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"Continuous Potable Water Supply for Fallout Shelters and Other Isolated Sites"

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INTRODUCTION

One possible method of urine purification that requires no external source of power is the alteration of urine by purely chemical means. Most of the research in urine purification has been aimed at producing a potable water through the utilization of techniques such as distillation, freeze-drying, and electrolysis. The relative advantage of a given process depends upon the characteristics of the feed, the desired quality of the effluent, and the amount of power available. The quality required for a potable water supply differs considerable from that needed for a supply intended for other purposes such as bathing.

The chemical and physical characteristics of urine vary with the diet of the individual and the length of time elapsed between excretion and analysis. For example, the excretion of urea ranges from 4.7 gm/day for persons on a low protein diet to 31.5 gm/day for persons on a high protein diet (1); ammonia may be present in large amounts as a result of the decomposition of urea. Table 1. gives a partial listing of the constituents of urine and their respective average daily quantities excreted by normal healthy individuals. Any listing of the constituents is necessarily incomplete because of the presence of many trace compounds and the difficulties associated with microanalysis for these compounds. The two gross constituents are urea and sodium chloride. Other materials which are excreted in aqueous

Table 1. Analysis of Normal Human Urine

Constituents	Grams/1250 cc.
urea	30
creatinine	1.15
ammonia	0.7
uric acid	0.7
hippuric acid	0.7
amino acids	0.5
glucose	0.3
aromatic hydroxy acids	0.06
oxalates	0.015
sodium chloride	15.0
phosphates (as P_2O_5)	2.5
inorganic sulfates (as SO_3)	2.0
organic sulfates (as SO_3)	0.3
neutral sulfur (as SO_3)	0.3
potassium (as K_2O)	2.5
calcium (as CaO)	0.2
magnesium (as MgO)	0.2
silicates	0.4
iron	0.005

greater than one gram per day are creatinine, potassium, phosphates and sulfates.

The investigation currently being conducted involves the alteration of the characteristics of urine using chemical means which require no external source of power and apply under ambient conditions. The entire process being considered involves four steps: urea removal, ion exchange, adsorption, and disinfection.

INITIAL STUDIES

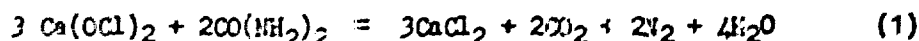
A preliminary investigation was made to determine the extent to which urea and sodium chloride could be removed from urine, utilizing chemical and biochemical reactions and ion exchange.

Reaction of Urea

Oxidation of urea by nitrous acid and calcium hypochlorite, hydrolysis of urea by urease, and precipitation of urea as urea nitrate were explored as possible methods of urine alteration prior to demineralization by ion exchange. The schemes of urea removal were suggested by available methods for the analytical determination of urea in urine. The oxidation of urea by nitrous acid giving water, nitrogen, and carbon dioxide as end products is attractive if the urine is to be subsequently treated by ion exchange. Unfortunately, nitrous acid is known only in solution, and must be prepared as needed by the reaction of ammonia and hydrogen peroxide, sodium nitrite and sulfuric acid, or similar schemes. Urea concentrations in aqueous solution and in urine were determined with the McLean Ureometer (3). As with many reactions involving organic compounds, the oxidation of urea does not proceed quantitatively as outlined by the stoichiometric equation.

Approximately twice the stoichiometric amount of nitrous acid was found to be necessary for the oxidation of urea in aqueous solution and in urine. For 100% removal of urea from an aqueous solution and from urine at concentrations of 20 gm/l. approximately 75 gm/l. of nitrous acid were reacted.

Utilizing calcium hypochlorite, a stronger oxidizing agent, better results were obtained.



Approximately 120% of the stoichiometric amount of calcium hypochlorite was needed for the oxidation at room temperature of urea in aqueous solution. For 100% reaction of urea from a 20 gm/l. solution, approximately 120 gm/l. of calcium hypochlorite were required. The reaction product, calcium chloride, must be precipitated from solution, if ion exchange is used later as a treatment process.

The possibility of precipitating urea as urea nitrate may be dismissed because of its relatively high solubility in aqueous solution.

The hydrolysis of urea by the enzyme urease is most rapid when the solution is buffered at a pH of 7 and the concentration of urea is in the order of magnitude of 1 - 2 gm/l (4). The dependence of hydrolysis of a high concentration of urea (20 gm/l.) as a function of urease concentration, under ambient temperature and with the solution not buffered with respect to pH, is shown below. A 90 percent hydrolysis of urea in 24 hours was observed for urease concentrations ≥ 2 gm/l. The presence of urease and the hydrolysis product ammonium carbonate is a serious disadvantage if ion exchange is to be employed as a subsequent treatment process.

