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Feasibility of a Modified Chloramine Process for the Production of UDMH and MMH

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The feasibility of a chemical process for the production of unsymmetrical dimethylhydrazine (UDMH) and monomethylhydrazine (MMH) by a gas phase generation of chloramine followed by its subsequent reaction with an amine solution was investigated by Martin Marietta Corporation under the direction of Air Force Space and Missile Systems Organization/The Aerospace Corporation. This report summarizes the study and analyzes the economics of the process. Information on the optimum operating conditions required for scaleup of the unit to a 40 lb/hr pilot plant system was obtained.

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Data obtained from the chloramine reaction and the substituted hydrazine reaction were used to derive statistical equations describing the reactions as functions of operating parameters. These derived equations were then used to develop a mathematical model to estimate the raw materials, recycling, and utility costs per pound of fuel produced. The overall economics of the process was assessed by determining the raw material and utility of a 400 lb/hr production plant and adding other costs such as capital, labor, maintenance, amortization, overhead, and profits. This analysis was used to recommend regions of process operating conditions for further investigation in a future pilot plant program.

PREFACE

The feasibility of a chemical process for the production of unsymmetrical dimethylhydrazine (UDMH) and monomethylhydrazine (MMH) by a gas phase generation of chloramine followed by its subsequent reaction with an amine solution was investigated by Martin Marietta Corporation under the direction of Air Force Space and Missile Systems Organization (SAMSO)/The Aerospace Corporation. This report summarizes the study and analyzes the economics of the process. Information on the optimum operating conditions required for scaleup of the unit to a 40 lb/hr pilot plant system was obtained.

Data obtained from the chloramine reaction and the substituted hydrazine reaction were used to derive statistical equations describing the reactions as functions of operating parameters. These derived equations were then used to develop a mathematical model to estimate the raw materials, recycling, and utility costs per pound of fuel produced. The overall economics of the process was assessed by determining the raw material and utility of a 400 lb/hr production plant and adding other costs such as capital, labor, maintenance, amortization, overhead, and profits. This analysis was used to recommend regions of process operating conditions for further investigation in a future pilot plant program.

The authors wish to express appreciation to Martin Marietta Corporation personnel, especially Dr. J. M. Murphy and Mr. T. Shupert for conducting the study and cooperating in providing the experimental data. Appreciation is also extended to Mr. J. P. Leary who provided the statistical analyses of the data, and to Dr. C. C. Badcock who developed the chloramine gas mixtures analysis method and provided valuable suggestions on the operation of the chloramine gas generator.

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I. INTRODUCTION

A. OBJECTIVES AND SCOPE

A critical potential shortage of unsymmetrical dimethylhydrazine (UDMH), a rocket fuel, arose in 1973 when the sole source manufacter in the United States terminated production of this propellant. The unavailability of this fuel, which is a major propellant ingredient in the Titan and Agena rocket boosters, would seriously cripple the U.S. space programs. The Air Force requested the development of an alternative amine fuel production process for the longrange resolution of the UDMH shortage and related problems. The specific objective was to develop an economical, non-hazardous, and bio-environmentally sound process capable of producing both UDMH and MMH (monomethylhydrazine).

SAMSO contracted with Martin Marietta Corp (MMC) to carry on a program that had been initiated at Naval Ordnance Station by Air Force Logistics Command (AFLC). This program was to determine the feasibility and economics of producing the two hydrazine fuels, UDMH and MMH, by adapting a process originally investigated on a laboratory scale by Dr. H. Sisler of University of Florida. In this process, the amine fuel is produced in a two-step reaction. The first step involves a gas phase reaction of chlorine with excess NH_3 to form chloramine (CA) gas and solid ammonium chloride. The resultant CA is reacted with a methyl substitute amine to yield the corresponding hydrazine fuel plus hydrogen chloride. The reactions are as follows:

$$2\,NH_3 + Cl_2 \longrightarrow NH_2Cl + NH_4Cl$$

$$NH_2Cl + CH_3\text{–}NHR \longrightarrow CH_3\text{–}NR\text{–}NH_2 + HCl$$

where R is H or CH_3 group.

It was hypothesized that a relatively concentrated product could be produced by these reactions, resulting in a low cost.

This report summarizes the result of the MMC study conducted from March 1975 through February 1978. Although the program covered approximately a 3-year span, the study was performed intermittently in five phases necessitated by uncertainties in available funding, and shifting priorities. This resulted in some inefficiency and a greater expenditure of funds than otherwise would have been required. Detailed description of the accomplishments for Phases I through IV can be found in the MMC reports.[1-4] Since under contractual agreement, MMC was not required to report on Phase V, a more detailed description of that phase is provided in this report.

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[1] Amine Fuels Production Feasibility Demonstration, MCR-75-466, Final Report and Addendum I - Data Book, Martin Marietta Corporation, Denver (November 1975) Contract F04701-74-C-0039.

[2] Amine Fuels Production Feasibility Demonstration, MCR-75-466, Addendum II to Final Report, Martin Marietta Corporation, Denver (February 1976) Contract F04701-74-C-0039.

[3] Amine Fuels Production Feasibility Demonstration, MCF-75-466, CDRL A305, Volume I, Final Report; Volume II Economic Analyses; and Volume III, Data Book; Martin Marietta Corporation, Denver (October 1976) Contracts F04701-74-C-0039 and F04701-76-C-0181.

[4] Amine Fuels Production Scalability Demonstration, MCR-78-33, Volume I, Final Report; Volume II, Data Book; and Volume III, Pilot System, Martin Marietta Corporation (February 1978).

B. HISTORY

From the early 1960s to the early 1970s, UDMH was produced by FMC, the sole manufacturer in this country, by the nitrosamine process. With the decreasing consumption of this fuel in the space program and the categorization of N-nitrosodimethylamine as a strong carcinogen, FMC made a corporate decision to terminate production of UDMH. The Air Force requested the supplier to produce an additional 4 million lb of this fuel, representing a 4-year stockpile, during which time an alternative production source could be developed. Under a decree from the Assistant Secretary of Air Force for Logistics, F.F. Shrontz, Air Force Systems Command (AFSC) directed SAMSO to assist AFLC for the resolution of this problem.

In conjunction with AFLC, SAMSO initiated development of two alternative processes to synthesize UDMH and MMH. One process study (based on chloramine chemistry) was awarded to MMC in March 1975; the second study (based on urea chemistry) was awarded to IIT Research Institute (IITRI) in April 1976. The objectives of these studies were to (a) demonstrate feasibility of amine fuel production, and (b) obtain sufficient data for starting a pilot plant program. At the completion of these studies, one process was to be selected for further investigation in a pilot plant system capable of producing 40 lb/hr of fuel.

In September 1976, representatives of SAMSO, AFLC, and Air Force Rocket Propulsion Laboratories (AFRPL) evaluated the results of the MMC chloramine and IITRI urea processes. It was concluded that both were feasible, but capital costs for the urea process would be considerably greater. The chloramine process was selected for further development primarily on an economic basis. Because MMC had not yet completed all tasks necessary to proceed to the pilot phase, additional funding for pre-pilot investigations was awarded in the Phase IV study to resolve outstanding problems. During Phase IV, SAMSO requested an add-on Phase V study, which entailed a major modification of the first-stage reactor to determine the efficiency of the first step, the NH_3 - Cl_2 reaction. The program was concluded in February 1978.

After the completion of the urea process study, SAMSO awarded a contract to IITRI to investigate the product purification and waste product disposal of the crude amine fuel mixtures resulting from the chloramine process. This study was initiated to facilitate the ongoing work at MMC by taking advantage of the expertise developed at IITRI on hydrazine chemistry. The work is still in progress and will be the subject of another report.

In November 1977, representatives from Headquarters USAF, AFSC, SAMSO, AFLC, and AFRPL convened to review the overall status of the hydrazine fuel availability. This working group reassessed the production resources in relation to stockpile and requirements. Also considered were manufacturing bioenvironmental concerns and the potential categorization of all hydrazine fuels as suspected carcinogens. At this meeting SAMSO recommended that, since further development of a new process plant was no longer required, the MMC chloramine process be completed only through the prepilot stage. This recommendation, with concurrence from the working group, led to the decision to conclude the MMC program with the completion of the Phase V study in February 1978.

II. PROCESS DEVELOPMENT

The Martin Marietta Corporation chloramine process study can be conveniently divided into two discrete sections, one involving the generation of CA in the gas phase and removal of the NH_4Cl byproduct, and the second involving the reaction of the resultant CA with amine to yield the desired product. The early phases of the work were conducted on a laboratory scale to establish design criteria and to determine the specific approaches from among several alternatives for construction of a continuous amine fuels production system. Several subtasks were also carried out to obtain information for development of the final system.

Data accumulated during Phases I through III on the laboratory scale apparatus were used to design, construct, and operate a bench scale unit (2 lb/hr) utilized during the latter part of Phase III. This unit was further modified for the experimental runs conducted during Phase IV. The test results from Phases IV and V served as the basis for the determination of optimum operating conditions and the final economic evaluation of the merits of the MMC chloramine process.

A. CA GENERATOR/NH_4Cl REMOVAL SYSTEM

The first CA generator, constructed as an all-glass unit, was sized to produce approximately 0.4 lb/hr of fuel. This apparatus appeared to produce relatively high yields of CA but was severely limited in long-term operation due to NH_4Cl plugging the Cl_2 injector tip. After this problem was circumvented by preheating the NH_3 and Cl_2, the operation was further plagued by clogging of the downstream transfer line by NH_4Cl buildup. Despite the condensation of NH_4Cl, sufficient quantity of this material still remained in the gas stream to adversely affect the second stage reaction. Several different methods, from glass wool to filter bags, were used in an attempt to remove fine NH_4Cl particles from the gas mixture, but relatively little success was achieved. Despite its limitation, this apparatus was used to obtain preliminary data on the CA-amine reaction and on the type of second-stage reactor that would be suitable for continuous operation.

The successful separation of NH_4Cl from the gas stream was achieved during the Phase III study. The CA generator used in the 2 lb/hr bench scale unit was mounted atop a cylindrical NH_4Cl chamber (thermal precipitator) 24 in. in diameter and 80 in. in height (Figure 1). The relatively large volume of the thermal precipitator (TP) serves to slow the gas stream velocity, allowing the solids to agglomerate and settle over a long hold up time. Separation of the bulk of the solid particles from the product mixture was further facilitated by forcing the flowing gas stream to make a 180 deg turn after exiting the thermal precipitator (TP) section. Finally, the residual NH_4Cl was eliminated by the application of an electrical field across the gas stream in an electrostatic precipitator (EP).

Other modifications made during Phase III on the first stage reactor included the following:

a. Conversion of construction material of the TP lid to a CA-compatible Inconel metal because the mild steel lid corroded and decomposed the product.

b. Installation of several vibrators on the TP wall to dislodge the deposited NH_4Cl that formed an insulating layer on the heat exchanger wall.

c. Installation of thermocouples at several locations to monitor the gas temperature. (Protrusion of thermocouples greater than several inches from the TP wall resulted in a buildup of NH_4Cl on the thermocouple with resultant false gas temperature readings.)

A major modification of the first stage reactor made during Phase IV was the installation of an attachment (hopper) at the bottom of the TP for mechanical removal of accumulated NH_4Cl (Figure 1). Periodic removal of the byproduct without test interruption was made possible by this device,

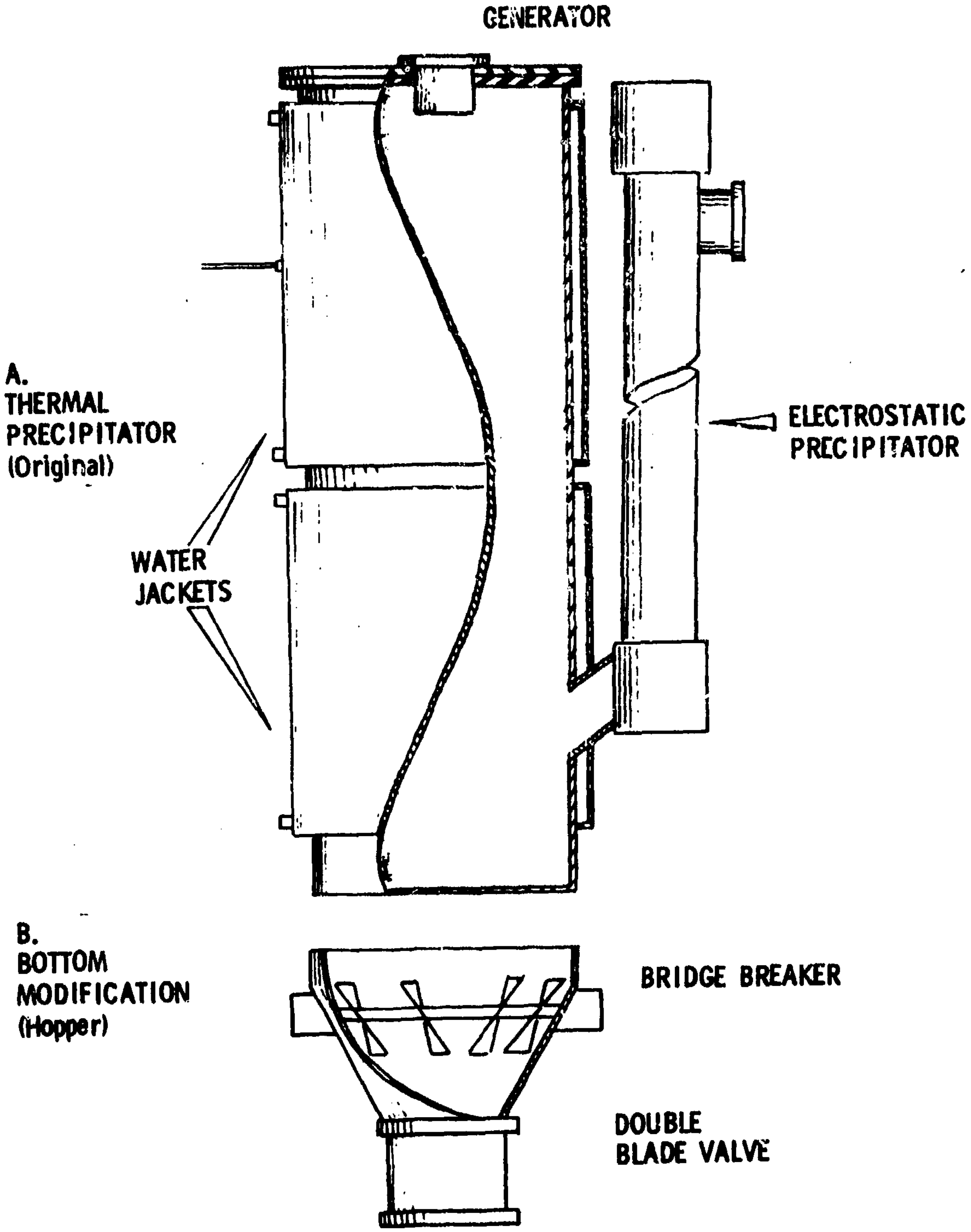

Figure 1. Generator/Precipitator Assembly

permitting long run time. This attachment consisted of a gas-tight, double-slide valve arrangement which isolated the NH_4Cl in a compartment from which the solids could be removed. However, the NH_4Cl formed from the NH_3 - Cl_2 reaction tended to agglomerate and form a bridge across the walls without falling into the bottom compartment. The problem was solved by installing a revolving blade system above the upper slide valve to break up the agglomerated solids. This mechanical system for the physical removal of NH_4Cl functioned satisfactorily and was used during Phase IV and V runs. After the conclusion of the study, some deterioration was observed of the Teflon seal on the revolving blade shaft and of the Teflon coating on the slide valves.

Other changes made on the reactor during Phase IV included installation of two TP water jackets for controlling the upper and lower wall temperatures, additional thermocouples and vibrators, and an improved electrostatic precipitator. A temperature conditioning system was also constructed for maintaining a constant temperature of the feed gases and liquids to improve the accuracy of the flow measurements.

B. CA-AMINE REACTOR

Preliminary studies of the fuel yield from the second-stage reaction between CA and amine were carried out using the 0.4 lb/hr laboratory scale CA generator. Several different contactor designs were initially investigated, including a wetted wall, a packed column, and a stirred-bubbler system. The first two approaches yielded little or no product, but the last design successfully produced reasonable yields of UDMH. In these stirred-bubbler tests, CA gas was introduced into a set quantity of the amine in a vessel. Based on these results, a stirred-bubbler design modified for continuous operation was selected during Phase II for further investigation.

Several options still remained in the choice of the reaction media for the second stage reactor. Among those investigated early in the program with varied results included anhydrous amine; amine/NaOH/H_2O; and various combinations of liquid NH_3, ethanol, NaOH, CaO, and water. Only the first two systems among those tried gave fair yields of the desired fuel without complications. However, the reaction of CA with amine in the absence of caustic and water had to be conducted at -70° C. Kinetic studies of second step reactions conducted as a subtask indicated that at higher temperatures (~ 0° C), CA reacted preferentially with the desired hydrazine product rather than with the amine. Although the CA-amine reaction at -70° C gave high yields, it was relatively slow, requiring hours for completion, and had the further disadvantage of being a costly production process. The most attractive reaction media appeared to be the amine/NaOH/H_2O system; therefore, it was adopted for final development study.

The second stage reactor (Figure 2) used during Phase III to the end of the program was designed for continuous operation. It consisted of a 12 in. by 8.25 in. ID stainless steel vessel equipped with a vent system, a CA gas inlet orifice at the bottom, and a second inlet for the amine/NaOH/H_2O feed line on the side wall close to the bottom. A 9-in. standpipe was used to remove the reaction mixture and to maintain a constant liquid level during a run. Another feature of this contactor was a coil installed inside the vessel for circulating fluid to control the temperature of the reaction mixture.

An important consideration of the second stage reactor was the stirrer design. This stirrer must not only homogenize the liquid and eliminate stagnation areas but also disperse incoming gases into tiny bubbles that will rapidly dissolve in the liquid media. A separate study was conducted using simulated gas and fluid to investigate the effects of different blade configurations, stirrer locations on the shaft, and rpm values. Details of this study are described in the MMC Phase III report.[3] From the results of this study, an arrangement consisting of two 3-in. diameter stirrers attached to a single shaft 2.5 and 6.5-in. from the botton was selected. The lower stirrer dispersed the gas; the upper stirrer homogenized the fluid.

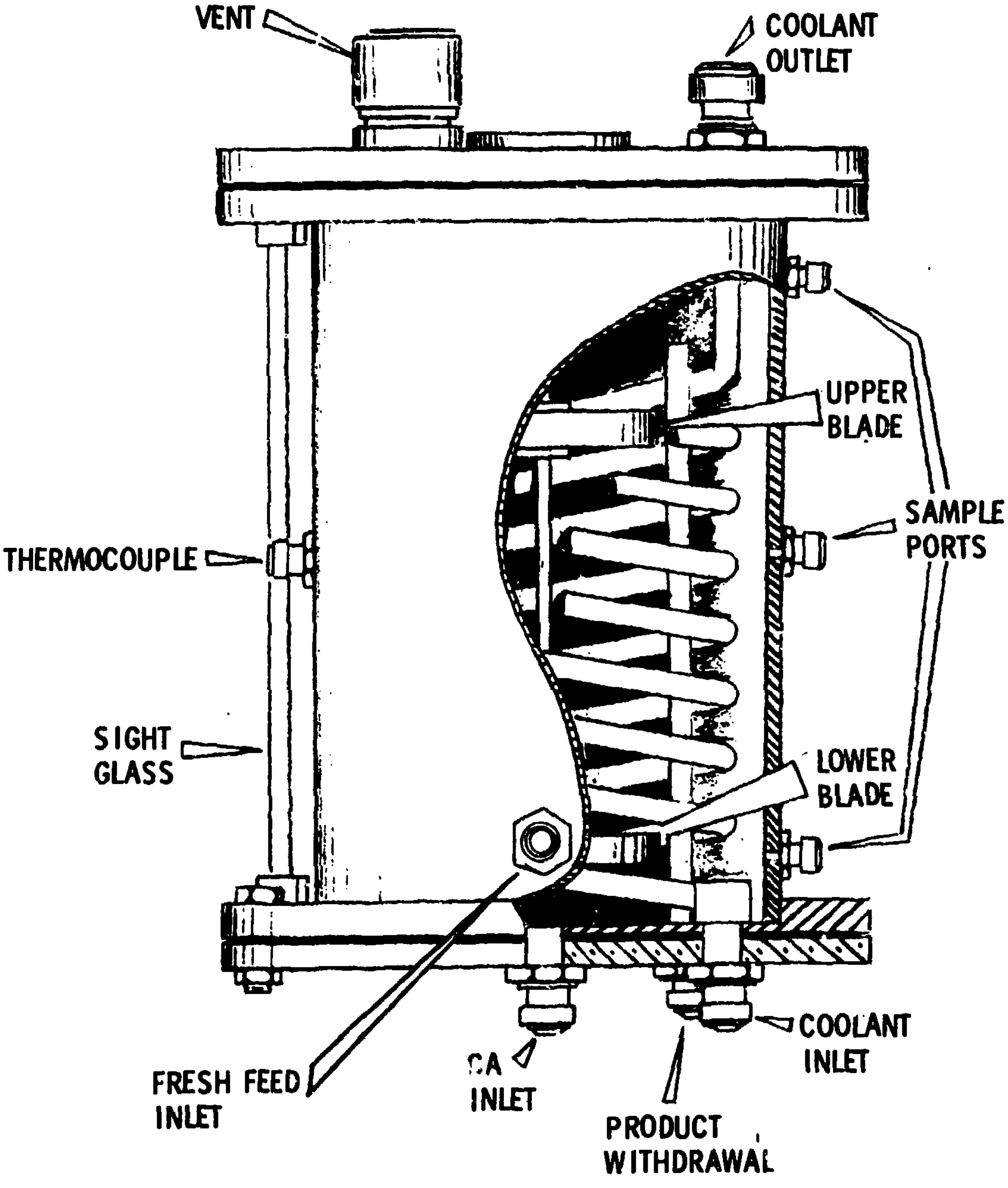

Figure 2. CA-Amine Reactor

This contactor design operated satisfactorily during the study, but difficulties were encountered under certain reaction conditions. At high amine concentrations, especially with simultaneous high caustic concentrations, a large quantity of the amine was lost by vaporization into the vent system. This effect restricted the operation of the contactor reaction conditions and prevented the attainment of experimental data desired for the statistical evaluation of the CA-amine reaction; however, it was probably representative of the real limit of operating conditions. The volatilization of amine was especially acute in the region where the amine/NaOH/H_2O system separated into two phases consisting of an amine-rich upper and a caustic-rich lower aqueous phase.

To identify this two-phase region, a phase diagram study of the three-component systems consisting of DMA/NaOH/H_2O and of MMA/NaOH/H_2O was conducted. The results are shown in Figures 3 and 4. This study is described in greater detail in the MMC Phase IV report.[4] Because the reactant concentrations are expressed as moles per unit volume throughout this report, the three-component phase diagrams were translated into moles per liter values for both caustic and the amine. The phase boundary is shown as a solid line for the DMA/NaOH/H_2O and MMA/NaOH/H_2O systems in Figures 5 and 6, respectively. Since some of the amine and caustic are consumed in the contractor by the incoming CA, the resultant product mixture will be homogeneous if the incoming mixture is not too far into the two-phase region. Although dependent to some extent on the CA concentration, the dashed line in these figures represents the region below which single-phase product mixture could be obtained. For the majority of the Phase IV test runs and all Phase V runs, contactor conditions were selected where the resultant product mixtures were expected to be single phase solutions.

The high volatility of the amines caused other operational difficulties. The major one was the quantitative determination of the effluent product mixture volume which was required for calculations of yields and the amount

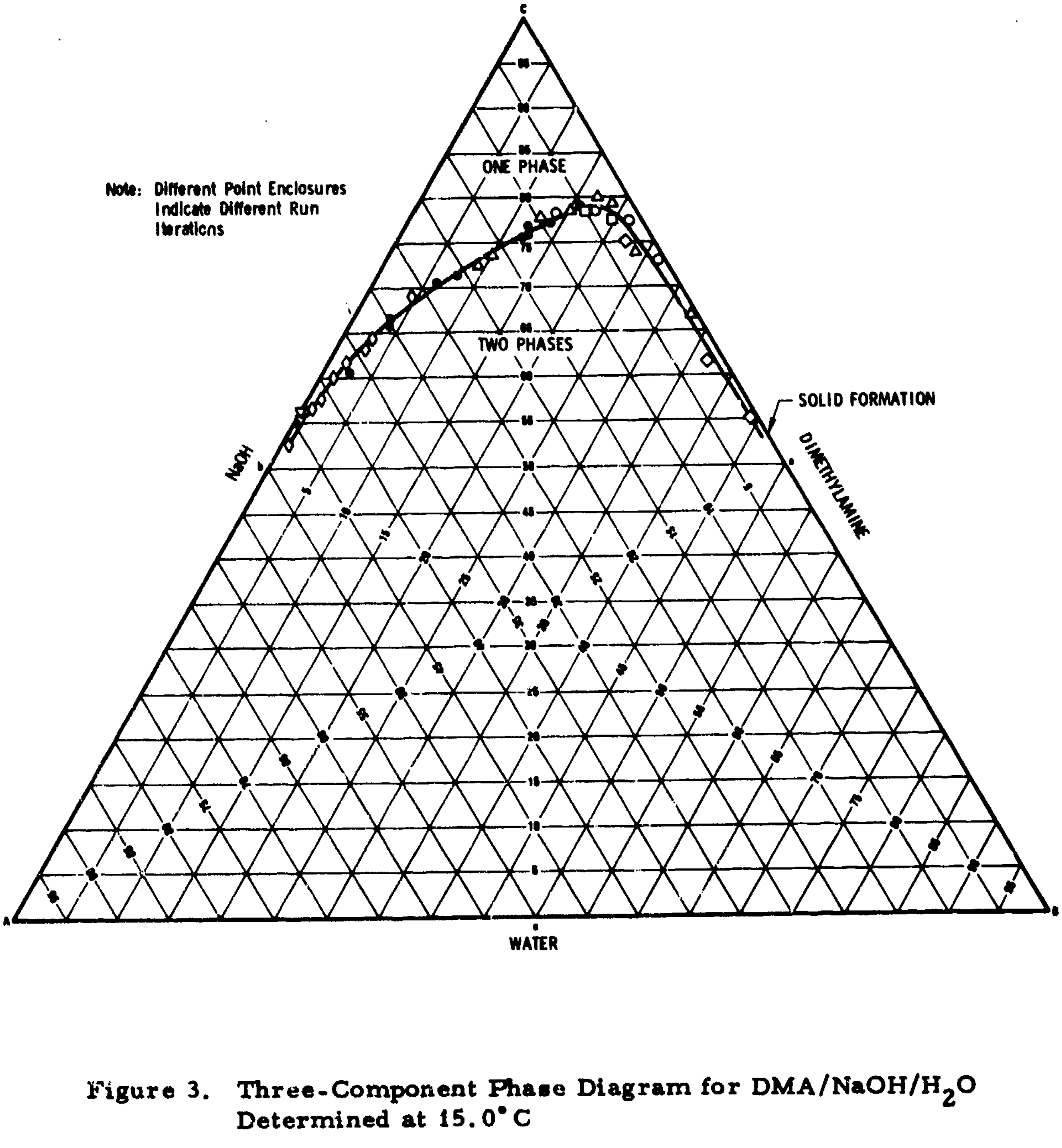

Figure 3. Three-Component Phase Diagram for DMA/NaOH/H_2O Determined at 15.0°C

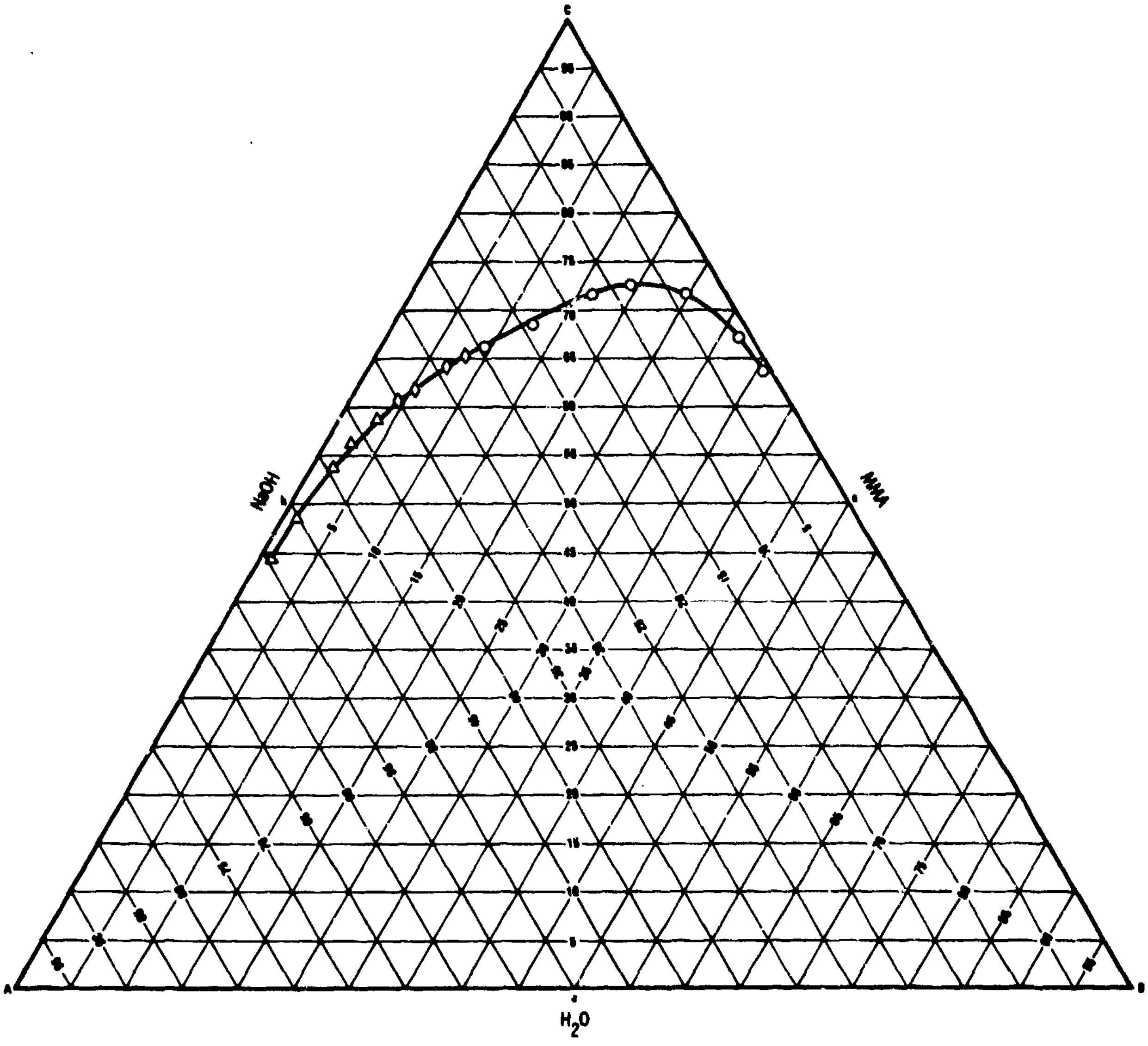

Figure 4. Three-Component Phase Diagram for MMA/NaOH/H_2O Determined at 15°C

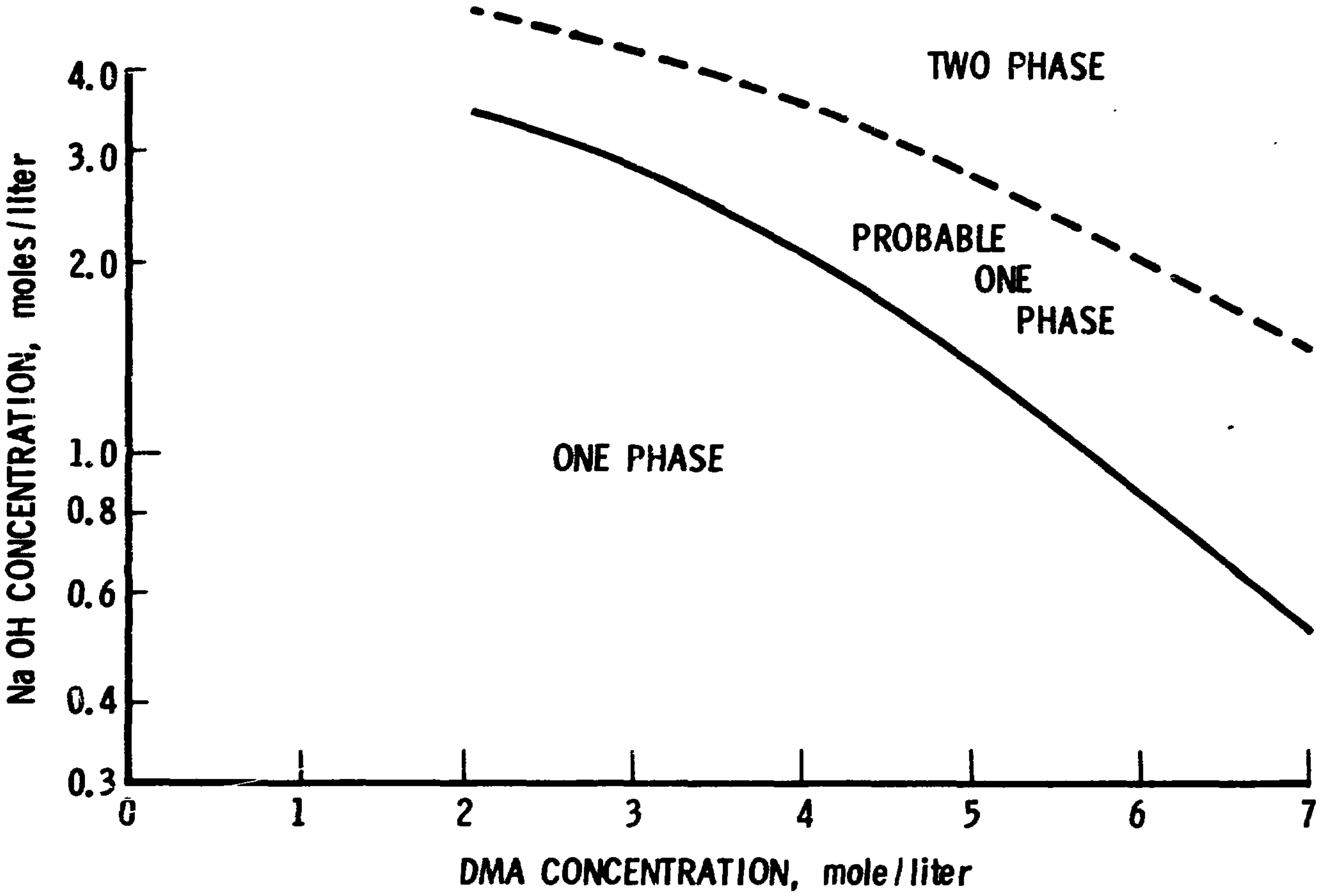

Figure 5. Contactor Product Mixture Homogeneity - UDMH System

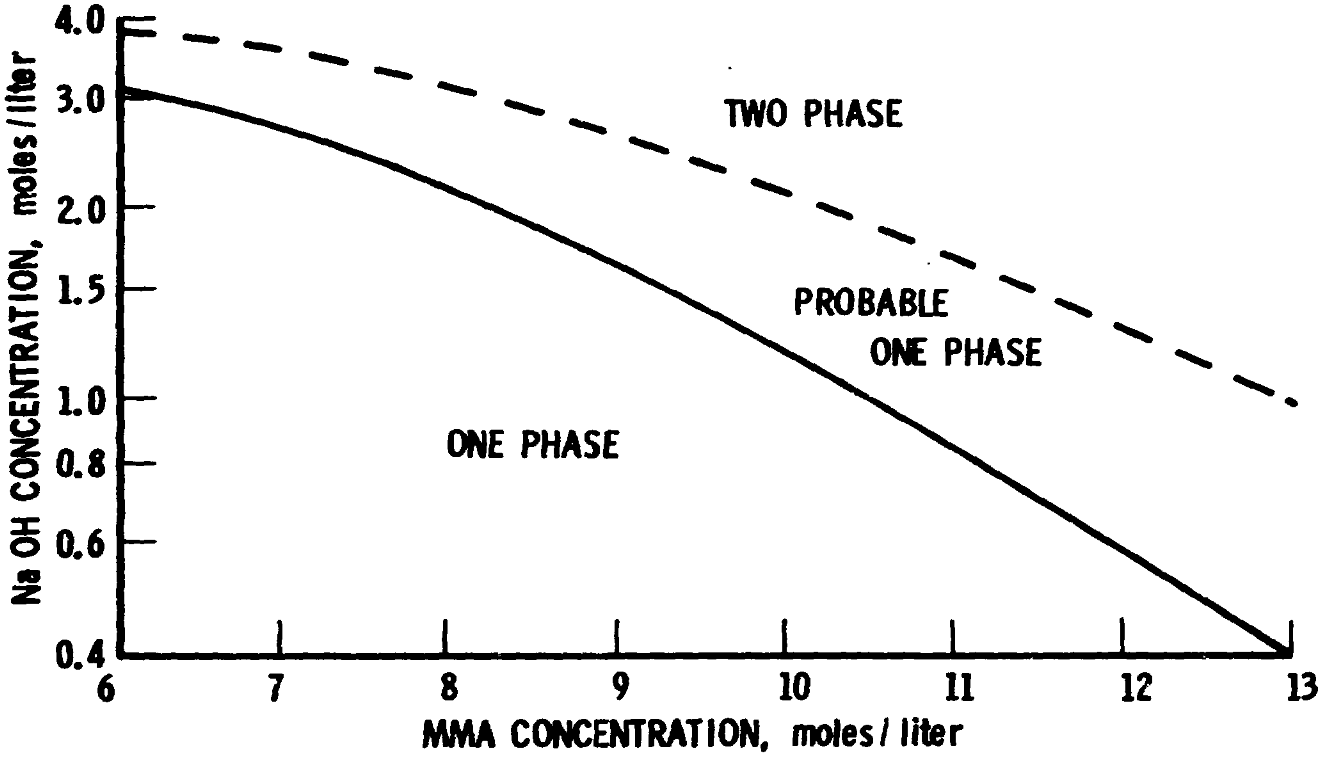

Figure 6. Contactor Product Mixture Homogeneity - MMH System

of product formed. The installation during Phase IV of a small gas separator vessel in the contactor effluent line followed by a mass flow meter did not resolve the problem.

