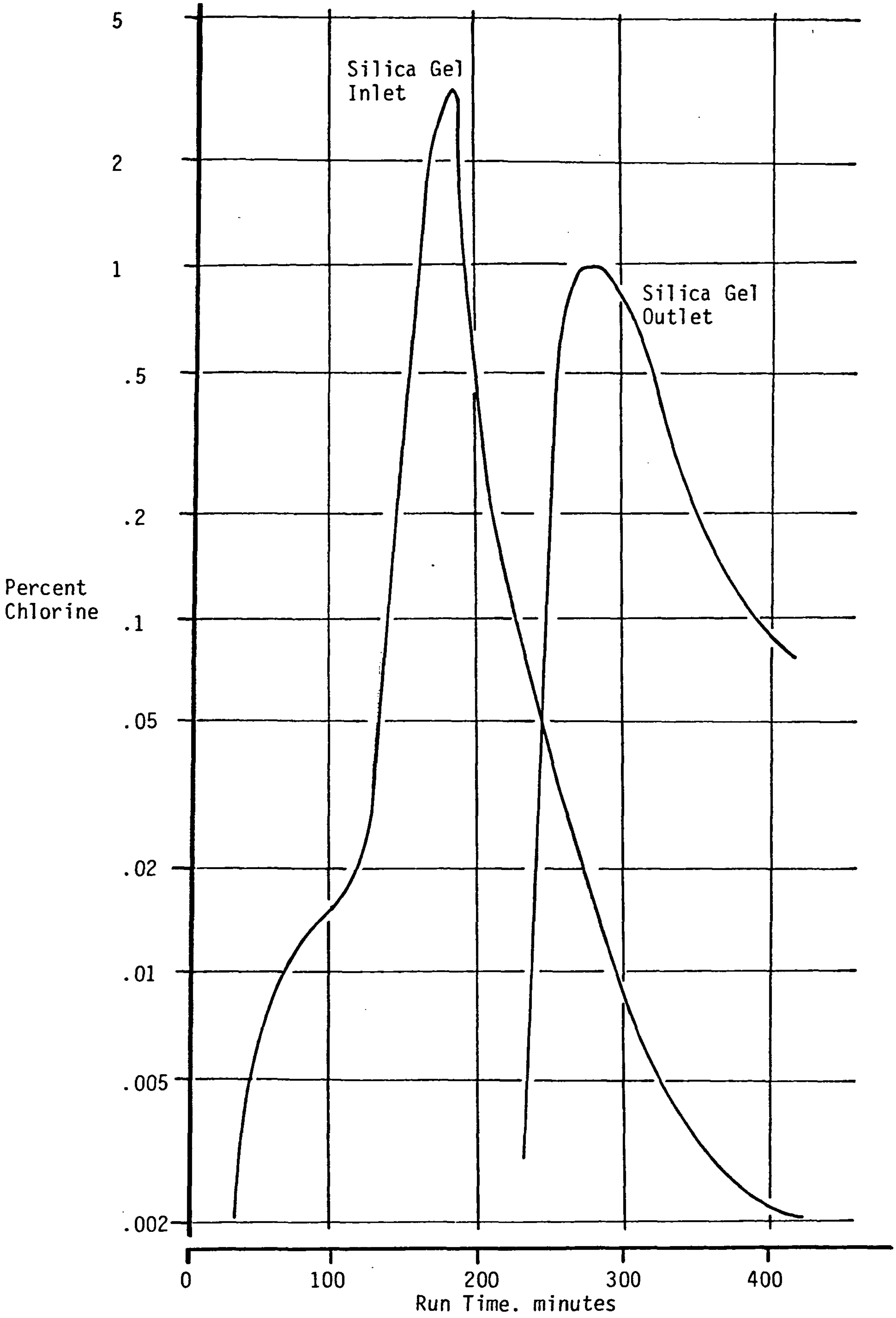


Figure 6-4, Chlorine Breakthrough From Silica Gel tails. The front of the breakthrough peak is even sharper than is evident in Figure 6-4. The breakthrough-indicating bubbler accumulates chlorine from the gas stream (the total exhaust from the bed) until enough color appears to be recognized. However, during the run the change is almost instantaneous when it occurs. Before breakthrough, the scrubbing liquid was periodically measured for optical density to see if any chlorine was accumulating. No color change had been measured up to the time of breakthrough as indicated by an obvious color change. The amount of chlorine necessary to produce a visually recognizable color in the 20 ml scrubber is approximately one microgram. Since breakthrough normally occurs near 210 minutes, at nominal flow of 400 ml/minute the integrated average chlorine concentration of the silica gel exhaust is approximately 4 x 10^{-6} parts per million; and even less for detection spectrophotometrically.

It is apparent that the chlorine moves through the bed in a manner similar to a peak through a gas chromatograph column. Thus, with proper bed design the chlorine may be trapped in a bed, and easily regenerated with minimum energy required. Successful regeneration was accomplished at 448 K (350°F) with a purge of dry nitrogen. Silica gel manufactures recommend regeneration at 623 K (675° F), however regeneration appeared satisfactory as evidenced by the indicator color at the lower temperature. A Silica Gel bed was regenerated three times with no apparent loss of performance.

A sketch of the Silica Gel bed after Run 3 is shown in Figure 6-5. Initially, all of the Silica Gel was blue. The blue color is due to the cobalt chloride indicator, which changes color to white when wet, thereby indicating the presence of H_2O. The black and green bands are probably caused by one or more of the absorbed hydrocarbons. After thermal regeneration at 350°F the bed returns to its normal blue color.

Previous studies, Reference 2, as shown in Table 3-2 found a number of trace organics were produced during the electrolysis process. This was initially investigated using gas chromatography the results of which are shown in Figure 6-6. The only peak initially identified was that of chloroform, occuring at 16 mm. Figure 6-6 also shows that the Silica Gel was quite effective in removing the majority of the hydrocarbons; even the early peaks were reduced by a factor of eight or more.