Urease Concentration gm./l.	Percent Urea Hydrolyzed
1.0	72
2.0	92
6.0	88
10.0	90

Ion Exchange

Introduction:

The utilization of ion exchange as a method of treating urine has been explored to a very limited extent. Bonting (5) made use of ion exchange resins in a laboratory analysis of urine. He established the total cation content of urine by removing all of the cations with a small ion exchange column and by determining the quantity of hydrogen ions in the effluent. He reported that better than 99 percent removal was achieved from contacting 0.7 meq. of cations with the exchange resin; he did not consider the capacity of the resin. McKeel (6) attempted to develop a method of waste disposal in aircraft by passing urine through a combined anion-cation exchanger and then by permitting the effluent to be picked up by a large wick for evaporation into a warm air stream. It was found that the resins were exhausted after the passage of 400 ml. of urine. A few additional references have been made in the literature to the use of ion exchange as a final purification technique following distillation or freeze-drying.

The ion exchange resins adopted for use in this study were produced by the Rohm and Haas Company. Amberlite MB 402, a quaternized chloromethylated polystyrene, was chosen as the anion exchanger. It is a strongly basic resin with a moisture content of 40-50% and a suggested capacity of 4.4 meq. per gram (dry basis). Amberlite IRC 50, a poly (methacrylic acid), was

chosen as the cation exchanger. It is a weakly acidic resin with a moisture content of 44-55% and a suggested capacity of 8.0-10.0 meq. per gram (dry basis). It was decided that for the initial studies untreated urine should be the influent to the resins.

Cylindrical glass columns, 24 inches in length with an inside diameter of 1 7/8 inches, were used to hold the resins. At the bottom of each column was placed 3/4 inch of glass wool followed by six or seven glass marbles and by another 3/4 inch layer of glass wool. The resin was then added and occupied approximately 12 1/2 inches of length of the column, 330 grams (dry basis). Another 3/4 inch of glass wool was placed on top of the resin to prevent splattering of the influent and to diffuse the influent over the entire surface of the resin. The influent was delivered at 10 ml. per minute through a 600 ml. separatory funnel attached at the top of the column.

Two columns were operated in series with untreated urine being passed through the column containing IRA 402 (anion exchanger) and with the effluent from the anion exchanger being passed through IRC 50 (cation exchanger). This order was adopted to provide an influent to the IRC 50 with a high pH. The urine was collected from six male adults over a period of several days and was stored at a temperature of 1-4° C. During the storage periods there were no signs of degradation; the color, odor, and pH remained unchanged. The effluents were generally collected in 250 ml. batches and were kept refrigerated until being subjected to analysis.

Analyses were carried out to determine the average characteristics of the influent to the anion exchanger and of the influent to the cation exchanger. Similar tests were applied to a series of effluent samples from

each column. Hydrogen ion concentrations were determined with a Beckman pH meter. Absorptivity measurements were made with a Klett-Summerson photoelectric colorimeter, using a number 42 filter (400-465 m μ). The zero point of the instrument was set with distilled water, and the absorptivity measurements were converted to color units by comparison with standard solutions containing potassium chloroplatinate and cobaltous chloride (7). Total solids were determined by evaporation of the samples to dryness at 103-105° C. Sulfate ion was analyzed by precipitation with barium chloride, followed by ignition of the filter residue at 800° C. Chloride was determined volumetrically using silver nitrate and potassium dichromate, and sodium and potassium ion concentrations were measured with a flame photometer. Calcium was analyzed by a modification of the standard permanganate test and by the EDTA titration method (7). An analysis of untreated urine is presented in Table 2. This represents the average composition of the influent to the anion exchange column.

Experimental Results:

The first series of observations was made by passing four liters of untreated urine through the anion exchanger. The first 2000 ml. of effluent were collected in 250 ml. samples, while the last 2000 ml., after the resin break point had been reached, were collected in 500 ml. samples. The prescribed set of analyses was made on each sample and the results were tabulated on the basis of cumulative volume of effluent (throughout volume). The analysis for each 250 ml. sample was assumed to apply to 125 ml. of

Table 2. Analysis of Untreated Urine

Test	Results
pH	6.6
color	1143 units
chloride	6.8 gm./l.
sulfate	2.5 gm./l.
calcium	0.15 gm./l.
sodium	3.7 gm./l.
potassium	3.3 gm./l.
total solids	37.6 gm./l.

throughput volume and for each 500 ml. sample, to 250 ml. of volume.

Figure 1 presents the relation of the pH of the effluent to the throughput volume. The first point indicated applies to the second sample collected; the first sample did not provide a proper measure of the effectiveness of the resin, since a large portion of the first 250 ml. consisted of the water which was used in preparing the resin for operation. It may be observed that a sharp break in the effectiveness of the resin occurs at 1600 ml. of throughput and that the resin is virtually exhausted when 2000 ml. of effluent have been collected.

