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FOREWORD

This study was initiated by the Biomedical Laboratory, 6570th Aerospace Medical Research Laboratories, Wright-Patterson Air Force Base, Ohio. The research was conducted by Ionics, Incorporated, Cambridge, Massachusetts, under Contract No. AF 33(657)-7710 in support of Project No. 6373, "Equipment for Life Support In Aerospace," Task No. 637304, "Waste Recovery and Utilization." Lt. Donald H. Jensen of the Respiratory Equipment Branch, Biotechnology Division, Biomedical Laboratory, was the contract monitor.

The principal investigators for Ionics were Mr. Robert W. Lindstrom, Senior Scientist, in charge of the pretreatment studies, and Mr. J. Douglas Smith, Senior Engineer, in charge of the electrolysis experiments. This contract was conducted under the supervision of Mr. Daniel L. Brown, Manager, Bionomics Section, Ionics, Inc. The research sponsored by this contract was performed between December 1961 and November 1962.

The authors wish to acknowledge the valuable assistance of Mr. Robert J. Allen, who conducted much of the experimental work on the kinetics of urea-urease reactions.

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ABSTRACT

A laboratory-scale experimental program was conducted which clearly indicated the feasibility of membrane electrodialysis for recovering potable water from urine. Since electrodialysis removes electrolytes from urine, pretreatment techniques were investigated for the removal of nonelectrolytes. These techniques were: charcoal adsorption, chemical oxidation, and enzymatic hydrolysis of urea to ammonium carbonate.

A model of a urine reclamation system was built, comprised of two main processes: charcoal adsorption and electrodialysis. The system weighs 15 pounds, is less than one ft³ in size, and has a peak power requirement of 45 watts for 8 hours operation for the daily recovery of 3000 ml of potable water from urine. The daily energy requirement is 310 watt-hrs, including 160 watt-hrs for charcoal regeneration. Excellent quality water is recovered in yields of 92%. The model has good reliability and can be readily adapted for conditions of weightlessness.

PUBLICATION REVIEW

This technical documentary report has been reviewed and is approved.

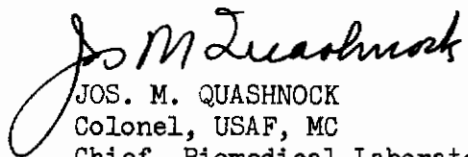

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RESEARCH ON THE RECOVERY OF WATER FROM URINE BY MEMBRANE ELECTRODIALYSIS

SECTION I

INTRODUCTION

As the scope of manned space flights becomes more ambitious in terms of crew size and duration of mission, it is increasingly necessary to develop improved life support equipment. One of the most critical life support areas is that concerned with the reclamation of water since, except for the shortest space flights, water must be purified and reused. A major source of waste water (over three pounds per man-day) is in the urine excreted by crew members. However, because of the quantity and chemical nature of the impurities in urine, it is difficult to process in order to provide purified water.

It was the purpose of the program presented herein to determine by means of laboratory investigations the feasibility of the membrane electro dialysis method for recovering potable water from human urine. As part of this program, it was also required to investigate techniques for removing urine nonelectrolytes since electro dialysis does not normally remove these components. Based on the results of these investigations, an optimum process for the recovery of 3000 ml. of potable water per day from urine was to be specified in accordance with the following criteria as applied to mission lengths in excess of fourteen days:

1. Minimum weight
2. Minimum volume
3. Minimum power requirements
4. Reliability of individual components
5. Operation under conditions of weightlessness
6. Necessity of component replacement

Electrodialysis is a relatively new unit operation in the chemical industry. In particular, it is used commercially in the demineralization of brackish water to produce potable water. Essentially, it involves the migration of ionized particles in a direction perpendicular to the direction of flow under the influence of an impressed direct current. Migration occurs across plastic membranes which are highly selective for the transport of either positively-charged ions (cations) or negatively-charged ions (anions). Cations migrate toward the cathode from one cell compartment to another as long as there is a cation-transport membrane separating the two compartments. However, cations are effectively "trapped" in any compartment which has an anion-transport membrane on the cathode side of the compartment. In a similar manner, anions migrate toward the anode until they are "trapped" in a compartment which has a cation-transport membrane on the anode side of the compartment. By the proper arrangement of ion-transport membranes, an electrolyte-containing

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stream can be separated into a pure water stream and a concentrated brine. (For a detailed discussion of membrane electrodialysis, see references 8 and 9.)

Thus, in principle, electrodialysis is the antithesis of the more familiar water purification techniques, especially distillation, since these techniques remove water from the solutes. However, there have been serious disadvantages noted in the operation of distillation equipment for urine water reclamation. For example, some of the solutes in urine, notably urea, tend to distill over with the water or are decomposed with the subsequent contamination of the recovered water by the decomposition products. Also, it is difficult to perform distillation under conditions of weightlessness. Furthermore, a large amount of energy is required to vaporize all the water unless the distillation is carried out in vacuum, in which case a considerable weight penalty must be paid because the low temperatures require large heat transfer area.

Thus, electrodialysis, which removes solute from urine to leave potable water, has the potential advantages of much lower energy and/or volume requirements than distillation. The main problem is that the solutes in urine are about 50% ionized inorganic salts (electrolytes) and 50% nonionized organic compounds (nonelectrolytes). Techniques, other than electrodialysis, have to be used to remove the organic compounds. Three such techniques were investigated during the course of this work and they could be employed separately or in various combinations. The techniques considered were:

1. Enzymatic conversion of urea (the major organic component) to ammonium carbonate, an ionizable inorganic salt
2. Adsorption of organic compounds
3. Chemical oxidation of organic compounds

Based on past experience, it was decided that the nonelectrolyte removal steps should precede any electrodialysis operations. Therefore, the techniques mentioned above have been termed pretreatment steps. Under certain circumstances post-treatment steps were also required to produce high quality water.

At the conclusion of this investigation two processes were evaluated for aerospace application of electrodialysis to recover potable water from urine based on the criteria previously mentioned. Process A has the following sequence of operations:

1. Enzymatic conversion of urea to ammonium carbonate
2. Adsorption on charcoal
3. Electrodialysis
4. Post-treatment clean-up

Process B has the following sequence of reactions:

1. Adsorption on charcoal
2. Electrodialysis

Both processes produce potable water and appear feasible for aerospace applications.

SECTION II

REMOVAL OF NONELECTROLYTES FROM URINE

About 50% of the solutes normally found in urine consist of nonionized organic compounds (nonelectrolytes). A list of the average concentration of major solute constituents is presented in Table 1 (ref. 3). Since electro dialysis is a process for the removal of electrolytes from water, other techniques must be applied in order to remove nonelectrolytes in urine. Moreover, to ensure optimum operation of the electro dialysis equipment, it is preferable to remove the nonelectrolytes before electro dialyzing the urine. Thus, the following techniques were investigated as possible pretreatment steps:

1. The addition to raw urine of an enzyme which converts urea, the major nonelectrolyte constituent, to ammonium carbonate, which is an ionizable inorganic salt
2. The contacting of either raw urine or the solution resulting from the enzyme reaction with charcoal adsorbents to remove nonelectrolytes
3. The addition of chemical oxidizing agents in order to remove additional nonelectrolytes

1. UREASE TREATMENT

Enzymatic Conversion of Urea to Ammonium Carbonate

One of the more promising pretreatment steps in a system using electro dialysis to recover potable water from urine is the enzymatic conversion of urea to ammonium carbonate. By this method about 90% of the nonelectrolyte concentration is converted to a compound which can be removed by electro dialysis equipment. The enzyme used for this conversion is urease which is a highly stable, commercially available enzyme (see ref. 2). In the presence of water, urease hydrolyzes urea according to the following reaction (see ref. 1C).



Reaction 1 is well-known and forms the basis of many analytical procedures for the determination of urea (see ref. 4). Once converted to ammonium carbonate, the samples are analyzed for the ammonium ion concentration.

TABLE I
COMPOSITION OF A TYPICAL NORMAL URINE

<u>Constituent</u>	<u>Daily Excretion in Grams</u>
Water	1200.0
Solids	60.0
Urea	30.0
Uric Acid	0.7
Hippuric Acid	0.7
Creatinine	1.2
Indican	0.01
Oxalic Acid	0.02
Allantoin	0.04
Amino Acid Nitrogen	0.2
Purine Bases	0.01
Phenols	0.2
Chloride as NaCl	12.0
Sodium	4.0
Potassium	2.0
Calcium	0.2
Magnesium	0.15
Sulfur, total, as S	1.0
Inorganic Sulfates as S	0.8
Neutral Sulfur as S	0.12
Conjugated Sulfates as S	0.08
Phosphate as P	1.1
Ammonia	0.7

In order to evaluate the merits of this pretreatment step, it was necessary to determine the quantities of urease required as a function of reaction times. While much has been reported in the literature regarding urease-urea kinetics, little work has been done with urea in raw urine. Because solution pH has been found to be such an important variable for urea-urease reactions, most investigators have standardized the reactions by employing a suitable buffer so that the pH remains fairly constant during the entire reaction period. It was felt that for aerospace applications, the use of buffers would add unnecessary complexity to a water reclamation system and also present an additional weight penalty. Therefore, in the experiments reported here, urease was added to samples of raw urine in order to establish the concentration of urease required to hydrolyze at least 98% of the original urea concentration to ammonium carbonate in a two hour period. This time limitation was not a magic number, but merely represented an allowable time interval for this part of a water reclamation system.

To obtain reaction rate data, samples of freshly collected urine were analyzed for free ammonium ion and urea concentrations. Initial free ammonium concentrations were measured because the urea-urease reaction was followed by analyzing the samples for increases in the ammonium concentrations. Once the free ammonium concentration in urine was known, any increase in its value could be directly related to the hydrolysis of urea.

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Two grades of commercial urease were investigated, "Jack Bean Meal" urease powder and "Soluble" urease powder. The latter material is more highly purified and, thus, has a higher activity per unit weight. Because of the relatively high cost of the "Soluble" urease, most of the preliminary reaction rate experiments were conducted using "Jack Bean Meal" urease. In these experiments known amounts of urease powder were added to fresh urine and after gently swirling for 30 seconds, the samples were capped and set aside without further agitation. Periodic ammonium ion analysis indicated the progress of the reaction. The reproducibility of this type of experiment was checked and found to be about 6% for the same samples under the same conditions.

Samples were analyzed for ammonium ion concentration in the following manner. The ammonium ion in an aliquot sample was converted to ammonia by the addition of a strong base (potassium carbonate), which also quenched the hydrolysis reaction. The solution was then aerated for one hour to liberate the ammonia, which bubbled into a known equivalent of hydrochloric acid. The resultant acid solution was back-titrated with sodium hydroxide to a methyl red end point. The analyses of per cent urea converted were good to $\pm 2\%$.

Table 2 lists the data obtained for three runs (at room temperature) employing 200 mg of urease per 100 ml of fresh urine. A plot of the log of the concentration of urea remaining at time t versus time t , Figure 1, indicates that the urease-urea (urine) hydrolysis is apparently a first order reaction, i.e., a reaction in which the rate is found to be directly proportional to the concentration of the reacting substance (urea). Since the amount of material which reacts depends on the amount that is present, and, if the volume is kept constant the situation can be described mathematically as follows:

$$-\frac{dc_A}{dt} = kC_A \quad (2)$$

Where C is the concentration of the reacting material A , k is a proportionality factor, t is the time, and $-dc/dt$ is the rate at which the concentration decreases.

The proportionality constant k , which is referred to as a velocity constant, can be evaluated by multiplying the slope of the line by 2.303. The value of k may also be found for any time interval by employing the following equation:

$$k = \frac{2.303}{t} \times \log \frac{C_0}{C} \quad ()$$

Where C_0 is the concentration at the beginning of the reaction (at $t=0$) and C is the concentration after time t has elapsed.

Reaction rates may be expressed by giving the numerical value of dc/dt (moles/liter) or by giving the period of half-life, $t_{1/2}$, that is, the time necessary for half a given quantity of material to decompose. For a first order

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TABLE 2

UREASE CONVERSION RATE DATA

Run 1			Run 2		
Physical and Chemical Analysis of Urine			Physical and Chemical Analysis of Urine		
pH = 5.65 Temp = 22.6°C			pH = 5.85 Temp = 22.5°C		
* Corrected Specific Gravity = 1.016 Moles/liter free NH_4^+ = 0.0508 Moles/liter Urea = 0.2598 (15.6 g/l)			* Corrected Specific Gravity = 1.017 Moles/liter free NH_4^+ = 0.0557 Moles/liter Urea = 0.3472 (20.8 g/l)		
Rate Data			Rate Data		
Time (min)	M/l Urea Converted	% Converted	Time (min)	M/l Urea Converted	% Converted
30	0.0881	33.9%	30	0.0917	26.4%
75	0.1496	57.6%	60	0.1357	39.1%
110	0.2166	83.3%	123	0.2012	58.0%
165	0.2346	90.3%	150	0.2332	67.3%
210	0.2466	94.9%	210	0.2752	79.3%
Ave. k = 0.014 min ⁻¹ t $\frac{1}{2}$ = 49.5 min.			Ave. k = 0.0075 min ⁻¹ t $\frac{1}{2}$ = 92.5 min.		

Run 3

Physical and Chemical Analysis of Urine

pH = 6.35
Temp = 23.1°C

* Corrected Specific Gravity = 1.014
Moles/liter free NH_4^+ = 0.0317
Moles/liter Urea = 0.2892 (17.35 g/l)

Rate Data

Time (min)	M/l Urea Converted	% Converted
30	0.0742	25.7%
60	0.1157	40.0%
130	0.2062	71.3%
170	0.2332	80.7%
210	0.2501	86.6%

Ave. k = 0.0097 min⁻¹ t $\frac{1}{2}$ = 71.3 min.

* Specific gravity was taken with a urinometer calibrated for 15.6°C.
The suggested temperature correction was employed to give the values indicated.