During Run 5, gas samples were taken in evacuated stainless steel sample bottles provided by NASA and returned to JSC for analysis by Gas Chromatography/Mass Spectrometry. The results of these analyses are summarized in Table 6-2. The majority of the compounds are chlorinated light hydrocarbons,

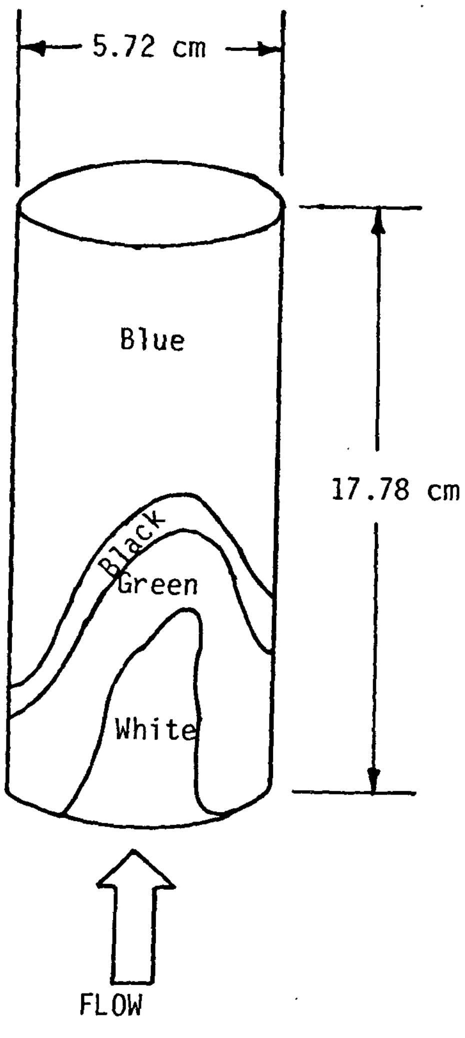


Figure 6-5, Silica Gel Bed After Run 3

TABLE 6-2 GC-MS ANALYSES OF GAS STREAMS FROM RUN #5*

COMPOUND	Feed to Silica Gel, Test Point 1, Figure 6-1 Test time 60 min. parts per million	Exhaust from Silica Gel Test Point 2, Figure 6-1 Test time 208 min. parts per million	Feed to Silica Gel, Test Point 1, Figure 6-1 Test time 220 min. parts per million
Methyl Chloride	230	<5	3000
Chloroethylene	<5		<5
Methyl Bromide	<5		<5
Freon 114	<5		<5
Chloroethane	15		80
1,1, Dichloroethene	<5		<5
Dichloromethane	16		300
Carbon Tetrachloride	<5		<5
Chloroform	185		900
Dichloroethane	225		200
1,1, Dichloropropane	<5		5
1,1,2 Trichloroethane	<5		20
Nitromethane	20		10
Trichloroethylene	<5		<5
Dichloroacetylene	<5		<5
Chloroacetylene	<5	<5	
Acetonitrile	5		15
Acetone	<5		<5
Methanol	20		25
Propylene	2	<1	<1
Butene	<5		
Propanal	<5		
Ethanol	7		
Acetylene	20	7	<1
Ketene	>1	<1	<1
1,1,1 Trichloroethane			<5

*from NASA-JSC

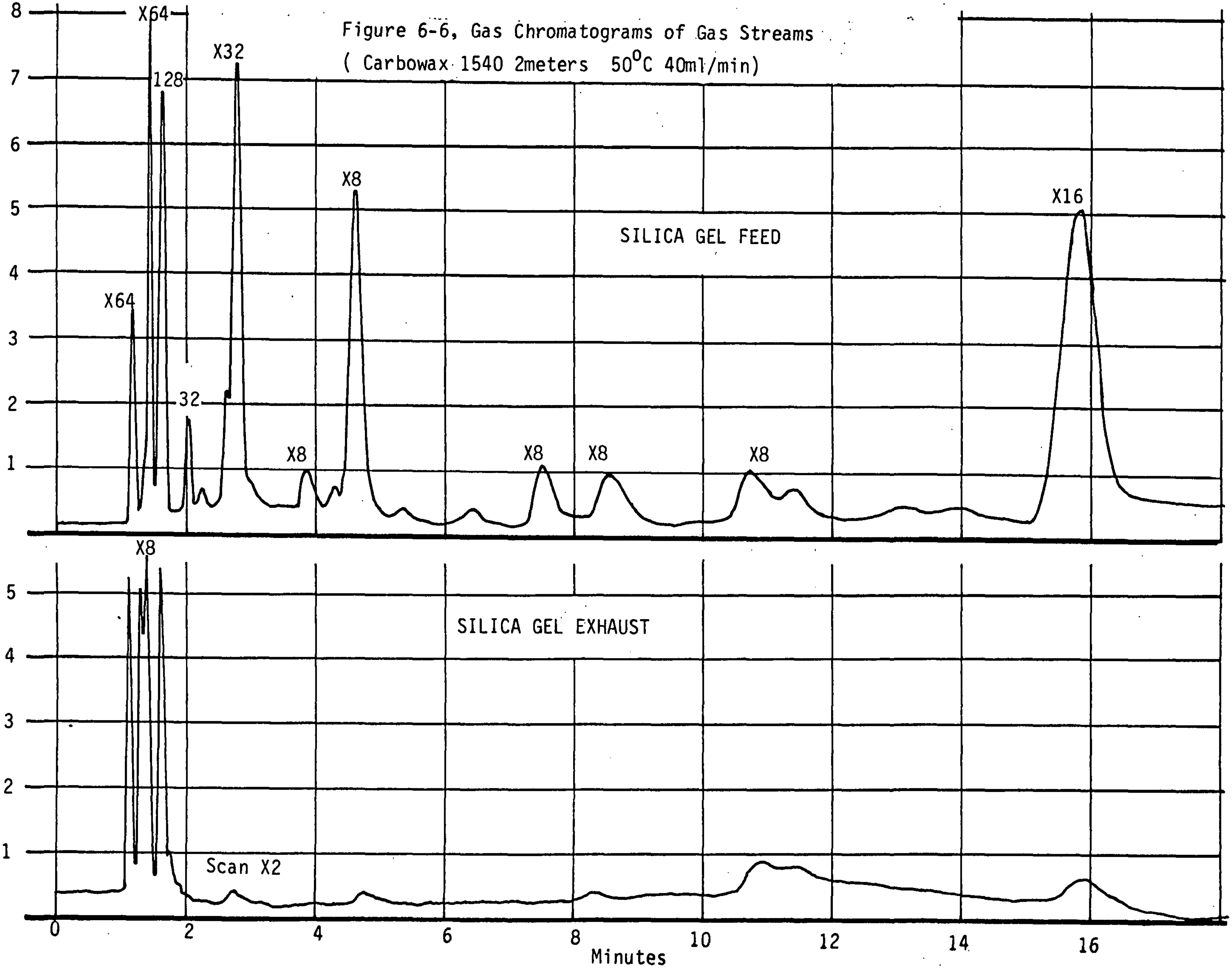


Figure 6-6, Gas Chromatograms of Gas Streams
(Carbowax 1540 2meters $50^{\circ}C$ 40ml/min)

apparently the result of the incomplete oxidation of the organic constituents of the urine by the chlorine generated in the electrolysis cell. The effectivity of Silica Gel in removing most of the compounds is again quite obvious.

