NASA CR 152227

FOUR-MAN RATED DUAL CATALYST SYSTEM FOR THE RECOVERY OF WATER FROM URINE

Final Report

by

P. Budininkas

November, 1978

Prepared Under Contract No. NAS2-9715

by

GATX

GARD, INC.

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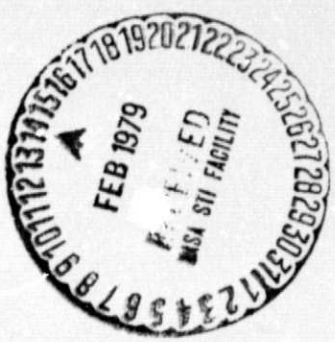

for

AMES RESEARCH CENTER

NATIONAL AERONAUTICS AND SPACE ADMINISTRATION

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FOREWORD

The investigation described herein was conducted by GARD, INC/GATX during the period of September, 1977 through December, 1978 under NASA Contract NAS2-9715. The Project Engineer was P. Budininkas who was assisted by E. K. Krug and M. M. Sternisha. The Technical Monitor was P. D. Quattrone, Chief, Advanced Life Support Office, NASA Ames Research Center, Moffett Field, California.

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SUMMARY

A four-man capacity dual catalyst system for the recovery of water from urine was designed and constructed. The catalytic system was then integrated with a 4-man rated urine wick evaporator constructed by the Umpqua Research Company. The integrated system was tested with untreated urine. During operation, urine vapor produced by the wick-evaporator is treated in the catalytic system to remove ammonia and volatile hydrocarbons, and water is recovered by condensation in a water cooled condenser. After start-up the system operates completely automatically and requires no manual adjustments, except periodic supply of urine and removal of the recovered water.

The testing program consisted of short preliminary and optimization tests, and three endurance tests of up to 94 hours of uninterrupted operation. The capacity of the integrated system was limited by the rate of urine vapor generation by the wick-evaporator. Although the system was designed for treating 0.325 kg urine per hour, this rate could be achieved only with a fresh wick, then gradually decreased as the wick became saturated with urine solids. The average urine treatment rates achieved during each of the three endurance tests were 0.137, 0.217, and 0.235 kg/hr.

The quality of the recovered water meets drinking water standards, with the exception of a generally low pH.

INTRODUCTION

A number of urine evaporation schemes have been proposed, developed, and tested for possible water recovery applications. Besides inherent hardware problems specific for each method, the presence of ammonia in urine vapor presents a considerable difficulty common for most of these processes. To inhibit the decomposition of urea into ammonia, pretreatment chemicals are usually added to the urine, and the evaporation is accomplished at low temperatures.

Previous studies (NASA CR 151930) have demonstrated the feasibility of water recovery from untreated urine by an evaporative process utilizing a dual catalyst system for the removal of ammonia present in urine vapor. That investigation indicated that the use of urine pretreatment chemicals may no longer be necessary in evaporative water recovery process.

The objective of this investigation was to design and fabricate a four-man rated dual catalyst system and to integrate it with a wick-evaporator fabricated by the Umpqua Research Company for the recovery of water from untreated urine. Both short and endurance tests were performed with the integrated system using untreated urine to obtain operational data and to establish the quality of the recovered water and the composition of the vent gas.

To achieve the objectives of this investigation, the program consisted of the following tasks:

1. Catalyst Optimization.- Bench scale tests were performed on commercially available platinum and ruthenium type catalysts selected on the basis of previous investigation to establish data required for catalyst bed sizing.

2. Design and Fabrication of the Catalyst System.- A catalyst system adequate for treating the urine of a four-man crew and compatible with the wick-evaporator was designed. Since the concentration of urine vapor in the gas stream produced by the wick evaporator is relatively low, the size of the catalytic system is dictated by the total gas flow rate required for evaporating urine at the required rate. The catalytic system was fabricated using commercially available components.

3. Wick-Evaporator/Catalytic System Integration.- The system was integrated, functionally tested and adjustments made to ascertain a balanced operation.

4. Integrated System Tests.- The system was tested using untreated urine. Short preliminary and optimization tests and endurance tests lasting up to 94 hours were performed. The quality of the recovered water was determined by appropriate tests. The overall urine treatment rate of the system was limited by the rate of vapor production of the wick-evaporator.

DESIGN AND FABRICATION OF CATALYTIC SYSTEM

Catalyst Optimization

To establish data required for catalyst bed sizing and design of a 4-man rated system, bench scale optimization tests were performed on selected commercially available platinum and ruthenium type catalysts. These catalysts were selected on the basis of previous investigation (NASA CR 151930) indicating that the most effective catalysts are platinum for the low temperature oxidation of ammonia and ruthenium for the decomposition of nitrous oxide.

Catalyst optimization tests were performed in a flow-through, single pass system depicted schematically in Figure 1. Basically it consists of a heated glass reactor containing the catalyst, gas supply lines and mixing chamber, an electric heater, and thermocouples for temperature control and measurement. Ports are provided for taking samples of the feed and of the product gas for analysis. The bottom portion of the reactor contains a 50cc heated bed of 1/8" alumina pellets which supports a 50cc bed of the catalyst. Passing through the hot alumina pellets, the feed gas becomes preheated to the reaction temperature before entering the catalyst bed. The temperature is controlled by a thermocouple located at the interface of the alumina and catalyst beds. By adjusting the position of the reactor within the surrounding electric heater, a temperature difference between the bottom and the top of the catalyst bed of less than ±5°C is achieved. For N_2O decomposition tests, a portion of the feed gas