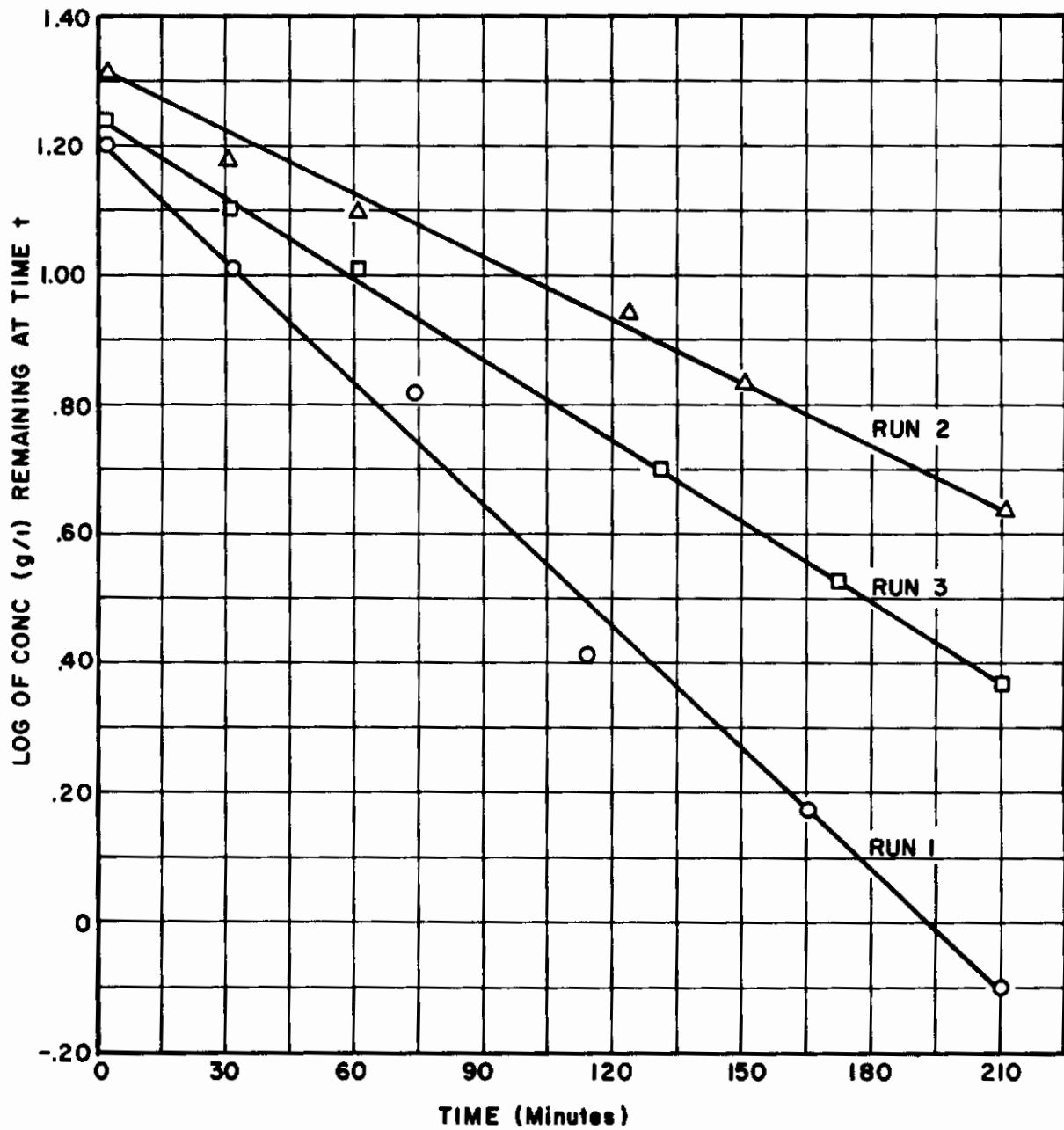


FIGURE 1

PLOT OF LOG OF UREA CONCENTRATION AT TIME t VS. TIME

reaction the period of half-life is calculated as follows:

$$t_{1/2} = \frac{0.693}{k} \quad (4)$$

The determination of the velocity constant from equation 3 and subsequently the half-life of the reaction makes possible a calculation of the time necessary for a 98% conversion without actually obtaining data up to the final time. The time necessary for a 98.4% conversion is six times the half-life of the reaction. This time, or the time necessary for any other conversion percentage, can be calculated from equation 3. A more convenient form of equation 3 is as follows:

$$\frac{C}{C_0} = e^{-kt}$$

A comparison of the reaction rates from Table 2 and Figure 1 indicates that the reaction rates did not vary proportionately with the initial concentration of urea. In other words, any one reaction follows an apparent first order rate but each sample of different urea concentration reacts with a different velocity constant. This was somewhat expected since other investigators needed rigid pH control before obtaining acceptable data.

An attempt was made to correlate the data obtained in similar tests with that shown in Table 2. Since most of this new data was not taken specifically for reaction rate determinations, the velocity constants were calculated from one or two analyses. These data were evaluated with respect to reaction rate versus initial urea concentration. From the wide discrepancies observed it was concluded that some other parameter besides urea concentration was an important factor. The most obvious parameter was pH.

An experiment was designed in which the pH of the same urine was varied without essentially changing the volume and the reaction rates determined. These data, which are tabulated in Table 3 - Column A, show a very pronounced relationship between pH and reaction rate over the pH range investigated. (This pH range corresponds to that normally found in fresh urine). As the pH was increased, the reaction rate decreased proportionately. If the velocity constants from Column A are multiplied by their respective pH value and a new set of reaction rates calculated, as has been done in Table 3 - Column B, a fairly consistent reaction rate is obtained. The usual first rate equation was then modified to be as follows:

$$-\left(\frac{dc}{dt}\right) = k'c \quad \text{where } k' = \text{pH} \times k$$

There are probably other methods of expressing this pH correction. Whether they be more appropriate or more precise is not questioned. The modified equation involving k' is just an expression of one possibility.

The data being correlated were then compared, Table 4, with respect to the calculated values of

$$-\frac{dc}{dt} \text{ and } -\left(\frac{dc}{dt}\right)'$$

versus urea concentration. These data are portrayed in Figure 2 as a plot of log rate versus log urea concentration - Curve A - and of log rate prime versus log urea concentration - Curve B. If one takes the liberty of drawing a straight line of exactly the same slope through both sets of points, it appears that Curve B is a better fit of the data points than Curve A. It is felt that if a set of correction factors could be evolved, especially one relating to ionic strength, the points on a plot of log rate versus log concentration of urea would more nearly fall on a straight line.

TABLE 3

VARIATION OF REACTION RATE WITH pH AND CORRECTION FOR SAME

Physical and Chemical Data of Urine

pH = 5.75
 Temp = 23°C
 Corrected Specific Gravity = 1.015
 Moles/liter free NH_4^+ = 0.0476
 Moles/liter Urea = 0.2762 (16.6 g/l)

<u>A</u>				<u>B</u>			
<u>Data Calculated by Regular Method</u>				<u>Data Calculated by Modified Method</u>			
pH	Velocity Constant min^{-1}	$-\frac{dc}{dt}$	% Deviation from ave. rate	pH	Velocity Constant min^{-1}	$-\left(\frac{dc}{dt}\right)'$	% Deviation from ave. rate
4.70 ¹	0.017	0.00470	+ 27.7%	4.70	0.080	0.0221	+ 1.4%
5.75 ²	0.013	0.00359	- 0.25%	5.75	0.075	0.0207	- 5.1%
8.20 ³	0.010	0.00276	- 25.0%	8.20	0.082	0.0226	+ 3.7%
ave. = 0.00368				ave. = 0.0218			

- 1 pH adjusted with 2 drops of conc. H_2SO_4 (100 ml urine)
- 2 as obtained
- 3 pH adjusted with 1 KOH pellet (100 ml urine)

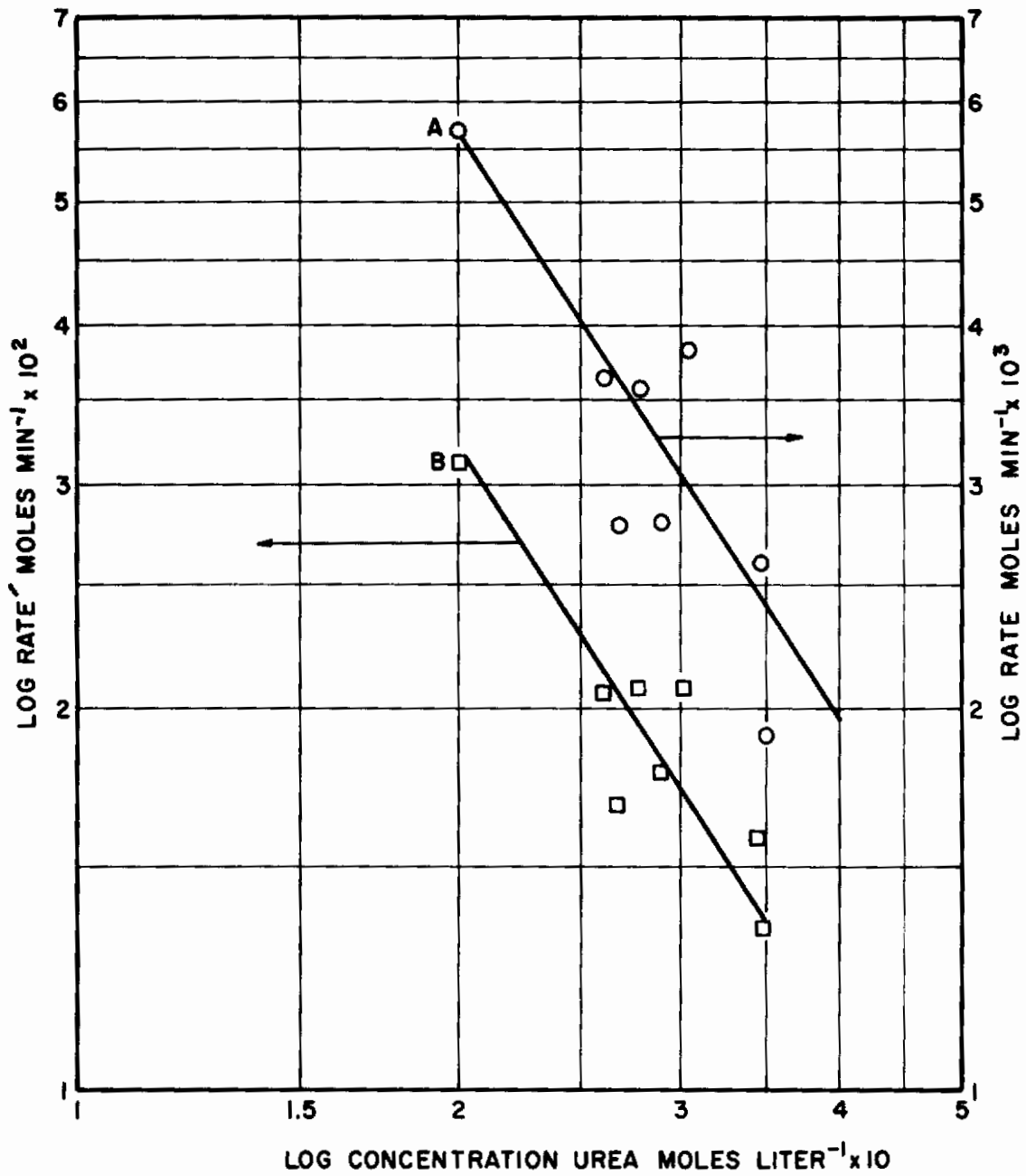


FIGURE 2

PLOT OF $\text{LOG} - \frac{dc}{dt}$ AND $-\frac{dc'}{dt}$ vs. LOG CONCENTRATION UREA

TABLE 4

TABULATION OF $-\frac{dc}{dt}$ AND $-(\frac{dc}{dt})'$ FOR 200 mg UREASE IN 100 ML OF FRESH URINE

Urea Conc. g/l	Urea Conc. m/l	pH	Velocity Constants min ⁻¹	$-\frac{dc}{dt}$	$-(\frac{dc}{dt})'$
12.22	.202	5.50	.028	.00566	.0311
15.60	.261	5.65	.014	.00365	.0206
15.98	.266	6.05	.0105	.00279	.0169
16.60	.276	5.75	.013	.00358	.0207
17.35	.289	6.35	.0097	.00280	.0178
18.20	.303	4.34	.0127	.00385	.0209
20.80	.347	5.85	.0075	.0026	.0157
20.91	.349	6.95	.0055	.0019	.0133

$$-\frac{dc}{dt} = kc$$

$$-(\frac{dc}{dt})' = k'c \text{ where } k' = pH \times k$$

Somewhat analogous data for the concentration range studied have been reported by Laidler and Hoare (ref. 6). Here again, however, these investigators obtained data for a urea-buffer solution. A comparison of the slope of the curve obtained by Laidler and Hoare and that obtained in the present investigation (log rate prime versus log conc.) is not possible since the former curve represents an extremely wide range of concentrations. Consequently, that portion of the curve corresponding to our study is extremely small. However, they also obtain a negative slope at these concentrations and assign an apparent negative order to the reaction. They also show that as the concentration of urea increases from 0.005 m/l to 0.14 m/l the reaction is first order; from 0.14 m/l to 0.2 m/l the reaction is zero order; and from 0.2 m/l to a level not previously determined, the reaction reverses to a negative order. (An experiment at this laboratory for an artificial 0.5 m/l urea solution indicated that the negative order still applied.) Laidler and Hoare state that the high concentrations of Urea (above 0.2 m/l) inhibit the catalysis of the enzyme due to absorption on the reaction sites.

Since it is highly unlikely that either pH or urea concentrations of the urine being processed in a space vehicle will be controlled, it is necessary to provide sufficient urease for the worst combination (high pH and high urea concentration). To illustrate the wide variation in per cent urea converted to ammonium carbonate in two hours as a function of urea concentration and pH, the data in Table 4 have been retabulated and are presented in Table 5. It should be remembered that this is for the addition of 200 mg. of "Jack Bean Meal" urease per 100 ml of urine.

TABLE 5
PERCENT UREA CONVERTED* IN TWO HOURS FOR VARIOUS
RAW URINE SAMPLES

<u>Urea Conc.</u> <u>g/l</u>	<u>pH</u>	<u>Velocity Constants</u> <u>(min⁻¹)</u>	<u>Urea Converted</u> <u>in 2 hours (%)</u>
12.22	5.50	0.028	96.5%
15.60	5.65	0.014	81.4
15.98	6.05	0.0105	71.6
16.60	5.75	0.013	79.0
17.35	6.35	0.0097	68.7
18.20	4.34	0.0127	78.2
20.80	5.85	0.0075	58.3
20.91	6.95	0.0055	48.4

* Converted to ammonium carbonate by the addition of 200 mg. of "Jack Bean Meal" urease to 100 ml. of urine.

Effect of Enzyme Concentration on Reaction Rate

Two tests were run in which the quantity of "Jack Bean Meal" urease was varied from the normal 200 mg per 100 ml of urine. To offer a comparison, a sample containing the usual amount of urease was run simultaneously. These data, which appear in Table 6, show that the reaction rate is essentially proportional to the concentration of urease for those levels tested.

Reactivity of Different Urease Preparations

During the course of this investigation many commercially available grades of urease were examined. Three were of particular interest. One material, which was in pellet form, was essentially nonreactive because the pellet did not disintegrate in urine. Grinding of the pellet yielded comparable data to those presented above. Another was the "Jack Bean Meal" urease powder, which was used to obtain much of the information concerning the kinetics and mechanism of the urease-urea reactions in urine. This material was inexpensive (40 cents per gram) but was not completely soluble in urine and showed a relatively low order of reactivity per unit weight. The third grade of urease was in the form of a completely soluble powder, having an activity of 1500 Sumner Units per gram. It was moderately expensive (\$12.50 per gram in small quantities) but proved to be about 6 times as active as the "Jack Bean Meal" powdered urease. A comparison of the reaction rates of this "Soluble" urease with "Jack Bean Meal" urease is presented in Table 7.