As a result of the NASA data, the gas chromatograph was calibrated for the four major chlorinated hydrocarbons, methyl chloride, dichloromethane, dichloroethane, and chloroform in addition to some light hydrocarbons methane through hexane. During Run 6, frequent gas chromatographic analyses were performed to provide hydrocarbon generation profiles. These data are shown in Figure 6-7. Attention should be directed to the methyl chloride peak which is shown reduced by a factor of 10 to permit inclusion on the same figure.

The chlorinated hydrocarbon peaks appear to closely follow the chlorine peak while the light hydrocarbons (mostly unsaturated) appear to decrease. This may be explained by the strong susceptibility of unsaturated hydrocarbons to halogenation, ie the light hydrocarbons are being converted to methyl chloride etc. Several samples were taken during the run and sent to JSC for MS/GC analysis. These points are also shown, and in general substantiate the curves. The dichloromethane data agree extremely well. The complete NASA data are summarized in Table 6-3. The methyl chloride data in the NASA samples are substantially lower, while the more highly chlorinated compounds, chloroform and dichloroethane are higher in the NASA samples. A possible explanation is, in the time between sampling at URC and analysis at JSC (approximately 2 weeks), in the presence of up to 3% chlorine, the methyl chloride may be further chlorinated to dichloroethane and chloroform.

The effect of increasing the length of the Silica Gel bed during Run 6 is compared to the previous Runs in Table 6-4.

6.2 Urine Scrubbing Evaluation

Runs 7, 8, and 9 were conducted to demonstrate and evaluate the use of raw urine to remove the excess chlorine generated during the electrolysis process. Run 7 demonstrated the effectiveness of the processes, Run 8 included Silica Gel to remove the trace chlorine not scrubbed, and Run 9 included all the processes of a complete system to condition and reuse the gasses generated by the electrolytic pretreatment of urine.

6.2.1 Urine Scrubbing Apparatus and Procedure

The apparatus used in Runs 7 and 8 is shown in Figure 6-8. The basic urine electrolysis loop was the same as previously described in Section 6.1.1. The urine scrubbing loop was composed of a 7.56 liter (2 gallon)

