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

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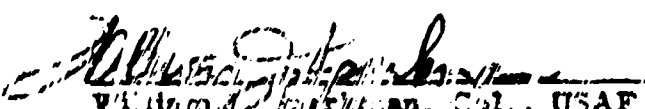
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PREFACE

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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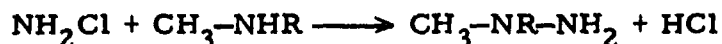
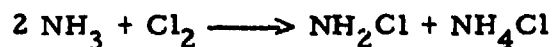
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I. INTRODUCTION

A. OBJECTIVES AND SCOPE

A critical potential shortage of unsymmetrical dimethylhydrazine (UDMH), a rocket fuel, arose in 1973 when the sole source manufacturer 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:



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.

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 $\text{NH}_3 - \text{Cl}_2$ reaction. The program was concluded in February 1978.

After the completion of the urea process study, SAMSO awarded a contract to ITRI 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 ITRI 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,

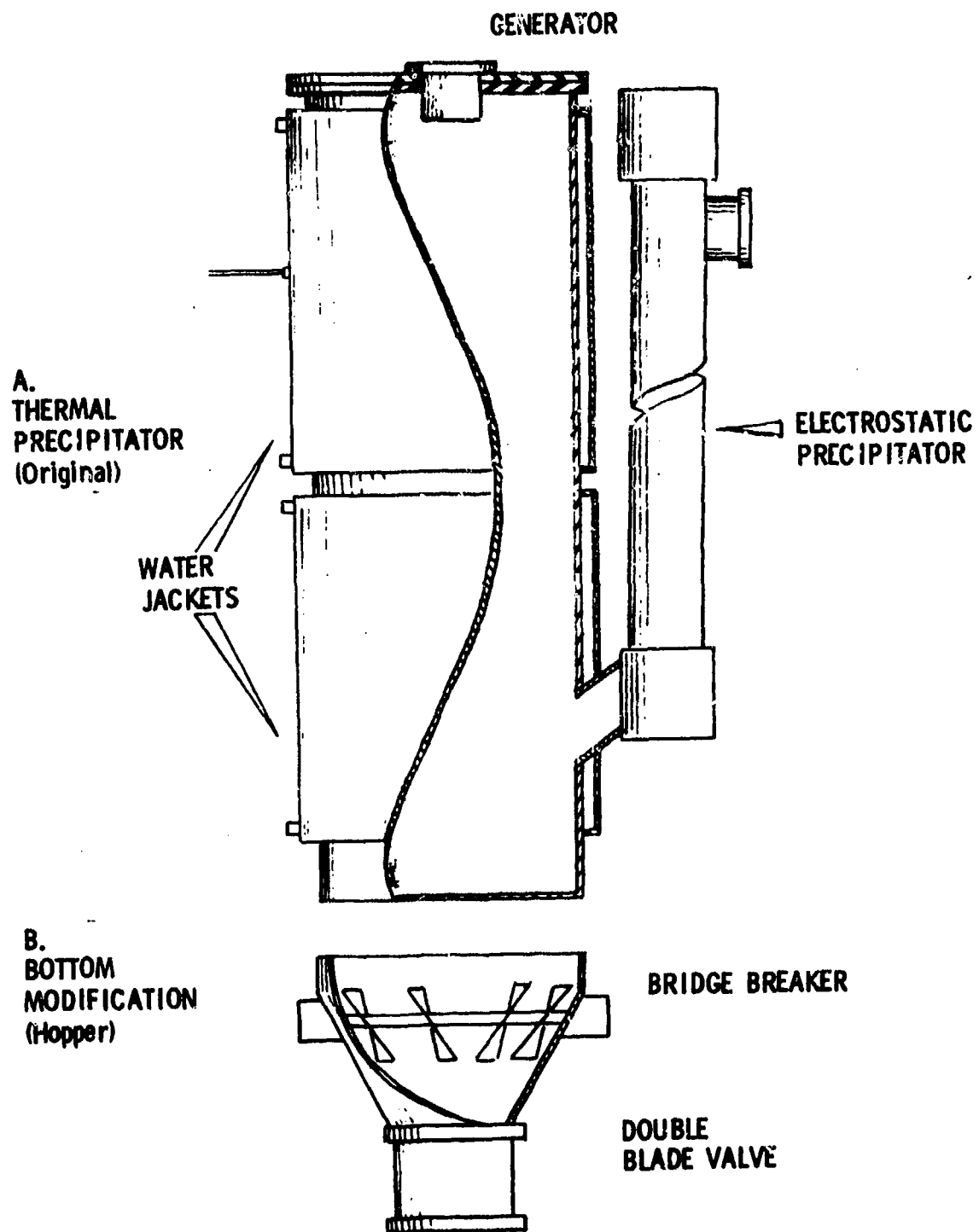


Figure 1. Generator/Precipitator Assembly

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 $\text{NH}_3 - \text{Cl}_2$ 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/ $\text{NaOH}/\text{H}_2\text{O}$; 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 ($\sim 0^\circ\text{C}$), 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/ $\text{NaOH}/\text{H}_2\text{O}$ 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/ $\text{NaOH}/\text{H}_2\text{O}$ 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 bottom was selected. The lower stirrer dispersed the gas; the upper stirrer homogenized the fluid.

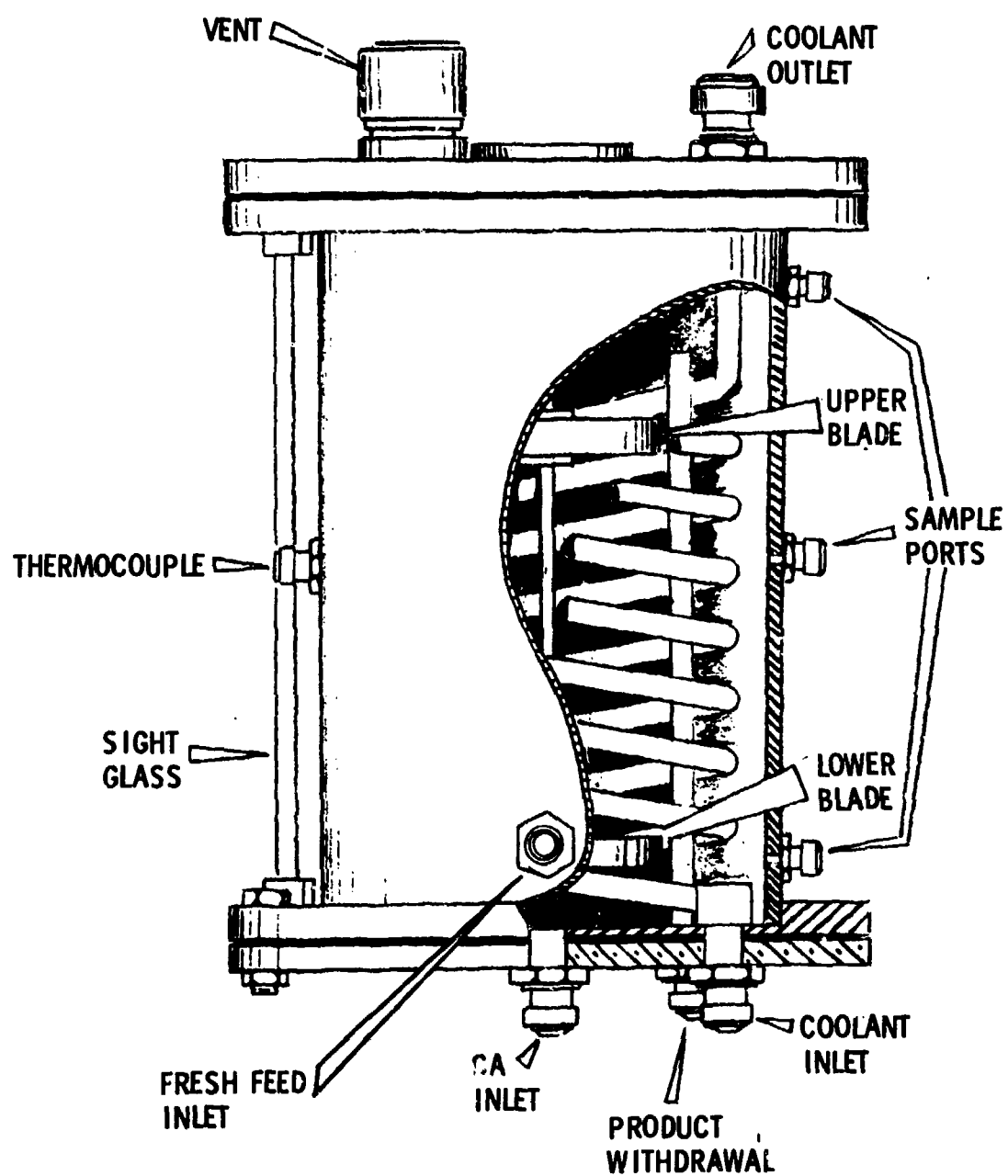


Figure 2. CA-Amine Reactor

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/H₂O 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/H₂O and of MMA/NaOH/H₂O 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/H₂O and MMA/NaOH/H₂O 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