TABLE 6

VARIATION OF REACTION RATE WITH UREASE CONCENTRATION

Test 1

Physical and Chemical Analysis of Urine

pH = 5.45
 Temp = 20°C
 Corrected Specific Gravity = 1.015
 Moles/liter free Ammonium ion = 0.0482
 Moles/liter urea = 0.3030 (18.2 g/l)

<u>mg Urease/100 ml Urine</u>	<u>Calculated -dc/dt</u>	<u>Ratio Urease Added</u>	<u>Ratio $\frac{dc}{dt}$ Found</u>
200 mg	.00385	10 to 1	8.5 to 1
20 mg	.00043		

Test 2

Physical and Chemical Analysis of Urine

pH = 6.95
 Temp = 25.6°C
 Corrected Specific Gravity = 1.021
 Moles/liter free Ammonium ion = .0232
 Moles/liter Urea = .3484 (20.9 g/l)

<u>mg Urease/100 ml Urine</u>	<u>Calculated -dc/dt</u>	<u>Ratio Urease Added</u>	<u>Ratio $\frac{dc}{dt}$ Found</u>
400 mg	.0037	2 to 1	1.95 to 1
200 mg	.0019		

TABLE 7

COMPARISON OF UREASE REACTION RATES

<u>Urease</u>	<u>Urea Conc. g/l</u>	<u>pH</u>	<u>Vol. Urine ml</u>	<u>Urease mg</u>	<u>Velocity Constants min⁻¹</u>	<u>t 1/2 min</u>
Soluble	12.26	6.0	100	50	0.041	16.9
Soluble	12.26	6.0	100	200	0.164	4.2
Jack Bean Meal	12.22	5.5	100	200	0.028	24.7

Urease Requirements

For the water reclamation system proposed as a result of this program, "Soluble" urease is recommended for use in converting urea to ammonium carbonate. As previously mentioned, this urease has six times the reactivity of "Jack Bean Meal" urease.

The data on a wide variation of urea concentration and pH presented in Tables 4 and 5 have been recalculated based on the use of 200 mg of "Soluble" urease per 100 ml of urine. These data are presented in Table 8, which shows a further comparison between the two grades of urease.

It is apparent that even for the worst urine sample, this level of "Soluble" urease addition is sufficient to hydrolyze 98% of the urea within two hours. Thus, for treating 3000 ml of urine per day, 6 grams of "Soluble" urease are required. For a 14 day mission only 84 grams of urease must be carried. One pound of urease on take-off would handle 3000 ml of urine per day for 76 days.

TABLE 8
PERCENT UREA CONVERTED* IN TWO HOURS FOR VARIOUS
RAW URINE SAMPLES

Urea Conc. g/l	pH	<u>"Jack Bean Meal" Urease</u>		<u>"Soluble" Urease</u>	
		Velocity Constants (min ⁻¹)	Urea Converted in 2 hours (%)	Velocity Constants (min ⁻¹)	Urea Converted in 2 hours (%)
12.22	5.50	0.028	96.5%	0.168	99.9%
15.60	5.65	0.014	81.4	0.084	99.9
15.98	6.05	0.0105	71.4	0.063	99.9
16.60	5.75	0.013	79.0	0.078	99.9
17.35	6.35	0.0097	68.7	0.0582	99.9
18.20	4.34	0.0127	78.2	0.0762	99.9
20.80	5.85	0.0075	58.3	0.045	99.6
20.91	6.95	0.0055	48.4	0.033	98.1

* Converted to ammonium carbonate by the addition of 200 mg. of urease to 100 ml of urine.

2. CHEMICAL OXIDATION OF NONELECTROLYTES

The first pretreatment step described, that of the addition of urease to raw urine, is highly specific in nature in that it can only be used for the removal (or conversion) of urea. However, after this step, there still remains at least 4 grams of organic compounds (nonelectrolytes) per liter of urine. Among the compounds normally present in significant quantities are creatinine, uric acid, hippuric acid, unreacted urea and urease. Because of

Contrails

the widely varying chemical nature of these compounds, a nonspecific method of nonelectrolyte removal must be employed to ensure the complete removal of all organic compounds.

One of the methods investigated was the addition of a strong chemical oxidizing agent to the urease-reacted urine. The agents used were sodium hypochlorite and calcium hypochlorite, commonly used bleaches. These materials contain available chlorine which can react with the ammonia present from the hydrolysis of urea to form amines; NH_2Cl , NHCl_2 , and NCl_3 (ref. 1). These amines are also oxidizing agents and bacteriacidal agents, as in bleach.

Since the color of urine is due mainly to an organic constituent, urochrome, the effect of hypochlorite addition on organic removal was determined by measuring the degree of decolorization of urine samples. The measurements were made on a colorimeter set at 430 millimicrons. The darkest urine samples available were used to set the scale at 0% transmittance. Distilled water was used as a sample of 100% transmittance.

It was noted that on adding the minimum amount of hypochlorite necessary to provide a titratable excess of chlorine, there was a darkening in the color of the urine. Not until a large excess of hypochlorite was added did any decolorization take place. At any reasonable levels of bleach addition residual color was always present in the urine samples, thus indicating that some non-electrolytes still remained.

Three bleaches were tested during the program; two grades of calcium hypochlorite powder 35% and 70% active in chlorine, and sodium hypochlorite liquid 5% to 6% active in chlorine. As expected, one-half the weight of 70% active calcium hypochlorite was necessary to produce the same decolorization as the 35% active material. In addition, on a weight basis the 70% active calcium hypochlorite was a more effective oxidizing agent than sodium hypochlorite (see Table 9).

Evaluation of the feasibility of using these oxidizing agents for pretreating urine in a membrane electrodialysis process produced the following conclusions:

1. Large weight penalties were indicated because of the high concentrations of these chemicals required to significantly lower the total organic content of urease-reacted urine.
2. Even at high concentrations, the hypochlorite bleaches were unable to completely remove all traces of urine color and odor.
3. Upon addition of bleach to urine, gas evolution occurred (including chlorine) which would complicate zero-gravity operations.
4. Calcium hypochlorite addition to urease-reacted urine produced a precipitate of calcium carbonate which had to be filtered out of the solution before further processing was possible.
5. High concentrations of bleach increased the power required to demineralize urine in an electrodialysis stack because of the increase in total salt content.

TABLE 9

COMPARISON OF HYPOCHLORITES AS CLARIFYING AGENTS FOR URINE

<u>Hypochlorite</u>	<u>ml per liter</u>	<u>Recovered Urine (initial vol. 100 ml)</u>	<u>% Transmission</u>
NaClO ₃ (6% Active Chlorine)	0	100 (no charcoal)	0
	25	102.5	49.0
	50	105.0	48.5
	100	110.0	53.0
	200	120.0	70.9
Ca(ClO ₃) ₂ (70% Active Chlorine)	<u>gm per liter</u>		
	2.5	91	44.0
	5.0	84	43.3
	10.0	80	46.1
	20.0	64	36.2

For these reasons it was decided to abandon the use of any type of hypochlorite oxidant for urine. The use of hydrogen peroxide as an oxidizing agent was considered. However, it was rejected because even in very dilute concentrations it reacted with charcoal, the use of which still was required to completely eliminate color and odor. The reaction took the form of gas production which caused severe bed binding.

3. CHARCOAL ADSORPTION STUDIES

In view of the many disadvantages found in using chemical oxidizing agents to remove the residual nonelectrolyte constituents in urease-reacted urine, that pretreatment step was abandoned. In place of that technique, the use of charcoal adsorbents for organic removal was investigated. Preliminary experiments had shown that urine percolated through a charcoal bed exhibited no residual color or odor. Thus, two approaches were taken for the use of charcoal adsorbents in pretreating urine. In one, urease-reacted urine was contacted with charcoal to remove the residual organic content. In the other, raw urine was contacted with charcoal to remove all the urea and other non-electrolytes. Both approaches proved to be feasible.

Figure 3 presents a transmittance curve of urine that was reacted with urease, filtered and passed through a charcoal column. The charcoal used was granular cocoanut charcoal (50-200 mesh) which previously had proved superior to other varieties of charcoal. The column had a contact time of 8.9 minutes. At this flow it would indicate a charcoal use of approximately 250 grams per liter of urine. Subsequent tests indicated that one liter of urine could be passed through 200 grams of cocoanut charcoal with a contact time of 15.4 minutes and give a product having transmittance greater than 90% based on the standard of 100% transmittance for distilled water.

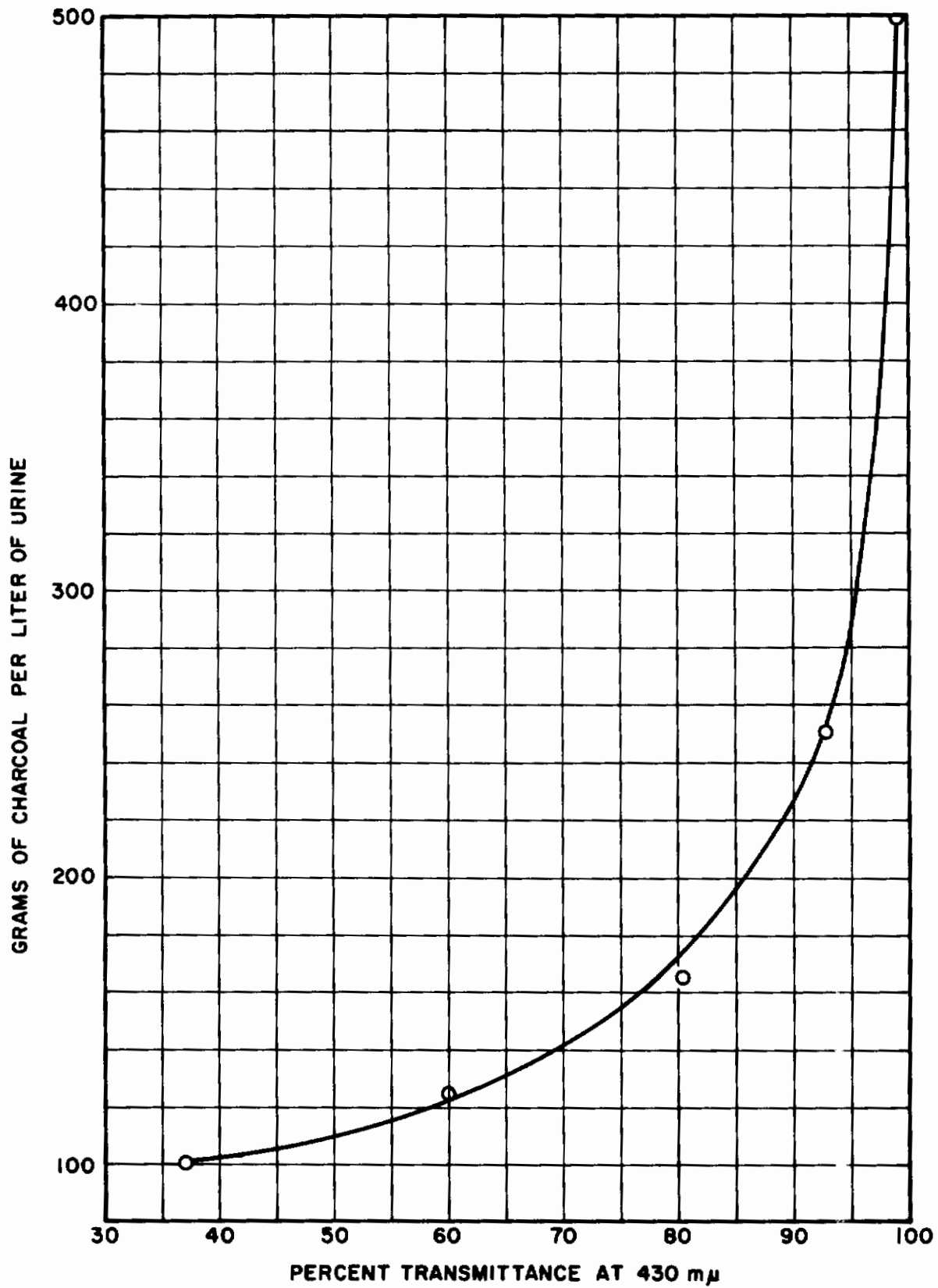


FIGURE 3
DECOLORIZATION OF URINE WITH CHARCOAL

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When raw urine was contacted with cocoanut charcoal, about twice as much charcoal was required to remove urea and other organics as was required to remove the organics remaining after urea-urease reaction. A representative plot of urea concentration versus volume of urine passed through a charcoal column is presented in Figure 4. The percentage of urea removed as a function of charcoal requirements was determined from this figure. The values were as follows:

<u>Charcoal Requirements</u>	<u>Urea Removed</u>
400 gms/liter	99.8%
300 gms/liter	98.4%
200 gms/liter	35.8%

By doubling the residence time of urine in a charcoal column compared to the residence time used for Figure 4, the urea was removed below detectable levels. Thus, it appeared that 400 grams of cocoanut charcoal were sufficient to remove almost all the urea present in one liter of urine.

Although the weight of charcoal required in the treatment of raw urine was double that of removing the residual organic from urease-reacted urine, there were distinct advantages in using charcoal adsorption as the only pretreatment technique.

1. The final salt concentration of the charcoal pretreated urine was roughly one quarter the concentration of the urease plus charcoal pretreated urine (5000-9000 ppm versus 20,000-50,000 ppm). In electrodialysis, as ions are transferred across the membranes, water is carried along with them (called endosmotic water). This is the major source of water loss in the system. Therefore, the lower the initial salt concentration in the feed to an electrodialysis stack, the greater is the yield of potable water.

2. The concentration of ammonium ion in the charcoal pretreated urine was about one-fiftieth the concentration of the urease plus charcoal pretreated urine (140 ppm versus 6000 ppm). Since public health service standards require low ammonium ion concentration in drinking water (less than 1 ppm), the urine from the urease plus charcoal steps had to be almost completely demineralized in the electrodialysis unit or by post-treatment steps, e.g., ion exchange, to meet this standard. However, the urine pretreated with charcoal alone easily met this standard when the total dissolved solids concentration was lowered to about 100 ppm in the electrodialysis stack. This additional demineralization requirement for urease plus charcoal pretreated urine resulted in even lower potable water yields for the reason stated above.

3. Because the initial salt concentration was lower and demineralization requirements were not so stringent, the power requirements to electrodialyze urine with only a charcoal pretreatment step were lower in addition to potable water recoveries being higher.