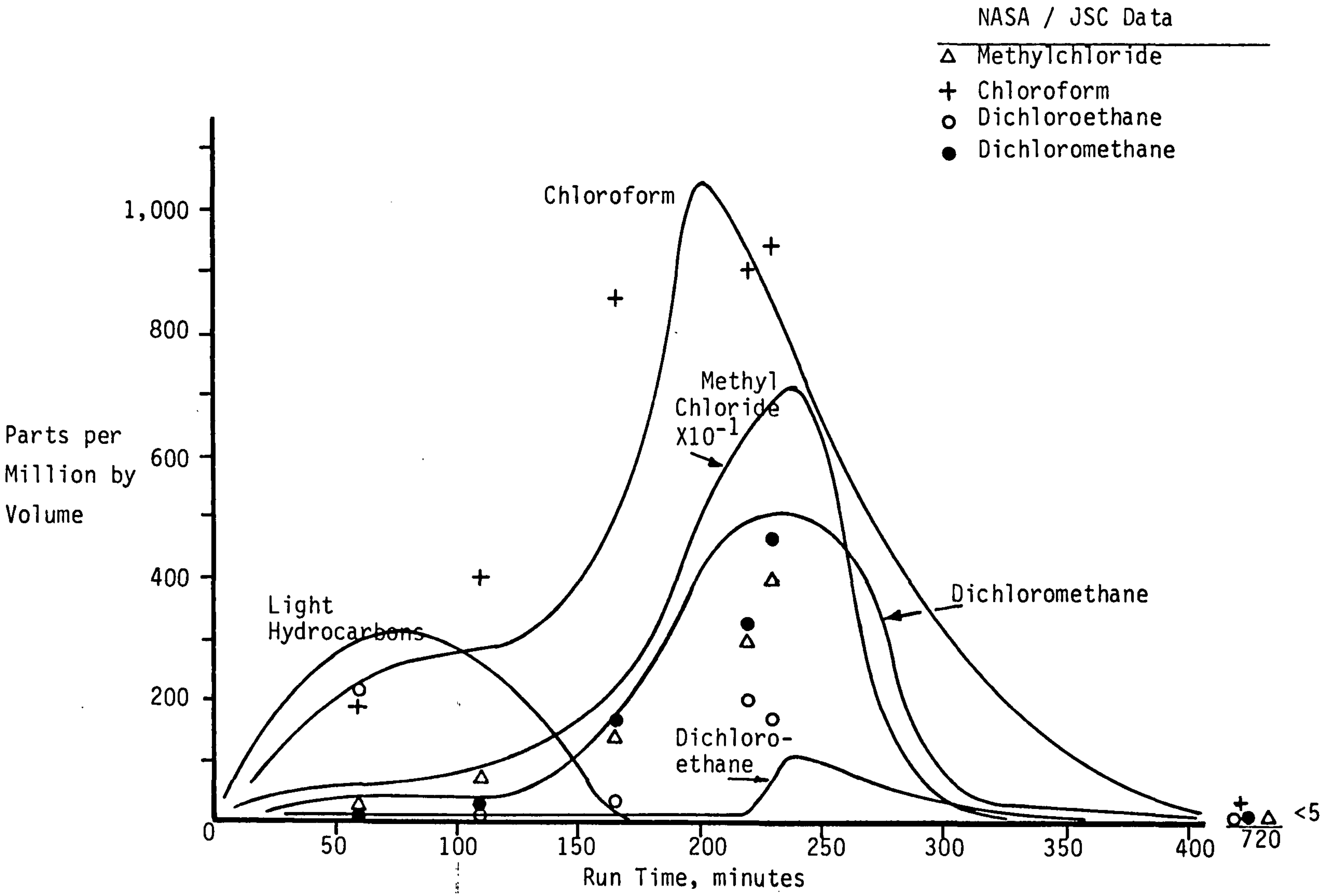


Figure 6-7, Hydrocarbon Generation Profile , Run 6

TABLE 6-3 GC-MASS SPEC ANALYSES OF GAS STREAMS FROM RUN #6*

Test Time	110 minutes		165 minutes		230 minutes		720 minutes	
Test Stream	Feed	Exhaust	Feed	Exhaust	Feed	Exhaust	Feed	Exhaust
Test Point, Fig. 6-1	1	2	1	2	1	2	1	2
COMPOUND	ppm	ppm	ppm	ppm	ppm	ppm	ppm	ppm
Methane	14	14	6	7	3	3	<1	<1
Ethylene	16	54	-	9	<1	-	<1	<1
Acetylene	8	13	1	21	<1	13	-	<1
Ethane	<1	<1	<1	<1	<1	<1	<1	<1
Propylene	<1	-	<1	-	<1	-	<1	-
Propane	<1	<1	<1	<1	<1	<1	<1	<1
Methyl Chloride	790	-	1550	-	4000	-	<5	-
Methyl Bromide	<5	-	<5	-	-	-	-	-
Dichloromethane	35	-	175	-	460	-	<5	-
Chloroform	400	-	860	<1	950	<1	9	<1
Bromodichloromethane	<5	-	<5	-	<5	-	-	-
Ethyl Chloride	60	-	50	-	36	-	-	-
1,1 Dichloroethane	<5	-	19	-	25	-	-	-
1,2 Dichloroethane	8	-	35	-	170	-	<5	-
1,1,2 Trichloroethane	<5	-	11	-	16	-	-	-
1,1,1,2 Tetrachloroethane	-	-	<5	-	-	-	-	-
Chloroethylene	50	-	-	-	5	-	<5	-
1,1 Dichloroethene	45	-	<5	-	<5	-	-	-
1,2 Dichloroethene	6	-	-	-	-	-	-	-
Trichloroethylene	<5	-	-	-	-	-	-	-
Tetrachloroethylene	<5	-	<5	-	-	-	-	-
Freon 114 B_2	<5	-	<5	-	-	-	-	-
Methanol	21	-	-	-	50	-	12	-
Ketene	>5	-	>5	-	-	-	-	-
Methyl Acetate	<5	-	-	-	-	-	-	-
Nitromethane	10	-	-	-	<5	-	-	-
Cyanogin Chloride	<5	-	135	-	-	-	-	-
Methyl Nitrate	<5	-	<5	-	45	-	5	-
Acetonitrile	40	-	-	-	19	-	-	-
Chlorobenzene	-	-	<5	-	-	-	-	-
Bromobenzene	-	-	<5	-	-	-	-	-

*from NASA-JSC

TABLE 6-4 EFFECT OF SILICA GEL BED SIZE ON CHLORINE RETENTION

Run No.	Diam cm	Length cm	Volume cm^3	Initial Bed Weight g	Final Bed Weight g	Increase in Bed Weight g	Time of Cl_2 Peak (inlet) min	Time of Cl_2 Breakthrough (exit) min	Time from Cl_2 Peak to Cl_2 Breakthrough min
1	5.72	15.88	408.04	320.1	325.0	4.9		205	
2	"	"	"	322.5	327.1	4.6	185	210	25
3	"	"	"	321.7	327.4	5.7	155	180	25
5	"	"	"				193	211	18
6	5.08	30.48	617.79	490.6	495.6	5.0	178	228	50

Run No.	Amount of Cl_2 Adsorbed g	Amount of H_2O adsorbed g	Amount of unidentified material adsorbed* g
1	-	1.8	-
2	1.2	1.9	1.5
3	1.4	1.6	2.7
5	-	-	-
6	0	3.7	1.3

