LOW TEMPERATURE VAPOR PHASE CATALYTIC OXIDATION UNIT

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TABLE OF CONTENTS

LIST OF ILLUSTRATIONS

LIST OF ILLUSTRATIONS

ABSTRACT

A low temperature vapor phase catalytic oxidation unit using Ardox catalyst has been designed, built and tested for its ability to oxidize organics entrained in vapor from the vacuum distillation of urine.

Performance data for the unit are included in this report, and estimates are made for catalyst life, power requirements, and capabilities of the unit.

SUMMARY

A low temperature, vapor phase catalytic oxidation unit using Ardox catalyst has been developed. It consists of a heated catalyst bed (containing a thermistor, temperature controller, and thermocouple) and an insulated heat exchanger.

The unit has been tested and was found to operate satisfactorily in the temperature range 140F to 250F. It is felt that lower operating temperatures may also be effective, and it is known that temperatures to 350F are tolerable.

The unit is used in conjunction with simple laboratory glassware, with which urine is distilled through the catalyst bed in the presence of air, and the vapors condensed.

The catalyst reduces the level of organics in urine vapor, as measured by COD and TOC values. Ammonia is not effectively oxidized by the catalyst, but when a pretreatment of the urine is carried out to prevent hydrolysis of urea to ammonia, a high quality water is recovered. Estimates of catalyst life have been made from the data collected.

Power requirements and capacity of the unit have been estimated. It is believed that minor modifications in design and in catalyst composition may be introduced to greatly increase the capacity of the unit and catalyst life, with only modest increases in weight.

INTRODUCTION

The recovery of potable water from urine,wash water and other sources is essential for long duration space missions.

A number of promising approaches to purification appear feasible, including air evaporation, membrane vapor diffusion, vacuum and compression distillation, electrodialysis, with electrolytic pretreatment and reverse osmosis.

Vacuum distillation of water from space wastes effects a substantial improvement in quality of such wastes, but alone is insufficient to yield a potable product. Three major pollutants may occur in such recovered water: 1) bacteria, 2) ammonia, and 3) volatile organic contaminants whose offensive odor and taste make the product non-potable.

Charcoal has been used to trap these odiferous compounds, but does not have sufficient capacity nor effectiveness. High temperature catalytic oxidation may be used effectively, but introduces a weight penalty due to the power required and weight of thermal insulation needed to shield the cabin from excess heat.

A low temperature catalyst, Ardox was developed at ARDE which effectively removes these organics with lower power requirements.

Under a contract with NASA (NAS 1-7025) this catalyst was incorporated into a designed light-weight piece of hardware consisting of a low temperature catalyst bed with an insulated heat exchanger. This unit was then tested by distillation of aqueous solutions of various organic compounds, aqueous solutions of ammonia, and urine through the catalyst bed.

The distillation of dilute solutions of pure organic compounds through the bed yields information on the expected behavior of constituents of urine during distillation. It also pointed out necessary modifications in the laboratory test set-up.

The distillation of urine was then carried out under varied conditions of distillation temperature and catalyst bed temperature, and the recovered water analyzed. This work constitutes the main subject matter of this report.

DESIGN

Catalyst Bed

The catalytic oxidation unit can be broken down into two component sections; a heated catalyst bed; and an insulated heat exchanger whose purpose is to minimize power requirements for the unit.

The catalyst bed consists of an annular cylindrical vessel which contains a helical bed heater, thermocouple, thermistor control, and approximately 130 grams of supported catalyst.

Heat Exchanger

The heat exchanger consists of a bundle of teflon tubes held between two aluminum perforated plates. Teflon was used to prevent axial heat loss in the exchanger tubes through conduction. The entering vapor passes around the heat exchanger tubes, through the heated catalyst bed, and down through the exchanger tubes out into a plenum chamber. The heat exchanger is housed in a teflon cylinder. Around the teflon cylinder is contained a powdered insulation consisting of finely divided aluminum power and santocel. The outer aluminum jacket is sealed to the heat exchanger via "O" rings at the top and bottom of the exchanger. The annular insulation chamber is evacuated during use to complete the insulation system.

EXPERIMENTAL

A. Assembly and Start Up of Unit

After completion of fabrication of the catalytic oxidation unit, the unit was assembled, and the catalyst bed charged with granular alumina (8 to 14 mesh). Alumina is the catalyst substrate, and was installed for the sake of running control experiments.

Insulation was poured into the vacuum jacket, and the unit sealed. A distillation apparatus was assembled from conventional laboratory glassware to allow vacuum distillation of test solutions and urine through the catalytic unit (see Figure 1). The distillation apparatus used in initial tests consisted of a three-neck five-gallon pyrex evaporator with provisions for air inlet, thermometer, entrainment separator and vapor tube to the catalyst unit. The catalyst unit was mounted on a conventional laboratory "flexaframe" support. The exit port of the catalyst unit was connected to two Graham-type condensers in series, which led to a receiving flask chilled in ice-water. The outlet of the receiving flask was connected to a liquid nitrogen trap, followed by a vacuum pump. Air was fed into the evaporator through a gas-flow meter to ascertain flow rate of air entering the unit.

A thermocouple, heater and temperature sensing probe are part of the catalyst bed make up, and served to monitor and control the catalyst bed temperature.

During start up, difficulties were encountered with a leak in the vacuum jacket. Rather than delay testing to find this leak, most of the testing was carried out without vacuum insulation. In addition, after some heater repairs and replacements, testing was carried out without powdered insulation, which was tedious and time consuming to install. Since the objective of these initial tests was to determine the oxidizing capability of the unit rather than establishing the power requirements, operations with insulation for power tests were deferred to later in the test program. Removing the insulation required operation at higher power levels. However, operation at higher power levels has no bearing on the validity of the oxidation tests.

B. Pure Compound Runs

Initial check out tests of the apparatus were carried out with distilled water. Then, dilute solutions of several pure compounds were consecutively distilled through the catalytic unit with alumina in the catalyst bed. These constituted the control distillations. Pure compounds chosen for the initial runs were acetic acid, formic acid, hippuric acid, phenol, creatine, skatole and ammonia. Ten liters of each solution were prepared and fractionally distilled.

Several distillate samples were collected on each run and the corresponding liquid nitrogen trap contents added to each sample. The total sample volume was measured and aliquots saved for analysis. Samples of the distilland were also set aside for analysis. The analyses used were chemical oxygen demand (COD)

for the organics, and colorimetric analysis using Nessler's reagent for ammonia. Each pure chemical was distilled in sequence without purge of the distillation system. Subsequent analysis of the data showed that tailing of earlier runs into later ones occurred. This tailing interfered with the precise interpretation of test data.

In later distillations, a one-neck distillation bottle was substituted for the three-neck bottle with no loss of convenience, and connecting hoses between system components were shortened as much as possible. The latter change reduced the tailing effects between consecutive runs by reducing the amount of fluid retained by the system (See Figure 2).

After the pure compound control-test distillations were carried out, the catalytic unit was opened and the alumina replaced with Ardox catalyst. Dilute solutions of pure compounds were then distilled through Ardox catalyst.

Ammonia was distilled through the unit in both a control run with alumina in the bed and a catalytic oxidation test run. These tests were first carried out in the same manner as the dilute solutions of organic compounds. However, it was found that considerable losses of ammonia occurred and that therefore material balances and interpretation of oxidation test data were impossible. The ammonia distillation tests were repeated, using rigorous precautions to trap the volatile ammonia. These precautions consisted of a train of three sulfuric acid traps in addition to a liquid nitrogen trap, partially filled with dilute sulfuric acid.

Data obtained in this experiment showed a good material balance, as can be seen in Table XIII.