Figure 2. indicates the relation between the ratio of the effluent chloride concentration to that in the influent and the throughput volume. A similar relation for sulfate is presented as Figure 3. The resin appears to be exhausted with respect to chloride removal sooner than with respect to sulfate removal and sulfate is still being removed until 4000 ml. of effluent have been collected. The resin's greater capacity for the divalent ion is to be expected. The results of the tests for total solids show a fraction of 0.53 of the solids in the influent remaining for 500 ml., 0.79 for 1000 ml., and 0.97 for 1500 ml. The value of this determination is lessened by the presence of large amounts of urea.

Two sets of color tests were run on the effluent samples; one set was made one day after the column operation, and the other, three days after operation. The results are presented as Figure 4. In the first day tests a definite turbidity began to appear in the samples which corresponded to 1500 ml. or more of throughput volume. This in part accounts for the relative maximum in the first day curve. The third day tests were made to see if the turbidity was reduced by sedimentation to any extent; some reduction had

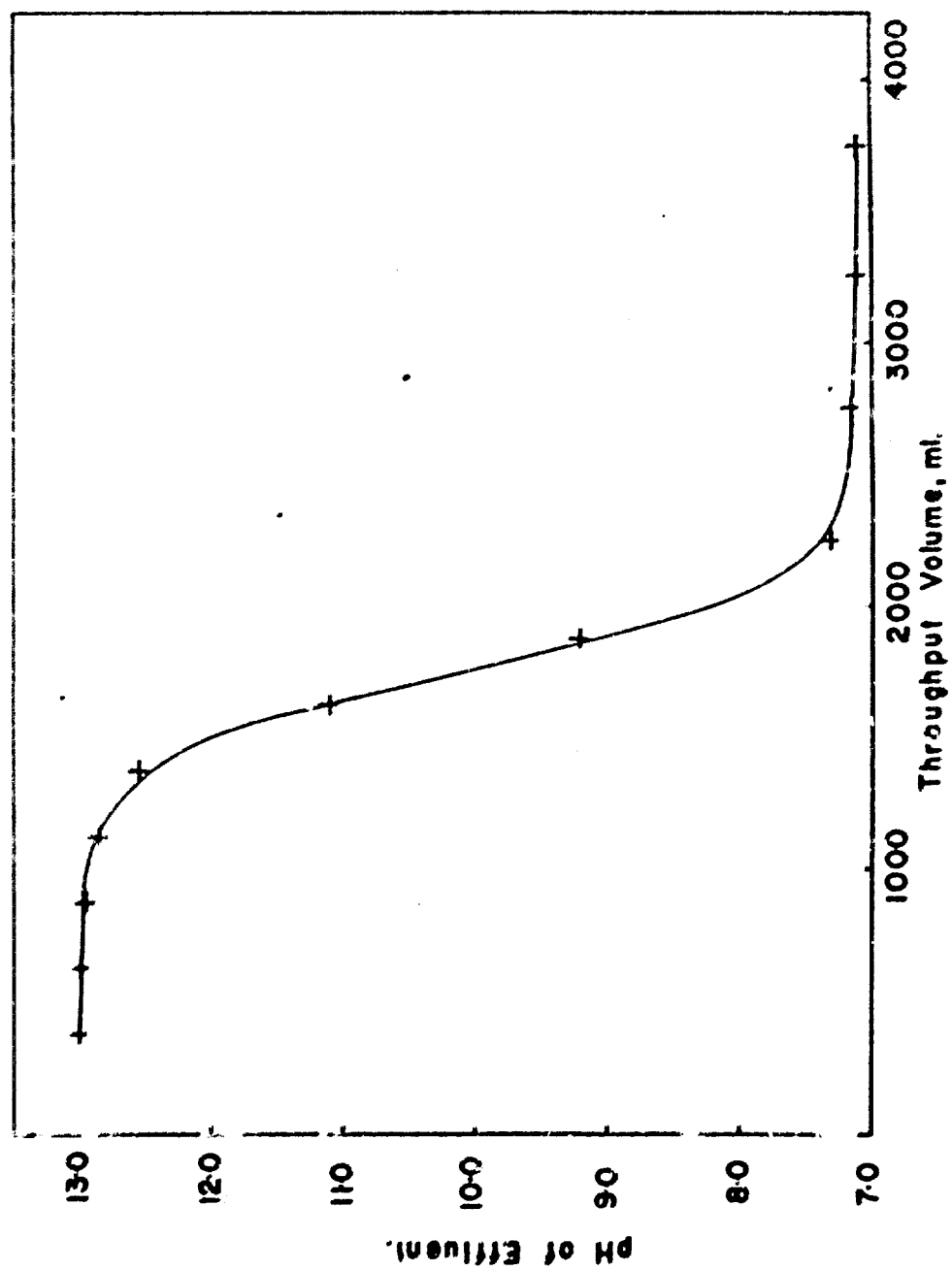


Figure 1. Variation in pH of effluent from anion exchanger.