*Calculated as: Increased bed weight less Cl_2 and H_2O absorbed

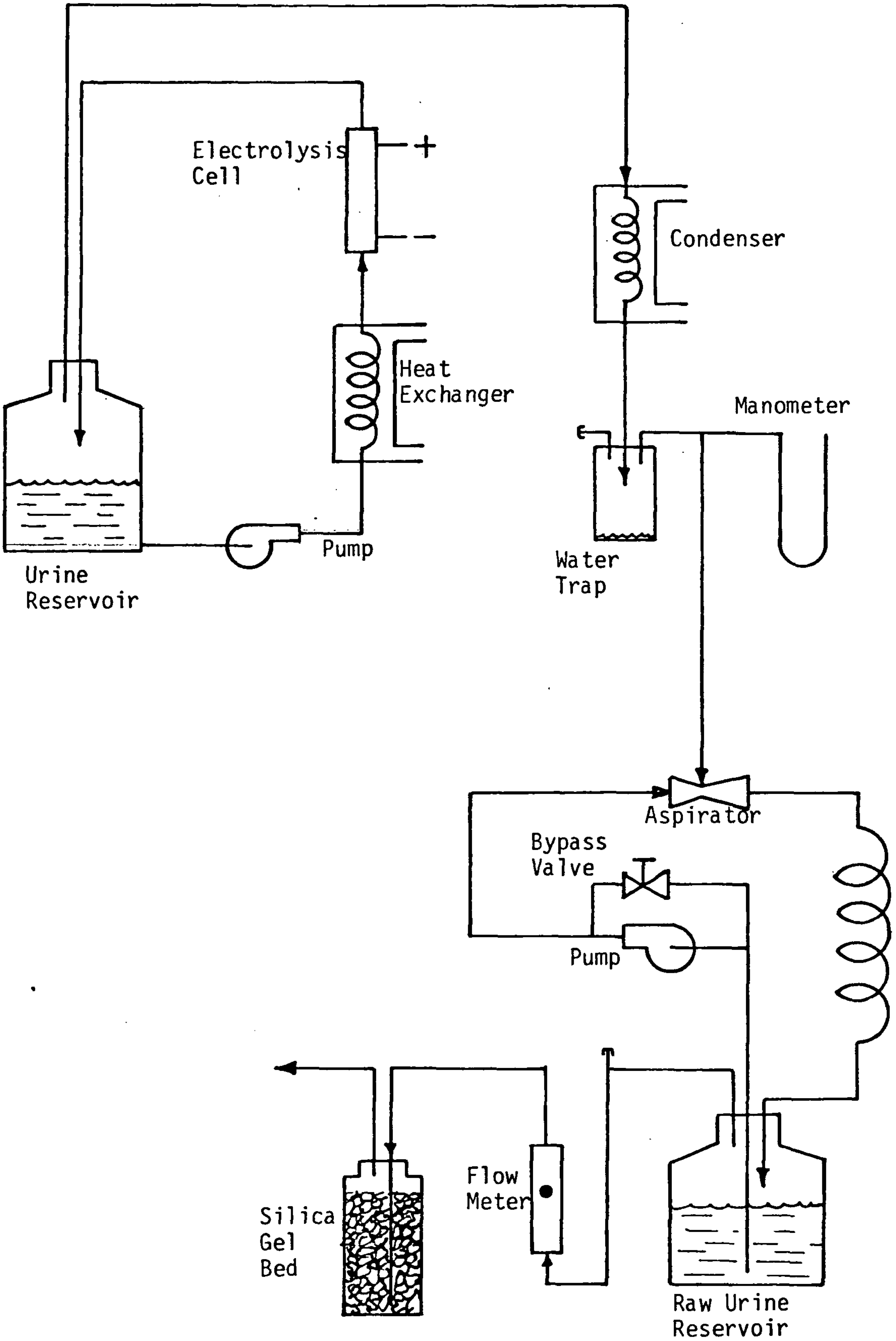


Figure 6-8, Test Schematic, Runs 7-8

plastic bottle with tubes fitted through a rubber stopper in the neck. The raw urine was pumped from the bottom of the bottle using a bronze gear pump. The effluent from the pump passed through a plastic venturi which created a negative pressure at the neck, pulled the gas stream from the electrolysis loop into the system, and created intimate mixing of the gas and liquid streams due to the high shear forces created. Flow in the urine scrubbing loop was maintained in the neighborhood of 3 liters per minute by means of a by pass valve around the pump, since the pump proved to be oversized for the requirements. The suction of the venturi, and consequently the pressure in the urine elctrolysis loop could be controlled to allow for flow changes in the gas generation loop by adjusting the by pass valve. The venturi was a commercially available laboratory aspirator, Nalgene #6140. The mixed stream from the aspirator was then passed through a 7.62 meter (25 ft) coil of 9.5 mm id (3/8 inch) polypropylene tubing, to provide contact time, and back to the 7.56 liter (2 gallon) bottle where the gas was liberated from the liquid. Approximately five drops of antifoam was added to each 2 liter batch of raw urine. The highly surfactant-like properties of urine provided a froth appearing mixture in the coil which provided excellent mass transfer conditions. In the bottle, however, degassing occurred without incident and foaming was not a problem. The gas stream exited from the bottle through the rotameter and was fed to the Silica Gel, etc.

The urine used for scrubbing in Run 7 was electrolyzed during Run 8, with fresh urine used in the scrubber. Likewise, the scrubbing urine from Run 8 was electrolyzed during Run 9.

6.2.2 Test Results, Runs 7 through 9

Figure 6-9 shows the results of chlorine scrubbing by raw urine in Runs 7, 8 and 9. The efficiency is obvious by comparing the chlorine peaks in the streams fed to the urine scrubber, 6-9a, and the streams leaving the scrubber, 6-9b. The scale of Figure 6-9b is expanded by a factor of 100 over that of 6-9a, indicating that the urine scrubber has an efficiency of greater than 99%. This is further illustrated by Table 6-5 which compares the amount of chlorine in the stream calculated by integrating the areas under the chlorine peaks.

The chlorine absorbed by the scrubbing urine is put to beneficial use, oxidizing more organics. Table 6-6 summarizes TOC and TKN data on the urine before and after use as a chlorine scrubber. Another effect is evident in Figure 6-9a. The chlorine peaks for the urine being electrolyzed in Runs 8 and 9 both occurred earlier than in previous Runs, and both had been previously used as a chlorine scrubber. The relative sizes of the peaks from 8 and 9 cannot be

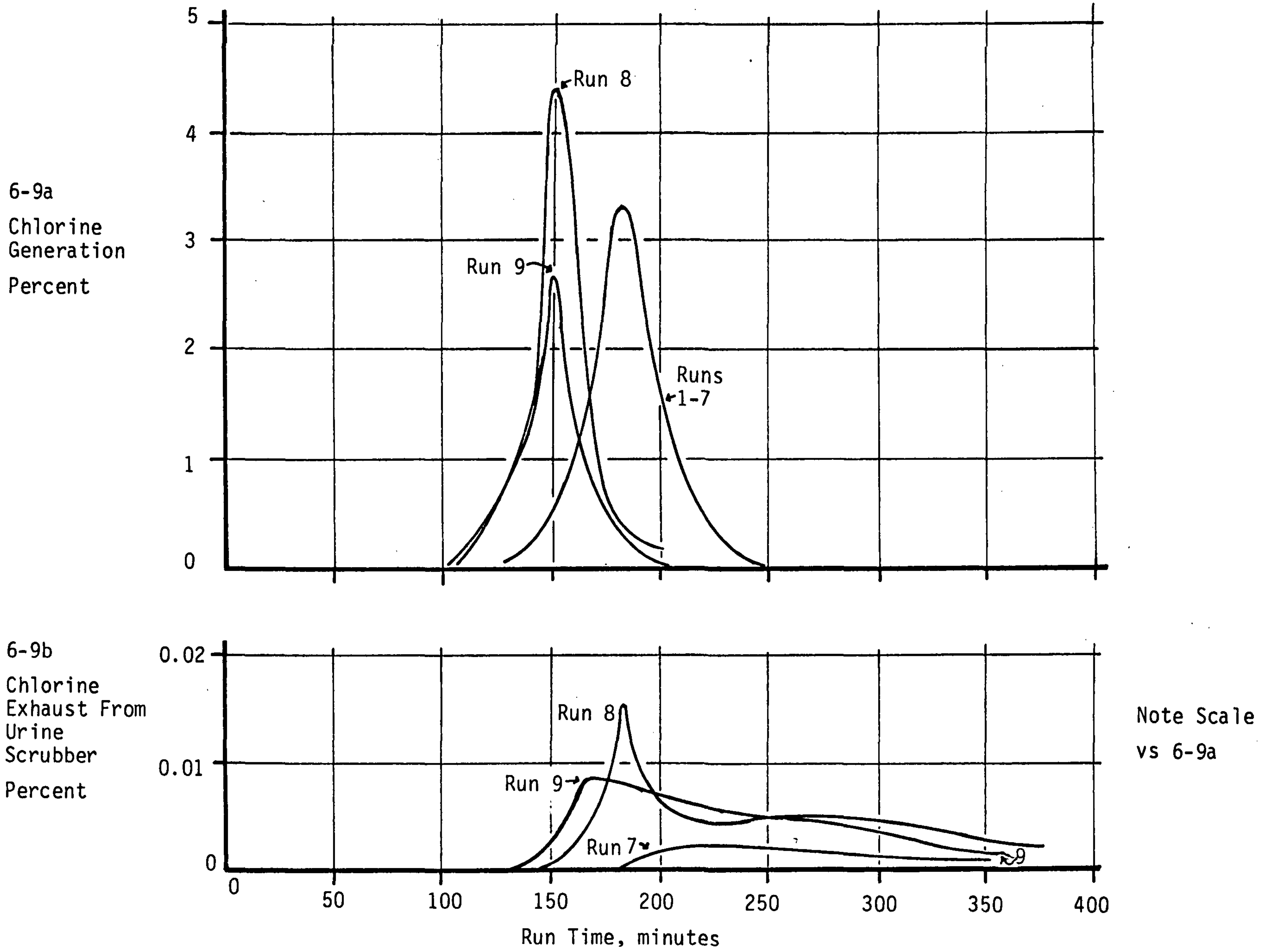


Figure 6-9, Effect of Raw Urine Scrubbing

TABLE 6-5 CHLORINE ABSORPTION BY URINE SCRUBBER

Run Number	Stream	gm Cl_2	% Cl_2 Removed
7	Feed to Urine Scrubber	1.14	
7	Urine Scrubber Exhaust	0.0035	99.69
8	Feed to Urine Scrubber	1.582	
8	Urine Scrubber Exhaust	0.0046	99.71
9	Feed to Urine Scrubber	1.291	
9	Urine Scrubber Exhaust	0.0057	99.56

TABLE 6-6 EFFECT OF CHLORINE SCRUBBING ON RAW URINE

Test Number	Total Organic Carbon		TKN	
	Initial	Final	Initial	Final
7	5000	4500	6268	5711
8	5500	4940	6880	6708
9	6000	4890	7500	7060
Average % Reduction	13		6	

explained at this time, although the urine that generated peak 8 was used to scrub chlorine generated by a batch of raw urine, while the urine from 9 was used to scrub chlorine from the partially oxidized urine from Run 8.