C. Urine Distillation

Urine distillations were carried out in a manner similar to the pure compound runs except that the apparatus was modified to allow urine vapor to be passed either through the catalyst bed, or condensed directly without contact with the catalyst. This change was instituted because urine composition varies from batch to batch as well as with time and consequently so does the urine vapor composition. With this modification of testing procedure, it was possible to monitor the urine vapor composition whenever desired. In practice, a sample of urine vapor (A, Table XV) was condensed before the distillation test through the catalyst. After each distillation test was completed, a second sample of urine vapor (B, Table XV) was collected. Bypass samples A and B were analyzed for TOC, while the oxidized distillate samples were analyzed both for TOC and COD. Data for these "bypass" samples were compared to data on samples distilled through the catalyst in order to ascertain the effectiveness of the catalyst in reducing organics in urine vapor.

In the last four urine distillations, the urine was pretreated with a sulfuric acid - chromic acid mixture, which serves to arrest urea hydrolysis. This pretreatment permitted the determination of catalyst effectiveness in the absence of ammonia.

D. Power Requirements

Upon completion of testing the catalytic unit was opened, the used catalyst removed, fresh catalyst installed and insulation put in place. The outer vacuum jacket was evacuated and was brought to a vacuum of about 500 microns. It was suspected that some naphtha, (which is present in the aluminum powder) might be distilling off and hence limiting the vacuum. However, it was also found that the limiting vacuum of 500 microns was close to the maximum capability of the vacuum pump. Power tests were run at this level of vacuum insulation. A better vacuum should reduce power requirements levels measured here.

In order to establish power requirements, water was distilled through the catalyst unit, and inlet and outlet temperatures and pressures recorded. Voltage and amperage to the heater were measured. From these data, and the time required for distillation of a measured volume of water, an estimate of power requirements was made for use of the unit at different distillation temperatures.

In addition, a pyrometer was used to measure the outer jacket wall temperature of the catalytic unit, in order to compare analytic estimates of power losses by convection from the outer wall, with measured losses.

RESULTS AND DISCUSSION

A. Control Distillations of Pure Compounds

Of particular importance in the investigation of the pure compounds is to determine whether they tend to distill or remain with the residue. Control distillations were already necessary for the study of oxidation of these pure compounds. These experiments afforded a convenient means of obtaining direct information relating to their distillation characteristics.

Acetic acid was distilled through the catalyst at two distilland concentrations; 5 ml of glacial acetic acid per 10 liters solution (see data in Table I) and 10 ml of glacial acetic acid per 10 liters solution (see Table II). The control distillations showed a marked fractionation of the binary mixture with acetic acid tending to remain behind in the evaporator as water-rich vapor preferentially distills over.

In order to clarify the significance of data in the tables, a detailed interpretation of data in Table I is given here. Samples A through G represent successive batches of distillate collected from the vacuum distillation of a solution of five milliliters of glacial acetic acid dissolved in ten liters of distilled water. The sample labelled "residue" represents the acetic acid solution remaining in the distillation flask at the end of the experiment. The sample labelled control is an aliquot of the original solution before distillation.

The COD value for each sample is the chemical oxygen demand, expressed in mg oxygen per liter as determined by laboratory analysis. The volume for each sample is expressed in liters. The total under this column is the sum of volumes of all samples measured in the experiment. The difference between this value (9.760 l) and 10.000 l represents material losses incurred during the experiment.

The data in the column labelled "Total COD" is the multiplication product of the data in the column "COD"in mg/liter and the data in the "volume" column, which gives the total number of milligrams of oxygen required to oxidize all of the organic content of the collected sample. The sum of these data in the "Total COD" column (30001.1 mg) is the total mg of oxygen needed to oxidize all of the samples and residue and control as measured experimentally. The number in parenthesis (4861) is calculated from the COD of the control sample in mg/liter, multiplied by 10 to give the total starting COD for the entire starting solution.

The difference between 4861 and 3001.1 represent experimental losses of material, oxidation occurring in the absence of catalyst, or any other losses of COD, due to experimental error or other factors.

In experiments where the measured sum of the total COD is greater than theory, it indicates that contamination from

the previous run was carried over into the collected samples. An example of this situation is seen in Table IV.

Formic acid likewise fractionated in the same way as acetic acid, and tended to remain behind in the evaporator, as is readily apparent from analysis of the data in Table III.

Examination of the data in Table IV for hippuric acid shows that the hippuric acid (as measured by COD values) remains completely behind in the evaporator residue. Phenol preferentially distilled over and therefore concentrated in the distillate (Table V). Creatine evidently has little tendency to distill, as can be seen from the residue concentration (in Table VI) for this compound.

The distillation of a dilute skatole solution caused considerable difficulty during the control tests. Skatole preferentially distilled over, and condensed to a solid in the catalytic oxidation unit and in the condensers, blocking the vapor flow. Because of the great difficulties with skatole, only two fractions of distillate were collected (see Table VII).

In the next consecutive control, ammonia was distilled (see Table VIII). The ammonia samples were contaminated with skatole, which, however, probably did not interfere with the Nessler's test for ammonia.

In summary then, phenol and skatole distill over readily while hippuric acid and creatine do not appear to distill over. Acetic and formic acids lie in between in terms of this characteristic.

A particular problem encountered during these runs was the tailing-off of a compound into the next consecutive run. This made the distillate data difficult to interpret.

For example, distillation of dilute hippuric acid solution showed a drop off of distillate COD for consecutive samples (see Figure 3).

However, since analysis of the evaporator residue showed that hippuric acid was not distilling over, it is obvious that the COD found in the distillate samples on this run actually represents formic acid contamination from the previous run. This is a clear cut indication of tailing of organics from one run into the following run, and this problem causes some difficulty of interpreting these test results.

Similarly, examination of the "creatine data" on the histogram (see Figure 4) leads to the conclusion that the COD observed in the distillate was phenol, which was still tailing into the distillate from the previous experiment. Note that in order for this COD to actually represent creatine, the distillate concentration would have to <u>increase</u> with time as

the residue concentration of creatine increased. The drop off in COD in successive distillate samples is convincing evidence that the COD is actually phenol.

B. Catalytic Oxidation of Pure Compounds

Only acetic acid, formic acid, phenol and ammonia were distilled through Ardox catalyst. Hippuric acid and creatine were not run with the catalyst because they were found not to volatilize in the control runs. Skatole was not run because of the great difficulties encountered with it in the control run. The data from the catalytic oxidation runs is shown in Tables IX to XII, and in Figure 5. Evaluation of this data is discussed below.

The basis of comparison used to evaluate the effect of the catalyst is to compare, for any given volume of solution distilled, the total COD observed in runs with and without catalyst. This approach is valid because for any given concentration of an initial binary mixture, the final cumulative concentration of the distillate depends only on the fraction of the starting volume distilled over. However, this is true when all of the distillate is caught and when no interferences (such as the tailing effect previously noted) occur.

Let us first consider the effect of loss of distillate from the system. The volumes of water lost were small, usually

under 5%, and could be corrected for by assuming that the loss was proportional to the volume of the fraction distilled. The loss of solute from the distillate depends on its volatility. For acetic acid and formic acid, these losses were probably negligible. For phenol, these losses would not be negligible, but are proably equal in the control and catalytic oxidation runs.

Let us next consider the errors due to the tailing of one run into the next. In some of these experiments, the quantity of material tailing was large compared to the amount apparently oxidized under the process conditions used in these tests, and therefore, it is difficult to interpret such data quantitatively. However, we have sufficient data to permit the comparison of catalyst and control runs for acetic acid and phenol.

1. Acetic Acid Data

The control runs on acetic acid were the first made on the alumina control bed. Therefore, no interference from tailings of a previous run was encountered. Two concentrations of acetic acid were run, however, and it might be argued that the first acetic acid run affected the second. Referring to the acetic acid data in Tables I and II indicates the residue of the dilute acetic acid control (residue 830 COD) run was virtually the same as the initial concentration of the starting

material in the second acetic acid run (initial concentration 1025 COD). On this basis the error due to tailings from the first acetic acid control run were considered to be negligible in the second. Some small amount of initial dilution of the distillate in the second run is all that might be expected.