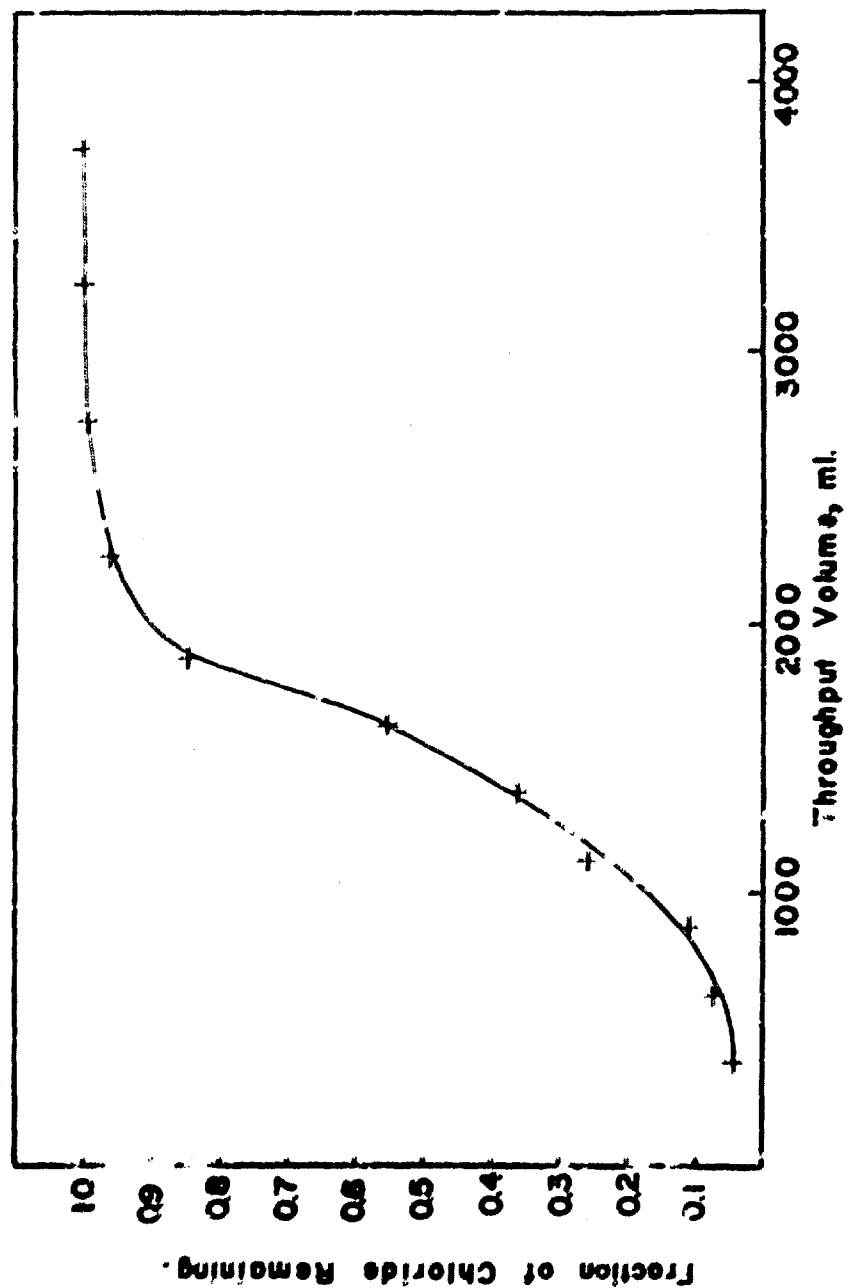


Figure 2. Variation in chloride concentration of effluent from anion exchanger.