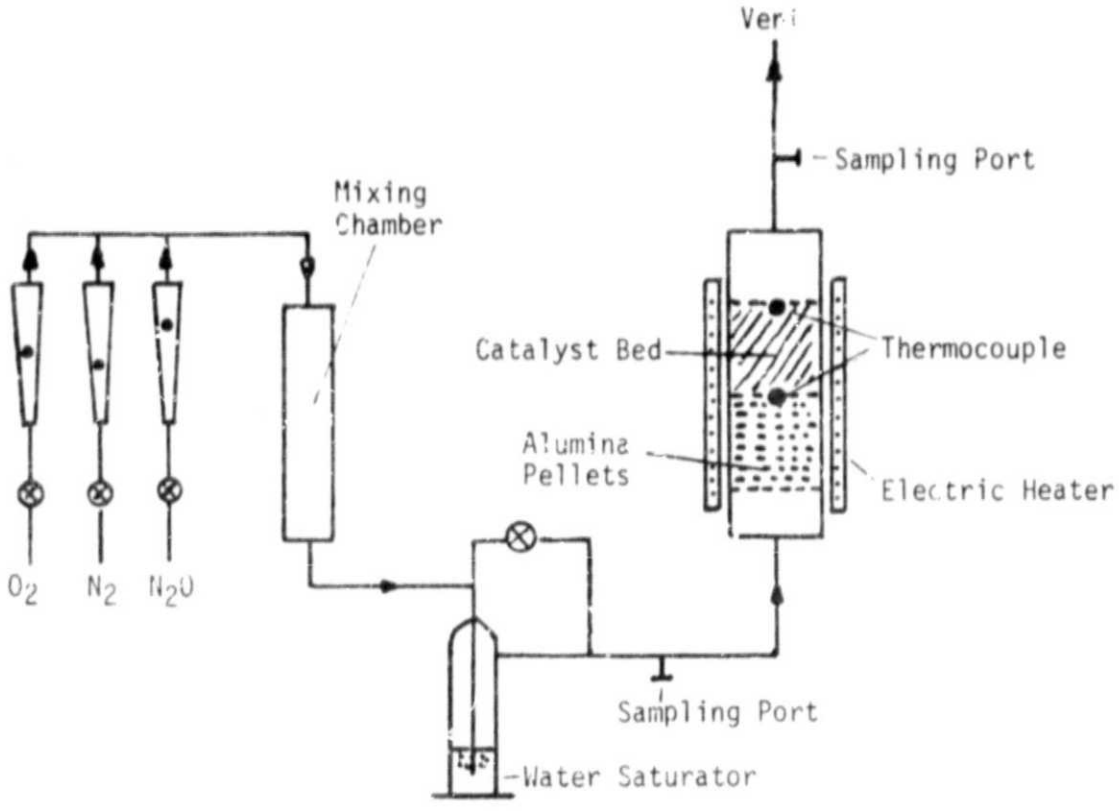

Figure 1 CATALYST TESTING SYSTEM

mixture required for adding desired water vapor concentrations was passed through a dispersion tube submerged in water. For NH_3 oxidation tests, the desired moisture concentration was achieved by adding steam to the feed. The concentration of water vapor in feed was calculated from the amount of condensate collected from the product stream.

Optimization tests were performed using artificial gas mixtures allowing a convenient establishment of the desired gas compositions and flow rates. The variables tested were:

a. Temperature
b. NH_3 or N_2O concentration
c. O_2 concentration
d. H_2O vapor concentration
e. Space velocity

The ranges of the NH_3, N_2O, O_2, and H_2O vapor concentrations investigated were primarily those anticipated in feed stream provided by the present air evaporation techniques. In addition, some consideration was given to gas compositions achievable either by high efficiency air evaporation or by other evaporative techniques, i.e., feed streams with high H_2O, NH_3, N_2O and low O_2 concentrations.

The basic purpose of these tests was to determine the maximum space velocities at the minimum catalyst temperatures under various experimental conditions and to gain an insight how they are affected by the feed gas composition. The overriding requirements for the acceptable performance of catalysts under all conditions is a complete removal of NH_3 or N_2O, depending on catalyst tested. In addition, the

catalysts should not form NO_x in significant concentrations under the selected operating conditions.

The requirement that the dependent variable, i.e., the removal of NH_3 or N_2O, must always be 100% determines the mode of testing. To minimize the number of tests, an experimental test program was selected which employs a central factorial design for changing the levels of the feed gas composition and the space velocities but increases the test temperature stepwise up to the level which achieves a complete removal of NH_3 or N_2O. For the oxidation of NH_3, testing started at 150° - 175°C with each selected gas composition and space velocity, then the temperature of the catalyst was increased in increments of 25°C until a complete removal of NH_3 was obtained. Similarly, for the decomposition of N_2O each test was started at 325° - 350°C and the temperature increased in increments of 25°C until a complete removal was achieved.

The results of the NH_3 oxidation tests are summarized in Table 1. It appears that the feed gas composition has only minimal effect; however, high concentrations of water vapor increase the temperature required for a 100% removal of NH_3 by 30° - 50°C. Space velocity higher than 14,400 hr^{-1} was not tested because higher flows tend to disturb the catalyst bed and produce channeling.

The results of the N_2O decomposition are summarized in Table 2. From the obtained data, the following conclusions can be made:

1. An increase of any of the three gas composition variables tested, i.e., concentrations of O_2, N_2O, and water vapor,

and the space velocity increases the temperature required for a complete N_2O decomposition,

2. A complete decomposition of N_2O is achieved at temperatures ranging from 350°C to 425°C, depending on feed gas composition,
3. To achieve a complete decomposition of N_2O at the catalyst temperatures not exceeding 425°C, water vapor will have to be removed to a concentration not exceeding 0.8% by volume (dew point of 4°C).

Table 1. SUMMARY OF NH_3 OXIDATION TESTS

Space Velocity, hr^{-1}	Feed Composition			Temperature for 100% Removal of NH_3, °C
	NH_3, ppm	O_2, %	H_2O Vapor, Vol.%	
9,600	2,500	5	0	175
9,600	2,500	21	0	200
9,600	500	21	0	175
14,400	2,500	21	0	200
14,400	2,500	21	11	230
14,400	2,500	21	22	250
14,400	2,500	21	45	230

It is anticipated that the system eventually may be operated in a recycle mode. In this mode, the entire gas stream used for evaporation and transport of urine vapor will be recycled, with only small amounts of oxygen required for maintaining a constant O_2 concentration added to it and equivalent amounts of product gas vented. In a recycle system,

Table 2. SUMMARY OF N_2O DECOMPOSITION TESTS

Space Velocity, hr^{-1}	Variables			Temp. for 100% Decomposition, °C
	N_2O, ppm	O_2,%	Water,%	
2,400	2,500	21	-	400
2,400	2,500	10	-	400
2,400	2,500	5	-	375
2,400	2,500	0	-	350
2,400	500	21	-	375
2,400	500	10	-	375
2,400	500	5	-	3.
2,400	500	0	-	350
2,400	2,500	21	1.8	425
2,400	2,500	10	1.8	425
2,400	2,500	5	1.8	425
2,400	2,500	0	1.8	400
2,400	2,500	21	0.8	425
2,400	2,500	10	0.8	425
4,800	2,500	0	-	375-400
4,800	2,500	5	-	425
4,800	2,500	10	-	425
4,800	500	10	-	400
4,800	500	21	-	425
4,800	2,500	0	1.8	425
4,800	2,500	10	1.8	475
4,800	2,500	21	1.8	500
4,800	2,500	21	0.8	450
9,600	2,500	0	-	450
9,600	2,500	0	1.8	475-500

a complete removal of N_2O would be required only from the vent gas, provided the presence of N_2O in the recycling gas does not interfere with the oxidation of NH_3.

To determine the effect of N_2O on the catalytic oxidation of NH_3, tests were performed using feed gas containing 2500 ppm NH_3, 21% O_2, and N_2O concentrations ranging up to 5700 ppm. In the temperature range of 175° - 250°C, the presence of N_2O in feedgas had no measurable effect on the catalytic oxidation of NH_3. Additional tests indicated that N_2O is not oxidized to NO_x by the NH_3 oxidation catalyst.

Design of a Four-Man Catalytic System

The catalytic system must be adequate for treating the output of urine of a 4-man crew and be compatible with the air evaporator supplied by the Umpqua Research Co. Since the concentration of urine vapor in the gas stream produced by an air evaporator is relatively low, the size of the catalytic system is basically dictated by the total gas flow rate necessary to evaporate and transport the required amount of urine vapor. Thus, the anticipated urine vaporization capability, gas flow rate and blower pressure of the Umpqua air evaporator were combined with the required daily processing rate of urine and data obtained during the catalyst optimization tests to provide catalyst system design conditions summarized in Table 3.

Table 3. SYSTEM DESIGN CONDITIONS

1.	Processing Rate	
	Urine	1.56 kg/man-day
	Flush Water	0.39 kg/man-day
	Total Four-man Output	7.8 kg/day
	Processing rate (continuous operation)	0.325 kg/hr.
2.	Umpqua Wick Evaporator	
	Maximum gas flowrate	340 l/min.
	Maximum blower pressure	69 cm H_2O
3.	Catalytic Oxidation of NH_3	
	Operational Temperature	250°C
	Maximum space velocity	14,400 hr^{-1}
4.	Catalytic Decomposition of N_2O	
	Operational Temperature	450°C
	Maximum space velocity	9,600 hr^{-1}
5.	Water Condensation Temperature	4°C

Based on these system design conditions, the sizes and geometry of the catalyst beds, interconnecting ducting and elbows, and the water condenser were calculated as shown in Table 4. It is estimated that the pressure drop across the entire dual catalyst system at maximum anticipated flow rates and operating temperatures will not exceed 48 cm H_2O. Thus, the system is compatible with Umpqua wick evaporator and is adequate for treating the output of urine of a 4-man crew.