The final test series (Phases IV and V) of the second stage reactor system provided data suitable for design and operation of a pilot plant.

C. CHEMICAL ANALYSIS

Evaluation of a chemical process requires development of reliable and reproducible analytical procedures. Hydrazines and associated product analyses represent a unique facet of analytical chemistry not readily available even in laboratories normally performing chemical analysis. Therefore, considerable time and effort were expended in developing suitable techniques applicable to the MMC chloramine process.

1. CA ANALYSIS

In Phase I, the effluent gas mixture from the CA generator containing CA was withdrawn through a gas sampling port, dissolved in toluene, and analyzed by titration with potassium iodide. Considerable decompostion of the product in the sampling line was observed and this approach was abandoned.

Another approach involved an on-line spectroscopic measurement of the gas mixture where a portion of the effluent stream was diverted through a spectrometer. The presence of solid NH_4Cl in the mixture, either originally present or deposited by CA decomposition, coated the windows sufficiently so that meaningful readings could not be obtained. Intermittent attempts to remove the coatings by heating under vaccum were not successful in providing accurate CA concentrations in the gas stream.

A third gas analysis procedure utilized was a method based on NH_3 to Cl_2 molar ratio developed by The Aerospace Corporation Laboratory Operations. This method is described in Appendix A. Although this method worked well at Aerospace on a small glass CA generator, its use on the

2 lb/hr unit at MMC yielded widely fluctuating, inconsistent values and was eventually abandoned.

The CA analysis used was based on titrimetric determination of the chloride concentration in the contactor. This method takes advantage of the fact that essentially all of the NH_4Cl is removed from the gas mixture upstream of the second-stage reactor by the combination of thermal and electrostatic precipitators. The chloride concentration, together with contactor product mixture volume per unit time, allowed the calculation of CA yield based on Cl_2 feed rate. This method requires very exact flow determinations.

2. AMINE FUEL ANALYSIS

The hydrazine product in the contactor was analyzed by direct injection of the product mixture into a gas chromatograph. One difficulty encountered using this method was the rapid deterioration of the chromatography column packing due to the accumulation of solids NaOH and NaCl found in the reaction mixture, especially during long test runs. This problem was alleviated by modification of the sample injection port to prevent the major portion of the solids from reaching the packed column section. The gas chromatograph was calibrated immediately prior to each run, using standards of the hydrazine dissolved in amine-water solution.

A major improvement in fuel analysis was obtained during Phase IV by the incorporation of an internal standard, pyridine, in the analysis solution. Even with these precautions and modifications, the results of the regression analysis of the experimental data showed a consistent unidirectional discrepancy in the empirical fit of data obtained from several Phase IV test runs. This observation is suggestive of an error in calibration for those runs.

D. ASSOCIATED STUDIES

During the MMC program, several problem areas arose which necessitated initiation of associated studies. These investigations provided pertinent information required for the development of the final system or for facilitating the attainment of process conditions suitable for continuous operation:

a. Kinetics of CA-amine and CA-fuel reactions
b. CA stability in solution
c. Efficiency of the contactor stirrer
d. DMA/NaOH/H_2O and MMA/NaOH/H_2O phase diagrams
e. Product purification by extraction
f. Side product analysis

The results of these studies can be found in the MMC reports.[1-4]

E. PHASE V STUDY

The Phase V study is discussed in greater detail here because MMC was not required to report on this phase.

The original design of the 2 lb/hr first-stage reactor used in Phase IV consisted of a generator section with poor temperature control and a thermal precipitator section that was considerably oversized. Large, rapid fluctuations in the gas temperature near the injector outlet were frequently observed, indicating an unstable NH_3/Cl_2 reaction. The system was further complicated by the large precipitator volume masking the CA concentration changes occurring in the gas phase. The effects of variations in generator operating conditions on CA yield could not be accurately assessed because of these difficulties and had to be deferred. Phase V study was conducted to resolve these problems.

The primary objective of this last phase of the study was to determine the first reaction yield versus input variables in the modified CA generator/TP system. The secondary objectives were to collect crude MMH product mixture for IITRI product purification studies and to obtain additional second reaction data for the statistical analysis.

The new, modified CA generator/TP system, referred to as the "Top Hat" design, had a conical configuration with the injector outlet situated at the vertex (Figures 7 and 8). The cone section, constructed of CA-compatible Inconel metal, was equipped with a thermocouple and heaters to maintain the temperature at the desired level. The gaseous reaction between NH_3 and Cl_2 to yield CA takes place in this section of the unit. The byproduct of this reaction, NH_4Cl, condenses as a solid at lower temperatures ($<650°F$). Heaters imbedded in the cone maintain a temperature high enough to prevent formation of NH_4Cl in the reaction zone, yet not high enough to decompose the product. A smaller cooling section was attached to the generator and inserted inside the original TP as indicated in Figure 7. The reduced volume of the NH_4Cl settling chamber for this new system was intended to improve response.

The performance of the modified CA generator/TP system was investigated by using the following experimental test matrix:

Parameter	Matrix Test Points				
	-K	-1	O	+1	+K
NH_3Cl_2 molar ratio	4	6	8	10	12
N_2/Cl_2 molar ratio	0	1	2	3	4
Cone Temp., °F	440	500	560	620	680

Three test runs were conducted to obtain experimental data for the matrix points. Other parameters that were held constant during these runs were NH_3 preheat, 450°F; Cl_2 preheat, 200°F; generator, 750°F; and TP water panels, 140°F at 3 GPM. The vibrators on the generator/TP system were operated continuously, whereas those on the electrostatic precipitator were actuated at 2-min intervals. Ammonium chloride accumulating during the test was removed after every 30 min of run time.

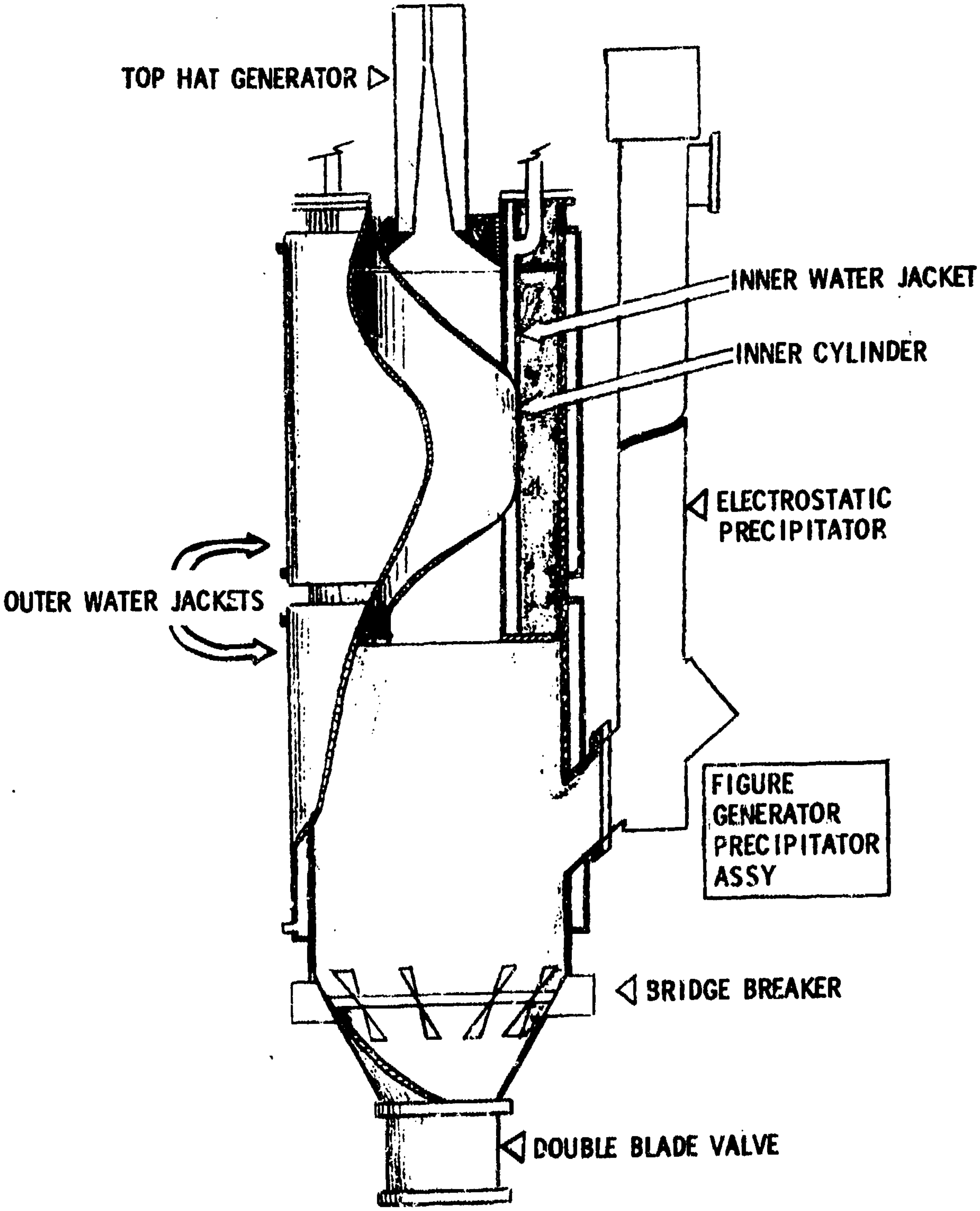

Figure 7. Top Hat Generator/Thermal Precipitator Assembly

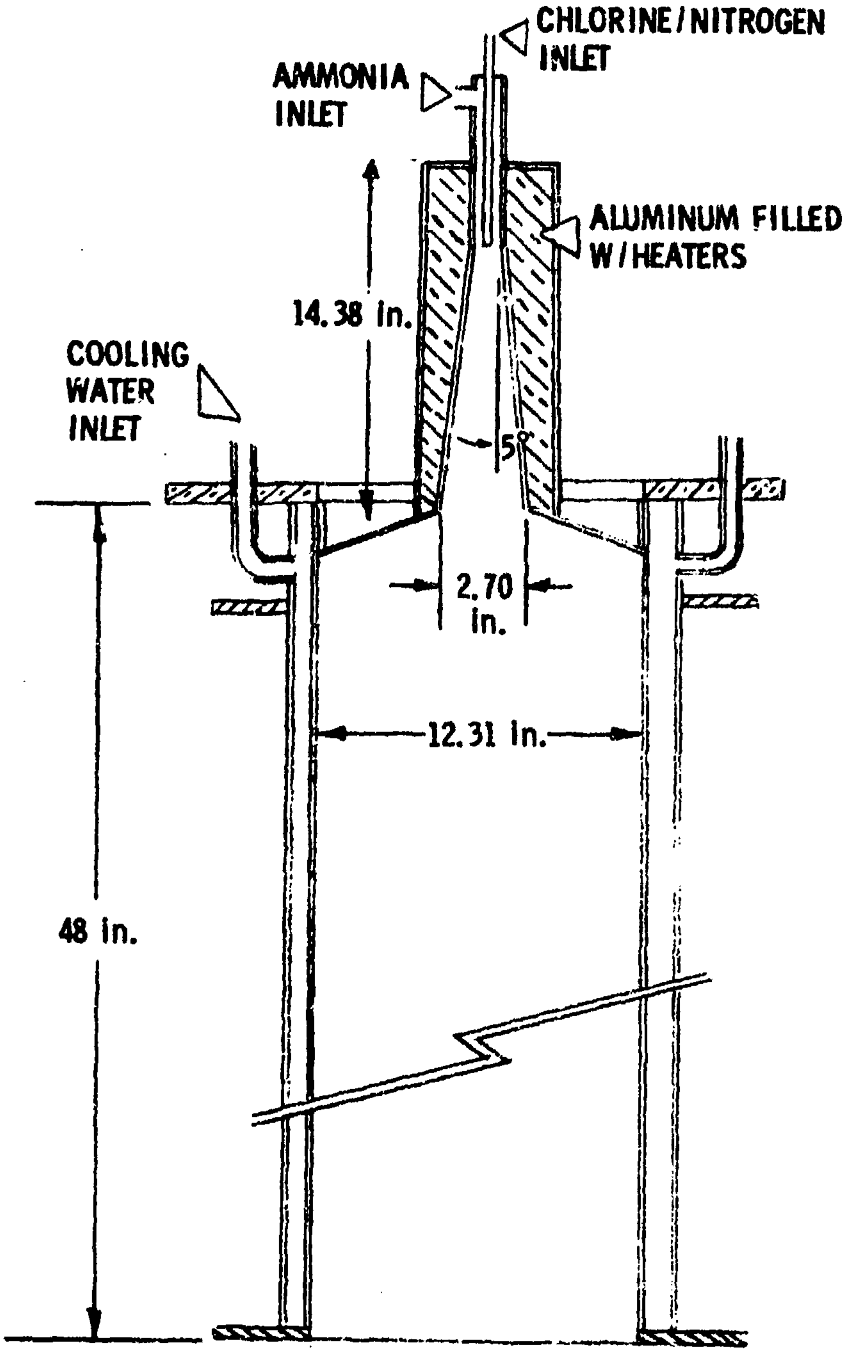

Figure 8. Top Hat Generator Assembly

For Run 1 conducted with the MMH system, the target reactant concentrations for the second stage reactor were MMA (X_1) = 9.0, NaOH (X_2) = 1.1, and CA (X_3) = 0.70 mole/liter X_3 was calculated assuming a 70 percent efficiency of the CA generator; however, these concentrations varied with generator performance and with changes in product mixture volume resulting from the use of different NH_3/ N_2/Cl_2 ratios.

Both Runs 2 and 3 were UDMH tests where DMA was used as the amine. Contactor conditions were selected primarily to obtain data at caustic concentrations missing from Phase IV study, which were required for a meaningful statistical evaluation of the second-stage reaction. The target concentrations for the reactants in these tests were as follows:

Parameter	Concentration, m/l	
	Run 2	Run 3
DMA (X_1)	2.9	3.2
NaOH (X_2)	2.3	1.9
CA (X_3)	0.60	1.1

In Run 3, an inadvertent error in the set point of the DMA flowmeter resulted in low X_1 values for most of the test.

Determinations of the CA yields during the first test (Run 1) were attempted by two methods: (1) sampling the gas mixture at the exit line of the electrostatic precipitator, which would have given real time data, and (2) analysis of the chloride concentrations in the contactor, which gave data after the fact. The former gave inconsistent values and was abandoned for subsequent runs.

CA yield determination by chloride ion analysis in the contactor requires an accurate knowledge of the product mixture volume. This volume can vary even while maintaining constant input flows of the amine, aqueous caustic, and water to the contactor since the amount of NH_3 remaining in the mixture is a variable based on the CA generator operating conditions. Nevertheless, the mixture volume can be estimated by measuring the liquid flows and using the experimentally determined concentration of dissolved NH_3 and product solution density. This approach appeared to be the method of choice for calculating product mixture volume and, hence, for CA yield provided that the flowmeters were closely monitored. The CA yield values obtained by this method were used in this report for discussion of the performance of the modified generator/TP system.

A modification of the above method for estimating volume used by MMC was based on a caustic mass balance. In this method, the NaOH (g/min) passed into the contactor was divided by the equivalent caustic (g/l) in the mixture. The calculated volume, however, is relatively sensitive to fluctuations in caustic input flows, and consequently larger variations in calculated CA yields resulted using this method.

Results of the Phase V study together with earlier work are discussed below.

F. DATA ANALYSIS

1. CA GENERATOR

Experimental data obtained in Runs 1 through 3 of Phase V are given in Appendix E. Raw data obtained from MMC were reduced by Aerospace into CA yield and contactor concentration values amenable to statistical evaluation. Reduced data are shown in Appendix C.

CA yields obtained for individual samples as a function of run times for the three Phase V tests are plotted in Figure 9, and the averages for each condition are summarized in Table 1. In Run 1, conditions A and E were duplicate tests, yet considerably different values were obtained. Comparison with other values indicated condition A yield was inconsistent; it was not included in the statistical analysis of the data. For similar reasons,

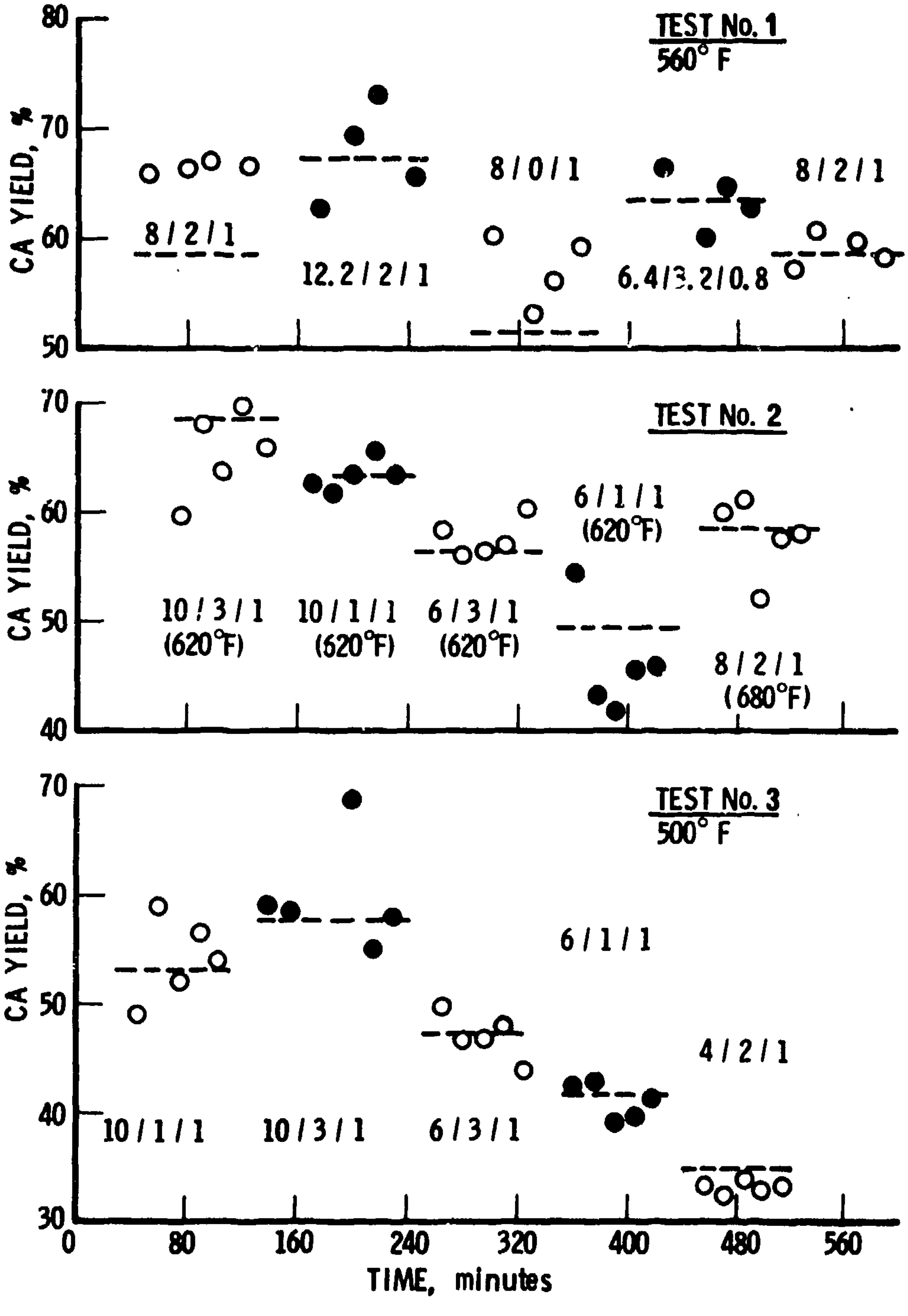

Figure 9. Top Hat Runs, CA Yields vs Run Times

Table 1. CA Generator/Thermal Precipitator Performance

Run No. 4 Condition		$NH_3/N_2/Cl_2$[a]	Cone Temp., °F	CA Yield, %
I	A	8/2/1	560	66.46[b]
	B	12.2/2/1	560	67.74
	C	8/0/1	560	57.13[b]
	D	8/4/1[c]	560	63.38
	E	8/2/1	560	59.10
II	A	10/3/1	620	66.85
	B	10/1/1	620	63.26
	C	6/3/1	620	57.72
	D	6/1/1	620	44.08[b]
	E	8/2/1	680	59.16
III	A	10/1/1	500	54.14
	B	10/3/1	500	57.62
	C	6/3/1	500	47.84
	D	6/1/1	500	41.22
	E	4/2/1	500	33.36

[a] Molar ratio based on Cl_2 flow of 37.5 g/min

[b] Inconsistent data. Not used in statistical analyses.

[c] Based on Cl_2 flow of 30.0 g/min

values for Run 1 condition C, and Run 2 condition D, were excluded in the derivation of the CA generator performance equation.

Evaluation of the CA generator data by regression analysis (Appendix D) gave an expression for CAyield as follows:

$$Y_2 = A[1 - e^{-B\lambda}] \qquad (1)$$

where, Y_2 = yield in decimal

$A = a + bT + cT^2$
$a = -2.44769530$
$b = 0.0103631115$
$c = -8.33124808 \times 10^{-6}$
T = cone temperature in °F

and where $B\lambda = (NH_3/Cl_2)\,[d + f\,(N_2/Cl_2)]$ and

$d = 0.14683221$
$f = 0.0234630876$

A very good fit was obtained. The correlation coefficient is 0.975, and the error mean square between data and the equation predictions is 0.0008 for 12 data points. This equation relating CA yield to operating conditions is applicable to the Top Hat design of the MMC 2-lb/hr CA generator. The generator efficiency, however, is dependent on equipment design and to some extent on the specific apparatus. Nevertheless, the derived equation correlates the basic operating parameters with yields expected upon scaleup to a CA generator having a similar configuration.

Representative plots of the variations in CA yield as a function of NH_3/Cl_2 molar ratios without N_2 and with N_2 added are shown in Figure 10. CA generator efficiency increases more rapidly with increasing NH_3 at lower molar ratios, but the effect is less pronounced at higher ratios. An increase of approximately 7 percent in CA yield is observed upon dilution of

Cl_2 with 2 moles of N_2 in comparison to the case where no N_2 was added. However, this increase is somewhat less when 4 moles are added. Although not obvious in Figure 10, N_2 addition at very high NH_3/Cl_2 ratios has little effect improving the CA yield. These results are consistent with the known chemistry of NH_3/Cl_2 reaction to form CA.

Figure 11 shows the changes in CA yield by variations in the cone temperature. The yield curve reaches a maximum at about 620° F, above which presumably a decomposition of the product occurs.

The derived mathematical expression provides a reasonable prediction of CA yields within the region of the experiments. Due to the paucity of data, however, extrapolations to other conditions outside the test region would result in less reliable values. A mathematical expression (Eq. 1) was used for the first stage reactor in the economic analysis of amine fuel production.

2. CA-AMINE REACTION

A statistical evaluation of the second-stage reaction of CA and amine in aqueous caustic solution was performed. Data used in this regression analysis included the results from Runs 20 through 24 and Top Hat Runs 2 and 3 for the UDMH system (Appendixes C and E). MMH test results include Runs 2B-8 and Run 1 (Appendies C and E). These data are tabulated in Appendix E and the regression analysis approach used is given in Appendix D.

An equation for the resultant fuel concentration in moles per liter (Y_3) was derived as a function of reactant concentrations of amine (X_1), NaOH (X_2), and CA (X_3) in the contactor prior to any reaction taking place. No independent variable of an order greater than 2 was considered. Several interaction terms were eliminated after computer analyses indicated they were not significant.

The empirical equations that describe the fuel concentrations resulting from the second stage reaction have the following form:

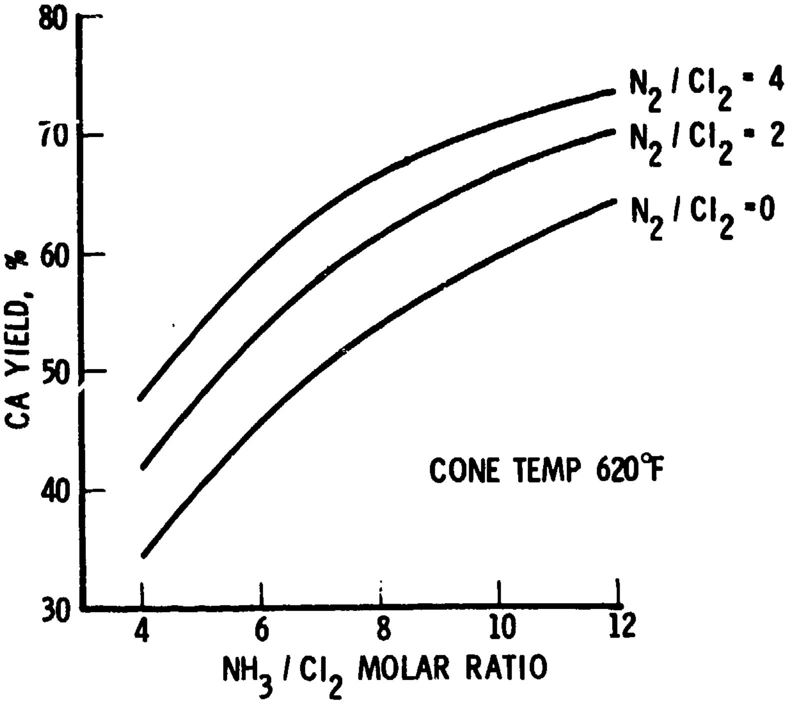

Figure 10. Effect of NH_3/Cl_2 Ratio on CA Generator Performance

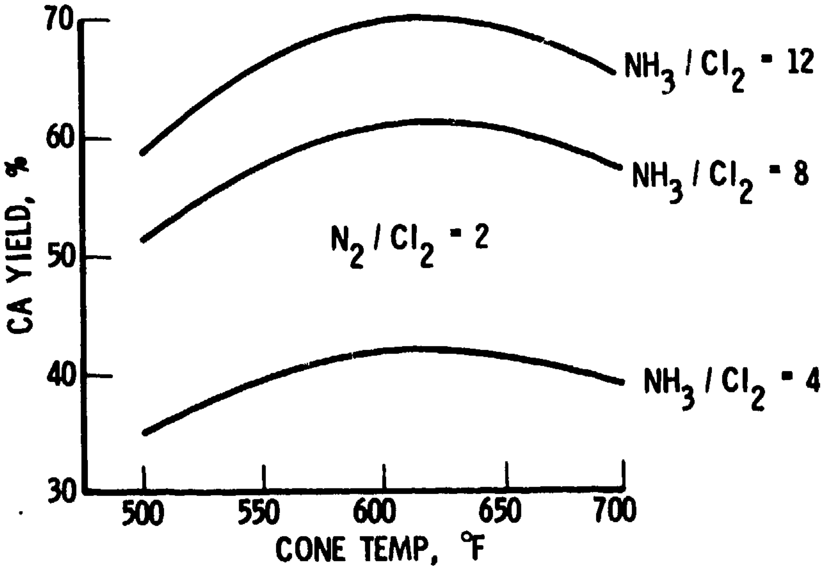

Figure 11. CA Yield vs Generator Cone Temperature

$$Y_3 = aX_1X_3 + bX_1X_3^2 + cX_1X_2X_3 + dX_1^2X_2X_3 + eX_1X_2^2X_3 + fX_1^2X_3 + gX_1X_2X_3^2 + hX_1^2X_3^2 + iX_1^2X_2^2X_3 + jX_1X_2^2X_3^2 + kX_1^2X_2X_3^2 + lX_1^2X_2^2X_3^2 \qquad (2)$$

where

	UDMH	MMH
a =	0.73778364	0.032871037
b =	0.46098250	000
c =	0.27815976	0.21449172
d =	0.048612608	-0.016759911
e =	0.080617422	-0.068893162
f =	-0.097538708	000
g =	0.23704285	-0.13920114
h =	0.036948483	000
i =	-0.018880704	0.0056778572
j =	-0.049607992	0.053908614
k =	000	0.011814990
l =	000	-0.0049383710

In both cases a very close fit was obtained in which the error mean square between the experimental data and the equation was 0.00064 for 143 and 222 data points for UDMH and MMH systems, respectively. The correlation coefficients were 0.988 and 0.967, respectively. Although several terms in the two equations differ, the resultant curves of product concentrations with variations in reactants exhibit similar shapes. The similarity would be expected from the known chemistry of the reactions. It should be emphasized that the equations are applicable primarily for the region of reaction conditions where the data were obtained.

The effects on fuel concentration of changes in DMA, NaOH, and CA concentrations as derived from Eq. (2) for the UDMH system are shown in Figures 12 through 14. In Figure 12, at constant caustic value, the fuel concentration exhibits a maximum with increasing DMA. CA-amine chemistry would predict a plateau, rather than a maximum. The decrease of the fuel concentration at high DMA value may be a reflection of the two-phase condition being reached in the contactor. The shift in curve maximum to higher DMA values for the higher CA values supports this interpretation, since the HCl from the amine-CA reaction acts to lower the residual caustic concentration. (See Figures 5 and 6). This drives the solution to single phase. DMA concentrations above 5.5 m/l could not be reached because of vaporization.

Many attempts were made to obtain experimental data for the second-stage reaction deep into the two-phase region of the amine/NaOH/H_2O phase diagram. These regions of interest were for amine and caustic concentrations of $>$ 5.5 and $>$ 2.5 m/l, respectively, for the UDMH system. Corresponding concentrations for the MMH system were $>$ 13 and $>$ 2.0 m/l. All of these attempts were unsuccessful due to the boil-off of the amine with resultant considerable decrease in the amine concentration.

Figure 13 shows the variations in UDMH concentration with caustic at two levels of DMA and CA. The operating range in terms of caustic is limited by having insufficient NaOH to neutralize HCl on one side and the two-phase separation of the mixture on the other. NaOH concentration in the contactor must be equal to or exceed the CA value, since the HCl formed in the reaction must be neutralized and converted to sodium chloride. Insufficient caustic results in CA attack on the product with concomitant decrease in yield.