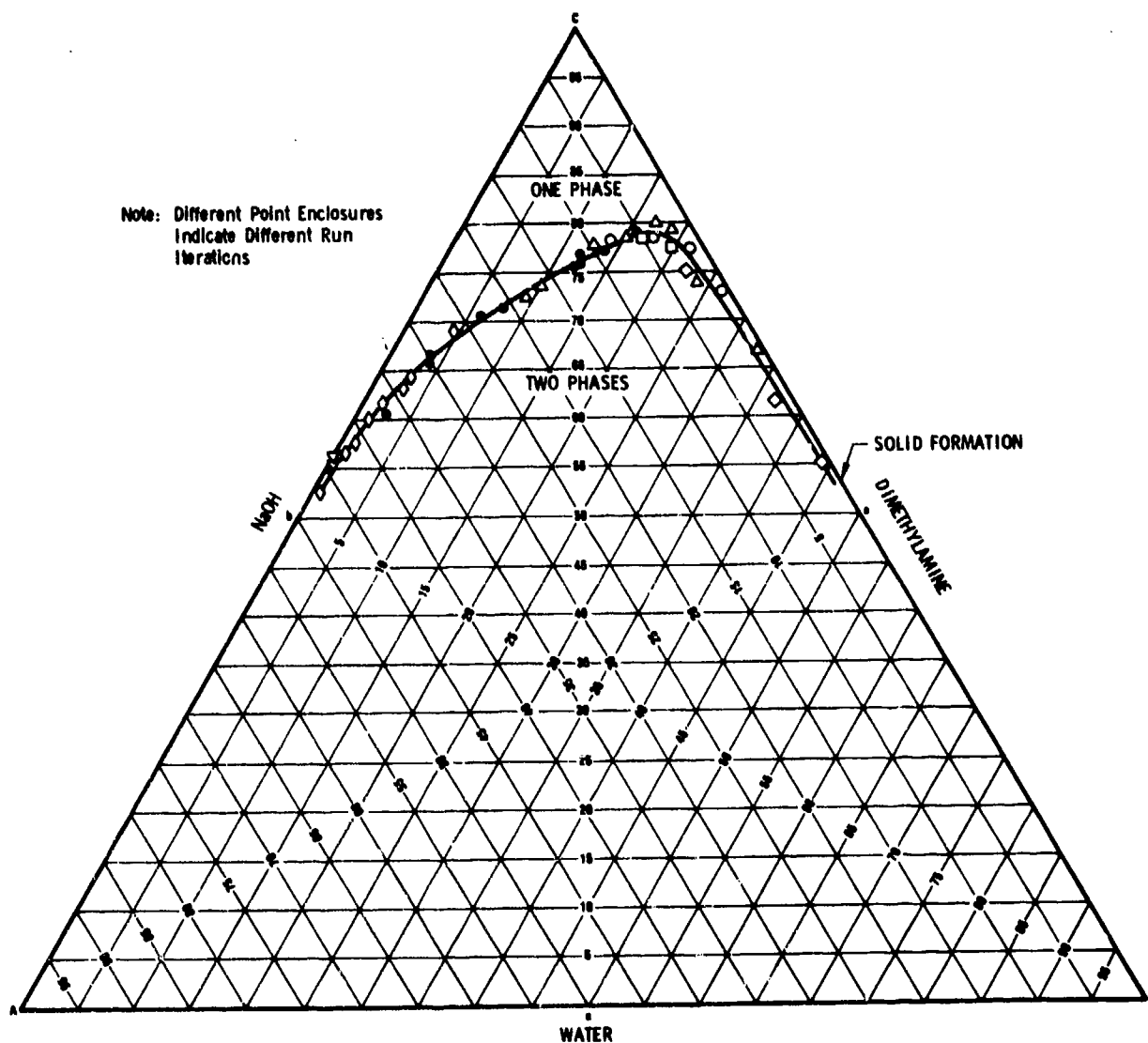


Figure 3. Three-Component Phase Diagram for DMA/NaOH/H₂O
Determined at 15.0°C

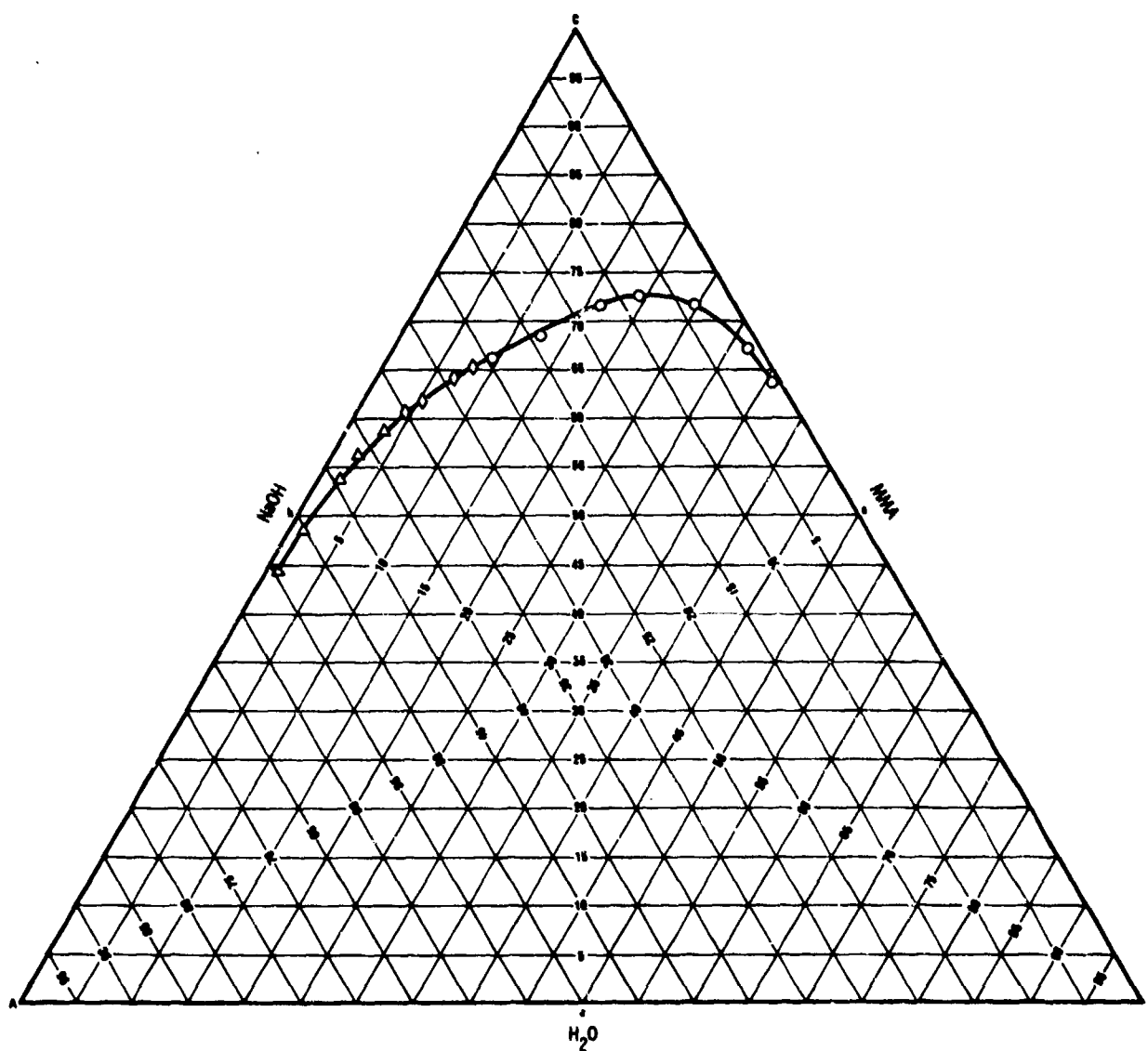


Figure 4. Three-Component Phase Diagram for MMA/NaOH/H₂O
Determined at 15° C

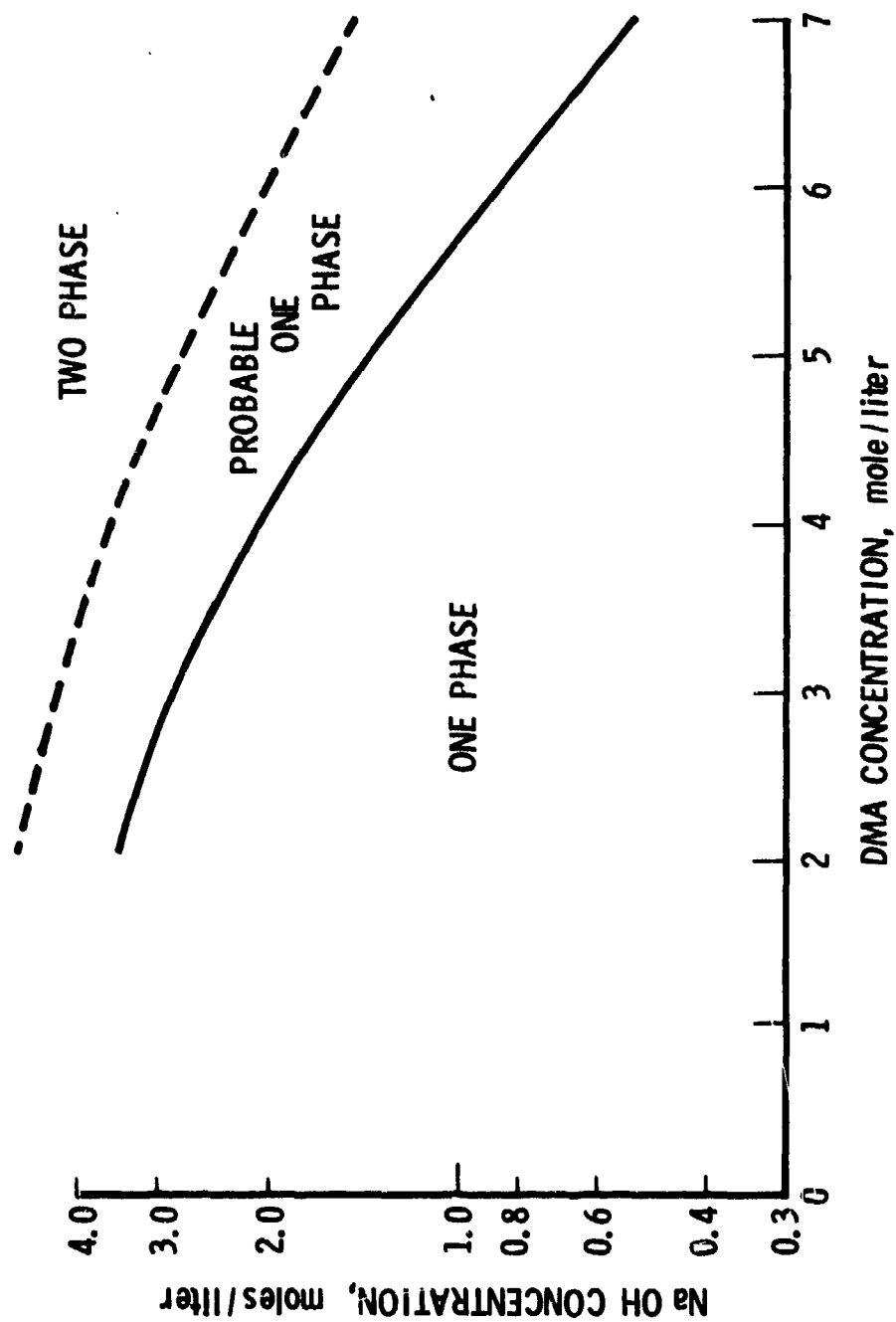


Figure 5. Contactor Product Mixture Homogeneity - UDMH System

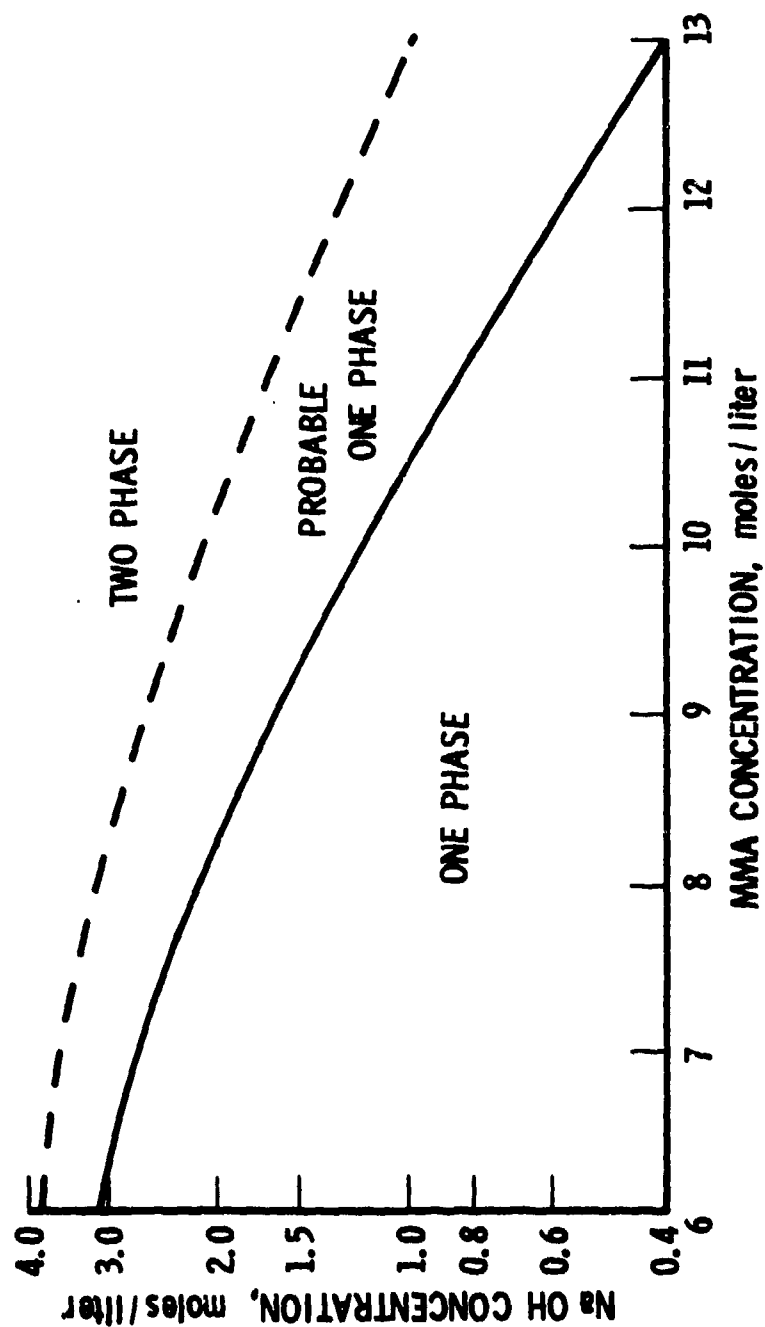


Figure 6. Contactor Product Mixture Homogeneity - MMH System

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 decomposition 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 vacuum 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/H₂O and MMA/NaOH/H₂O 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 NH₃/Cl₂ 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 ITRI 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 ($< 650^\circ\text{F}$). 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	0	+1	+K
NH_3/Cl_2 molar ratio	4	6	8	10	12
N_2/Cl_2 molar ratio	0	1	2	3	4
Cone Temp., $^\circ\text{F}$	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.

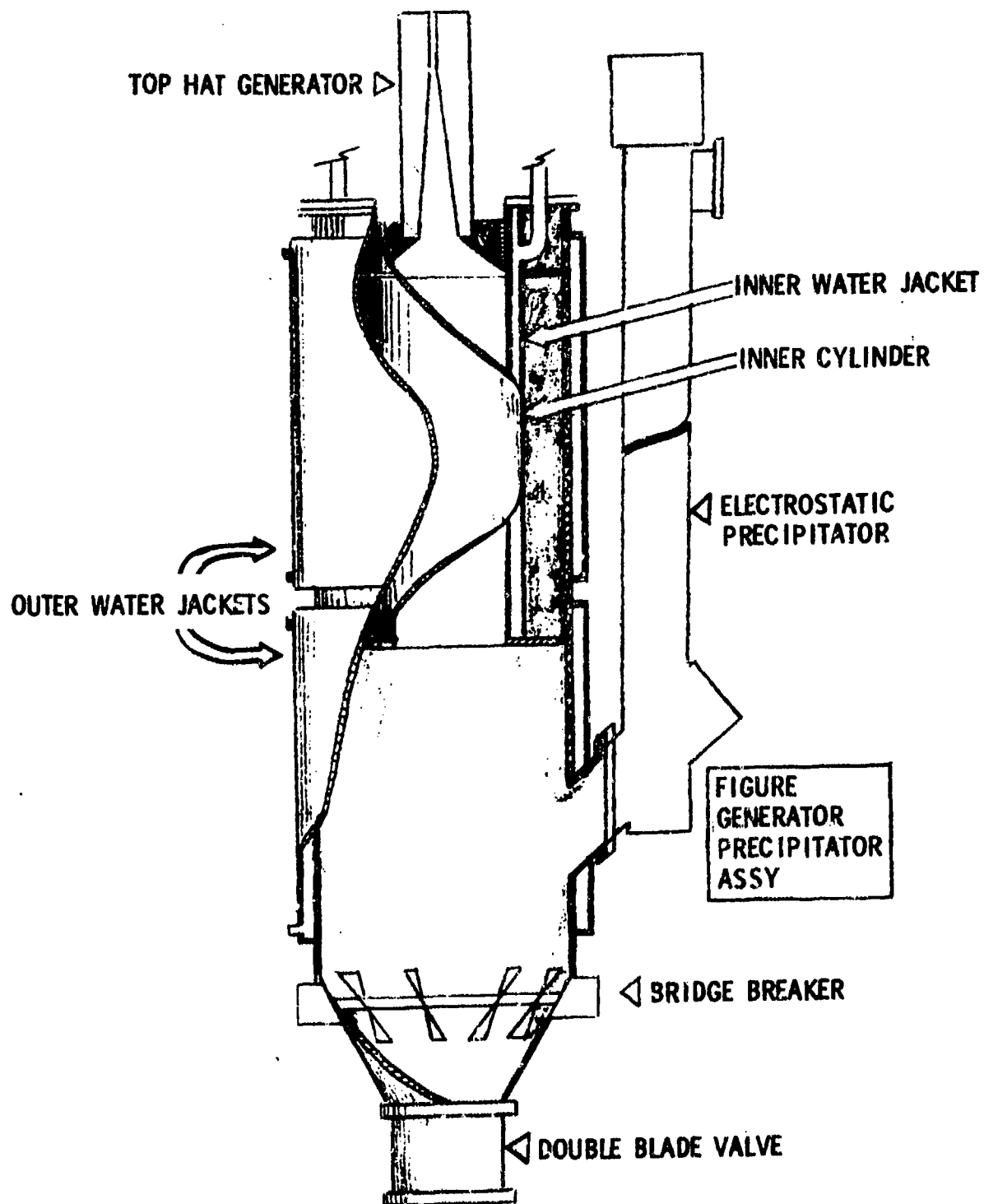


Figure 7. Top Hat Generator/Thermal Precipitator Assembly

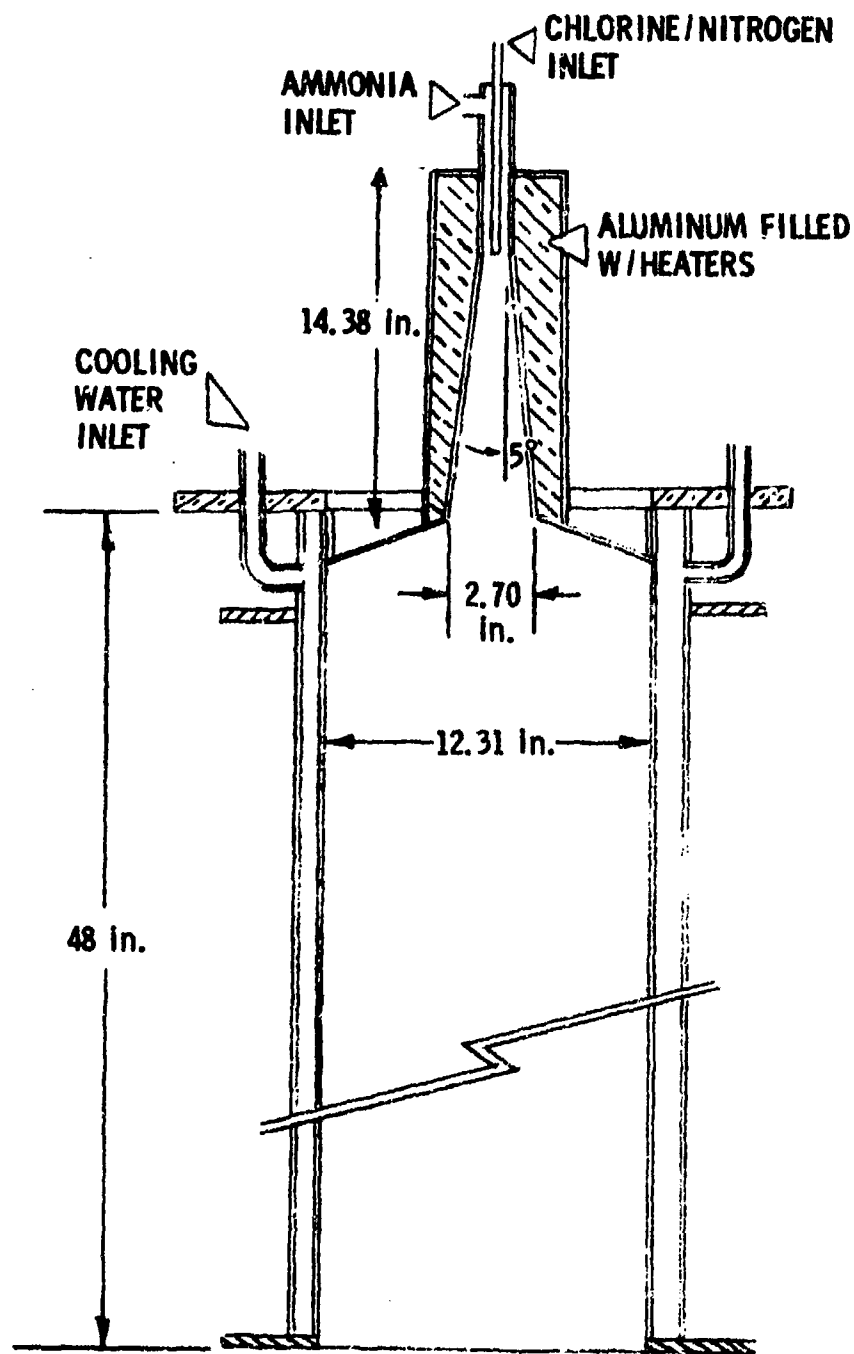


Figure 8. Top Hat Generator Assembly

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 $\text{NH}_3/\text{N}_2/\text{Cl}_2$ 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,

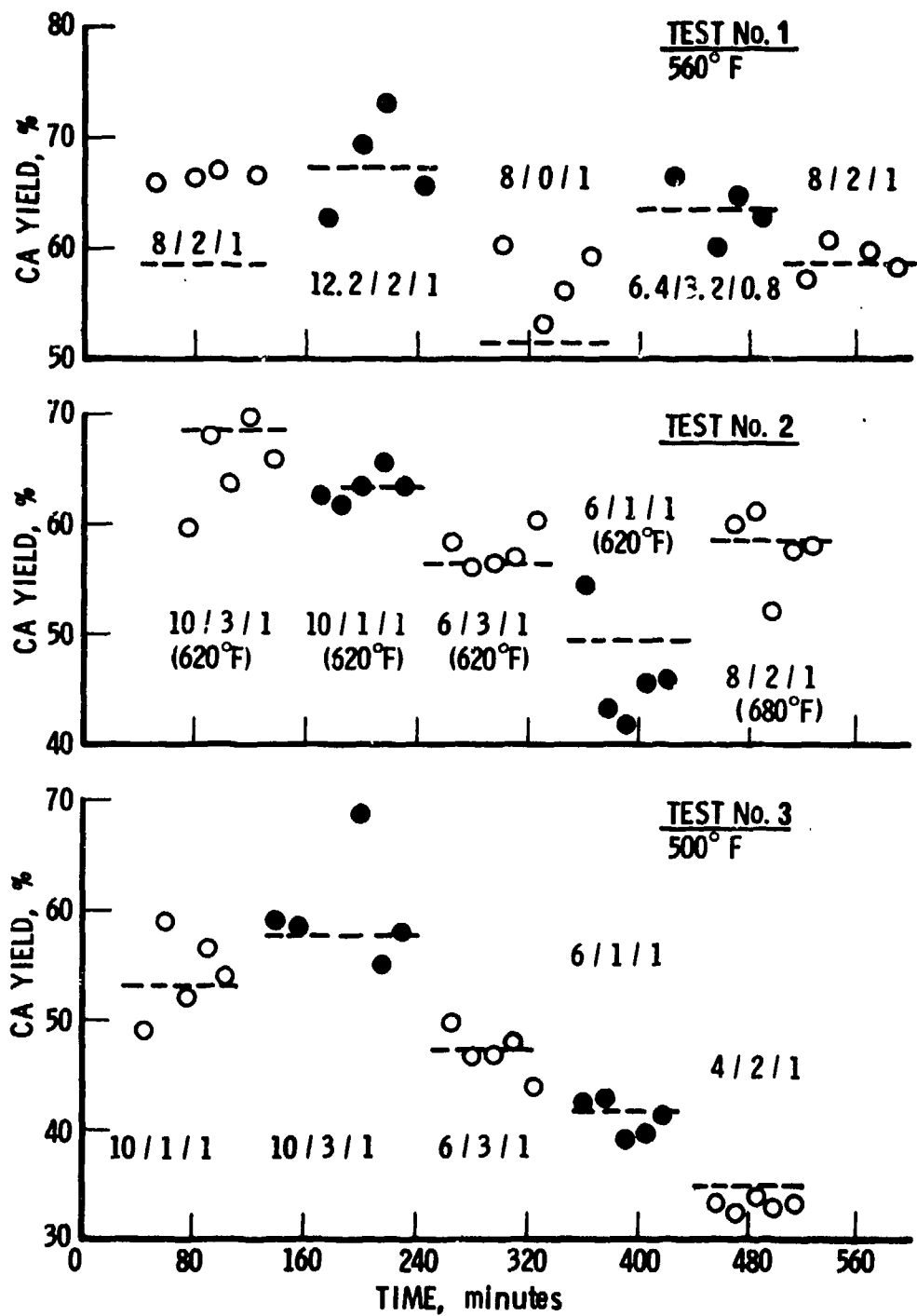


Figure 9. Top Hat Runs, CA Yields vs Run Times

Table 1. CA Generator/Thermal Precipitator Performance

Run No. & 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

^aMolar ratio based on Cl₂ flow of 37.5 g/min

^bInconsistent data. Not used in statistical analyses.