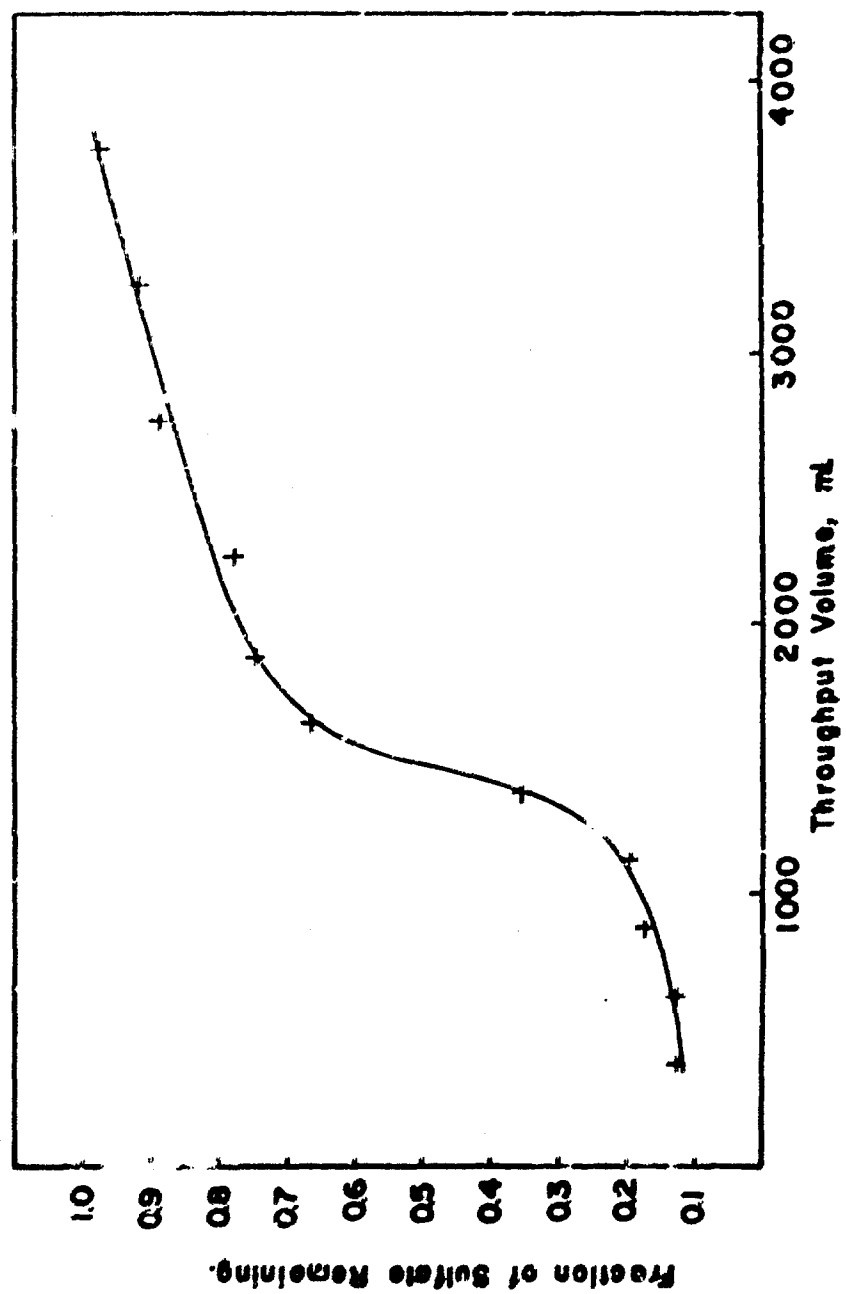


Figure 3. Variation in sulfate concentration of effluent from anion exchanger.