Shown in Figure 14 are the representations of UDMH concentration and yield versus CA concentrations. At low CA concentrations the yield is high, but it decreases with increasing CA concentration. At low DMA values, the

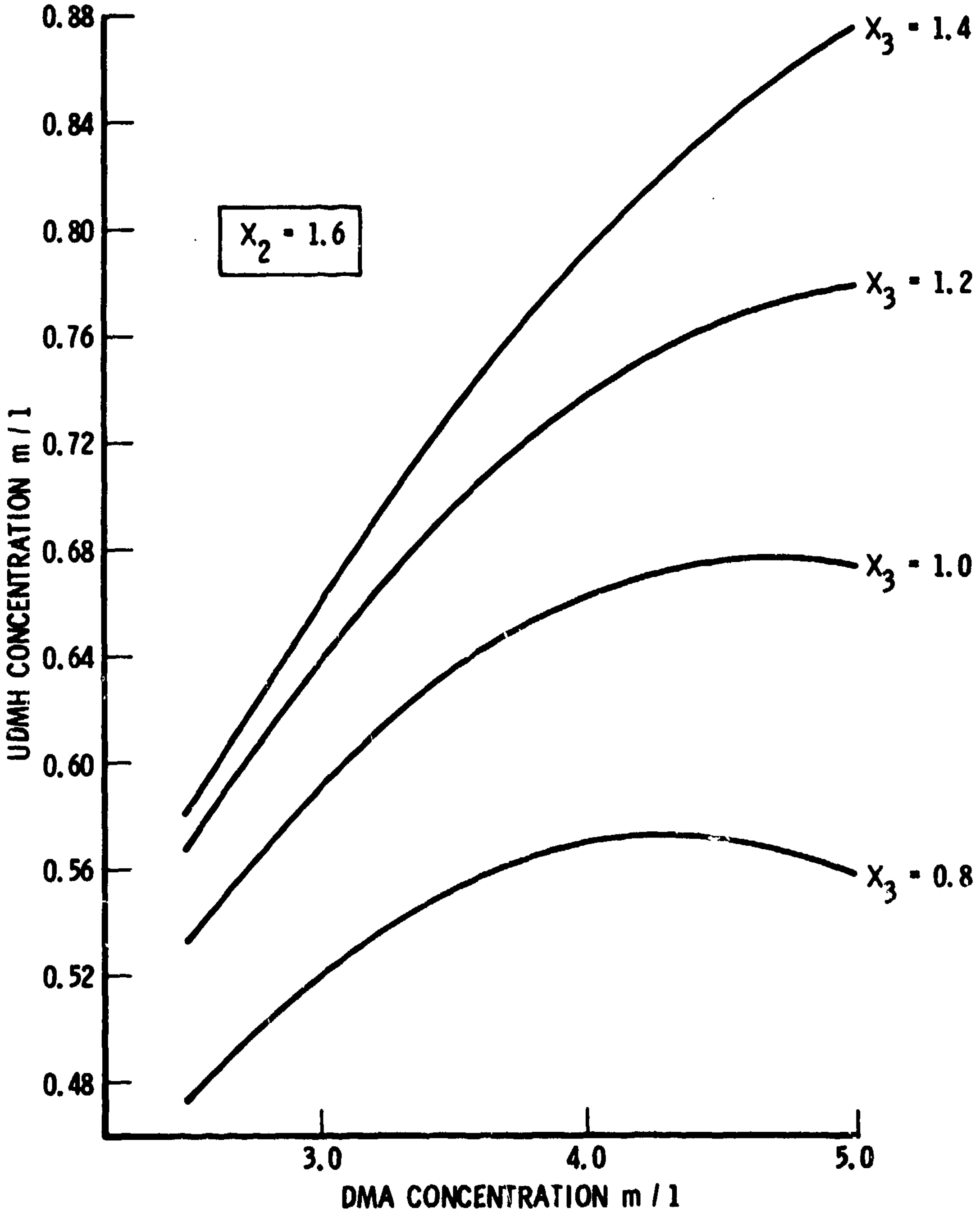

Figure 12. UDMH vs DMA Concentration

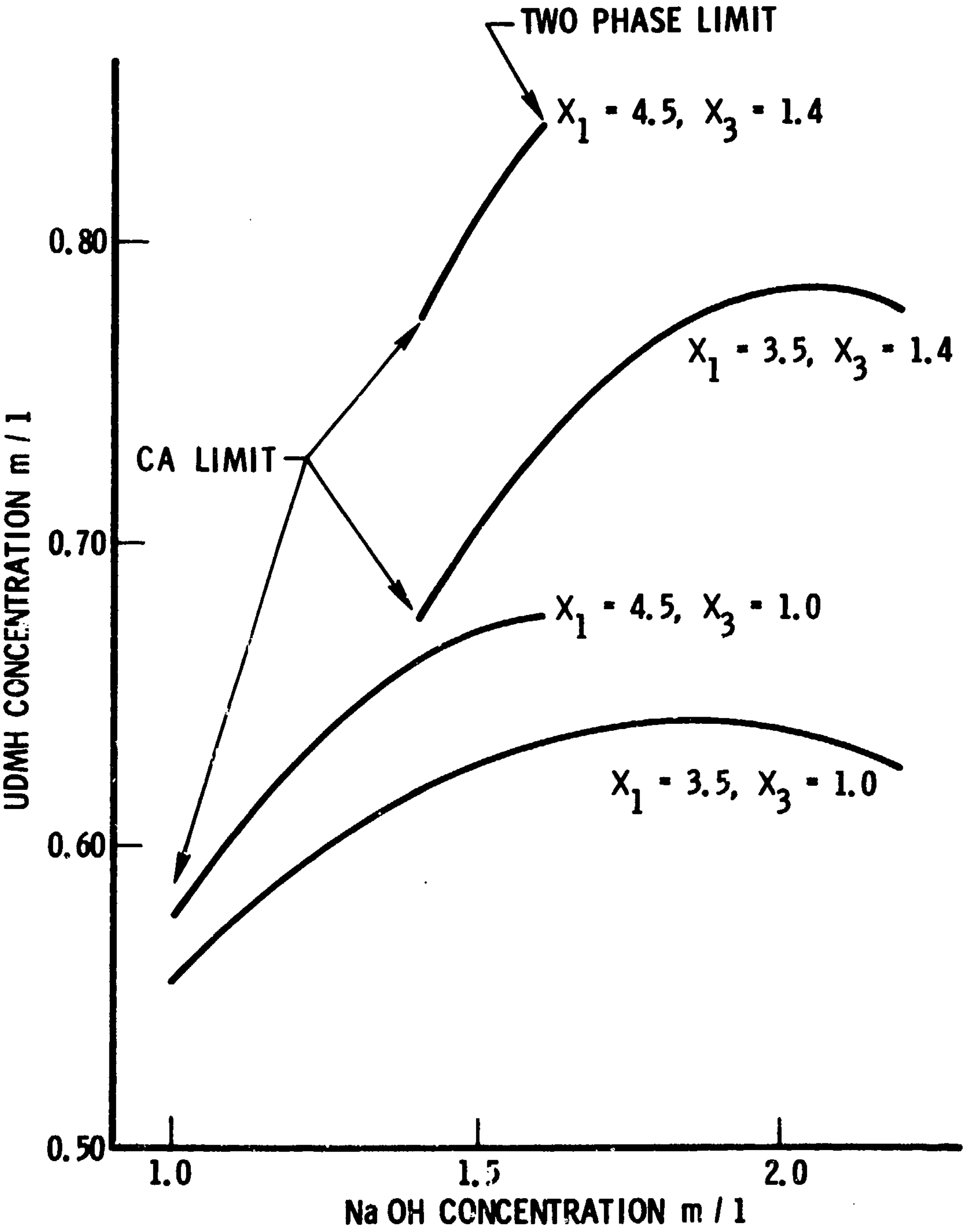

Figure 13. UDMH vs NaOH Concentration

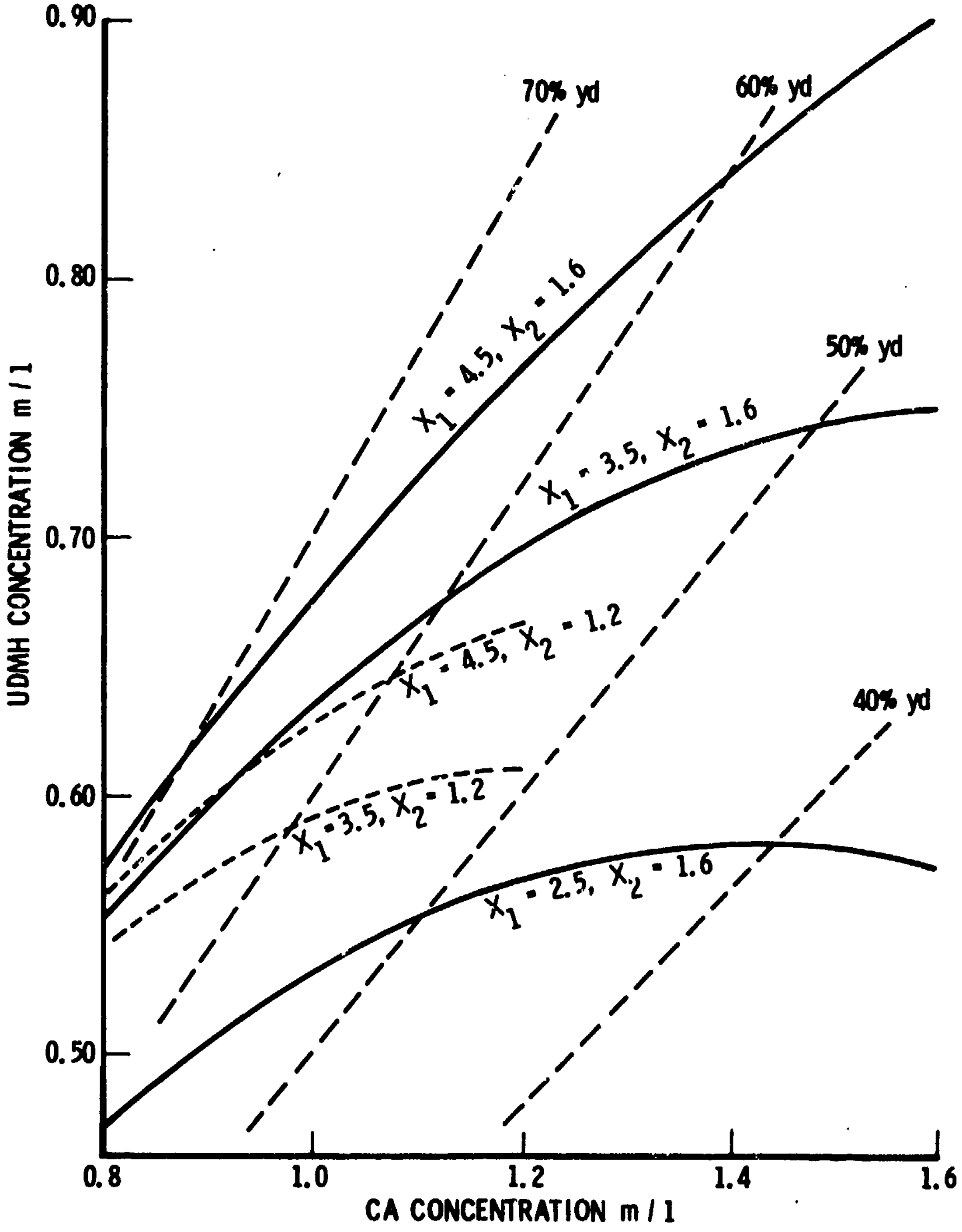

Figure 14. UDMH vs CA Concentration

fuel concentration increases to a maximum and then decreases with increasing CA level. Although a similar situation exists for the higher amine conditions, the region of maximum fuel value is not reached before all the caustic is depleted. As mentioned earlier, the derived equation for UDMH concentration is not valid when CA exceeds the amount of NaOH present in the reactor. Under such conditions, another CA-amine reaction mechanism is believed to prevail where side product formation becomes the predominant reaction.

The plots of Eq. (2) for the MMH systems, Figures 15 through 17, exhibit similar shapes albeit at different reactant concentrations. The CA-MMA reaction requires higher amine and lower CA concentrations to effectively produce the fuel. As a consequence, the attainable MMH concentration in the contactor is lower than in the UDMH system. These factors contribute to a higher cost per pound of fuel for MMH.

G. SUMMARY

The design of the final 2 lb/hr amine fuel production system is suitable for scaleup to a larger pilot size. However, the effects of volume (hold-up time) and area are not defined for scaling.

The Top Hat configuration of the CA generator/TP unit permits better temperature control and a more stable operation of the first-stage reactor. It consists of the following:

a. A CA generator with a conical section constructed of Inconel metal and a cylindrical NH_4Cl settling chamber (TP).

b. A hopper at the bottom of the TP equipped with a revolving blade to prevent NH_4Cl bridging.

c. An adjoining isolated compartment with double-slide valves for mechanical removal of NH_4Cl without interrupting the run.

d. An improved electrostatic precipitator for separating residual NH_4Cl from the effluent gas stream.

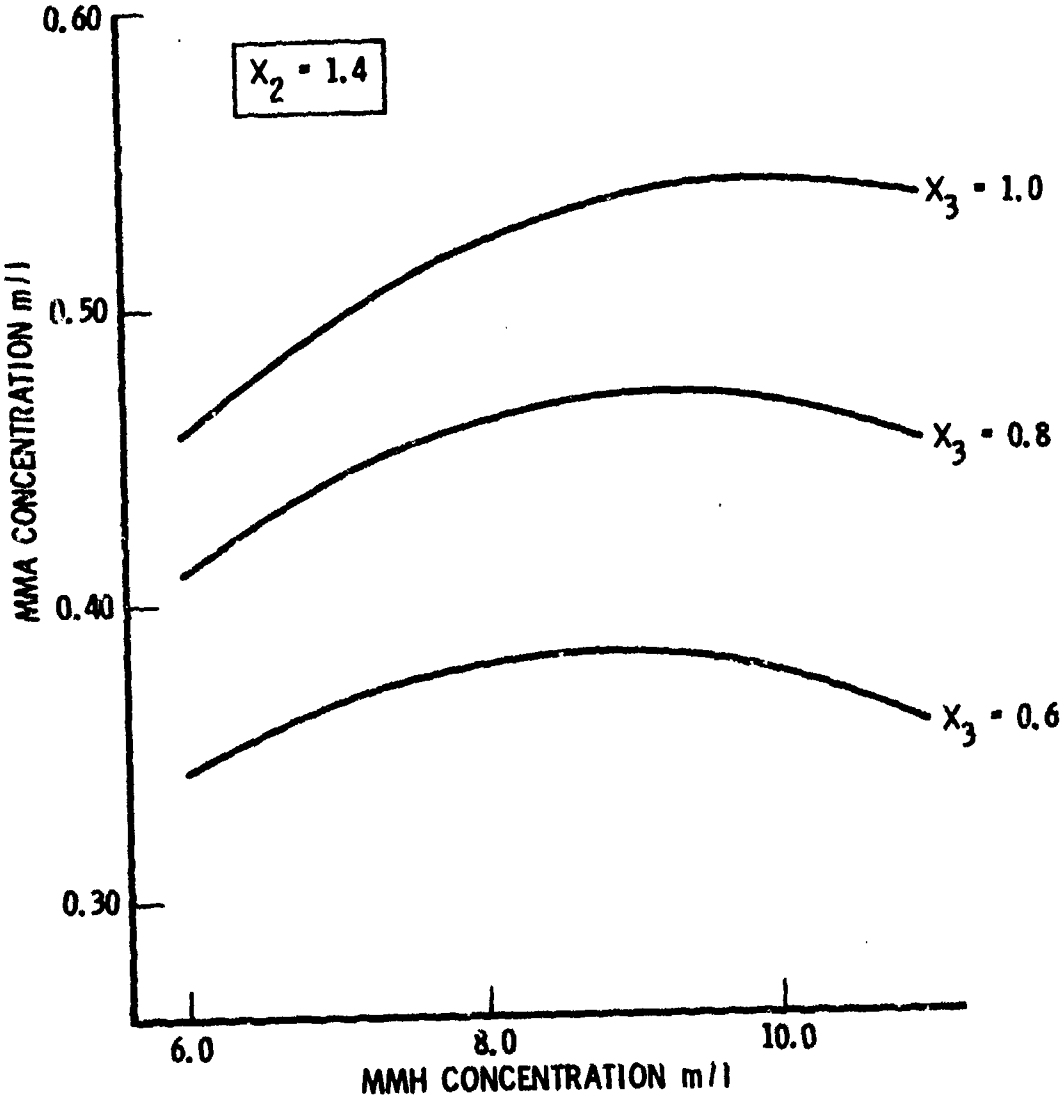

Figure 15. MMH vs MMA Concentration

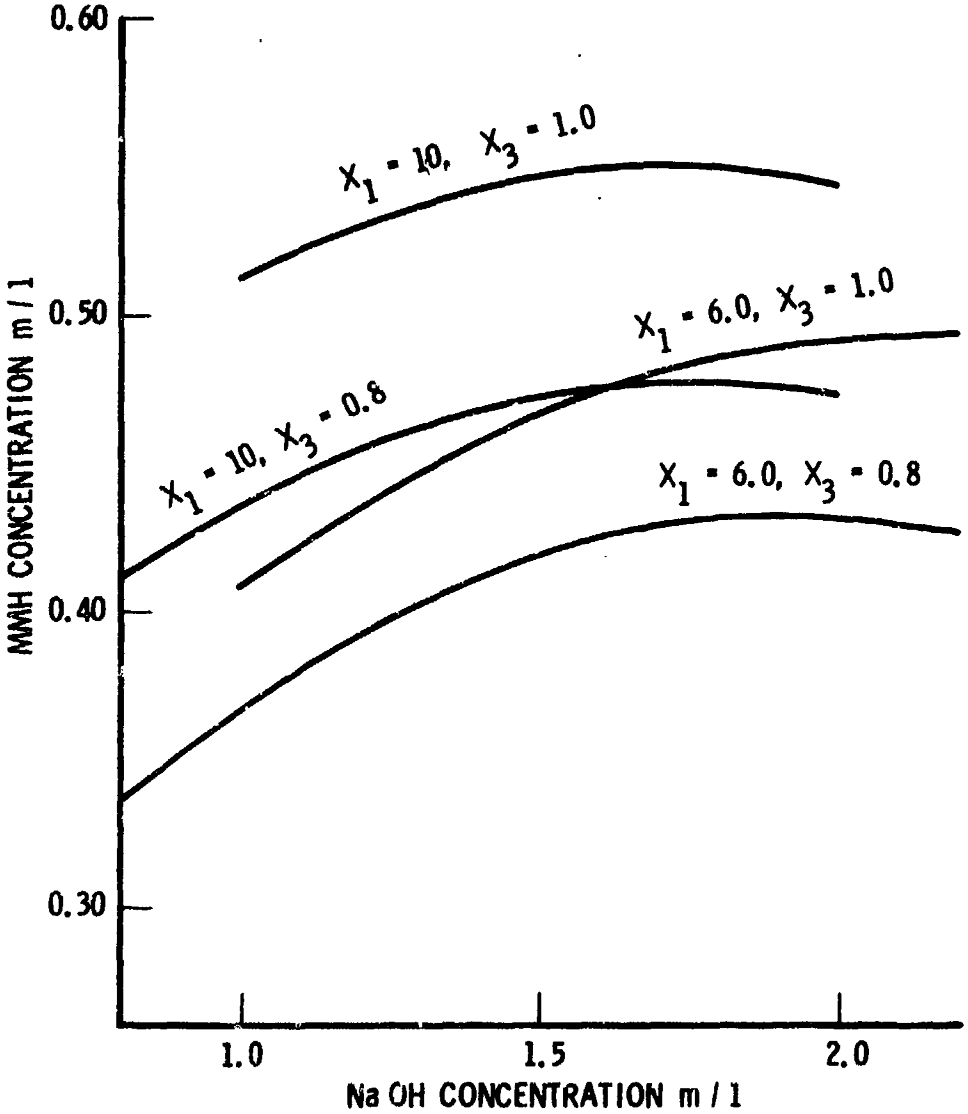

Figure 16. MMH vs NaOH Concentration

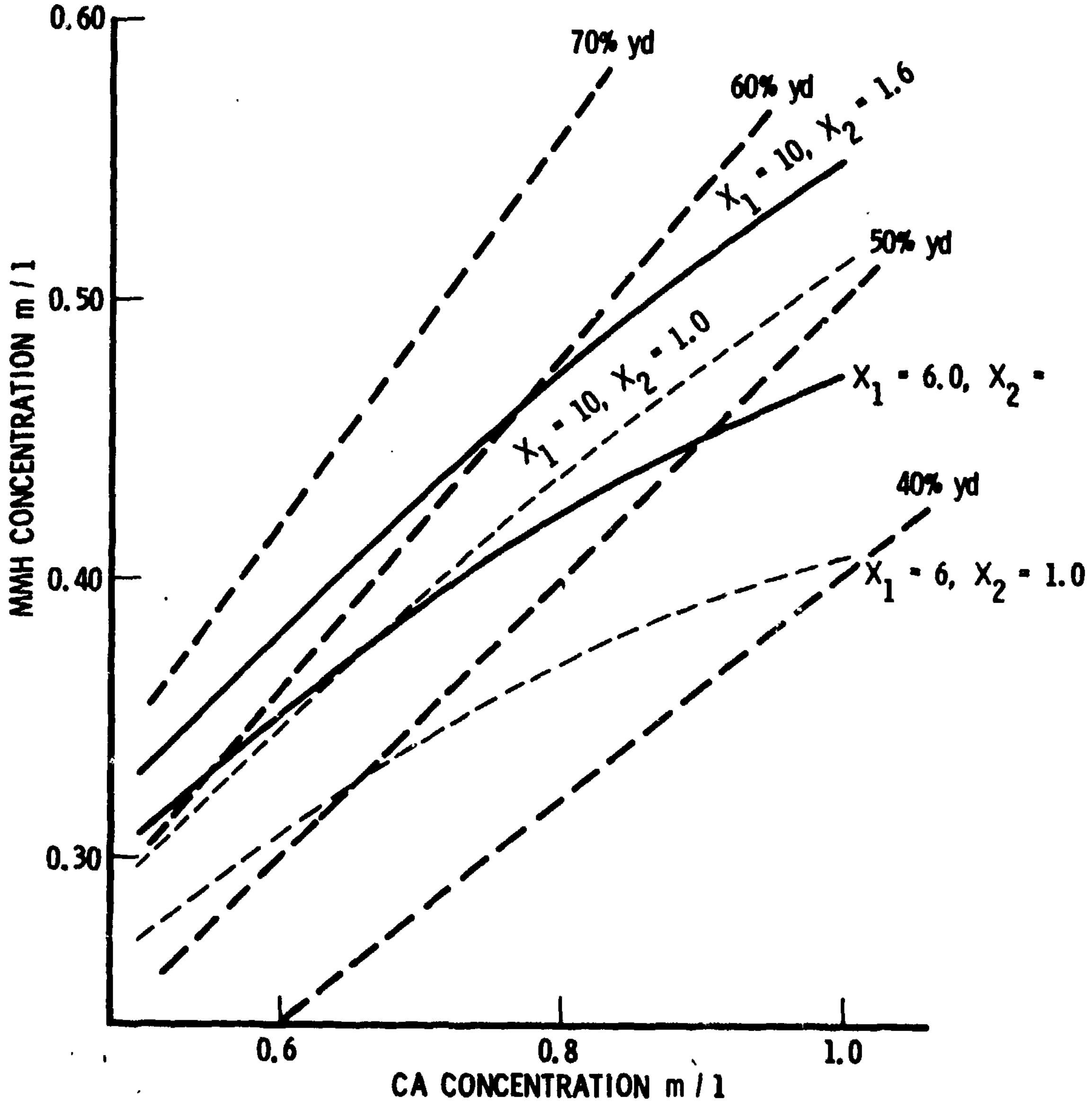

Figure 17. MMH vs CA Concentration

e. A 180 deg bend of the gas stream exiting the TP to facilitate solid settling.

f. Thermocouples appropriately located for monitoring temperature.

g. Vibrators on the TP and electrostatic precipitator to prevent NH_4Cl accumulation on the wall.

The Top Hat configuration of the CA generator/TP unit (Figure 5) was still oversized and should be reduced in scale. Additional design consideration should include arrangement of the components that will permit ready access for maintenance and repair. The first-stage reactor system so constructed should be capable of long-term operation.

The stirred-bubbler system (Figure 2) described in Section 3.2 served satisfactorily as the second stage reactor. The stirrer design appeared to be critical in obtaining good yields.

The development of techniques to analyze constituents in the second-stage reactor from which both yields and quantity of product formed could be calculated resolved chemical analysis problems. The procedure requires stringent control of the input gas and liquid feeds necessitating use of reliable, calibrated flow meters.

The final system, developed after much time and effort, is believed to be capable of long-term operation. The MMC chloramine process is a feasible process for the production of both UDMH and MMH. However, the scaling factors of the chloramine generator - precipitator are not understood. Before attemping a 200 lb/hr plant, a 20 lb/hr generator size should be designed and tested.

Regression analyses of the first- and second-stage reaction data obtained during the latter phases of this study gave mathematical expressions correlating product yields and concentrations with process conditions. The derived equations not only show the sensitivity of the system to variations in a specific parameter but also indicate the operating regions where the process would be economically attractive.

No attempt was made to investigate the production of hydrazine in the same facility; however, the higher pressure requirements indicated in the literature makes the gaseous chloramine process appear to be unattractive because of the potential explosive hazard of pressurized chloramine.

III. ECONOMICS

A. RAW MATERIALS/UTILITIES

1. GENERAL APPROACH

The major economic variable of the chloramine process for UDMH and MMH production was estimated by deriving a general cost equation. The expression yields the basic operating cost of raw materials and utilities per pound of product, as a function of parameters such as reactant concentrations, reaction yields, and recycle stream purification. Equations 1 and 2 (pages 32 and 35), which predict the CA generator efficiency and contactor product concentration, respectively, served as the primary bases for this cost equation. From the stoichiometry of the reaction, the composition of the product mixture and various process streams was calculated. The size of each stream in pounds per hour was determined by using an assumed production rate of 400 lb of fuel per hour of operation. The excess NH_3 and amines in the product stream and a portion of the water are recycled. The residual caustic is neutralized with HCl and converted to NaCl and water.

The product costs were derived by considering the specific costs of raw materials, recycle and waste water clean-up, and utility. Raw material costs* per pound are:

Chemical	Cost, $/lb	Chemical	Cost, $/lb
NH_3	0.07	MMA or DMA	0.295
Cl_2	0.07	NaOH	0.08
N_2	0.03	HCl (anhyd.)	0.29

The same source lists the price of NH_4Cl as $0.125/lb, but no credit for the sale of this material was taken in calculating the initial operating cost.

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*From Chemical Marketing Reporter, 26 September 1977.

However, a partial credit can be taken in determining final cost after adding fixed costs. The value of the second by-product, NaCl, was assumed to cover handling.

Other assumptions which were made included \$0.01/lb of product for the purification of water and \$0.03/lb for both NH_3 and amine purification in the recycle streams, plus a 5 percent mass loss during recycle. A utility cost expression was derived from the values given by Rockwell International.[5] It was estimated that 100 lb of unspecified structured side products were formed for every 400 lb of fuel produced. Finally, no loss of product was considered in the purification process. A 10 percent loss would raise the final cost at least an equivalent 10 percent.

For derivation of the economics equation, the following notations were used throughout:

CA Generator Reaction

NH_3/Cl_2 = molar ratio

N_2/Cl_2 = molar ratio

Y_2 = CA yield based on Cl_2 (in decimal)

CA-Amine Reaction

X_1 = Amine conc. (mole/liter)

X_2 = NaOH conc. (mole/liter)

X_3 = Ca conc. (mole/liter)

Y_1 = Amine fuel yield based on CA (in decimal)

Y_3 = Amine fuel conc. (mole/liter)

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[5] F. E. Raniere and M. Frankel, Hydrazine Manufacturing Process, Final Report, AFRPL-TR-76-4, Rockwell International, Canoga Park (March 1976).

The general approach used in the derivation of the economics equation involves mass flows and assignment of cost/lb. The total cost is the sum of the following:

Total = Raw Material + Recycle and Clean-up + Utility

where

Raw Materials = [NH_3 and Amine]$_{\text{consumed \& recycle loss}}$ + Cl_2 + N_2 + NaOH + HCl

Recycle and Clean-up = NH_3 + Amine + H_2O

Utility = product distillation and purification

The required quantity of all reactants was ultimately related to the amount of CA needed to produce 400 lb/hr of fuel, as predicted by the statistical equations derived from the regression analysis of the data obtained on the 2 lb/hr unit. From the CA value, the quantity of NH_3 consumed or recycled was calculated based on the amount of Cl_2 used. CA yield, and the stoichiometry of the chemical equations (step 1) for the formation and destruction of CA.

$$2\,NH_3 + Cl_2 \longrightarrow NH_2Cl + NH_4Cl \qquad \text{(Reaction 1)}$$

$$3\,NH_2Cl + 2\,NH_3 \longrightarrow 3\,NH_4Cl + N_2 \qquad \text{(Reaction 2)}$$

Of the residual NH_3, 5 percent was assumed to be lost during recycle.

The amine cost was calculated based on second-stage reaction yield and on the required concentration and volume flow in the contractor. The contactor reaction is:

NH_2Cl + Amine + NaOH ⟶ Hydrazine + NaCl + H_2O + Side Products

(Reaction 3)

Total moles of amines consumed were assumed to be equal to the equivalent amount of fuel produced plus an additional estimated quantity consumed in yielding the side products as follows:

$$\text{Amines}_{(\text{moles consumed})} = \frac{1 + Y_1}{2} [CA_{\text{moles}}]$$

During recycle 5 percent of the residual amine was also estimated to be lost. For both NH_3 and amine, the recycled quantity can be represented by:

$$\text{Recycled} = [\text{Total Input Flow}] - [\text{Consumed}]$$

The total amount of water to be purified was calculated by subtracting the masses of all the constituents from the total weight. Average densities of 0.95 and 0.91 g/ml were used for UDMH and MMH product mixtures, respectively, to obtain the total weight. Other constituents, either consumed or produced, were determined based on the known stoichiometry of the chemical equations given in Reactions 1, 2, and 3.

The utility costs, which represent the energy expense involved in product distillation and purification, were derived from the data provided in the Rockwell report.[5] This report gave values for cost as a function of wt% of the fuel in the product mixture from which the following empirical equation was derived:

$$\text{Cost } (\$/\text{lb}) = \frac{1.011}{\text{wt\%}} - 0.01404$$

After applying a density correction for the two fuel systems, this equation was converted to an expression involving amine fuel concentration in moles per liter.

The results of these derivations are shown in Table 2 for a 400 lb/hr UDMH and MMH systems. The columns list the compound, its disposition, and the equation yielding pounds per hour of the material in the stream for the two hydrazine fuels.

In Table 3 are the cost equations for each item per pound of fuel produced. The price per pound of each item utilized is also shown so that future variations in the itemized cost can easily be incorporated to modify the equations. The derived cost expressions were used to calculate the individual costs as a function of the reaction conditions, and thus its impact on the overall price of the fuel.

2. CA GENERATOR CONDITIONS

The overall cost of the fuel is significantly affected by variations in operating conditions of the CA generator. Equation 1 (Section 3.6.1), which was used for this analysis, gives CA yield as a function of NH_3/Cl_2; N_2/Cl_2; and generator cone temperature. UDMH cost as a function of NH_3Cl_2 molar ratio where no nitrogen was used is shown in Figure 18. A constant downstream contactor condition for DMA (X_1) of 4.5 m/l, NaOH (X_2) of 1.6 m/l, and CA (X_3) of 1.4 m/l was maintained so that a comparison of the cost would reflect the effect of the efficiency of the first reaction.

Variations in the final costs shown represent the differences in raw material cost in generating CA and in the recycling of excess NH_3. At low NH_3/Cl_2 ratio, the higher cost is due to the low CA yield; at high ratios, where the yield is high, the recycling cost of the excess NH_3 becomes a significant cost factor. The optimum yield at NH_3/Cl_2 ratio of 10 is 60 percent. Although the addition of N_2 to dilute the Cl_2 results in a higher CA yield, this increase is offset by the additional cost of N_2. The net effect is an increase of a few cents per pound on the overall cost of the amine fuel.

Table 2. Process Mass Flow for 400 lb/hr Plant

Chemical	Description	Mass Flow (lb/hr)	
		UDMH	MMH
NH_3	Consumed	$\frac{1}{Y_1}\left[\frac{303.33}{Y_2} - 75.61\right]$	$\frac{1}{Y_1}\left[\frac{394.36}{Y_2} - 98.63\right]$
NH_3	Recycled	$\frac{1}{Y_1}\left[\frac{113.36\,(NH_3/Cl_2) - 302.33}{Y_2} + 75.61\right]$	$\frac{1}{Y_1}\left[\frac{147.87\,(NH_3/Cl_2) - 394.36}{Y_2} + 93.63\right]$
NH_3	Loss during recycle (5%)	$\frac{1}{Y_1}\left[\frac{5.67\,(NH_3/Cl_2) - 15.12}{Y_2} + 3.78\right]$	$\frac{1}{Y_1}\left[\frac{7.393\,(NH_3/Cl_2)}{Y_2} - \frac{19.72}{Y_2} + 4.93\right]$
Cl_2	Consumed	$\frac{471.97}{Y_1 Y_2}$	$\frac{615.65}{Y_1 Y_2}$
NH_4Cl	Produced	$\frac{712.11}{Y_1 Y_2} - \frac{356.05}{Y_1}$	$\frac{928.89}{Y_1 Y_2} - \frac{464.44}{Y_1}$
N_2	Required	$\frac{186.46}{Y_1 Y_2}\,(N_2/Cl_2)$	$\frac{243.23\,(N_2/Cl_2)}{Y_1 Y_2}$
CA	Required	$\frac{342.64}{Y_1}$	$\frac{446.94}{Y_1}$
MMA	Consumed	$\frac{600.12}{1 + Y_1}$	$\frac{539.27}{1 + Y_1}$
MMA	Recycled	$\frac{300.06\,X_1}{Y_3} - \frac{600.12}{1 + Y_1}$	$\frac{269.64\,X_1}{Y_3} - \frac{539.27}{1 + Y_1}$
MMA	Loss during recycle (5%)	$\frac{15.00\,X_1}{Y_3} - \frac{30.01}{1 + Y_1}$	$\frac{13.48\,X_1}{Y_3} - \frac{26.96}{1 + Y_1}$
NaOH	Total consumed	$\frac{256.26\,X_2}{Y_3}$	$\frac{347.31\,X_2}{Y_3}$
HCl	Neutralize X's NaOH	$\frac{242.70\,X_2}{Y_3} - \frac{242.70}{Y_1}$	$\frac{316.58\,X_2}{Y_3} - \frac{316.58}{Y_1}$
NaCl	Produced	$\frac{389.04\,X_2}{Y_3}$	$\frac{507.48\,X_2}{Y_3}$
H_2O	Produced	$\frac{119.91\,X_2}{Y_3}$	$\frac{156.41\,X_2}{Y_3}$
Product Mixture	Total wt.	$\frac{6322.8}{Y_3}$	$\frac{7900.3}{Y_3}$

H_2O - Total in Product Mixture (lb/hr)

UDMH System

$$\frac{1}{Y_3}\left[6332.8 - 300.06\,X_1 - 389.04\,X_2\right] - \frac{1}{Y_1}\left[\frac{113.36\,(NH_3/Cl_2) - 302.33}{Y_2} + 75.61\right] + \frac{600.12}{1 + Y_1} - 500$$

MMH System

$$\frac{1}{Y_3}\left[7900.3 - 269.64\,X_1 - 507.48\,X_2\right] - \frac{1}{Y_2}\left[\frac{147.87\,(NH_3/Cl_2) - 394.36}{Y_2} + 98.63\right] + \frac{539.27}{1 + Y_1} - 500$$

Table 3. Individual Process Cost Breakdown

Item		Unit Cost $/lb	UDMH	MMH
Raw Materials				
NH_3	(consumed + recycle loss)	0.07	$\frac{1}{Y_1}\left[\frac{0.0010\,(NH_3/Cl_2) + 0.0503}{Y_2} - 0.0126\right]$	$\frac{1}{Y_1}\left[\frac{0.0013\,(NH_3/Cl_2) + 0.0656}{Y_2} - 0.0164\right]$
Cl_2		0.07	$\frac{0.0826}{Y_1Y_2}$	$\frac{0.1077}{Y_1Y_2}$
N_2		0.05	$\frac{0.0140\,(N_2/Cl_2)}{Y_1Y_2}$	$\frac{0.0182\,(N_2/Cl_2)}{Y_1Y_2}$
Amine	(consumed + recycle loss)	0.295	$\frac{0.0111\,X_1}{Y_3} + \frac{0.4205}{1 + Y_1}$	$\frac{0.0099\,X_1}{Y_3} + \frac{0.3779}{1 + Y_1}$
NaOH		0.08	$\frac{0.0533\,X_2}{Y_3}$	$\frac{0.0695\,X_2}{Y_3}$
HCl		0.29	$\frac{0.1760\,X_2}{Y_3} - \frac{0.1760}{Y_1}$	$\frac{0.2295\,X_2}{Y_3} - \frac{0.2295}{Y_1}$
Recycle-Cleanup				
NH_3		0.03	$\frac{1}{Y_1}\left[\frac{0.0085\,(NH_3/Cl_2) - 0.0227}{Y_2} + 0.0057\right]$	$\frac{1}{Y_1}\left[\frac{0.0111\,(NH_3/Cl_2) - 0.0296}{Y_2} + 0.0074\right]$
Amine		0.08	$\frac{0.0225\,X_1}{Y_3} - \frac{0.0450}{1 + Y_1}$	$\frac{0.0202\,X_1}{Y_3} - \frac{0.0404}{1 + Y_1}$
H_2O		0.01	$\left\{\frac{1}{Y_3}\left[0.1583 - 0.0075\,X_1 - 0.0097\,X_2\right]\right.$ $+ \frac{0.0150}{1 + Y_1}$ $- \frac{1}{Y_1}\left[\frac{0.0028\,(NH_3/Cl_2) - 0.0076}{Y_2}\right.$ $\left.- 0.0019\right] - 0.0125$	$\left\{\frac{1}{Y_3}\left[0.1975 - 0.0067\,X_1 - 0.0127\,X_2\right]\right.$ $+ \frac{0.0135}{1 + Y_1}$ $- \frac{1}{Y_1}\left[\frac{0.0037\,(NH_3/Cl_2) - 0.0099}{Y_2}\right.$ $\left.- 0.0025\right] - 0.0125$
Utility		-	$\frac{0.1598}{Y_3} - 0.0140$	$\frac{0.1997}{Y_3} - 0.0140$

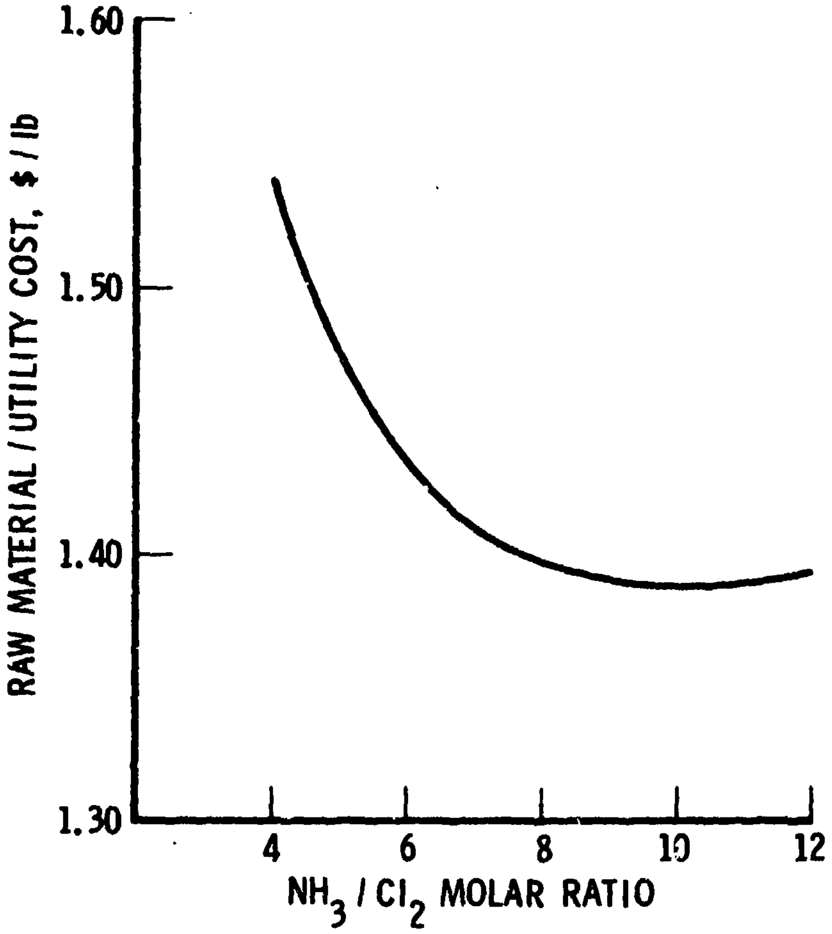

Figure 18. Effect of NH_3/Cl_2 Ratio on Fuel Cost

As indicated previously, the maximum first reaction yield attained in the 2 lb/hr unit was about 75 percent (NH_3/Cl_2 = 12, N_2/Cl_2 = 4), whereas laboratory tests in small glass hardware indicated yields as high as 90 percent. However, even if the yield in this example was raised from 60 to 70 percent without changing input conditions, the cost improvement would only be 8 cents/lb.