^cBased on Cl₂ 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 CA yield as follows:

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

where, Y_2 = yield in decimal

$$A = a + bT + cT^2$$

$$a = -2.44769530$$

$$b = 0.0103631115$$

$$c = -8.33124808 \times 10^{-6}$$

$$T = \text{cone temperature in } ^\circ\text{F}$$

and where $B\lambda = (\text{NH}_3/\text{Cl}_2) [d + f (\text{N}_2/\text{Cl}_2)]$ 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 on 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 (Appendices 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:

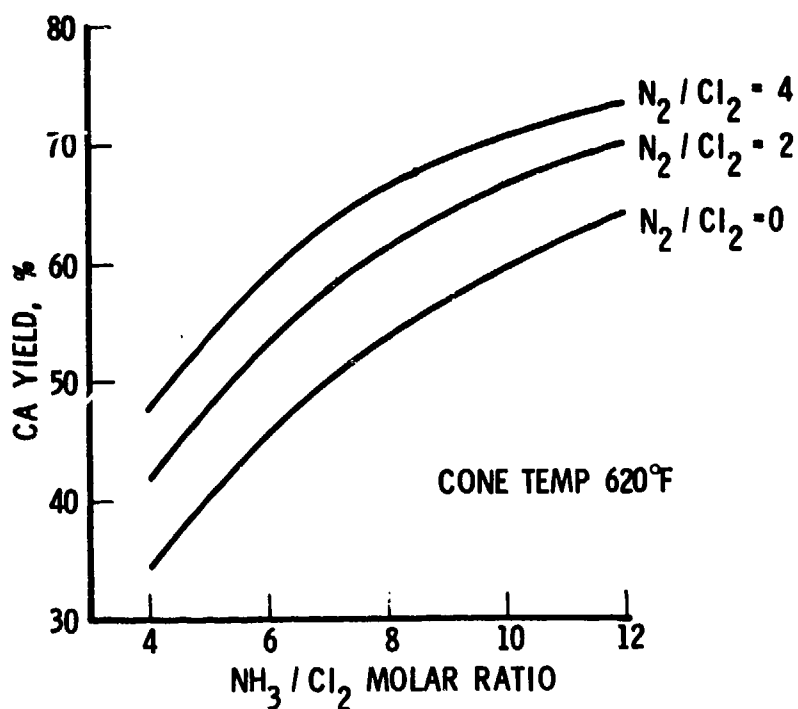


Figure 10. Effect of NH_3/Cl_2 Ratio on CA Generator Performance

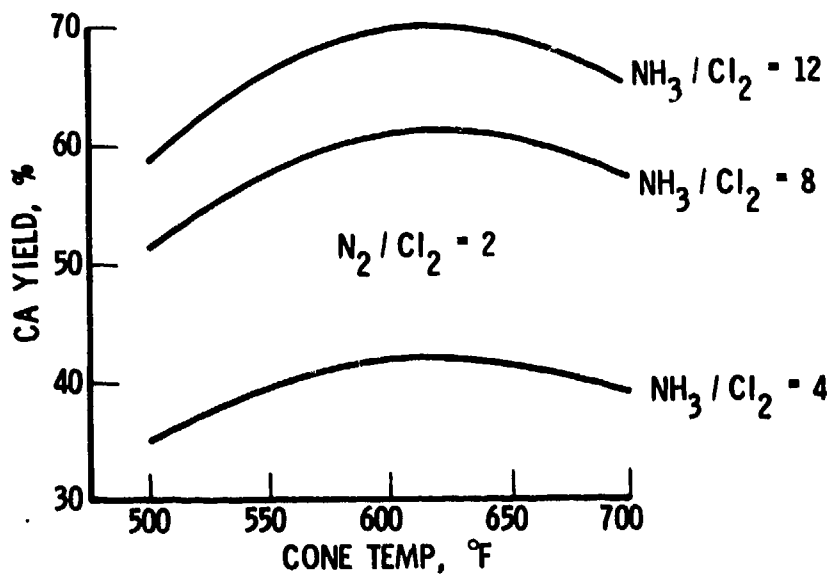


Figure 11. CA Yield vs Generator Cone Temperature

$$\begin{aligned}
 Y_3 = & aX_1X_3 + bX_1X_3^2 + cX_1X_2X_3 + dX_1^2X_2X_3 + eX_1X_2^2X_3 \\
 & + fX_1^2X_3 + gX_1X_2X_3^2 + hX_1^2X_3^2 + iX_1^2X_2^2X_3 + jX_1X_2^2X_3^2 \\
 & + kX_1^2X_2X_3^2 + lX_1^2X_2^2X_3^2
 \end{aligned}
 \tag{2}$$

where

<u>UDMH</u>	<u>MMH</u>
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/H₂O 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

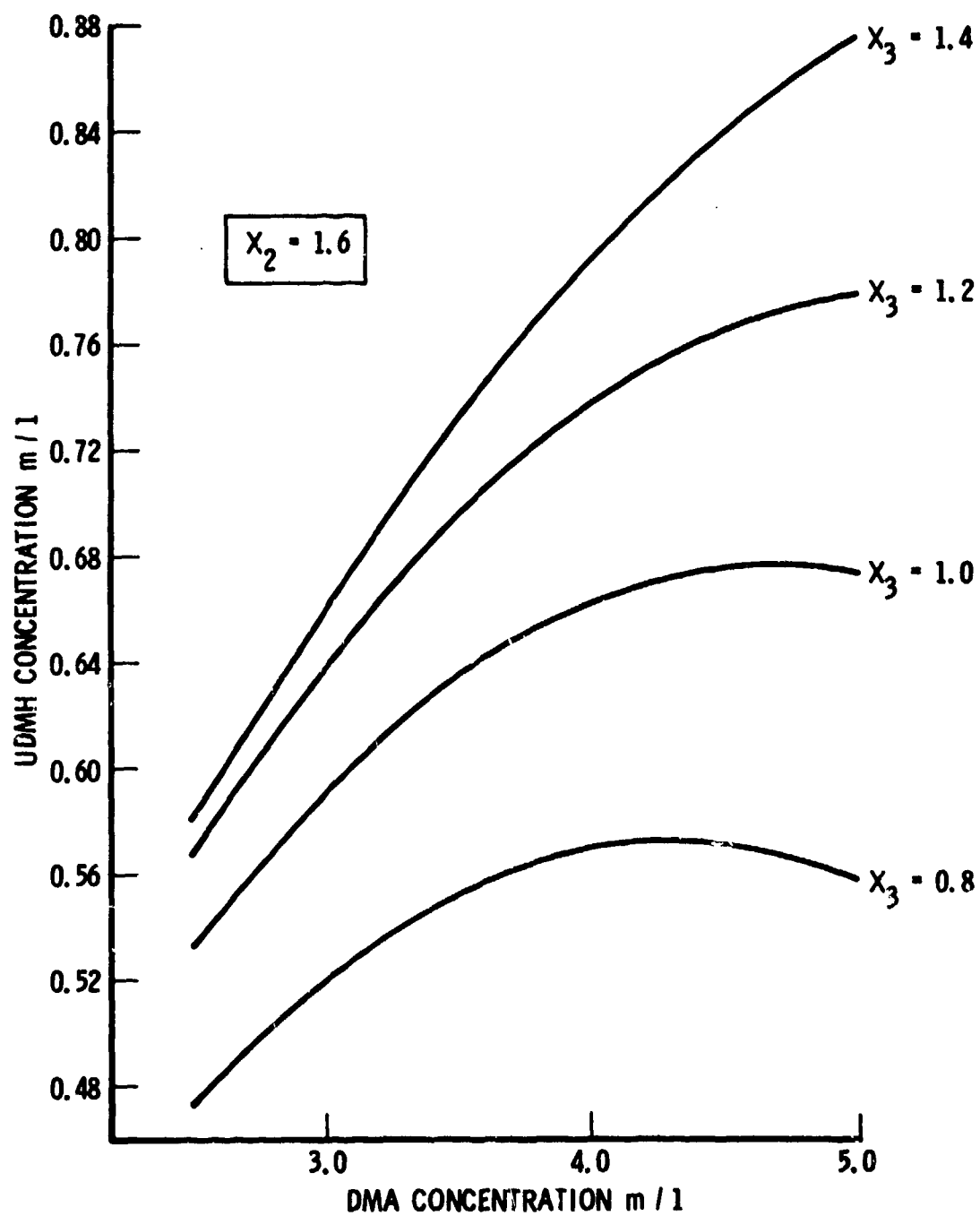


Figure 12. UDMH vs DMA Concentration

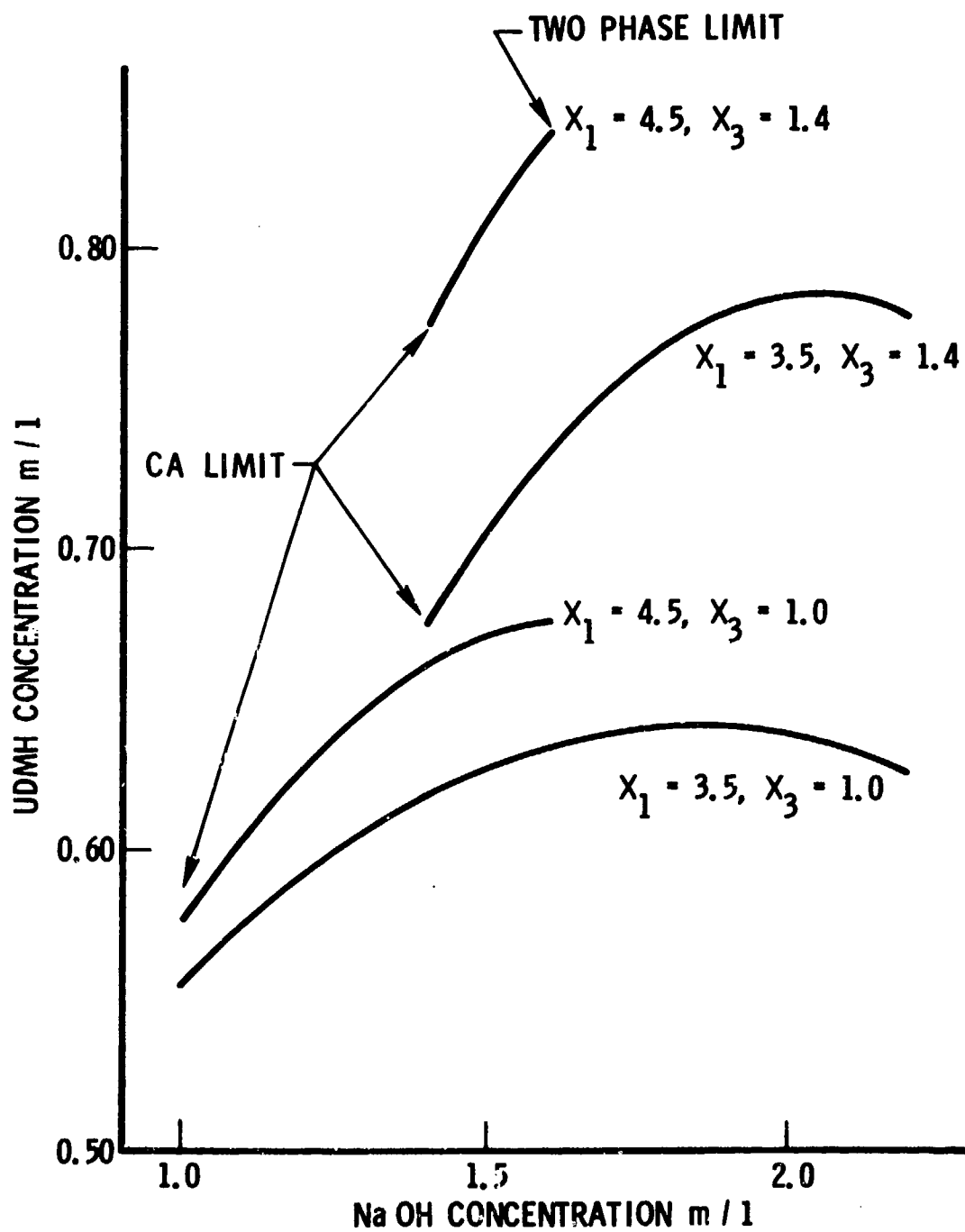


Figure 13. UDMH vs NaOH Concentration

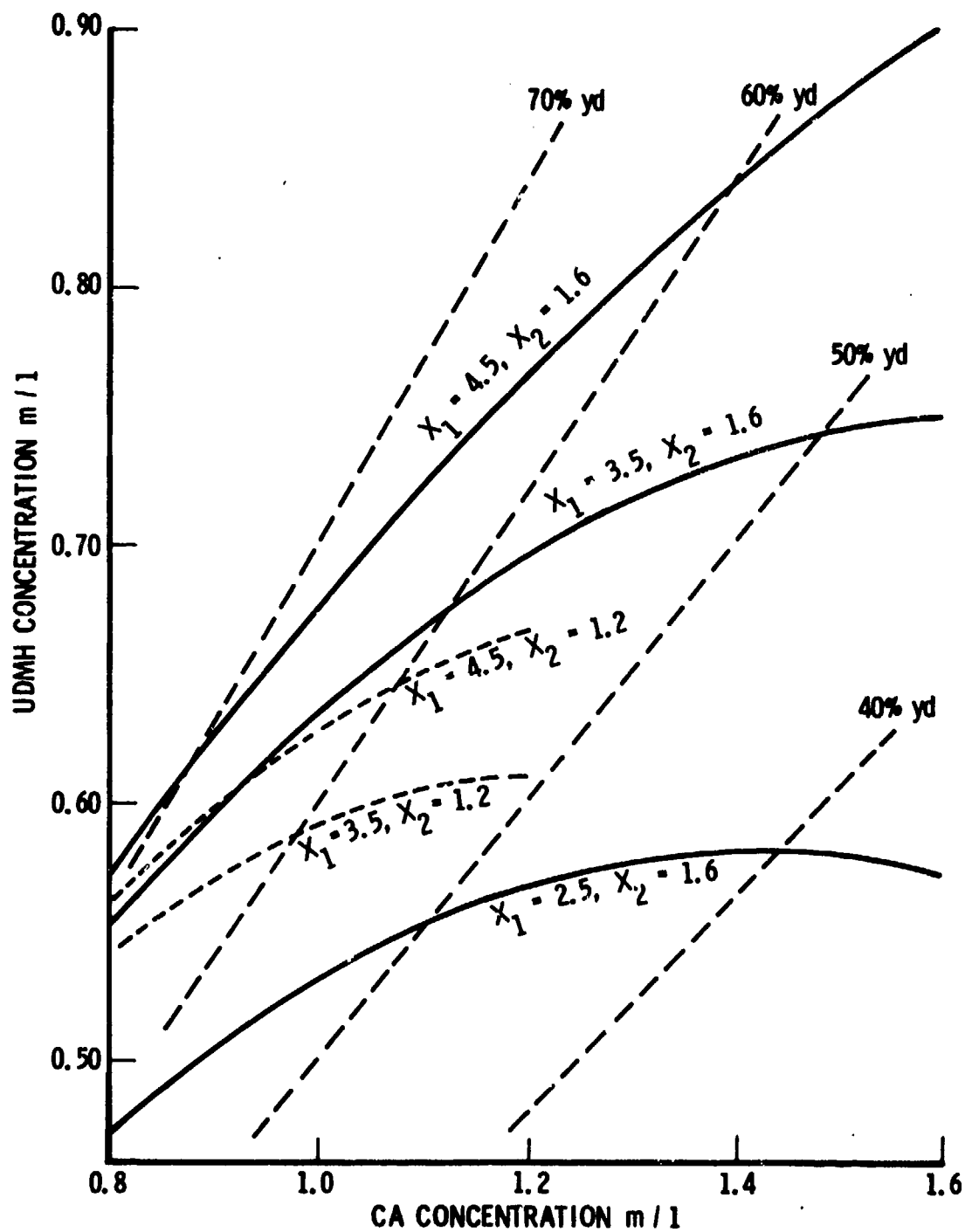


Figure 14. UDMH vs CA Concentration

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.