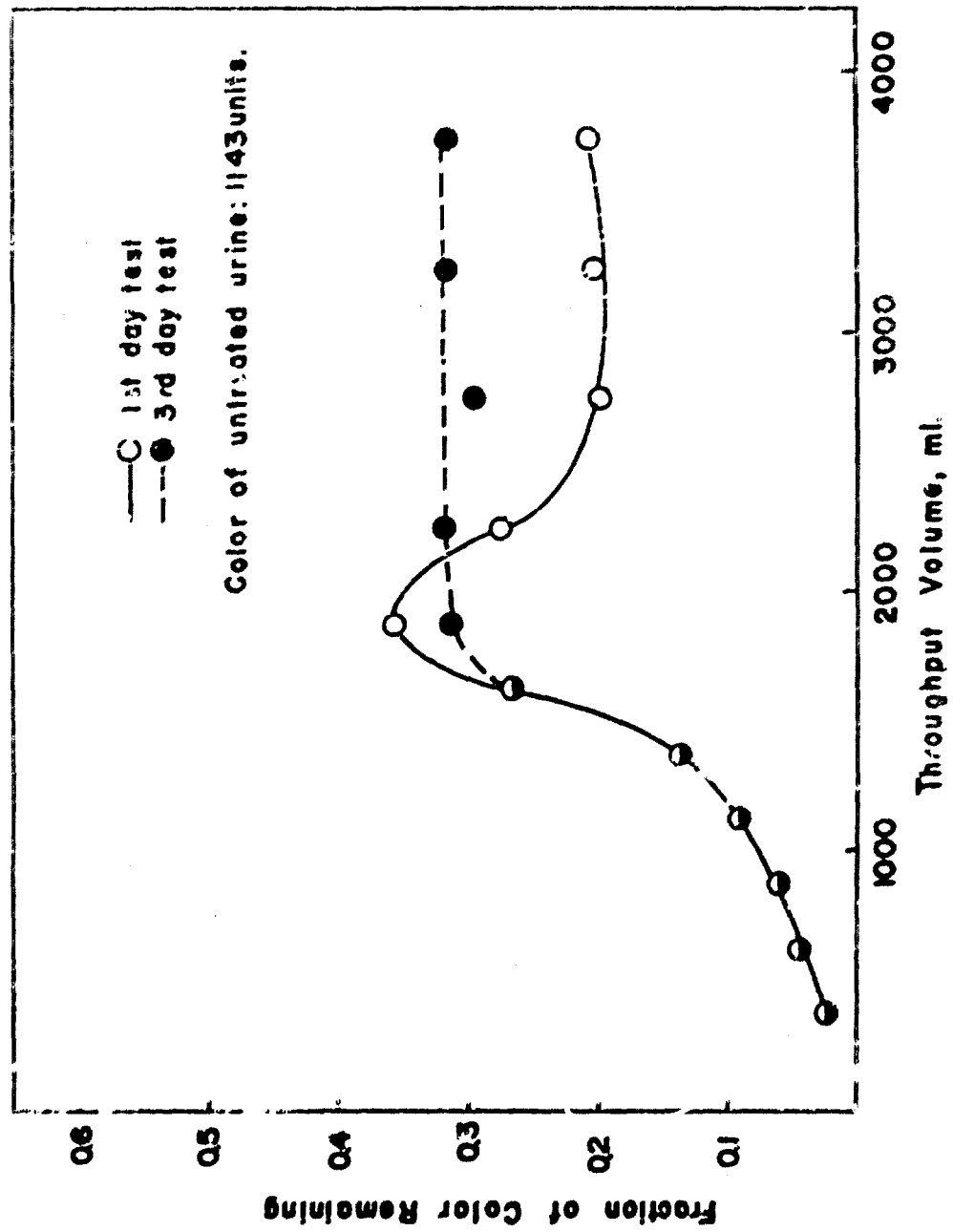


Figure 4. Variation in color of effluent from anion exchanger

taken place, but a definite cloudiness appeared in the last three samples. Filtration of these samples caused no significant change in the absorptivity. The turbidity effects can be explained in part by the sloughing-off of calcium phosphate by the resin. It is also possible that bacterial action could have occurred; however, the samples were kept refrigerated except during analysis.

The second series of observations was made by passing the combined effluent from the anion exchanger through the column containing IRC 50. It was decided that more representative results could be obtained by using only the initial effluent from the anion exchanger as feed for the cation exchanger. The first 1600 ml. of throughput volume was of rather uniform concentration and represented the effluent from an effectively operating anion column. As a result, three anion exchange columns were set up and 1600 ml. of untreated urine were passed through each column. The 4750 ml. of effluent were combined into one sample and analyzed. The results of the analysis are presented in Table 3. The total absence of calcium ion should be noted. It is likely that the calcium was precipitated out in the anion exchanger as calcium phosphate; this was enhanced by the elevated pH at which the anion exchanger operated. Although no quantitative analysis was made for magnesium, it seems probable that this cation was also precipitated out as magnesium hydroxide; this was qualitatively determined from the permanganate calcium analysis.

About 4750 ml. of the combined anion exchanger effluent were passed through the cation exchanger. Table 4 indicates the variation of the pH of the effluent with throughput volume. It may be observed that the column was still operable after 4100 ml. of effluent had been collected. The break point

Table 5. Analysis of Influent to Cation Exchanger

<u>Test</u>	<u>Results</u>
pH	12.6
color	90.0 units
chloride	1.0 gm./l.
sulfate	0.5 gm./l.
calcium	0.0 gm./l.
sodium	3.7 gm./l.
potassium	3.3 gm./l.
total solids	28.4 gm./l.

Table 4. Analysis for pH, Sodium, and Potassium of
Effluent from Cation Exchanger

Throughput Volume (ml.)	pH	Sodium (gm./l.)	Potassium (gm./l.)
125	3.4	1.0	0.95
625	3.4	0.8	0.7
2125	4.0	0.55	0.8
4125	5.0	0.55	0.8
4625	10.4	0.75	1.8

occurs at approximately 4500 ml. of throughput. The results of the sodium and potassium analyses are also presented in Table 4. It can again be seen that the column was operating effectively up to the 4500 ml. point. The remaining fraction of total solids in the effluent during the period before exhaustion was on the average approximately 0.35 that of the influent. The color of the effluent was reduced to about 0.5 of that of the influent until a throughput volume of 3000 ml. was reached. It then rose sharply due to turbidity.

From the results which have been presented it may be observed that the anion exchanger removed approximately 260 meq. of chloride and 80 meq. of sulfate before the resin was exhausted. This corresponds to 1.03 meq. per gram of dry resin. Approximately 1500 ml. of untreated urine were processed before the resin approached exhaustion, although sulfate was still being removed until the 2500 ml. point. This corresponds to four ml. of effluent per cm^3 of resin. In addition, significant quantities of calcium and magnesium were removed, apparently by precipitation; this may have influenced the rapid exhaustion of the anion exchanger by forming a film to block available exchange sites. The cation exchanger removed approximately 500 meq. of sodium and 250 meq. of potassium before the resin was exhausted. This corresponds to 2.27 meq. per gram of dry resin. About 4500 ml. of effluent were collected before the resin approached exhaustion; this corresponds to 11 ml. of effluent per cm^3 of resin. The combination of both columns produced an effluent with a total solids content 0.27 of the value for the influent. The total solids removal is greater than would be expected from the sulfate, chloride, sodium, and potassium analyses. This may be attributed to exchange of other ions, some urea removal, and the filtering

action of the exchange beds. The color of the effluent was 40 units compared to 1143 units for the influent. Urea removal was negligible.

