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PROCESS ENGINEERING DESIGN FOR MANUFACTURE OF GUANIDINE NITRATE

N. W. Steele, et al

Hercules, Incorporated

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N. W. Steele, J. A. Doyle, M. G. Whippen, J. A. Gorton

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This report (Vol. II) completes the technical, operating and economic studies for a 3-year pilot plant demonstration of the urea/ammonium nitrate (U/AN) route to guanidine nitrate (GN) as an intermediate for manufacture of nitroguanidine (NQ). Vol. I covered the laboratory and pilot plant program through 1971-72; this Vol. II summarizes the 1973 effort, including plant modifications, production of 10 tons of specification GN, economic comparison of the U/AN and British Aqueous Fusion (BAF) processes for GN, and layaway of the GN pilot plant. As summarized in Vol. I, catalyst lifetime problems due to impurities in the feed AN were encountered in the first production efforts, and these were satisfactorily resolved in early 1973. The pilot plant subsequently operated well, and an economic catalyst (Houdry CP-532 silica bead) lifetime was shown in the reactors which are basically production prototypes in design and size. Following limited equipment and line modifications, the GN pilot plant was operated as a totally integrated unit for nearly 3 months. Actual cumulative operations time was 62 days. About 10 tons of 95.9% average purity GN from the 1973 campaign and 827 lb GN (95%) made in 1972 were shipped to Cyanamid of Canada and converted to NQ. The NG product met all military specifications. The concept of the U/AN process for manufacturing GN via a melt reactor plus a single aqueous crystallization step was demonstrated. Finally, results from this program are sufficient to yield a confident plant design, and the U/AN process can be recommended for commercialization.

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Packed bed tubular reactors						
Catalyst mileage						

PROCESS ENGINEERING DESIGN FOR MANUFACTURE OF GUANIDINE NITRATE

Final Report

Volume II of Volumes I and II

N. W. Steele
J. A. Doyle
M. G. Whippen
J. A. Gorton

HERCULES INCORPORATED
KENVIL, NEW JERSEY

The findings in this report are not to be construed as an official Department of the Army position.

ABSTRACT

This report (Volume II) completes the technical, operating and economic studies for a 3-year pilot plant demonstration of the urea/ammonium nitrate (U/AN) route to guanidine nitrate (GN) as an intermediate for manufacture of nitroguanidine (NQ). Volume I covered the laboratory and pilot plant program through 1971-72; this Volume II summarizes the 1973 effort, including plant modifications, production of 10 tons of specification GN, economic comparison of the U/AN and British Aqueous Fusion (BAF) processes for GN, and layaway of the GN pilot plant.

As summarized in Volume I, catalyst lifetime problems due to impurities in the feed AN were encountered in the first production efforts, and these were satisfactorily resolved in early 1973. The pilot plant subsequently operated well, and an economic catalyst lifetime was shown in the reactors which are basically production prototypes in design and size.

Following limited equipment and line modifications, the GN pilot plant was operated as a totally integrated unit for nearly three months. Actual cumulative operations time was 62 days, resulting in 6,456 total reactor hours. About 10 tons of 95.9% average purity GN from the 1973 campaign and 827 lb GN (95%) made in 1972 were shipped to Cyanamid of Canada and converted to nitroguanidine. The NQ product met all military specifications.

The concept of the U/AN process for manufacturing GN via a melt reactor plus a single aqueous crystallization step was demonstrated. The plant, employing prototype production equipment and exact models of production reactors, ran well. About 30 to 40 system turnovers were experienced with no evidence of impurities buildup in the recycle stream. System upsets and process variations were encountered, and the process demonstrated its stability by accepting changes without any serious adverse effects.

Houdry CP-532 silica bead catalyst was used throughout the run. A minimum mileage of 188 lb GN/lb catalyst was obtained, a reasonable value for production plant operation. Some of the packed bed tubular reactors became plugged from a buildup of ammelide in the system. This problem can be minimized in a plant by specifying suitable process equipment and optimizing the feed AN/U ratio. Good guanidine nitrate yields based on urea and ammonium nitrate were demonstrated: urea = 79.5% (2-mole stoichiometry); ammonium nitrate = 105.5%. Expected yields in a production plant are > 90% and > 95%, respectively. Data showed that nitrates are conserved in the reactor and that high concentration urea feed results in urea losses other than those expected from hydrolysis. The economic analysis used conservative yield values.