Table 4. DESIGN SIZES AND GEOMETRY OF SYSTEM COMPONENTS

1. Catalytic Oxidation of NH_3	
Reactor dimensions	24 cm. high, 12 cm. dia
Depth of catalyst bed	12.4 cm
Volume of catalyst bed	1.4 liters
ΔP across bed at oper. cond.	8.4 cm H_2O
2. Catalytic Decomposition of N_2O	
Reactor dimensions	24 cm. high, 12 cm. dia
Depth of catalyst bed	18.6 cm
Volume of catalyst bed	2.1 liters
ΔP across bed at operating conditions	10.7 cm H_2O
3. Connecting ducts and elbows	
Diameter	3.8 cm (1.5")
ΔP at operating conditions	21.6 cm H_2O
4. Water Condenser	
Operating temperature	4°C
ΔP at operating conditions	7.6 cm H_2O

Fabrication of the Catalytic System

The basic components of the system are the two catalytic reactors, the water vapor condenser, and the recovered water collector; these components are interconnected by stainless steel tubing. All the components were fabricated using commercially available parts and materials. The catalytic reactors and the condenser were fabricated from stainless steel, the water collector was a polyethylene wide-mouth bottle with a drain for the removal of the recovered water. To conform to readily available stainless steel tubing, the diameters of the reactors were changed somewhat from the dimensions indicated in the original design considerations. Thus, the final dimensions of the reactors were:

a. NH_3 oxidation reactor: O.D. = 10.2 cm., I.D. = 9.8 cm., length = 34.3 cm.

b. N_2O decomposition reactor: O.D. = 10.2 cm., I.D. = 9.8 cm., length = 40.6 cm.

With the exception of their lengths, the geometries of both catalytic reactors were identical, as schematically depicted in Figure 2. Catalyst support screens were installed 7.6 cm above the bottom of the reactors; this arrangement created a plenum that minimizes channeling of the gas flow. Similarly, a 6.8 cm. long cavity was left above the catalyst beds for minimizing channeling and as a reservoir for adding more catalyst, if required, for attaining the desired processing rate. Four thermocouples were installed for monitoring the catalyst bed temperature at the inlet, center, and top of the bed; the

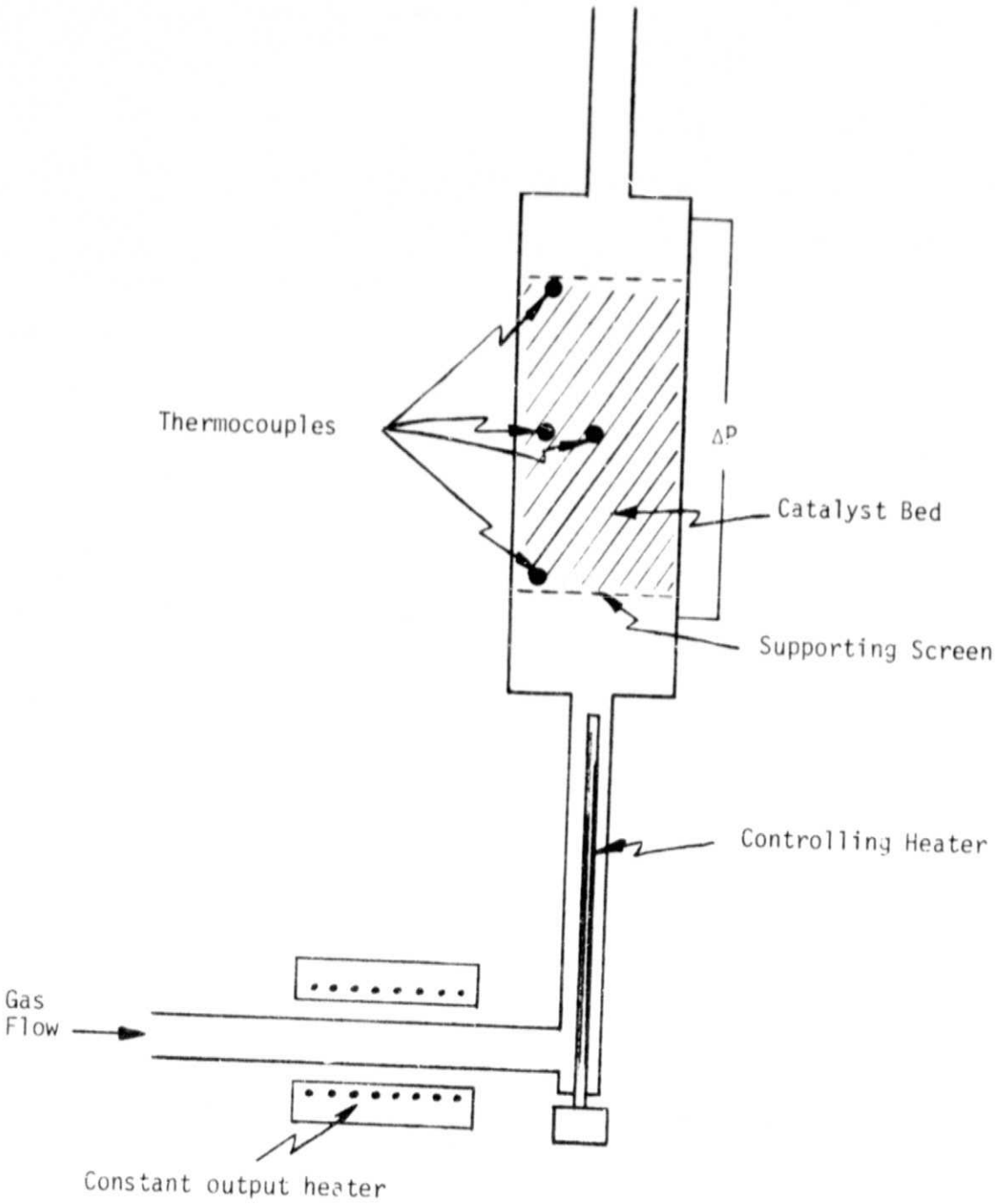

Figure 2 CONFIGURATION OF CATALYTIC REACTORS

fourth thermocouple, located in the center, is provided for temperature control.

Heating of the gas stream is provided by two electric heaters. The first is a constant output tubular heater capable of increasing the gas temperature close to the lowest anticipated operational level; the second, a cartridge type heater whose output is varied by a stepless temperature controller as required for achieving a constant operational temperature.

The following amounts of catalysts required for processing a nominal 4-man output were placed in the catalytic reactors:

a. NH_3 oxidation, 1,500 cc (1,490 g) of Engelhard Industries, Inc. 0.5% Pt on 1/8" alumina pellets catalyst, creating a 19.9 cm. deep bed,

b. N_2O decomposition, 2,200 cc (2,206 g) of Engelhard Industries, Inc. 0.5% Ru on 1/8" alumina pellets catalyst, creating a 29.2 cm deep bed.