OXIDATION AND PRECIPITATION OF UREA

Based upon the results obtained in the initial studies, it was determined that the use of calcium hypochlorite appeared to be the most promising method of urea removal. Aqueous urea solutions were then used for further studies on calcium hypochlorite as an oxidizing agent. The concentration of urea in the solutions was adjusted to 24 gm/l. corresponding approximately to the average reported urea content of urine. HTH calcium hypochlorite with an available chlorine content of 65% was used.

Complete oxidation of urea was achieved with a 125% of the stoichiometric quantity of calcium hypochlorite. The oxidation reaction was accompanied by precipitation of calcium carbonate. Approximately 24% of the calcium content of calcium hypochlorite added was thus removed in the oxidation step.

Precipitation of calcium and chloride ions in solution resulting from oxidation of urea would be necessary if ion exchange were to follow. Introduction of silver and carbonate ions into a solution containing calcium and chloride ions seemed likely to precipitate silver chloride and calcium carbonate since the solubility products of these compounds are extremely low. Four precipitation methods were evaluated: 1. use of a saturated solution of silver carbonate in ammonium hydroxide, 2. use of dry silver carbonate, 3. use of silver carbonate followed by silver oxide, and 4. use of dry silver oxide. Complete removal of both calcium and chloride ions was effected by methods 1 and 2, using approximately the stoichiometric quantity of silver carbonate (based on chloride content). The calcium to

chloride content was 0.7 on an equivalent basis. Analysis showed that addition of silver carbonate beyond the requirement for complete precipitation of calcium continued to precipitate chloride, but introduced undesirable carbonate ions into solution. It was therefore decided to attempt a two stage precipitation (method 3) using silver carbonate for precipitation of all the calcium and part of the chloride, followed by silver oxide for removal of the remaining chloride. The use of ammonium hydroxide as a solvent for the silver carbonate did not seem to aid the precipitation reaction appreciably; only silver salts were used in all further experiments. Complete removal of both calcium and chloride ions was effected by the two stage precipitation. Silver carbonate additions were in stoichiometric quantities based on the calcium content and silver oxide, in 130% of the stoichiometric quantity based on the remaining chloride content. The solution after complete precipitation of calcium and chloride was highly alkaline. This could presumably be attributed to hydrolysis of silver oxide.

In alkaline solution dissolved carbon dioxide exists as the carbonate ion and in the presence of calcium ions is likely to precipitate as calcium carbonate. Since silver oxide has both the property of removing chloride ions in solution as well as increasing the alkalinity of the solution, oxidation of urea with calcium hypochlorite in the presence of silver oxide (method 4) seemed likely to precipitate both the reaction products, calcium and chloride. An aqueous solution of urea was contacted with 106% of the stoichiometric quantity of calcium hypochlorite and 130% of the stoichiometric quantity of silver oxide based on chloride content. Complete precipitation of both calcium and chloride ions was effected. However, only 90% of urea removal was effected, indicating some interference.

Two methods for the oxidation of urea with calcium hypochlorite and for the precipitation of the reaction products, calcium and chloride ions, seem feasible:

Oxidation with hypochlorite followed by precipitation first with silver carbonate, then with silver oxide. (method 3),

Simultaneous oxidation and precipitation, using calcium hypochlorite and silver oxide. (method 4).

Table 3 compares the properties of the solutions after treatment by the above methods and presents a comparison of the chemical requirements. It can be seen that the economics of method 4 seem more favorable than that of method 3, since the price of silver oxide is approximately half that of silver carbonate.

Studies were then initiated using calcium hypochlorite and silver carbonate and/or silver oxide for urea removal from urine. The addition of calcium hypochlorite to urine caused the formation of a rather stable foam phase above the solution. This was brought about by a combination of gas evolution due to chemical reaction with the existence of certain surface-active constituents of urine. Three methods were considered to overcome this difficulty: 1. the use of a screen as a mechanical foam breaker, 2. the addition of a small amount of defoaming agent to the urine (such as a silicone derivative) which could later be adsorbed from the urine, 3. the use of a preliminary adsorption step to remove the surface-active agents present in urine. The third method seemed most promising, particularly if some urea removal could be accomplished.