The catalytic oxidation run with acetic acid, Table IX, was again the first material through fresh catalyst bed. Just prior to this run, about 6.6 liters of NH_3 - water mixture had passed through the system, effectively cleaning it reasonably free from contaminants showing COD. This run was made with acetic acid solution at approximately the same concentration as the second acetic acid control run. A comparison between these runs was therefore considered useful and the results are shown in Figure 6. The plots show the cumulative COD of the distillate as a function of the cumulative volume distilled. Note that when 70% of each starting solution was distilled that a difference in cumulative COD (i.e., total COD collected) of 650 mg/l is observed. The control showed a total COD of 2530 at this point. A reduction of 25.7% was attributable to the presence of the catalyst.

2. Phenol Data

A similar comparison of distillate COD from control run and catalyst run was made with phenol. Examination of the control run data indicates that the run with hippuric acid is

expected to have little effect upon the phenol control run. The COD of the last sample of 1.3 liters from the hippuric acid control was only 40 mg/l which is negligible in comparison with the COD's obtained with phenol. The only effect would be slight dilution of the initial fraction of phenol control distillate. The catalyst run, however, did suffer somewhat more of an effect from the tailings of the run preceding it. Formic acid run (Table X) was conducted prior to the catalyst test with phenol. However, the COD of the last two liters from the formic acid run exceeded that from the hippuric acid run. It would seem, therefore, that comparison of the phenol control run with the catalyst run could reflect only errors which tended to diminish the effect of the catalyst.

The comparison of catalytic oxidation and control runs on phenol (see Figure 7) indicates that when 70% of each initial material has distilled over, a difference in 3200 mg/l is observed. Since at this point a total of 16200 mg/l had distilled from the control, a reduction in COD of 19.7% could be attributed to the catalyst.

3. Ammonia Data

The ammonia data in Table VIII show a very poor material balance. Subsequent work showed that unless very careful precautions were exercised ammonia was lost through the liquid nitrogen trap into the vacuum pump, and also through

routine handling of the samples in the laboratory. Therefore, the data on this control run could not be used for evaluation of the catalytic oxidation of ammonia.

After these preliminary experiments with ammonia demonstrated the difficulty of achieving successful material balances, special precautions were instituted to insure that ammonia would not be lost from samples through handling or through the liquid nitrogen trap into the pump. The result of this exercise was quite interesting, and points out the difficulty of working with ammonia solutions.

For example, in Table XIII, the data collected in a control distillation of ammonia are shown. In this experiment there was no contact with catalyst, and therefore no opportunity for catalytic oxidation. A comparison of analytical data collected on the distillate sample with the trap samples shows that the trap picked up almost twice the amount of ammonia as was found in the distillate sample (2.08 grams as compared to 1.16 grams). Therefore, if special precautions had not been taken to catch all of the distilled ammonia, approximately two-thirds of the distilled ammonia would have been lost.

Another point to be observed in these data is that the trapping techniques used were effective in accounting for all of the ammonia, and all of the water used in the experiment

(103.6% and 99.0% respectively). This gives us confidence that the data in the catalytic oxidation test with ammonia (shown in Table XIV), which were obtained under the same precautions should be just as accurate and reliable.

In the catalytic oxidation test, 91.5% of the ammonia, and 98.5% of the water were recovered. These data indicate that a small amount of ammonia (8.5%) may have been oxidized in this test. However, the low level of removal (oxidation, adsorption, or otherwise) does not hold out much promise for effective oxidation of ammonia with Ardox catalyst.

C. Physical Chemistry of Urine Distillation

Examination of the distillation data on dilute solutions of pure compounds serves a useful purpose. It allows us to reflect on what happens during the distillation of such a complex mixture as urine.

1) It is immediately obvious (from the hippuric acid and creatine data) that certain components of urine would not distill, and therefore do not need to be oxidized or otherwise removed from the vapor. Therefore, one can look at an analysis of urine (such as is given in the NASA Bioastronautics Data Handbook) and predict that the greatest weight fraction of dissolved constituents of urine will not distill, and therefore really are not a problem (except that they constitute waste

solids which must eventually be disposed of or stored). This group of constituents includes the amino acids, salts, sugars, hormones, vitamins, urea and many other constituents.

2) On the other hand, there are other highly volatile and strongly odiferous constituents in urine, many of which have not been identified in the urine analyses, which distill much more easily than water (an important constituent of this group is ammonia). These compounds as a group are largely found in the liquid nitrogen trap after a urine distillation. Although their total mass (or volume) contribution to urine is small, their pungent odors allows them to contaminate water recovered during a urine distillation to render it non-potable. These constituents must be destroyed or separated from the vapor. It would seem worthwhile to consider a flash distillation of these volatiles from urine out to space, to eliminate them from the urine without a significant loss of water vapor, as part of any water recovery system.

3) A third class of urine constituents represent compounds which have volatilities comparable to that of water. These cannot be separated quantitatively from the water vapor in any practical way by distillation and must be removed by catalytic oxidation, adsorption, or other procedures.

D. Urine Distillations

A series of consecutive distillations of one-day old human (male) urine were made through the catalytic oxidation unit to determine the unit's efficiency to oxidize the organics and ammonia present in urine vapor. The tests were also carried out to compare urine distillations at different vaporization temperatures. A second variable was introduced, that of the catalyst bed operating temperature, in order to collect data for estimation of the unit's power requirements. The distillation rate was generally kept slow, but distillations were carried out over a range of distillation rates 51-396 ml/hour. Distillate from initial runs showed excellent removals of organics. Ammonia levels started low but quickly built up to unacceptable levels in successive runs. With continued use there was a gradual fall off in catalytic oxidation efficiency with a rather large spread in efficiency in day to day tests. The large spread may partly be due to differences in operational parameter levels used in the series of experiments, and may also be due to differences in the quality and composition of urine used in the tests. Nevertheless, the data show a trend, and from this trend one can extrapolate a useful life for the catalyst, before regeneration is necessary.

There is reason to believe that a part of the loss of catalyst efficiency is caused by chemical degradation of the catalyst by chemically reactive species in urine vapor. The

relative fraction of efficiency loss caused by side reactions and that caused by need for regeneration has not yet been determined, but it is hoped that chemical analysis of the catalyst bed used for the urine distillations will provide an answer to this question.

The results of urine distillations carried out are shown in Table V. As can be seen from these data, distillation of urine through the catalyst bed results in a reduction in chemical oxygen demand (COD) and total organic carbon (TOC) of the recovered water as compared to the condensed urine vapor which was diverted past the catalyst bed.

We believe that the efficiency of oxidation is dependent on the "residence" or "stay" time of vapor in the catalyst bed.

When the catalyst unit was used at the design goal distillation rate, poor efficiency of oxidation of organics resulted. However, at one third this flow rate, good results were obtained. These results indicate that a longer residence time is required for the urine vapor in the catalyst bed. This can be achieved by increasing the weight of catalyst used in the unit, which, fortunately, does not require a proportional increase in hardware weight.

Water obtained during the test runs had good reductions in organic levels, but was contaminated with ammonia, which

caused a noticeable odor. When ammonia was eliminated by pretreatment with a chromic and sulfuric acid mixture, efficiency of removal of organics was approximately the same as without pretreatment. However, elimination of the ammonia results in water with excellent odor. Therefore, it would seem that pretreatment of urine to eliminate ammonia followed by distillation of the urine through Ardox catalyst at a (low) elevated temperature would offer an effective route to recovery of potable water from urine.

In other tests, not part of this program, it has been found that water obtained by distillation of urine through Ardox catalyst at 300°F is essentially free of pathogenic types of bacteria normally associated with urine. Bacterial tests were not carried out on samples collected in this program but we believe that the water recovered was probably likewise essentially sterile.

Analyses of "by-pass" samples of urine vapor in these runs show that a substantial fraction of the volatile organics are eliminated by the pretreatment.

The consequence of this fact is that pretreatment reduces the load on the catalyst bed, and therefore would no doubt greatly extend the life of the catalyst bed beyond the estimate made here on the basis of non-treated urine.