To prevent possible carryover of catalyst, a plug of loosely rolled-up stainless steel screen was installed in the outlet of each catalytic reactor.

A 91 cm. long, 10.2 cm. O.D. stainless steel shell and tube heat exchanger (manufactured by American Standard Co.) with a nominal 10,000 cm^2 heat exchanging area was installed for condensing the recovered water. The heat exchanger was cooled by laboratory tap water.

The overall view of the catalytic system is shown in Figure 3. Both catalytic reactors and their inlet tubes serving as gas preheaters were thermally insulated by blanket type, high temperature insulation.

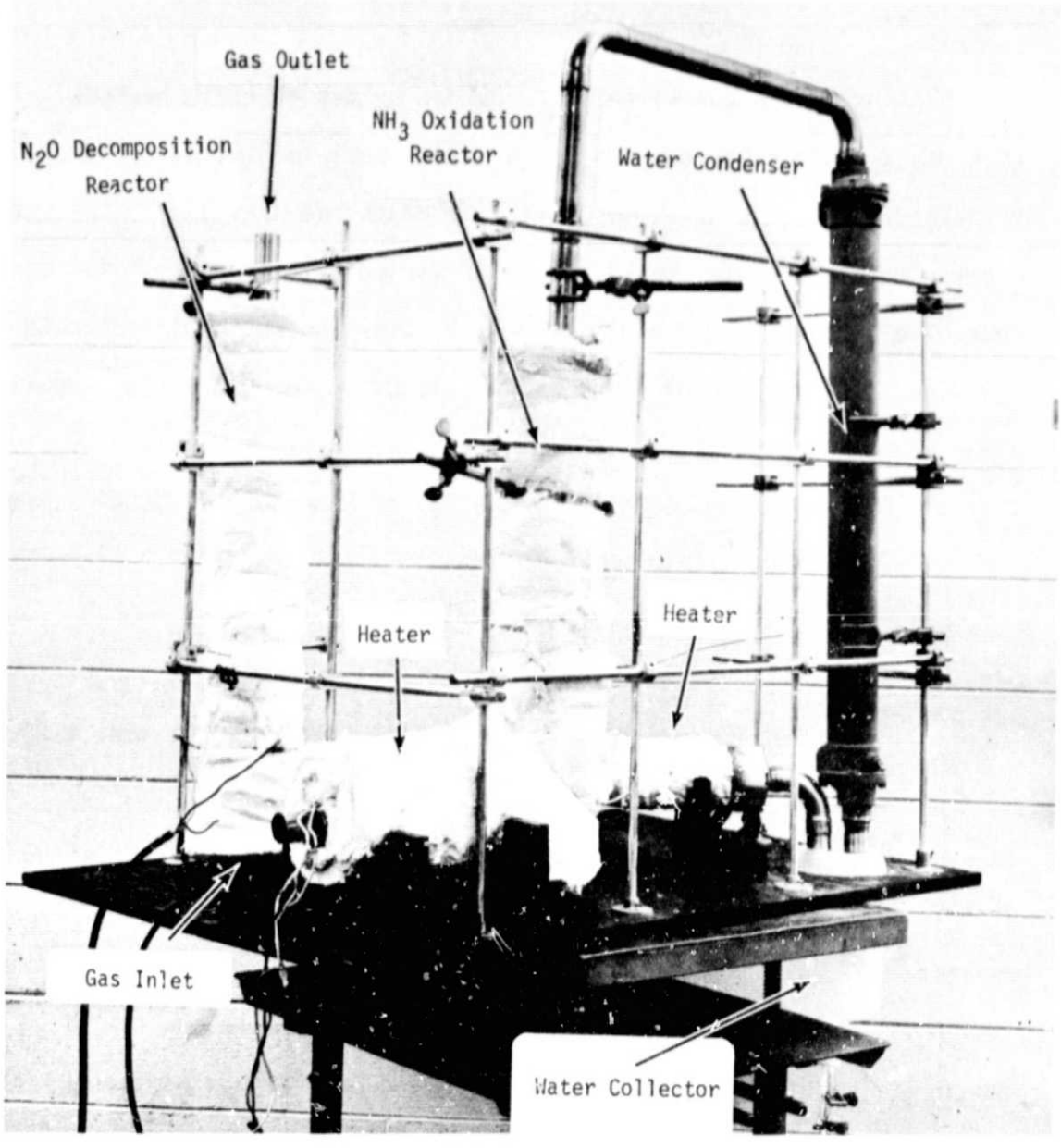

Figure 3 DUAL CATALYST SYSTEM

The gas inlet tube is designed for integration with the air evaporator and accepts the gas stream carrying urine vapor. This gas stream is preheated to approximately 250°C and passed through the NH_3 oxidation reactor. The product, which does not contain any NH_3, is passed through the water cooled condenser where the entire stream is cooled to 4° - 16°C, depending on the temperature of the tap water, and the recovered water collected in the water collector. The gas, freed from water, is then preheated to approximately 450°C, passed through the N_2O decomposition catalyst, and vented.

Functional Testing

Functional testing of the dual catalyst system was performed to observe functional behavior of the system and to compare the observed levels of parameters with the design values. A test plan shown in Table 5, which was previously submitted to and approved by NASA/ARC, was followed. The results of these tests are summarized in Tables 6, 7 and 8.

The ΔP measurements, shown in Table 6, were obtained at room temperature and at the operational temperatures of the catalyst beds. Because of limitations of the available blower, ΔP measurements at the operational temperatures were obtained only at the gas flow rate of 227 l/min. The observed ΔP values are higher than those predicted by design calculations. The calculated ΔP value at operational temperatures and maximum flow rate of 340 l/min was 48.3 cm. H_2O, as compared with the measured value of 50.8 cm. H_2O at a flow rate of 227 l/min.

Table 5. TEST PLAN FOR THE FUNCTIONAL TESTING OF THE DUAL CATALYST SYSTEM

A. SYSTEM PARAMETERS

1. Leak test the entire system
2. Start air blower and adjust air flow to 227-340 l/min
3. Measure ΔP across each catalyst bed and the entire system
4. Preheat the catalyst beds to their operating temperatures, i.e., NH_3 oxidation catalyst to 250°C, N_2O decomposition catalyst to 450°C
5. Determine ΔP as in step 3
6. Determine temperature distribution for each catalyst bed
7. Determine temperature of gas leaving the water condenser
8. Cool down the system and stop air blower.

B. PERFORMANCE OF CATALYSTS

1. Start blower and adjust air flow to 227 l/min.
2. Preheat catalyst beds to operating temperatures
3. Feed gaseous NH_3 into the air stream to give a 1000 ppm concentration
4. Analyze product gas as follows:
    a. Effluent from the NH_3 oxidation catalyst, analyze for NH_3, N_2O, NO_x
    b. Effluent from the N_2O decomposition catalyst, analyze for N_2O
    c. Condensed water, analyze for NH_3
5. Cool down the system and stop air blower.