Run 9 was conducted using the apparatus shown in Figure 6-10. The urine electrolysis loop and urine scrubber were as described above. The effluent from the urine scrubber was passed through the Silica Gel bed and then diluted with a 12 liter/minute stream of laboratory compressed air to dilute the hydrogen below the lower explosive limit. This mixed stream was then fed to a catalytic burner, Matheson Model No. OR-100, in which the hydrogen was burned to water. During this dilution process the hydrogen concentration was reduced from the 50% to 60% level of the electrolysis exhaust to between 1 and 3% as shown in Figure 6-11. These data were used to back calculate the volume flows from the electrolysis loop as previously mentioned.

Gas samples were taken and sent to NASA-JSC for analysis. These results are presented in Table 6-7. This is the first hydrocarbon data on the stream from the urine scrubber. The only significant difference as compared to the Silica Gel feed at 165 min shown in Table 6-4 is the absence of the cyanogen chloride which was reported in Run 6 without urine scrubbing.

The most important result of this test is the analysis of the catalytic burner exhaust. The levels of the few compounds were reported as less than 5 ppm, which the lab reports when their presence is indicated but is too low to quantify. The conclusion is, however, that the system produced a gas stream of a quality suitable for return to a space cabin.

During the initial period of Run 9, before the chlorine peaks, the Silica Gel bed used during Run 8 was regenerated using the arrangement shown in Figure 6-10. The bottle was maintained at 448 K (350^{0}F) while being purged with dry nitrogen. This apparently had no effect on the chlorine scrubbing process. The hydrocarbons liberated during the regeneration were not scrubbed by the urine. In a "no-dump" spacecraft, carbon or other type of sorbent for the trace chlorinated hydrocarbons would have to be incorporated to prevent their buildup. Data reported in Reference 5 indicate that a Hopcalite toxin burner is immune to poisoning by halogenated hydrocarbons. This may be a viable solution to this potential problem. Additionally, a carbon bed should also be used in front of a platinum or palladium containing catalytic burner to prevent any traces of methyl chloride and other halogenated hydrocarbons that escapte the Silica Gel