3. CONTACTOR REACTION CONDITION

The effects of the variations in CA-DMA reaction conditions in the second-stage reactor on the overall cost of the fuel is shown in Figures 19 and 20. These results are based on the product concentration and yield predicted by Eq. (2) for the UDMH system (page 35). For these analyses, the CA generator operating conditions were held constant at $NH_3/N_2/Cl_2$ molar ratios of 10/0/1 and a cone temperature of 620°F, giving CA yield of 60 percent. The generator condition using no N_2 was selected, since the economic analysis indicated its use would slightly increase the cost of the fuel.

In Figure 19 resultant UDMH costs per pound are plotted against contactor fuel concentrations for a constant CA (X_3) condition of 1.4 m/l. The solid lines represent loci of three values (3.5, 4.0, and 4.5 m/l) of DMA (X_1) for a range of NaOH (X_3) concentrations. The latter values are increased in 0.2 increments from 1.4 to 2.0 m/l. The dashed lines connect points of constant X_2 in a plane of constant X_3. Under these conditions, it is apparent that fuel costs are markedly affected by amine concentration and to a somewhat lesser degree by the caustic. Higher DMA levels would be preferred costwise, but such conditions are not attainable due to the resultant separation of the mixture into two phases which, in turn, results in amine boiloff.

Figure 20 shows a plot of UDMH cost versus product concentration where X_1 was held constant at 4.0 m/l. Four values of CA concentrations at 1.1, 1.4, 1.7, and 2.0 m/l are given by the solid lines, and the caustic

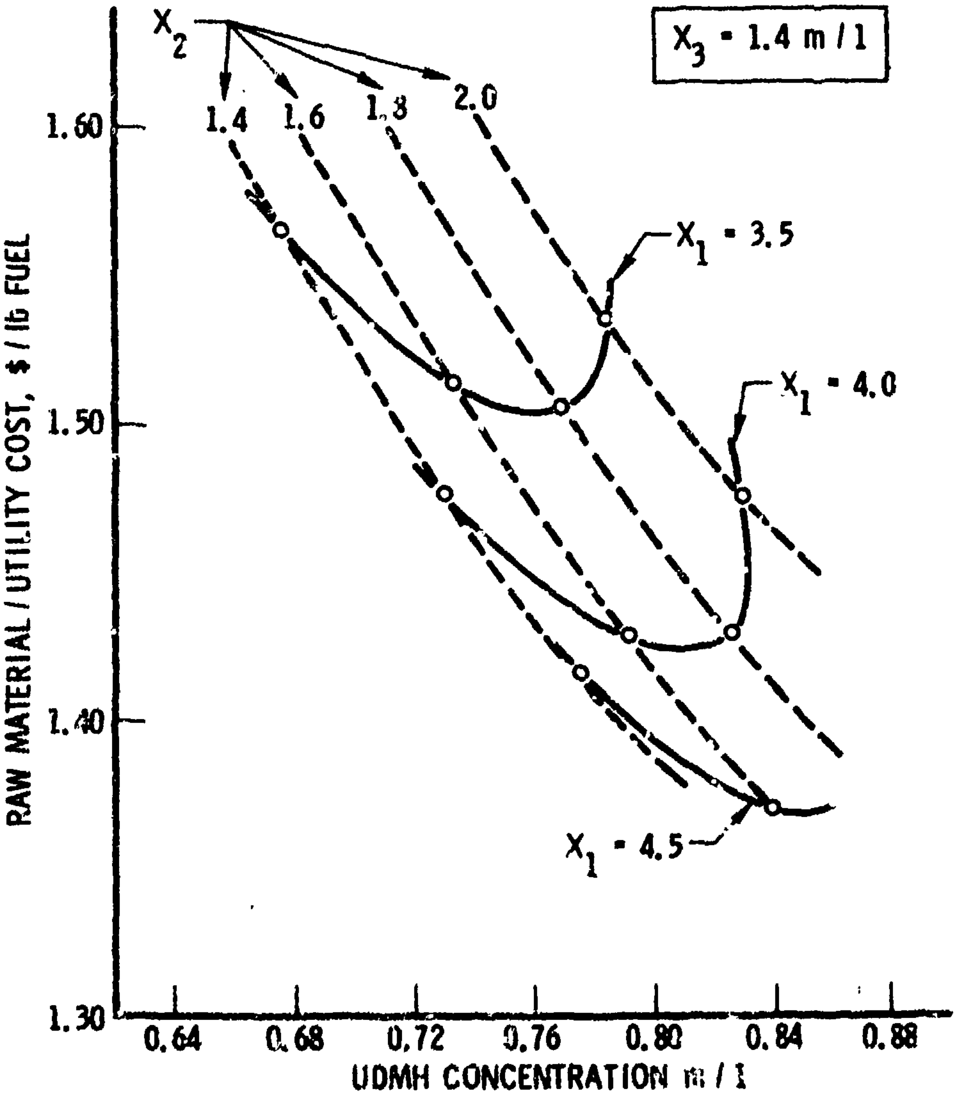

Figure 19. UDMH Cost at Constant CA Condition

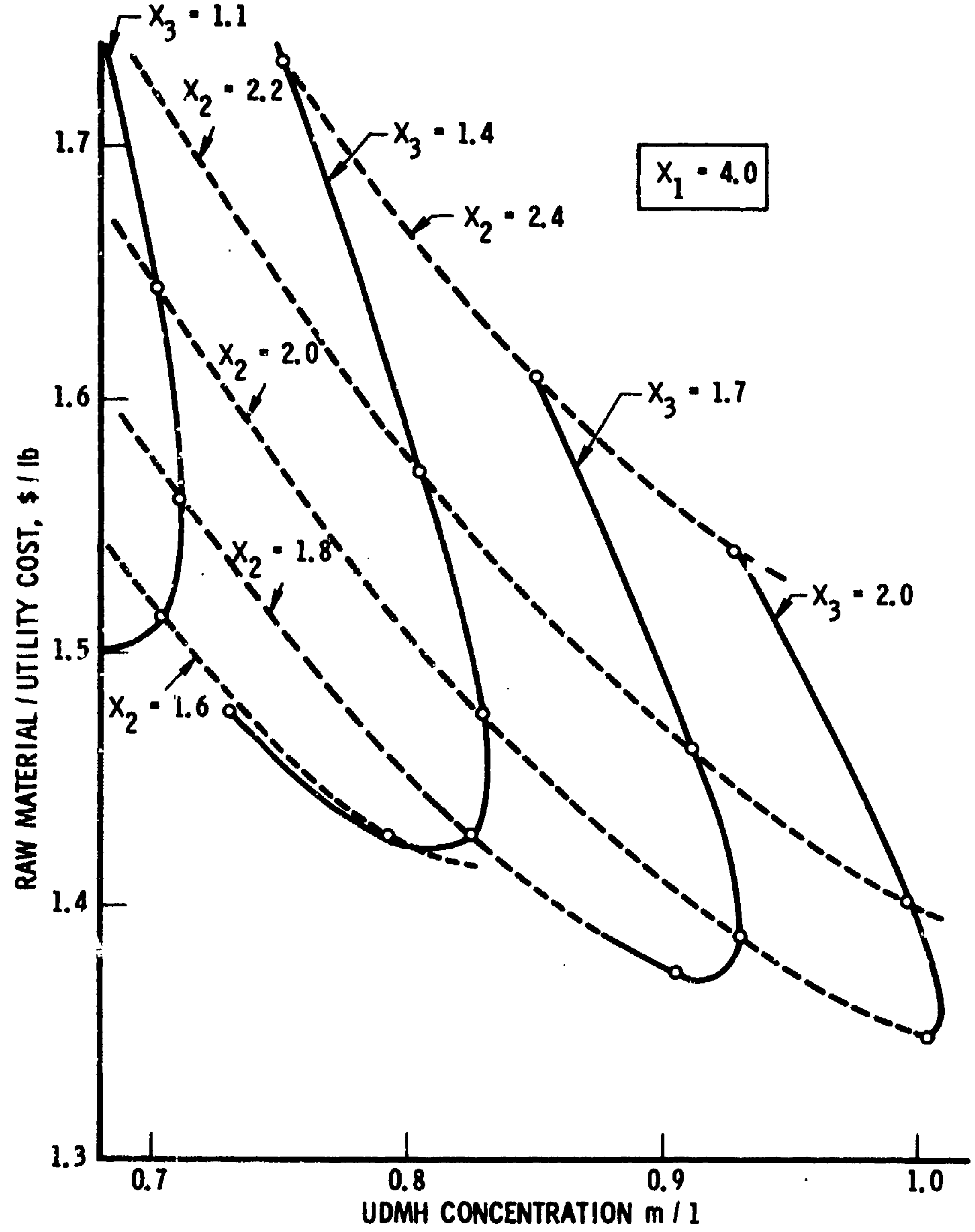

Figure 20. UDMH Cost at Constant DMA Condition

by the dashed curves from 1.6 to 2.4 m/l. As indicated in this graph, the economics of the process is greatly affected by the CA concentration. However, as CA increases, caustic must be increased to neutralize the HCl formed in the reaction. Increased caustic drives the solution to two phase, limiting the attainable amine concentration.

Similar graphs showing the effects of variations in the contactor operating condition on MMH cost are plotted in Figures 21 and 22 for constant CA and MMA conditions, respectively. In general, the comments made earlier for the UDMH system apply equally well to the MMH production process. However, the resultant MMH cost per pound of fuel is considerably higher than UDMH since the former requires higher amine concentrations, yet lower product concentrations are obtained.

Some representative examples of operating conditions for the CA generator and the contactor and their effects on the individual item costs for UDMH and MMH production are shown in Tables 4 and 5, respectively. Although the discussion in the previous sections dealt primarily with the UDMH system, the comparison of the values provided in these two tables shows the basic items which contribute to the higher overall cost of MMH production.

The economic analysis of the chloramine process for hydrazine fuel production indicates that the cost of the fuel is not overly sensitive to the operating parameters described in this section provided reasonable judgments are made in the choice of process conditions. The recommended initial pilot plant-phase operating conditions based on the current available information are as follows:

<u>CA Generator</u>

$NH_3/N_2/Cl_2$ molar ratio 10/0/1

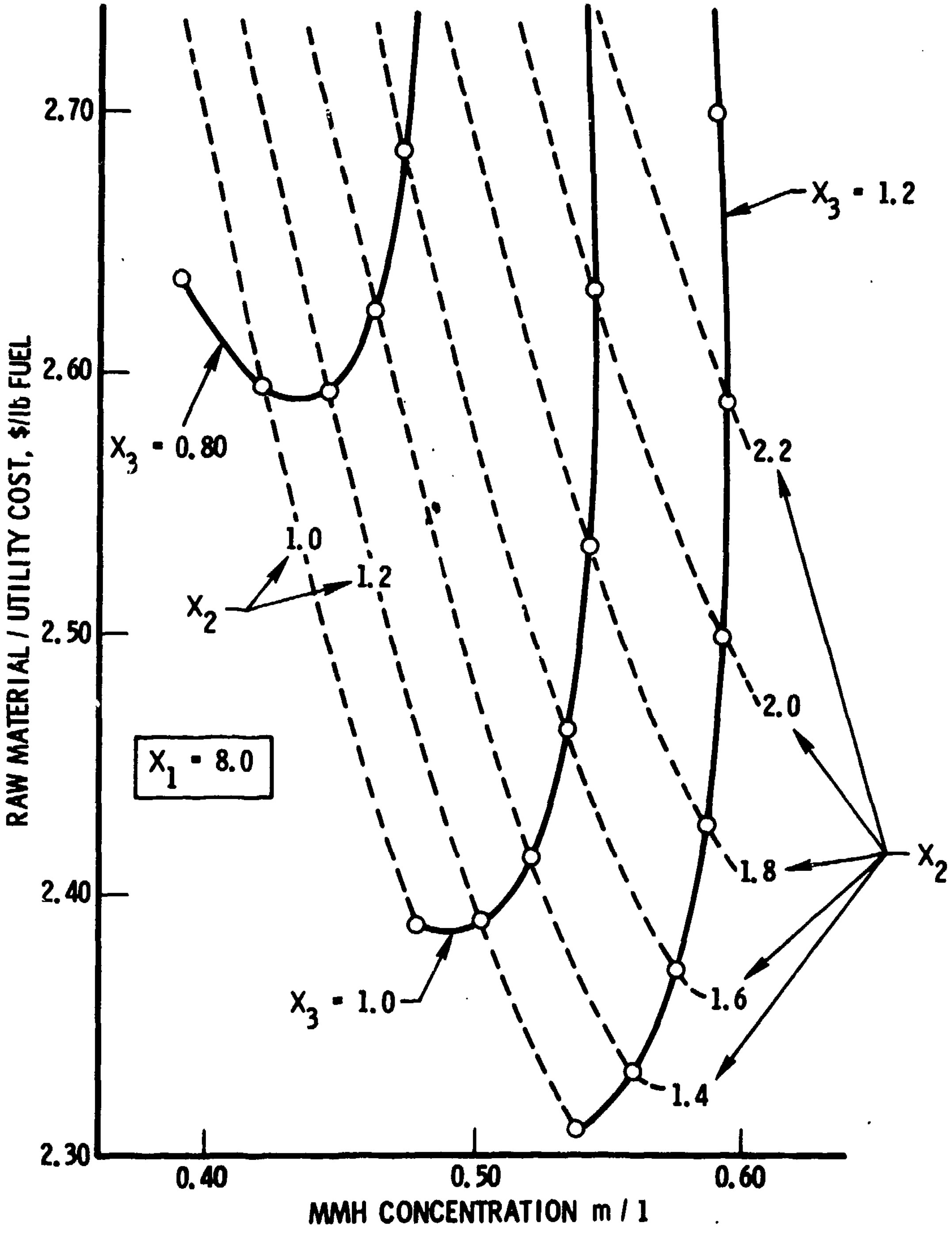

Figure 21. MMH Cost at Constant CA Condition

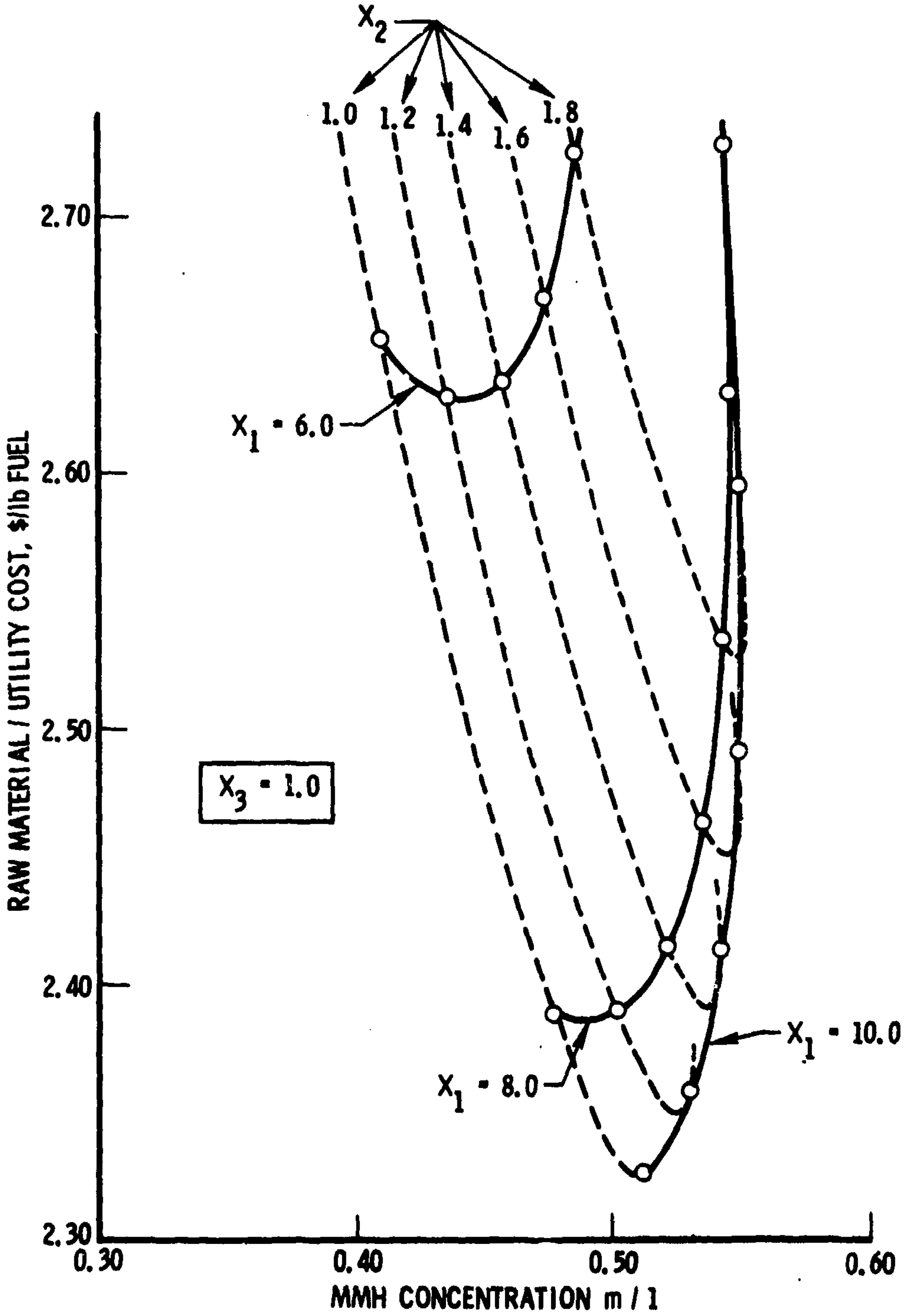

Figure 22. MMH Cost at Constant MMA Condition

Table 4. UDMH System, Process Condition, and Cost Breakdown

Item	Operating Parameter and Individual Cost, $/lb							
Process Condition								
NH_3/Cl_2	10	7	12	10	10	10	10	10
N_2/Cl_2	0	3	0	0	0	0	0	0
CA Yd., Y_2	0.596	0.606	0.642	0.596	0.596	0.596	0.596	0.596
DMA, X_1	4.5	4.5	4.5	3.5	5.0	4.5	4.5	4.5
NaOH, X_2	1.6	1.6	1.6	1.6	1.6	1.6	2.0	1.2
CA, X_3	1.4	1.4	1.4	1.4	1.4	1.0	1.4	1.0
UDMH, Y_3	0.839	0.839	0.839	0.733	0.875	0.676	0.857	0.628
UDMH Yd., Y_1	0.600	0.600	0.600	0.524	0.625	0.676	0.612	0.628
Raw Material Cost								
NH_3	0.148	0.137	0.141	0.169	0.142	0.131	0.145	0.141
Cl_2	0.231	0.228	0.215	0.264	0.222	0.205	0.226	0.220
N_2	00	0.116	00	00	00	00	00	00
DMA	0.322	0.322	0.322	0.329	0.322	0.325	0.319	0.338
NaOH	0.102	0.102	0.102	0.116	0.097	0.126	0.124	0.102
HCl	0.042	0.042	0.042	0.048	0.040	0.156	0.123	0.056
Total	0.845	0.946	0.822	0.927	0.823	0.943	0.938	0.857
Recycle Cost								
NH_3	0.184	0.111	0.216	0.210	0.176	0.163	0.180	0.175
DMA	0.093	0.092	0.095	0.078	0.101	0.123	0.090	0.134
H_2O	0.073	0.096	0.062	0.094	0.065	0.109	0.066	0.125
Total	0.349	0.300	0.370	0.382	0.342	0.395	0.336	0.434
Utility Cost	0.177	0.177	0.177	0.204	0.169	0.223	0.173	0.241
Grand Total	1.370	1.422	1.368	1.514	1.334	1.561	1.447	1.531

Table 5. MMH System, Process Condition, and Cost Breakdown

Item	Operating Parameter and Individual Cost, $/lb							
Process Condition								
NH_3/Cl_2	10	7	10	10	10	10	10	10
N_2/Cl_2	0	0	2	0	0	0	0	0
CA Yd., Y_2	0.596	0.498	0.663	0.596	0.596	0.596	0.596	0.596
MMA, X_1	9.0	9.0	9.0	7.0	11.0	9.0	9.0	9.0
NaOH, X_2	1.2	1.2	1.2	1.2	1.2	1.2	1.6	1.6
CA, X_3	1.0	1.0	1.0	1.0	1.0	0.8	1.0	1.4
MMH, Y_3	0.521	0.521	0.521	0.473	0.530	0.454	0.548	0.624
MMH Yd., Y_1	0.521	0.521	0.521	0.473	0.530	0.567	0.548	0.446
Raw Material Cost								
NH_3	0.222	0.257	0.196	0.244	0.218	0.203	0.211	0.259
Cl_2	0.347	0.416	0.312	0.382	0.341	0.318	0.330	0.405
N_2	00	00	0.105	00	00	00	00	00
MMA	0.420	0.420	0.420	0.403	0.453	0.438	0.407	0.404
NaOH	0.160	0.160	0.160	0.176	0.158	0.184	0.203	0.178
HCl	0.088	0.088	0.088	0.097	0.087	0.202	0.251	0.074
Total	1.236	1.340	1.281	1.302	1.256	1.345	1.402	1,320
Recycle Cost								
NH_3	0.276	0.200	0.250	0.304	0.272	0.254	0.263	0.323
MMA	0.323	0.323	0.323	0.271	0.393	0.375	0.306	0.263
H_2O	0.148	0.174	0.157	0.192	0.121	0.189	0.132	0.088
Total	0.747	0.696	0.730	0.768	0.786	0.818	0.700	0.674
Utility Cost	0.370	0.370	0.370	0.409	0.364	0.427	0.351	0.307
Grand Total	2.353	2.406	2.381	2.478	2.405	2.590	2.453	2.301

Contactor		UDMH	MMH
Amine (X_1), moles/liter	=	4.5	9.0
NaOH (X_2), moles/liter	=	1.6	1.2
CA (X_3), moles/liter	=	1.4	1.0

B. MODEL

The method used to estimate production cost evolved from a simple technique of estimating by the number of process steps (i.e., each process step was charged with a fixed gross cost) through a refined method[6] and finally to a detailed process cost analysis involving individual equipment costs, manpower costs, process chemistries, and operating costs. The final cost analysis accomplished for the Modified Chloramine Process is discussed below.

The process physical model was based on a nominal production rate of 2,000,000 lb year (400 lb/hr) equally divided between UDMH and MMH, to provide a "cost-per-pound" basis for the analysis. Costs were then defined to include the following major categories:

- Capital Costs
  - Equipment Costs
  - Installation and Construction Costs
  - Site Preparation Costs
- Operating Labor
- Maintenance
- Raw Materials/Utilities
- Miscellaneous
- Administrative Overhead
- Profit

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[6] John H. Perry, ed., Chemical Engineers Handbook, McGraw-Hill, New York, 1973; Section 25.

Each major category was further broken down to provide a realistic basis for predicting cost of production. The process cost analysis is summarized in Table 6. The derivation of the major category costs are described in Appendix B.

C. SUMMARY

The economic analysis, which is the overall evaluation of the feasibility of the chloramine process for the production of UDMH and MMH, indicates that this process would be satisfactory if there were no commercial alternatives. This analysis indicated the 1978 price of UDMH and MMH would be \$4.53 and \$6.12, respectively. It is estimated that these prices are accurate to ±20 percent. The price includes a 6-1/2-yr capital amortization. If the amortization were extended to 10 yr, the price would be reduced 28 cents to \$4.25 and \$5.84, respectively. If the cost of borrowing money at 10 percent were included in a 10-yr amortization, the price would increase 5 cents to \$4.58 and \$6.17 for the two fuels. A credit for ammonium chloride calculated at 6 cents a pound net (\$0.125/lb market price) would reduce the price of UDMH by about 20 cents a pound to \$4.38, and of MMH by about 30 cents a pound to \$5.87. No allowance for product purification losses was made. Costs would be increased by the percent loss - perhaps as much as 6 percent - to \$4.64 and \$6.22.

This analysis indicates that the modified chloramine process would be preferable to other alternatives considered by the Air Force (nitramine process for UDMH about \$15/pound, and methylation of hydrazine about \$8/pound UDMH and \$12/pound MMH). It is comparable to the estimated cost of UDMH from the nitrosamine process that only makes UDMH but offers the advantage that a carcinogenic intermediate is not used. The price is probably somewhat cheaper than the Raschig Process fuel which currently produces MMH at a price of \$6.73/lb. (The price of UDMH has not yet been established.)

Table 6. Variations in Amine Fuels Production Cost by the Modified Chloramine Process

	1978	1981
Fabricated Equipment	1,371,960	1,731,920
Process Machinery	1,241,980	1,721,630
Machinery/Equipment	534,990	700,480
Pumps/Valves	214,840	282,240
Electrical	697,870	886,460
Installation	2,648,160	2,961,660
Buildings	508,660	666,000
Siting/Facilities	1,966,340	2,199,120
	9,184,800	10,779,510

	1978		1981	
	MMH	UDMH	MMH	UDMH
Amortization	706,520	706,520	829,190	829,190
Operating Labor	749,720	749,720	905,630	905,630
Maintenance	177,160	177,160	213,990	213,990
Raw Materials*	2,046,000	1,230,000	2,798,200	1,682,200
Burden (40%)	818,400	492,000	1,119,300	672,900
Utilities	369,000	158,000	491,140	210,310
Miscellaneous	199,950	199,950	266,140	266,140
Production Cost*				
Total	5,066,750	3,713,350	6,623,550	4,780,310
$/lb	5.07	3.71	6.62	4.78
Admin O/H (24%)	348,600	298,000	432,100	364,700
Profit (13%)	704,000	521,500	917,200	668,850
Total Cost*	6,119,350	4,532,850	7,972,850	5,813,860
$/lb	6.12	4.53	7.97	5.81

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*1,000,000 lb/yr production rate

(Note: Does not include NH_4Cl credit)

IV. CONCLUSIONS

From the process development studies and the economic analysis, it is concluded that an alternative source of UDMH and MMH that is clean and economical could be produced by the modified Sisler chemistry investigated by the Martin Marietta Corp. It is recommended that only a contractor proficient in the manufacture of hydrazine should attempt such a project. The 40-lb/hr pilot-plant phase is not recommended unless a decision is made to proceed with a production plant. However, only the chloramine generator-precipitator needs scale up information.

APPENDIX A

ANALYSIS OF CHLORAMINE IN GAS STREAMS

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The analysis of chloramine (CA) in gas streams is complicated by its relative instability and also by the inevitable presence of other constituents in the gas streams (unreacted NH_3, by-products, etc.). Hence, if the yields from a chloramine generator are determined by the concentration of chloramine product, additional information on the composition of the gas stream is required as a function of the stoichiometry of both CA formation and loss mechanisms. Chloramine is formed by the reaction of ammonia with chlorine.

$$2NH_{3(g)} + Cl_{2(g)} \rightarrow NH_2Cl_{(g)} + NH_4Cl_{(s)} \qquad (A\text{-}1)$$

Three moles of gaseous reactants yield only one mole of gaseous product. The assumed decomposition pathway consumes five moles of gaseous reactants to produce one mole of gaseous product.

$$2NH_{3(g)} + 3NH_4Cl_{(g)} \rightarrow N_{2(g)} + 3NH_4Cl_{(s)} \qquad (A\text{-}2)$$

Presented are the details of sampling and analysis procedures that are designed to allow determination of the yield from a chloramine generator rapidly and with minimal interference. The entire process entails gas-phase sampling into a fixed, evacuated volume; extraction of the gas with water to dissolve CA, NH_3, and NH_4Cl; dilution of the solution to a known volume; and determination of the CA and unreacted NH_3 by spectrophotometry and acid-base titration, respectively. From the moles of CA and NH_3 in the sample and the ratio of NH_3 to Cl_2 input to the reactor, the yield can be computed if the reactions for production and loss are known.

$$\% \text{ CA yield} = \frac{E(R_i - 2.667)}{1 - 1.667\,E} \times 100 \qquad \text{(A-3)}$$

where

E = moles CA/(moles NH_3 + moles CA) from the analysis

and

R_i = moles NH_3/moles Cl_2 input to the reactor

The numbers in Eq. (A-3) were derived from the stoichiometry of (A-1) and (A-4). It is assumed that all Cl_2 is consumed by reaction in the presence of excess NH_3.

The analytical techniques for determination of chloramine and ammonia will be presented first, followed by a description of the sampling process.

A. ANALYSIS FOR CHLORAMINE

The quantitative analysis of chloramine produced by the gas-phase reaction of ammonia with chlorine has commonly been based upon either the iodometric titration of the product,[1] or the measurement of the amount of chloride (as the ammonium chloride) resulting from complete decomposition of the chloramine.[2] Both are viable analytical techniques, but each offers its own characteristic disadvantage.

For the analysis by iodometry, the gaseous chloramine is extracted with a suitable solvent (diethyl ether or water). An aliquot of the sample solution is then added to aqueous acidified potassium iodide and the liberated iodine is back-titrated with standard thiosulfate. The entire procedure must be performed with dispatch owing to the evident instability of chloramine

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[1] H. H. Sisler, R. M. Kren, and K. Utvary, Inorg. Chem., 8(9), 2007 (1969).

[2] H. H. Sisler, F. T. Neth, R. S. Drago, and D. Yaney, J. Am. Chem. Soc., 76, 3906 (1954).

with time[3] and to the gradual air oxidation of iodide to iodine in an acidic medium. Although this technique has been proven reliable, the requirement for routine preliminary work (preparation of reagents, standardization of the titrant, repeated cleaning of glassware) along with having to perform an expeditious titration for each of possibly many critical samples can be tedious, time-consuming, and not always convenient.

The determination by chloride yield involves the comparison of the initial weight of the chlorine entering the reactor with the weight of chlorine retained in the reactor as the ammonium chloride after decomposition of the chloramine in the presence of excess ammonia either as a condensate or in solution.[2] However, this technique is not only an indirect rather than direct analysis for chloramine, but also requires several hours (e.g., 12 hr) for complete decomposition to occur.

Spectrophotometry offers a potentially attractive means of analysis provided that the preliminary sample preparation is uncomplicated. A spectral scan requiring no more than a few minutes (3-5 min) from sample loading to the end of the scan can supply both qualitative and quantitative information. Light scattering due to the production of NH_4Cl in the gas stream as a smoke precludes in situ gas-phase spectroanalysis; however, the feasibility of a spectrophotometric technique for chloramine in solution has been verified by Kleinberg, Tecotzky, and Audrieth.[4]

The following is a suggested spectrophotometric means of analysis for gas-phase generated chloramine that is free of interference from NH_4Cl smoke.

B. CALIBRATION OF CA SPECTRA

Spectra were taken against a water reference (reference spectrum was not affected by NH_3, NH_4Cl) of analytes prepared by dilution with

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[3] L. F. Audrieth and R. A. Rowe, J. Am. Chem. Soc., 77, 4726 (1955).