Comparison of results on treated and non treated urine samples shows no great difference in % reduction of organics. Since the untreated urine vapor contained copious amounts of ammonia, this "lack of difference" between treated and untreated urine suggests that ammonia does not have an adverse effect on the action of the catalyst.

E. Energy Requirements

Energy requirements were calculated from test data carried out under various operations. The energy requirements are summarized in Table XVI.

Energy requirements ranged from 4.76 to 7.74 watt hrs. per lb. of distillate. The tests were made at flow rates ranging from .76 to 3.12 lbs. per hr. The previously described vacuum pump limitation permitted a jacket pressure only as low as 500 microns to be achieved. It is expected that more efficient evacuation of the insulating vacuum jacket would further reduce the energy requirements. However, the low energy level of 5 watt hours per lb. of distillate, required at anticipated flow rates, is certainly a useful feature of the Ardox reactor design.

CONCLUSIONS

1) The use of Ardox catalyst for the catalytic oxidation of urine vapor constituents results in a marked reduction in concentration of organic species.

2) Ammonia is not effectively oxidized by Ardox.

3) The catalyst unit has minimum power requirements of about 5 watt-hours per pound of water collected.

4) Pretreatment of urine with chromic acid-sulfuric acid mixture, followed by distillation through Ardox yields water with no noticeable odor and a low concentration or organic contaminants.

5) It is estimated that 50 pounds of water can be recovered from urine per pound of catalyst used, if no catalyst regeneration is used.

6) Catalyst regeneration would no doubt allow much more water to be recovered before the catalyst loses its effectiveness.

7) A simple modification of catalyst preparation to include a higher percentage of active material could further increase the output of the catalyst unit in terms of pounds of water recovered per pound of catalyst used.

RECOMMENDATIONS

The catalyst unit should be redesigned, as shown in Figure 8 to allow more catalyst to be used in the unit. This will not cause a great increase in weight, but would triple the output of the catalytic unit.

Ardox should be used to oxidize organics in urine vapor from pretreated urine or vapor otherwise freed from ammonia.

Tests should be carried out at still lower catalyst bed temperatures, since the optimum bed temperature has not been established.

The distillation procedure for recovering potable water from urine should include a flash distillation of ammonia and highly volatile organics, prior to pretreatment, to gain greatest benefit from any water recovery system.

The distillation apparatus should provide opportunity to collect the distillate hot, and for reflux of the vapors from the hot distillate to drive off volatiles. The adaptation of this idea to zero gravity systems, however, will require some creative imaginative engineering.

TABLE I

TEST RESULTS

DISTILLATION OF CONTROL SAMPLES

Acetic Acid 5 ml/10L

SAMPLE	COD mg/L	VOLUME Liters	TOTAL COD mg
A	39.2	.190	7.0
B	27.4	.920	25.2
C	15.7	.940	14.8
D	15.6	.820	12.8
E	19.5	.990	19.3
F	54.6	1.700	92.8
G	85.8	.770	66.1
Residue	830.7	3.180	2641.6
Control	486.1	.250	121.5
Total		9.760	3001.1

(Calculated value 4861.0)

TABLE II

TEST RESULTS

DISTILLATION OF CONTROL SAMPLES

Acetic Acid 10 ml/10L

SAMPLE	COD mg/L	VOLUME Liters	TOTAL COD mg
A	115.2	1670	192.4
B	276.5	1725	477.0
C	564.5	3290	1857.2
Residue	Lost (Est.* 1755)	2810	4930.4 Est.
Control (Taken after 1670 MLS Distilled)	1213.4	300	364.0
Total		9795	7821.0 Est.
		(Calculated value 10256.0)	

* Estimate based on previous experiment (Acetic Acid 5 ml/10L

TABLE III

TEST RESULTS

DISTILLATION OF CONTROL SAMPLES

Formic Acid 10 ml/10L

SAMPLE	COD	VOLUME	TOTAL COD
A	389.6	3390	1320.7
B	420.2	1390	584.1
C	301.8	820	247.5
D	500.4	1310	655.5
Residue	1062.0	2680	2846.2
Control	405.0	~300	121.5
Total		9890	5775.5

(Calculated value 4050.0)

TABLE IV

TEST RESULTS

DISTILLATION OF CONTROL SAMPLES

Hippuric Acid 10 grams/10L

SAMPLE	COD	VOLUME	TOTAL COD
A	272.0	1965	534.5
B	228.0	920	209.8
C	76.0	1450	110.2
D	52.0	1310	68.1
E	40.0	1300	52.0
Residue	7400.0	2620	19,388.0
Control	804.0	300	241.2
Total		9865	20,603.8

(Calculated value 8040.0)

TABLE V

TEST RESULTS

DISTILLATION OF CONTROL SAMPLES

Phenol 10 grams/10L

SAMPLE	COD	VOLUME	TOTAL COD
A	2567.5	4150	10,655.2
B	2074.0	2665	5,527.2
C	1521.0	450	684.5
D	1363.0	610	831.4
Residue	711.0	1680	1,194.5
Control	2172.5	300	651.8
Total		9855	19,544.5

(Calculated value 21,725.0)

TABLE VI

TEST RESULTS

DISTILLATION OF CONTROL SAMPLES

Creatine 10 Grams/10L

SAMPLE	COD	VOLUME	TOTAL COD
A	867.0	1300	1153.1
B	200.9	1780	357.6
C	59.1	2040	120.6
D	35.5	2350	83.4
Residue	3388.5	2020	6844.8
Control	685.6	300	205.7
Total		9820	8765.2
		(Calculated value	6856.0)

TABLE VII

TEST RESULTS

DISTILLATION OF CONTROL SAMPLES

Skatol 10 Grams/10L

SAMPLE	COD	VOLUME	TOTAL COD
A	2788.5	2900	8,086.7
B	1681.0	2220	3,731.8
Residue	267.5	3620	968.4
Control*	515.5	300	154.7
Total		9040	12,941.6

(Calculated value 5155.0)*

* Taken after distillation of Sample A

TABLE VIII

TEST RESULTS

DISTILLATION OF CONTROL SAMPLES

Ammonia 10 Grams/10L

SAMPLE	AMMONIA	VOLUME	TOTAL AMMONIA
A	380.0	1260	478.8
B	40.0	1940	77.6
C	7.0	1370	18.9
D	4.0	1850	7.4
Residue	0.0	3040	0.0
Control	380.0	300	114.0
Total		9760	696.7

(Calculated value 3800.0)

TABLE IX

TEST RESULTS

DISTILLATION OF SAMPLES THROUGH ARDOX CATALYST

Acetic Acid 10 Grams/10L

SAMPLE	COD	VOLUME	TOTAL COD
A	168.6	1270	214.1
B	174.2	1710	297.8
C	202.3	1860	376.3
D	528.3	1860	982.6
Residue	2304.0	2890	6,658.6
Control	1124.0	~300	337.2
Total		9890	8,866.6

(Calculated value 11,240.0)

TABLE X

TEST RESULTS

DISTILLATION OF SAMPLES THROUGH ARDOX CATALYST

Formic Acid

SAMPLE	COD	VOLUME	TOTAL COD
A	521.4	1000	521.4
B	134.3	1315	176.6
C	118.5	2390	283.2
D	208.4	1990	414.7
Residue	1113.9	2460	2,740.2
Control	426.6	300	128.0
Total		9455	4,264.1

(Calculated value 4266.0)

TABLE XI

TEST RESULTS

DISTILLATION OF SAMPLES THROUGH ARDOX CATALYST

Phenol

SAMPLE	COD	VOLUME	TOTAL COD
A	1921.0	1080	2,074.7
B	2489.0	2260	5,625.1
C	2088.0	1470	3,069.4
D	1607.5	1360	2,186.2
Residue	1097.5	2780	3,051.1
Control	2332.5	~425	991.3
Total		9375	16,997.8
		(Calculated value	23,325.0)