Table 6. MEASUREMENTS OF ΔP

Gas Flow, l/min.	NH_3 Oxidation Bed		N_2O Decomposition Bed		Entire System
	Temp., °C	ΔP, cm. H_2O	Temp., °C	ΔP, cm. H_2O	ΔP, cm. H_2O
226	R.T.	7.9	R.T.	12.1	29.8
283	R.T.	11.1	R.T.	16.5	41.9
340	R.T.	15.2	R.T.	22.2	57.2
226	250	12.7	460	29.2	50.8

Table 7. TEMPERATURE DISTRIBUTION IN CATALYST BEDS

Catalyst Bed	Inlet, °C	Center, °C	Outlet, °C
NH_3 Oxidation	249	246	241
N_2O Decomposition	463	449	443

Table 8. PERFORMANCE OF CATALYST BEDS

NH_3 oxidation Catalyst: 250°C

N_2O decomposition catalyst: 450°C

Feed: 227 l/min air containing 280 ppm NH_3

Product leaving NH_3 oxidation catalyst: NH_3 <0.1 ppm (detection limit is 0.1 ppm)

Product leaving N_2O decomposition catalyst: N_2O, not detectable (detection limit is 5 ppm)

Temperature of condenser outlet: 16°C

Condensed water: None collected

The distribution of temperatures within the catalyst beds is presented in Table 7. The temperature distribution through the NH_3 oxidation catalyst is very good and shows only 8°C difference between the inlet and the outlet. The spread of temperature through the N_2O decomposition catalyst is 20°C, and can be considered acceptable.

The performance of the catalytic system was tested by treating an air stream containing NH_3 and analyzing the product for NH_3, N_2O, and NO_x. The results of this run are summarized in Table 8 and indicate that the system removes NH_3 as expected.

INTEGRATION WITH WICK EVAPORATOR

Wick Evaporator

The wick evaporator was fabricated by the Umpqua Research Co., Myrtle Creek, Oregon. Details of its design, construction, and operation are presented in a separate report to NASA CR 152226; however, basic information pertinent to the operation of the integrated system is presented here. Basically, a blower takes in room air and blows it continuously through an electrical preheater where the temperature of the air stream is raised to approximately 95° - 110°C. The hot air passes through the wick saturated with urine, raises its temperature, and evaporates some of the urine. The air stream containing urine vapor is then introduced into the catalytic system for the removal of NH_3 and volatile organics.

The heart of the evaporator is the wick. Basically, it consists of an array of 32 strips of wicking material and 31 strips of plastic foam placed alternately next to each other in a package of approximately 35 cm x 35 cm x 5.5 cm size. The plastic foam strips provide longitudinal passages for the hot air which heats and evaporates urine present in the adjacent strips of the wicking material. Urine is pumped to the wick by a variable speed diaphragm pump; the overall pumping rate is varied by adjusting the pump speed and, in addition, the time of pumping and waiting cycles. Urine is pumped into a manifold located at the downstream end of the wick package and distributed into the wick through small openings in the distributing tubes located

on top of the strips of the wicking material. Flooding of the wick is prevented by controlling the amount of urine absorbed in the wick structure. Since an excess of urine causes cooling of the gas stream, a thermocouple located in the gas stream downstream of the wick serves as a controller. When the gas temperature decreases below a desired set-point indicating cooling caused by an excess of urine in the wick, the power of the urine pump is cut-out and remains so until the temperature increases again to the set point; then power to the pump is restored and pumping begins again.

Integrated System

The integrated system consisting of the wick evaporator and the dual catalyst system is shown in Figure 4. The operation of the system is initiated by blowing air through the wick evaporator at room temperature while the catalytic reactors are preheated. When the catalytic reactors reach their operational temperatures, both the air preheater and the urine pump are switched on. When the wick becomes preheated above the pump cutout temperature, pumping of urine is initiated automatically. After the start-up, the system operates automatically and requires no manual adjustments. At the end of a test, the sequence is reversed, i.e., the urine pump and the air heater are switched off, then the catalytic reactors cooled, and the air blower stopped.

Typical test conditions of the integrated system are shown in Table 9. The operating parameters of the wick evaporator are those recommended and/or preset by its fabricator, i.e., Umpqua Research Co. The system

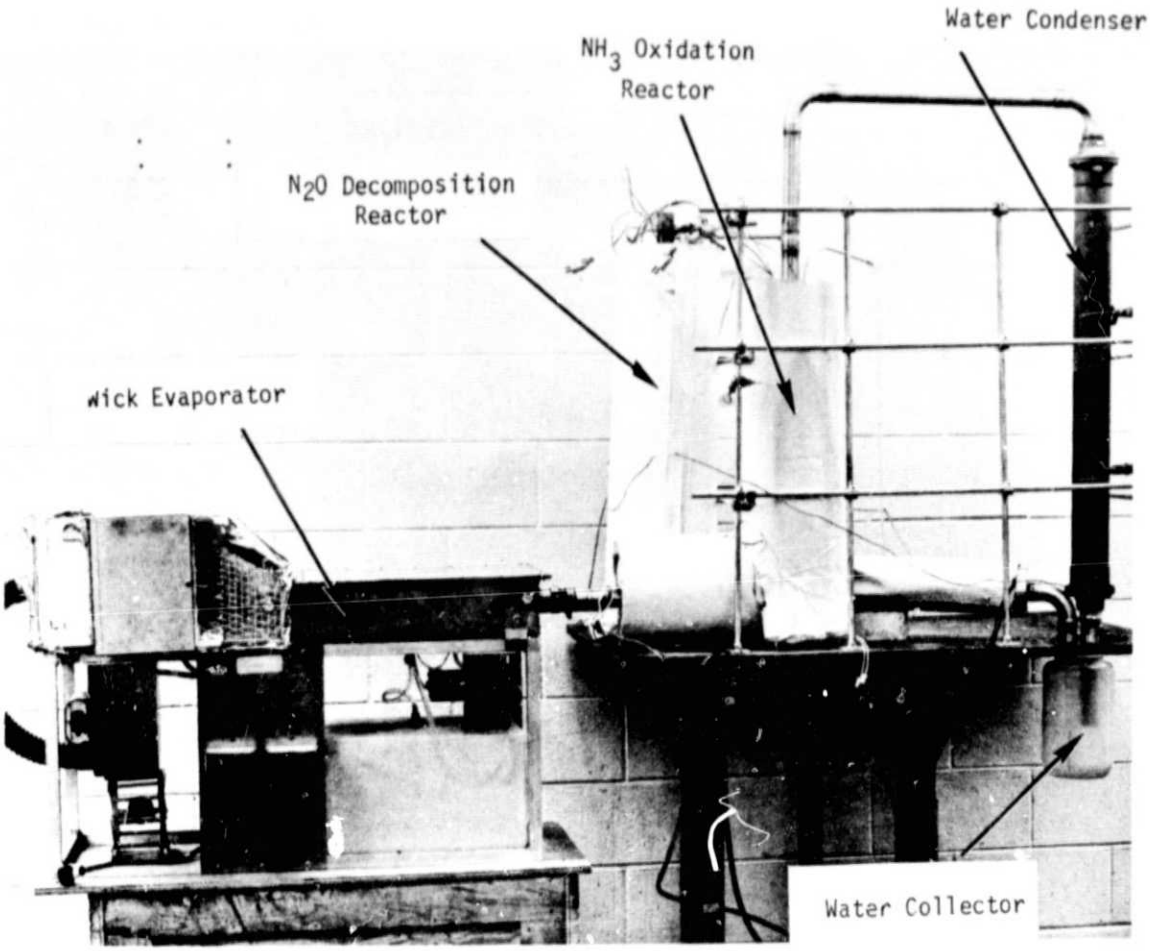

Figure 4 URINE TREATMENT SYSTEM

Table 9 TYPICAL TEST CONDITIONS

Catalytic System:		
	NH_3 oxidation catalyst	250°C
	N_2O decomposition catalyst	425°C
	Condenser	13°C
Wick Evaporator:		
	Air flow rate	250-300 l/min
	Air preheater	100°C
	Pump cutout	55°C
	Pumping cycle	5 sec
	Waiting cycle	90 sec
	Pump volume dial	8

was designed to achieve the maximum air flow rate of 340 l/min; however, only 300 l/min could be reached with the actual system. The difference was caused by the ΔP values higher than anticipated from the initial design calculations.