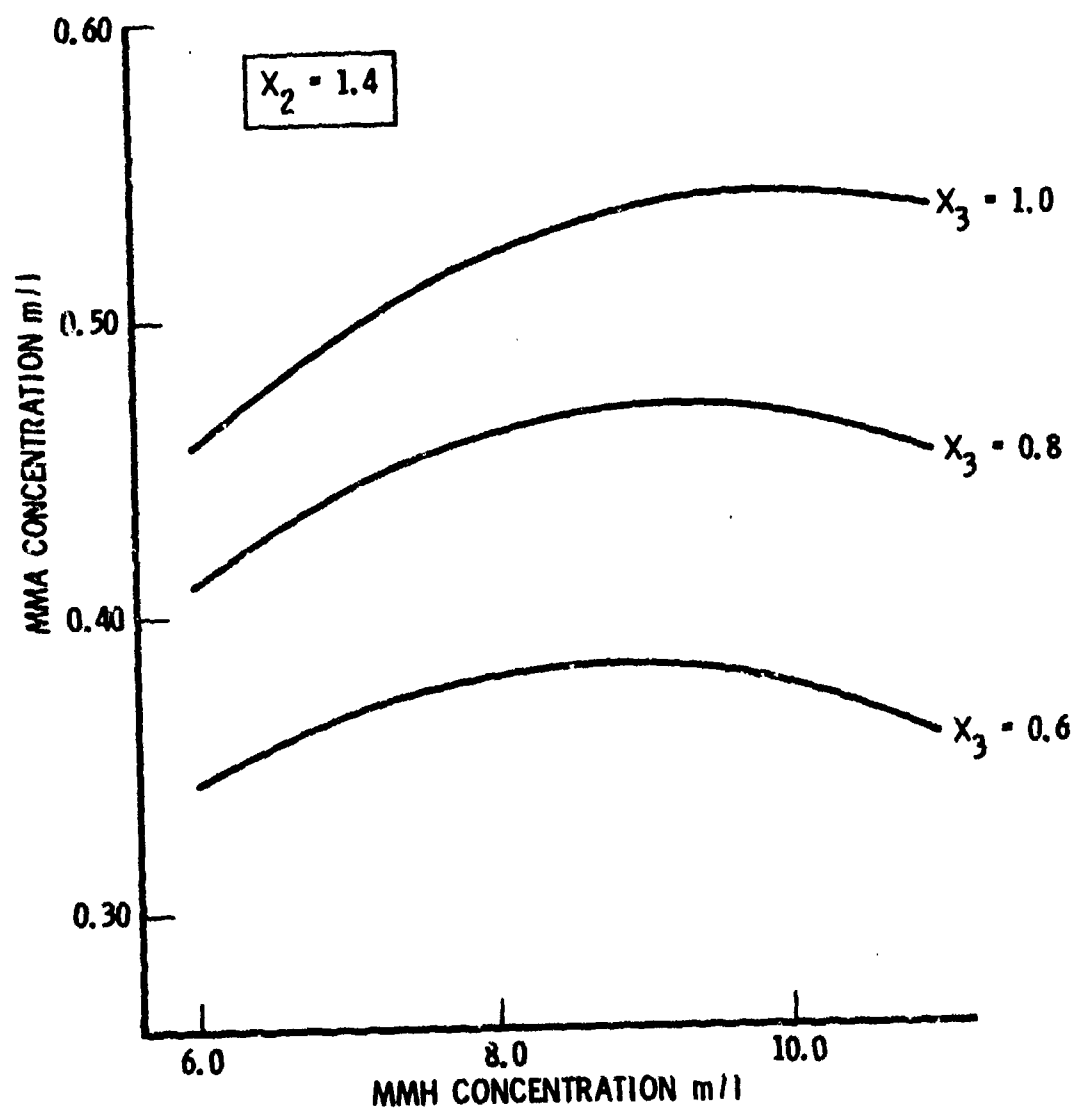


Figure 15. MMH vs MMA Concentration

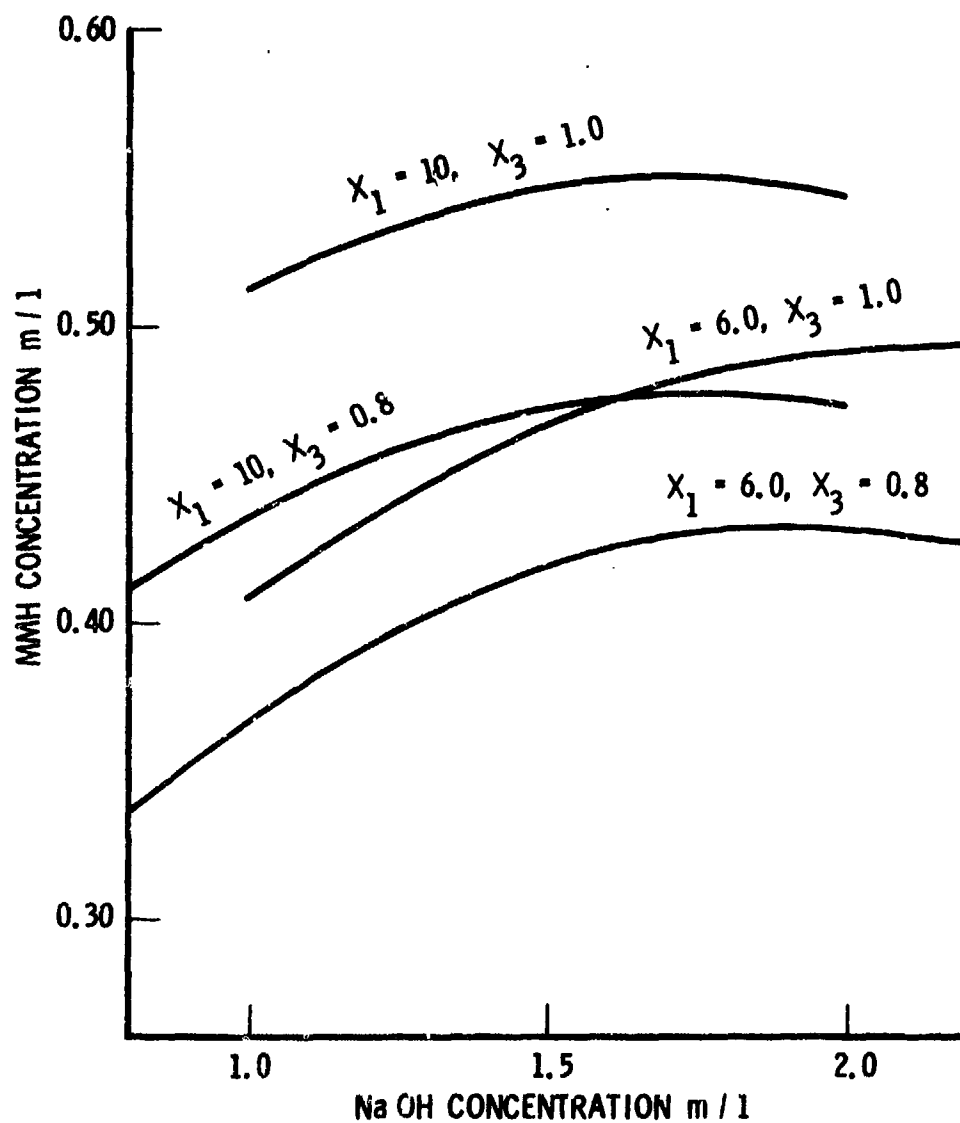


Figure 16. MMH vs NaOH Concentration

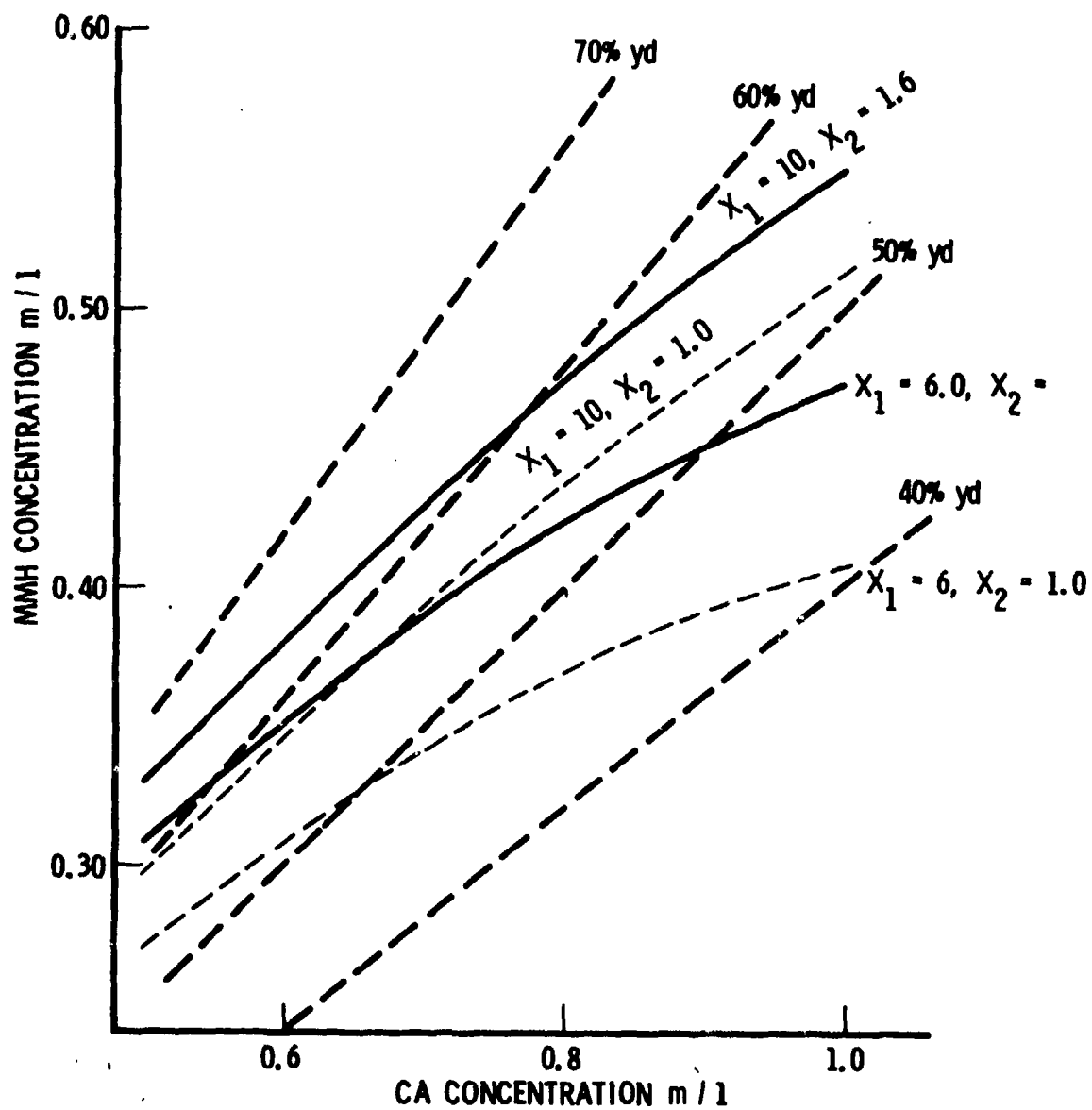


Figure 17. MMH vs CA Concentration

- 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 attempting 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.

*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)

⁵F. 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:

$$\text{Total} = \text{Raw Material} + \text{Recycle and Clean-up} + \text{Utility}$$

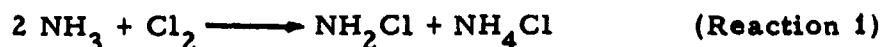
where

$$\begin{aligned} \text{Raw Materials} = & [\text{NH}_3 \text{ and Amine}]_{\text{consumed \& recycle loss}} \\ & + \text{Cl}_2 + \text{N}_2 + \text{NaOH} + \text{HCl} \end{aligned}$$

$$\text{Recycle and Clean-up} = \text{NH}_3 + \text{Amine} + \text{H}_2\text{O}$$

$$\text{Utility} = \text{product distillation and purification}$$

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.



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 contractor reaction is:



(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:

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

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:

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

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 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	$\frac{1}{Y_1} \left[\frac{113.36 (NH_3/Cl_2) - 302.33}{Y_2} + 75.61 \right]$	$\frac{1}{Y_1} \left[\frac{147.87 (NH_3/Cl_2) - 394.36}{Y_2} + 98.63 \right]$
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 ₄ Cl	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	Neutralise 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 ₂ O	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 ₂ O - Total in Product Mixture (lb/hr)			
UDMH System			
$\frac{1}{Y_3} [6322.8 - 300.06 X_1 - 389.04 X_2] - \frac{1}{Y_1} \left[\frac{113.36 (NH_3/Cl_2) - 302.33}{Y_2} + 75.61 \right] + \frac{600.12}{1 + Y_1} - 500$			
MMH System			
$\frac{1}{Y_3} [7900.3 - 269.64 X_1 - 507.48 X_2] - \frac{1}{Y_2} \left[\frac{147.87 (NH_3/Cl_2) - 394.36}{Y_2} + 98.63 \right] + \frac{539.27}{1 + Y_1} - 500$			

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	$\frac{0.0826}{Y_1 Y_2}$	$\frac{0.1077}{Y_1 Y_2}$
N ₂	0.05	$\frac{0.0140 (N_2/Cl_2)}{Y_1 Y_2}$	$\frac{0.0182 (N_2/Cl_2)}{Y_1 Y_2}$
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 ₂ O	0.01	$\left\{ \begin{aligned} &\frac{1}{Y_3} \left[0.1583 - 0.0075 X_1 - 0.0097 X_2 \right] \\ &+ \frac{0.0150}{1 + Y_1} \\ &- \frac{1}{Y_1} \left[\frac{0.0028 (NH_3/Cl_2) - 0.0076}{Y_2} \right. \\ &\quad \left. - 0.0019 \right] - 0.0125 \end{aligned} \right\}$	$\left\{ \begin{aligned} &\frac{1}{Y_3} \left[0.1975 - 0.0067 X_1 - 0.0127 X_2 \right] \\ &+ \frac{0.0135}{1 + Y_1} \\ &- \frac{1}{Y_1} \left[\frac{0.0037 (NH_3/Cl_2) - 0.0099}{Y_2} \right. \\ &\quad \left. - 0.0025 \right] - 0.0125 \end{aligned} \right\}$
Utility	-	$\frac{0.1598}{Y_3} - 0.0140$	$\frac{0.1997}{Y_3} - 0.0140$

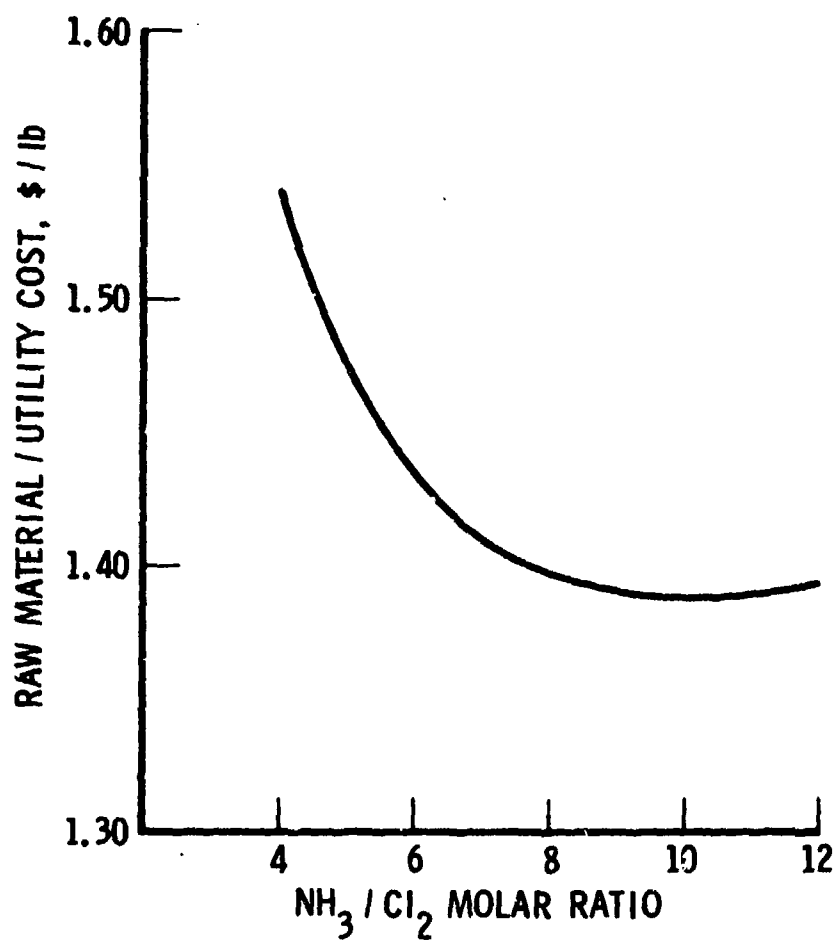


Figure 18. Effect of NH_3/Cl_2 Ratio on Fuel Cost

As indicated previously, the maximum first reaction yield attained in the 2 lb/hr unit was about 75 percent ($\text{NH}_3/\text{Cl}_2 = 12$, $\text{N}_2/\text{Cl}_2 = 4$), 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 $\text{NH}_3/\text{N}_2/\text{Cl}_2$ 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_2) 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

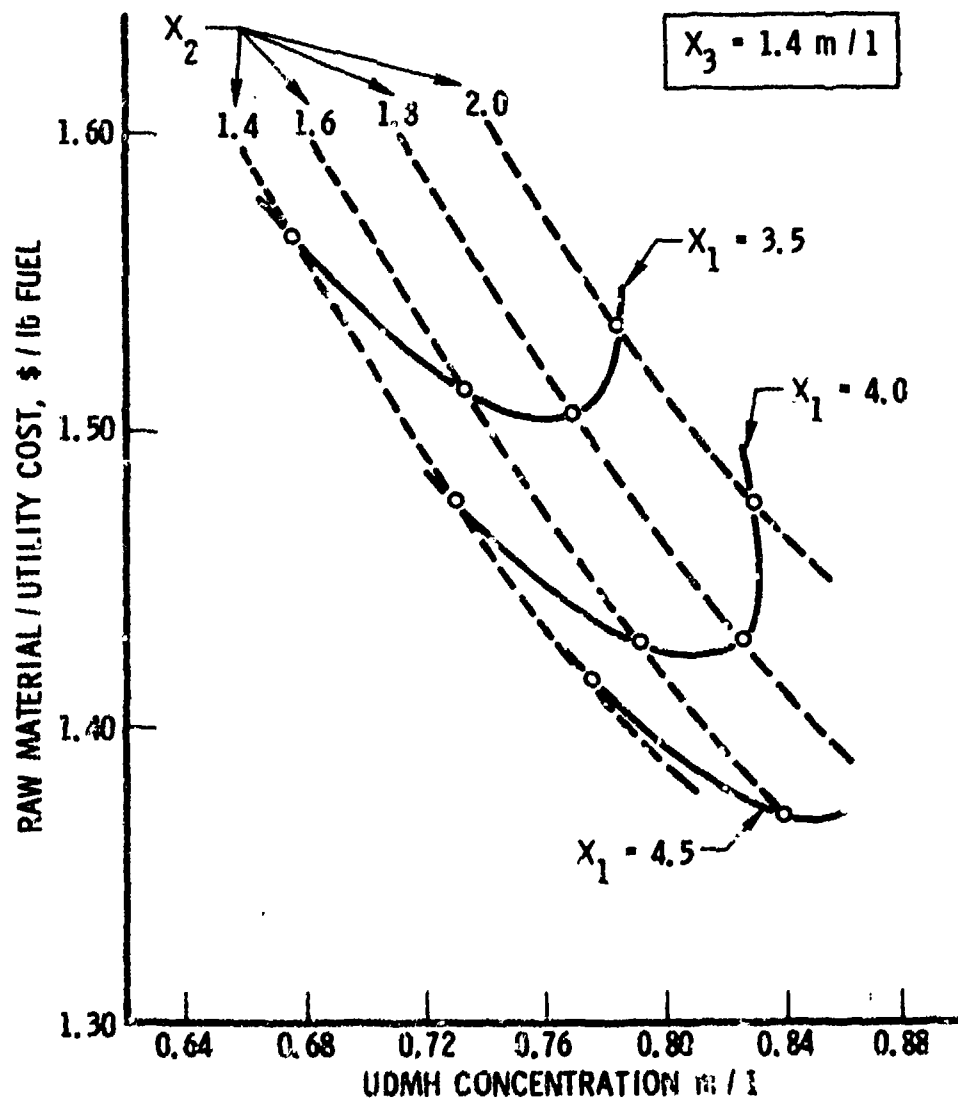


Figure 19. UDMH Cost at Constant CA Condition

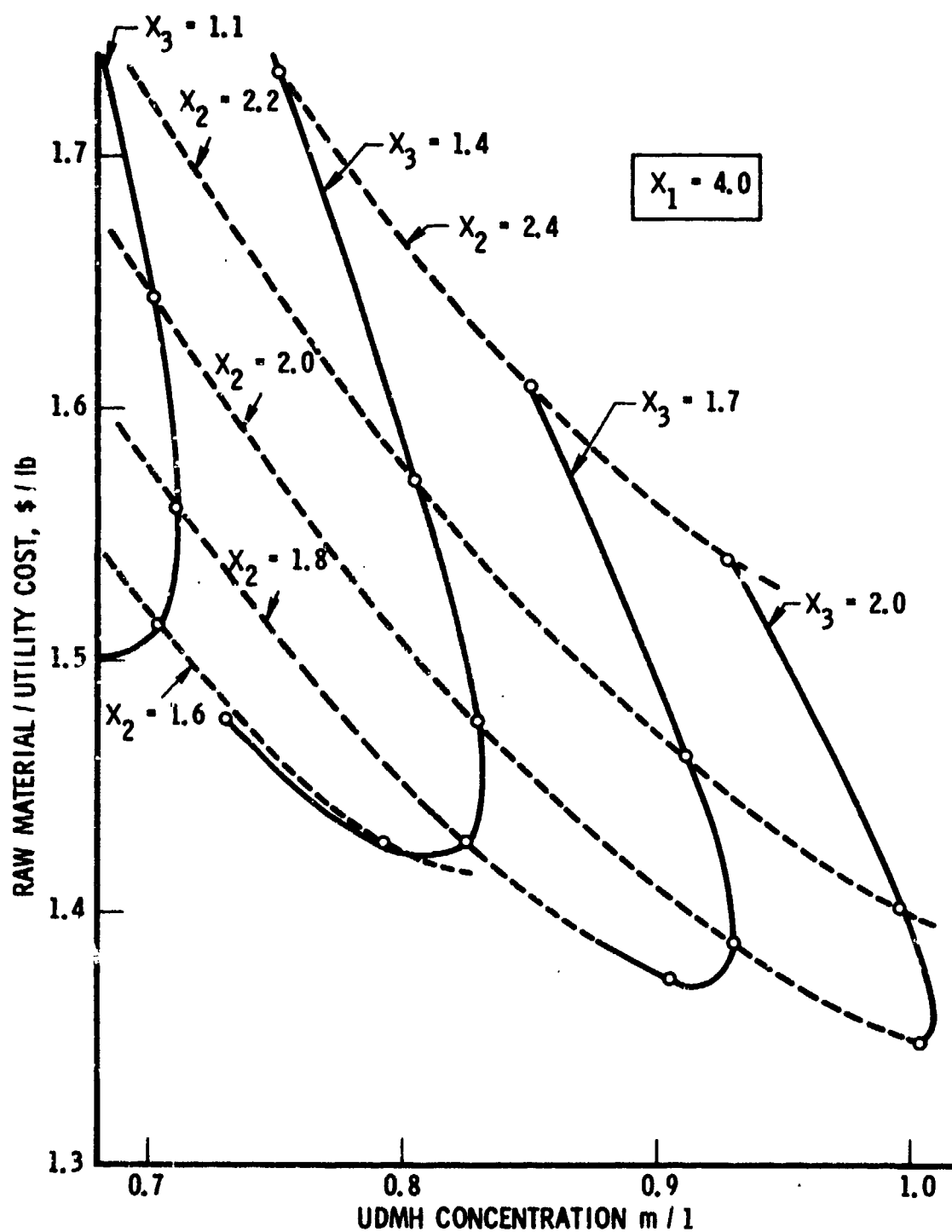


Figure 20. UDMH Cost at Constant DMA Condition

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:

CA Generator

$\text{NH}_3/\text{N}_2/\text{Cl}_2$ molar ratio 10/0/1

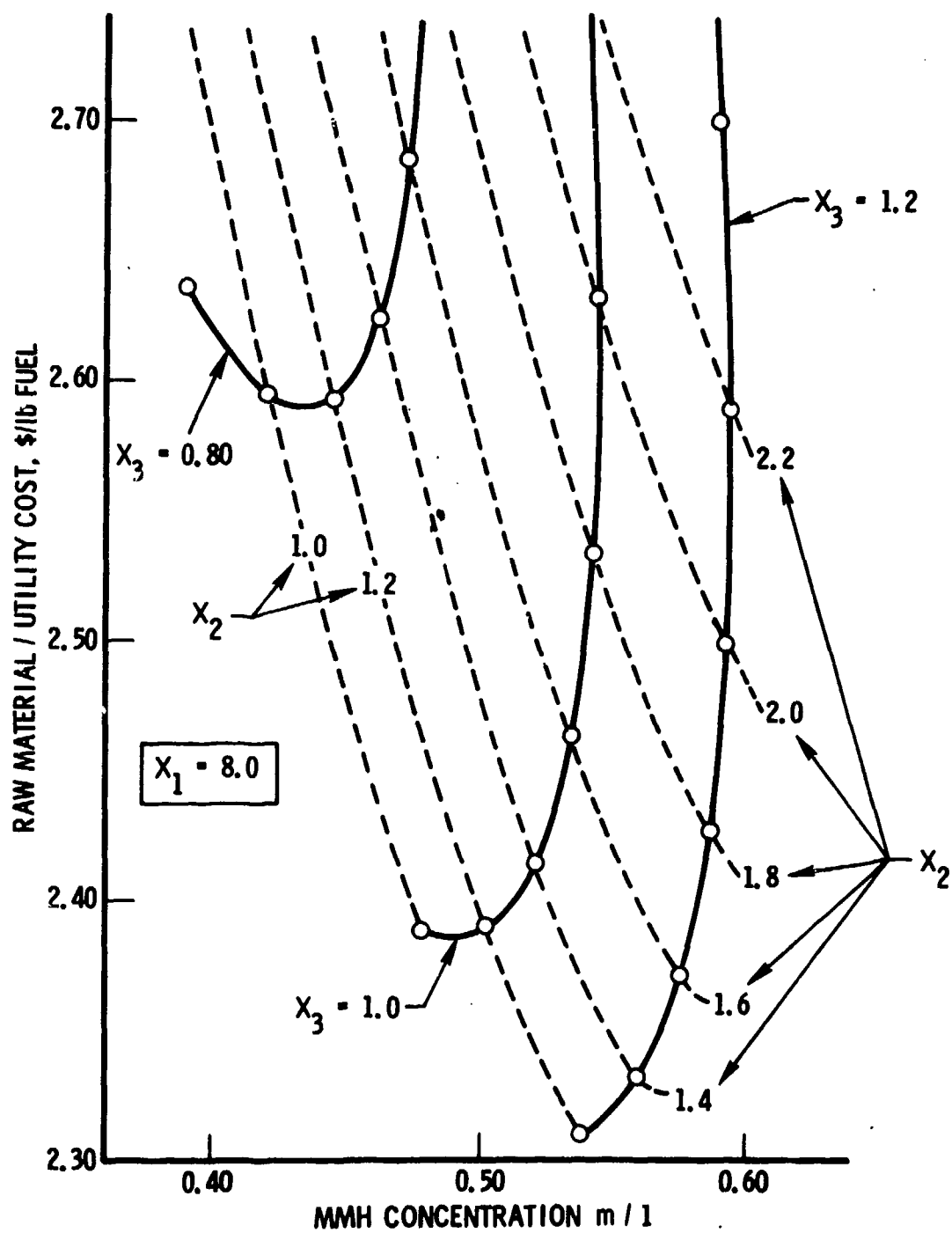


Figure 21. MMH Cost at Constant CA Condition

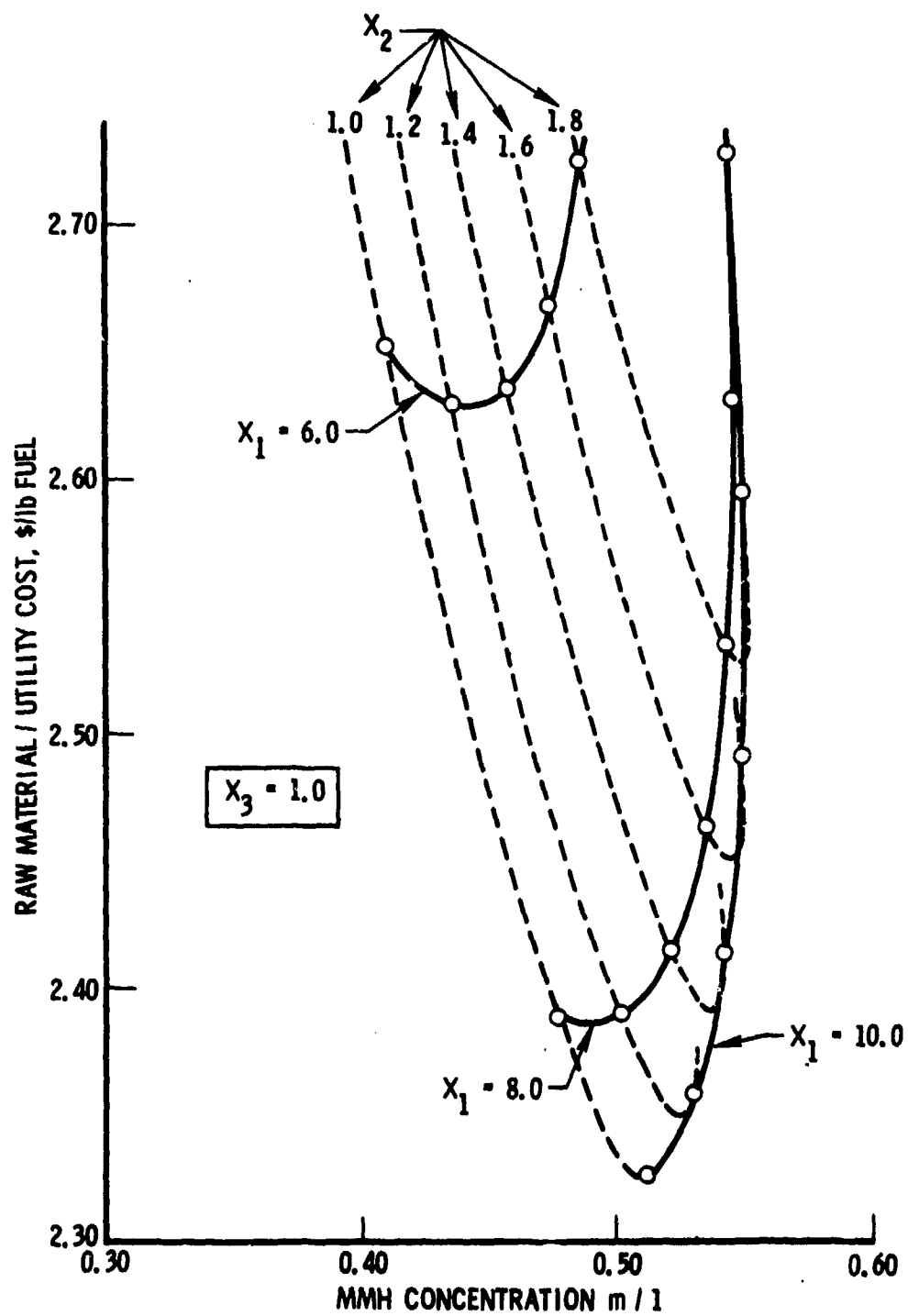


Figure 22. MMH Cost at Constant MMA Condition

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

Item	Operating Parameter and Individual Cost, \$/lb							
<u>Process Condition</u>								
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
<u>Raw Material Cost</u>								
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
<u>Recycle Cost</u>								
NH ₃	0.184	0.111	0.216	0.210	0.176	0.163	0.180	0.175
DMA	0.093	0.092	0.093	0.078	0.101	0.123	0.090	0.134
H ₂ O	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							
<u>Process Condition</u>								
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
<u>Raw Material Cost</u>								
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
<u>Recycle Cost</u>								
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 ₂ O	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

<u>Contactor</u>		<u>UDMH</u>	<u>MMH</u>
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 method⁶ 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

⁶John 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**

	<u>1978</u>	<u>1981</u>
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
	<u>9,184,800</u>	<u>10,779,510</u>

	<u>1978</u>		<u>1981</u>	
	<u>MMH</u>	<u>UDMH</u>	<u>MMH</u>	<u>UDMH</u>
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 *	<u>6,119,350</u>	<u>4,532,850</u>	<u>7,972,850</u>	<u>5,813,860</u>
\$/lb	6.12	4.53	7.97	5.81

* 1,000,000 lb/yr production rate
(Note: Does not include NH₄Cl 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.

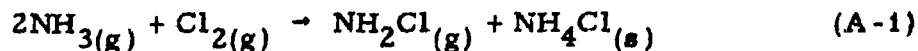
APPENDIX A

**ANALYSIS OF CHLORAMINE IN
GAS STREAMS**

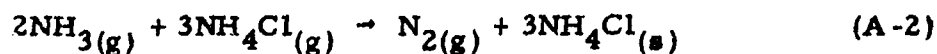
APPENDIX A

ANALYSIS OF CHLORAMINE IN GAS STREAMS

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.



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.



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.

$$\% \text{ CA yield} = \frac{E(R_1 - 2.667)}{1 - 1.667 E} \times 100 \quad (\text{A-3})$$

where

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

and

R_1 = 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

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 time³ 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.

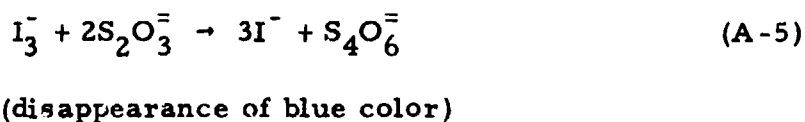
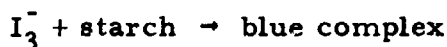
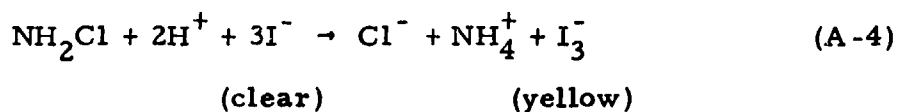
B. CALIBRATION OF CA SPECTRA

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

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).

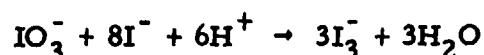
0.05 N NH_3 of parent chloramine solutions from the sampler (sampler is discussed later). These parent solutions were kept in an ice bath and shielded from light for the duration of the analyses. While an analyte was being scanned, concurrent iodometry was performed on another aliquot of the analyte 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.



All chemicals used were reagent grade. The starch indicator was made as prescribed by Vogel⁵ using mercuric iodide as preservative. The thiosulfate solution was likewise prepared as described by Vogel using

⁵A. 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.



$$\text{IO}_3^- : \text{S}_2\text{O}_3^{2-} = 1:6 \quad (\text{A-7})$$

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 acidifying 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 ($\sim 23^\circ\text{C}$) 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 ($\lambda_{\text{max}} \sim 294$ nm);

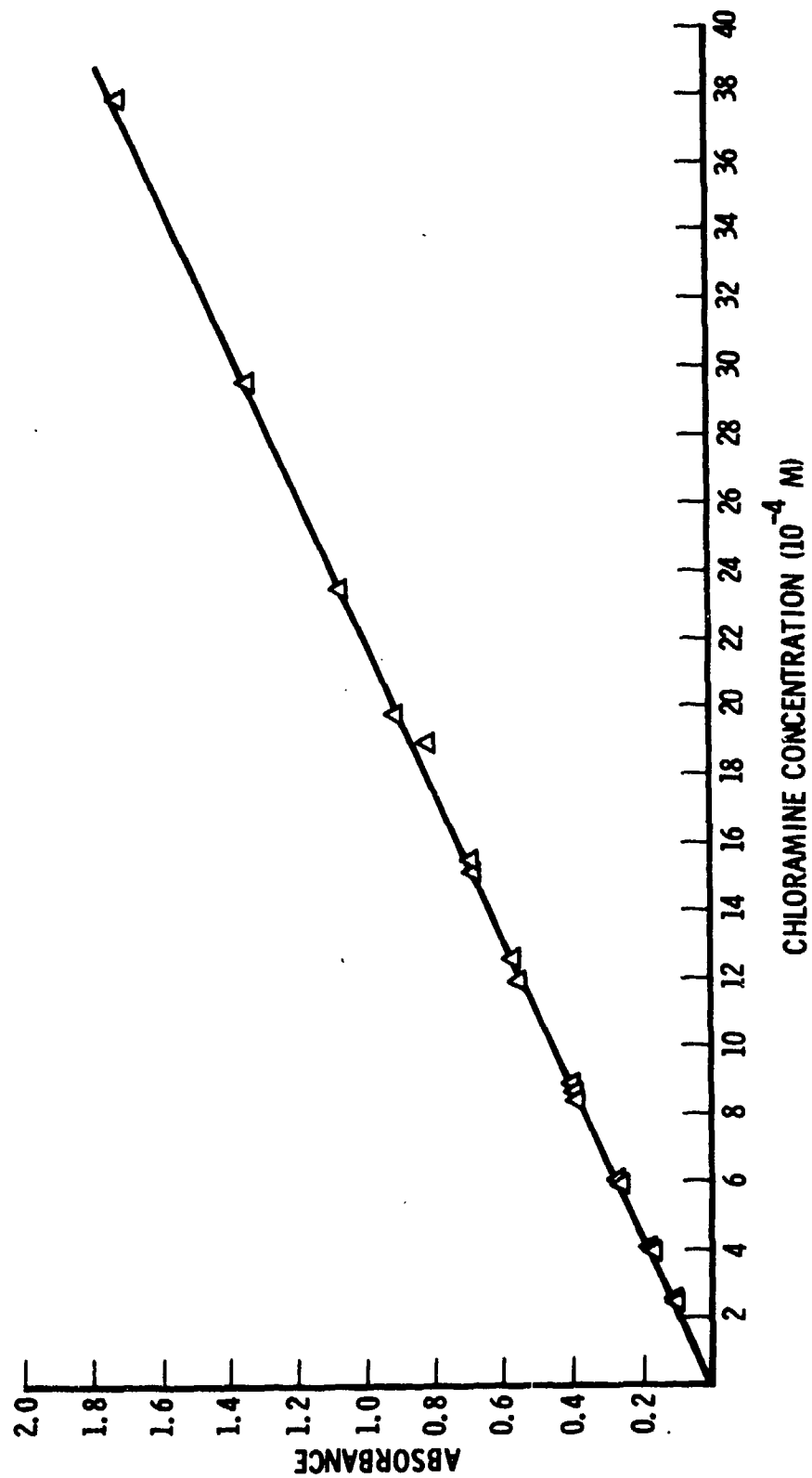


Figure A-1. Absorbance vs Concentration of Aqueous Chloramine at 243-243.5 nm 1 cm path length, $\epsilon = 453 \pm 6$ l/mole-cm

and at pHs of 0.4 to 0.8, the trichloramine appeared ($\lambda_{\text{max}} \sim 330-340 \text{ nm}$).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 ($\text{pK}_b \sim 10^{-5}$) with an equivalence point which occurs near $\text{pH} = 5$. 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.