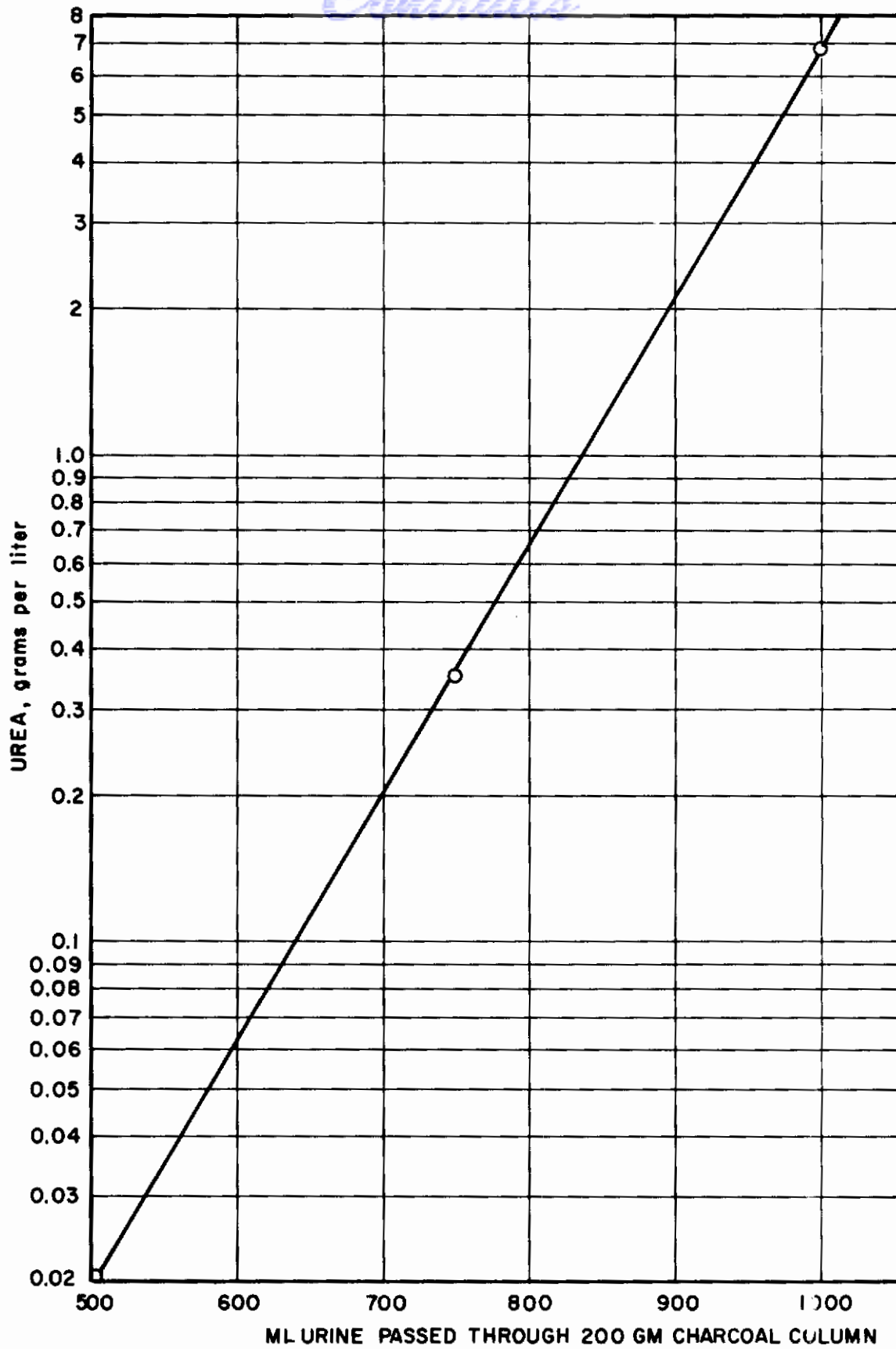


FIGURE 4
CHARCOAL REQUIREMENTS FOR UREA REMOVAL
INITIAL UREA 10.9 GRAMS PER LITER

4. ADDITIONAL PRETREATMENT TECHNIQUES

Urine, following reaction with urease and percolation through charcoal, contained very high levels of ammonium ion concentration. Because of this, energy requirements and water losses were higher in the subsequent electro-dialysis step than in cases where only charcoal was used for nonelectrolyte removal. In an effort to lower ammonium ion concentrations, two additional pretreatment steps were evaluated. One involved pulling a vacuum on the solution to remove ammonia gas, which is in equilibrium with ammonium ion. The other involved the addition of a chemical compound which reacted with ammonium ion to form a precipitate.

After reaction with urease, urine will contain between 6000 and 14,000 ppm of ammonium ion and have a pH of approximately 9.15. This terminal pH was observed for all of the urease-reacted urine samples and represents the hydrolysis pH of ammonium carbonate formed in the urine. Since the possibility exists of being able to use the vacuum of space, an attempt was made to remove ammonia from urine by application of vacuum.

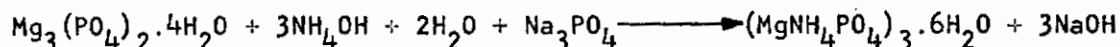
For these tests, a suitable vacuum was applied to the sample and the vapor removed was passed through an acetone-dry ice trap and measured. The results of these tests, Table 10, indicated that unless a vacuum capable of removing considerable water vapor was applied, the removal of ammonia was not spectacular. Lower vacuums than those listed in Table 10 were not applied for any length of time because considerable frothing occurred at these low vacuums. Because these results were not particularly encouraging, the alternative of direct precipitation of ammonium was investigated.

TABLE 10
VACUUM REMOVAL OF AMMONIA FROM UREASE-CONVERTED URINE

Run	Time (hrs)	Ave. Absolute Vacuum	% Water Lost	% Ammonia Lost
1	2	20 mm Hg	> 1	5.7
2	2	10 mm Hg	≈ 6	27.2
3*	1-½	25 mm Hg	≈ 1	9.5
4*	5	20 mm Hg	≈ 1	8.0

* Samples were stirred during test.

It was noted that the solubility product of magnesium ammonium phosphate decreases with increasing pH values. Since the pH of urine is 9.15 after the urease reaction, magnesium ammonium phosphate should form a relatively insoluble precipitate. According to Jacobson's Encyclopedia of Chemical Reactions (ref. 5), the reaction with ammonium is:

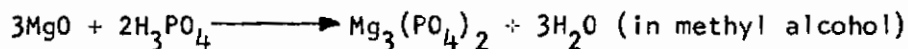


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Four different sources of magnesium phosphate were evaluated:

1. A mixture of Na_2HPO_4 and MgSO_4
2. A commercial grade $\text{Mg}_3(\text{PO}_4)_2$
3. Freshly precipitated $\text{Mg}_3(\text{PO}_4)_2$, oven dried
4. Freshly precipitated $\text{Mg}_3(\text{PO}_4)_2$, air dried

The freshly precipitated material was made for test purposes according to the following reaction:



The portion that was air dried retained about 10% residual moisture.

A comparison of the effectiveness of each source of magnesium phosphate in lowering the ammonium ion concentration is presented in Table II. It can be seen that only moist, freshly precipitated materials (1 and 4) were reasonably effective at the levels of addition used (100 grams/liter). This represented a distinct disadvantage to the use of this method in a space environment because of the added complexity to the system. In addition, the necessity of using 100 grams of precipitant per liter of urine represented a high weight penalty for missions of long duration. Furthermore, the excess magnesium ions which remained in the urine were more difficult to remove by electro dialysis than ammonium ions. Thus, for the reasons mentioned above, this pretreatment technique was deemed unsuitable for use in a process to recover potable water from urine by membrane electro dialysis.

TABLE II
DIRECT PRECIPITATION OF NH_4^+

Source	Precipitating Agent 100 grams/liter of urine	Initial Normality of NH_4^+	Normality of NH_4^+ After Precipitation	Percent Reduction of NH_4^+
1	Na_2HPO_4 ; $\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$	0.31	0.06	80.7%
2	$\text{Mg}_3(\text{PO}_4)_2$ U.S.P.	0.44	0.36	18.2%
3	$\text{Mg}_3(\text{PO}_4)_2$ Freshly ppt; oven dried	0.39	0.32	18.0%
4	$\text{Mg}_3(\text{PO}_4)_2$ Freshly ppt; not dried	0.39	0.17	56.4%

SECTION III

MEMBRANE ELECTRODIALYSIS

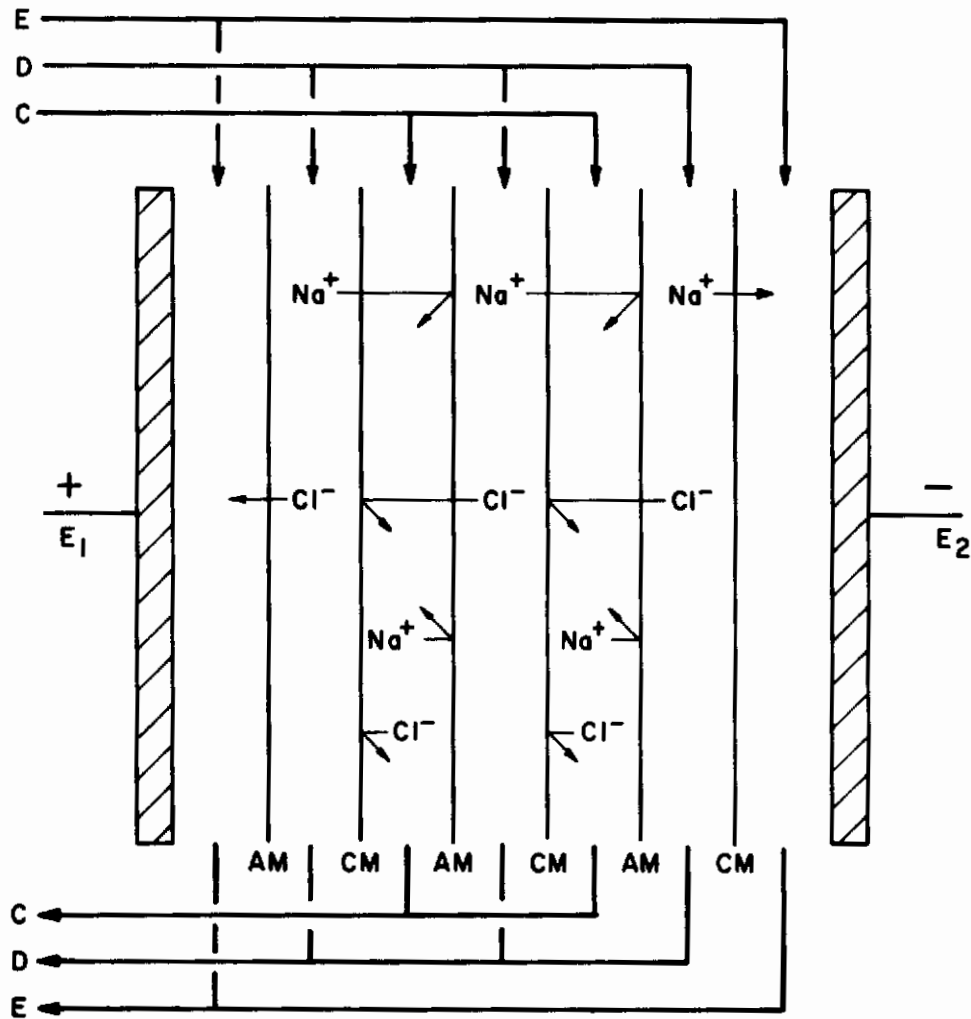
I. PRELIMINARY DISCUSSION

On the basis of the experimental results obtained for the various pretreatment steps, two techniques were selected which completely removed the nonelectrolytes from urine. One technique consisted of adding urease to urine to convert most of the urea to ammonium carbonate and then percolating the urine through cocoanut charcoal to remove the residual organic constituents. The other technique involved merely contacting raw urine with sufficient cocoanut charcoal to remove all the nonelectrolytes. The organic-free solutions resulting from both techniques were then fed to electro dialysis stacks where demineralization took place. Comparisons were made between the two pretreatment techniques on the basis of the energy requirements and potable water recoveries in the electro dialysis step.

Before proceeding with a discussion of the electro dialysis experiments performed, it will be necessary to present a brief explanation of this process and to define the more commonly used terms.

Electro dialysis is a process in which ionized molecules or atoms are transferred through highly selective ion-transfer membranes under the influence of a direct current. If a solution containing positively and negatively-charged ions is fed to an electro dialysis cell, the positively-charged ions (cations) will be attracted to the negatively-charged cathode and the negatively-charged ions (anions) will be attracted to the positively-charged anode. The nature of the ion-transfer membrane between the solution and electrode (anode or cathode) determines whether or not an ion can migrate through it or be retained in the solution. Anion-transfer membranes will allow anions to pass through them but exclude cations, while cation-transfer membranes will allow the passage of cations but not anions. These membranes are highly selective for either anion or cation transfer.

As an illustration of an electro dialysis process, the demineralization of a salt solution is considered, as shown in Figure 5. If an ionized salt solution is fed to all the compartments of an electro dialysis stack in which multiple anion and cation membranes are alternated between an anode and cathode, anions will be transferred out of compartments where anion membranes are between the solution and the anode, and cations will be transferred out of the same compartments since cation membranes will be between the solution and the cathode. Thus, in this manner alternate compartments are being demineralized to produce pure water or being concentrated to produce a brine stream. The demineralized compartment is referred to as the dilute compartment and the brine compartment is referred to as the concentrate compartment. The two adjacent compartments



C = CONCENTRATE STREAM
D = DILUTING STREAM
E = ELECTROLYTE STREAM
CM = CATION MEMBRANE
AM = ANION MEMBRANE

FIGURE 5
TYPICAL ELECTRODIALYSIS UNIT

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are termed a cell pair. It is possible in this sort of arrangement to place as many as 500 cell pairs between a single pair of electrodes. The combination of cell pairs and electrodes is referred to as an electrodialysis stack.

2. ELECTRODIALYSIS STACK CONFIGURATIONS

During the course of this investigation many electrodialysis stack configurations were tested. The first configuration was designated Type A and is illustrated in Figure 6. The distinguishing feature of Type A was the ion-exchange resin packing between ion-transfer membranes. At the latter stages of a demineralization process, the electrical resistance of the dilute stream may become very great. Thus, the resin served chiefly to prevent a high voltage drop from occurring across the dilute cell compartments.