A comparative economic analysis showed that the U/AN-GN process has definite cost advantages over the BAF route for a total GN-NQ facility. Results from this program are sufficient to yield a confident plant design. The U/AN process can be recommended for commercialization.

FOREWORD

This program was conducted by Hercules Incorporated, Kenvil, New Jersey, under contract DAAA 21-71-C-0193 with Picatinny Arsenal. The Contract Project Officer for this contract at Picatinny Arsenal was Mr. C. H. Nichols. The Hercules Incorporated Program Manager was Mr. N. W. Steele (Kenvil). Hercules/Kenvil principal investigators were Messrs. J. A. Doyle and M. G. Whippen. Technical assistance was supplied by the following Corporate Home Office (Wilmington, Delaware) personnel: Dr. J. A. Gorton, Mr. A. R. Bookout and Mr. D. M. Clarke, Engineering Department; and Dr. R. S. Voris, Systems Group.

TABLE OF CONTENTS

TABLE OF CONTENTS (CONTINUED)

TABLE OF CONTENTS (CONTINUED)

LIST OF FIGURES

LIST OF FIGURES (CONTINUED)

LIST OF FIGURES (CONTINUED)

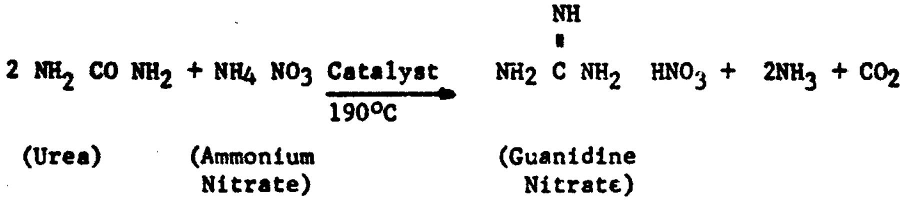
LIST OF TABLES

LIST OF TABLES (CONTINUED)

I. DIGEST

A. OBJECTIVE AND BACKGROUND

The objectives of this program (Contract DAAA21-71-C-0193) were to develop basic design data and to produce a quantity of guanidine nitrate (GN) of suitable quality for conversion to nitroguanidine at the Welland Cyanamid of Canada plant. The process for producing GN is based on the catalytic reaction of ammonium nitrate and urea at about 190°C according to the following equation:



$$2\ NH_2\ CO\ NH_2 + NH_4\ NO_3 \xrightarrow[190^{o}C]{Catalyst} NH_2\ \overset{NH}{\overset{\|}{C}}\ NH_2\ \ HNO_3 + 2NH_3 + CO_2$$

(Urea) (Ammonium Nitrate) (Guanidine Nitrate)

The original patent (U.S. 2,783,286)(1) for the above chemical route to GN was issued to L. G. Boatright and J. S. Mackay, American Cyanamid Company on February 26, 1957.

This program was designed for four separate phases ranging from laboratory process confirmation/kinetic studies through pilot plant GN production and culminating in battery limits process design criteria. Program results from October 1970 through January 1973 were presented in a Final Report - Volume I,(2) dated August 1973. This Volume II completes the summary of pilot plant and economic studies through September 1973.

B. SUMMARY OF VOLUME I

Laboratory and pilot plant experiments confirmed the soundness of the process as described in the literature. Mathematical models and computer programs developed on the basis of laboratory kinetic and process variable data proved to be capable of predicting the behavior of packed bed tubular reactors. A semi-continuous pilot plant with a nominal capacity of 50 lb GN/hr was constructed on the basis of laboratory data and subsequently operated as an integrated unit. The plant, containing eight parallel 4-inch-diameter tubular reactors, employed production plant prototype equipment. About one ton of 96+% purity GN was produced using a single, aqueous crystallization step for product recovery. This material was capable of being converted to nitroguanidine comparable to NQ produced in the Cyanamid production facilities. Total production was not met because of catalyst poisoning which was traced to phosphates and perhaps borates in commercial grade ammonium nitrate. When commercial additive-free AN and urea were used with Houdry CP-532 macroporous silica beads (the only known suitable catalyst for the packed bed reactors) in a 4-inch-diameter production plant model reactor, a minimum mileage of 38 lb GN per lb catalyst was obtained. Accompanying this mileage were a urea conversion of 83% and a urea-to-GN yield (based on 2-mole stoichiometry) of 95%. A minimum mileage of 65 gm GN per gm catalyst was demonstrated in a 1-inch-diameter downflow reactor. Unreacted AN and urea were not recycled for either of these experiments. These results represented a significant technological advancement for the manufacture of GN via the urea/ammonium nitrate process, but did not provide an adequate engineering base for reliable, economic production design.