Table 5. Analysis of Solutions Before and
After Precipitation Treatment

Property	Initial Solution	Final Solution Method 3	Final Solution Method 4
pH	7.6*	12.2	12.4
Ca^{+2} , gm./l.	33.2	---	---
Chloride, gm./l.	66.6	---	---
Alkalinity as CaCO_3			
OH^- , gm./l.	---	23.20	18.50
CO_3^{-2} , gm./l.	---	3.14	9.50
HCO_3^- , gm./l.	---	---	---
Urea, gm./l.	24.0*	---	2.50

*before oxidation

Chemical Requirements

Chemical	Consumption gm./l. urea solution Method 3	Consumption gm./l. urea solution Method 4
Calcium hypochlorite	150.9	169.0
Silver carbonate	180.0	---
Silver oxide	82.5	305.8

ADSORPTION STUDIES

Activated charcoal (coconut, 6-14 mesh) was used in two batch experiments to determine urea removal. Aqueous urea solutions were prepared containing urea concentrations varying from 1.5 to 115 gm./l. 50 ml of each of these solutions were contacted with 10 gm. of activated charcoal and an adsorption isotherm was determined at 26° C. A plot of log gm. urea removed/gm. charcoal vs. log of equilibrium concentration of the urea solution gave a straight line, as would be predicted from Freundlich's Equation. For a urea solution of 25 gm./l., 0.047 gm. urea/gm. charcoal were removed, indicating approximately 40% removal, for the given charcoal dosage. A single experiment was conducted using 50 ml. of urine (urea concentration: 24 gm./l.). 40 gm. of charcoal yielded approximately 60% urea removal.

The adsorption characteristics of a 7/8 in. I.D., 23 in. deep activated charcoal column were determined using urine as the influent. For the 102 gm. of charcoal, approximately 100% urea removal was obtained for the first 110 ml. of throughput volume (corrected for the water used to prepare the column). For the next 280 ml. of throughput, the urea removal decreased almost linearly until, at the 400 ml. point, 20% urea was being removed. The effluent remained colorless until the 400 ml. point and from 400-500 ml. the effluent had a faint coloration. The use of columns of greater depth should improve considerably the efficiency of urea removal.

Further studies will be conducted on the adsorption phase of the process, using charcoal columns and determining urea removal and the breakpoint for the columns. Then, an optimum arrangement will be worked out, combining preliminary adsorption with oxidation and precipitation. It is anticipated that a considerable saving in overall cost and space can be accomplished by using a preliminary adsorption step.

FUTURE STUDIES

15 January, 1963 - 1 August, 1963

Upon completion of the adsorption, oxidation, and precipitation phase of the process, further ion exchange experiments will be conducted. Urine after treatment by calcium hypochlorite, silver carbonate, activated carbon, etc. will be passed through columns containing acid and base exchange resins. Various combinations of weak acid, strong acid, weak base, and strong base resins will be employed using single and mixed beds. The effluent will be tested for pH, sulfate, phosphate, chloride, sodium, potassium, silver, and magnesium. Color and turbidity will be checked and total and volatile solids will be determined. Based upon influent and effluent hydrogen ion concentrations, upon the removal of both anions and cations, and upon resin efficiencies, the optimum ion exchange combination will be selected. It is anticipated that a value of approximately 8 throughput volumes per unit volume of combined resins can be obtained. This estimate is based upon the efficient removal of chloride and calcium in the oxidation and precipitation step.

The effluent from the ion exchange phase of the process will then be passed through a terminal bed of activated carbon. This should accomplish the removal of trace quantities/^{of} remaining organic matter, including color and taste and odor producing substances. The effluent will be tested for organic, ammonia, and nitrate nitrogen and for fixed and volatile solids. Various bed sizes will be tested and the optimum value will be selected. The quality of the effluent after oxidation, precipitation, ion exchange and adsorption can be expected to be quite high, but additional treatment in the form of pH adjustment and terminal disinfection may be necessary.

1 August, 1963 - 1 August, 1964.

A preliminary analysis will then be conducted to estimate the relation between capacity and cost and size for the entire urine conversion unit. Size considerations will include the storage volume of chemicals needed in the oxidation, precipitation, and disinfection steps and the volume of resins and activated carbon needed in the ion exchange and adsorption steps. Various combinations will then be attempted experimentally, such as the reduction of the precipitation dosage and the increase of the ion exchange bed volume to see if an optimum arrangement may exist. It is likely that the size and cost of the unit will depend largely upon the effluent quality desired. Once criteria have been established, dosages and bed volumes will be varied to achieve that standard.

The shape and size of the final unit will depend upon volume required for chemical contact and separation and upon effective contact and detention times for the ion exchange and adsorption steps. The flow rate and velocity distribution through each of the porous media and the effect upon head loss of packing and grain size of the porous media will be determined. The optimum shape and overall length to characteristic cross-section can be selected on the basis of the above information.

A laboratory scale model of the urine conversion process will be fabricated and operated at the rate of five quarts per day. An effort will be made to include all of the components in a compact unit which would be operated with ease and would require minimum maintenance. Full-scale capacities can then be obtained by using a battery of the laboratory-scale units in parallel and overall performance and operation characteristics can then be predicted from the known characteristics of the laboratory-scale

model. The essential criterion for the effectiveness of the entire unit will be the quality of the effluent. In addition to the usual physical and chemical tests on water, analyses of trace metals and short term animal toxicity studies involving body weight gain and gross examinations of body organs for lesions will be conducted, as time and funds allow.

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