[4] J. Kleinberg, M. Tecotzky, and L. F. Audrieth, Anal. Chem., 26 (8), 1388 (1954).

0.05 N NH_3 of parent chloramine solutions from the sampler (sampler is discussed later). These parent solutions were kept in an ice bath and shielded from light for the duration of the analyses. While an analyte was being scanned, concurrent iodometry was performed on another aliquot of the analyte for chloramine content. A 5 ml aliquot of the analyte was added to ~20 ml of acidified potassium iodide. Due to air oxidation of the iodide, an excess of KI (0.5 - 1.0 g) was added to the 0.1 N H_2SO_4 solution just prior to analyte addition. With a stirring bar, the titer was continuously mixed while standard thiosulfate was quickly added dropwise until the color of the solution faded to a pale yellow due to consumption of iodine. At this point several drops of starch indicator were added, producing a deep violet-blue hue due to complexation with residual iodine. Addition of thiosulfate continued until disappearance of the coloration.

$$\underset{\text{(clear)}}{NH_2Cl + 2H^+ + 3I^-} \rightarrow \underset{\text{(yellow)}}{Cl^- + NH_4^+ + I_3^-} \qquad \text{(A-4)}$$

$$I_3^- + \text{starch} \rightarrow \text{blue complex}$$

$$I_3^- + 2S_2O_3^= \rightarrow 3I^- + S_4O_6^= \qquad \text{(A-5)}$$

(disappearance of blue color)

$$NH_2Cl: S_2O_3^= = 1:2 \qquad \text{(A-6)}$$

All chemicals used were reagent grade. The starch indicator was made as prescribed by Vogel[5] using mercuric iodide as preservative. The thiosulfate solution was likewise prepared as described by Vogel using

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[5] A. I. Vogel, "A Textbook of Quantitative Analysis," 3rd. ed., 343-349, John Wiley and Sons, Inc. (1961).

sodium carbonate as preservative. The thiosulfate solution was standardized against iodine liberated by the reaction of iodate with iodide in dilute acid.

$$IO_3^- + 8I^- + 6H^+ \rightarrow 3I_3^- + 3H_2O$$

$$IO_3^- : S_2O_3^= = 1:6 \qquad \text{(A-7)}$$

KIO_3 was dried overnight in an oven at 110°C, and about 1 g of this was accurately weighed out and dissolved in preboiled distilled water to 100 ml. The solution was kept in a container away from light. A KI solution was made by dissolving ~6 g in 50 ml of preboiled distilled water. The KI was tested for iodate contamination by acififying and then adding starch solution. The absence of an immediate violet-blue coloration indicated the KI was iodate-free. A 5 ml aliquot of the KIO_3 solution was then mixed with ~10 ml of the KI solution in an Erlenmeyer flask. 5 ml of 2 N H_2SO_4 was gradually added with stirring. Titration with thiosulfate solution followed, using the starch solution as indicator.

For the titration of analytes giving absorbance readings less than 2, it was necessary to dilute the 0.1 N thiosulfate standard prescribed by Vogel. Excessively low volumes of thiosulfate titrant at 0.1 N concentration were required, resulting in the tendency toward inaccurate volumetric dispensing. The titrant solution was therefore diluted by a factor of 10 or 20 with solvent made up of 0.1 g Na_2CO_3 in 1 liter of preboiled distilled water.

For reliable quantitation it was necessary to maintain a sufficiently low pH in the iodide titer solution. Insufficient acidity in the iodide solution prevented complete reaction of the chloramine to produce iodine resulting in low concentration determinations. The iodide solutions were therefore made in 0.1 N H_2SO_4. It should be noted, however, that if the pH becomes too low, rapid air oxidation of iodide and poor indicator performance can occur. A test to determine the presence of unreacted chloramine after titration would be to add more dilute acid; the immediate appearance of the starch-iodine blue color would indicate that the titer was not initially acidic enough.

Figure A-1 shows the experimental calibration curve. The chloramine maxima occurs at 243 to 243.5 nm with $\epsilon = 453 \pm 6$ l/mole-cm, in good agreement with Kleinberg, et al.[4]

1. STABILITY OF THE CHLORAMINE SOLUTIONS

Both the spectral scan and iodometry performed on a sample required no more than 10 to 15 min. The reliability of the analyses depends upon the stability of the chloramine solutions.

a. Dependence on Time and Temperature

An aqueous chloramine sample kept at room temperature (~23°C) and protected from light showed a gradual decrease in absorbance readings of 0.079 ± 0.016 per hr (5 percent per hr) over a 3.3-hr period (initial absorbance = 1.7). However, another sample showed no decrease in absorbance within a 2.5 hr span while standing in an ice water bath.

b. Dependence on NH_4Cl and NH_3 Present in Solution

Working with a sample initially at pH ~10.2, as much as 1 g/100 ml of NH_4Cl was added. Although the pH decreased to 9.7 there was no apparent change from the original absorbance. Also, there was no change in absorbance when added to an equal amount of NH_3 solution at pH = 12. Finally, no change in absorbance was seen when both NH_3 and NH_4Cl were added.

c. Dependence on pH

The pH of a sample solution was varied with HCl or NH_3. At pHs between 5 and 11.6, only monochloramine was evident; at pHs between 1.7 and 4, only dichloramine appeared to be present (λ_{max} ~294 nm);

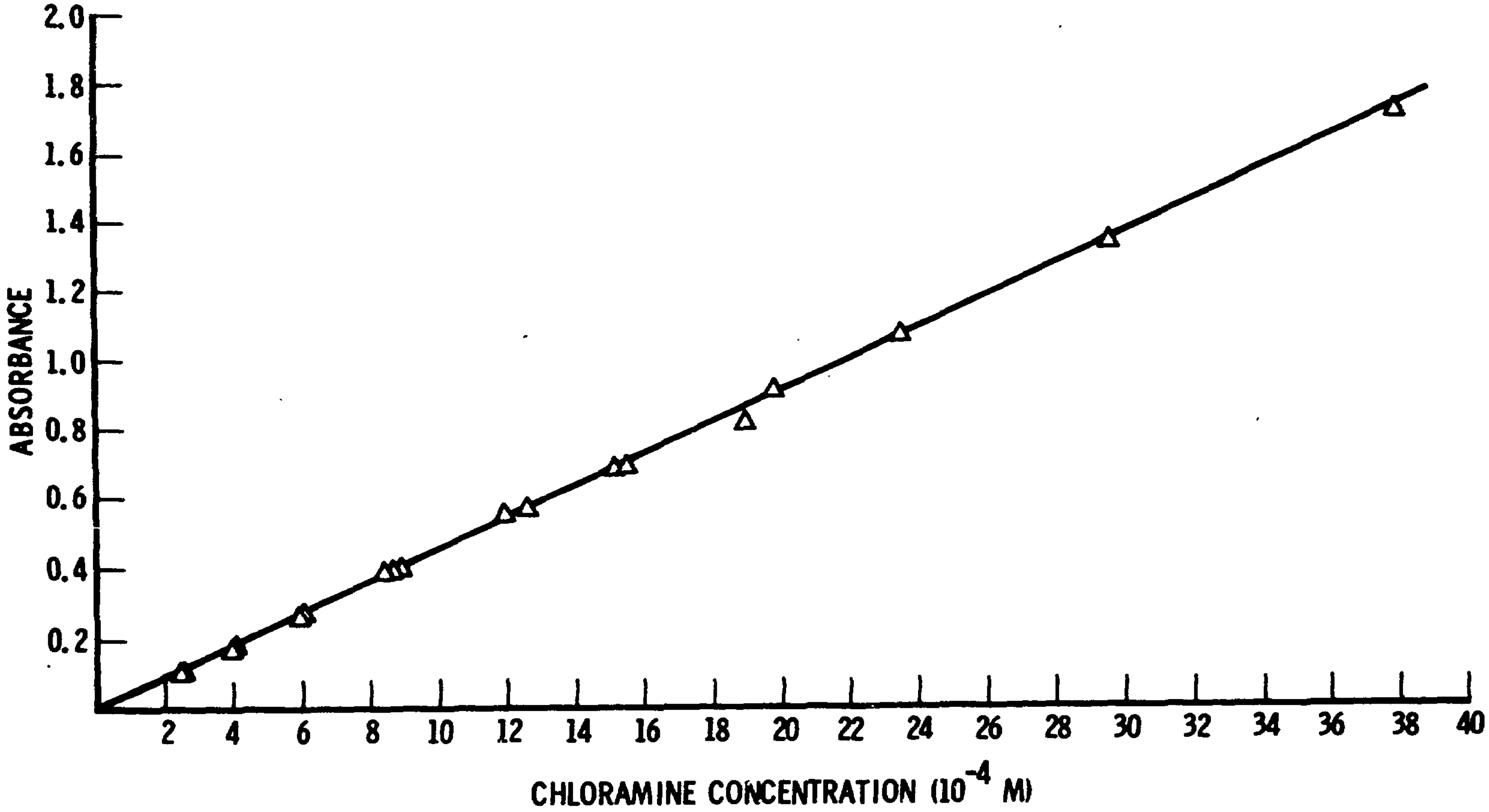

Figure A-1. Absorbance vs Concentration of Aqueous Chloramine at 243-243.5 nm 1 cm path length, $\epsilon = 453 \pm 6$ ℓ/mole-cm and at pHs of 0.4 to 0.8, the trichloramine appeared ($\lambda_{max} \sim 330\text{-}340$ nm).[6] The spectra are shown in Figure A-2.

From the above observations the following conclusions are made:

- The aqueous samples have moderate stability at room temperature. Analysis should be performed within 15 to 30 min of sampling to keep losses to less than ~2 percent. If a sample is to be stored for longer periods, it should be cooled to 0-5°C. Under these conditions the absorbance and iodometric quantitation of the sample should remain virtually unchanged for at least 2 hr.
- Variation in the amounts of NH_4Cl or NH_3 have little or no effect on the absorbance of aqueous chloramine. Additionally, no spectral interferences due to NH_4Cl or NH_3 occur.
- Aqueous samples from the chloramine generator have exhibited pHs between 10.2 and 10.6, presumably by the buffering action of the NH_3 and NH_4Cl also present. This pH range is favorable to the existence of the one chloramine in solution; i.e., monochloramine will not favorably decompose to the di- or tri-chloramine in this pH range. Also, a slight increase in pH upon dilution with 0.05 N NH_3 or decrease by dilution with water will not affect the monochloramine in solution.

C. ANALYSIS FOR AMMONIA

Ammonia is a weak base ($pK_b \sim 10^{-5}$) with an equivalence point which occurs near pH = 5. Hence, a suitable indicator for ammonia titration against acid is bromcresol green. For analysis, an aliquot (ca. 20 ml) was withdrawn from a sample solution and dispensed into a flask with 3 to 5 drops of bromcresol green indicator solution (0.1 % w/v in 1:1 ethanol-water). Titration ensued against 0.1 N HCl solution prepared by dilution of commercial vials of standard HCl. The end point was indicated by the color change from light blue to straw yellow.

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[6] W. S. Metcalf, J. Chem. Soc., 148, 1942.

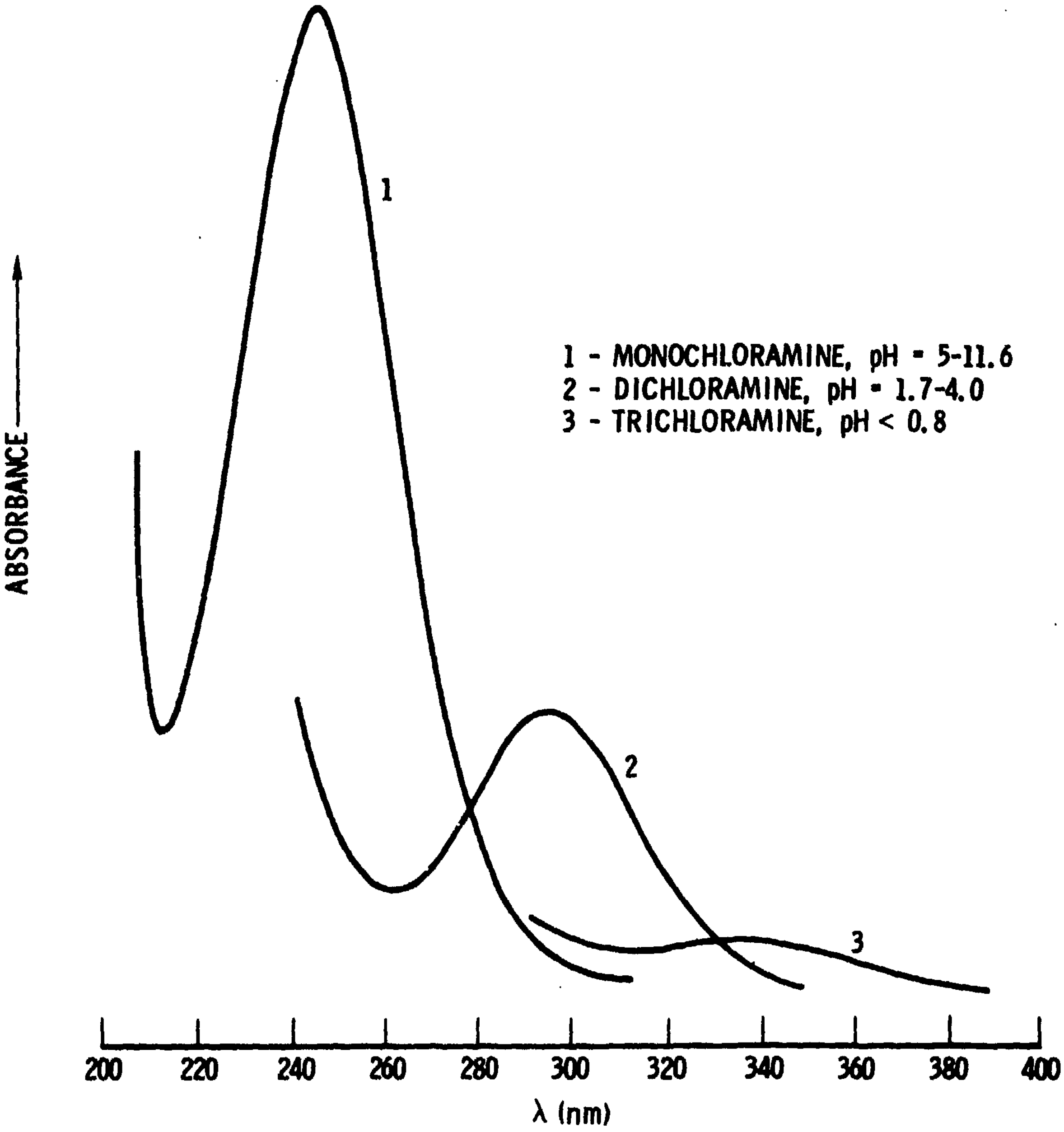


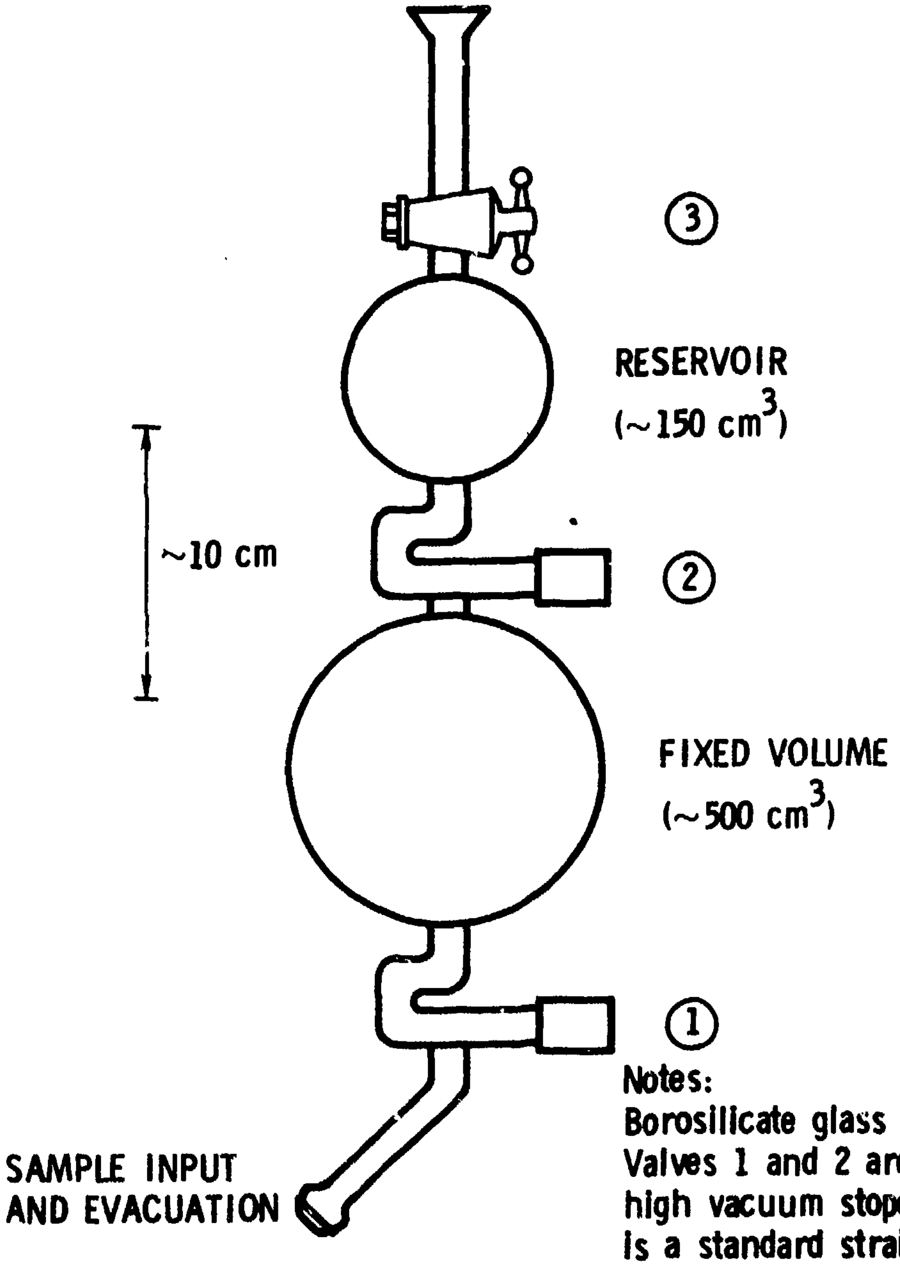
Figure A-2. Absorbance Spectra of the Chloramines in Aqueous Solution

D. SAMPLING OF THE CA REACTOR GAS STREAM

A sampling system was devised to accommodate subsequent analysis with minimal loss of chloramine and without interference by the presence of NH_4Cl smoke. A sampler was designed to acquire an anhydrous sample of fixed volume from the reactor gas stream (the volume need not be known (Eq. (A-3)); to rapidly dissolve the NH_2Cl, NH_3, and NH_4Cl in water; and to readily deliver that sample solution to the analysis procedure.

The sampler is shown in Figure A-3. Since the instability of chloramine in contact with glass or NH_4Cl at room temperature is not indicated in the scientific literature (excluding some ambiguous patent disclosure statements), no provision for the heating of sampler chambers or for NH_4Cl particle filtration was included. Borosilicate glass was chosen to facilitate observation and cleaning. The valves (1 and 2) isolating the fixed volume are of the Teflon high-vacuum type with Teflon-to-glass seating surfaces (Viton O-ring seats are acceptable). These valves were necessary to maintain vacuum between evacuation and sampling. Valve 3 which isolates the water reservoir is a standard Teflon-plug 6 mm stopcock; it only serves to maintain sufficient vacuum to load the water and to isolate the system during extraction. The fitting through which the sample volume is evacuated and also through which the sample is introduced will depend on the application. The sampler used here was fitted with an 18/9 O-ring ball joint.

The sampling procedure is as follows: All stopcocks, valve plugs, and O-rings are removed from the sampler (no grease is used) and cleaned. The glass sampler is thoroughly rinsed and dried in an oven (~110°C). The sampler is reassembled and attached to a water aspirator. After partial evacuation all valves are closed. The water reservoir is filled with approximately 125 mil water by inverting over a beaker of deionized water and then opening the stopcock (3). The fixed volume is then evacuated to less than 1 Torr using a mechanical pump. The prepared sampler is placed on a reactor port and a sample is collected by opening valve 1 to the fixed



Notes:
Borosilicate glass construction.
Valves 1 and 2 are ≥ 5 mm Teflon high vacuum stopcocks. Valve 3 is a standard straight-bore 6 mm Teflon stopcock.

Figure A-3. Chloramine Gas Sampler (~1:2.5)

volume. After the pressure has equilibrated between the gas stream and the fixed volume, the valve is closed. With the sampler in near-vertical orientation, valve 2 is opened to admit the water to the fixed volume. The sampler is shaken vigorously and the vacuum is broken by opening valve 3. A funnel is placed in a 250 ml volumetric flask and the contents of the sampler are drained into it through valve 1. The sampler is rinsed twice by closing valve 1 and adding ~20 ml water through valve 3 from a wash bottle. The sample is immediately made up to volume with deionized water.

E. STABILITY OF GASEOUS CHLORAMINE

Preliminary tests were performed to determine the stability of chloramine samples in the gaseous phase. Samples generated under identical initial reactor conditions produced results that agree within 2 percent in CA yield. In another experiment, three samplers were connected to the reactor at approximately the same position in the gas stream. Samples were then taken simultaneously. One sample was immediately extracted into water; another sample was extracted after 5 min; and the third sample was extracted after 15 min. Each sample was analyzed just after extraction. The first two sample yields were indistinguishable within experimental error: 2.68×10^{-4} mole CA (24.7 percent yield) for the first, and 2.71×10^{-4} mole CA (25.4 percent yield) for the second. The third sample analyzed at 2.52×10^{-4} mole CA (23.3 percent yield) after 15 min of standing as a gas. On the basis of data from this third sample, a decomposition rate of 0.4 percent per minute for gaseous chloramine is estimated.

The design and operation of the sampler appears adequate. Samples are readily recovered and analyzed. Although more work on the stability of gaseous chloramine must be done, the gas phase stability appears adequate for convenient sampling. However, it is recommended that the gas samples be extracted as soon as possible and at least within 5 min after the sample is generated.

F. ATTACHMENTS

Attachments A1 through A4 to this Appendix present step-by-step directions for the following analyses: (1) Sampling of the reactor gas stream; (2) Chloramine analysis; (3) Iodometric analysis for chloramine; and (4) Analysis of NH_3 and CA yield.

Attachment A.1 - Sampling of the Reactor Gas Stream

(See Figure A-3)

1. Partially evacuate the reservoir and fixed volume of a cleaned and dried sampler. Shut all valves.

2. Fill the reservoir with 100 to 125 ml deionized water by immersing the reservoir inlet in a beaker of water and opening stopcock 3.

3. Complete the evacuation of the fixed volume with a mechanical vacuum pump.

4. Attach the sampler to the reactor and open valve 1. Close valve 1 after the pressure has equilized.

5. With the reservoir above the fixed volume in a vertical orientation, open valve 2 and shake vigorously. This should be done within 5 min of sampling.

6. Open valve 3 to equalize the pressure and transfer the solution to a 250 ml volumetric flask through valve 1.

7. Rinse the sampler twice by shaking with approximately 20 ml of water and transfer the rinse to the volumetric flask.

8. Fill the volumetric flask to volume and mix.

Attachment A2 - Chloramine Analysis

1. Withdraw enough sample solution to fill a 1.0 cm path length uv-grade cuvette.

2. Place sample into a spectrophotometer and take the sample's absorption spectra from 200 nm to 400 nm relative to a water reference in a matched cell.

3. If the absorbance is greater than the range of the instrument, dilute by a factor of 4 with water and remeasure (dilutions can be conveniently accomplished directly in the cuvette with the use of a 1.0 ml automatic pipette with disposable tips).

4. Determine the CA absorbance at λ_{max} = 243 to 243.5 nm

5. Calculation for moles of CA:

   (moles of chloramine) = (Absorbance/453) × (volume of sample volumetric flask in liters)

   For a 250 ml volumetric flask,

   (moles of chloramine) = (Absorbance) $\times 5.52 \times 10^{-4}$

Attachment A3 - Iodometric Analysis for Chlormaine

1. To ~20 ml of 0.1 N H_2SO_4 in an Erlenmeyer flask, add 0.5 to 1.0 g KI. Immediately after the addition of the KI, add a measured aliquot (5-10 ml) of sample solution.

2. With the aid of a magnetic stirring bar, continuously mix the flask contents for the duration of the titration.

3. Quickly add dropwise from a buret, standard thiosulfate solution (0.005-0.01 N) until the solution fades to a pale yellow.

4. Add ~0.5 ml of starch indicator (the solution should turn into a violet-blue color).

5. Continue the addition of thiosulfate until the solution changes from blue to clear.

6. Calculation of chloramine concentration:

$$(\text{CA conc.}) = \frac{(S_2O_3^{=}\ \text{conc.}) \times (\text{volume } S_2O_3^{=}\ \text{in ml})}{2(\text{volume sample aliquot in ml})}$$

Attachment A4 - Analysis of NH_3 and CA Yield

1. Transfer an accurately known volume of the sample solution into an Erlenmeyer flask (a 20 ml aliquot is sufficient).

2. Add 3 to 5 drops of bromcresol green indicator and titrate with 0.1 N HCl until the indicator changes from blue to straw yellow.

3. Calculation for moles of NH_3:

   (moles of NH_3) = (conc. HCl) × (volume HCl in liters) × (volume of sample volumetric flask)/volume of sample titrated.

   For a titration using 0.1 N HCl titrant, and a 20 ml aliquot from a sample solution originally made up to 250 ml in a volumetric flask,

   $$(\text{moles of } NH_3) = (\text{vol. HCl in ml}) \times 1.25 \times 10^{-3}$$

4. CA yield:

   $$E = \frac{(\text{moles of chloramine})}{(\text{moles of } NH_3 + \text{moles of chloramine})}$$ in the product sample

   $$R_i = \frac{(\text{moles/min of } NH_3)}{(\text{moles/min of } Cl_2)}$$ of input reactants

   $$\text{Yield } (\%) = \frac{E(R_i - 2.667)}{1 - 1.667\,E} \times 100$$

APPENDIX B

CAPITAL AND MISCELLANEOUS COSTS

APPENDIX B

CAPITAL AND MISCELLANEOUS COSTS

Following the precedent established by the agreement between AF Logistics Command and Teledyne McCormick-Selph, the total capital costs were amortized in 5-1/2 years. Nothing was allowed for the cost of money.

A. SITE ACQUISITION

No cost was assessed for purchase of land for plant siting due to the extreme variation in land cost across the nation. For the most part, a major chemical company would build on its own property and Government construction could easily be accommodated at a number of Department of Defense sites, obviating land related costs.

B. OPERATING LABOR

Operating labor costs were estimated on the basis of labor rates for skilled and technical personnel derived from the California State Department of Labor. Base rates were increased by a 100 percent overhead burden to account for shift differentials, accounting and administrative overhead, and related factors. Manning levels were estimated based on departmentalizing the plant operation; i.e., the manpower structure is intended to reflect an independent operation within a parent organization and incorporates essential functional elements from general management through routine maintenance and clerical staff (Table B-1).

C. EQUIPMENT COSTS

Equipment was broken down into subcategories as follows:

Fabricated Equipment
Process Machinery
Other Machinery/Equipment

Table B-1. Integral Plant Manpower

		Number	Shifts	Annual	Sub Totals
Plant Manager	30, 000	1	1	30, 000	
Clerical	10, 000	1	1	10, 000	
					40, 000
Production Supervisor	30, 000	1	1	30, 000	
Shitf Supervisor	20, 000	1	2	40, 000	
Operators	18, 000	3/2	1/2	126, 000	
Technicians	15, 000	3	3	135, 000	
Maintenance	15, 000	2	3	90, 000	
					421, 000
Materials/Scheduler	20, 000	1	1	20, 000	
Quality Control	20, 000	1	1	20, 000	
Lab Tech	15, 000	1	3	45, 000	
					85, 000
Support Supervisor	25, 000	1	1	25, 000	
Admin Clerk	10, 000	2	1	20, 000	
Janitorial	10, 000	1	1	10, 000	
Medical Nurse	18, 000	1	1	18, 000	
Technicians	14, 000	1	3	42, 000	
					115, 000
				Sub-Total	661, 000
				O/H (100%)	661, 000
				Total	1, 322, 000

Pumps, Valves
Electrical Equipment
Buildings

Baseline cost data by equipment item were extracted from various sources, including Martin Marietta Aerospace, Naval Ordnance Station, Teledyne-McCormick-Selph, and where no specific data were available, by equipment similarity. An item/category cost inflation factor was derived from industry data for the 1975 to 1977 period and used to adjust 1976 baseline cost data to present and future levels (Table B-2).

D. INSTALLATION COSTS

Installation costs were estimated by characterizing various detailed tasks, scheduling the tasks and estimating completion time. Labor levels and costs were determined for each task, and an overhead rate of 100 percent was applied. A rental charge for heavy equipment was assumed and a 10 percent contingency charge was added (Table B-3).

E. SITING/SITE PREPARATION

Siting and site preparation were estimated by preparing a plant layout drawing based on assumed square-feet-per-process unit estimates. The plant layout included equipment layout, access roads, underground waterlines, drains, diking, storage area revetments, and a control room/ maintenance building layout. Based on the layout drawing, concrete slab and paving areas were calculated and linear measurements for water systems, drains, and fencing were taken. Costs were calculated from base rates supplied by Martin Marietta Aerospace for similar tasks. Survey and grading/fill costs were estimated (Table B-4).

F. MAINTENANCE

The design life of the plant was taken as 20 yr; i.e., over the 20-yr period, 90 percent of all equipment items were assumed to have either been replaced or to have required major refurbishment equivalent to the original

Table B-2. Equipment Cost Estimate

Item Description	Number	Unit Cost	Total
Tanks			
Amine, 15, 000 gallon	2	30, 000	60, 000
Caustic, 15, 000 gallon	1	30, 000	30, 000
Chlorine, 15, 000 gallon	1	30, 000	30, 000
Ammonia, 15, 000 gallon	1	30, 000	30, 000
Water, 25, 000 gallon	1	20, 000	20, 000
Surge, 10, 000 gallon	1	22, 000	22, 000
Product, 15, 000 gallon	2	30, 000	30, 000
			222, 000
Columns			
Stripping	1	100, 000	100, 000
Evaporator	1	60, 000	60, 000
Separator	2	20, 000	40, 000
Azeotrope/Concentrator	1	45, 000	45, 000
Finishing	1	70, 000	70, 000
Caustic Concentrator	1	30, 000	30, 000
Crystallizer	1	70, 000	70, 000
			415, 000
Pumps			
Typical	55	1, 500	75, 000
Conveyor/Hopper Assy	2	15, 000	30, 000
Boilers			
Heavy Duty	5	10, 500	52, 500
Light Duty	5	6, 500	32, 500
Condensers			
Typical	10	3, 500	35, 000
Heat Exchangers			
Typical	6	3, 000	18, 000
Mixers			
Caustic/H20	1	20, 000	20, 000
Neutralizer	1	6, 000	6, 000
Preheaters			
Ammonia	1	10, 000	10, 000
Chlorine	1	5, 000	5, 000

Table B-2. Equipment Cost Estimate (Continued)

Item Description	Number	Unit Cost	Total
Vaporizer Assy			
Ammonia	1	8,000	8,000
Chlorine	1	8,000	8,000
Filters			
Salt	2	25,000	50,000
Reactor Assy			
Reactor	1	78,000	78,000
Heat Ex	1	9,800	9,800
Generator/Precipitator	1	500,000	500,000
Cooling Assy's			
Cooling Tower	1	135,000	135,000
Chiller, 500 Ton	2	52,000	104,000
Refrig 250 Ton	1	485,000	485,000
Valves		90,000	90,000
Sub-Total			2,381,000
Instrumentation/Control (25%)			595,000
Installation			2,457,813
Total			5,433,813
Optional Add-On Costs			
Rail Service Facility			
Equipment, Rail	150,000		
Equipment, Service	150,000		
Siting	300,000		
Total (Est)		$650,000	
Drum Service Facility			
Equipment, Service	75,000		
Siting	110,000		
Total (Est)		$185,000	
Total Equipment			5,808,813
Total Siting			2,240,000
A&E Service (10%)			685,000
Total			8,733,813

Table B-3. Installation Cost Estimate Breakdown

Task	Detail
Site Prep	Grading; leveling; road-bed prep; revetments cooling pond; rail-line prep; concrete-work
Installation	Structural Steel (Sub-structures/overheads)
	Process Equipment Emplace Interconnect
	Control Equipment Emplace Interconnect
	Non-Process Plumbing FIREX/Fire Safety Equipment Air Supply Stations
	Instrumentation
	Electrical Support Equipment Process Equipment
	Support Buildings Erect/Elect/Plumb Finish

- Structural Steel
  - Install sub-structures, equipment supports, overheads; field welding, lay-out
    - Rigging & Assembly
    - Lay-out
    - Scheduling
    - Material (Non-Structural)
    - Heavy Equipment (Rental)(Crane)
    - Labor
      - Skilled
      - Unskilled
      - Supervision

Table B-3. Installation Cost Estimate Breakdown (Continued)

- Process Equipment
  - Emplace
    - 25 Major Assemblies
      - 15 Columns & Equivalent
      - Cooling Towers
      - Electrical Substation
      - Carbon Units
      - Refrigeration Plant
      - Supporting Assemblies
    - 50 Minor Assemblies
      - Hold/Surgetanks
      - Filter Units
      - Conveyors
      - Pumps
  - Interconnect
    - Riggers
    - Assemblies (Pneumatic, hydraulics)

- Control Equipment (Control Room)
  - Data Display/Record
  - Control Computer Console
  - Manual Control Console
  - Material Processing Controls (Product Raw Material)
  - Install/Interconnect

- Non-Process Equipment
  - Environmental/Occupational Monitors
  - Safety Equipment
    - FIREX
    - Fire
    - Air Supply
    - Equipment Stations
    - Safety Showers

- Instrumentation (Install/Interconnect)
  - Process Control
  - Environmental/Occupational

Table B-3. Installation Cost Estimate Breakdown (Continued)

- Electrical (Hook-up)
    - Support Equipment
    - Process Equipment

- Support Buildings
    - Erect
    - Plumb & Wire
    - Finish

1. Structural - 6 months

    1-1/2 months pre-installations (33 days × 8 = 264 hours)
    4-1/2 months installation (99 days × 8 = 792 hours)

2. Process Equipment - 9 months

    4 mos - major assemblies (88 × 8 = 704)
    3 mos - minor assemblies (66 × 8 = 528)
    2 mos - interconnect (44 × 8 = 352)

3. Control Equipment - 3 months (66 × 8 = 528)

4. Non-Process Equipment - 4 months (88 × 8 = 704)

5. Instrumentation - 3 months (66 × 8 = 528)

6. Electrical - 6 months (132 × 8 = 1056)

7. Support Buildings - 3 months (66 × 8 = 528)

Structural		
Super - Job × $60,000		$ 60,000
Foreman - 4 × 20,000		80,000
Welders - 4 × 8.50 × 1056		34,904
Riggers - 4 × 7.50 × 1056		31,680
Layout - 2 × 7.000 × 1056		14,784
Heavy Equipment - 2 × 15.00 × 1056		31,680
Labor - 10 × 4.00 × 1056		42,240
	Cum. Total	$ 296,288

Table B-3. Installation Cost Estimate Breakdown (Continued)

Process Equipment		
Super - Job × 60, 000		$ 60, 000
Foreman - 5 × 20, 000		100, 000
Installer - 20 × 8.50 × 1232		209, 440
Mechanic - 5 × 7.50 × 600		22, 500
	Cum. Total	$ 688, 228
Control		
Foreman - 2 × 20, 000		40, 000
Install - 4 × 8.50 × 528		17, 952
	Cum. Total	$ 746, 180
Non-Process Equipment		
Foreman - 2 × 20, 000		40, 000
Install - 6 × 7.50 × 704		31, 680
Lay-out - 2 × 7.50 × 704		10, 560
	Cum. Total	$ 828, 420
Instrumentation		
Foreman - 1 × 20, 000		20, 000
Technicians - 4 × 9.50 × 528		20, 064
	Cum. Total	$ 868, 484
Electrical		
Foreman - 2 × 20, 000		40, 000
Technicians - 6 × 10.50 × 1056		66, 528
Technicians - 4 × 10.50 × 528		22, 176
	Cum. Total	$ 997, 188
Total Labor		$ 997, 188
Overhead (100%)		997, 188
Heavy Equipment Rental ($20, 000/mos × 12 mos)		240, 000
Sub Total		2, 234, 376
Contingency (10%)		223, 437
Total		$2, 457, 813

Table B-4. Facility/Site Preparation

Concrete Work				
Slabs				
Raw Material Areas				
1' × 60' × 60'	=	3,600 ft^2 @ \$3.50	\$ 12,600	
Product Storage				
1' × 30' × 40'	=	1,200 ft^2 @ \$3.50	4,200	
Plant				
1' × 100' × 120'	=	12,000 ft^2 @ \$3.50	42,000	
Control Center				
0.5" × 50' × 80'	=	4,000 ft^2 @ \$3.50	14,000	
				\$ 72,800
Retaining Walls				
Raw Material Area				
1' × 12' × 180	=	180 @ \$7.00	1,260	
1' × 3' × 60	=	60 @ \$7.00	420	
Product Storage				
1' × 12' × 100'	=	100' @ \$7.00	700	
1' × 3' × 40'	=	40' @ \$7.00	280	
Plant				
1' × 3' × 960	=	960' @ \$7.00	6,720	
Control Center				
1' × 8' × 100'	=	100' @ \$7.00	700	
				\$ 10,080
Asphalt Paving				
30,000 ft^2 @ \$2.50				\$ 75,000
Cooling Pond				
Excavation, 400 yds^3 @ \$80			32,000	
Lining, Gunite, Steel			90,000	
				\$ 122,000
Perimeter Drain				
1' × 1' × 500', Channel @ \$10				\$ 5,000
Perimeter Fence				
8' × 4000', Chain Link, @ \$10				\$ 80,000
Sub-Surface Drain				\$ 50,000
Fire-Hydrant/Fire-Ex System				\$ 110,000

Table B-4. Facility/Site Preparation (Continued)

Control Center		
Building, Pre-Fab (50' × 80')	$120,000	
Quality Control Lab	100,000	
Medical Facility	75,000	
Office Equipment	75,000	
Maintenance Equipment	50,000	
Installation (100%)	420,000	
		$ 840,000
Site Preparation		
Survey	65,000	
Grading/Fill	400,000	
		$ 465,000
Total		$1,830,000

item cost. Twenty-five percent of the maintenance cost for the first 5 years was used as a baseline inflated at 6 percent per year. The annual cost was calculated and a 15 percent contingency factor added (Table B-5).