A proposal was submitted to Picatinny Arsenal to continue the program, with some changes in the scope of work, through the third quarter of 1973. The executed contract modification covered additional pilot plant operations, an economic study to compare the relative nitroguanidine manufacturing costs via both the British Aqueous Fusion and urea/ammonium nitrate guanidine nitrate processes and layaway of the pilot plant.

C. SUMMARY OF VOLUME II

1. Modifications

The pilot plant was modified to effect smoother operations and to minimize operating downtime. Examples of changes included routing of 150 psig plant steam to the pilot plant, installation of larger diameter and better steam traced melt process lines, installation of individual feed flow controllers on each reactor, relocation of some equipment for improved and safer operations, etc.

2. Production

The pilot plant was operated from May 21 until August 10, 1973 (except for a scheduled two-week plant shutdown) as an integral unit employing fresh makeup plus recycle ammonium nitrate and urea as feed for the reactors. The number of reactors on stream at any one time varied from one to eight. Total cumulative reactor time was 6,456 hours, resulting in the production of 19,240 lb of on-grade guanidine nitrate. This material, plus 827 lb of GN produced during the 1972 campaign, was shipped to Cyanamid of Canada and subsequently converted to nitroguanidine which met all military specifications. Average chemical analysis of GN produced during the 1973 pilot plant campaign and shipped to Cyanamid is shown below. The minimum target GN content of production material for producing nitroguanidine is included. Higher GN assays are desirable to minimize sulfuric acid purging in the NQ plant.

	Average Analysis of 1973 GN to Cyanamid, %	Target GN Product Analysis
Guanidine Nitrate	95.9	> 94.0
Ammonium Nitrate	2.4	< 4.0
Urea	0.4	< 1.0
Water Insolubles	0.5	< 1.25
Water	0.8	< 2.0

3. Catalyst Performance

Reactor R-200 was the first reactor placed in service and consequently was the reactor with the greatest number of cumulative operating hours. During its period of performance, the other operating reactors were removed temporarily from service two times to determine that the catalyst in R-200 was still active. Reactor R-200, stopped and started on seven occasions, was in service for a total of 1,026 hours. On the basis of productivity calculations, using reactor feed plus product analysis and a nitrate conservation calculation technique, a mileage of 188 lb GN per lb catalyst was demonstrated. Average productivity of the pilot plant reactors during this 1,026-hour operating period was about 4.5 lb GN/hr/reactor tube at 190°C, about 22 lb feed/hr, and total recycle. Ultimate mileage of the Houdry CP-532 silica bead catalyst was not determined; however, a conservative value of 200 lb GN/lb catalyst was used in the economic study. Commercial, additive-free ammonium nitrate and urea were used for pilot plant feed stock.

Some attrition of the Houdry CP-532 silica bead catalyst was noted when it was inspected after 37 days of cumulative operating time. About 10% (2.5 lb) of the catalyst was gone. This represents a catalyst consumption rate of about 0.6 lb catalyst per 1000 lb GN produced. If the catalyst depleted to about 60% [illegible] original depth, the urea conversion would be about 80% of the value for a fully loaded reactor based on mathematical model predictions. Using this criterion as the minimum acceptable urea conversion level, a single reactor tube could conceivably produce about 17,000 lb of GN before charging new catalyst.

Houdry CP-532 silica bead catalyst is presently a pilot plant material. Air Products and Chemicals, Inc., will either produce the catalyst or supply a design for a government plant to produce catalyst. A review of Air Products proprietary data does not indicate any major problems regarding the manufacture of this catalyst.