The main purpose of the electrodialysis experiments conducted on Stack Type A was to gain some insight into the basic variables involved. The experiments were conducted on "synthetic urine" solutions, which were prepared by dissolving the following salt concentrations in distilled water:

$(\text{NH}_4)_2 \text{CO}_3 \cdot \text{H}_2\text{O}$	40 g/l	0.700 N
NaCl	10 g/l	0.171 N
K_2SO_4	4 g/l	0.045 N
	<hr/>	<hr/>
	54 g/l	0.916 N

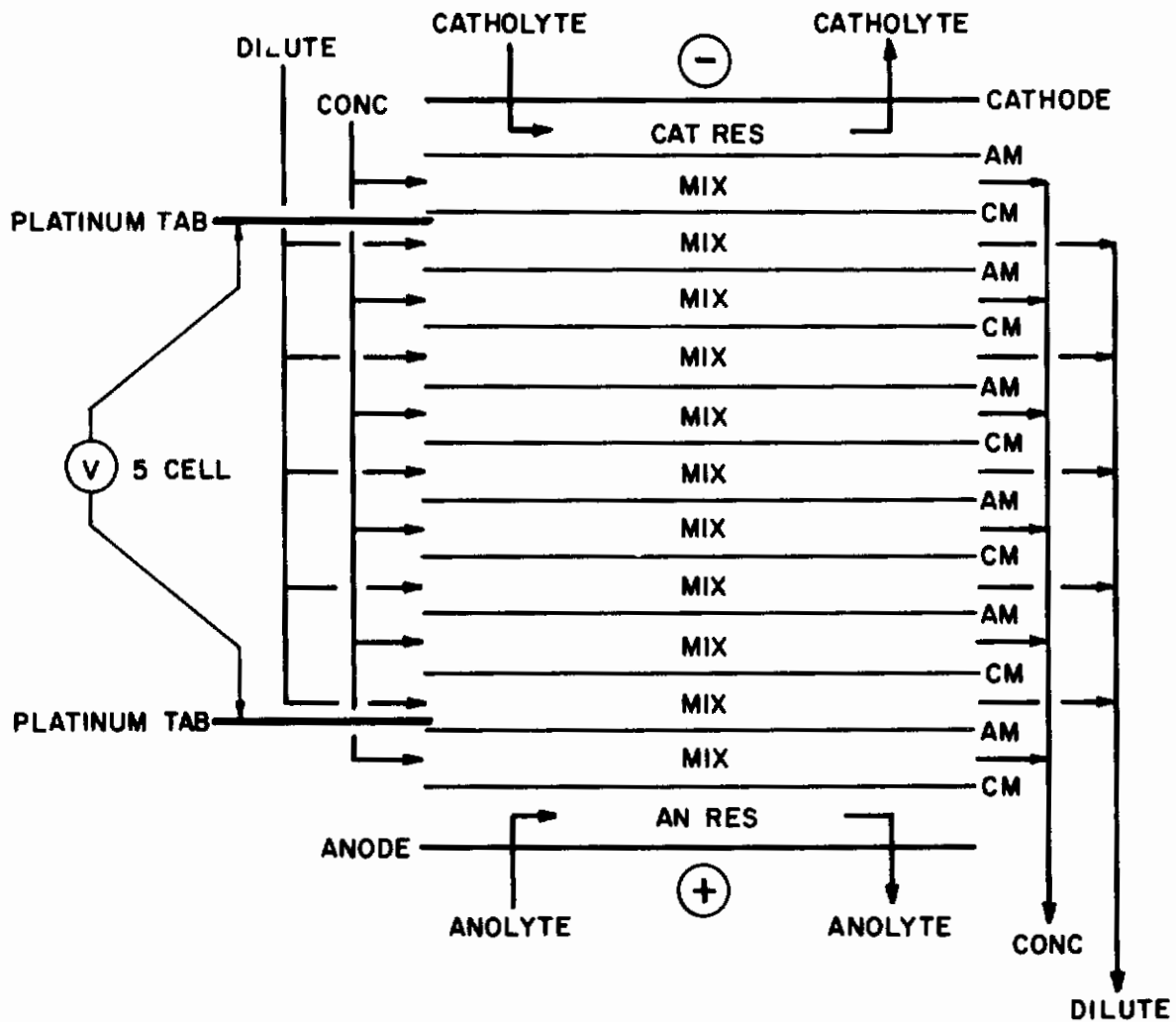
This simulated the conditions expected when real urine was pretreated with urease and charcoal and was based on the figures for salt concentrations presented in Table I.

In these experiments a batch of "synthetic urine" was recirculated through the dilute cells until the desired demineralization had been accomplished. In a typical run (Run A-2) the normality of a four liter batch was reduced from 0.916 N to 0.022 N in 90 minutes. This was equivalent to a reduction from 54,000 ppm to 1320 ppm. Other results were as follows:

Water recovery	= 63%
Current efficiency	= 50%
Energy consumption	= 79 watt-hrs/liter

The energy consumption figure did not include the energy consumed at the electrodes.

Another run (Run A-4) was designed to estimate limiting current densities for the resin-filled electrodialysis stack being tested. Ions are transferred to the membranes by diffusion and by the turbulence of the dilute feed stream. If the current density (amps/cm²) of an electrodialysis stack is so large that ions are being transferred across



CM = CATION MEMBRANE CR-61 STD.
 AM = ANION MEMBRANE AR-III STD.
 CAT RES = CATION EXCHANGE RESIN
 AN RES = ANION EXCHANGE RESIN
 MIX = 50-50 EXCHANGE RESIN MIX

NO. CELL PAIRS = 5
 CELL SPACING = 0.052 IN.
 EFF. MEMBRANE AREA = 1.875 IN. x 16 IN. = 30 SQ. IN.

FIGURE 6
STACK TYPE A

Contrails

the membrane more rapidly than ions can be transported to the membranes, a condition known as polarization will occur. Water will be dissociated and OH^- and H^+ ions will be transferred across the membranes. This will result in increased water losses and power consumption and in decreased current efficiencies. Overheating or charring of membranes can also result from excessive current densities. For efficient operation and prevention of equipment deterioration therefore, it was necessary that these limiting current densities not be exceeded.

In these experiments an additional dilute stream reservoir was added to the system rather than continually recycling into the same reservoir. The "synthetic urine" solution was pumped from reservoir A, through the test unit, to reservoir B. When "A" was emptied, the two reservoirs were interchanged so that the partially demineralized solution was then pumped from "B" to "A", and so on until the solution approached the required degree of demineralization. In this manner, an influent solution of constant concentration was continuously being fed to the test unit and the effluent solution concentration was being monitored with a conductivity cell. A steady state condition was achieved when the effluent concentration became constant with time. During each pass of the "synthetic urine" solution through the test unit, the influent concentration and flow rate were held constant while the current was gradually increased.

A plot of E/i versus $1/i$ for each pass revealed a break in the curve which indicated the limiting current for the particular solution concentration and flow rate involved (see Figure 7).

where E = voltage drop across the 5 cell pairs

i = current density.

In this manner, the limiting current density was determined for several combinations of flow rates and concentrations. From this information the relationship between limiting current density, solution normality and influent flow rate was calculated. This relationship is shown in Figure 8.

The next electro dialysis stack configuration tested was termed Stack Type B. It was very similar to Type A except that the ion-exchange resins in the electrode compartments were replaced with expanded plastic spacers in order to reduce the flow resistance in these compartments where gas was being evolved. The results obtained in a typical run (Run B-1) on "synthetic urine" are given below:

Initial normality	= 0.916 (54,000 ppm)
Final normality	= 0.026 (1560 ppm)
Water recovery	= 61%
Current efficiency	= 46%
Energy consumption	= 44 watt-hrs/liter

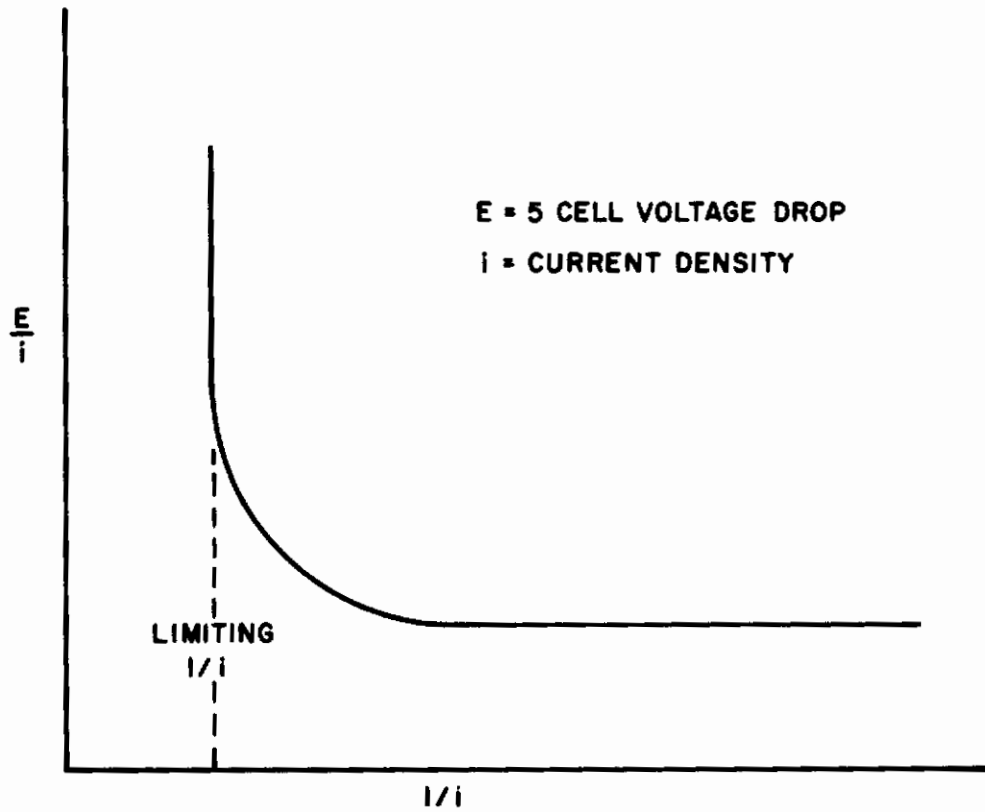


FIGURE 7
LIMITING CURRENT DENSITY STUDY

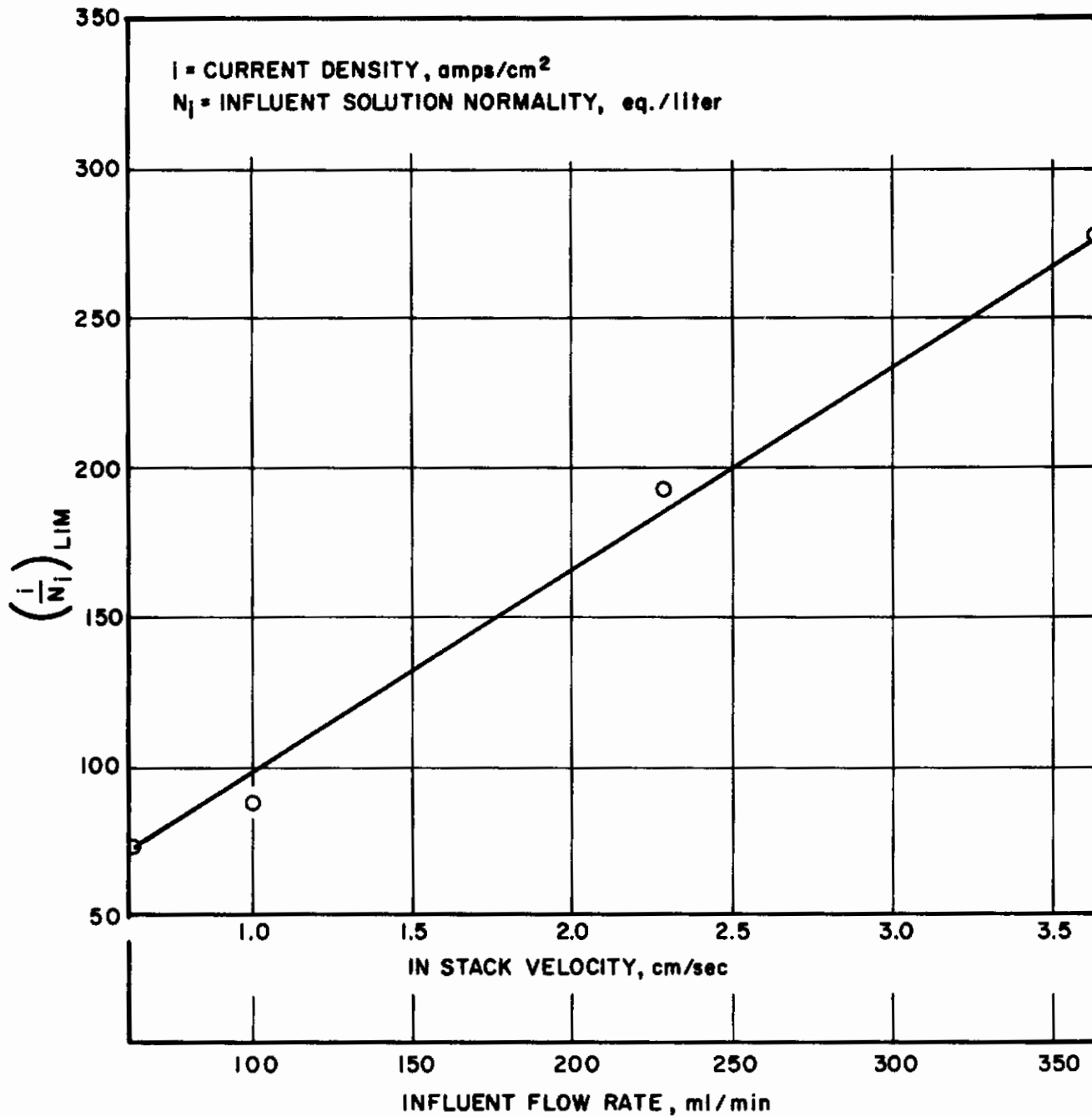


FIGURE 8
LIMITING CURRENT DENSITY RELATIONSHIP

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Comparison of these values with those obtained for Run A-2 indicated that although water recovery was not improved, the energy consumption figure had been reduced. This was an unexpected result since the energy consumption figures should not have been very different. The most reasonable explanation, based on past experience with resin-filled cells, was that slight variations in packing procedures could produce relatively large differences in voltage drops across cells.

An additional run with this stack configuration (Run B-2) was made in order to collect gas samples from the anode and cathode streams. Gas flow from the cathode stream was measured at approximately 110 ml/min at room temperature with 15 amps being maintained through the stack. Gas flow from the anode stream was measured at about 1/5 of the cathode gas flow.

A chromatographic analysis of the two gas streams showed the cathode stream to be essentially all hydrogen gas with a slight amount (8%) of oxygen. The anode stream was essentially oxygen with a trace (1%) of carbon dioxide. No chlorine was detected in the anode stream.

The oxygen volume produced would be expected to be 1/2 the hydrogen volume produced solely on the basis of electron transfer. The volumetric solubility of oxygen in water is approximately twice that of hydrogen. The combination of these two factors would explain the difference in the gas volumes collected.

Following these experiments, a new stack configuration (Type C) was tested in which the ion-exchange resins were removed from the remaining compartments of the Type B stack. The resins were replaced with expanded plastic spacer material of the type used in the electrode compartments. It was important to prove that this type of stack operated as well as Stack Types A and B in order to eliminate the use of ion-exchange resins. These resins were difficult to pack between membranes and the reproducibility of packing was poor unless extreme care was taken.

Run C-1 was conducted in the same manner as Run A-4 to obtain polarization information on this new configuration. The information obtained indicated that the limiting current density relationship was still the same as that shown in Figure 8. These results indicated that no essential performance differences existed between stack Types B and C so that the further use of ion-exchange resins was obviated.