SYSTEM TESTING

Preliminary Tests

A series of tests was performed to become familiar with the overall operation of the integrated system and to ascertain that the operational conditions are adequate for the production of water having acceptable quality. For these tests, the system was restarted every morning, operated during the day, and shut down in the evening. Generally, the test conditions were the same as indicated in Table 9.

The experimental data obtained during the preliminary tests are summarized in Table 10. The overall quality of the recovered water does not change appreciably, except the concentration of NH_3 increases with each test. Apparently this is caused by the transfer of untreated urine vapor during the start-up stage, before the NH_3 oxidation catalyst reaches its operational temperature. As the wick becomes increasingly saturated with urine solids, more urea is decomposed and progressively higher concentrations of untreated NH_3 are transferred to the water. This is demonstrated by the results obtained during the last three tests. Tests No. 12 and 13 were performed on consecutive days without cooling the NH_3 oxidation catalyst at night, and resulted in a complete removal of NH_3. Then the catalyst bed was cooled overnight and test No. 14 restarted in the morning. The high NH_3 concentration found in product water clearly demonstrates the transfer of untreated urine vapor containing NH_3 during the start-up stage before the NH_3 oxidation catalyst is preheated.

Table 10. EXPERIMENTAL DATA OF THE PRELIMINARY TESTS

Test No.	Test Duration, min.	Water Collected		Analysis											
				Effluent gas			Product Water								
		Total, ml	Average Rate, ml/hour	NH_3, ppm	NO_x, ppm	N_2O, ppm	pH	NH_3, ppm	NO_x, ppm	Conductivity, Mhos/cm	Turbidity**	Color**	Odor	$SO_4^{=}$*	Cl^-*	Org. C, mg/l
1	330	1,250	227				4.4	N.D.	0.08	13×10^{-6}	< 0.2	< 5	None	+	+	0
2	330	820	149				4.6	0.2	0.1	14×10^{-6}	< 0.2	< 5	None	+	+	0
3	355	1,040	176				3.4	0.8	0.1	60×10^{-6}	< 0.2	< 5	None	+	+	0
4	340	1,700	300				4.3	0.6	0.08	25×10^{-6}	< 0.2	< 5	None	+	+	0
5	365	1,880	309	N.D.	N.D.	N.D.	4.1	1.1	0.2	20×10^{-6}	< 0.2	< 5	None	+	+	0
6	240	1,450	362	N.D.	N.D.	N.D.	4.4	0.9	0.2	28×10^{-6}	< 0.2	< 5	None	+	+	0
7	435	1,930	266				6.5	2.4	0.2	33×10^{-6}	< 0.2	< 5	None	+	+	0
8	317	1,180	223				7.0	4.8	0.3	53×10^{-6}	< 0.2	< 5	None	+	+	1.5
9	400	780	117				6.7	4.5	0.08	63×10^{-6}	< 0.2	< 5	None	+	+	0
10	358	295	49				4.9	5.4	0.4	68×10^{-6}	< 0.2	< 5	None	+	+	0
11	445	600	81				6.8	7.1	0.4	82×10^{-6}	0.4	< 5	None	+	+	5.3
12	355	910	154	N.D.	N.D.	N.D.	4.9	0	0.3							N.D.
13	445	1,353	182	N.D.		N.D.	3.7	0	0.1							N.D.
14	440	1,510	206	N.D.		N.D.	4.0	7.3	0.4							

* $SO_4^{=}$ and Cl^- present in very small concentrations

** Turbidity - JTU; color - chloroplatinate units

N.D. = Not detected.

Table 10 indicates that the average throughput rate varies unpredictably. An air stream preheated to 100°C and flowing at a 270 l/min rate has sufficient heat for raising the temperature from 25°C to 55°C and evaporating approximately 360 ml/hr of urine. Thus, the output of the wick evaporator operating at 100% efficiency cannot exceed 360 ml/hr using this flow rate of air. The observed rates indicate the evaporation efficiency ranging from 14 to 100%. In separate tests, attempts were made to determine the wick conditions that would result in the maximum output; however, no clear trends could be established. When the collection of water decreased to a very low rate, the preliminary tests were discontinued. An inspection of the wick indicated that the urine distribution tubes were filled with urine solids which partially closed the small openings and impeded the flow of urine to the wicking material, causing a decrease in the processing rate. Similar difficulty was experienced with all the wicks used for later tests.

Duration Tests

Three duration runs lasting from 73 to 94 hours were performed, using a new wick element for each run. During each run, the system was operated continuously until the output of the evaporator became too low for obtaining a significant rate of water recovery. An inspection of the wick elements after each run indicated a failure of the tubes distributing the urine through the wicking material. In each case the small distribution tubes were partially blocked up by urine solids, hindering transfer of urine to the wicking material and increasing the internal

pressure which caused slippage of the urine feed tube. Initially, this caused a decrease in the evaporation rate; however, eventually the increased pressure caused leaks at the connections of the supply tube and transfer of liquid urine into the catalytic system.

During each duration run, the entire system was operated automatically and did not require any manual adjustments; in fact it was attended only during the normal day working hours and was left to operate unattended during the night. Only untreated urine was used through the entire testing period. All tests were performed in a flow-through mode. Initially, operation of the system in a recycle mode was contemplated; however, recycling tests could not be performed because of leaks in the air evaporator.

Analytical Procedures.- Sampling and analyses were performed to obtain the processing rate and material balance, to characterize the feed and vent gases, and to determine the quality of the recovered water. To follow the progress of the process and possible changes occuring with time, two sets of samples were collected for analysis every 24 hours: one during the day; another, during the night.

Since this is a flow-through system using room air, the total moisture content of the feed consists of air moisture and the evaporated urine; similarly, the total product is the sum of the collected water and the moisture content of the vent gas, i.e., moisture + evaporated urine = condensed water + vent moisture. Therefore, the material balance cannot be obtained from the volumes of urine pumped to the wick and water collected alone. For this reason, the total

moisture contents of the feed and of the vent gas were determined from water collected by passing measured proportioned sampling streams of gas through silica gel traps. These measured values were then combined with the volume of recovered water to obtain the mass balance. Similarly, integrated samples of NH_3 and volatile hydrocarbons in the gas were collected by bubbling proportional sampling streams through a known volume of water. Gas samples were used for the analysis of the vent gas for NO_x and N_2O.