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

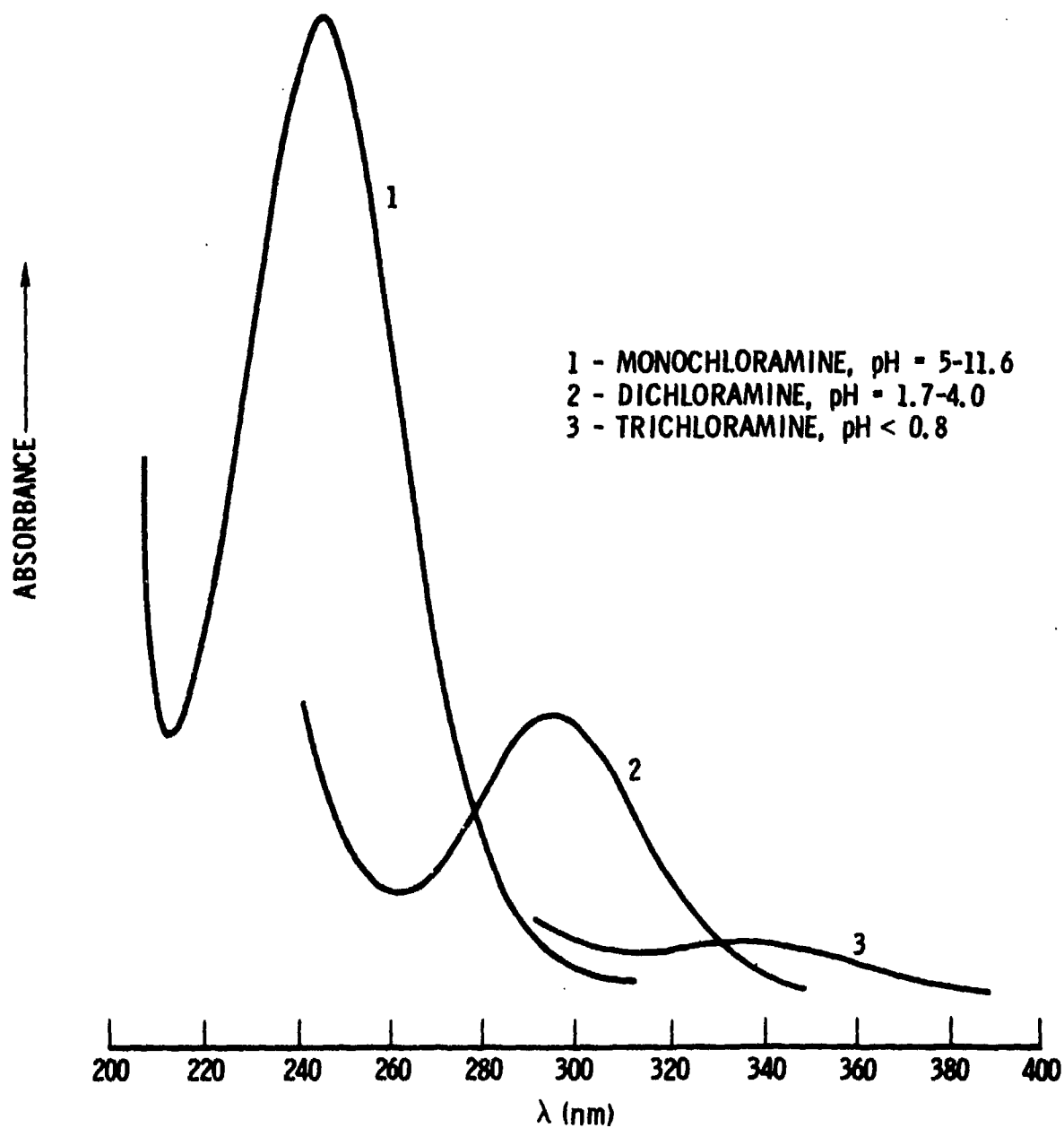


Figure A-2. Absorbance Spectra of the Chloramines in Aqueous Solution

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 ($\sim 110^\circ\text{C}$). 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 ml 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

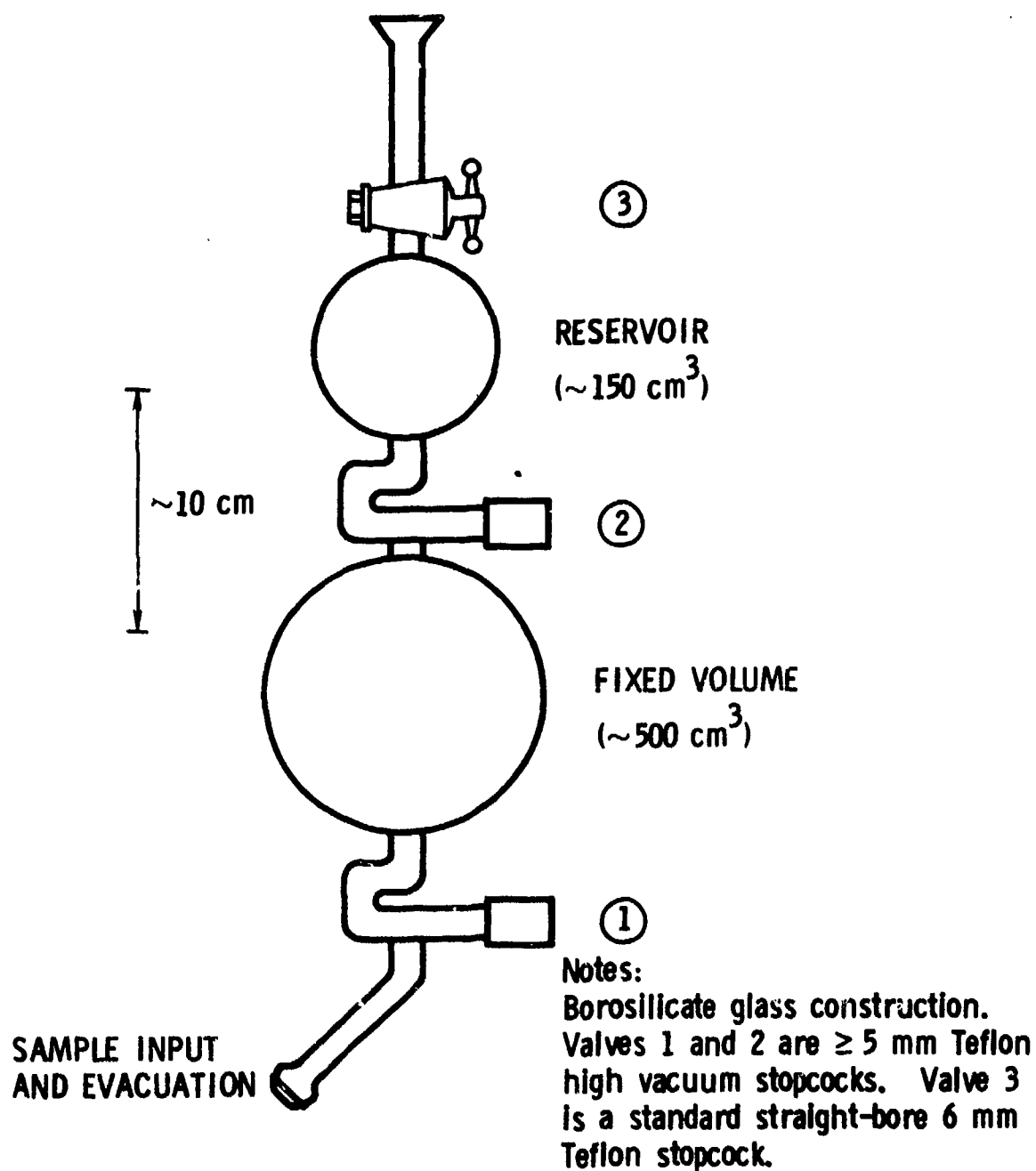


Figure A-3. Chloramine Gas Sampler (~1:2.5)

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 $\lambda_{\text{max}} = 243 \text{ to } 243.5 \text{ nm}$
5. Calculation for moles of CA:

(moles of chloramine) = (Absorbance/453) $\times$ (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 Chloramine

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:

$$(\text{CA conc.}) = \frac{(\text{S}_2\text{O}_3^{\equiv} \text{ conc.}) \times (\text{volume S}_2\text{O}_3^{\equiv} \text{ in ml})}{2(\text{volume sample aliquot in ml})}$$

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 :

$$\begin{aligned} (\text{moles of } \text{NH}_3) &= (\text{conc. HCl}) \times (\text{volume HCl in liters}) \\ &\times (\text{volume of sample volumetric flask}) / \text{volume of sample titrated.} \end{aligned}$$

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,

$$(\text{moles of } \text{NH}_3) = (\text{vol. HCl in ml}) \times 1.25 \times 10^{-3}$$

4. CA yield:

$$E = \frac{(\text{moles of chloramine})}{(\text{moles of } \text{NH}_3 + \text{moles of chloramine})} \text{ in the product sample}$$

$$R_i = \frac{(\text{moles/min of } \text{NH}_3)}{(\text{moles/min of } \text{Cl}_2)} \text{ of input reactants}$$

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

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	<u>10,000</u>	40,000
Production Supervisor	30,000	1	1	30,000	
Shift 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	<u>90,000</u>	421,000
Materials/Scheduler	20,000	1	1	20,000	
Quality Control	20,000	1	1	20,000	
Lab Tech	15,000	1	3	<u>45,000</u>	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	<u>42,000</u>	
					<u>115,000</u>
				Sub-Total	<u>661,000</u>
				O/H (100%)	<u>661,000</u>
				Total	<u>1,322,000</u>

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 water-lines, 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
<u>Tanks</u>			
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
			<u>222,000</u>
<u>Columns</u>			
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
			<u>415,000</u>
<u>Pumps</u>			
Typical	55	1,500	75,000
<u>Conveyor/Hopper Assy</u>	2	15,000	30,000
<u>Boilers</u>			
Heavy Duty	5	10,500	52,500
Light Duty	5	6,500	32,500
<u>Condensers</u>			
Typical	10	3,500	35,000
<u>Heat Exchangers</u>			
Typical	6	3,000	18,000
<u>Mixers</u>			
Caustic/H2O	1	20,000	20,000
Neutralizer	1	6,000	6,000
<u>Preheaters</u>			
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
<u>Vaporizer Assy</u>			
Ammonia	1	8,000	8,000
Chlorine	1	8,000	8,000
<u>Filters</u>			
Salt	2	25,000	50,000
<u>Reactor Assy</u>			
Reactor	1	78,000	78,000
Heat Ex	1	9,800	9,800
<u>Generator/Precipitator</u>	1	500,000	500,000
<u>Cooling Assy's</u>			
Cooling Tower	1	135,000	135,000
Chiller, 500 Ton	2	52,000	104,000
Refrig 250 Ton	1	485,000	485,000
<u>Valves</u>		<u>90,000</u>	<u>90,000</u>
Sub-Total			2,381,000
Instrumentation/Control (25%)			595,000
Installation			<u>2,457,813</u>
Total			5,433,813
<u>Optional Add-On Costs</u>			
<u>Rail Service Facility</u>			
Equipment, Rail	150,000		
Equipment, Service	150,000		
Siting	300,000		
Total (Est)		\$650,000	
<u>Drum Service Facility</u>			
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

<u>Task</u>	<u>Detail</u>
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

- o 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)**

- o **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)
- o **Control Equipment (Control Room)**
 - Data Display/Record
 - Control Computer Console
 - Manual Control Console
 - Material Processing Controls (Product Raw Material)
 - Install/Interconnect
- o **Non-Process Equipment**
 - Environmental/Occupational Monitors
 - Safety Equipment
 - FIREX
 - Fire
 - Air Supply
 - Equipment Stations
 - Safety Showers
- o **Instrumentation (Install/Interconnect)**
 - Process Control
 - Environmental/Occupational

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

- o Electrical (Hook-up)
Support Equipment
Process Equipment
- o Support Buildings
Erect
Plumb & Wire
Finish

1. Structural - 6 months
1-1/2 months pre-installations ($33 \text{ days} \times 8 = 264 \text{ hours}$)
4-1/2 months installation ($99 \text{ days} \times 8 = 792 \text{ hours}$)
2. Process Equipment - 9 months
4 mos - major assemblies ($88 \times 8 = 704$)
3 mos - minor assemblies ($66 \times 8 = 528$)
2 mos - interconnect ($44 \times 8 = 352$)
3. Control Equipment - 3 months ($66 \times 8 = 528$)
4. Non-Process Equipment - 4 months ($88 \times 8 = 704$)
5. Instrumentation - 3 months ($66 \times 8 = 528$)
6. Electrical - 6 months ($132 \times 8 = 1056$)
7. Support Buildings - 3 months ($66 \times 8 = 528$)

Structural

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

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

Table B-4. Facility/Site Preparation

Concrete Work
Slabs

Raw Material Areas

1' x 60' x 60' = 3,600 ft² @ \$3.50 \$ 12,600

Product Storage

1' x 30' x 40' = 1,200 ft² @ \$3.50 4,200

Plant

1' x 100' x 120' = 12,000 ft² @ \$3.50 42,000

Control Center

0.5' x 50' x 80' = 4,000 ft² @ \$3.50 14,000

\$ 72,800

Retaining Walls

Raw Material Area

1' x 12' x 180 = 180 @ \$7.00 1,260

1' x 3' x 60 = 60 @ \$7.00 420

Product Storage

1' x 12' x 100' = 100' @ \$7.00 700

1' x 3' x 40' = 40' @ \$7.00 280

Plant

1' x 3' x 960 = 960' @ \$7.00 6,720

Control Center

1' x 8' x 100' = 100' @ \$7.00 700

\$ 10,080

Asphalt Paving

30,000 ft² @ \$2.50

\$ 75,000

Cooling Pond

Excavation, 400 yds³ @ \$80

32,000

Lining, Gunite, Steel

90,000

\$ 122,000

Perimeter Drain

1' x 1' x 500', Channel @ \$10

\$ 5,000

Perimeter Fence

8' x 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)

<u>Control Center</u>		
Building, Pre-Fab (50' x 80')	\$120,000	
Quality Control Lab	100,000	
Medical Facility	75,000	
Office Equipment	75,000	
Maintenance Equipment	50,000	
Installation (100%)	<u>420,000</u>	\$ 840,000
 <u>Site Preparation</u>		
Survey	65,000	
Grading/Fill	<u>400,000</u>	\$ 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.

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.

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

\$177, 160 (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-H₂O

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
		8.60	0.99	0.67	0.358	53.69	66.42
		8.42	0.99	0.67	0.367	54.65	66.89
		8.61	1.08	0.67	0.352	52.38	66.71
							<u>66.46</u>
B 1	12.2/2/1	7.95	1.02	0.61	0.343	56.63	62.89
		7.29	1.08	0.67	0.367	55.16	69.38
		7.40	1.07	0.70	0.380	54.59	73.12
		7.39	0.85	0.64	0.343	53.75	65.58
							<u>67.74</u>
C 1	8/0/1	8.35	0.93	0.59	0.358	60.44	60.19
		8.40	0.86	0.53	0.343	65.23	53.05
		8.53	0.88	0.56	0.330	59.44	56.03
		7.71	0.98	0.58	0.343	58.71	59.20
							<u>57.13</u>
D 1	8/4/1	8.48	1.28	0.55	0.343	62.81	66.40
		8.42	0.93	0.50	0.313	62.97	60.03
		9.29	0.86	0.53	0.343	64.37	64.80
		9.15	1.00	0.52	0.326	62.84	62.27
							<u>63.38</u>
D 1	8/2/1	8.04	1.05	0.59	0.326	55.18	57.12
		7.80	0.95	0.62	0.362	58.47	60.93
		8.25	0.92	0.61	0.380	62.23	60.37
		8.10	0.98	0.60	0.362	60.85	57.97
							<u>59.10</u>

^aCone temp. 560°F

^bCl₂ flow of 37.5 g/mm except condit D which was 30 g/min

^cBased on CA

^dBased on Cl₂ 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
		2.89	2.21	0.61	0.444	72.62	67.90
		2.83	2.19	0.57	0.411	71.57	63.81
		3.43	2.32	0.61	0.493	80.96	69.72
		3.64	2.11	0.58	0.519	90.10	65.97
B 1	10/1/1	3.27	2.38	0.55	0.384	69.64	66.85
		3.26	2.32	0.55	0.384	70.32	62.50
		3.32	2.36	0.55	0.391	70.95	61.81
		3.34	2.31	0.57	0.391	68.16	62.87
		3.07	2.22	0.56	0.378	67.74	65.75
C 1	6/3/1	2.94	2.52	0.56	0.329	58.85	63.35
		3.26	2.64	0.53	0.349	65.52	63.26
		3.05	2.53	0.54	0.343	63.36	58.45
		3.20	2.49	0.54	0.343	63.23	56.21
		3.33	2.44	0.57	0.363	63.23	56.65
D 1	6/1/1	3.48	2.40	0.52	0.343	66.30	56.99
		3.26	2.50	0.42	0.296	70.72	60.28
		3.23	3.19	0.41	0.243	59.36	57.72
		3.61	2.64	0.44	0.323	73.84	54.16 ^e
		3.65	2.70	0.44	0.296	66.93	43.40
E 1	8/2/1	3.14	2.63	0.56	0.364	55.32	41.66
		3.18	2.35	0.56	0.391	69.29	45.43
		3.26	2.25	0.49	0.336	68.73	45.83
		3.40	2.22	0.53	0.391	73.32	44.08
		3.23	2.20	0.54	0.378	70.56	60.10
							61.18
							52.17 ^e
							57.58
							57.77
							59.16

^a Cone temp. 620°F except condit E which was 680°F^b Cl₂ flow of 37.5 g/min^c Based on CA^d Based on Cl₂ 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
		1.89	2.04	0.97	0.458	47.09	59.04
		1.91	1.99	0.88	0.438	50.00	52.11
		2.08	2.05	0.93	0.506	54.53	56.50
		2.00	2.04	0.89	0.512	57.65	53.93
							<u>54.14</u>
B 1	10/3/1	1.79	2.18	1.00	0.478	47.63	59.01
		1.86	2.36	0.98	0.486	49.36	58.54
		1.94	1.90	1.13	0.478	42.37	68.81 ^e
		1.85	2.25	0.92	0.458	49.68	54.92
		1.75	2.41	0.98	0.451	46.18	57.99
							<u>57.62</u>
C 1	6/3/1	2.05	2.38	0.94	0.478	50.56	49.68
		2.11	2.41	0.89	0.484	54.47	46.66
		2.23	2.41	0.90	0.493	54.93	46.91
		2.19	2.47	0.92	0.519	56.48	48.12
		2.10	2.40	0.85	0.451	53.15	43.95 ^e
							<u>47.84</u>
D 1	6/1/1	2.26	2.40	0.80	0.451	56.44	42.48
		2.27	2.30	0.80	0.418	51.90	42.98
		2.32	2.37	0.73	0.451	61.83	39.33
		2.31	2.20	0.73	0.438	59.68	39.82
		2.32	2.16	0.77	0.451	58.93	41.49
							<u>41.22</u>
Σ 1	4/2/1	3.72	1.94	0.59	0.458	77.22	33.49
		3.81	1.99	0.49	0.438	74.70	32.48
		4.01	1.90	0.62	0.431	70.06	34.04
		4.61	1.95	0.59	0.458	77.55	32.93
		4.18	2.04	0.61	0.451	73.54	33.86
							<u>33.36</u>

^aCone temp. 500°F^bCl₂ flow of 37.5 g/min^cBased on CA^dBased on Cl₂ flow^eNot 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

$$Y = a_1x_1 + a_2x_2 + a_3x_3 + \dots$$

$$a_{11}x_1^2 + a_{22}x_2^2 + a_{33}x_3^2 + \dots$$

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

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

	-1	0	+1
x_1	-a	0	a
x_2	-b	0	b
x_3	-c	0	c

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

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

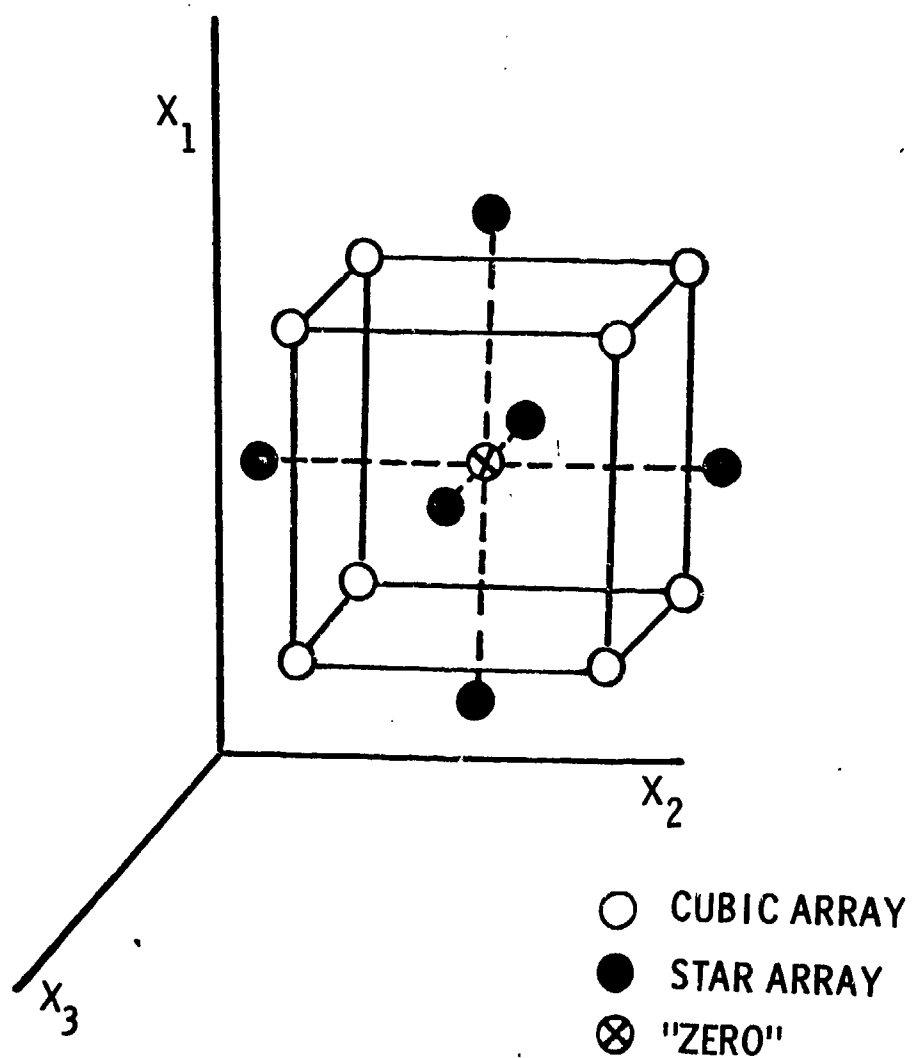
Thus 16 discrete test points are sufficient to approximate the response function (in this instance a surface) with two replicates to provide an error estimation. The actual test points are tabulated in Attachment D-1 and displayed in Attachment D-2.