The membranes used in all the experiments on electrodialysis stack Types A, B and C were Ionics' standard cation and anion-transfer membranes. These were the type of membranes used in Ionics' commercial water demineralizers. However, in conjunction with other company activity, ion-transfer membranes had been developed which exhibited lower endosmotic water transfer rates, apparently due to their having smaller average pore size. These membranes are referred to as "tight" membranes. Since the volume of water which transferred endosmotically from the dilute to concentrate compartments represented the major loss of water, an electrodialysis stack was constructed using these low water-transfer membranes. This configuration was identical in all other respects to that shown in Figure 6 and was termed Stack Type D.

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Four runs were conducted on Stack Type D still using "synthetic urine" as the dilute stream feed solution. Results of two of these runs (Runs D-2 and D-4) are presented in Table 12. For the sake of comparison the data already presented for Runs A-2 and B-1 are included. It was obvious from the data obtained that the use of "tight" membranes had resulted in much higher water recoveries (up to 85%).

TABLE 12
SUMMARY OF ELECTRODIALYSIS RUNS

Run No.	A-2	B-1	D-2	D-4
Stack Type	A	B	D	D
Operation	Batch	S.S.*	Batch	S.S.*
Flow Rate, ml/min	400	267	185	210
In Stack Velocity, cm/sec	4.0	2.7	1.9	2.1
Initial Concentration, eq/l	0.916	0.916	0.916	0.916
ppm	54,000	54,000	54,000	54,000
Final Concentration, eq/l	0.022	0.026	0.065	0.028
ppm	1,320	1,560	3,900	1,680
% Water Recovery	63	61	77	85
Current Efficiency (%) **	50	46	68	76
Energy Consumption, watt-hrs per liter treated	79	44	81.5	

* S.S. operation indicates steady state.

** Current efficiencies calculated on water recovered basis.

Energy consumption values for Run D-4 were not included because of a lack of sufficient voltage measurements.

It should be noted here that the energy consumption values listed in Table 12 were higher than expected. This was due to the fact that all runs were conducted with some polarization present, especially at the latter stages of the demineralization process. The runs, therefore, were less efficient from an energy consumption standpoint than they would be in a practical application.

Up to this stage of the experimental program all electrodialysis runs had been conducted using "synthetic urine" as the dilute feed. These experiments were necessary in order to obtain the basic information required for the electrodialysis of urine, such as current and voltage relationships, cell resistances as a function of stream normalities, and limiting current density to normality ratios as a function of flow rates. Two additional pieces of information were necessary before a final electrodialysis stack design could be prepared. One was to operate a stack so that no polarization occurred, and the other was to determine what effect, if any, real urine had on stack operation. To obtain this information a new electrodialysis

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stack was constructed (Type E). It contained "tight" ion-transfer membranes, tortuous path spacers between membranes, and ten cell pairs between electrodes, rather than five as shown in Figure 6.

Following the determination of limiting current density relationships for this stack configuration, a run (Run E-2) was conducted on a batch basis on a "synthetic urine" feed. At all times during this run, the current density was maintained well below polarization conditions. The results are summarized below:

Initial normality	= 0.916 (54,000 ppm)
Final normality	= 0.016 (960 ppm)
Flow rate	= 375 ml/min
In-stack velocity	= 10.75 cm/sec
Water recovery	= 79%
Current efficiency	= 77%
Energy consumption	= 68 watt-hrs/liter

When compared with the results for Run D-2 in Table 12, it can be seen that the energy consumption has been reduced about 20% by eliminating polarization.

In another experiment on this stack (Run E-5), human urine was utilized as the feed solution. Samples were collected, reacted with urease, heated to 95°C for two hours, filtered, and then clarified by passing through a bed of coconut charcoal. The heating step resulting in the precipitation of calcium carbonate, which has an inverse temperature-solubility relationship, such that the calcium level was lowered to below detectable limits. In addition, heating to 95°C also served to pasteurize the solution for any pathogenic organisms that were present. Run E-5 was carried out successfully with the total salt content lowered to less than 500 ppm. At this low level the ammonia present was still slightly detectable due to its low odor threshold. Therefore, a small mixed bed ion-exchange column containing 10 ml of wet settled resin was used as a final clean up step. A charcoal column containing the same volume was also tried. Both methods removed the residual ammonia below any detectable taste or odor level.

The product from the electrodialysis stack with and without the post-treatment steps were compared on a Beckman Recording Spectrophotometer. Once the interfering ions such as ammonium and chloride were removed from the urine, ultraviolet absorption could be used as a measure of the organic material present. Figure 9 depicts the results of this test. It will be noted that the spectra of the product from the small charcoal column lies between laboratory tap water and distilled water.

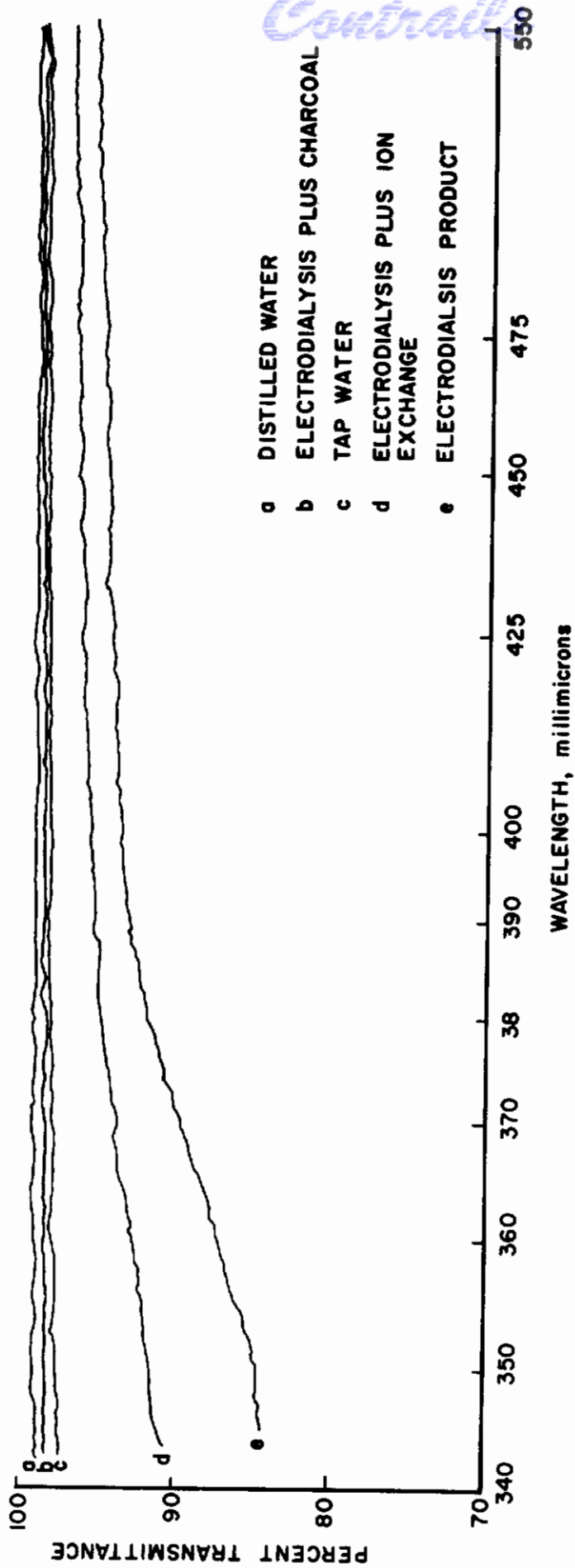


FIGURE 9
ABSORPTION SPECTRA OF URINE AFTER ELECTRODIALYSIS

3. OPERATION OF A LABORATORY MODEL OF A URINE RECLAMATION UNIT

In view of the encouraging results obtained for the demineralization of urine in electro dialysis stack configuration Type E, a batch-operated, laboratory pilot model of a urine reclamation system was designed and constructed. The electro dialysis portion of this unit was designed to recover potable water from urine at a rate of approximately 30 ml/min. A flow diagram for this system is illustrated in Figure 10; the electro dialysis stack configuration (Stack Type F) is presented in Figure 11. A photograph of the urine reclamation system model is presented in Figure 12.

The system operated as follows:

1. Pretreatment - Urine was reacted with urease, filtered, heated to 95°C for two hours for sterilization and calcium precipitation, refiltered and placed in storage vessel (A).
2. Charcoal adsorption - The urease-treated urine was pumped at 30 ml/min through an activated charcoal column (B) (to remove color and organic residue) to the dilute reservoir (C). Approximately 200 g. of charcoal per liter of urine permitted some factor of safety. In cases where no urease step was used, urine was pumped at 30 ml/min through approximately 400 g. of charcoal per liter of urine.
3. Electrodialysis - The urine was circulated from the dilute reservoir (C) through the 10-cell pair electro dialysis stack (D) and back to the dilute reservoir. The circulating flow rate was approximately 250 ml/min. Direct current was 5.8 - 6.0 amps initially and dropped to 0.5 - 0.8 amps finally, when the voltage across the stack electrodes was maintained at 12.4 volts. Urine was demineralized at 40-50 ml/min, depending on initial concentration.
4. Final clean up step - When the salt concentration was reduced to below 500 ppm, as indicated by conductivity monitoring cell (F), the demineralized urine was shunted through a small, mixed-resin and charcoal column (G) at 30 ml/min to the product reservoir (H). 5 ml of wet ion-exchange resin (mixed anion and cation) and 5 ml of activated charcoal per liter of processed urine removed the last small traces of ammonia and organic material with a factor of safety greater than 2.
5. Concentrate and Electrode Streams - An initial quantity of charcoal adsorbed urine was placed in the concentrate reservoir (E). In the test operations of the unit, 200-250 ml of initial concentrate volume were used per liter of initial dilute stream volume. The concentrating stream was circulated at approximately 250 ml/min through the electro dialysis stack and back to the concentrate reservoir. The electrode stream was also circulated from this reservoir, through the anode and cathode compartments of the stack and back to the reservoir. Electrode stream flow rate was approximately 140-150 ml/min.

Ten electro dialysis runs were completed on the laboratory pilot model. These runs are summarized in Table 13.

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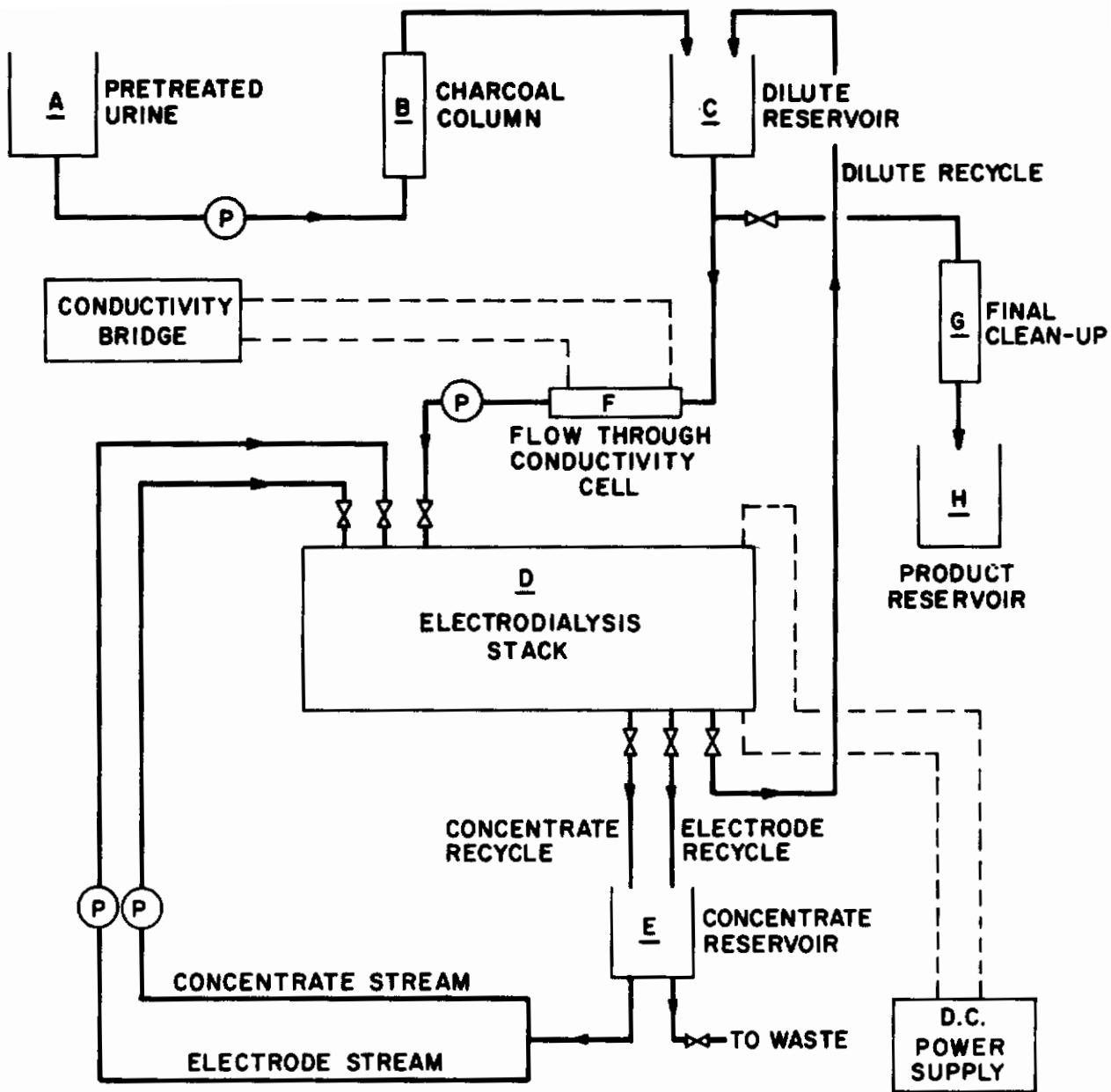
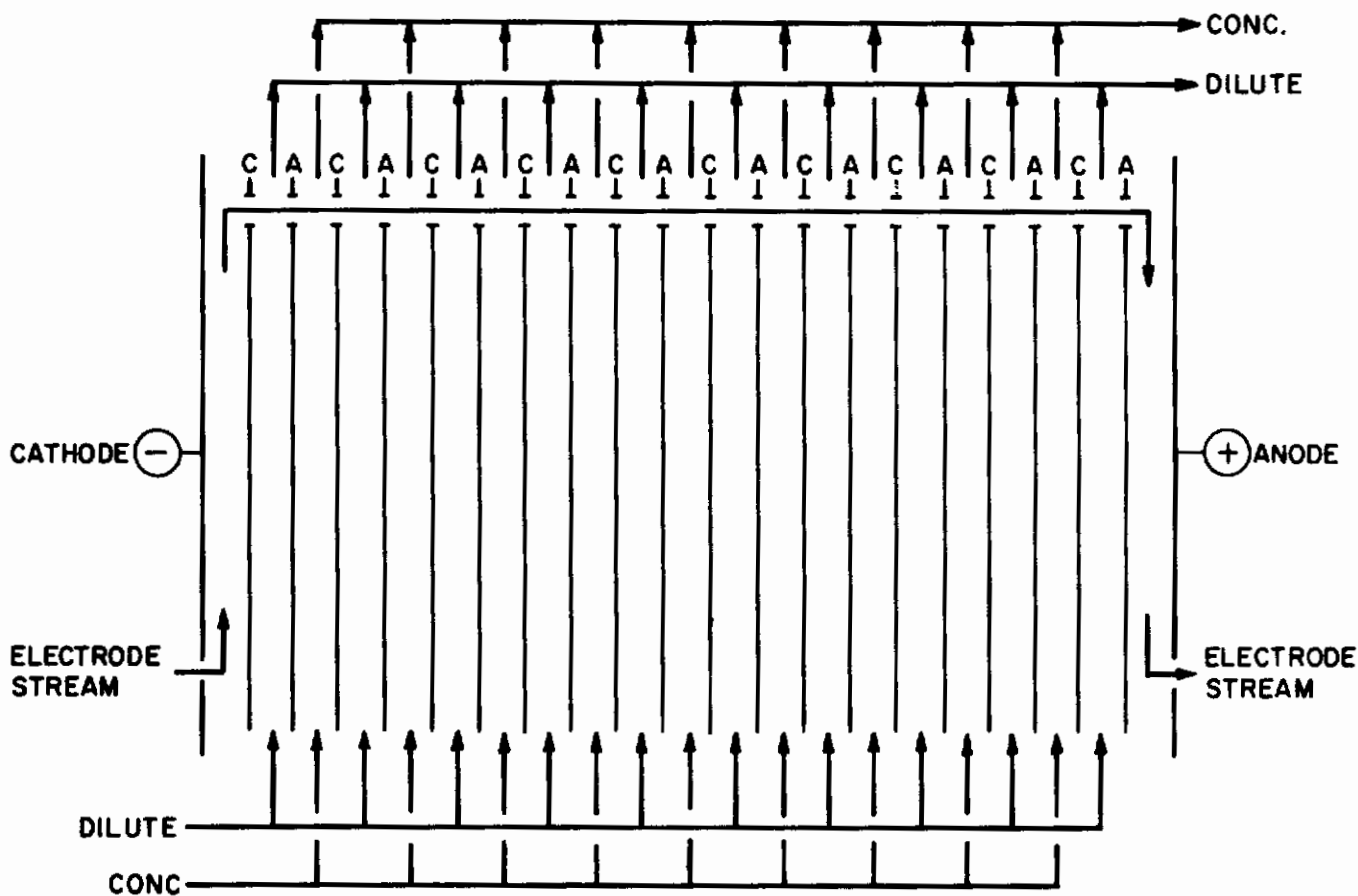