G. MISCELLANEOUS

Other costs include clean-up of salt stream and caustic streams and rail car rental (Table B-6).

H. RAW MATERIALS/UTILITIES

A 40 percent burden is added to cover the drive contractor's cost of handling purchased materials.

I. ACCURACY

An attempt was made to determine the validity of the model by applying it to the Teledyne-McCormick-Selph production plant. The model predicted a UDMH production cost of around $4.85/lb compared to the Teledyne-McCormick-Selph cost estimate of $4.65. However, the model represents a hybrid of several types of estimating methods. Thus the accuracy probably lies somewhere in the range of the "budget estimate" method, or ±20 percent.

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Table B-5. Maintenance Cost

Maintenance Cost Basis

25% replacement over 5 year period

$1, 037, 475

At 6% cost growth rate

$1, 388, 375

Average cost per year

$277, 675

Cost per fuel per year

$138, 840

Add Misc. Annual Maintenance (15%)

Total Maintenance Cost	<u>$177, 160</u> (1978)

Table B-6. Byproduct Purification Cost

Carbon Clean-Up
Remove 50 lb/hr contaminants from two process streams

Salt Stream
3 lb contamination in 3500 lb salt-H_2O
100% duty cycle
40% loading
7.5 lb/hr carbon

Use: 1500 lb carbon unit
2 × 750, regenerate twice a week

Initial carbon cost: 1500 × $0.30			$450.00
Regeneration			
Steam: 1500 × $1.00/1,000		$1.50	
Utility: 150 kW-hr × 0.01		1.50	
Manpower: 2 man days	$40	80.00	
Carbon make-up 75 lbs	$0.30	22.50	
Equipment Cost			
Carbon Assy		$130,000	
Support Equipment		31,000	
		$161,000	
Depreciation		$3.65/hr	
Cost per hour		$4.28/hr	
Cost per lb fuel		0.01	

APPENDIX C

REDUCED TOP HAT DATA

APPENDIX C

REDUCED TOP HAT DATA

Run 1

Condit & Sample		$NH_3/N_2/Cl_2$[b]	Concentration, m/l				Yield, %	
			MMA (X_1)	NaOH (X_2)	CA (X_3)	MMH (Y_3)	MMH[c] (Y_1)	CA[d] (Y_2)
A	1	8/2/1	8.70	0.84	0.66	0.343	51.90	65.82
	2		8.60	0.99	0.67	0.358	53.69	66.42
	3		8.42	0.99	0.67	0.367	54.65	66.89
	4		8.61	1.08	0.67	0.352	52.38	66.71
								66.46
B	1	12.2/2/1	7.95	1.02	0.61	0.343	56.63	62.89
	2		7.29	1.08	0.67	0.367	55.16	69.38
	3		7.40	1.07	0.70	0.380	54.59	73.12
	4		7.39	0.85	0.64	0.343	53.75	65.58
								67.74
C	1	8/0/1	8.35	0.93	0.59	0.358	60.44	60.19
	2		8.40	0.86	0.53	0.343	65.23	53.05
	3		8.53	0.88	0.56	0.330	59.44	56.03
	4		7.71	0.98	0.58	0.343	58.71	59.20
								57.13
D	1	8/4/1	8.48	1.28	0.55	0.343	62.81	66.40
	2		8.42	0.93	0.50	0.313	62.97	60.03
	3		9.29	0.86	0.53	0.343	64.37	64.80
	4		9.15	1.00	0.52	0.326	62.84	62.27
								63.38
D	1	8/2/1	8.04	1.05	0.59	0.326	55.18	57.12
	2		7.80	0.95	0.62	0.362	58.47	60.93
	3		8.25	0.92	0.61	0.380	62.23	60.37
	4		8.10	0.98	0.60	0.362	60.85	57.97
								59.10

[a] Cone temp. 560°F

[b] Cl_2 flow of 37.5 g/mm except condit D which was 30 g/min

[c] Based on CA

[d] Based on Cl_2 flow

Run 2a

Condit & Sample #		$NH_3/N_2/Cl_2$[b]	Concentration, m/l				Yield, %	
			MMA	NaOH	CA	MMH	MMH[c]	CA[d]
A	1	10/3/1	2.91	2.18	0.53	0.418	78.14	59.66[e]
	2		2.89	2.21	0.61	0.444	72.62	67.90
	3		2.83	2.19	0.57	0.411	71.57	63.81
	4		3.43	2.32	0.61	0.493	80.96	69.72
	5		3.64	2.11	0.58	0.519	90.10	65.97
								66.85
B	1	10/1/1	3.27	2.38	0.55	0.384	69.64	62.50
	2		3.26	2.32	0.55	0.384	70.32	61.81
	3		3.32	2.36	0.55	0.391	70.95	62.87
	4		3.34	2.31	0.57	0.391	68.16	65.75
	5		3.07	2.22	0.56	0.378	67.74	63.36
								63.26
C	1	6/3/1	2.94	2.52	0.56	0.329	58.85	58.45
	2		3.26	2.64	0.53	0.349	65.52	56.21
	3		3.05	2.53	0.54	0.343	63.36	56.65
	4		3.20	2.49	0.54	0.343	63.23	56.99
	5		3.33	2.44	0.57	0.363	63.23	60.28
								57.72
D	1	6/1/1	3.48	2.40	0.52	0.343	66.30	54.16[e]
	2		3.26	2.50	0.42	0.296	70.72	43.40
	3		3.23	3.19	0.41	0.243	59.36	41.66
	4		3.61	2.64	0.44	0.323	73.84	45.43
	5		3.65	2.70	0.44	0.296	66.93	45.83
								44.08
E	1	8/2/1	3.14	2.63	0.56	0.364	65.32	60.10
	2		3.18	2.35	0.56	0.391	69.29	61.18
	3		3.26	2.25	0.49	0.336	68.73	52.17[e]
	4		3.40	2.22	0.53	0.391	73.32	57.58
	5		3.23	2.20	0.54	0.378	70.56	57.77
								59.16

[a] Cone temp. 620°F except condit E which was 680°F

[b] Cl_2 flow of 37.5 g/min

[c] Based on CA

[d] Based on Cl_2 flow

[e] Not included in the average

Run 3a

Condit & Sample #		$NH_3/N_2/Cl_2^b$	Concentration, m/l				Yield, %	
			MMA	NaOH	CA	MMH	MMH[c]	CA[d]
A	1	10/1/1	1.86	1.82	0.82	0.444	54.19	49.14
	2		1.89	2.04	0.97	0.458	47.09	59.04
	3		1.91	1.99	0.88	0.438	50.00	52.11
	4		2.08	2.05	0.93	0.506	54.53	56.50
	5		2.00	2.04	0.89	0.512	57.65	53.93
								54.14
B	1	10/3/1	1.79	2.18	1.00	0.478	47.63	59.01
	2		1.86	2.36	0.98	0.486	49.36	58.54
	3		1.94	1.90	1.13	0.478	42.37	68.81[e]
	4		1.85	2.25	0.92	0.458	49.68	54.92
	5		1.75	2.41	0.98	0.451	46.18	57.99
								57.62
C	1	6/3/1	2.05	2.38	0.94	0.478	50.56	49.68
	2		2.11	2.41	0.89	0.484	54.47	46.66
	3		2.23	2.41	0.90	0.493	54.93	46.91
	4		2.19	2.47	0.92	0.519	56.48	48.12
	5		2.10	2.40	0.85	0.451	53.15	43.95[e]
								47.84
D	1	6/1/1	2.26	2.40	0.80	0.451	56.44	42.48
	2		2.27	2.30	0.80	0.418	51.90	42.98
	3		2.32	2.37	0.73	0.451	61.83	39.33
	4		2.31	2.20	0.73	0.438	59.68	39.82
	5		2.32	2.16	0.77	0.451	58.93	41.49
								41.22
E	1	4/2/1	3.72	1.94	0.59	0.458	77.22	33.49
	2		3.81	1.99	0.49	0.438	74.70	32.48
	3		4.01	1.90	0.62	0.431	70.06	34.04
	4		4.61	1.95	0.59	0.458	77.55	32.93
	5		4.18	2.04	0.61	0.451	73.54	33.86
								33.36

[a] Cone temp. 500°F

[b] Cl_2 flow of 37.5 g/min

[c] Based on CA

[d] Based on Cl_2 flow

[e] Not included in the average

APPENDIX D

STATISTICAL DATA SAMPLING

APPENDIX D

STATISTICAL DATA SAMPLING

The approach taken in conducting testing for both the first and second reaction was a modified statistical method. This was dictated by the complexity of the relationship between the reaction efficiency (yield, concentration) and the process parameters (feed rate, temperatures, raw material concentration). The statistical approach greatly reduced the number of discrete test points while enabling a thorough evaluation of the reactions.

The primary assumption is made that there exists some functional relationship between the response of interest (in this case yield or concentration of product) and selected reaction variables which can be mathematically approximated by a Taylor series expansion in a confined region. Since the intent of the test program was to identify the characteristics of the process in a region of optimum response, for subsequent large-scale (pilot plant) testing, the precise form of the response function was not necessary. What was needed was a determination of the region of optimum response and a mapping of that region.

Inherent in the assumption of a valid series approximation is that the response function is "smooth" and mathematically well behaved in the region of interest. That is, the reaction chemistries are relatively simple and not subject to abrupt transitions or changes in reaction mechanisms. Thus, the response function can be approximated by the lower order terms of a Taylor series

$$Y = ax_1 + a_2x_2 + a_3x_3 + \dots$$

$$a_{11}x_2^1 + a_{22}x_2^2 + a_{33}x_3^2 \; X \dots$$

$$a_{12}x_1x_2 + a_{13}x_1x_3 + a_{23}x_2x_3 + \dots$$

where

Y = response; yield or concentration

X_i = reaction parameters

A_{ig} = coefficients

The test-point ("zero" point) is estimated to be at or near the point of optimum response, as determined by earlier laboratory testing and from the literature. In the test series, the response is considered to be a function of three variables

$$Y = f(x_1, x_2, x_3)$$

in the region around (x_{01}, x_{02}, x_{03}) and bounded by upper and lower values on each x_{ok} to constrain the region to the area of interest. Thus, the discrete test points are displayed in the matrix form, a cubic array of 2^k points

$$\begin{array}{cccc} & -1 & 0 & +1 \\ x_1 & -a & 0 & a \\ x_2 & -b & 0 & b \\ x_3 & -c & 0 & c \end{array}$$

where

a, b, c, 0 = quantitative values of the independent variables x_i

Because of the unknown extent of the response to the parameters, a "star" design was imposed on the cubic, such that the matrix is expanded by 2^k points

	-	-1	0	1	+
x_1	-na	-a	0	a	+na
x_2	-nb	-b	0	b	+nb
x_3	-nc	-c	0	c	+nc

where

$$n = (2^k)^{1/4}$$

Thus for k = 3 variables, n = 1.6817 and the discrete matrix points are

x_1	-1.68 a	-a	0	+a	+1.68 a
x_2	-1.68 b	-b	0	+b	+1.68 b
x_3	-1.68 c	-c	0	+c	+1.68 c

The center, or zero point, is replicated to provide an estimate of experimental error. The total number of test points becomes

$$N = n_0 + 2k + 2^k$$

where

n_0 = zero point replicates

$2k$ = star design points

2^k = cubic array

For the three-variable system k = 3 and

$$N - 2 + 6 + 8 = 16 \text{ (2 zero replicates)}$$

Thus 16 discrete test points are sufficient to approximate the response function (in this instance a surface) with two replicates to provide an error estimation. The actual test points are tabulated in Attachment D-1 and displayed in Attachment D-2.

The procedure briefly described above was modified to accommodate test equipment limitations and physical constraints of the reaction mixture. In the latter instance, phase separation in the reaction mixture made chemical analysis inaccurate and the final matrix was modified to eliminate the regions of separation.

Attachment D-1

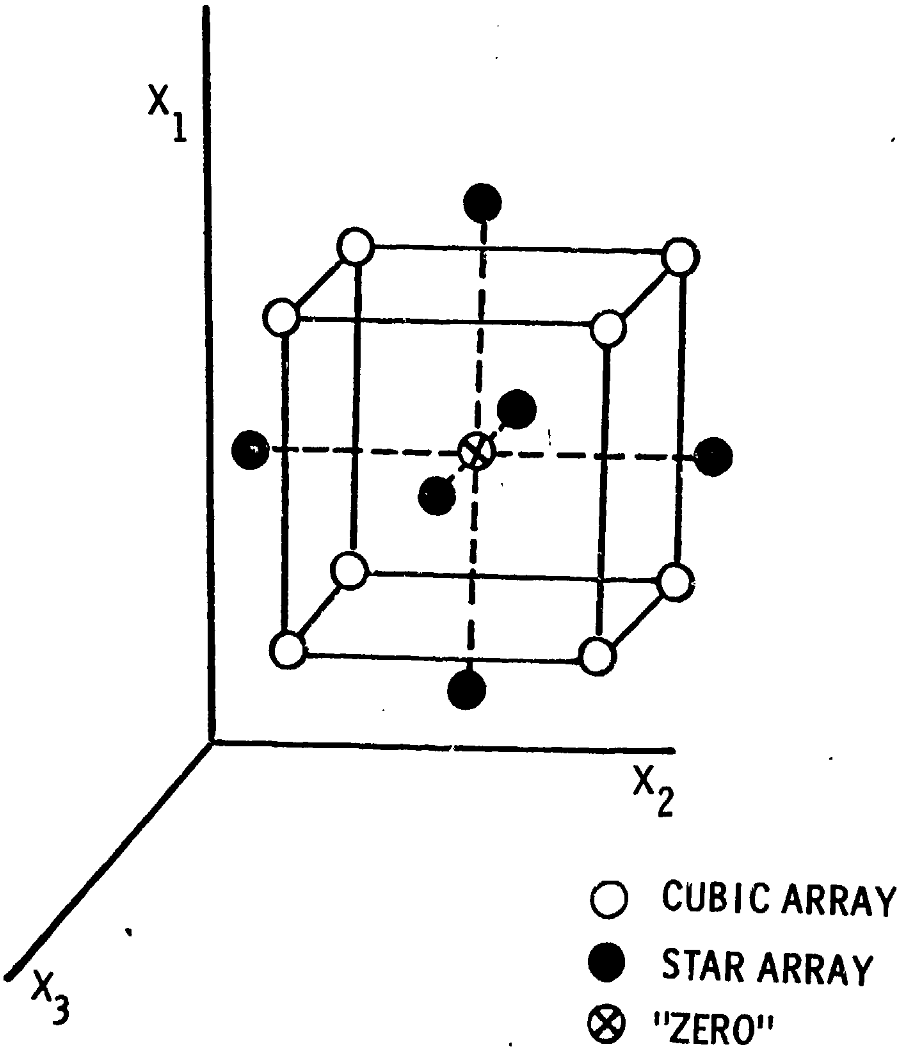
Test Point Tabulation

3-Variable System

	x_1	x_2	x_3	
1	0	0	0	...
2	+a	+b	+c	+++
3	+a	+b	-c	++-
4	+a	-b	+c	+-+
5	+a	-b	-c	+--
6	-a	+b	+c	-++
7	-a	+b	-c	-+-
8	-a	-b	+c	--+
9	-a	-b	-c	---
10	-1.68a	0	0	
11	+1.68a	0	0	
12	0	+1.68b	0	
13	0	-1.68b	0	
14	0	0	+1.68c	
15	0	0	-1.68c	
16	0	0	0	

Attachment D-2

Spatial Array, Test Matrix



APPENDIX E

UDMH AND MMH DATA

APPENDIX E

A: UDMH DATA

Run No.	Con- dition	Run Time (min)	DMA (m/l)	NaOH (m/l)	CA (m/l)	UDMH (m/l)	UDMH (Yd %)	Remark
20	A	65	2.95	2.46	1.48	0.782	53.01	
		85	2.89	2.69	1.63	0.714	43.89	
		105	2.92	2.58	1.60	0.727	45.35	
		125	3.57	2.80	1.55	0.641	41.35	
			3.66	2.59	1.46	0.654	44.86	
	B	200	4.04	1.45	0.92	0.612	66.25	
		200	4.31	1.54	0.86	0.626	72.35	
		240	3.91	1.48	0.79	0.552	70.33	
		260	5.18	1.41	0.73	0.546	74.60	
			5.22	1.47	0.78	0.546	70.32	
			4.94	1.45	0.77	0.506	65.58	
21	A	50	3.74	1.54	1.39	0.727	52.35	
		65	3.89	1.59	1.42	0.754	53.15	
		85	3.80	1.52	1.39	0.740	53.36	
		105	4.06	1.65	1.39	0.789	56.59	
		125	3.88	1.75	1.52	0.789	51.93	
			3.92	1.58	1.37	0.782	57.14	
			3.52	1.50	1.29	0.740	57.31	
	B	175	4.30	1.53	1.50	0.862	57.36	
		190	4.39	1.67	1.64	0.895	54.45	
		210	4.69	1.60	1.53	0.849	55.33	
		230	4.11	1.47	1.40	0.760	54.48	
		250	4.81	1.56	1.43	0.895	62.68	
			4.78	1.49	1.36	0.862	63.38	
			4.47	1.52	1.41	0.849	60.19	
	C	300	4.19	2.10	1.55	0.869	55.86	
		315	4.46	2.23	1.60	0.930	57.96	
		335	4.19	2.22	1.60	0.902	56.38	
		355	4.36	2.41	1.77	0.862	48.76	
		375	4.30	2.35	1.70	0.869	51.15	
			4.17	2.38	1.70	0.895	52.65	

A: UDMH DATA (Continued)

Run No.	Con-dition	Run Time (min)	DMA (m/l)	NaOH (m/l)	CA (m/l)	UDMH (m/l)	UDMH (Yd %)	Remark
22	A	135	2.64	0.72	0.38	0.296	77.79	
		150	2.85	0.75	0.38	0.316	82.42	
		170	2.60	0.73	0.47	0.378	80.68	
		190	2.53	0.76	0.47	0.358	75.95	
		210	2.36	0.80	0.51	0.371	73.21	
			2.36	0.81	0.52	0.364	70.60	
			2.32	0.77	0.48	0.364	75.34	
23	A	55	2.67	0.73	0.65	0.438	67.17	
		70	2.69	0.78	0.67	0.444	65.91	
		90	2.64	0.76	0.67	0.464	69.51	
		110	2.52	0.74	0.66	0.444	67.40	
		130	2.64	0.84	0.72	0.464	64.05	
			2.68	0.81	0.69	0.478	69.62	
			2.74	0.84	0.72	0.451	62.95	
	B	180	2.61	1.52	0.75	0.464	62.00	
		195	2.51	1.63	0.76	0.451	59.26	
		215	2.52	1.55	0.77	0.478	62.41	
		235	2.53	1.55	0.78	0.478	60.99	
		255	2.50	1.59	0.75	0.451	60.13	
			2.55	1.62	0.80	0.578	59.43	
			2.46	1.56	0.76	0.464	61.21	
	C	305	2.91	1.39	0.55	0.378	68.12	
		320	2.92	1.39	0.59	0.378	63.71	
		340	2.83	1.59	0.57	0.364	64.31	
		360	2.75	1.64	0.51	0.358	70.27	
		380	2.88	1.56	0.56	0.404	72.51	
			2.98	1.56	0.58	0.418	72.45	
			2.83	1.53	0.56	0.378	67.60	
	D	435	2.47	0.92	0.68	0.404	59.54	
		450	2.53	0.98	0.69	0.404	58.61	
		470	2.52	0.90	0.69	0.404	58.66	
		490	2.59	1.11	0.68	0.404	59.12	
		510	2.52	1.06	0.63	0.404	64.49	
			2.47	1.10	0.66	0.418	63.04	
			2.46	1.12	0.67	0.391	58.38	

A: UDMH DATA (Continued)

Run No.	Condition	Run Time (min)	DMA (m/l)	NaOH (m/l)	CA (m/l)	UDMH (m/l)	UDMH (Yd %)	Remark
23	E	560	2.82	0.51	0.48	0.364	75.21	
		575	2.99	0.71	0.61	0.384	63.12	
24	A	50	3.58	0.78	0.61	0.431	70.68	
		65	3.58	0.76	0.56	0.451	80.99	
		80	3.60	0.80	0.58	0.478	82.19	
		100	3.61	0.75	0.55	0.444	80.95	
		125	3.71	0.82	0.60	0.464	77.64	
			3.87	0.85	0.63	0.458	72.72	
			3.77	0.83	0.61	0.464	75.85	
	B	175	3.36	1.13	0.68	0.444	65.61	
		190	3.57	1.37	0.65	0.451	69.51	
		210	3.61	1.34	0.65	0.464	71.22	
		230	3.89	1.26	0.66	0.499	75.31	
		250	3.65	1.25	0.75	0.539	72.32	
			3.52	1.19	0.68	0.506	73.96	
			3.67	1.28	0.77	0.526	68.04	
	C	300	4.48	0.86	0.64	0.486	75.42	
		315	4.48	0.83	0.64	0.499	78.21	
		335	4.64	0.84	0.67	0.478	71.20	
		355	4.54	0.96	0.63	0.478	76.10	
		375	4.57	1.08	0.64	0.454	70.52	
			4.55	1.09	0.66	0.451	68.77	
			4.62	1.08	0.63	0.486	77.22	
	D	425	4.24	1.60	0.64	0.451	70.09	
		440	4.08	1.63	0.62	0.431	69.42	
		490	3.63	0.80	0.67	0.444	65.91	
		505	3.51	0.99	0.65	0.438	66.88	
		555	5.32	1.02	0.42	0.316	74.88	
		570	5.47	0.86	0.42	0.336	80.09	

B: MMH DATA

Run No.	Condition	Run Time (min)	MMA (m/l)	NaOH (m/l)	CA (m/l)	MMH (m/l)	MMH (Yd %)	Remark
2-B	A	75	7.87	1.60	0.31	0.176	56.51	
		99	8.13	1.60	0.35	0.204	57.96	
		110	8.36	1.65	0.36	0.208	58.17	
		129	8.77	1.81	0.33	0.204	61.05	
		142	7.91	1.82	0.40	0.230	58.14	Not Used
		162	8.06	1.61	0.34	0.204	60.89	
		173	8.06	1.60	0.33	0.204	60.94	
		194	7.70	1.76	0.34	0.184	55.60	
	B	220	7.71	1.58	0.33	0.215	65.28	
		235	7.88	1.48	0.32	0.234	72.15	
		260	8.11	1.49	0.29	0.215	74.40	
		273	7.77	1.55	0.28	0.195	68.71	
		288	8.27	1.58	0.26	0.193	75.76	
		298	8.15	1.53	0.32	0.208	65.44	
	C	326	8.40	1.43	0.29	0.208	70.83	
		342	8.56	1.53	0.24	0.195	81.39	
		358	8.18	1.51	0.23	0.165	71.85	
		372	7.93	1.50	0.32	0.215	68.02	
		402	8.46	1.49	0.25	0.187	74.36	
	D	452	7.96	1.62	0.35	0.245	69.46	
		467	7.70	1.50	0.34	0.256	75.93	
		482	8.10	1.49	0.28	0.204	73.22	
		497	7.73	1.65	0.31	0.208	66.38	
		526	7.79	1.47	0.33	0.243	72.92	
	E	553	7.87	1.61	0.33	0.211	64.63	
		569	8.16	1.50	0.32	0.230	71.93	
		580	6.83	1.55	0.31	0.206	65.86	Not Used
		598	7.30	1.47	0.24	0.182	74.99	
		615	7.96	1.56	0.24	0.161	67.63	
		630	7.80	1.43	0.23	0.174	75.26	
	F	664	7.90	1.52	0.24	0.171	70.53	
		675	7.85	1.54	0.26	0.180	69.28	
		690	8.29	1.48	0.28	0.200	72.02	
		704	7.22	1.39	0.28	0.184	64.89	
	G	724	7.15	1.41	0.27	0.184	67.65	
		740	7.83	1.34	0.28	0.184	65.15	
		754	7.93	1.46	0.265	0.195	73.76	

B: MMH DATA (Continued)

Run No.	Con-dition	Run Time (min)	MMA (m/l)	NaOH (m/l)	CA (m/l)	MMH (m/l)	MMH (Yd %)	Remark
3-A	A	40	7.35	1.31	0.45	0.250	55.77	
		55	7.81	1.32	0.42	0.260	62.31	
		70	8.07	1.40	0.41	0.271	65.75	
		85	8.30	1.19	0.39	0.243	62.55	
		100	7.89	1.24	0.40	0.256	64.00	
		115	8.12	1.36	0.41	0.306	74.37	Not Used
		130	8.22	1.34	0.41	0.258	62.73	
	B	150	8.11	1.47	0.38	0.250	65.56	
		180	8.03	1.47	0.41	0.265	64.44	
		195	7.72	1.53	0.39	0.247	63.99	
		209	7.83	1.57	0.35	0.239	68.38	
		239	7.74	1.47	0.35	0.243	68.62	
		255	8.22	1.50	0.31	0.221	72.15	
	C	274	7.28	2.01	0.39	0.256	65.47	
		288	6.81	2.27	0.46	0.282	61.00	
		304	6.91	2.09	0.45	0.291	64.57	Not Used
		318	7.37	2.19	0.37	0.258	68.91	
		335	7.44	2.35	0.38	0.260	67.85	
		348	7.01	2.15	0.41	0.269	64.96	
		364	7.21	2.22	0.38	0.254	66.30	
	D	386	6.61	2.26	0.37	0.230	62.41	
		400	6.59	2.11	0.39	0.230	58.94	
		415	6.97	2.25	0.41	0.265	64.97	Not Used
		430	7.47	2.09	0.34	0.239	69.85	
		445	7.21	2.17	0.34	0.234	69.84	
		460	7.02	2.22	0.35	0.226	63.72	
		475	6.73	2.20	0.35	0.23	65.56	

B: MMH DATA (Continued)

Run No.	Condition	Run Time (min)	MMA (m/l)	NaOH (m/l)	CA (m/l)	MMH (m/l)	MMH (Yd %)	Remark
3-B	A	39	7.12	1.47	0.40	0.258	64.63	
		54	7.55	1.52	0.39	0.278	71.64	
		70	7.71	1.48	0.35	0.260	74.24	
		84	7.38	1.46	0.37	0.284	76.14	
		99	7.48	1.47	0.39	0.286	74.15	
		114	7.62	1.33	0.35	0.269	77.77	Not Used
		129	7.82	1.42	0.32	0.243	74.88	
	B	170	7.99	1.51	0.29	0.234	79.76	
		230	8.22	1.52	0.28	0.247	87.47	
		243	8.22	1.49	0.25	0.226	91.68	
		258	8.08	1.62	0.23	0.221	94.23	
		276	8.00	1.61	0.25	0.234	94.02	
		292	8.15	1.57	0.24	0.226	93.72	
		320	8.39	1.51	0.215	0.202	93.92	
	C	369	8.27	1.48	0.31	0.256	83.69	
		384	8.20	1.53	0.23	0.204	83.75	
		399	8.29	1.52	0.18	0.178	97.08	
		414	8.26	1.64	0.18	0.178	99.69	
		403	8.52	1.57	0.18	0.174	97.26	
		455	8.59	1.54	0.16	0.156	98.59	
		460	8.66	1.54	0.12	0.130	104.70	Not Used

B: MMH DATA (Continued)

Run No.	Condition	Run Time (min)	MMA (m/l)	NaOH (m/l)	CA (m/l)	MMH (m/l)	MMH (Yd %)	Remark
3-C	A	40	7.37	1.77	0.59	0.367	61.67	
		90	7.75	1.94	0.60	0.410	68.64	
		105	7.95	1.94	0.60	0.428	70.98	
		130	8.10	1.96	0.64	0.425	66.07	Not Used
			8.78	1.97	0.62	0.397	64.07	
			8.23	2.02	0.62	0.451	72.96	
	B	164	7.89	1.61	0.48	0.358	74.04	
		180	7.65	1.59	0.47	0.347	74.49	
		195	7.93	1.61	0.46	0.334	71.91	Not Used
			8.66	1.57	0.43	0.334	77.31	
			7.82	1.61	0.43	0.328	76.05	

B: MMH DATA (Continued)

Run No.	Con-dition	Run Time (min)	MMA (m/l)	NaOH (m/l)	CA (m/l)	MMH (m/l)	MMH (Yd %)	Remark
4A	A	35	8.88	0.75	0.63	0.293	46.24	
		65	10.06	0.80	0.70	0.362	51.93	
		85	10.46	1.01	0.72	0.369	51.04	
		105	10.08	0.95	0.69	0.378	55.13	
		125	10.73	0.90	0.62	0.349	56.24	
			10.65	0.97	0.66	0.358	54.33	
			10.40	0.96	0.67	0.358	53.24	
		155	9.48	0.84	0.49	0.276	55.94	

B: MMH DATA (Continued)

Run No.	Con-dition	Run Time (min)	MMA (m/l)	NaOH (m/l)	CA (m/l)	MMH (m/l)	MMH (Yd %)	Remark
4-B	A	65	9.91	0.98	0.66	0.384	58.16	
		85	10.31	1.01	0.67	0.406	60.75	
		105	9.79	1.14	0.69	0.375	54.05	
		125	9.92	1.07	0.67	0.367	54.58	
			10.00	1.03	0.64	0.371	57.57	
			10.13	1.07	0.70	0.402	57.71	

B: MMH DATA (Continued)

Run No.	Condition	Run Time (min)	MMA (m/l)	NaOH (m/l)	CA (m/l)	MMH (m/l)	MMH (Yd %)	Remark
4-C	A	35	9.68	0.95	0.55	0.397	71.60	
		65	10.70	1.08	0.60	0.319	52.79	
		85	10.66	1.10	0.64	0.336	52.87	
		105	10.82	1.07	0.69	0.345	50.17	
		125	9.93	1.12	0.68	0.295	43.63	Not Used
	B	175	12.34	1.14	0.571	0.295	51.74	
		190	11.48	1.13	0.51	0.278	54.76	
		205	12.33	1.06	0.52	0.302	58.26	
	C	252	11.10	1.19	0.58	0.362	62.91	
		267	10.10	1.19	0.64	0.375	58.68	
		283	10.63	1.22	0.63	0.358	56.46	
	D	330	12.20	1.11	0.64	0.389	60.98	
		345	10.99	1.20	0.67	0.375	56.18	

B: MMH DATA (Continued)

Run No.	Con-dition	Run Time (min)	MMA (m/l)	NaOH (m/l)	CA (m/l)	MMH (m/l)	MMH (Yd %)	Remark
4-D	A	35	10.53	0.75	0.61	0.347	57.00	
		65	10.52	0.84	0.80	0.423	52.95	
		85	10.92	0.74	0.70	0.402	57.36	
		105	11.70	0.84	0.63	0.313	49.32	
		125	10.58	0.74	0.70	0.371	52.98	Not Used
			10.96	0.74	0.71	0.375	52.85	
			10.22	0.71	0.67	0.375	55.89	
	B	175	8.52	0.75	0.59	0.321	54.78	
		190	8.72	0.77	0.60	0.334	55.64	
		205	8.82	0.77	0.64	0.339	52.89	
			8.66	0.77	0.64	0.330	51.33	
			8.66	0.78	0.66	0.330	50.33	
	C	260	11.82	0.63	0.57	0.286	50.05	
		270	9.65	0.63	0.61	0.269	44.00	
		290	10.49	0.50	0.49	0.221	45.43	
			9.65	0.51	0.49	0.265	54.55	
			10.35	0.47	0.46	0.234	50.86	
	D	345	10.49	1.34	0.49	0.221	44.85	
		360	9.71	1.46	0.60	0.265	43.87	
		375	10.25	1.43	0.55	0.230	41.47	
			10.59	1.43	0.56	0.243	43.03	
			8.29	1.44	0.56	0.239	42.99	Not Used
	E	410	11.48	0.88	0.58	0.304	52.40	
		455	11.88	0.85	0.61	0.256	42.10	
		470	10.79	0.84	0.57	0.239	42.12	