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)	JMA (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.	Con- dition	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	
		175	3.36	1.13	0.68	0.444	65.61	
			3.57	1.37	0.65	0.451	69.51	
			3.61	1.34	0.65	0.464	71.22	
			3.89	1.26	0.66	0.499	75.31	
			3.65	1.25	0.75	0.539	72.32	
		250	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.	Con- dition	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	Not Used
		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	
		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	Not Used
		569	8.16	1.50	0.32	0.230	71.93	
		580	6.83	1.55	0.31	0.206	65.86	
		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	Not Used
		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	
		130	8.22	1.34	0.41	0.258	62.73	
	B	150	8.11	1.47	0.38	0.250	65.56	Not Used
		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	Not Used
		288	6.81	2.27	0.46	0.282	61.00	
		304	6.91	2.09	0.45	0.291	64.57	
		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	Not Used
		400	6.59	2.11	0.39	0.230	58.94	
		415	6.97	2.25	0.41	0.265	64.97	
		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.	Con- dition	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	Not Used
		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	
		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	Not Used
		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	

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-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	
			8.78	1.97	0.62	0.397	64.07	
	B		8.23	2.02	0.62	0.451	72.96	Not Used
		164	7.99	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	
			8.66	1.57	0.43	0.334	77.31	
			7.82	1.61	0.43	0.328	76.05	Not Used

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	
			10.73	0.90	0.62	0.349	56.24	
		125	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.	Con- dition	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	Not Used
		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	
	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	Not Used
		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	
			10.58	0.74	0.70	0.371	52.98	
		125	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	
			8.82	0.77	0.64	0.339	52.89	
		205	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	
			10.49	0.50	0.49	0.221	45.43	
		290	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	
			10.25	1.43	0.55	0.230	41.47	
		375	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	
		103	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	
	D		13.26	1.85	0.94	0.469	49.86	Not Used
		560	12.92	2.03	0.94	0.415	44.14	
		580	13.72	1.88	0.76	0.393	51.44	
		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	Not Used
		545	10.94	2.52	0.40	0.213	53.07	
		560	10.33	2.59	0.54	0.310	57.86	
		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	
			9.00	1.05	0.65	0.408	62.88	
		125	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	
			8.25	2.80	0.88	0.473	53.48	
		240	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	Not Used
		215	5.95	1.66	1.24	0.477	38.57	
		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	Not Used
		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	
			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	
	E		5.50	1.41	0.94	0.402	42.93	
		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

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Temperatures - °F Refer To Instrumentation List

Rev.

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Temperatures - °F Refer To Instrumentation List

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Temperatures - °F Refer To Instrumentation List

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Temperatures - °F Refer To Instrumentation List

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Temperatures - °F Refer To Instrumentation List

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Temperatures - °F Refer To Instrumentation List

Rev.

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Test Contractor's Notes		Test Contractor's Notes	
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0-3 Chemical Analysis
Run Number 2
Sample Name UDMH

UDMH

Sample Lot: 12345										Analysis Date: 10/26/2023										Analysis Time: 14:30										Analysis Location: Lab 1										Analysis Operator: J. Doe										Analysis Instrument: GC/MS										Analysis Method: GC/MS										Analysis Method: GC/MS										Analysis Method: GC/MS										Analysis Method: GC/MS										Analysis Method: GC/MS										Analysis Method: GC/MS										Analysis Method: GC/MS										Analysis Method: GC/MS										Analysis Method: GC/MS										Analysis Method: GC/MS										Analysis Method: GC/MS										Analysis Method: GC/MS										Analysis Method: GC/MS										Analysis Method: GC/MS										Analysis Method: GC/MS										Analysis Method: GC/MS										Analysis Method: GC/MS										Analysis Method: GC/MS										Analysis Method: GC/MS										Analysis Method: GC/MS										Analysis Method: GC/MS										Analysis Method: GC/MS										Analysis Method: GC/MS										Analysis Method: GC/MS										Analysis Method: GC/MS										Analysis Method: GC/MS										Analysis Method: GC/MS										Analysis Method: GC/MS										Analysis Method: GC/MS										Analysis Method: 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THIS PAGE IS BEST QUALITY PRACTICABLE
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UDOHMMH PLANT RUN DATA SHEETS

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X JCMH, Test Conductor J. S. Shoup

Page 1 of 1

Run Time	Core Min.	Test Matrix Point	1st Stage Feed Rate	2nd Stage Feed Rate	System Pressures				Stirrer RPM at 300	Core Level (Inches)	MAN Level (Inches)	Sample Taken			Vibrator	Flow Rates	T.P. Jacket Upper C/P	Electrode AC	Proportionator DC	
Core Min.	Core Min.		Cl ₂ g/min	H ₂ g/min	MAN g/min	Water Man. Min.	Amine Man. Min.	T.P. Poly	L.P. Poly	Spent Poly			Y-1Y-2Y-3	PR 4066	Flow Min.	Force PSI	Product MAN	Valve g/min	Spent Min.	Alkalinity mA
1000	5	W	6.0	1.0	1.0	2.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0
1005	5	W	6.0	1.0	1.0	2.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0
1010	5	W	6.0	1.0	1.0	2.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0
1015	5	W	6.0	1.0	1.0	2.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0
1020	5	W	6.0	1.0	1.0	2.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0
1025	5	W	6.0	1.0	1.0	2.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0
1030	5	W	6.0	1.0	1.0	2.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0
1035	5	W	6.0	1.0	1.0	2.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0
1040	5	W	6.0	1.0	1.0	2.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0
1045	5	W	6.0	1.0	1.0	2.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0
1050	5	W	6.0	1.0	1.0	2.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0
1055	5	W	6.0	1.0	1.0	2.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0
1060	5	W	6.0	1.0	1.0	2.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0
1065	5	W	6.0	1.0	1.0	2.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0
1070	5	W	6.0	1.0	1.0	2.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0
1075	5	W	6.0	1.0	1.0	2.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0
1080	5	W	6.0	1.0	1.0	2.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0
1085	5	W	6.0	1.0	1.0	2.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0
1090	5	W	6.0	1.0	1.0	2.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0
1095	5	W	6.0	1.0	1.0	2.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0
1100	5	W	6.0	1.0	1.0	2.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0
1105	5	W	6.0	1.0	1.0	2.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0
1110	5	W	6.0	1.0	1.0	2.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0
1115	5	W	6.0	1.0	1.0	2.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0
1120	5	W	6.0	1.0	1.0	2.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0
1125	5	W	6.0	1.0	1.0	2.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0
1130	5	W	6.0	1.0	1.0	2.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0
1135	5	W	6.0	1.0	1.0	2.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0
1140	5	W	6.0	1.0	1.0	2.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0
1145	5	W	6.0	1.0	1.0	2.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0
1150	5	W	6.0	1.0	1.0	2.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0
1155	5	W	6.0	1.0	1.0	2.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0
1160	5	W	6.0	1.0	1.0	2.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0
1165	5	W	6.0	1.0	1.0	2.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0
1170	5	W	6.0	1.0	1.0	2.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0
1175	5	W	6.0	1.0	1.0	2.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0
1180	5	W	6.0	1.0	1.0	2.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0
1185	5	W	6.0	1.0	1.0	2.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0
1190	5	W	6.0	1.0	1.0	2.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0
1195	5	W	6.0	1.0	1.0	2.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0
1200	5	W	6.0	1.0	1.0	2.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0
1205	5	W	6.0	1.0	1.0	2.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0
1210	5	W	6.0	1.0	1.0	2.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0
1215	5	W	6.0	1.0	1.0	2.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0
1220	5	W	6.0	1.0	1.0	2.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0
1225	5	W	6.0	1.0	1.0	2.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0
1230	5	W	6.0	1.0	1.0	2.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0
1235	5	W	6.0	1.0	1.0	2.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0
1240	5	W	6.0	1.0	1.0	2.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0
1245	5	W	6.0	1.0	1.0	2.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0
1250	5	W	6.0	1.0	1.0	2.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0
1255	5	W	6.0	1.0	1.0	2.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0
1260	5	W	6.0	1.0	1.0	2.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0
1265	5	W	6.0	1.0	1.0	2.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0
1270	5	W	6.0	1.0	1.0	2.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0
1275	5	W	6.0	1.0	1.0	2.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0
1280	5	W	6.0	1.0	1.0	2.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0
1285	5	W	6.0	1.0	1.0	2.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0
1290	5	W	6.0	1.0	1.0	2.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0
1295	5	W	6.0	1.0	1.0	2.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0
1300	5	W	6.0	1.0	1.0	2.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0
1305	5	W	6.0	1.0	1.0	2.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0
1310	5	W	6.0	1.0	1.0	2.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0
1315	5	W	6.0	1.0	1.0	2.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0
1320	5	W	6.0	1.0	1.0	2.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0
1325	5	W	6.0	1.0	1.0	2.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0
1330	5	W	6.0	1.0	1.0	2.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0
1335	5	W	6.0	1.0	1.0	2.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0
1340	5	W	6.0	1.0	1.0	2.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.0	1.

0-1 Process

UDMH PLANT RUN DATA SHEETS

MMH, Date 2/20/78, Test No 2

UDMH, Test Conductor J.C. Saupers

Page 01

Run Time	Test Matrix	1st Stage Feed Rate	2nd Stage Feed Rate	System Pressures						Shower RPM	Card. Level	MMOH Level	Sample Taken	Vibrator	Flare Rates	T.P. Jacket	Electrode	Preheater					
Card Min.	Card Min.	O ₂ g/min	H ₂ g/min	MMOH M/Min	Water M/Min	Amine M/Min	T.P. P.Si	E.P. P.Si	Card. P.Si	Spand P.Si	inches	Y-1 Y-2 Y-3 Y-4 Y-5	Freq. Min.	Force PSI	Product M/Min	Vent g/min	Upper GPM	Lower GPM	Volt	Amps	Spd. Min	AC Volts	AC Amperes
4:20	10	2	87.5	43.8	14.8	198.8	246.3	146.7	5.2	0.8	0.3	5.3	2000	2	2000	3	3	4.9	1.7	20	25	2.5	2.5
4:55	18	2	87.5	43.8	23.7	198.8	246.3	146.7	1.4	1.6	0.3	1.6	2000	2	2000	3	3	4.9	2.0	20	32	2.5	2.5
4:40	20	2	87.5	43.8	23.7	198.8	246.3	146.7	1.6	1.7	0.4	1.9	2000	2	2000	3	3	4.9	1.0	20	26	2.5	2.5
4:45	25	2	87.5	43.8	23.7	198.8	246.3	146.7	1.7	1.8	0.3	1.9	2000	2	2000	3	3	4.9	0.9	20	26	2.5	2.5
4:50	30	2	87.5	43.8	23.7	198.8	246.3	146.7	1.9	1.8	0.3	1.8	2000	2	2000	3	3	4.9	0.8	20	26	2.5	2.5
4:55	35	2	87.5	43.8	23.7	198.8	246.3	146.7	1.8	1.8	0.3	1.8	2000	2	2000	3	3	4.9	0.7	20	26	2.5	2.5
4:58	38	2	87.5	43.8	23.7	198.8	246.3	146.7	1.8	1.8	0.4	2.0	2000	2	2000	3	3	4.9	0.4	20	26	2.5	2.5
4:59	40	2	87.5	43.8	23.7	198.8	246.3	146.7	1.8	1.8	0.4	2.0	2000	2	2000	3	3	4.9	0.4	20	26	2.5	2.5

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(D-4 Test Conductors Notes)		
Date 2-28-17 Run No. 2		
Run Cont.	Time @ Cond.	Test Conductor's Notes
870	10	Conduct at 82% - add 5% to adjust
88		As mine flow - adding heat to boiler
86		Dump MHC, still working mine problem
45		on line - with mine - (1) Running line smelter
60	15	Dump MHC
95		Dump MHC, conduct at 80.5%
127	82	TC 29 smelter in gas stream = 2; will be unable to get more data
180	10	Dump MHC
182	12	Change MHC boiler
187	17	Hopper chopper stopped; upper valve not operating on discharge
189	42	Upper valve now operating; completed dump; working on hopper chopper
184		Hopper chopper now working
288		Dump MHC; conduct at 80.1%
283		Dump MHC: 0
295		Dump MHC
300	0	Dump MHC; upper valve not closing; working problem
885		Upper valve working, seems to work constant increasing and decreasing
381		Dump MHC; upper valve still under ticking; conduct 80%; EP eaching above 29 KV, 1.9 mb

(D-4 Test Conductors Notes)		
Date 2-28-17 Run No. 2		
Run Cont.	Time @ Cond.	Test Conductor's Notes
425		Dump MHC; playing with valve, again
455		EP is continuing to hit up, dump MHC's gas run warning even if EP is present
460		EP reaching at 30, backed down to 30
465		EP back at 30 KV and 1.5 mb
485		Dump MHC
545		Shut down; could not push practice in reasonable period of time; had some especially smelter; sorry for this

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Pg. 2

8-3 Census' Agency
Date 2/29, Box Number 3
Agency San Jose

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(B-3 Chemical Analysis)
Date _____, Page Number _____

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②-1 Chemical Analysis
Date: Run Number: Analyst:

Run No.	Sample	Q ₁	Q ₂	Q ₃	Q ₄	Q ₅	Q ₆	Q ₇	Q ₈	Q ₉	Q ₁₀	Q ₁₁	Q ₁₂	Q ₁₃	Q ₁₄	Q ₁₅	Q ₁₆	Q ₁₇	Q ₁₈	Q ₁₉	Q ₂₀	Q ₂₁	Q ₂₂	Q ₂₃	Q ₂₄	Q ₂₅	Q ₂₆	Q ₂₇	Q ₂₈	Q ₂₉	Q ₃₀	Q ₃₁	Q ₃₂	Q ₃₃	Q ₃₄	Q ₃₅	Q ₃₆	Q ₃₇	Q ₃₈	Q ₃₉	Q ₄₀	Q ₄₁	Q ₄₂	Q ₄₃	Q ₄₄	Q ₄₅	Q ₄₆	Q ₄₇	Q ₄₈	Q ₄₉	Q ₅₀	Q ₅₁	Q ₅₂	Q ₅₃	Q ₅₄	Q ₅₅	Q ₅₆	Q ₅₇	Q ₅₈	Q ₅₉	Q ₆₀	Q ₆₁	Q ₆₂	Q ₆₃	Q ₆₄	Q ₆₅	Q ₆₆	Q ₆₇	Q ₆₈	Q ₆₉	Q ₇₀	Q ₇₁	Q ₇₂	Q ₇₃	Q ₇₄	Q ₇₅	Q ₇₆	Q ₇₇	Q ₇₈	Q ₇₉	Q ₈₀	Q ₈₁	Q ₈₂	Q ₈₃	Q ₈₄	Q ₈₅	Q ₈₆	Q ₈₇	Q ₈₈	Q ₈₉	Q ₉₀	Q ₉₁	Q ₉₂	Q ₉₃	Q ₉₄	Q ₉₅	Q ₉₆	Q ₉₇	Q ₉₈	Q ₉₉	Q ₁₀₀	Q ₁₀₁	Q ₁₀₂	Q ₁₀₃	Q ₁₀₄	Q ₁₀₅	Q ₁₀₆	Q ₁₀₇	Q ₁₀₈	Q ₁₀₉	Q ₁₁₀	Q ₁₁₁	Q ₁₁₂	Q ₁₁₃	Q ₁₁₄	Q ₁₁₅	Q ₁₁₆	Q ₁₁₇	Q ₁₁₈	Q ₁₁₉	Q ₁₂₀	Q ₁₂₁	Q ₁₂₂	Q ₁₂₃	Q ₁₂₄	Q ₁₂₅	Q ₁₂₆	Q ₁₂₇	Q ₁₂₈	Q ₁₂₉	Q ₁₃₀	Q ₁₃₁	Q ₁₃₂	Q ₁₃₃	Q ₁₃₄	Q ₁₃₅	Q ₁₃₆	Q ₁₃₇	Q ₁₃₈	Q ₁₃₉	Q ₁₄₀	Q ₁₄₁	Q ₁₄₂	Q ₁₄₃	Q ₁₄₄	Q ₁₄₅	Q ₁₄₆	Q ₁₄₇	Q ₁₄₈	Q ₁₄₉	Q ₁₅₀	Q ₁₅₁	Q ₁₅₂	Q ₁₅₃	Q ₁₅₄	Q ₁₅₅	Q ₁₅₆	Q ₁₅₇	Q ₁₅₈	Q ₁₅₉	Q ₁₆₀	Q ₁₆₁	Q ₁₆₂	Q ₁₆₃	Q ₁₆₄	Q ₁₆₅	Q ₁₆₆	Q ₁₆₇	Q ₁₆₈	Q ₁₆₉	Q ₁₇₀	Q ₁₇₁	Q ₁₇₂	Q ₁₇₃	Q ₁₇₄	Q ₁₇₅	Q ₁₇₆	Q ₁₇₇	Q ₁₇₈	Q ₁₇₉	Q ₁₈₀	Q ₁₈₁	Q ₁₈₂	Q ₁₈₃	Q ₁₈₄	Q ₁₈₅	Q ₁₈₆	Q ₁₈₇	Q ₁₈₈	Q ₁₈₉	Q ₁₉₀	Q ₁₉₁	Q ₁₉₂	Q ₁₉₃	Q ₁₉₄	Q ₁₉₅	Q ₁₉₆	Q ₁₉₇	Q ₁₉₈	Q ₁₉₉	Q ₂₀₀	Q ₂₀₁	Q ₂₀₂	Q ₂₀₃	Q ₂₀₄	Q ₂₀₅	Q ₂₀₆	Q ₂₀₇	Q ₂₀₈	Q ₂₀₉	Q ₂₁₀	Q ₂₁₁	Q ₂₁₂	Q ₂₁₃	Q ₂₁₄	Q ₂₁₅	Q ₂₁₆	Q ₂₁₇	Q ₂₁₈	Q ₂₁₉	Q ₂₂₀	Q ₂₂₁	Q ₂₂₂	Q ₂₂₃	Q ₂₂₄	Q ₂₂₅	Q ₂₂₆	Q ₂₂₇	Q ₂₂₈	Q ₂₂₉	Q ₂₃₀	Q ₂₃₁	Q ₂₃₂	Q ₂₃₃	Q ₂₃₄	Q ₂₃₅	Q ₂₃₆	Q ₂₃₇	Q ₂₃₈	Q ₂₃₉	Q ₂₄₀	Q ₂₄₁	Q ₂₄₂	Q ₂₄₃	Q ₂₄₄	Q ₂₄₅	Q ₂₄₆	Q ₂₄₇	Q ₂₄₈	Q ₂₄₉	Q ₂₅₀	Q ₂₅₁	Q ₂₅₂	Q ₂₅₃	Q ₂₅₄	Q ₂₅₅	Q ₂₅₆	Q ₂₅₇	Q ₂₅₈	Q ₂₅₉	Q ₂₆₀	Q ₂₆₁	Q ₂₆₂	Q ₂₆₃	Q ₂₆₄	Q ₂₆₅	Q ₂₆₆	Q ₂₆₇	Q ₂₆₈	Q ₂₆₉	Q ₂₇₀	Q ₂₇₁	Q ₂₇₂	Q ₂₇₃	Q ₂₇₄	Q ₂₇₅	Q ₂₇₆	Q ₂₇₇	Q ₂₇₈	Q ₂₇₉	Q ₂₈₀	Q ₂₈₁	Q ₂₈₂	Q ₂₈₃	Q ₂₈₄	Q ₂₈₅	Q ₂₈₆	Q ₂₈₇	Q ₂₈₈	Q ₂₈₉	Q ₂₉₀	Q ₂₉₁	Q ₂₉₂	Q ₂₉₃	Q ₂₉₄	Q ₂₉₅	Q ₂₉₆	Q ₂₉₇	Q ₂₉₈	Q ₂₉₉	Q ₃₀₀	Q ₃₀₁	Q ₃₀₂	Q ₃₀₃	Q ₃₀₄	Q ₃₀₅	Q ₃₀₆	Q ₃₀₇	Q ₃₀₈	Q ₃₀₉	Q ₃₁₀	Q ₃₁₁	Q ₃₁₂	Q ₃₁₃	Q ₃₁₄	Q ₃₁₅	Q ₃₁₆	Q ₃₁₇	Q ₃₁₈	Q ₃₁₉	Q ₃₂₀	Q ₃₂₁	Q ₃₂₂	Q ₃₂₃	Q ₃₂₄	Q ₃₂₅	Q ₃₂₆	Q ₃₂₇	Q ₃₂₈	Q ₃₂₉	Q ₃₃₀	Q ₃₃₁	Q ₃₃₂	Q ₃₃₃	Q ₃₃₄	Q ₃₃₅	Q ₃₃₆	Q ₃₃₇	Q ₃₃₈	Q ₃₃₉	Q ₃₄₀	Q ₃₄₁	Q ₃₄₂	Q ₃₄₃	Q ₃₄₄	Q ₃₄₅	Q ₃₄₆	Q ₃₄₇	Q ₃₄₈	Q ₃₄₉	Q ₃₅₀	Q ₃₅₁	Q ₃₅₂	Q ₃₅₃	Q ₃₅₄	Q ₃₅₅	Q ₃₅₆	Q ₃₅₇	Q ₃₅₈	Q ₃₅₉	Q ₃₆₀	Q ₃₆₁	Q ₃₆₂	Q ₃₆₃	Q ₃₆₄	Q ₃₆₅	Q ₃₆₆	Q ₃₆₇	Q ₃₆₈	Q ₃₆₉	Q ₃₇₀	Q ₃₇₁	Q ₃₇₂	Q ₃₇₃	Q ₃₇₄	Q ₃₇₅	Q ₃₇₆	Q ₃₇₇	Q ₃₇₈	Q ₃₇₉	Q ₃₈₀	Q ₃₈₁	Q ₃₈₂	Q ₃₈₃	Q ₃₈₄	Q ₃₈₅	Q ₃₈₆	Q ₃₈₇	Q ₃₈₈	Q ₃₈₉	Q ₃₉₀	Q ₃₉₁	Q ₃₉₂	Q ₃₉₃	Q ₃₉₄	Q ₃₉₅	Q ₃₉₆	Q ₃₉₇	Q ₃₉₈	Q ₃₉₉	Q ₄₀₀	Q ₄₀₁	Q ₄₀₂	Q ₄₀₃	Q ₄₀₄	Q ₄₀₅	Q ₄₀₆	Q ₄₀₇	Q ₄₀₈	Q ₄₀₉	Q ₄₁₀	Q ₄₁₁	Q ₄₁₂	Q ₄₁₃	Q ₄₁₄	Q ₄₁₅	Q ₄₁₆	Q ₄₁₇	Q ₄₁₈	Q ₄₁₉	Q ₄₂₀	Q ₄₂₁	Q ₄₂₂	Q ₄₂₃	Q ₄₂₄	Q ₄₂₅	Q ₄₂₆	Q ₄₂₇	Q ₄₂₈	Q ₄₂₉	Q ₄₃₀	Q ₄₃₁	Q ₄₃₂	Q ₄₃₃	Q ₄₃₄	Q ₄₃₅	Q ₄₃₆	Q ₄₃₇	Q ₄₃₈	Q ₄₃₉	Q ₄₄₀	Q ₄₄₁	Q ₄₄₂	Q ₄₄₃	Q ₄₄₄	Q ₄₄₅	Q ₄₄₆	Q ₄₄₇	Q ₄₄₈	Q ₄₄₉	Q ₄₅₀	Q ₄₅₁	Q ₄₅₂	Q ₄₅₃	Q ₄₅₄	Q ₄₅₅	Q ₄₅₆	Q ₄₅₇	Q ₄₅₈	Q ₄₅₉	Q ₄₆₀	Q ₄₆₁	Q ₄₆₂	Q ₄₆₃	Q ₄₆₄	Q ₄₆₅	Q ₄₆₆	Q ₄₆₇	Q ₄₆₈	Q ₄₆₉	Q ₄₇₀	Q ₄₇₁	Q ₄₇₂	Q ₄₇₃	Q ₄₇₄	Q ₄₇₅	Q ₄₇₆	Q ₄₇₇	Q ₄₇₈	Q ₄₇₉	Q ₄₈₀	Q ₄₈₁	Q ₄₈₂	Q ₄₈₃	Q ₄₈₄	Q ₄₈₅	Q ₄₈₆	Q ₄₈₇	Q ₄₈₈	Q ₄₈₉	Q ₄₉₀	Q ₄₉₁	Q ₄₉₂	Q ₄₉₃	Q ₄₉₄	Q ₄₉₅	Q ₄₉₆	Q ₄₉₇	Q ₄₉₈	Q ₄₉₉	Q ₅₀₀	Q ₅₀₁	Q ₅₀₂	Q ₅₀₃	Q ₅₀₄	Q ₅₀₅	Q ₅₀₆	Q ₅₀₇	Q ₅₀₈	Q ₅₀₉	Q ₅₁₀	Q ₅₁₁	Q ₅₁₂	Q ₅₁₃	Q ₅₁₄	Q ₅₁₅	Q ₅₁₆	Q ₅₁₇	Q ₅₁₈	Q ₅₁₉	Q ₅₂₀	Q ₅₂₁	Q ₅₂₂	Q ₅₂₃	Q ₅₂₄	Q ₅₂₅	Q ₅₂₆	Q ₅₂₇	Q ₅₂₈	Q ₅₂₉	Q ₅₃₀	Q ₅₃₁	Q ₅₃₂	Q ₅₃₃	Q ₅₃₄	Q ₅₃₅	Q ₅₃₆	Q ₅₃₇	Q ₅₃₈	Q ₅₃₉	Q ₅₄₀	Q ₅₄₁	Q ₅₄₂	Q ₅₄₃	Q ₅₄₄	Q ₅₄₅	Q ₅₄₆	Q ₅₄₇	Q ₅₄₈	Q ₅₄₉	Q ₅₅₀	Q ₅₅₁	Q ₅₅₂	Q ₅₅₃	Q ₅₅₄	Q ₅₅₅	Q ₅₅₆	Q ₅₅₇	Q ₅₅₈	Q ₅₅₉	Q ₅₆₀	Q ₅₆₁	Q ₅₆₂	Q ₅₆₃	Q ₅₆₄	Q ₅₆₅	Q ₅₆₆	Q ₅₆₇	Q ₅₆₈	Q ₅₆₉	Q ₅₇₀	Q ₅₇₁	Q ₅₇₂	Q ₅₇₃	Q ₅₇₄	Q ₅₇₅	Q ₅₇₆	Q ₅₇₇	Q ₅₇₈	Q ₅₇₉	Q ₅₈₀	Q ₅₈₁	Q ₅₈₂	Q ₅₈₃	Q ₅₈₄	Q ₅₈₅	Q ₅₈₆	Q ₅₈₇	Q ₅₈₈	Q ₅₈₉	Q ₅₉₀	Q ₅₉₁	Q ₅₉₂	Q ₅₉₃	Q ₅₉₄	Q ₅₉₅	Q ₅₉₆	Q ₅₉₇	Q ₅₉₈	Q ₅₉₉	Q ₆₀₀	Q ₆₀₁	Q ₆₀₂	Q ₆₀₃	Q ₆₀₄	Q ₆₀₅	Q ₆₀₆	Q ₆₀₇	Q ₆₀₈	Q ₆₀₉	Q ₆₁₀	Q ₆₁₁	Q ₆₁₂	Q ₆₁₃	Q ₆₁₄	Q ₆₁₅	Q ₆₁₆	Q ₆₁₇	Q ₆₁₈	Q ₆₁₉	Q ₆₂₀	Q ₆₂₁	Q ₆₂₂	Q ₆₂₃	Q ₆₂₄	Q ₆₂₅	Q ₆₂₆	Q ₆₂₇	Q ₆₂₈	Q ₆₂₉	Q ₆₃₀	Q ₆₃₁	Q ₆₃₂	Q ₆₃₃	Q ₆₃₄	Q ₆₃₅	Q ₆₃₆	Q ₆₃₇	Q ₆₃₈	Q ₆₃₉	Q ₆₄₀	Q ₆₄₁	Q ₆₄₂	Q ₆₄₃	Q ₆₄₄	Q ₆₄₅	Q ₆₄₆	Q ₆₄₇	Q ₆₄₈	Q ₆₄₉	Q ₆₅₀	Q ₆₅₁	Q ₆₅₂	Q ₆₅₃	Q ₆₅₄	Q ₆₅₅	Q ₆₅₆	Q ₆₅₇	Q ₆₅₈	Q ₆₅₉	Q ₆₆₀	Q ₆₆₁	Q ₆₆₂	Q ₆₆₃	Q ₆₆₄	Q ₆₆₅	Q ₆₆₆	Q ₆₆₇	Q ₆₆₈	Q ₆₆₉	Q ₆₇₀	Q ₆₇₁	Q ₆₇₂	Q ₆₇₃	Q ₆₇₄	Q ₆₇₅	Q ₆₇₆	Q ₆₇₇	Q ₆₇₈	Q ₆₇₉	Q ₆₈₀	Q ₆₈₁	Q ₆₈₂	Q ₆₈₃	Q ₆₈₄	Q ₆₈₅	Q ₆₈₆	Q
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FROM COPY FURNISHED TO DDC

UDMHMMH PLANT RUN DATA SHEETS

(D-1) Process
 _____ MMH, Date _____, Test No. _____
 _____ UDMH, Test Conductor _____
 Page _____ of _____

Run Time	Test Matrix	1st Stage Feed Rate		2nd Stage Feed Rate		System Pressures				Solvent RPM ± 100	Cond. Level L/Hr.	NACH Level Inches	Sample Taken					Vibrator	Flow Rates		T.P. Jacket	Electrostatic Precipitator						
Cont. Min.	Cont. Min.	Cl ₂ g/min	H ₂ g/min	H ₂ g/min	NACH ML/min	Water ML/min	Amine ML/min	T.P. P.S.I.	E.P. P.S.I.	Cont. P.S.I.	Spout P.S.I.		Y-1	Y-2	Y-3	Y-4	Y-5	From Min.	Force PSI	Product ML/min	Ward g/min	Upper GPM	Lower GPM	Volts	Amps	Sparks/min	Kilowatts	mA
21K 45	D	97.5	13.5	10.8	1.5	1.7	50.5	10.0	1.3	2.3	1870																	
22K 45	D	97.5	13.5	10.8	1.5	1.7	50.5	10.0	1.3	2.3																		
23K 45	D	97.5	13.5	10.8	1.5	1.7	50.5	10.0	1.3	2.3																		
24K 45	D	97.5	13.5	10.8	1.5	1.7	50.5	10.0	1.3	2.3																		
25K 45	D	97.5	13.5	10.8	1.5	1.7	50.5	10.0	1.3	2.3																		
26K 45	D	97.5	13.5	10.8	1.5	1.7	50.5	10.0	1.3	2.3																		
27K 45	D	97.5	13.5	10.8	1.5	1.7	50.5	10.0	1.3	2.3																		
28K 45	D	97.5	13.5	10.8	1.5	1.7	50.5	10.0	1.3	2.3																		
29K 45	D	97.5	13.5	10.8	1.5	1.7	50.5	10.0	1.3	2.3																		
30K 45	D	97.5	13.5	10.8	1.5	1.7	50.5	10.0	1.3	2.3																		
31K 45	D	97.5	13.5	10.8	1.5	1.7	50.5	10.0	1.3	2.3																		
32K 45	D	97.5	13.5	10.8	1.5	1.7	50.5	10.0	1.3	2.3																		
33K 45	D	97.5	13.5	10.8	1.5	1.7	50.5	10.0	1.3	2.3																		
34K 45	D	97.5	13.5	10.8	1.5	1.7	50.5	10.0	1.3	2.3																		
35K 45	D	97.5	13.5	10.8	1.5	1.7	50.5	10.0	1.3	2.3																		
36K 45	D	97.5	13.5	10.8	1.5	1.7	50.5	10.0	1.3	2.3																		
37K 45	D	97.5	13.5	10.8	1.5	1.7	50.5	10.0	1.3	2.3																		
38K 45	D	97.5	13.5	10.8	1.5	1.7	50.5	10.0	1.3	2.3																		
39K 45	D	97.5	13.5	10.8	1.5	1.7	50.5	10.0	1.3	2.3																		
40K 45	D	97.5	13.5	10.8	1.5	1.7	50.5	10.0	1.3	2.3																		

(D-2) Temperatures

UDMHMMH PLANT RUN DATA SHEETS

MMH, Date 2-22-78, Test No. 2

UDMH, Test Conductor J. C. H

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[illegible]

Run Cont.	Time @ Cond.	Test Conductor's Notes
		On same condition, per Run plan #2. As Williams made second fill above the condenser.
0200		Temperature, pressure, and flow measurements Condensate at 28.7%, resulting in five condensate tanks being in hydrocarbon in danger.
		Condensate with water inside. Condensate - expanded 1 pump away from it.
		Condensate with water inside. Condensate - expanded 1 pump away from it.
1200		30% condensate pump. Cuts out.
		Change to 100% condensate pump. OK now.
		30% condensate pump. Still cut out. Inquiries made - must have water inside and water
		and again 30% condensate pump at pressure in line ready.
RWD at 2		Long Way 1:11:30. Pressure now 10% condensate pump and inhibited. Permit condensate pump
		and steam condenser.
		Up at 1:00 for Run 3. System power on at 1:10, before activation. Very windy.
		1:10 at 1:11:30 - address 10% - This was caused by the change out of 30% pump
		and observation of 10% in system (system) for shut out
1210		but no time for condition 1, because more the 10% already above the 10%.
		10% to 10% 1
1220		Condensate at 10%, continuing to add 10%.
1230		Dump 10%.
1240		Switching off boiler.
1250		Dump 10%.
1300		Condensate at 10%; attempting to go back to cond. 1. 10%.
1310		Condensate at 10%.
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ID-4 Test Conductors Notes		
Date 4-23-71 Run No. 2		
Run Card	Time of Card	Test Conductor's Notes
001		Runup 1000
002		T.C. 20 sec. Runup 1000; 1st in scheduled position
003		2nd Runup to 2000 at 2.4 sec; reaching at 2.5 sec
004		Runup 1000
005		2nd Runup to 2000 at 2.4 sec; reaching at 2.5 sec
006		Runup 1000
007		Runup 1000
008		Runup 1000
009		Runup 1000
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011		Runup 1000
012		Runup 1000
013		Runup 1000
014		Runup 1000
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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 ₂ O	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 ₂ Cl	chloramine

NH ₄ Cl	ammonium chloride
SAMSO	Space and Missile Systems Organization
TP	thermal precipitator
UDMH	unsymmetrical dimethylhydrazine