To summarize, the following samples were collected:

a. Feed gas: analysis for moisture, NH_3, organic carbon

b. Vent gas: analysis for moisture, NH_3, N_2O, NO_x, organic carbon

The following analytical techniques were used.

a. A Nesslerization method was used for ammonia. After the development of color by the Nessler reagent, ammonium ion concentration in the absorbing water is determined from spectral absorbance at 425 nm wavelength and is then related to the NH_3 concentration in the gas. The sensitivity of this method is approximately 0.3 mg/l of NH_3 in liquid which corresponds to 0.1 - 0.5 ppm in gas depending on the duration of collection.

b. A gas chromatographic method using a thermal conductivity detector and a Porapak Q column was used for measuring the N_2O concentrations. The detection limit of this method is approximately 1 ppm of N_2O in gas.

c. A modified Saltzman method was used for determining the NO_x (i.e., $NO + NO_2$) in vent gas and recovered water. Color is developed by reacting NO_2 with a reagent consisting of a mixture of sulfanilic acid, N- (1-naphthyl) ethylenediamine dihydrochloride, and acetic acid, and the concentration is read from the absorbance at 550 nm wavelength. Since this method measures only the concentration of NO_2, NO present in product gas must first be converted into NO_2. This is accomplished readily by mixing a sample of product gas with equal volume of oxygen. By employing adequate sample volumes, 0.1 ppm of combined NO and NO_2 in gas can be detected. Nitrous oxide (N_2O) does not interfere and is not detected by this method.

d. The organic carbon is determined by Beckman Total - Organic Carbon Analyzer. Organic carbon is catalytically oxidized to CO_2 which is then detected by an infrared analyzer. Using the above described collection techniques, it is estimated that approximately 0.2 ppm of organic C carbon in gas can be detected.

e. The analysis of the recovered water was obtained using methods described in "Standard Methods for the Examination of Water and Wastewater", 14th Edition, or equivalent procedures. The recovered water was routinely tested for pH, conductivity, NH_3, TOC, and NO_x. In addition, integrated samples of the recovered water were submitted to the Umpqua Research Co. for an independent and complete analysis.

The results of the analyses of feed and vent gases, and water collected during each day's/night's test are summarized in Table 11.

Table 11. DAILY ANALYSIS

Test No.	Time, min.	Air Flow, l/min.	Feed Gas			Vent Gas					Recovered Water				
			Moisture, g/l	NH_3, ppm	Organic C, ppm	Moisture, g/l	NH_3, ppm	Organic C, mg/l	N_2O, ppm	NO_x, ppm	pH	Conductivity, µMhos/cm	NH_3, mg/l	T.O.C., mg/l	NO_x, mg/l
Duration Run No. 1															
1	420	179	0.02612		N.D.	0.01696	N.D.	N.D.		0.1	5.7	15	N.D.	N.D.	0.6
2	1,020	151	0.02604	85	N.D.	0.01624	N.D.	N.D.		0.9	4.1	37	N.D.	N.D.	0.5
3	425	192	0.02704	53	N.D.	0.01572	N.D.	N.D.	N.D.	0.9	3.9	49	N.D.	N.D.	0.3
4	975	192	0.00960			0.01044								N.D.	
5	360	255	0.02178	5	N.D.	0.01572	N.D.	N.D.		1.0	3.7	70	N.D.	N.D.	0.1
6	975	243		51	0.6		N.D.	N.D.		0.9	3.6	146	N.D.	N.D.	0.1
7	200	250	0.01385	28	N.D.	0.01363	N.D.	N.D.	N.D.	0.6	3.1	146	N.D.	N.D.	< 0.1
Duration Run No. 2															
1	342	272	0.02613	4	14	0.01513	N.D.	N.D.	N.D.	0.3	3.5	52	0.4	N.D.	0.1
2	975	370	0.02950	57	125	0.01506	N.D.	N.D.		0.3	4.0	44	N.D.	N.D.	1.0
3	465	266	0.02949	67	11	0.01431	N.D.	N.D.		0.3	3.9	54	1.1	N.D.	0.5
4	975	272	0.02597	46	3	0.01503	N.D.	N.D.	N.D.	0.2	3.6	86	N.D.	N.D.	0.3
5	470	270	0.02708	44	7	0.01502	N.D.	N.D.	N.D.	0.3	3.6	84	N.D.	N.D.	0.4
6	970	272	0.02282	34	9	0.01493	N.D.	N.D.	N.D.	0.1	3.1	180	N.D.	N.D.	0.2
7	475	278	0.01875	6	26	0.01454	N.D.	N.D.	N.D.	0.1	3.1	270	N.D.	N.D.	0.3
8	980	269	0.01582	13	6	0.01461	N.D.	N.D.	N.D.		2.9	400	N.D.	N.D.	0.2
Duration Run No. 3															
1	290	191	0.01694	< 1	N.D.	0.01359	N.D.	N.D.	N.D.		5.7	22	N.D.	N.D.	0.4
2	965	170	0.02174	13	3	0.01399	N.D.	N.D.	N.D.	0.1	4.7	11	N.D.	N.D.	0.3
3	465	191	0.02169	47	8	0.01410	N.D.	N.D.	N.D.	0.1	4.0	39	N.D.	N.D.	0.3
4	975	219	0.02205	39	4	0.01391	N.D.	N.D.	N.D.	0.1	4.0	31	N.D.	N.D.	0.2
5	465	241	0.02031	59	12	0.01338	N.D.	N.D.	N.D.	< 0.1	4.0	30	N.D.	N.D.	0.2
6	975	229	0.01928	44	8	0.01333	N.D.	N.D.	N.D.	0.1	3.8	52	N.D.	N.D.	0.2
7	465	259	0.01638	24	32	0.01368	N.D.	N.D.	N.D.	< 0.1	3.5	114	N.D.	N.D.	0.1
8	980	234	0.01349	20		0.01291	N.D.	N.D.	N.D.	0.1	3.3	210	N.D.	N.D.	0.2

In addition to water vapor, the feed gas contains varying concentrations of NH_3 and volatile organic compounds. However, the vent gas contains no NH_3, organic carbon or N_2O.

Water Mass Balance.- Mass balance for each test of the three duration runs is presented in Table 12. For each test, the total amounts of water in feed and in vent gas were calculated from measured moisture contents, test duration, and test air flow rate indicated in Table 11. The difference between the moisture in feed and the vent loss indicates the calculated amount of water which should be collected as condensate. Since condenser was cooled by tap water to approximately 15° - 18°C, the vent losses were large; cooling to lower temperature would increase the volume of water collected as condensate. As anticipated, calculations indicate that the measured moisture content in vent gas equals the equilibrium vapor pressure of water at the condenser temperature, i.e., gas leaving the condenser is saturated.

The mass balances of the three duration runs are summarized in Table 13. The agreement between the calculated and the actually measured water collection values appears to be reasonable, particularly since the calculated values are based on measurements involving several variables, such as weight gains, gas flow rates, gas temperatures. Based on the above variables, it is estimated that the maximum possible error in the calculated amount of water collected is ± 16-17%. The contribution of the air moisture can be seen by comparing the measured content of water in feed gas with the volume

Table 12. DAILY WATER MASS BALANCE

Test No.	Water Mass Balance			Actually Collected, ml
	Feed, ml	Vent, ml	Calculated Collection, ml	
Run No. 1				
1	1,964	1,275	689	775
2	4,011	2,501	1,510	1,795
3	2,206	1,283	923	868
4	1,797	1,954	- 157	- 100
5	1,999	1,443	556	576
6	-	-	-	-
7	693	682	11	20
Run No. 2				
1	2,431	1,407	1,024	1,018
2	10,642	5,433	5,202	4,750
3	3,647	1,770	1,877	1,643
4	6,887	3,986	2,901	2,839
5	3,437	1,906	1,531	900
6	6,021	3,939	2,082	1,310
7	2,476	1,920	556	465
8	4,170	3,851	319	380
Run No. 3				
1	938	752	186	287
2	3,566	2,295	1,271	1,714
3	1,926	1,252	674	855
4	4,708	2,970	1,738	2,277
5	2,276	1,499	777	923
6	4,305	2,976	1,329	1,743
7	1,973	1,648	325	364
8	3,094	2,960	134	359

Table 13. MASS BALANCE OF WATER RECOVERY

Run No.	Duration, hrs.	Urine Treated		Water Mass Balance			Actually Collected, ml.	Difference,* %
		Total, ml.	Avg. Rate, ml/hr.	Feed, ml.	Vent Loss, ml.	Calculated Collection, ml		
1	53	7,260	137	11,977	8,456	3,521	3,914	+ 10
2	94	22,080	235	39,711	24,212	15,499	13,305	- 16
3	93	20,230	217	22,786	16,352	6,434	8,522	+ 24

* $\% = \frac{\text{Calculated - actually collected}}{\text{actually collected}} \times 100$

of urine pumped. During Run No. 1 the average relative humidity of room air was 40%, and the contribution of the air moisture was 39%; run No. 2 average relative humidity 44%, air moisture contribution 44%; run No. 3 average relative humidity 36%, air moisture contribution 19%.