B: MMH DATA (Continued)

Run No.	Con-dition	Run Time (min)	MMA (m/l)	NaOH (m/l)	CA (m/l)	MMH (m/l)	MMH (Yd %)	Remark
5-A	A	65	11.06	1.00	0.77	0.280	36.43	Not Used
		85	11.10	0.99	0.76	0.315	41.48	Not Used
		105	11.75	0.93	0.66	0.328	49.41	
		125	11.66	0.88	0.58	0.302	52.36	
	B	280	12.75	1.99	1.02	0.469	45.97	
		290	11.57	2.13	1.04	0.486	46.87	
		315	12.30	2.01	1.00	0.501	50.05	
		340	12.02	2.13	1.01	0.469	46.23	
			11.85	2.17	1.03	0.477	46.54	
			11.64	2.13	1.01	0.469	46.30	
	C	405	12.83	1.74	0.77	0.273	35.40	Not Used
		425	13.03	1.75	0.86	0.417	48.56	
		445	12.47	1.82	0.91	0.438	48.25	
		465	12.25	1.81	0.94	0.469	49.71	
			11.21	1.80	0.86	0.443	51.47	
			13.26	1.85	0.94	0.469	49.86	
	D	560	12.92	2.03	0.94	0.415	44.14	
		580	13.72	1.88	0.76	0.393	51.44	Not Used
		615	11.71	1.39	0.65	0.334	51.42	
		630	11.23	1.26	0.59	0.286	48.91	
		645	10.95	1.18	0.58	0.300	51.68	

B: MMH DATA (Continued)

Run No.	Con-dition	Run Time (min)	MMA (m/l)	NaOH (m/l)	CA (m/l)	MMH (m/l)	MMH (Yd %)	Remark
6	A	100	11.12	1.95	0.75	0.341	45.15	Not Used
		120	11.69	2.28	0.75	0.354	47.01	
		140	12.70	1.84	0.68	0.362	53.44	
		160	12.70	1.11	0.64	0.345	53.90	
			12.16	1.13	0.63	0.332	52.73	
			12.71	1.89	0.61	0.323	52.77	
	B	230	9.77	2.40	0.87	0.391	45.02	Not Used
		245	10.52	2.13	0.89	0.408	45.73	Not Used
		265	10.06	2.03	0.90	0.434	48.14	Not Used
		285	10.19	1.99	0.86	0.499	58.36	
			9.11	2.05	0.89	0.477	53.46	
			9.88	2.21	0.90	0.488	54.36	
	C	371	11.73	1.33	1.03	0.536	52.12	
		410	11.58	1.42	1.08	0.501	46.44	
		430	11.59	1.22	1.00	0.532	53.32	
		450	12.35	1.28	0.98	0.545	55.39	
			11.85	1.22	0.98	0.527	53.75	
	D	530	10.84	1.17	0.30	0.211	69.96	
		545	10.94	2.52	0.40	0.213	53.07	
		560	10.33	2.59	0.54	0.310	57.86	Not Used
		590	12.33	2.09	0.37	0.256	68.33	
			12.65	1.99	0.37	0.265	71.07	
			11.52	2.21	0.38	0.204	53.98	

B: MMH DATA (Continued)

Run No.	Con-dition	Run Time (min)	MMA (m/l)	NaOH (m/l)	CA (m/l)	MMH (m/l)	MMH (Yd %)	Remark
7	A	65	8.45	1.05	0.67	0.371	55.18	
		85	8.18	1.05	0.67	0.358	53.37	
		105	9.01	1.05	0.67	0.404	60.12	
		125	9.00	1.05	0.65	0.408	62.88	
			8.37	0.99	0.62	0.371	59.41	
			8.41	1.11	0.69	0.358	51.64	
	B	185	7.60	2.57	0.87	0.469	53.97	
		205	7.58	2.74	0.91	0.493	54.00	
		225	7.98	2.60	0.94	0.493	52.41	
		240	8.25	2.80	0.88	0.473	53.48	
			7.77	2.76	0.90	0.438	48.73	
			8.30	2.82	0.96	0.460	48.10	

B: MMH DATA (Continued)

Run No.	Con-dition	Run Time (min)	MMA (m/l)	NaOH (m/l)	CA (m/l)	MMH (m/l)	MMH (Yd %)	Remark
8	A	65	5.94	1.34	0.92	0.436	47.29	
		85	5.72	1.27	0.91	0.406	44.38	
		105	5.49	1.33	1.05	0.397	37.75	
		125	5.78	1.24	0.93	0.445	47.94	
			5.70	1.25	0.93	0.436	46.72	
			5.71	1.30	0.99	0.441	44.39	
	B	195	5.71	1.41	1.05	0.464	44.14	
		215	5.95	1.66	1.24	0.477	38.57	Not Used
		235	5.68	1.40	0.93	0.460	49.42	
		255	5.85	1.40	0.95	0.517	54.35	Not Used
			5.76	1.40	0.94	0.449	47.69	
			5.62	1.40	0.95	0.464	48.67	
	C	320	5.65	1.44	0.97	0.428	44.15	
		340	5.54	1.47	0.93	0.445	47.98	
		360	5.41	1.64	0.94	0.428	45.53	
		380	6.10	1.86	1.18	0.473	40.00	Not Used
			5.34	1.67	0.97	0.432	44.64	
			5.79	1.64	0.93	0.449	48.21	
	D	445	5.78	1.52	0.89	0.441	49.66	
		465	5.73	1.60	0.95	0.436	46.09	
		485	5.84	1.52	0.91	0.441	48.41	
		505	6.10	1.33	0.88	0.458	51.80	
			5.83	1.40	0.96	0.460	47.98	
			5.50	1.41	0.94	0.402	42.93	
	E	575	5.75	1.57	0.89	0.449	50.27	
		600	5.99	1.55	0.85	0.436	51.34	

APPENDIX F

DATA SHEETS

Ⓢ-3 Chemical Analysis
Date ____ Run Number 1
Analyst Kruber

Run Time		Sample Taken					Process Data Flow Rate			Product Analysis Results										Output Data Calculated									
																	Gas Samples			NH$_2$Cl Yield (%) Based On				Hydrazine Yield (%) Based On					
Cont. Min.	Θ Cont. Min.	Y-1	Y-2	Y-3	Y-4	Y-5	Cl$_2$ g/Min	NH$_3$ g/Min	Product ML/Min	NH$_3$ g/l	Amine g/l	S.P. g/l	Hydrazine g/l	NaOH g/l	Cl$^-$ g/l	pH	NH$_3$	Iodometric NH$_2$Cl	U.V. NH$_2$Cl	Total Cl$^-$	Iodometric	U.V.	Hydrazine g/Min	Cl$^-$	Cl$_2$ Feed	Iodometric NH$_2$Cl	U.V. NH$_2$Cl	Calc. Proc.	Remarks
					3.2				480.9											62.65				47.04	35.26				
					5.2				502.5											70.20				[illegible]	37.10				
					7.8				521.3											70.45				49.50	38.46				
					10.2				492.1											70.94				52.64	37.20				
					12.8				505.9											61.25				50.72	35.30				
					15.2				504.7											67.32				47.80	33.28				
					17.8				498.9											70.79				49.74	38.32				
					20.2				506.8											65.26				53.91	34.56				
					22.8				507.5											55.65				40.61	33.33				
					25.2				518.9											65.85				46.92	32.20				
					27.8				492.4											51.15				37.61	30.09				
					30.2				512.9											53.93				38.92	30.67				
					32.8				476.5											65.42				62.98	40.31				
					35.2				504.8											63.93				63.14	37.82				
					37.8				500.2											62.85				64.82	40.12				
					40.2				501.9											60.99				62.08	38.09				
					42.8				500.9											65.53				60.84	34.30				
					45.2				498.8											60.63				65.69	37.65				
					47.8				500.1											59.69				62.98	35.36				
					50.2				503.2											60.34				64.02	36.62				

↑ BASED ON NaOH MASS BALANCE

MMH

Ⓢ-3 Chemical Analysis
Date ____ Run Number 1
Analyst ____

Run Time		Sample Taken					Process Data Flow Rate			Product Analysis Results										Output Data Calculated									
																	Gas Samples			NH$_2$Cl Yield (%) Based On				Hydrazine Yield (%) Based On					
Cont. Min.	Θ Cont. Min.	Y-1	Y-2	Y-3	Y-4	Min Bag Sheet	Cl$_2$ g/Min	NH$_3$ g/Min	Product ML/Min	NH$_3$ g/l	Amine g/l	S.P. g/l	Hydrazine g/l	NaOH g/l	Cl$^-$ g/l	pH	NH$_3$	Iodometric NH$_2$Cl	U.V. NH$_2$Cl	Total Cl$^-$	Iodometric	U.V.	Hydrazine g/Min	Cl$^-$	Cl$_2$ Feed	Iodometric NH$_2$Cl	U.V. NH$_2$Cl	Calc. Proc.	Remarks
						1	37.5	32.06	692.4					.036															
		2															2.125		.082			91.89							
					3.2					[illegible]	208.5	[illegible]	15.9	7.07	32.48					62.79			7.533	51.56	32.40				I NH$_2$Cl in spent
						4								.082															
				5													15.12		1.689			69.04							2
					5.2					[illegible]	208.1	520	16.5	12.73	28.16					56.34			9.225	52.95	35.05				
					7.8					[illegible]	205.9	519	16.9	12.80	28.06					53.79			9.086	50.71	34.97				@ 0.782
						8								.080															
				9													12.75		.966			52.56							4
					10.2					[illegible]	[illegible]	[illegible]	16.5	6.97	22.06					53.79			9.136	52.45	33.42				
						11	23.5	40	522.9					.097															
		12															13.75		.462			36.33							
					12.8					[illegible]	212.3	536	15.8	16.62	21.91					57.83			8.261	49.79	33.95				
						14								.062															
				15													13.25		.663			49.35							
					16.2					[illegible]	210.8	496	15.9	16.43	25.38					56.95			9.336	55.32	36.31				
					17.8					[illegible]	212.8	[illegible]	17.5	16.02	24.62					56.79			9.499	53.68	37.40				@ 0.923
						18								.041															
				19													15.75		.773			45.58							5
					20.2					20.0	213.3	532	16.8	1.290	22.62					52.68			8.261	53.81	33.95				
						21	37.5	32.06						.065															
		22															14.25		.882			30.31							6
					22.8					[illegible]	204.6	520	8.5	13.53	21.01					61.86			8.627	60.91	36.06				
						24																							
				[illegible]													17.25		1.146			38.7							8
					25.2					[illegible]	207.5	520	16.6	13.35	18.18					61.37			8.261	56.31	33.95				
					27.8					[illegible]	208.1	523	15.2	13.16	19.18					60.97			7.997	55.50	32.66				@ 0.920
						28								.062															
				30													16.25		.772			22.97							6
					30.2					16.3	226.0	500	16.8	15.99	20.71					53.95			8.261	52.78	33.65				

(B-1 Chemical Analysis)
Date ____, Run Number 1
Analyst ____

Run Time		Sample Taken					Process Data : Flow Rate			Product Analysis Results										Output Data Calculated									
																	Gas Samples			NH_4Cl Yield % Based On			Hydrazine	Hydrazine Yield % Based On				Cell Prec.	Remarks
Cont. Min.	@ Cond. Min.	Y-1	Y-2	Y-3	Y-4	FP wt% NaOCl	Cl_2 g/Min	NH_3 g/Min	Product ML/Min	NH_3 g/l	Amine g/l	[illegible] g/l	Hydrazine g/l	NaOH g/l	Cl^- g/l	pH	NH_3	Iodometric NH_4Cl	U.V. NH_4Cl	Total Cl^-	Iodometric	U.V.	g/Min	Cl^-	Cl_2 Feed	Iodometric NH_4Cl	U.V. NH_4Cl		
						[illegible]	37.5	72.45	533.8					.091															
		[illegible]															12.63		1.159			62.21							[illegible]
					38.2					[illegible]	218.6	528	16.6	29.20	19.34					58.98			8.738	64.96	55.99				
						[illegible]								.091															
				[illegible]													13.13		1.024			47.63							[illegible]
					[illegible]					[illegible]	298.8	526	14.4	17.95	23.60					49.01			7.529	63.06	30.94				
					[illegible]					[illegible]	235.0	528	16.8	13.02	16.82					52.61			8.244	68.95	32.95				[illegible]
						[illegible]								.098															
				[illegible]													12.38		1.097			47.91							
					[illegible]					[illegible]	231.1	528	16.0	15.38	[illegible]					56.21			7.998	[illegible]	33.33				
						[illegible]	37.5	72.45	517.8					.091															
					[illegible]					[illegible]	236.0	604	16.0	15.47	20.93					53.78			7.366	65.30	34.91				
					[illegible]					[illegible]	236.8	620	16.7	13.16	21.98					60.14			8.181	[illegible]	[illegible]				[illegible]
						[illegible]								.091															
					[illegible]					[illegible]	240.8	592	17.5	12.36	[illegible]					65.36			9.044	[illegible]	37.32				
					[illegible]					[illegible]	236.7	629	16.7	15.34	22.12					63.33			8.497	[illegible]	[illegible]				

UDMH/MMH PLANT RUN DATA SHEETS

(B-1 Process)
X MMH, Date [illegible], Test No [illegible]
____ UDMH, Test Conductor [illegible]
Page ____ Of ____

Run Time		Test Matrix Point	1st Stage Feed Rate			2nd Stage Feed Rate			System Pressures				Stirrer RPM x 100	Cont. Loss Liters	NaOH Level Inches	Sample Taken					Vibrator		Flow Rates		T.P. Jacket Flow Rate		Electrostatic Precipitator				
																											AC			DC	
Cont Min.	@ Cond Min.		Cl_2 g/Min	NH_3 g/Min	N_2 g/Min	NaOH ML/Min	Water ML/Min	Amine ML/Min	T.P. Psig	E.P. Psig	Cont. Psig	Spent Psig				Y-1	Y-2	Y-3	Y-4	FP & Y-6	Freq. Min.	Force PSI	Product ML/Min	Vent g/Min	Upper GPM	Lower GPM	Volts	Amps	Spark Min	Kilovolts	mA
[illegible]	[illegible]	[illegible]	[illegible]	72.05	[illegible]	[illegible]	210.4	[illegible]	1.1	1.3	0.2	1.25	1850	8							2				3	3	62	1.2	20	35	1.0
5	0	A	37.5	72.05	29.7	58.8	219.9	214	1.3	1.4	0.2	1.4															62	1.0	10	40	1.3
20	15	A	37.5	72	29	58.8	210	214	1.35	1.5	0.2	1.5								1							62	1.0	20	41	0.9
35	30	A	37.5	72	[illegible]	58	210	214	1.35	1.6	0.2	1.6				2											50	0.8	10	35	0.9
50	45	A	37.5	72	29	58	210	214	1.4	1.6	0.2	1.5							7.1	9							50	0.8	10	35	0.9
65	60	A	37.5	72	29	40	210	214	1.3	1.5	0.2	1.45						6									50	0.8	10	35	0.9
80	75	A	37.5	72	29	58.8	210	214	1.5	1.7	0.2	1.6							6.3								50	0.9	20	35	0.8
95	90	A	37.5	72	29	58.8	210	214	1.6	1.8	0.2	1.7							7.2	8							50	0.8	10	35	0.9
110	105	A	37.5	72	29	56.0	210	214	1.7	2.0	0.2	2.0															57	0.8	10	35	0.9
125	120	A	37.5	72	29	58.8	210	214	1.85	2.1	0.2	1.95						9	10.5								57	0.8	10	35	0.8
[illegible]	15	B	37.5	110	29	56.0	210	214	3.5	3.6	0.5	3.6								8							50	0.9	20	35	0.7
[illegible]	30	B	37.5	110	29	58.8	210	214	2.6	2.8	0.4	2.8				12											60	0.8	20	35	0.7
[illegible]	45	B	37.5	110	29	58.8	210	214	2.9	2.95	0.2	2.9							[illegible]	[illegible]							50	0.8	20	35	0.8
185	60	B	37.5	110	29	59	210	214	2.7	2.9	0.4	2.7						15									50	0.8	20	35	0.8
200	75	B	37.5	110	29	59	210	214	4.0	3.2	0.5	[illegible]							[illegible]								50	0.8	10	35	0.8
215	90	B	37.5	110	29	56	210	214	[illegible]	3.2	0.4	[illegible]							17.0	[illegible]							50	0.8	10	35	0.8
245	120	B																[illegible]	[illegible]												
270	15	C	37.5	72.05	[illegible]	59	210	214	2.7	2.9	0.0	1.9								[illegible]							50	0.8	20	35	0.8
285	30	C	37.5	72.05	[illegible]	59	210	214	2.3	2.6	0.2	2.6				22											52	0.8	20	35	1.0
300	45	C	37.5	72.0	[illegible]	59	210	214	2.2	2.4	0.2	2.7							[illegible]	[illegible]							52	0.8	20	35	0.8
315	60	C	37.5	72.0	[illegible]	59	210	214	2.3	2.6	[illegible]	2.5						26									51	0.9	10	35	0.8
330	75	C	37.5	72.0	[illegible]	57.5	210	214	2.3	2.5	[illegible]	2.35							[illegible]								52	0.9	10	35	0.8
345	90	C	37.5	72.0	[illegible]	59	210	214	2.3	[illegible]	[illegible]	2.30							[illegible]	[illegible]							53	0.8	20	35	0.8
365	110	C	37.5	72.0	[illegible]	59	210	214	2.5	2.5	0.2	2.7						28	[illegible]								53	0.8	10	34	0.8
385	15	D	30.0	57.5	47.1	58.8	210	214	3.6	1.75	0.2	3.6								31							55	0.9	10	35	1.1
410	30	D	30.0	57.5	47.0	59	210	214	4.0	4.2	0.7	4.05						32									55	0.7	10	35	1.1

(B-1 Process)

UDMH/MMH PLANT RUN DATA SHEETS

✓ MMH, Date 2-21-78, Test No 1
UDMH, Test Conductor
Page ___ Of ___

Run Time Cont. Min.	Run Time @ Cond. Min.	Test Matrix Point	1st Stage Feed Rate Cl₂ g/Min	1st Stage Feed Rate NH₃ g/Min	1st Stage Feed Rate N₂ g/Min	2nd Stage Feed Rate NaOH ML/Min	2nd Stage Feed Rate Water ML/Min	2nd Stage Feed Rate Amine ML/Min	System Pressures T.P. Psig	System Pressures E.P. Psig	System Pressures Cont. Psig	System Pressures Spool Psig	Stirrer RPM x 100	Cont. Level Liters	NaOH Level Inches	Sample Taken Y-1	Sample Taken Y-2	Sample Taken Y-3	Sample Taken Y-4	Sample Taken P-[illegible]	Vibrator Freq. Min.	Vibrator Force PSI	Flow Rates Product ML/Min	Flow Rates Vent g/Min	T.P. Jacket Flow Rate Upper GPM	T.P. Jacket Flow Rate Lower GPM	Electrostatic Precipitator AC Volts	Electrostatic Precipitator AC Amps	Sparks/Min	DC Kilovolts	DC mA
415	45	D	30.0	57.5	47.0	59	210	214	3.9	4.0	0.3	4.0							[illegible]	[illegible]							62	1.1	[illegible]	35	1.3
430	60	D	30.0	57.5	47.0	59	210	214	3.95	4.1	0.4	3.8						[illegible]									60	1.1	10	35	4.5
455	75	D	30.0	57.5	47.0	59	210	214	[illegible]	4.1	0.3	3.9							[illegible]								61	1.1	10	35	4.5
470	90	D	30.0	57.0	47.0	61	210	214	4.1	[illegible]	[illegible]	4.3							[illegible]	[illegible]							60	1.1	10	35	4.5
490	110	D	30.0	57.5	47.0	59	210	214	4.3	4.4	0.3	4.0						[illegible]	[illegible]								[illegible]	1.0	10	35	4.1
510	15	E'	27.5	110	29.7	59	210	214	5.0	5.2	0.25	4.9								[illegible]							[illegible]	1.0	23	[illegible]	1.4
525	30	E'	27.5	110	29.7	[illegible]	210	214	3.9	5.2	0.1	5.1							[illegible]								[illegible]	1.1	23	[illegible]	1.4
540	45	E'	27.5	110	29	59	210	214	[illegible]	[illegible]	0.0	4.7							[illegible]								[illegible]	1.5	21	[illegible]	[illegible]
555	60	E'	27.5	110	29	61	210	214	[illegible]	2.6	0.3	2.4							[illegible]								[illegible]	1.1	21	[illegible]	1.4
570	75	E'	27.5	110	29	59	210	214	2.3	2.6	0.1	2.4							[illegible]								[illegible]	1.0	21	[illegible]	1.4

TEST #1

(B-2 Temperatures)
Date 2-21-78 Run No. [illegible]

Temperatures - °F Refer To Instrumentation List 4 Rev.

	Run Time Cont.	Run Time @ Cond.	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18	19	20	21	22	23	24	25	26	27	28	29	30	31	32	33	34	35	36	37	38	39	40	41	42	43	44	45
Pre	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]				
	5	0	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]				
	20	15	81	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]				
	35	30	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]				
	50	45	80	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]				
	65	60	80	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]				
	80	75	80	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]				
	95	90	79	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]				
	110	105	79	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]				
A	125	120	80	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]				
B	145	15	70	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]				
	160	30	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]				
	175	45	75	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]				
	185	60	76	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]				
	200	75	74	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]				
	215	90	75	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]				
	230	15	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]				
	[illegible]	30	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]				
	[illegible]	45	69	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]				
C	315	60	71	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]				
	330	75	69	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]				
	345	90	72	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]				
	365	110	74	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]				
D	395	15	73	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]				
	410	30	76	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]				

See picture

T.C. #29 is the probe (moveable) in the new T.P. [illegible] when pushed in to ℄

(D-2 Temperatures)

SAT Date 3-21-78 Run No. 1

Temperatures - °F Refer To Instrumentation List ____ Rev. ____

Run Cont.	Time @ Cond.	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18	19	20	21	22	23	24	25	26	27	28	29	30	31	32	33	34	35	36	37	38	39	40	41	42	43	44	45
425	45	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	6	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]			
430	60	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	4	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]			
435	75	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	8	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]			
440	90	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	6	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]			
450	110	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	5	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]			
510	15	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	5	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]			
525	30	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	4	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]			
540	45	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	8	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]			
555	60	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	5	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]			
570	75	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	5	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]			

(D-4 Test Conductors Notes)

Date 3-21-78 Run No. [illegible]

Run Cont.	Time @ Cond.	Test Conductor's Notes
		Pre-test Conditions per Test Plan, Rev #1
0830		Conditions @ 30%
0845	30	[illegible]
		[illegible]
11	6	[illegible]
		[illegible]
20	15	Adjusting C.P. Bars to 35 KV from 40.
20	15	[illegible]
35	30	Dump HgCl
	30	[illegible]
		[illegible]
48	43	[illegible]
65	60	Dump HgCl
65	60	[illegible]
	75	[illegible]
	83	Conditions @ 30%
	90	Dump HgCl
	105	[illegible]
	120	Dump HgCl
140	10	Burned up reaction zone [illegible]
180	20	Dump XHgCl
185	25	Dump XHgCl
185	60	Upper [illegible] Discharge valve not operating to open position [illegible]
220	90	[illegible]
250		Upper gate valve repaired using crowbar; dump XHgCl
255		[illegible]
		[illegible]
300		Dump HgCl !
320		[illegible]
370		Dump HgCl
390		Tried to go to Condition [illegible]
		[illegible]

III-4 Test Conductor's Notes

SET 2

Date [illegible] Run No. 1

Run Cond.	Time @ Cond.	Test Conductor's Notes
310	30	[illegible]
315		Pump [illegible]
360		30% Caustic Pump out - running on Drum feed.
395		[illegible]
380		Liquid [illegible]
[illegible]		[illegible]
670		Normal shut down

III-5 Chemical Analysis

Date [illegible] Run Number 2

Analyst [illegible]

Run Time		Sample Tubes					Process Side Flow Rate			Product Analysis Results										Output Data Calculated								Cost Proc.	Remarks
																	Gas Samples			NH2Cl Yield (%) Based On				Hydrazine Yield (%) Based On					
Cond. Min.	Θ Cond. Min.	Y-1	Y-2	Y-3	Y-4	Y-5	Cl_2 g/min	NH_3 g/min	Product ml/min	NH_3 g/l	Caustic g/l	S.P. g/l	Hydrazine g/l	NaOH g/l	Cl^- g/l	pH	NH_3	Colorimetric NH_2Cl	U.V. NH_2Cl	Total Cl^-	Colorimetric	U.V.	Hydrazine g/min	Cl^-	Cl_2 Feed	Colorimetric NH_2Cl	U.V. NH_2Cl		
					[illegible]				[illegible]											58.21	[illegible]			[illegible]	[illegible]				
					[illegible]				[illegible]											[illegible]				[illegible]	[illegible]				
					[illegible]				[illegible]											[illegible]				[illegible]	[illegible]				
					[illegible]				[illegible]											[illegible]				[illegible]	[illegible]				
					[illegible]				[illegible]											[illegible]				[illegible]	[illegible]				
					[illegible]				[illegible]											[illegible]	[illegible]			[illegible]	[illegible]				
					[illegible]				[illegible]											[illegible]				[illegible]	[illegible]				
					[illegible]				[illegible]											[illegible]				[illegible]	[illegible]				
					[illegible]				[illegible]											[illegible]				[illegible]	[illegible]				
					[illegible]				[illegible]											[illegible]				[illegible]	[illegible]				
					[illegible]				[illegible]											[illegible]	[illegible]			[illegible]	[illegible]				
					[illegible]				[illegible]											[illegible]				[illegible]	[illegible]				
					[illegible]				[illegible]											[illegible]				[illegible]	[illegible]				
					[illegible]				[illegible]											[illegible]				[illegible]	[illegible]				
					[illegible]				[illegible]											[illegible]				[illegible]	[illegible]				
					[illegible]				[illegible]											[illegible]	[illegible]			[illegible]	[illegible]				
					[illegible]				[illegible]											[illegible]				[illegible]	[illegible]				
					[illegible]				[illegible]											[illegible]				[illegible]	[illegible]				
					[illegible]				[illegible]											[illegible]				[illegible]	[illegible]				
					[illegible]				[illegible]											[illegible]				[illegible]	[illegible]				
					[illegible]				[illegible]											[illegible]	[illegible]			[illegible]	[illegible]				
					[illegible]				[illegible]											[illegible]				[illegible]	[illegible]				
					[illegible]				[illegible]											[illegible]				[illegible]	[illegible]				
					[illegible]				[illegible]											[illegible]				[illegible]	[illegible]				
					[illegible]				[illegible]											[illegible]				[illegible]	[illegible]				

BASED ON NaOH MASS BALANCE

UDMH

Q-3 Chemical Analysis
Date 2/28 Run Number 2
Analyst Garth

Run Time: Cont. Min.	# Cond. Min.	Sample Taken: FF Vol	Y-2	Y-3	Y-4	Process Data Flow Rate: N_2	Cl_2 g/min	NH_3 g/min	Product ml/min	Product Analysis Results: NH_3 g/l	Amine g/l	[illegible] g/l	Hydrazine g/l	NaOH g/l	Cl^- g/l	pH	Gas Samples: NH_3	Iodometric NH_2Cl	U.V. NH_2Cl	NH_2Cl Yield % Based On: Total Cl^-	Iodometric	U.V.	Output Data Calculated: Hydrazine g/min	Hydrazine Yield % Based On: Cl^-	Cl_2 Feed	Iodometric NH_2Cl	U.V. NH_2Cl	Calc. Prec.	Remarks
60	15	1				[illegible]	37.5	99.2	516					.126															[illegible] .125
75	30				2.2					[illegible]	[illegible]	[illegible]	26.1	[illegible]	19.95					59.23			14.70	76.25	96.34				
90	45				3.2					[illegible]	[illegible]	[illegible]	26.7	[illegible]	20.[illegible]					67.77			15.65	72.76	[illegible]				
		9												.112															
105	60				5.2					[illegible]	[illegible]	[illegible]	24.7	[illegible]	20.3[illegible]					63.63			14.97	71.70	95.62				
120	75				6.2					[illegible]	[illegible]	[illegible]	29.6	[illegible]	21.57					62.90			17.35	86.12	97.67				
135	90				7.2					[illegible]	[illegible]	[illegible]	26.2	[illegible]	20.93					63.94			15.20	90.39	82.63				ρ=1.025
		9												.112															
150	15	9				[illegible]								.139															
165	30				[illegible]					[illegible]	126.5	[illegible]	23.1	72.20	19.53					64.15			13.[illegible]	69.77	98.67				
180	45				[illegible]					[illegible]	[illegible]	[illegible]	23.1	70.31	19.26					64.27			12.[illegible]	78.96	98.67				
		12												.12[illegible]															
195	60				[illegible]					[illegible]	[illegible]	142.1	22.5	72.29	19.49					61.07			13.77	71.09	98.91				
210	75				[illegible]					[illegible]	170.7	[illegible]	22.5	69.[illegible]	20.3[illegible]					63.58			12.71	64.27	98.91				
225	90				[illegible]					[illegible]	[illegible]	[illegible]	22.7	[illegible]	19.72					61.73			13.80	62.37	94.93				ρ=1.010
		14												.101															
240	15	15				[illegible]		62.9						.109															
255	30				[illegible]					[illegible]	132.3	[illegible]	19.3	74.55	19.85					82.93			14.60	75.96	96.37				
270	45				[illegible]					73.3	131.0	[illegible]	20.0	[illegible]	18.91					83.10			12.81	65.43	98.71				
		20												.106															
285	60				[illegible]					[illegible]	[illegible]	[illegible]	20.6	75.69	19.18					59.98			12.07	63.98	98.05				
300	75				[illegible]					[illegible]	[illegible]	[illegible]	20.6	73.93	19.24					60.86			12.07	63.07	98.05				
315	90				[illegible]					[illegible]	[illegible]	[illegible]	21.8	[illegible]	20.71					63.53			12.77	63.33	98.27				ρ=1.027
		24												.101															
330	15	25				[illegible]								.106															
345	30				[illegible]					[illegible]	[illegible]	[illegible]	20.6	[illegible]	19.33					82.36			13.07	66.91	98.05				
360	45				[illegible]					[illegible]	[illegible]	[illegible]	17.8	[illegible]	19.95					96.90			10.48	74.96	[illegible]				
		25												.105															
375	60				[illegible]					[illegible]	131.1	[illegible]	19.6	81.1	19.61					96.86			[illegible]	89.96	86.97				
390	75				[illegible]					[illegible]	[illegible]	[illegible]	19.9	87.93	15.70					93.95			11.87	92.95	96.53				ρ=1.026

Q-3 Chemical Analysis
Date 2/29 Run Number 2
Analyst Garth

Run Time: Cont. Min.	# Cond. Min.	Sample Taken: FF Vol	Y-2	Y-3	Y-4	Process Data Flow Rate: N_2	Cl_2 g/min	NH_3 g/min	Product ml/min	Product Analysis Results: NH_3 g/l	Amine g/l	[illegible] g/l	Hydrazine g/l	NaOH g/l	Cl^- g/l	pH	Gas Samples: NH_3	Iodometric NH_2Cl	U.V. NH_2Cl	NH_2Cl Yield % Based On: Total Cl^-	Iodometric	U.V.	Output Data Calculated: Hydrazine g/min	Hydrazine Yield % Based On: Cl^-	Cl_2 Feed	Iodometric NH_2Cl	U.V. NH_2Cl	Calc. Prec.	Remarks
[illegible]	90				[illegible]	[illegible]	37.5	58.8	536	55.5	[illegible]	[illegible]	17.8	90.97	15.69					99.04			10.53	62.09	82.91				
		32												.126															
[illegible]	15	33				39.1		[illegible]						.157															
[illegible]	30				[illegible]					[illegible]	100.7	[illegible]	21.9	82.98	19.71					61.82			12.93	65.50	90.05				
[illegible]	45				[illegible]					[illegible]	122.4	146.9	23.5	79.27	20.46					62.80			13.77	65.41	92.41				
		34												.124															
[illegible]	60				31.2					72.4	129.3	[illegible]	20.2	70.36	17.39					99.20			11.94	68.85	87.31				
[illegible]	75				[illegible]					[illegible]	132.4	[illegible]	23.5	67.27	15.91					69.10			13.77	73.45	93.91				ρ=1.029
[illegible]	90				[illegible]					[illegible]	125.1	[illegible]	22.7	66.36	18.95					59.30			13.80	70.70	91.52				
		[illegible]												.121															

(D-1 Process)