4. Process Yields

Productivity during the 1973 pilot plant operations, calculated on the basis of recovered product and total reactor operating hours, averaged about 3 lb of GN per reactor hour. Day-to-day calculations of reactor productivity based on a nitrate conservation technique, showed an overall average value of 4 to 4.5 lb GN/hour/reactor. The difference in the two values was due to observed process losses. It was shown that ammonium nitrate did not decompose in the reactor, that 1% of the reactor product melt was entrained in the NH_3 - CO_2 gas stream, and that approximately 1-1/2% of the AN in the evaporator feed stream was volatilized in the recycle evaporator. By comparing the predicted and actual quantities of NH_3 and CO_2 found in the off-gas scrubber outlets, it was shown that a nominal one mole of ammonium carbamate is produced for each mole of guanidine nitrate. Analyses of plotted calculated results showed that a reactor urea yield of 100% (based on a 2-mole stoichiometry) is reasonable at an AN/U feed molar ratio of about 1.5. At high urea concentrations ($AN/U \leq 1$), total urea losses are greater than those predicted from hydrolysis. At AN/U ratios greater than 1.5, urea hydrolysis efficiency decreases, indicating a yield advantage in operating the reactors with high AN feed.

A two-day material balance was made over the entire pilot plant with a reactor feed AN/U molar ratio approximating 1.5. Process materials resulting from leaks were returned to the system. Accountability of urea, ammonium nitrate and guanidine nitrate was accomplished by direct weighing, tank levels and numerous analyses. Extraneous material losses and urea equivalents were calculated on the basis of data presented above. Results of the material balance are as follows:

Total weight closure	100.7%
Ammonium nitrate weight balance	103.0
Urea weight balance	108.4
GN yield based on urea (2-mole ratio)	79.6
GN yield based on AN	105.5

The GN-from-urea yield loss can be attributed to hydrolysis from water in feed (ca 18%) and insolubles formation (ca 2%). For production plant design purposes, urea and AN yields of 90% and >95%, respectively, can be used.

5. Operations

Reactors were brought on stream sequentially, and within one month all eight reactors were operating. The plant operated very well mechanically with recycle of unreacted AN and urea. Analytical closures of the evaporator bottoms recycle stream were consistent throughout the total pilot plant operation, indicating that the recycle operation does not produce impurities. A shutdown of the eight reactors was required because of the inability to sustain individual reactor feed rates. Post examination of the elbows and catalyst retention screens at the tops of the reactors revealed heavy deposits of insolubles (ammelide). This condition was the direct result of having taken the solid bowl centrifuge (used for insolubles removal) out of service temporarily, thus permitting ammelide to build up in the system. This condition, perhaps the most serious upset condition that could be experienced, produced scaling in the recycle equipment and transfer lines. The performance of the recycle evaporator was adversely affected by this upset. Continuous removal of water insolubles from the system is important.

During the above shutdown, catalyst was changed in three reactors. On startup, seven of the eight reactors resumed operation. Following a scheduled two-week shutdown, only three of the previously six functioning reactors could be placed in service. At the very end of the campaign, only one reactor was functioning. The primary reason for reactor bed plugging has been attributed to ammelide. In a production plant with feed pumps developing higher discharge pressures than the pilot plant feed pump, the problem of feeding partially restricted reactor tubes would not be as serious. The problem of reactor bed plugging would be minimized by employing reliable insolubles removal equipment and by optimizing the AN/U feed molar ratio.

The pilot plant was subjected to other upset conditions and process variables such as AN/U feed ratio, water in feed, inadvertent diversion of reactor product from system, varying solids content of reactor quench stream, loss of reactor tubes, startup of reactor tubes, etc. Daily calculations of reactor productivity showed normal variations and major swings. The major productivity swings could be explained by a process variable change or an upset. It is interesting to note that there was not a gradual decline of productivity as a function of time, indicative that the catalyst activity was not decaying. Analysis of the data showed that the U/AN process for manufacturing GN is a stable operation that can accept natural process upsets and variations without deleterious effects on long-term operations or product quality.

Guanidine nitrate recovery via water quenching of the reactor product, removal of the insolubles, and single aqueous crystallization followed by a centrifuge operation was totally satisfactory. If all of the operations are performed properly, the resulting GN product will be of good quality and will be essentially independent of reactor operating conditions.

6. Corrosion

Corrosion coupons of different types of aluminum and stainless steel installed in three separate areas in the pilot plant showed that aluminum exhibits significant corrosion characteristics, whereas stainless steel was unaffected. Some of the aluminum corrosion could have been attributed to the short term presence of sodium carbonate wash solutions employed for removing ammelide from the system. Visual inspection of the all-stainless steel pilot plant equipment did not reveal any corrosion.