TABLE XII

TEST RESULTS

DISTILLATION OF SAMPLES THROUGH ARDOX CATALYST

Ammonia 5 Grams/10L

SAMPLE	AMMONIA	VOLUME	TOTAL AMMONIA
A	488.0	1540	751.5
B	14.6	2310	33.7
C	7.9	1480	11.7
D	8.5	1830	15.6
Residue	5.6	2720	15.2
Control	327.0*	0	0
Total		9880	827.7

(Calculated value 3270.0)

* Estimated

TABLE XIII

AMMONIA CONTROL

MATERIAL BALANCE

Sample Description	Initial Volume Liters	Initial Conc. NH_3 G/L	Grams Ammonia	Final Volume	Final Conc.	Grams Ammonia	% Recoverable
Initial Solution	10.000	0.585	5.850				
Aliquot	.045	0.585	0.026				
Net Initial Solution	9.955	0.585	5.824	9.720	0.287	2.790	
Trap #1	0.050	0.000		0.030	42.500	1.275	
#2	0.400	0.000		0.338	2.380	0.804	
#3	0.400	0.000		0.330	0.0017	0.0006	
LN_2 Trap	0.050	0.000		0.140	0.0017	0.0002	
H_2SO_4	-	0.000					
Distillate	-	-		0.185	6.280	1.162	
Total Volume	10.855			10.743			99.0
Total NH_3			5.824			6.032	103.6

TABLE XIV

AMMONIA CATALYTIC OXIDATION

MATERIAL BALANCE

Sample Description	Initial Volume Liters	Initial Conc. NH_3 G/L	Grams Ammonia	Final Volume	Final Conc.	Grams Ammonia	% Recoverable
Initial Solution	10.000	0.617					
Aliquot	0.100	0.617					
Net Initial Solution	9.900	0.617	6.108	8.540	0.051	0.436	
Trap #1	0.050	0.000		0.050	19.850	0.993	
#2	0.400	0.000		0.335	9.000	3.015	
#3	0.400	0.000		0.340	1.350	0.459	
LN_2	0.050	0.000		0.225	0.000	0.000	
H_2SO_4	-	0.000	-				
Distillate	-	-		1.150	0.600	0.690	
Total Volume	10.800			10.640			98.5
Total NH_3			6.108			5.593	91.6

TABLE XV

NASA CATALYTIC OXIDATION TEST DATA

Distillation Test #	Dist. Temp.	Cat. Bed Temp.	Dist. Rate L/H	Volume Collected	A TOC	B TOC	$\frac{A+B}{2}$	Sample TOC	COD	NH_3 MG/L	% TOC Red'n.	ΣV	Vol. Fresh Urine Added	$\overline{\Sigma V}$	ΔX	Remarks
1	150	255	0.179	460	170.0	35.2	102.6	3.0	20.3	0.0	97.1	460	New 4000	230	99.4	
2	157	245	0.0875	350	59.7	34.5	47.1	0.0	12.2	5.0	100.0	810	990	635	47.1	
3	139	240	0.0756	365	24.0	37.0	30.5	4.2	32.4	0.0	86.2	1175	720	993	26.3	
4	145	248	0.0510	270	49.8	21.7	35.8	25.0	24.3	39.6	38.5	1445	725	1310	10.8	
5	119	234	0.163	652	56.5	18.2	37.4	12.8	28.6	14.1	65.8	2097	New 4000	1771	24.6	
6	130	195	0.136	689	40.0	24.2	32.1	12.5	20.6	12.7	61.0	2786	1170	2442	19.6	
7	106	235	0.107	515	25.7	25.7	25.7	5.2	5.2	15.7	79.8	3301	1195	3044	20.5	
8	107	223	0.396	1519	51.7	40.0	45.9	37.5	31.8	685	16.1	4820	New 4000	4061	8.4	
9	90	200	0.100	564	60.5	63.0	61.8	16.8	33.9	760	72.9	5384	2100	5102	45.0	
10	92	140	0.091	455	30.0	15.0	22.5	13.4		1150	40.5	5839	1003	5602	9.1	
11	93	198	0.147	330	34.4	25.0	29.7	14.0	35.7	1772	52.9	6169	886	5954	15.7	
12	151	-	0.0855	185	-	-	-	-	-	6280	-	-	-	-	-	Ammonia Control
13	150	235	0.307	1150	-	-	-	-	-	600	-	-	-	-	-	Ammonia Test Run
14	153	~250	0.144	528	66.0	12.2	39.1	40.3	69.4		0	6697	New 6000	6433	-1.2	
15	150	248	0.144	730	10.8	9.2	10.0	3.5	12.5		65.0	7427	823	7062	6.5	
16	149	239	0.161	705	6.7	6.2	6.5	3.5	18.5		46.0	8132	1110	7780	3.0	
17	140	233	0.206	1010	66.6	17.1	41.9	25.8	78.2		38.4	9142	New 6000	8637	16.1	
18	143	245	0.116	675	15.3	11.0	13.2	8.0	24.5		39.4	9817	1240	9480	5.2	
19	141	243	0.215	1075	6.4	8.3	7.4	4.3	17.2		42.0	10892	New 6000	10355	3.1	Treated Urine
20	150	235	0.137	695	15.8	5.0	10.4	7.1	24.1		31.7	11587	1455	11240	3.3	Treated Urine
21	148	245	0.200	1000	6.1	5.0	5.6	2.5	12.0		55.4	12587	New 6000	12087	3.1	Treated Urine
22	138	240	0.216	1060	11.0	9.3	10.2	3.8	19.3		62.7	13647	1345	13117	6.4	Treated Urine

TABLE XVI

ENERGY REQUIREMENTS

SUMMARY OF RESULTS

Run	Flow (lb/hr.)	Ave. Inlet Temp. (°F)	Ave. Outlet Temp. (°F)	Δt Water (°F)	Jacket Pressure (Microns)	Energy (watt hr/lb)
P1	1.11	113.5	91.0	22.5	500	7.74
P2	.76	161.7	89.6	72.1	500	6.05
P3	3,12	146.5	108.7	37.8	500	4.76