Although the system was designed for treating urine at a rate of 0.325 kg/hr., this rate could be achieved only for a short time with a fresh wick. As the wick became saturated and urine solids were deposited in the distribution tubes, the output of the wick evaporator gradually decreased. The average urine treatment rates for the three duration tests were 137 ml/hr., 235 ml/hr., and 217 ml/hr. Because of this limitation imposed by the wick evaporator, the catalytic system was never exposed to its potential capacity for urine vapor.

There was no observable deterioration of the catalyst activity during the entire testing period. When a failure of the wick element caused introduction of liquid urine into the catalytic system, high concentrations of chloride appeared in the recovered water and corrosion of the stainless steel tubing connecting the oxidation catalytic reactor with the water condenser was observed; however, the removal of ammonia was not affected. To remove the urine solids deposited within the system, water condenser, connecting tubing, and the ammonia oxidation catalyst were backwashed with hot water. After drying with a stream of hot air, the catalyst remained active and showed no deterioration.

Quality of the Recovered Water.- The recovered water collected during each duration run was analyzed for pH, specific conductance, ammonia, TOC, turbidity, odor, color, and nitrate nitrogen. In addition,

samples were submitted to the Umpqua Research Co. for independent analysis. The results of these analyses are summarized in Table 14. The results obtained by GARD and by Umpqua Research Co. are in good agreement, particularly considering the small values and the amount of sample handling involved.

The quality of the recovered water can be judged by comparing the analytical results with the EPA Primary and Secondary Standards for Drinking Water incorporated into Table 14. The recovered water meets all the EPA drinking water requirements, except pH and mercury. The low pH is characteristic for the catalytic process primarily because small amounts of NO_x and Chloride are formed producing acid in the recovered water. In addition, it is possible that some volatile carbon compounds may be oxidized to acids which condense with the water. Since the recovered water is unbuffered, low concentrations of these acidic components produce a significant change in the pH value. The pH of the recovered water can be easily corrected either by adding alkalies or by passing through a small ion exchange bed. It is estimated that raising the pH to a value of 7 would require only 110-120 g of NaOH per year for a 4-man system. The high concentration of mercury found in water is due to contamination introduced by an inadvertent use of an open mercury manometer safety switch that would shut off the system in case of blower failure.

Table 14. ANALYSIS OF RECOVERED WATER

Parameter	Units	EPA Primary & Secondary Standards	Run No. 1		Run No. 2		Run No. 3	
			GARD	Umpqua	GARD	Umpqua	GARD	Umpqua
pH	pH units	6.5-8.5	4.0	3.8	3.6	3.6	4.1	3.9
Specific Conductance	umho/cm		43	54	76	92	27	40
Ammonia	mg/l		< 0.3	0.44	0.3	0.40	< 0.3	0.13
T.O.C.	mg/l		N.D.	2.5	N.D.	1.2	N.D.	0.9
Turbidity	JTU & F		< 0.01	0.8	< 0.01	N.D.@0.1	0.1	0.1
Odor	T.O.N.	3	N.D.	1	N.D.	N.D.@1	N.D.	N.D.@1
Color	Color Un.	15	< 5	5	< 5	5	< 5	5
Nitrogen, Nitrate	mg/l	10	0.4	1.0	0.5	0.7	0.6	0.9
Nitrogen, Nitrite	mg/l			N.D.@0.01		N.D.@0.01		0.13
Solids, total	mg/l	500				1.8		3.0
Solids, volatile	mg/l					N.D.@0.1		N.D.@0.1
Arsenic	mg/l	0.05		N.D.@0.01		N.D.@0.01		N.D.@0.01
Barium	mg/l	1.0		N.D.@0.1		N.D.@0.1		N.D.@0.1
Cadmium	mg/l	0.01		0.0019		0.0015		0.0014
Calcium	mg/l			0.73		0.69		0.92
Chromium	mg/l	0.05		N.D.@0.02		N.D.@0.02		N.D.@0.02
Copper	mg/l	1		0.14		0.04		N.D.@0.01
Iron	mg/l			0.38		0.29		0.09
Lead	mg/l	0.05		0.106		0.023		0.019
Magnesium	mg/l			0.09		0.09		0.04
Manganese	mg/l	0.05		0.01		0.01		N.D.@0.01
Mercury	mg/l	0.002		0.24		0.28		0.079
Selenium	mg/l	0.01		N.D.@0.002		N.D.@0.002		N.D.@0.002
Silver	mg/l	0.05		N.D.@0.01		N.D.@0.01		N.D.@0.01
Silica	mg/l					0.64		0.21
Zinc	mg/l	5		0.22		0.08		0.18
Chloride	mg/l	250	2.4	2.6	4.8	5.59	0.6	1.5
Fluoride	mg/l	1.4-2.4		1.0		1.9		0.8
Sulfate	mg/l	250	N.D.		N.D.	0.29	N.D.	N.D.@0.5
Cyanide	mg/l					N.D.@0.02		N.D.@0.02

N.D. = Not detectable at limits indicated.

CONCLUSIONS AND RECOMMENDATIONS

Based on test data obtained during this investigation, the following conclusions can be made:

1. Water recovered from untreated urine by the integrated dual catalyst-wick evaporator system meets the drinking water standards, with the exception of a low pH.

2. The system was capable of processing urine at the design processing rate of 0.325 kg/hr. with a fresh wick; however, the rate decreased as the wick became progressively saturated with urine solids. The average urine processing rates observed during the three uninterrupted runs lasting up to 94 hours were 0.137 kg/hr., 0.217 kg/hr. and 0.235 kg/hr.

3. The urine processing and water recovery rate is limited by the rate of urine vapor generation by the wick-evaporator.

4. After start-up, the system operates automatically and requires no adjustments or supervision, except periodic supply of urine and removal of the recovered water.

5. The activity of the catalysts remained unchanged during the entire testing program consisting of short duration runs and three successive endurance runs of continuous operation for 73, 94, and 93 hours.

6. The design of the wick and its controls requires additional investigation because the present configuration results in blockage of the urine distribution tubes by urine solids after a short time,

long before the wick intself is loaded with solids.

Since the wick-evaporator produces an air-vapor stream containing only 3-5% of urine vapor by volume, large quantities of air must be passed through the catalytic system to achieve the four-man rating, increasing the weight, volume, and power requirements. It is recommended to investigate the integration of the catalytic system with some evaporative system capable of producing high urine vapor concentrations. The use of feed containing high urine vapor concentrations would minimize the size and power requirements of the catalyst system. Since inhibition of urea decomposition would not be required, the urine pretreatment chemicals could be eliminated and the evaporative system could operate at higher temperatures, increasing its capacity.