7. Pilot Plant Layaway

Following completion of operations, the pilot plant was placed in a layaway condition by removing catalyst from all reactors, cleaning all equipment, painting pump frames, etc., tagging all equipment for proper identification, and locking all doors. Repairs and permanent installation of lines, pumps, etc. would be required to continue operations. The equipment can, however, be transported to another location. A 1000-lb lot of Houdry CP-532 silica beads, recently procured by Picatinny Arsenal, is also stored in the pilot plant building.

8. Reactor Off-Gas Disposition

An analysis was performed to determine the best of several alternatives for utilization of the ammonia and carbon dioxide off-gases from the U/AN process for manufacturing guanidine nitrate. Options centered around utilization of one or both of the off-gas components for producing feedstock or selling the off-gases and purchasing AN and urea. The case selected for use in the economic study, and approved by the Army, was as follows: Separate the NH_3 and CO_2 in the off-gas, vent the CO_2 to the atmosphere and liquefy the ammonia, and provide seven days storage for internal use or for sale.

9. Economic Study

An economic analysis study was performed to compare the total cost, nonrecurring and recurring, for the manufacture of nitroguanidine (NQ) via two alternatives:

(a) Utilizing GN manufacture via the British Aqueous Fusion Process (BAF), and

(b) Utilizing GN manufacture by the urea/ammonium nitrate (U/AN) process.

The analysis showed that the U/AN - GN process is more economical than the BAF - GN process. Over an economic life of ten years, the difference at 100% operating rate is $11,000,000 to $13,000,000 or 13.5% to 15.6% (based on U/AN - GN costs).

D. CONCLUSIONS AND RECOMMENDATIONS

The U/AN process concept for manufacturing guanidine nitrate has been demonstrated on a pilot-plant scale. The integrated pilot plant, employing a single aqueous GN crystallization step and recycle of unreacted AN and urea, was operated for a cumulative period of about 62 days. This represents about 30 to 40 system turnovers with no evidence of impurities buildup or catalyst activity decay. Data generated during this operating period are sufficient for a confident production plant design. Quality of GN product was repeatedly good, emphasizing that the wet work-up end of the process, if operated properly, is independent of the number of reactors on-stream or specific reactor conditions. The overall process is stable and can accept normal process upsets and/or variations. A buildup of ammelide in the system, resulting from unreliable process equipment, has detrimental effects throughout the system.

Guanidine nitrate shipped to Cyanamid of Canada (10 tons) was successfully converted to nitroguanidine meeting all military specifications. There is no reason to believe that NQ resulting from U/AN - GN cannot be used as a direct replacement for NQ now being incorporated into cannon propellants.

A catalyst mileage of about 200-lb GN per lb catalyst was demonstrated with physically superior Houdry CP-532 macroporous silica beads. An improved mileage is reasonable. Problems were encountered with catalyst bed plugging. This problem has been attributed to buildup of ammelide in the system. Proper selection of removal equipment and optimization of the reactor feed AN/U molar ratio would minimize the problem. Some catalyst attrition was evident, but the magnitude was not great. Houdry silica bead catalyst can be made available for a production plant.

Satisfactory urea and ammonium nitrate yields for a production plant design were demonstrated. Data showed that nitrates are conserved in the reactor. High AN/U molar ratios (e.g., ≥ 1.5) in the feed favor improved urea to GN yields. At low AN/U ratios (e.g., <1.0), urea yields are decreased and the two-mole urea stoichiometry is no longer valid.

Production prototype equipment was satisfactorily demonstrated for all of the basic unit operations except for drying GN. Laboratory data obtained from a Wyssmont Turbo Tray dryer will enable the selection of a suitable GN dryer. The pilot plant reactor configurations were identical to those envisioned for a production plant. Specifications of processing equipment for a production plant should not present any problems.

The U/AN - GN process is chemically and operationally simple. The U/AN - GN process utilizes readily available raw materials and does not produce any undesirable by-products.

Results of an economic analysis for the manufacture of nitroguanidine utilizing GN made by the U/AN and BAF processes show that the U/AN process has a significant 10-year life span cost advantage.

On the technical and economic grounds, the U/AN process for manufacturing guanidine nitrate can be recommended for commercialization.