FIGURE 10
FLOW DIAGRAM
LABORATORY PILOT PLANT



C = CAT.ION MEMBRANE CR 61-DYG
 A = ANION MEMBRANE AR III-DYG

NO. CELL PAIRS = 10
 CELL SPACING = 0.040 IN.
 FLOW PATH WIDTH = 0.25 IN.
 FLOW PATH LENGTH = 12 FT.
 EFFECTIVE MEMBRANE AREA = 32.81 SQ. IN.
 = 212 SQ. CM.

FIGURE II
PILOT PLANT ELECTRODIALYSIS STACK CONFIGURATION
STACK TYPE F

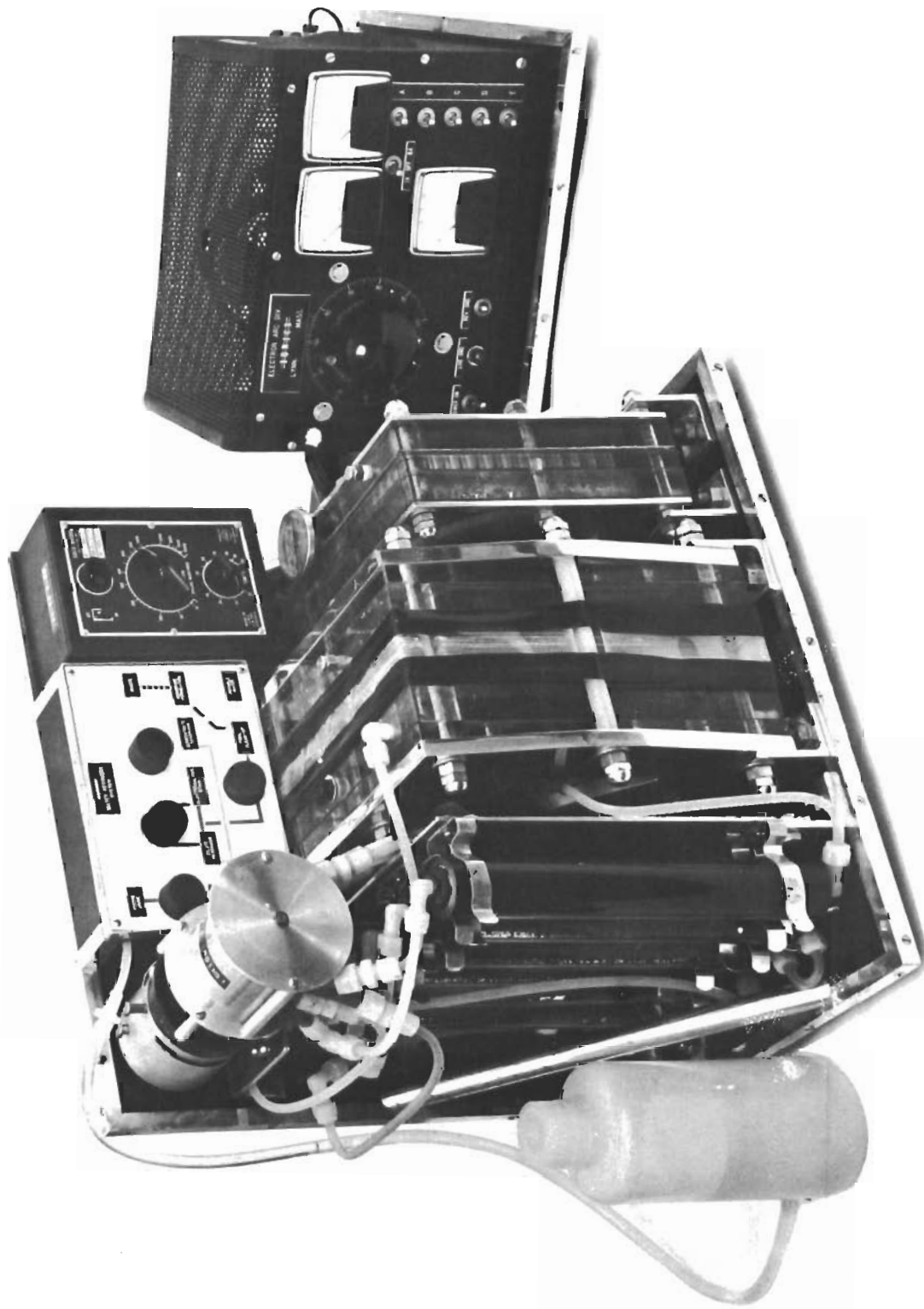


FIGURE 12
MODEL OF URINE RECLAMATION SYSTEM

TABLE 13
PILOT PLANT RUNS
(Batch Operation)

Run No.	F-1	F-3	F-4	F-5	F-6	F-7	F-9	F-10
Solution	"Synth"	"Synth"	"Synth"	"Synth"	"Synth"	"Synth"	Urine A ¹	Urine B ²
Dilute Volume, ml								
Initial	750	750	500	500	750	750	500	500
Final	453	500	300	332	330	480	460	407
Dilute Normality, eq/l								
Initial	0.916	0.916	0.916	0.916	0.916	0.916	0.125	0.425
Final	0.013	0.011	0.007	0.013	0.014	0.013	0.012	0.0015
Concentrate Volume, ml								
Initial	250	250	150	200	150	200	200	200
$\frac{\text{Initial Dilute Volume}}{\text{Initial Conc. Volume}}$	3.0	3.0	3.33	2.5	5.0	3.75	2.5	2.5
Total Stack Energy Consumption,								
$\frac{\text{watt-hours}}{\text{l. recovered}}$	56.9	70.8	66.8	38.4	65.0	53.7	4.56	5.33
Current Efficiency, %	43.5	48.8	37.7	66.0	37.3	46.0	66.4	63.4
Dilute Stream Water Recovered, %	60.5	66.6	60.0	66.4	44.0	64.0	92.0	90.0

(1) Urine A: Human urine passed through activated charcoal column only.
Two data points are presented for this run.

(2) Urine B: Human urine, urease reacted and otherwise pretreated as described.

Contrails

In previous runs (see Table 12), energy consumption data which was reported did not include electrode energy requirements. Data was reported in this manner so that design information could be extrapolated for variable numbers of cell pairs. It is to be noted, however, that the energy consumption figures of Table 13 are total stack energy consumption values: i.e., electrode energy consumption is included. The stack configurations used throughout this program have had total energy consumptions of approximately twice the values which were reported in Table 12. Energy consumption values from Table 13 should, therefore, be halved before comparing them with any previously reported runs. However, even including the electrode energy consumption, the total energy consumption values for Stack Type F are significantly lower than the values previously obtained. This was due to certain innovations in the techniques of preparation of the ion-transfer membranes, such that their electrical resistances were greatly reduced while still maintaining the desirable properties of high capacitance and low endosmotic water transfer rates.

In Run F-1, voltage was initially set at 10 volts and gradually decreased to 7.5 volts at the end of the run. Initial current was 5 amps and decreased to a final value of 0.3 amps. Initial dilute/concentrate volume ratio was 3 to 1. During this run, a discrepancy in the total water balance was noted.

A second run (F-2) was made to determine the location of the apparent leakage. No current was applied during this run. The concentrate stream volume was found to be diminishing by 1 to 3 ml/min, the leakage primarily occurring at a packing gland in the concentrate recycle pump. It was decided that this leakage would not interfere greatly with energy consumption determination, the primary purpose of this series of runs, so no corrective action was taken.

Run F-3 was conducted similarly to Run F-1 except higher voltages (24 volt maximum) were used. It can be seen from Table 13 that, although current efficiency and water recovery were slightly improved from Run F-1, energy consumption was greatly increased.

Runs F-4, 5, 6, and 7 were conducted in the same manner as Run F-1. The variable in these runs was the initial dilute/concentrate volume ratio. This ratio was, in fact, a measure of final concentrate stream normality. As the ratio increased, efficiency was impaired due to back-diffusion of ions during the final stages of demineralization. The results from Run F-5, which used the lowest ratio, illustrated this factor clearly.

Run F-8 was a polarization study. It was determined that a constant 10 volts could be applied throughout a run without incurring serious polarization problems. Subsequent runs were then conducted at this constant voltage.

Runs F-9 and F-10 were conducted to evaluate pretreatment methods using human urine. For Run F-9, human urine was passed through a column of activated charcoal (400 g. per liter of urine) as the pretreatment step. For Run F-10, the urine was subjected to the complete pretreatment process (urease reaction, heating, filtration, charcoal). The results of these

Contrails

runs indicated that a 50% saving in energy consumption could be realized at the cost of regenerating twice as much charcoal. In addition, the water recovery for urine pretreated with only charcoal was much greater than that obtained with the urease plus charcoal pretreatment.

It can be seen in Table 13 that the runs using real urine (F-9 and F-10) had higher water recoveries and lower energy consumptions than the runs on "synthetic urine". This was obviously due to the higher initial salt concentrations in the latter. Since the "synthetic urine" was prepared on the basis of the normal concentration of components in urine (see Table 1), apparently the urine used in Runs F-9 and F-10 was more dilute than normally expected. Additional analyses were done on samples of collected urine. In all cases, the concentrations of salts and urea were below those listed for the average male urine.

SECTION IV

DISCUSSION AND SUMMARY

1. EVALUATION OF URINE RECLAMATION SYSTEMS

Based on the experimental results obtained during this investigation, two urine reclamation systems were selected for further evaluation. Each system had been shown to be feasible in terms of recovering potable water from urine. The sequence of operations of the two systems are presented below:

<u>System A</u>	<u>System B</u>
1. urease addition	1. charcoal adsorption
2. heating	2. electrodialysis
3. filtration	
4. charcoal adsorption	
5. electrodialysis	
6. charcoal clean up	

Both systems require pretreatment steps for the removal of nonelectrolytes. Electrodialysis experiments conducted on raw urine clearly indicated that over 95% of the initial urea concentration remained in the demineralized product water. The urea which was removed probably was transferred across the membranes dissolved in the endosmotic water rather than by ion-transport mechanisms. Moreover, these experiments were conducted in electrodialysis stacks containing "standard" membranes. If similar experiments were conducted in stacks containing "tight" membranes, the amount of urea removal by electrodialysis would be even less. Since urea, which is a relatively small molecule and has a low, but measurable, ionization constant, was not removed by electrodialysis, it is very unlikely that the other nonelectrolytes were removed to any significant degree.

The pretreatment techniques employed in the two proposed systems differ greatly. In System A the initial step is the addition of soluble urease powder which enzymatically converts urea to ammonium carbonate. Although this step, in itself, involves very low weight requirements (2 grams of urease per liter of urine), it greatly affects the subsequent electrodialysis operation. The urease-urea reaction increases the total dissolved electrolyte concentration in urine from about 15,000 ppm to 54,000 ppm. The higher the salt concentration is in the feed to the electrodialysis stack, the higher the energy consumption is

and the lower the yield of potable water.

The next step in System A involves heating the solution to 95°C. This results in the precipitation of calcium carbonate and also serves to sterilize the urine. After filtering to remove the calcium precipitate, the solution is percolated through a coconut charcoal bed to remove the residual non-electrolytes. It was determined that approximately 200 grams of charcoal were required per liter of urine.

The urine, now free of nonelectrolytes, is fed to an electro dialysis stack where demineralization takes place. The electrolyte concentration is lowered from 54,000 ppm to 100-200 ppm. While this salt concentration is well within public health service standards for drinking water, the water would have poor taste and an objectionable odor because of the relatively high residual ammonia concentration. Because of this a post-treatment step is necessary to produce acceptable water. This step consists of passing the demineralized stream through a small charcoal bed.

On the other hand, System B is much simpler because it consists of only one pretreatment and no post-treatment steps. The pretreatment step involves the percolation of raw urine through a coconut charcoal bed to remove all the nonelectrolytes. It was determined that 400 grams of charcoal are required per liter of urine. Sterilization of the urine in System B is accomplished by passing the demineralized product water over an ultraviolet lamp.

In System B the feed solution to the electro dialysis stack has a salt concentration of approximately 15,000 ppm. The concentration after demineralization is about 100 ppm. The product water contains negligible ammonia and therefore requires no further treatment.