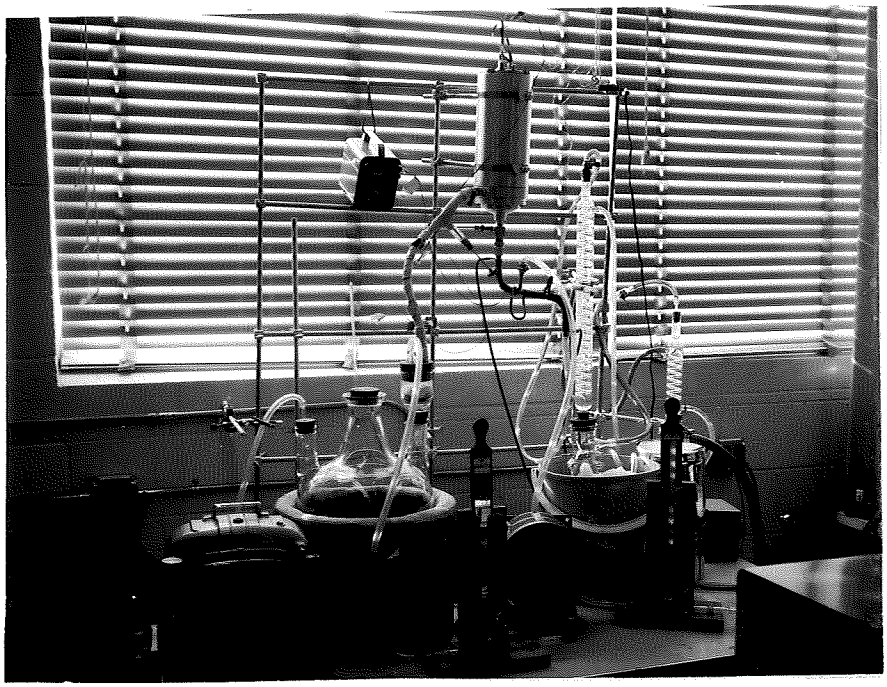


Figure 1

Original Lab Set-Up

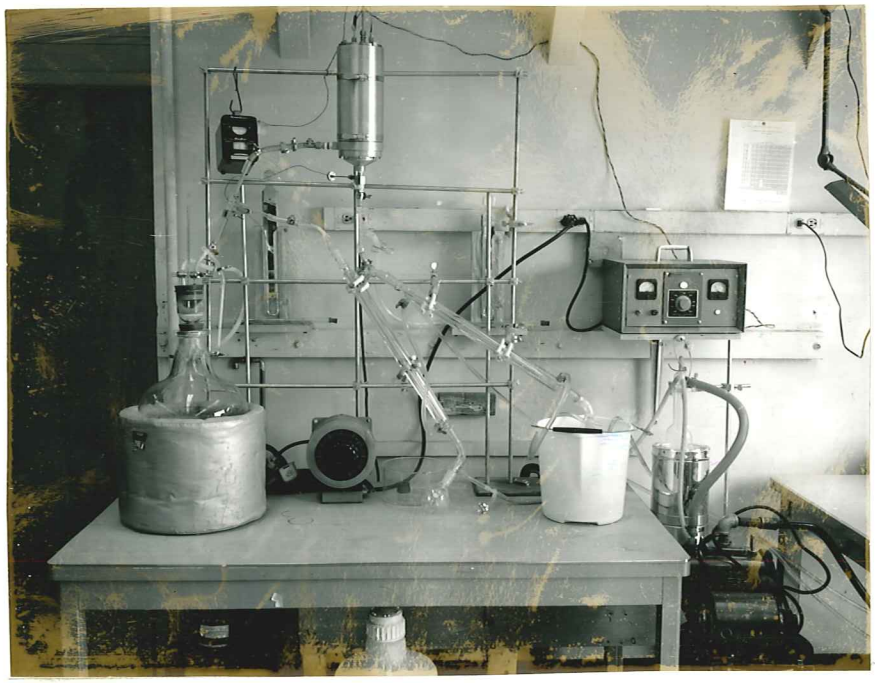


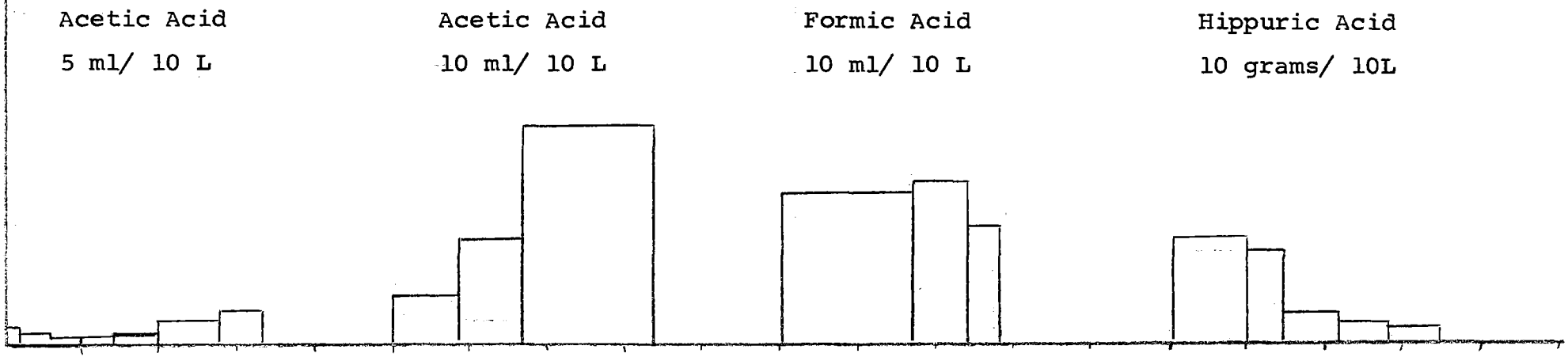
Figure 2

Modified Lab Set-Up

FIGURE 3

TEST RESULTS

DISTILLATION OF CONTROL SAMPLES