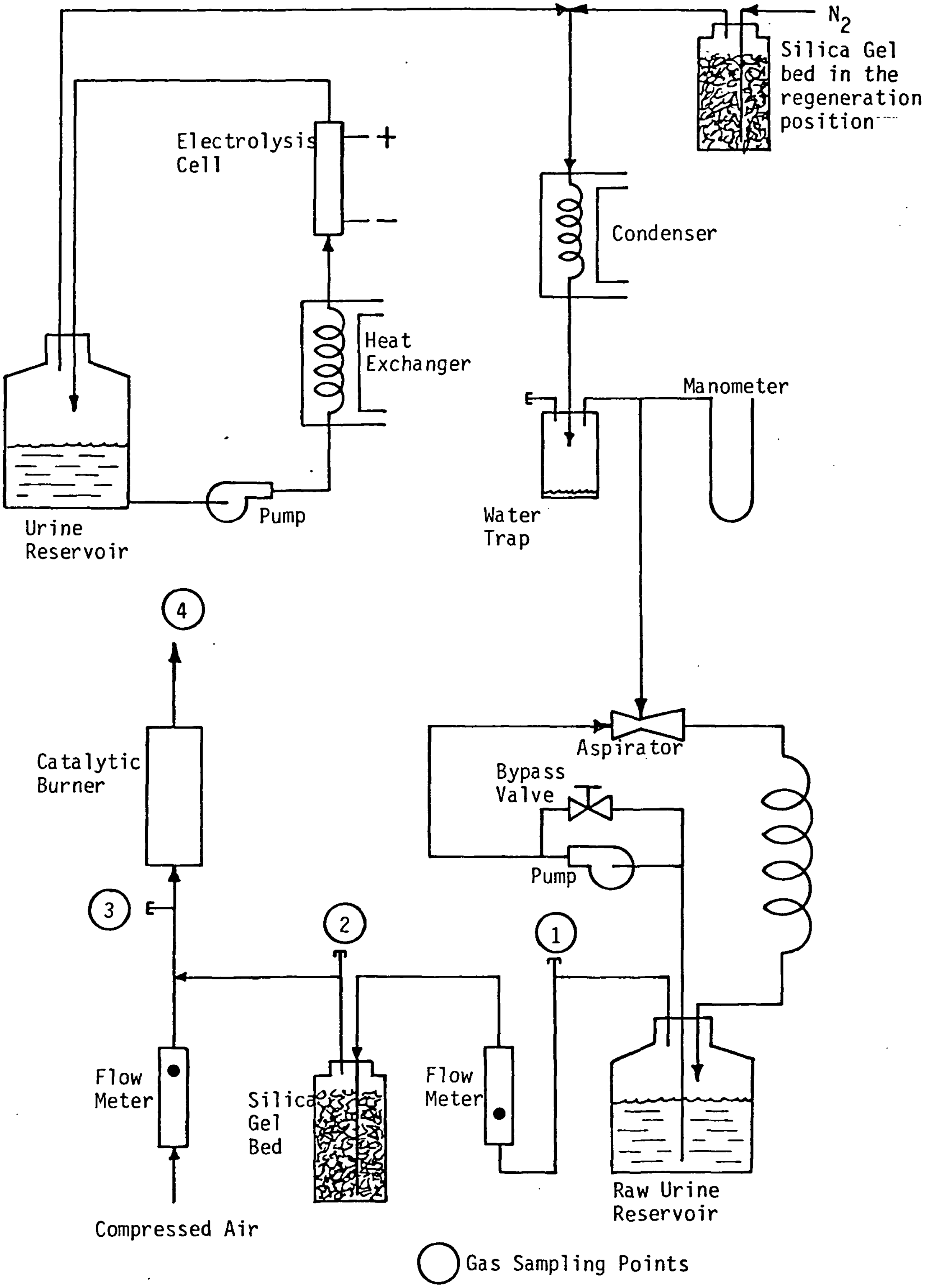


Figure 6-10, Integrated System Test Schematic, Run 9

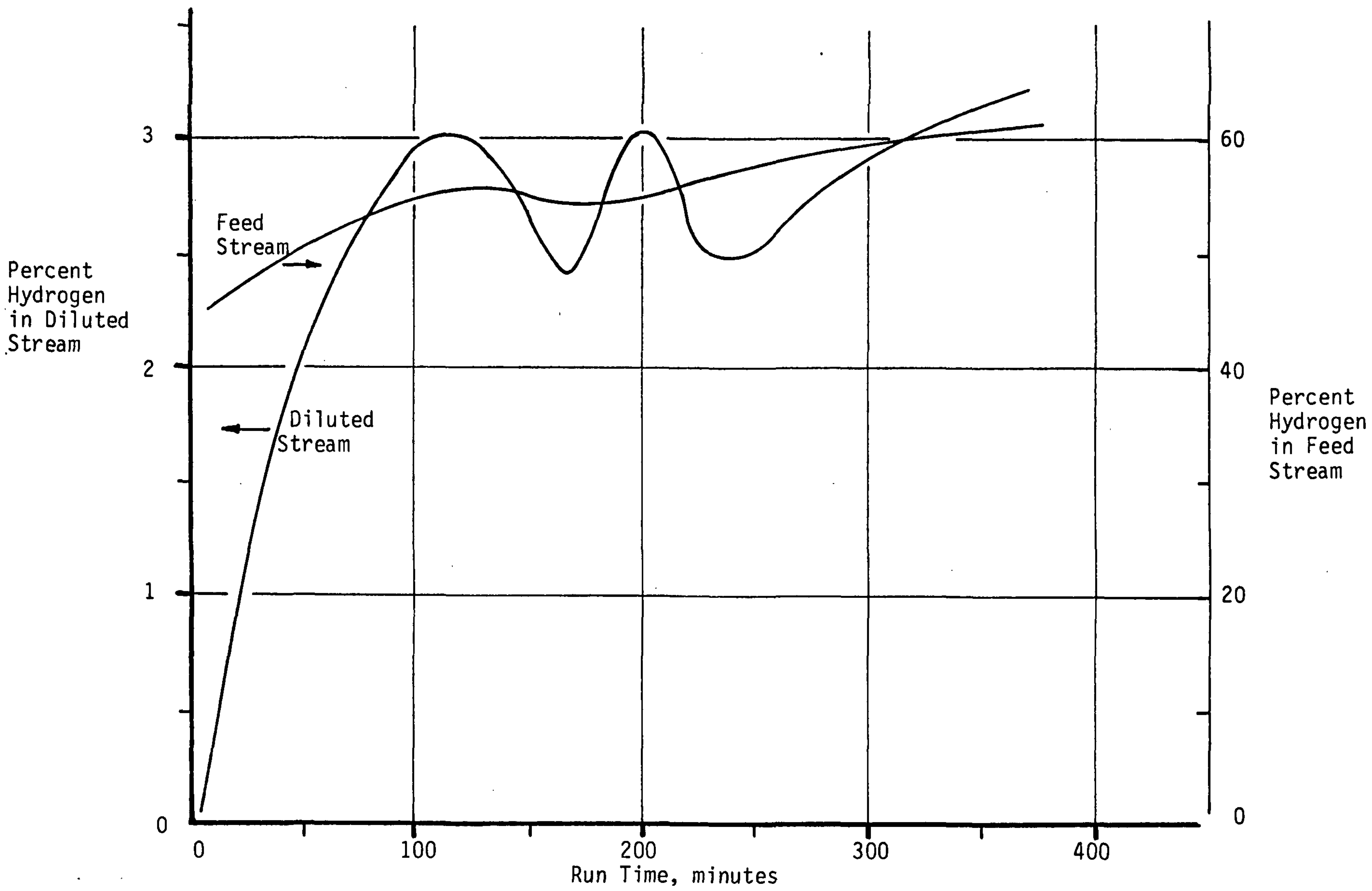


Figure 6-11, Hydrogen Levels After Dilution

TABLE 6-7 GC/MS ANALYSES OF INTEGRATED SYSTEM TEST (RUN 9)

Test Time	125 min	130 min	170 min	325 min	325 min	350 min
Test Stream	Silica Gel Exhaust	Catalytic Burner Exhaust	Urine Scrubber Exhaust	Silica Gel Exhaust	Catalytic Burner Exhaust	Catalytic Burner Feed
Test Point, Fig. 6-10	2	4	1	2	4	3
COMPOUND	ppm	ppm	ppm	ppm	ppm	ppm
Carbon Dioxide	+	+	+	+	+	+
Nitrous Oxide	-		-	-		
Methane	25	<5	16	<5	<5	<5
Ethylene	7		<5	<5		
Acetylene	10		<5	<5		
Propylene			<5	<5		
Ketene			33	10		<5
Methyl Chloride	<5		1200	63	<5	<5
Methyl Bromide			<5			
Dichloromethane			250	<5		<5
Chloroform		<5	900		<5	<5
Bromodichloromethane			<5			
Ethyl Chloride			94			
1,1, Dichloroethane			15			
1,2, Dichloroethane			53			
1,1,2, Trichloroethane			<5		<5	
1,1,1,2,Tetrachloroethane			<5			
Chloroethylene			<5			
1,1, Dichloroethene			25			
Trichloroethylene			<5			
Tetrachloroethylene			<5			
Chloroacetylene			<5	<5		
Dichloroacetylene			6			
1-Chloro-1-Propene			<5			
3-Chloro-1-Propyne			<5			
1,2, Dichloropropane			<5			
Nitromethane			<5			
Cyanogin Chloride			<5			
Methyl Nitrate			<5			
Acetonitrile			17			
Carbon Tetrachloride			<5			
Chlorobenzene			<5			
Ethanol			12			
Acetaldehyde			80			
Acetone			<5			
Toluene			<5			
1,1,1 Trichloroethane	<5					

bed from poisoning the catalyst. Again, a Hopcalite burner may solve this problem.

If overboard dumping is allowed, then the Silica Gel could be vacuum desorbed to space, possibly via the toilet to take advantage of the sterilizing capability of the desorbate gasses.

A note of caution should be added based on an experience encountered while shutting down Run 9. The dilution air stream was inadvertantly stopped too early, allowing the stream feeding the catalytic burner to exceed the lower explosive limit, and ignition occurred. The system was designed with this possibility in mind, (the extensive use of plastic, rubber stopper connections, etc.), and the only result was several of the stoppered joints came apart. A flight system must be designed to minimize the accumulation of explosive atmospheres, and fail safe instrumentation to prevent the occurence of ignition sources.

7.0 SYSTEM DESIGN AND TRADEOFFS

Based on the test results reported in Section 6, the following conclusions may be drawn concerning the tradeoffs presented in Section 3, and new process concepts suggested.

7.1 Methods 1 Through 4 - Section 3, Figure 3-1

Method 1 - Although this method is potentially the most attractive, it must be rejected from consideration since no viable approach has been identified to dispose of the chlorine produced during Silica Gel regeneration.