II. TECHNICAL ASSESSMENT

From the results of this program, it is concluded that the U/AN - GN process is an acceptable, proven operation. The reasons for this overall assessment are presented below.

During Phase I of this program, the 1955 work of Mackay (Pittsburgh Coke & Chemical Co.) was confirmed in one-liter batch reactor experiments. The extensive kinetic data were utilized to develop mathematical models for predicting the performance of both tubular and stirred tank packed bed reactors. Stirred tank reactors appeared marginally better than tubular reactors from the standpoint of conversion and yield. However, they did present problems in regard to practical and economic design and unknown hazards characteristics. Various catalysts were shown to be useful in the batch reaction of urea (U) and ammonium nitrate (AN) to yield guanidine nitrate (GN). The most promising were the Mobil Sorbeads, Grace 59 silica gel, and Houdry CP-532 macroporous silica beads. Houdry beads were subsequently determined to be the only one of the three candidate catalysts suitable for a packed bed reactor application. Analysis of off-gas (NH_3 and CO_2) from the batch reaction showed it to be equivalent to ammonium carbamate as predicted. Studies yielded a simple process where the reactor product melt is diluted with water to isolate ammelide which is then removed by centrifugation. The clear filtrate is cooled, crystallized and centrifuged to yield high-quality guanidine nitrate.

A 2-inch-diameter, continuous, packed bed reactor was built and operated to determine whether the transition from batch to continuous operations offered problems. Operations were performed only on a one shift per day basis, but the results were promising enough to justify proceeding to design and construction of a pilot plant.

A 50 lb guanidine nitrate per hour pilot plant was designed, built and operated at Hercules/Kenvil, N.J., to demonstrate reactor scale-up from 2-inch to 4-inch diameter, catalyst mileage, product work-up, and mother liquor recycle as well as to produce GN for a large-scale conversion to nitroguanidine at the Cyanamid of Canada Welland plant. The pilot plant was also operated to demonstrate prototype equipment for a large-scale manufacturing plant. Laboratory conversions of pilot plant GN to nitroguanidine at Welland were successful. Hazards analysis and sensitivity test results showed that in-process material would not transit from a deflagration to a detonation and that the overall process, if properly designed, was relatively safe. Certain anhydrous compositions will detonate if subjected to a sufficient external source shock stimuli.

For the most part, the objectives of the pilot plant program were achieved. Scale-up to the 4-inch-diameter tube was achieved with results in accord with those predicted from the mathematical model. Problems were encountered with physical breakdown of catalyst, but this problem would be minimized under proper operating conditions. After a disappointing operational campaign in 1972, marked by unreliable steam availability and rapid catalyst activity decay, a one-inch diameter reactor program was initiated. This program demonstrated that catalyst poisoning was caused by phosphates in the ammonium nitrate (AN). With reagent-grade AN, and 1% water in the reactor feed, a good mileage (65 gm GN/gm catalyst) was demonstrated with Houdry silica beads. A follow-up run in a single 4-inch-diameter reactor without recycle confirmed that a good catalyst mileage could be obtained with a demonstrated minimum mileage of 38 lb GN per lb of catalyst. The GN yield on the basis of urea consumed was 95%.

A second pilot plant campaign was performed in 1973, following equipment modifications and receipt of commercial, additive-free, uncoated ammonium nitrate. This particular grade of AN, supplied by Hercules Incorporated/Donora, meets reagent grade specifications. This campaign demonstrated the fundamental soundness of the recycle operation, the soundness of the process equipment (with the exception of the dryer), a minimum Houdry silica bead catalyst mileage for design purposes of 200 lb GN per lb catalyst, and the ability of the process to produce uniformly high quality GN. Shipments of GN product (95 + % purity) to Cyanamid of Canada totaled 10 tons. Nitroguanidine prepared from this GN met all military specifications. Separate laboratory studies on GN drying yielded sufficient information to permit specification of a GN dryer.

The chemical and operating simplicity of the U/AN process is attractive. The U/AN process does not require an operation for manufacturing a raw material, and recycling of unreacted AN and urea is a simple matter of water evaporation. Reactor off-gas disposition is independent of the plant operation. The U/AN process is envisioned as being easy to stop and start and can probably be operated easily at reduced rates.