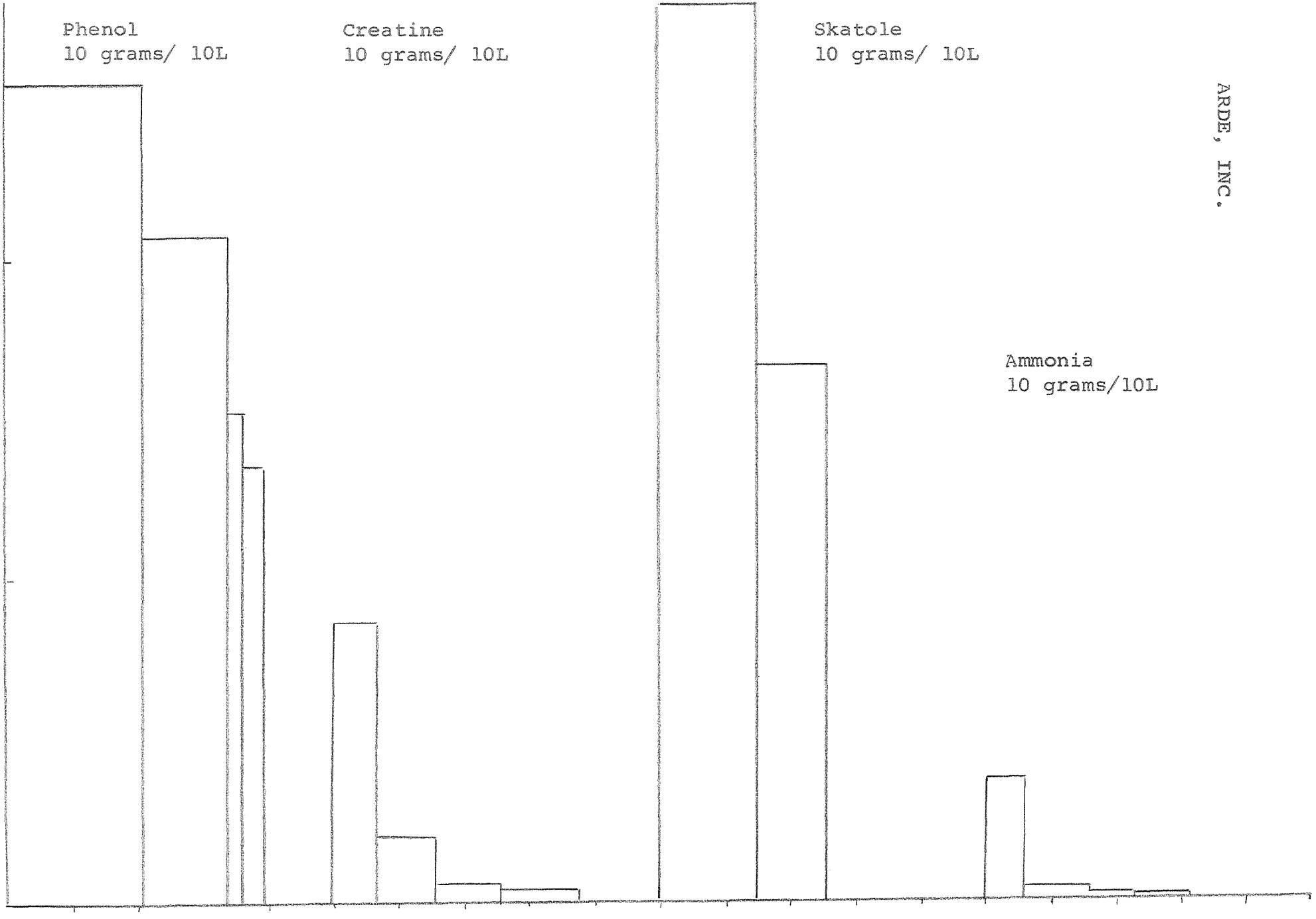


FIGURE 4

TEST RESULTS - DISTILLATION OF CONTROL SAMPLES

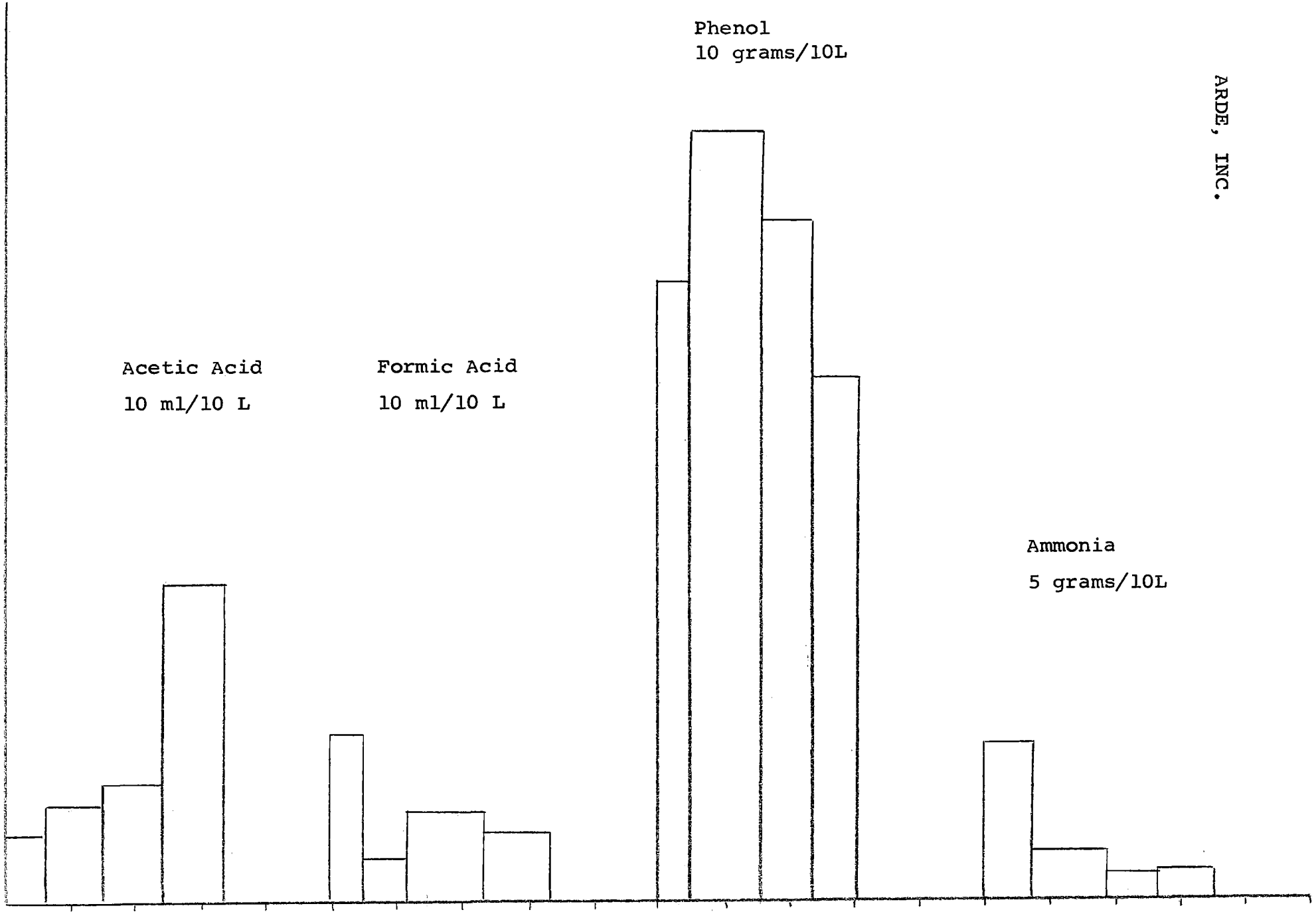


FIGURE 5

TEST RESULTS - DISTILLATION OF SAMPLES THROUGH ARDOX CATALYST

COMPARISON OF CONTROL RUN
WITH CATALYST RUN