2. SYSTEM SELECTION

A comparison of the more important features of Systems A and B is presented below based on the treatment of 3000 ml of urine per day.

	<u>System A</u>	<u>System B</u>
Urease requirement	6 g/day	0
Charcoal requirement	600 g/day	1200 g/day
Electrodialysis energy requirement	app. 120 watt-hrs.	app. 20 watt-hrs
Potable water yield	80%	92%
Post-treatment requirement	yes	no

The only apparent disadvantage to System B is that twice as much charcoal is required than for System A. If we assume that the charcoal will be regenerated for either system, then for missions over fourteen days in duration the yield of potable water becomes the limiting weight consideration. It seems clear that the charcoal will have to be regenerated in either system because of the large quantities involved. To illustrate this a table (Table 14) has been prepared showing the take-off weights of charcoal and make-up water as functions of the reclamation system employed, the mission duration, and whether or not charcoal is regenerated. This table is based on the treatment of 3000 ml of urine per day.

TABLE 14
WEIGHT REQUIREMENTS*

	<u>System A</u>		<u>System B</u>	
	<u>14 day mission</u>	<u>28 day mission</u>	<u>14 day mission</u>	<u>28 day mission</u>
<u>No Charcoal Regeneration</u>				
Charcoal weight	18.5 lb.	37.0 lb.	37.0 lb.	74.0 lb.
Make-up water	18.5 lb.	37.0 lb.	7.4 lb.	14.8 lb.
Total	37.0 lb.	74.0 lb.	44.4 lb.	88.8 lb.
<u>Charcoal Regeneration</u>				
Charcoal weight	1.3 lb.	1.3 lb.	2.6 lb.	2.6 lb.
Make-up water	18.5 lb.	37.0 lb.	7.4 lb.	14.8 lb.
Total	19.8 lb.	38.3 lb.	10.0 lb.	17.4 lb.

* Take-off weight requirements based on potable water yields from urine of 80% and 92% and charcoal requirements of 600 g/day and 1200 g/day for Systems A and B respectively.

Therefore, except for very short missions (less than four days) System B has a lower total weight requirement, assuming charcoal regeneration, because of its having a higher potable water yield.

In all other respects System B is superior to System A. Energy requirements are much less and the system is inherently simpler. In addition, raw urine can be processed immediately, while in System A both the urease and heating steps delay the start of the electro dialysis operation.

Thus, in view of its many advantages, System B has been selected as the optimum system for the recovery of potable water from urine using membrane electro dialysis.

3. ANALYSIS OF SELECTED SYSTEM

On the basis of the experimental results obtained during this program, a system for the recovery of potable water from urine has been selected. The system contains two main processes; pretreatment of urine with charcoal to remove the nonelectrolytes in urine, and membrane electro dialysis to remove the electrolytes. A schematic flow diagram is presented in Figure 13. The

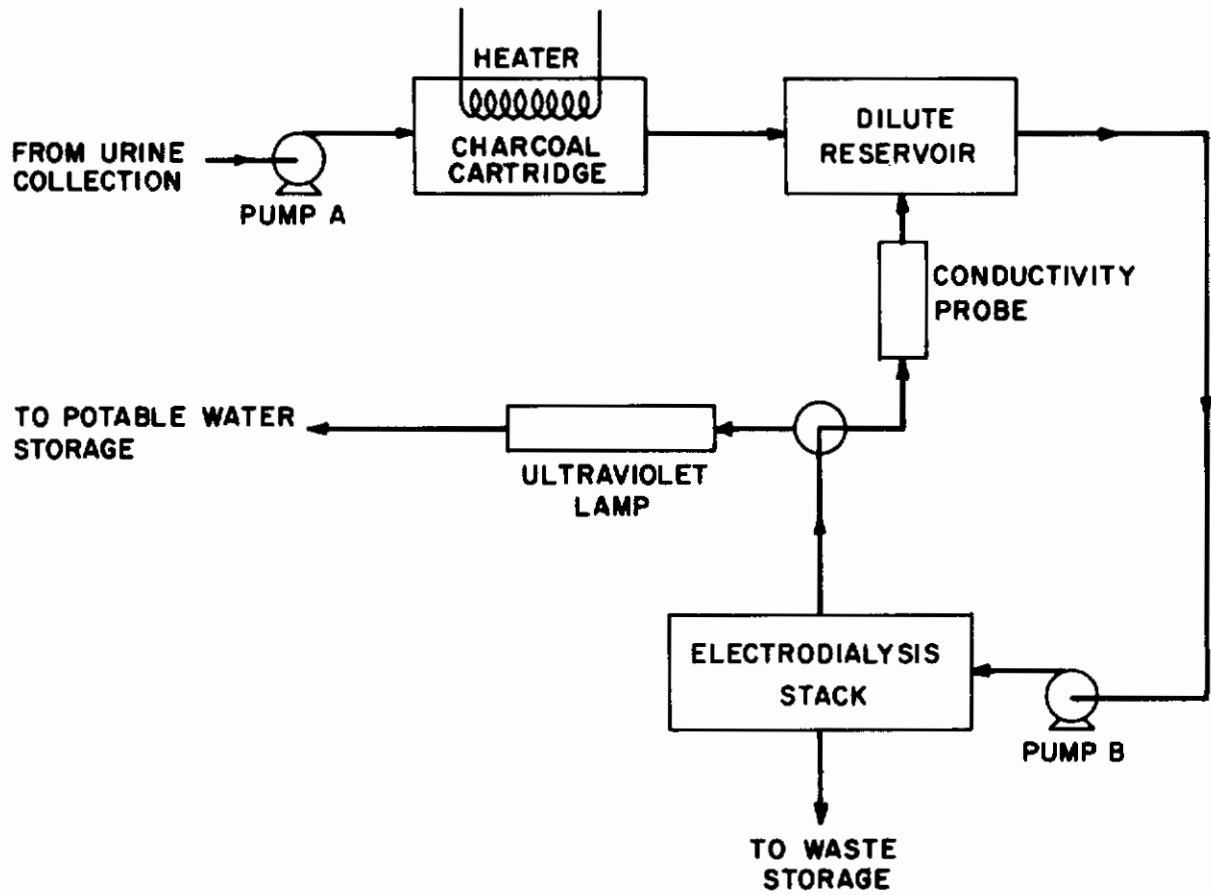


FIGURE 13
PROPOSED URINE RECLAMATION SYSTEM

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system has been shown to be feasible in that excellent quality water has been recovered from human urine in high yield and low energy consumption. The discussion which follows will be an evaluation of the merits of this system for aerospace applications. The criteria which will be applied are weight, volume, and energy requirements, reliability, component replacement, and zero-gravity operation. This discussion is based on the recovery of 3000 ml of urine per day for mission lengths in excess of fourteen days.

The system as shown in Figure 13 contains seven main components:

- a. Pump A, which pumps urine from its storage container through the charcoal adsorber to the dilute reservoir.
- b. Pump B, which recirculates the electrodialysis feed batch until the desired demineralization has occurred. At this time, the product stream is pumped across the ultraviolet lamp to the potable water reservoir.
- c. Charcoal adsorber cartridge containing a heating element for regeneration purposes.
- d. Dilute reservoir.
- e. Electrodialysis stack.
- f. Conductivity probe, which is used to monitor the degree of demineralization.
- g. Ultraviolet lamp for sterilization of the product stream.

In order to minimize the water inventory aboard the space vehicle, it is proposed that the urine reclamation system be operated four times a day for two hours each time. During the nonoperating period, the charcoal can be regenerated. This means that each raw urine batch will be approximately 800 ml and the charcoal required will be 320 grams.

The weight of the system can be estimated as follows:

Electrodialysis stack	8.0 lbs.
Pumps	1.5
Charcoal cartridge (full)	1.5
Dilute reservoir	0.5
Ultraviolet lamp	0.2
Conductivity probe	1.3
Valves and piping	2.0
	<hr/>
	15.0 lbs.

Contrails

Thus, the weight of the proposed urine reclamation system is about 15.0 lbs not including spare parts, power supply, or controls if automation is desired. The spare parts would include mainly extra charcoal cartridges, assuming that regeneration is not complete and, therefore, the cartridges should be changed every seven days. For missions over thirty days in length, spare ion-transport membranes would be included in the spare parts inventory.

The total volume of the system is less than 1 cubic foot. The laboratory model discussed previously (see Figure 12) was about 1 cubic foot in size but it had a higher urine reclamation capacity, and no serious effort had been made to minimize the size of components.

Based on treating 800 ml of urine in two hours, the power requirement of the electrodialysis stack is about 30 watts at the start of the demineralization process, or 24 watt-hours per day. The ultraviolet lamp requires 6 watt power. The pumps and conductivity probe require about 15 watts. An additional 40 watts are required for charcoal regeneration. However, not all of the various components operate simultaneously. The maximum power requirement occurs at the start of the demineralization process and amounts to approximately 45 watts (30 watts for the electrodialysis stack and 15 watts for the pumps and the conductivity probe). The total energy requirement per day can be calculated as follows:

<u>Component</u>	<u>Power</u>	<u>Operating Time</u>	<u>Energy</u>
Electrodialysis stack	30 watts (max)	6 hrs	24 watt-hrs
Pumps and Conductivity Probe	15	8	120
Ultraviolet Lamp	6	1	6
Charcoal Regeneration	40	4	160
			<u>310 watt-hrs</u>

The reliability of this system should be extremely good because of its inherent simplicity. This has been demonstrated in operations with the laboratory model. For example, no water is released to the potable water storage container until all the water is demineralized. Therefore, the quality of potable water is not only very high at all times, but is reproducible in that the quality is independent of variations in urine batches. In other urine reclamation systems the nonreproducibility of water quality has been a serious problem.

A study was undertaken to determine whether or not ion-transfer membranes deteriorated after prolonged exposure to urine. Samples of the membranes were placed in raw urine for periods up to three months in duration. At the conclusion of this time interval, the physical and chemical properties of these samples were measured and compared with samples stored for the same time in distilled water. Very little deterioration in properties was observed for the samples stored in urine.

Contrails

The one additional safety feature, which might be required to ensure high reliability, is a device to measure the organic content of the stream leaving the charcoal cartridge. This device could be a colorimeter set at one wavelength in the ultraviolet range to measure the transmittance of the effluent stream. In the experiments to date, this has not been necessary since an excess of fresh charcoal was always used for organic removal.

The final criteria to be discussed is the operation of this system under conditions of weightlessness. Electrodialysis is inherently a gravity-independent process. During the experimental program, the electrodialysis stacks were operated in many orientations with no decrement in performance. In addition, none of the other operations in the proposed system appear to be dependent upon gravity. The only possible problem area concerns the effluent electrode streams from the electrodialysis stacks. These are gas-liquid streams and, therefore, it will be necessary to include gas-liquid separators in the system in order to operate under conditions of weightlessness. Ionics has built gravity-independent gas-liquid separators for this purpose. These separators have worked efficiently in different orientations for the removal of the gases from the electrode liquid streams.

A summary of the essential features of Ionics' Urine Reclamation System using membrane electrodialysis is presented in Table 15. The numbers in this table are based on the recovery of 3000 ml of potable water from urine for aerospace missions in excess of fourteen days.

TABLE 15

SUMMARY OF IONICS' URINE RECLAMATION SYSTEM*

Weight	=	15.0 lbs.
Volume	=	less than 1 ft ³
Power Requirements (Peak)	=	45 watts
Potable Water Yield	=	92%
Water Reproducibility	=	Excellent
Reliability	=	Excellent
Pretreatment	=	Yes
Post-treatment	=	no
Operation in Zero-Gravity	=	Feasible

* System designed to recover 3000 ml of urine per day.

SECTION V

CONCLUSIONS

1. The use of membrane electrodialysis for the recovery of potable water is feasible for aerospace applications. Excellent quality water is produced in yields of 92%. The system selected has low weight (15 lbs.), volume (less than 1 ft³) and power requirements (45 watts) for the recovery of 3000 ml. of urine per day. Reliability and water quality reproducibility are excellent and operation under conditions of weightlessness appears feasible.
2. The system must contain a pretreatment step for the removal of nonelectrolytes. The use of coconut charcoal (400 g. per liter of urine) is recommended. No post-treatment step is required.
3. For missions in excess of fourteen days, charcoal must be regenerated in order to avoid large take-off or resupply weight penalties.
4. The use of urease to hydrolyze urea is feasible. Two grams of "soluble" urease powder (1500 Sumner Units/gram) per liter of urine are sufficient to hydrolyze at least 98% of the urea in urine within two hours. This step must be followed by charcoal adsorption to complete the removal of nonelectrolytes. However, these pretreatment steps are not as suitable as the use of charcoal alone for the removal of nonelectrolytes.
5. The use of hypochlorite bleaches to oxidize organic compounds is of little value for the proposed applications.

SECTION VI

TECHNICAL RECOMMENDATIONS

In view of the encouraging results obtained in this feasibility study, the following recommendations for future technical work are included:

1. Additional work should be undertaken in the production of even "tighter" ion-transfer membranes. Based on Ionics' experience in this field, the chances seem excellent of producing membranes which exhibit extremely low endosmotic water transfer rates while retaining their satisfactory strength, capacity and electrical resistance properties. The use of these membranes would enable the proposed system to obtain potable water yields of at least 95%.
2. Additional investigations should be performed in the area of optimizing the charcoal adsorption step. There are very good indications that the quantity of charcoal required to remove urea and other organics can be much less than 400 grams per liter of urine. All charcoal adsorption experiments performed to date have been done in a single pass column. This is the least efficient manner of operation in terms of obtaining minimum charcoal requirements, but adequate for providing organic-free solutions for the subsequent electrodialysis runs. Studies have shown that charcoal requirements can be sharply reduced by (1) increasing the liquid-solid contact time, (2) increasing the length of the charcoal column in a constant volume system, and (3) placing a number of columns in series, regenerating only one column at a time. By the use of such techniques, it is believed that the quantity of charcoal required to remove all the urea and other organic constituents from fresh urine can be reduced to about 200 grams per liter.
3. Since the ability to regenerate charcoal represents a significant reduction in weight penalties, an investigation should be conducted to determine the optimum method of charcoal regeneration. Techniques presently used in industry appear to be unsuitable for aerospace applications because of the high temperatures involved (600°C). Exploratory experiments conducted at Ionics have indicated that coconut charcoal can be 75% regenerated by heating the bed to 200°C and applying a low vacuum.
4. The only portions of the entire urine reclamation system which are presently gravity-dependent are the gas-liquid separators, which handle the effluent electrode streams from the electrodialysis stack. Results of preliminary investigations of a gas-liquid separator designed to operate under conditions of weightlessness have been encouraging. However, more work is required to assure the reliability of these components.
5. Either on a state-of-the-art basis or subsequent to the future experimental programs described above, a full-scale prototype of the urine reclamation system should be constructed. This should be designed to minimize weight, volume, and power requirements. The prototype can then be tested to determine the life of components, reliability, decrement in performance, and other factors which are almost impossible to predict without actually operating a full-scale prototype.

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