Method 2 - This method remains essentially unchanged. The first two parts have been experimentally verified, but the hydrogen separation remains untried.

Method 3 - This method has been experimentally verified with slight modification by the addition of a regenerable Silica Gel bed.

Method 4 - This method remains untried. No provision has been included for expendable carbon, it assumes use of previously expended beds from more critical areas.

7.2 Modified Methods 1 - 4

The apparent success in Run 4 in reducing the chlorine effluent by controlling the pH leads to a possible modification of the previously identified methods.

This modification should increase the weight penalty of each method by approximately 2 Kg fixed weight for a pH controller, and an additional 24 gm/day expendable penalty for the required NaOH. This penalty, however is roughly equivalent to a chemical urine pretreatment system which the electrochemical pretreatment system is proposed to replace.

Modified Method 1 - The Silica Gel bed would be reduced but the chlorine disposal problem remains.

Modified Method 2 - Very little benefit, possibly smaller Silica Gel bed.

Modified Method 3 - Possibly would make this method more reliable by reducing the load on the urine scrubber and Silica Gel.

Modified Method 4 - This method would benefit the most by pH control since it would greatly reduce the load on the carbon bed.

7.3 Suggested System Design

The most attractive system design at this time appears to be one based on Method 2. This system is shown in Figure 7-1.

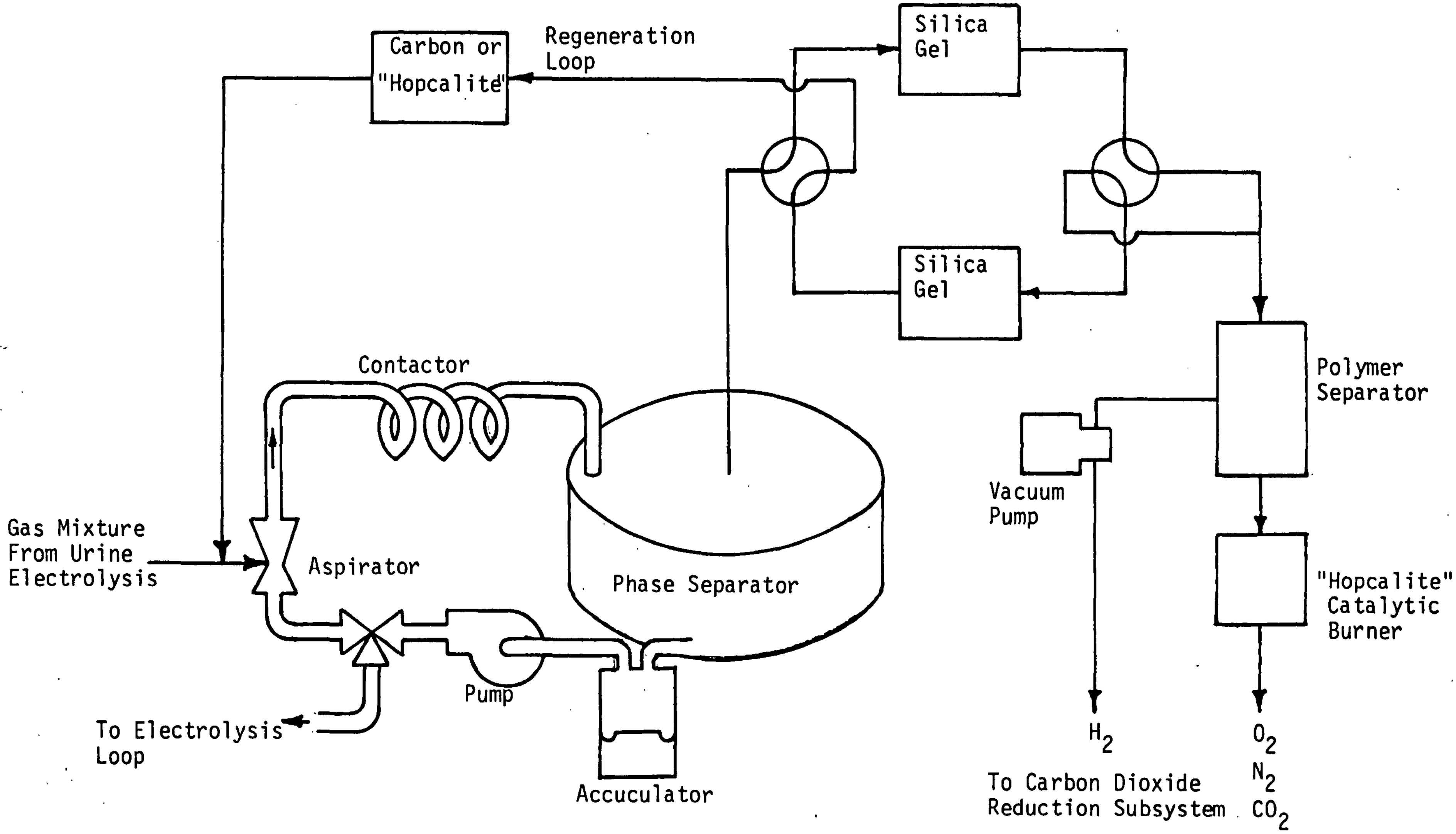


Figure 7-1, Proposed System Design

The gas mixture from the urine electrolysis unit is fed to a venturi through which raw urine is being circulated by a pump connected to a phase separator. The gas stream from the phase separator, with 99% of the chlorine removed, is fed to one Silica Gel bed which is maintained at room temperature where the residual chlorine, water vapor, and trace contaminants are removed. The gas mixture of dry hydrogen oxygen, nitrogen, and carbon dioxide then goes to the polymer separator where the hydrogen is removed by diffusion through the polymer membrane under the pressure differential produced by a vacuum pump. The hydrogen is then sent to the carbon dioxide reduction subsystem. Further treatment of this stream is probably not required since O_2, N_2 and CO_2 impurities will probably not impact the subsystem.

The remaining stream from the polymer separator containing oxygen, nitrogen, and carbon dioxide would best be sent to the carbon dioxide removal system due to its high carbon dioxide content at times. Further treatment of this stream may not be warranted, however trace hydrocarbons that escape the Silica Gel bed may cause problems, and require a carbon bed.

The second Silica Gel bed is regenerated by heating and purging with part of the oxygen, nitrogen, carbon dioxide stream during the first part of the batch cycle, before the chlorine peak is generated. Carbon or Hopcalite will be required to prevent buildup in the gas loop.

At the end of a process cycle, the urine in the scrubber loop is transferred to the electrolysis loop, and the scrubber loop is filled with fresh urine.

The only untried portion of the proposed system is the polymer separation. In the event that this component cannot be developed, an air dilution/Hopcalite catalytic burner system will produce an acceptable gas stream, however at an increased system penalty.

8.0 References

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