The acceptable catalyst for the U/AN process is at present made only by Air Products and Chemicals Incorporated. A review of proprietary information supplied by Air Products to the U. S. Government shows that the catalyst manufacturing operation is fundamentally simple and does not present any serious operating problems. A suitable means for disposing of by-product salts (ammonium nitrate and sodium sulfate) must be provided. The unique feature of the operation is a bead-forming machine invented by Air Products and used extensively for producing cracking catalysts since 1941. Air Products and the U. S. Government have signed an intent for a possible catalyst manufacturing facility at Sunflower Army Ammunition Plant (scheduled site of nitroguanidine facility) in the event that Air Products does not have production facilities. Related patents are as follows: U. S. patent 2,665,258, Bead Forming Process, January 5, 1954; and Canadian patent 646,409, Catalyst on Silica Support, August 7, 1962.

The minimum catalyst mileage of 200 lb GN per lb catalyst demonstrated in the pilot plant will likely be exceeded in a production plant. It is also possible that a cheaper catalyst will be found in the future which is a common experience for new processes.

The equipment used for the GN manufacture by the U/AN process is simple, conventional chemical plant equipment, and essentially of standard design. There is little risk of process failure inasmuch as plant prototypes of equipment were demonstrated in the Kenvil pilot plant. Furthermore, Kenvil pilot plant data suggest that the nominal 85% value used in the economic study for the yield of GN from urea (based on the 2-mole urea stoichiometry) can be improved on a production scale.

Based on pilot plant data, it is firmly believed that a production plant can be operated to produce GN at the design rate and yield levels. In a time-of-war atmosphere, a fertilizer manufacturer could set up quickly to make GN by the U/AN process.

It is concluded that the U/AN process is viable. The U/AN process is economically attractive in both operating and capital costs.

III. INTRODUCTION

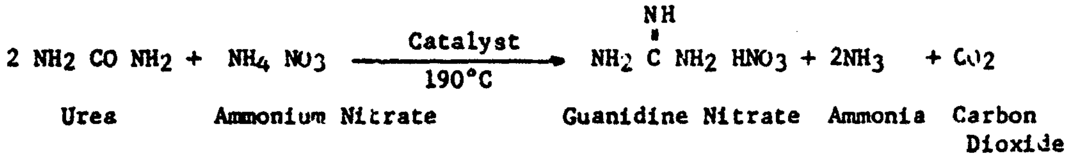
A. HISTORICAL BACKGROUND

Nitroguanidine is an important component of triple-base cannon propellants. It is cool burning and high in nitrogen content. These properties are of particular importance since they lead to formulations yielding a flashless exhaust and reduce erosion. Guanidine nitrate (GN), the intermediate from which nitroguanidine (NQ) is manufactured, is converted to nitroguanidine by sulfuric acid dehydration or "nitration" of GN, a process that is well understood from an engineering standpoint.

Presently, all nitroguanidine used by the U. S. military is manufactured in Canada by the Welland Process. However, the technology on which this process is based has been surpassed by several new processes. The lack of a domestic source of nitroguanidine has been a continuing concern of the Army Munitions Command (now Armament Command). Because of this concern, construction plans were prepared in the mid-1950's for building a facility at Pryor, Oklahoma, for production of guanidine nitrate based on a modified Welland process. These plans were not completed.

About twenty chemical routes exist for the production of guanidine nitrate, but most of these are quite expensive and impractical for commercial consideration since they involve uncommon and expensive raw materials and require technically difficult processing conditions. Four of the possible routes, however, have shown sufficient economic promise either to have been studied extensively on pilot scale or to have been commercialized. One route is represented by Welland ("Dicy") process and is currently the source of supply to the United States via Cyanamid of Canada. The British Aqueous Fusion (BAF) process, a more efficient version of the Welland process, is used by the British to produce nitroguanidine (NQ). Hercules Incorporated is now completing a final design employing advanced processing technology for the U. S. Government for production of NQ by the BAF process. This design includes safety site plans at the Sunflower Army Ammunition Plant. Both the Waterbury (ammonium thiocyanate) and the Roberts fusion processes have been studied extensively in pilot plants, but the need for and the feasibility of commercializing them have not materialized.