ACETIC ACID

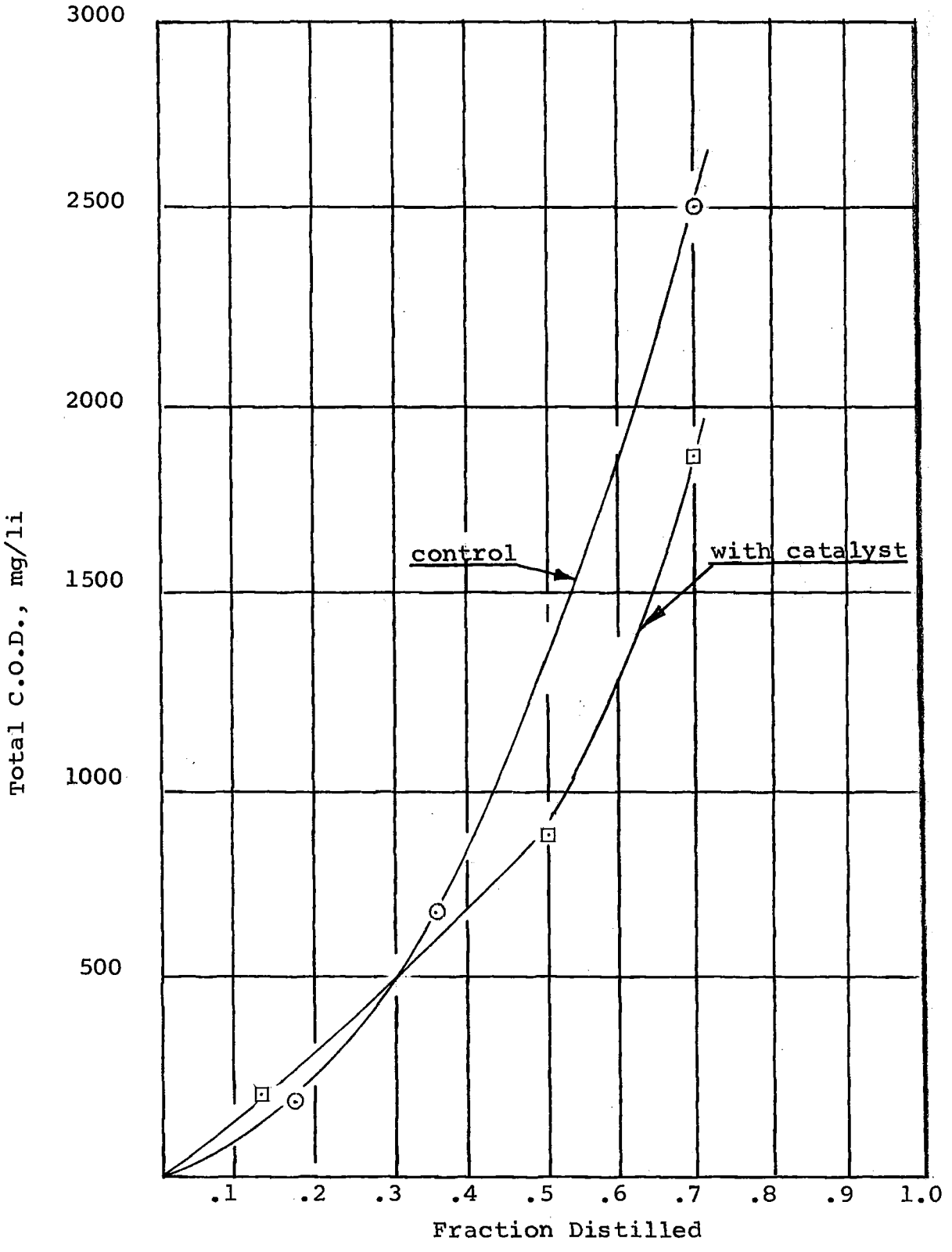
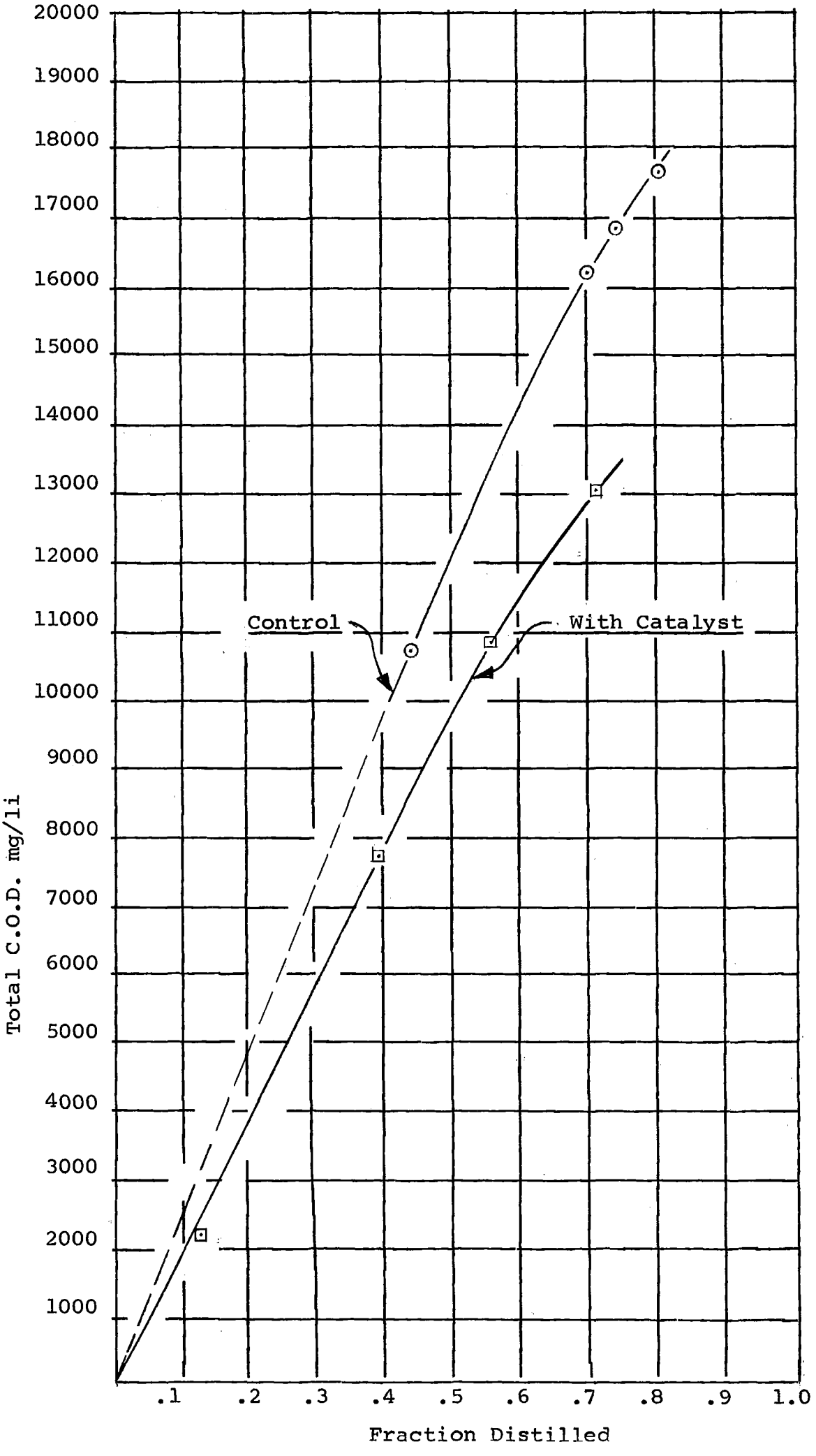


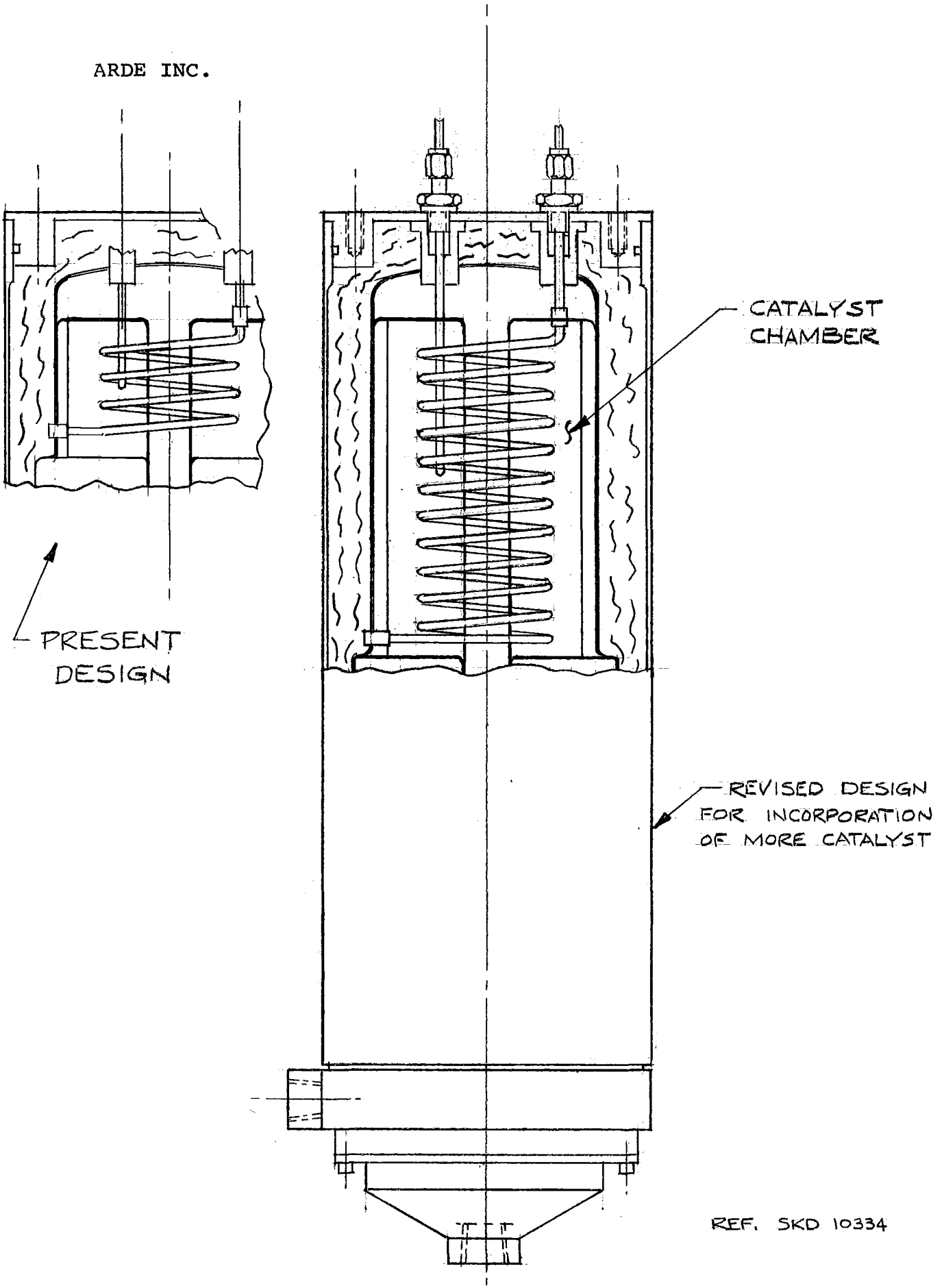
FIGURE 6



Comparison of Control Run with Catalyst Run

Phenol

FIGURE 7



PROPOSED
CATALYST CHAMBER REDESIGN
FIG. 8