UDMH/MMH PLANT RUN DATA SHEETS

____ MMH, Date 2-28-78, Test No 2
X UDMH, Test Conductor T. C. Shupert
Page 1 Of ____

Run Time Cont Min.	Run Time @ Cond Min.	Test Matrix Point	1st Stage Feed Rate Cl_2 g/Min	1st Stage Feed Rate NH_3 g/Min	1st Stage Feed Rate N_2 g/Min	2nd Stage Feed Rate NAOH ML/Min	2nd Stage Feed Rate Water ML/Min	2nd Stage Feed Rate Amine ML/Min	System Pressures T.P. Psig	System Pressures E.P. Psig	System Pressures Cont. Psig	System Pressures Spool Psig	Stirrer RPM x 100	Cont. Level Liters	NAOH Level Inches	Sample Taken Y-1	Sample Taken Y-2	Sample Taken Y-3	Sample Taken Y-4	Sample Taken Y-5	Vibrator Freq. Min.	Vibrator Force PSI	Flow Rates Product ML/Min	Flow Rates Vent g/Min	T.P. Jacket Flow Rate Upper GPM	T.P. Jacket Flow Rate Lower GPM	Electrostatic Precipitator AC Volts	Electrostatic Precipitator AC Amps	Electrostatic Precipitator Spark Min	Electrostatic Precipitator DC Kilovolts	Electrostatic Precipitator DC mA
Pre start			6.0	[illegible]	49.4	[illegible]	266.3	[illegible]	1.8	1.4	0.1	2.0	2100	8							2	min.			3	3					
5	[illegible]	[illegible]	27.5	59.5	49.4	148.3	266.3	←	1.3	2.0	0.2	2.0															52	0.8	20	35	1.0
15			27.5	59.5	49.9	148.3	266.3	←	[illegible]	1.6	0.8	1.5															52	0.8	20	35	1.0
50	5	A	27.5	59.0	49.4	148.3	266.3	116.7	1.8	1.5	0.2	1.5															53	0.8	20	35	1.0
60	15	A	27.5	59.0	49.4	148.3	266.3	116.7	1.5	1.7	[illegible]	1.9								1							52	0.8	20	35	0.9
75	30	A	27.5	59.2	49.4	148.3	266.3	116.7	2.3	2.5	[illegible]	0.6							2.2								54	0.8	20	35	0.9
90	45	A	27.5	59.4	49.4	148.3	266.3	116.7	2.8	2.5	0.2	0.5							2.2	4							54	0.8	20	35	0.9
105	60	A	27.5	59.5	49.4	148.3	266.3	116.7	2.65	2.3	0.3	0.45							5.2								54	0.9	20	35	0.9
120	75	A	27.5	59.5	49.4	148.3	266.3	116.7	2.45	2.85	0.2	0.4							6.2								54	0.8	20	35	0.9
135	90	A	27.5	59.2	49.4	148.3	266.3	116.7	2.8	2.6	0.2	0.5							7.2	8							55	0.8	20	35	0.9
155	15	B	27.5	59.2	14.8	148.3	266.3	116.7	1.8	2.0	0.2	0.5								9							58	0.8	20	35	0.4
170	30	B	27.5	59.2	14.8	148.3	266.3	116.7	1.8	2.1	0.1	1.0							10.5								54	0.8	20	35	0.9
185	45	B	27.5	59.2	14.8	148.3	266.3	116.7	1.9	2.1	0.2	2.0							10.2	12							53	0.8	20	35	0.9
200	60	B	27.5	59.8	14.8	148.3	266.3	[illegible]	2.1	2.5	0.5	2.0							11.4								53	0.9	20	35	0.6
215	75	B	27.5	59.8	14.8	148.3	266.3	116.7	2.3	2.5	0.2	2.5							14.2								53	0.9	20	35	0.9
230	90	B	27.5	59.8	14.8	148.3	266.3	116.7	2.6	2.7	◆	2.6							15.4	16							53	0.9	20	35	0.9
245	15	C	27.5	53.9	49.4	148.3	266.3	116.7	2.1	2.4	◆	2.8								17							53	0.9	20	35	0.9
265	30	C	27.5	53.8	49.4	148.3	266.3	116.7	2.4	2.7	0.4	2.7							18.3								53	0.8	20	35	1.0
280	45	C	27.5	53.8	49.4	148.3	266.3	116.7	2.5	2.7	0.1	2.7							19.2	20							52	0.8	20	35	0.8
295	60	C	27.5	53.8	49.4	148.3	266.3	116.7	2.6	2.8	0.2	2.8							24.2								53	0.8	20	35	0.9
310	75	C	27.5	53.8	49.4	148.3	266.3	116.7	2.7	2.9	0.2	2.8							27.2								58	0.8	20	35	1.0
325	90	C	27.5	53.8	49.4	148.3	266.3	116.7	2.8	2.9	0.2	2.7							23.5	24							58	0.8	20	35	1.0
345	15	D	27.5	53.8	[illegible]	148.3	266.3	116.7	1.8	2.0	0.2	2.0								25							52	0.8	20	34	1.0
360	30	D	27.5	53.8	14.8	148.3	266.3	116.7	2.0	2.1	0.1	2.1							26.4								53	0.8	20	34	1.0
375	45	D	27.5	53.8	14.8	148.3	266.3	116.7	1.6	1.7	0.2	1.65							27.3	28							53	1.3	20	35	1.9
390	60	D	27.5	53.8	14.8	148.3	266.3	116.7	2.0	2.1	0.2	2.1							29.2								56	1.8	20	30	2.2
405	75	D	27.5	53.8	14.8	148.3	266.3	116.7	2.4	2.5	0.2	2.5							30.4								58	1.2	20	29	1.8

(D-1 Process)

UDMH/MMH PLANT RUN DATA SHEETS

____ MMH, Date 2/28/78, Test No 2
✓ UDMH, Test Conductor J.C. Shupert
Page ____ Of ____

Run Time Cont Min.	Run Time @ Cond Min.	Test Matrix Point	1st Stage Feed Rate Cl_2 g/Min	1st Stage Feed Rate NH_3 g/Min	1st Stage Feed Rate N_2 g/Min	2nd Stage Feed Rate NAOH ML/Min	2nd Stage Feed Rate Water ML/Min	2nd Stage Feed Rate Amine ML/Min	System Pressures T.P. Psig	System Pressures E.P. Psig	System Pressures Cont. Psig	System Pressures Spool Psig	Stirrer RPM x 100	Cont. Level Liters	NAOH Level Inches	Sample Taken Y-1	Sample Taken Y-2	Sample Taken Y-3	Sample Taken Y-4	Sample Taken Y-5	Vibrator Freq. Min.	Vibrator Force PSI	Flow Rates Product ML/Min	Flow Rates Vent g/Min	T.P. Jacket Flow Rate Upper GPM	T.P. Jacket Flow Rate Lower GPM	Electrostatic Precipitator AC Volts	Electrostatic Precipitator AC Amps	Electrostatic Precipitator Spark Min	Electrostatic Precipitator DC Kilovolts	Electrostatic Precipitator DC mA
420	90	D	27.5	53.8	14.8	148.3	266.3	116.7	2.2	2.3	0.2	2.3	2100	8					31.2	32	2	min			3	3	63	1.7	20	33	2.2
455	15	E	27.5	33.0	29.7	148.3	266.3	116.7	1.4	1.6	0.3	1.6								33							65	2.0	20	32	2.8
470	30	E	27.5	33.0	29.7	148.3	266.3	116.7	1.6	1.7	0.4	1.8							34.1								62	1.0	20	35	1.4
485	45	E	27.5	33.0	29.7	148.3	266.3	116.7	1.7	1.8	0.3	1.8							35.2	36							55	0.9	20	35	1.2
500	60	E	27.5	33.0	29.7	148.3	266.3	116.7	1.5	1.5	0.2	1.5							37.2								55	1.0	20	35	1.2
515	75	E	27.5	33.0	29.7	148.3	266.3	116.7	1.65	1.8	0.1	1.8							38.1								54	0.9	20	35	1.1
530	90	E	27.5	33.0	29.7	148.3	266.3	116.7	1.8	2.0	0.4	2.0							39.2	40							58	1.2	20	34	1.1
	15	[illegible]																													

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(D-2 Temperatures)
Date 2-28-78 Run No. 2
TC #29 Extended 2

Run Time		Temperatures - °F Refer To Instrumentation List 4 Rev.																																												
Cont.	@ Cond.	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18	19	20	21	22	23	24	25	26	27	28	29	30	31	32	33	34	35	36	37	38	39	40	41	42	43	44	45
Pre-test		[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]			
35		[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]			
50	5	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]			
60	15	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]			
75	30	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]			
90	45	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]			
105	60	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]			
120	75	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]			
135	90	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]			
155	15	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]			
170	30	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]			
185	45	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]			
200	60	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]			
215	75	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]			
230	90	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]			
245	15	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]			
265	30	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]			
280	45	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]			
295	60	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]			
310	75	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]			
325	90	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]			
345	15	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]			
360	30	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]			
375	45	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]			
390	60	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]			
405	75	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]			

TC #29 is Retractable probe - Recorded in #29 when Retracted, Recorded in #44 when extended.

(D-2 Temperatures)
Date 2-28-78 Run No. 2

Run Time		Temperatures - °F Refer To Instrumentation List 4 Rev.																																												
Cont.	@ Cond.	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18	19	20	21	22	23	24	25	26	27	28	29	30	31	32	33	34	35	36	37	38	39	40	41	42	43	44	45
420	90	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]			
455	15	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]			
470	30	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]			
485	45	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]			
500	60	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]			
515	75	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]			
	15																																													

(D-4 Test Conductors Notes)

Date 2-28-78 Run No. 2

Run Cont.	Time @ Cond.	Test Conductor's Notes
0710 hours		Caustic at [illegible]% - adding 50% to adjust.
10		No amine flow - adding heat to bottle
36		Dump NH4Cl; still working amine problem
45		On line - with Amine - Run Time clock started
60	15	Dump NH4Cl
95		Dump NH4Cl, caustic at 30.5%
127	32	TC #29 stuck in gas stream ~2"; will be unable to get more data
150	10	Dump NH4Cl
152	12	Change NH3 bottles
157	17	Hopper chopper stopped; upper valve not operating on discharge.
182	42	Upper valve now operational; completed dump; working on hopper chopper
184		hopper chopper now working
228		Dump NH4Cl; caustic at 30%
263		Dump NH4Cl.
295		Dump NH4Cl
300	0	Dump NH4Cl; upper valve not closing; working problem
335	.	Upper valve working, seems to [illegible] [illegible] arcing and [illegible]
381		Dump NH4Cl; upper valve still needs tickling; caustic 30%; EP arching above 29 kV, 1.9 mA

(D-4 Test Conductors Notes)

Date 2-28-78 Run No. 2

Run Cont.	Time @ Cond.	Test Conductor's Notes
425		Dump NH4Cl; playing with valve again
455		EP is continuing to act up; damn [illegible] gonna run 480 min even if EP [illegible]
460		EP arching at 32, backed down to 30
465		EP back at 38 kV and 1.5 mA
485		Dump NH4Cl
545		Shut down; cooldown not practicable in reasonable period of time; [illegible] especially [illegible]; [illegible] [illegible]

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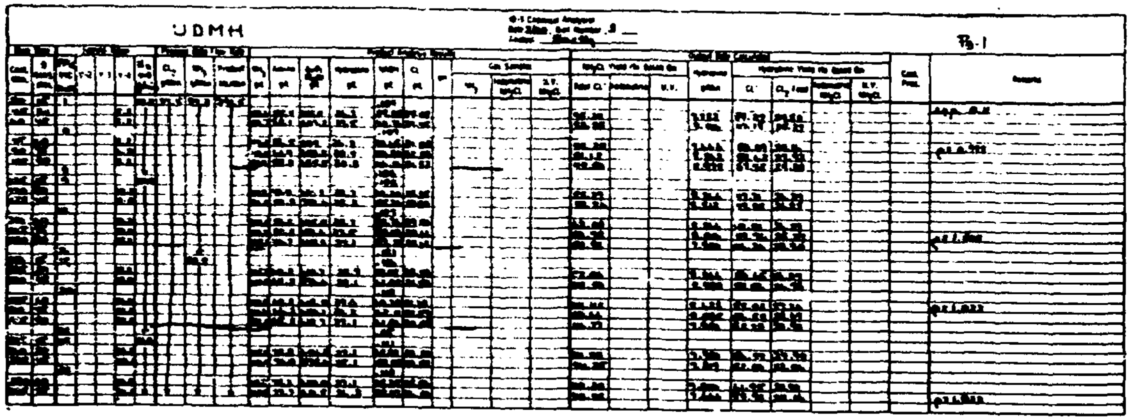
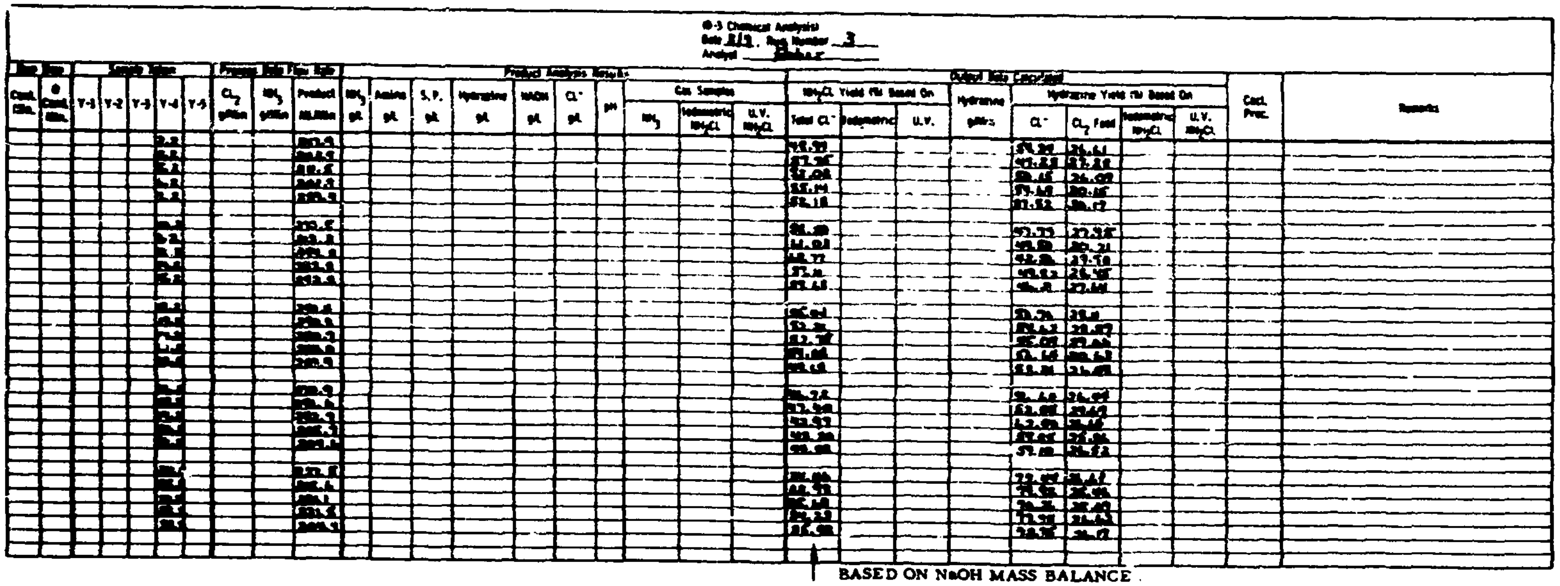


UDMH

Θ-3 Chemical Analysis

Fig. 1



Θ-3 Chemical Analysis
Date 2/13, Run Number 3
Analyst [illegible]

Cond. Min.	Θ Cond. Min.	Y-1	Y-2	Y-3	Y-4	Y-5	Cl_2 g/Min	NH_3 g/Min	Product ml/Min	NH_3 g/l	Amine g/l	S.P. g/l	Hydrazine g/l	NaOH g/l	Cl^- g/l	pH	Gas Samples NH_3	Gas Samples Iodometric NH_2Cl	Gas Samples U.V. NH_2Cl	NH_2Cl Yield (%) Based On Total Cl^-	NH_2Cl Yield (%) Based On Iodometric	NH_2Cl Yield (%) Based On U.V.	Hydrazine g/Min	Hydrazine Yield (%) Based On Cl^-	Hydrazine Yield (%) Based On Cl_2 Feed	Hydrazine Yield (%) Based On Iodometric NH_2Cl	Hydrazine Yield (%) Based On U.V. NH_2Cl	Calc. Prec.	Remarks

↑ BASED ON NaOH MASS BALANCE

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B-3 Chemical Analysis
Date 3/29 Run Number 3
Analyst [illegible]

Pg. 2

Run Time		Sample Taken					Process Rate Flow Rate			Product Analysis Results							Gas Samples			Output Data Calculated								Calc. Prec.	Remarks
																				NH2Cl Yield % Based On			Hydrazine	Hydrazine Yield % Based On					
Cond. Min.	@ Cond. Min.	[illegible] Vol.	Y-2	Y-3	Y-4	NH3 temp °C	Cl2 g/Min	NH3 g/Min	Product ML/Min	NH3 g/L	Amine g/L	Sp. Gr.	Hydrazine g/L	NaOH g/L	Cl⁻ g/L	pH	NH3	Iodometric NH2Cl	U.V. NH2Cl	Total Cl⁻	Iodometric	U.V.	g/Min	Cl⁻	Cl2 Feed	Iodometric NH2Cl	U.V. NH2Cl		
[illegible]	[illegible]				[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	23.1	[illegible]	27.13					42.18			7.390	59.03	25.96				
		[illegible]												.101															
[illegible]	[illegible]	[illegible]				[illegible]		[illegible]	[illegible]					.102															
[illegible]	[illegible]				[illegible]					[illegible]	[illegible]	[illegible]	27.5	[illegible]	21.01					36.54			8.480	77.36	27.29				
[illegible]	[illegible]				[illegible]					[illegible]	[illegible]	[illegible]	26.3	[illegible]	20.77					36.04			8.311	75.13	26.28				
		[illegible]												[illegible]															
[illegible]	[illegible]				[illegible]					[illegible]	[illegible]	[illegible]	25.9	[illegible]	[illegible]					[illegible]			9.194	70.47	26.90				
[illegible]	[illegible]				[illegible]					[illegible]	[illegible]	[illegible]	27.5	[illegible]	[illegible]					[illegible]			9.400	77.68	27.89				
[illegible]	[illegible]				[illegible]					[illegible]	[illegible]	[illegible]	27.1	[illegible]	[illegible]					[illegible]			9.304	73.44	26.50				ρ = 1.02
		[illegible]												.105															

B-3 Chemical Analysis
Date ______ Run Number ______
Analyst ______

Run Time		Sample Taken					Process Rate Flow Rate			Product Analysis Results							Gas Samples			Output Data Calculated								Calc. Prec.	Remarks
																				NH2Cl Yield % Based On			Hydrazine	Hydrazine Yield % Based On					
Cond. Min.	@ Cond. Min.	Y-1	Y-2	Y-3	Y-4	[illegible] Y-4	Cl2 g/Min	NH3 g/Min	Product ML/Min	NH3 g/L	Amine g/L	S.P. g/L	Hydrazine g/L	NaOH g/L	Cl⁻ g/L	pH	NH3	Iodometric NH2Cl	U.V. NH2Cl	Total Cl⁻	Iodometric	U.V.	g/Min	Cl⁻	Cl2 Feed	Iodometric NH2Cl	U.V. NH2Cl		
1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18	19	20	21	22	23	24	25	26	27	28		
[illegible]	[illegible]				[illegible]					[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]					63.87			9.399	88.27	29.51				
[illegible]	[illegible]				[illegible]					[illegible]	[illegible]	[illegible]	27.5	[illegible]	[illegible]					64.90			9.66	97.16	30.95				
[illegible]	[illegible]				[illegible]					[illegible]	[illegible]	[illegible]	26.3	[illegible]	[illegible]					63.43			9.339	50.29	28.12				
[illegible]	[illegible]				[illegible]					[illegible]	[illegible]	[illegible]	30.4	[illegible]	[illegible]					64.82			10.68	59.63	33.44				ρ = .995
[illegible]	[illegible]				[illegible]					[illegible]	[illegible]	[illegible]	30.8	[illegible]	[illegible]					63.06			10.32	57.75	30.10				
[illegible]	[illegible]				[illegible]					[illegible]	[illegible]	[illegible]	28.7	[illegible]	[illegible]					64.61			10.08	97.31	31.78				
[illegible]	[illegible]				[illegible]					[illegible]	[illegible]	[illegible]	29.2	[illegible]	[illegible]					65.38			10.26	49.45	32.33				
[illegible]	[illegible]				[illegible]					[illegible]	[illegible]	[illegible]	25.2	[illegible]	[illegible]					58.35			9.959	57.77	29.24				
[illegible]	[illegible]				[illegible]					[illegible]	[illegible]	[illegible]	24.3	[illegible]	[illegible]					61.30			9.536	52.97	26.91				
[illegible]	[illegible]				[illegible]					[illegible]	[illegible]	[illegible]	23.9	[illegible]	[illegible]					62.06			9.356	40.35	26.31				ρ = 1.004
[illegible]	[illegible]				[illegible]					[illegible]	[illegible]	[illegible]	25.5	[illegible]	[illegible]					62.78			9.952	[illegible]	27.34				
[illegible]	[illegible]				[illegible]					[illegible]	[illegible]	[illegible]	21.9	[illegible]	[illegible]					61.06			9.012	[illegible]	25.49				
[illegible]	[illegible]	[illegible]			[illegible]					[illegible]	[illegible]	[illegible]	27.5	[illegible]	[illegible]					64.05			8.660	[illegible]	30.95				
[illegible]	[illegible]				[illegible]					[illegible]	[illegible]	[illegible]	26.3	[illegible]	[illegible]					63.83			9.239	[illegible]	29.45				ρ = 1.032
[illegible]	[illegible]				[illegible]					[illegible]	[illegible]	[illegible]	23.3	[illegible]	[illegible]					[illegible]			9.536	47.38	26.41				
[illegible]	[illegible]				[illegible]					[illegible]	[illegible]	[illegible]	23.9	[illegible]	[illegible]					62.08			9.396	[illegible]	26.46				
[illegible]	[illegible]				[illegible]					[illegible]	[illegible]	[illegible]	23.3	[illegible]	[illegible]					63.92			9.324	[illegible]	30.14				
[illegible]	[illegible]				[illegible]					[illegible]	[illegible]	[illegible]	23.9	[illegible]	[illegible]					[illegible]			9.396	[illegible]	26.46				ρ

NO GOOD - REVISED

@-3 Chemical Analysis
Date ____, Run Number ______
Analyst ______________

Run Time		Sample Taken					Process Data Flow Rate			Product Analysis Results							Gas Samples			Data: Yield Calculated								Lect. Prec.	Remarks
																				NH2Cl Yield (%) Based On			Hydrazine	Hydrazine Yield % Based On					
Cont. Min.	@ Cond. Min.	Y-1	Y-2	Y-3	Y-4	Y-5	Cl_2 g/Min	N_2 g/Min	Product ML/Min	NH_3 g/L	Amine g/L	S.P. g/L	Hydrazine g/L	NAOH g/L	Cl^- g/L	pH	NH_3	Iodometric NH_2Cl	U.V. NH_2Cl	Total Cl^-	Iodometric	U.V.	g/Min	Cl^-	Cl_2 Feed	Iodometric NH_2Cl	U.V. NH_2Cl		
					58.2					[illegible]	72.3	141.8	33.5	86.53	66.8					98.71			3.255	83.41	76.02				ρ = 1.022
					67.3																								
						8.2								.81															
						23								.107															
					90.3																								

NO GOOD - REVISED

(@-1 Process)
X NMH, Date 2-23-72, Test No 2
UDMH, Test Conductor T. C. Shaver
Page ____ Of ____

UDMH/MMH PLANT RUN DATA SHEETS

Run Time		Test Matrix Point	1st Stage Feed Rate			2nd Stage Feed Rate			System Pressures				Stirrer RPM x 100	Cont. Level Liters	NAOH Level Inches	Sample Taken					Vibrator		Flow Rates		T.P. Jacket Flow Rate		Electrostatic Precipitator				
																											AC			DC	
Cont Min.	@ Cond Min.		Cl_2 g/Min	NH_3 g/Min	N_2 g/Min	NAOH ML/Min	Water ML/Min	Amine ML/Min	T.P. Psig	E.P. Psig	Cont. Psig	Spent Psig				Y-1	Y-2	Y-3	Y-4	FF	Freq. Min.	Force PSI	Product ML/Min	Vent g/Min	Upper GPM	Lower GPM	Volts	Amps	Spark Min	Kilovolts	mA
Pre-[illegible]	-07	[illegible]	0	87.8	49.4	[illegible]	149.1	0	1.98	2.1	0.3	2.1	1870	8						0					3	2	59	1.0	10	35	1.4
15	0	A	37.5	88.8	49.8	[illegible]	149	[illegible]	1.76	1.85	◆	1.55															51	0.9	20	35	0.9
30	15	A	37.5	88.8	49.8	64.6	147	70.2	1.8	1.9	◆	1.9								1							51	0.9	20	35	0.8
45	30	A	37.5	88.8	49.8	64.6	147	70.2	1.8	1.9	◆	1.8							9.2								51	0.8	20	35	0.8
60	45	A	37.5	88.8	49.8	65	147	70.2	2.0	2.3	◆	2.3							[illegible]	4							51	0.9	20	35	0.8
75	60	A	37.5	88.8	49.8	65	147	70.2	1.6	1.8	◆	1.8							6.2								51	0.8	20	35	0.8
90	75	A	37.5	88.8	49.8	65	147	70.2	1.9	2.1	◆	2.0							[illegible]								51	0.8	20	35	0.8
105	90	A	37.5	88.8	49.8	65	147	70.2	2.1	2.3	◆	2.3							7.3	8							51	0.8	20	35	0.8
120	15	B	37.5	88.8	49.4	65	147	70.2	2.0	2.1	◆	2.0								9							51	0.9	20	35	0.8
135	30	B	37.5	88.8	49.4	65	147	70.2	2.2	2.9	◆	2.9							[illegible]								51	0.8	20	35	0.8
150	45	B	37.5	88.8	49.4	65	147	70.2	3.3	3.4	◆	3.4							11.3	12							51	0.8	20	35	0.8
165	60	B	37.5	88.8	49.4	◆	◆	◆	3.3	3.5	◆	3.5							[illegible]								51	0.8	20	35	0.8
180	[illegible]	B	37.5	88.8	49.4	65	147	70.2	4.1	4.3	0.2	4.2							12.3								51	0.8	20	35	0.8
195	75	B	37.5	88.8	49.4	65	147	70.2	4.4	4.50	0.15	4.65							[illegible]								51	0.8	20	35	0.8
210	90	B	37.5	88.8	49.4	65	147	70.2	3.5	3.6	0.1	3.55							15.3	16							50	0.75	20	35	0.4
225	15	C	37.5	88.8	49.4	65	147	70.2	2.45	2.36	◆	2.36								17							50	0.75	20	35	0.55
240	30	C	37.5	88.8	49.4	65	147	70.2	2.8	3.0	◆	2.9							18.3								50	0.75	20	35	0.9
255	45	C	37.5	88.8	49.4	65	147	70.2	2.1	2.2	◆	2.2							19.3	20							50	0.75	20	35	0.9
270	60	C	37.5	88.8	49.4	65	147	70.2	2.7	2.9	◆	2.9							21.3								52	0.8	20	35	[illegible]
285	75	C	37.5	53.8	49.4	65	147	70.2	[illegible]	3.2	0.1	3.1							22.3								52	0.8	20	35	0.8
300	90	C	37.5	53.8	49.4	65	147	70.2	2.9	3.1	◆	3.0							23.3	24							52	0.8	20	35	0.9
315	15	D	37.5	53.8	19.8	65	147	70.2	2.1	2.4	◆	2.4								25							52	0.9	20	35	0.95
330	30	D	37.5	53.8	19.8	65	147	70.2	2.9	3.1	◆	3.0							26.3								52	0.9	20	35	1.0

[illegible]

(D-2 Process)

UDMH/MMH PLANT RUN DATA SHEETS

______ MMH, Date ______, Test No ______
______ UDMH, Test Conductor ______
Page ____ Of ____

Run Time Cont Min.	Run Time @ Cond Min.	Test Matrix Point	1st Stage Feed Rate Cl_2 g/Min	1st Stage Feed Rate NH_3 g/Min	1st Stage Feed Rate N_2 g/Min	2nd Stage Feed Rate NAOH ML/Min	2nd Stage Feed Rate Water ML/Min	2nd Stage Feed Rate Amine ML/Min	System Pressures T.P. Psig	System Pressures E.P. Psig	System Pressures Cont. Psig	System Pressures Spent Psig	Stirrer RPM x 100	Cont. Level Liters	NAOH Level Inches	Sample Taken Y-1	Sample Taken Y-2	Sample Taken Y-3	Sample Taken Y-4	Sample Taken Y-5	Vibrator Freq. Min.	Vibrator Force PSI	Flow Rates Product ML/Min	Flow Rates Vent g/Min	T.P. Jacket Flow Rate Upper GPM	T.P. Jacket Flow Rate Lower GPM	Electrostatic Precipitator AC Volts	Electrostatic Precipitator AC Amps	Electrostatic Precipitator Spark Min	Electrostatic Precipitator DC Kilovolts	Electrostatic Precipitator DC mA
375	45	D	33.5	53.8	19.8	65	147	[illegible]	3.0	3.3	◆	3.2	1130	8					[illegible]						3	3	55	1.0	20	35	1.2
390	60	D	33.5	53.8	19.8	65	147	[illegible]	3.0	3.2	◆	3.1							[illegible]	[illegible]							58	1.0	20	35	1.3
405	75	D	33.5	53.8	19.8	65	147	[illegible]	3.4	3.5	◆	3.4							[illegible]	[illegible]							60	1.1	20	35	1.6
415	90	D	33.5	53.8	19.8	65	147	[illegible]	3.45	3.45	◆	3.6							[illegible]	[illegible]							63	1.8	20	34	2.3
440	15	E	33.5	35.9	29.7	65	147	30.2	3.3	3.45	◆	2.85								[illegible]							65	1.8	20	34	2.3
455	30	E	33.5	35.9	29.7	65	147	30.2	2.6	2.9	◆	2.9							[illegible]								62	1.1	20	35	1.6
470	45	E	33.5	35.9	29.7	65	147	30.2	2.3	3.5	◆	2.4							[illegible]	[illegible]							61	1.1	20	35	1.6
485	60	E	33.5	35.9	29.7	65	147	30.2	2.8	2.6	◆	2.5							[illegible]								61	1.1	20	35	1.6
500	75	E	33.5	35.9	29.7	65	147	30.2	2.5	3.1	◆	2.6							[illegible]								60	1.0	20	35	1.4
515	90	E	33.5	35.9	29.7	65	147	30.2	2.5	2.7	◆	3.6							[illegible]	[illegible]							5.1	1.0	20	35	1.4

(D-2 Temperatures)

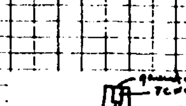
Date 3-22-78 Run No. 2

Temperatures - °F Refer To Instrumentation List 4 Rev.

TC#29 extended)

Run Time Cont.	Run Time @ Cond.	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18	19	20	21	22	23	24	25	26	27	28	29	30	31	32	33	34	35	36	37	38	39	40	41	42	43	44	45
[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	7	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]			
15	0	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	3	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]			
30	15	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	6	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]			
45	30	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	5	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]			
60	45	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	3	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]			
75	60	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	7	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]		[illegible]	
90	75	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	4	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]			
105	90	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	5	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]			
125	15	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	5	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]			
140	30	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	3	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]			
155	45	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	7	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]			
170	60	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	4	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]		[illegible]	
200 [illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	4	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]			
215	75	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	2	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]			
230	90	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	6	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]			
250	15	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	3	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]			
265	30	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	6	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]			
280	45	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	5	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]			
295	60	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	6	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]		[illegible]	
310	75	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	7	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]			
325	90	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	5	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]			
345	15	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	7	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]			
360	30	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	5	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]			

TC 29 is retractable, when recorded in #29 position - retracted, when recorded in 44 position extended



(D-2 Temperatures)

Date ________ Run No. ________

Temperatures - °F Refer To Instrumentation List ______ Rev. ____

Run Cont.	Time @ Cond.	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18	19	20	21	22	23	24	25	26	27	28	29	30	31	32	33	34	35	36	37	38	39	40	41	42	43	44	45
375	45	94	88	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	64	5	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	58	77	58	17	[illegible]	[illegible]	[illegible]	78	[illegible]	[illegible]			
390	60	87	85	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	63	6	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	58	81	58	19	[illegible]	[illegible]	[illegible]	73	[illegible]	[illegible]		105	
405	75	86	87	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	64	4	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	53	79	59	17	[illegible]	[illegible]	[illegible]	74	[illegible]	[illegible]			
415	90	83	85	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	67	7	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	57	78	58	19	[illegible]	[illegible]	[illegible]	78	121	[illegible]			
440	15	87	84	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	41	4	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	57	81	57	21	[illegible]	[illegible]	[illegible]	71	115	[illegible]			
455	30	84	79	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	96	6	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	58	79	57	17	[illegible]	[illegible]	[illegible]	76	115	[illegible]			
470	45	82	78	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	97	6	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	52	79	[illegible]	17	[illegible]	[illegible]	[illegible]	74	113	[illegible]			
485	60	79	76	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	96	4	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	51	78	[illegible]	15	[illegible]	[illegible]	[illegible]	73	113	[illegible]			
500	75	81	76	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	97	7	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	49	79	58	17	[illegible]	[illegible]	[illegible]	78	113	[illegible]			
515	90	82	77	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	96	5	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	52	75	58	17	[illegible]	[illegible]	[illegible]	72	113	[illegible]			

(D-4 Test Conductors Notes)

Date 2-22-78 Run No. 2

Pg 1 of 2

Run Cont.	Time @ Cond.	Test Conductor's Notes
		Pre-run conditions per Run plan #2. [illegible] [illegible] were flagged [illegible] during the conditioning.
		Romarco, [illegible], [illegible] & [illegible]
0800		Caustic at 30%, starting to fill Combustor. Water Temp to heat exchanger in [illegible].
		Found a [illegible] hose inside Combustor - repaired. Temp [illegible] on the
		[illegible] [illegible] pump not operating.
		30% Caustic Pump cuts out.
1000		Changed out [illegible] pump - OK now.
		30% Caustic Pump still cuts out: [illegible] [illegible] - will [illegible] [illegible] [illegible] and [illegible]
		and operate 30% caustic pump at [illegible] [illegible] [illegible] [illegible].
End of a		long day 2-22-78. [illegible] [illegible] 10% caustic Pump and installed. Rebuilt [illegible] pump
		and [illegible] [illegible].
2-23-78		Setting up at [illegible] for Day 2. System purge on at 0730, [illegible] [illegible]. [illegible] [illegible]
		up [illegible] 30% [illegible] pump.
		Caustic at 28.7% - adding 10%. This was caused by the change out of 30% pumps
		and [illegible] of [illegible] in system ([illegible]) for [illegible].
0915		Got on line for Condition 1, [illegible] [illegible] [illegible] [illegible] [illegible] the TIP.
		went to Condition 2
20	5	Caustic at 2%, continuing to add 10%
30	15	Dump [illegible]
60	45	Switching [illegible] bottles
75	60	Dump [illegible]
105	90	Dump NH4Cl; attempting to go back to cond. #1. 10:11
142	82	Dump NH4Cl
163		[illegible] flow of caustic and H2O; working problems, [illegible] in [illegible]; activated heater
172		[illegible] went off. Found problem [illegible] [illegible] [illegible] - [illegible] [illegible] the air supply
		and the [illegible] [illegible] [illegible] [illegible] at [illegible] 125 and Caustic at 60
		[illegible] [illegible] [illegible] [illegible] [illegible] again, [illegible] [illegible] [illegible] [illegible] for
		15 min. Took the [illegible] samples, then shut the [illegible] of caustic [illegible].
200	40	Dump NH4Cl
235	0	Dump NH4Cl
275	40	Dump NH4Cl
317	82	Dump NH4Cl
363		Dump NH4Cl

(D-4 Test Conductor's Notes)

Date 2-25-75 Run No. 3

Pg 2 of 2

Run Cond.	Time @ Cond.	Test Conductor's Notes
[illegible]		Dump NH_4Cl
[illegible]		TC #29 not longer usable; left in retracted position
413		EP down to 35 kV at 2.4 mA; arcing at 35 kV
425		Dump NH_4Cl
430		EP down to 30 kV at 1.6 mA; arcing at 35 kV
[illegible]		Dump NH_4Cl
[illegible]	91	Dump NH_4Cl → [illegible] shut-down

ABBREVIATIONS

AFLC	Air Force Logistics Command
AFRPL	Air Force Rocket Propulsion Laboratories
CA (X_3)	chloramine
CaO	calcium oxide
Cl_2	chlorine
Conc.	concentration
DMA (X_1)	dimethyl amine
°F	degrees fahrenheit
EP	electrostatic precipitator
GPM	gallons per minute
H_2O	water
HCl	hydrochloric acid
IITRI	IIT Research Institute
MMC	Martin Marietta Corporation
m/l	moles/liter
MMA (X_1)	monomethylamine
MMH	monomethylhydrazine
$NaOH$ (X_2)	sodium hydroxide
N_2	nitrogen
NH_3	ammonia
NH_2Cl	chloramine

NH_4Cl	ammonium chloride
SAMSO	Space and Missile Systems Organization
TP	thermal precipitator
UDMH	unsymmetrical dimethylhydrazine