All four of the above processes have disadvantages, either because they are not economical or because they pose technical or waste disposal problems. For these reasons, there is interest in a fifth process. This process for manufacturing guanidine nitrate is based on the reaction of molten urea and molten ammonium nitrate on silica catalyst. The process was patented by L. C. Boatright and J. S. Mackay, American Cyanamid, on February 26, 1957 (Patent U. S. 2,783,276).(1) The basic chemistry of the process is as follows:



$$2\ NH_2CONH_2 + NH_4NO_3 \xrightarrow[190°C]{Catalyst} NH_2\overset{NH}{\overset{\|}{C}}NH_2\ HNO_3 + 2NH_3 + CO_2$$

Urea Ammonium Nitrate Guanidine Nitrate Ammonia Carbon Dioxide

Subsequent patents were issued to J. S. Mackay, Pittsburgh Coke and Chemical Company (Patent U. S. 2,949,484, August 16, 1960)(3) for increased yields, etc., and to E. Roberts and T. Martin, Minister of Aviation in Her Majesty's Government (Patent U. S. 3,043,878, July 10, 1962)(4) for the eutectic crystallization of guanidine nitrate. This process (BMR) consists of contacting an equimolar mixture of urea and ammonium nitrate with silica gel at an elevated temperature (ca. 190°C) to form a melt rich in guanidine nitrate and an off-gas containing carbon dioxide and ammonia. The GN is recovered by crystallization and the resulting mother liquor is evaporated for recycle of the unreacted urea and ammonium nitrate plus guanidine nitrate. The Boatright-Mackay and BMR processes offer distinct advantages over the BAF process in terms of cheaper raw materials, less water usage, fewer and simpler unit operations, and production of minimum quantities of by-products.

B. PROGRAM SUMMARY THROUGH JANUARY 1973

In response to Picatinny Arsenal Solicitation No. DAAA-21-70-Q-0211, Hercules Incorporated proposed to undertake a process engineering design for the manufacture of guanidine nitrate (GN). Hercules Incorporated was subsequently awarded a 23-month contract effective October 1970. The approach used was based on the Boatright-Mackay-Roberts (BMR) process noted above. The original program consisted of four distinct phases listed below:

Phase I - Laboratory, Engineering, Economic and Technology Study
Phase II - Pilot Plant Design
Phase III - Guanidine Nitrate Pilot Plant Construction and Operation
Phase IV - Guanidine Nitrate Production Plant Battery Limit Process Design Criteria

Following successful completion of the laboratory and pilot plant construction phases, technical problems were encountered in the operating phase. The problem basically involved catalyst poisoning. The catalyst poisoning problem was resolved in an extension of Phase III, and an acceptable catalyst mileage was demonstrated. This technical breakthrough was the basis for program continuation from February 4, 1973, until October 1, 1973 (Contract Modification No. P-0013). The results obtained during the initial portion of the program (October 1970 to January 1973) have been presented in detail in Final Report - Volume I, August 1973.(2) To serve as a background for the results presented in this report, Volume I results are summarized below.

Laboratory investigations related to determination of kinetic data and mathematical models, hazards analysis, and installation/operation of a pilot plant were completed. Investigations verified previous literature as related to the basic reaction mechanism. The resulting data were employed to develop kinetic expressions and, subsequently, mathematical models for predicting type, size, and performance of catalytic reactors. Various catalysts were evaluated leading to the selection of three types for pilot plant use. Limited experimental data from a 2-inch-diameter up-flow reactor verified the soundness of the tubular reactor mathematical model. Hazards analysis of the U/AN process, based on selected pilot plant equipment, showed that none of the individual process materials or streams would transit from a deflagration to an explosion. Through a fault-tree analysis, it was shown that the process is basically safe. Preliminary economic cost analyses showed that the U/AN process has definite cost advantages. Recovery of guanidine nitrate from the reaction melt, completed in a single aqueous step, was simpler than the Roberts-Martin dual eutectic-aqueous crystallization system.

A pilot plant was designed and constructed at Hercules/Kenvil based on the results of the laboratory and bench-scale investigations. A schematic of the urea/ammonium nitrate/silica process for manufacturing guanidine nitrate is presented in Figure 1A. The pilot plant contained eight parallel tubular reactors, 4-inch diameter by 12 foot tall, backed up by the necessary support equipment and unit operations for a totally integrated plant. The reactors were sized to duplicate those envisioned for a production plant (on the basis of heat transfer, safety and predicted performance); most of the remaining pieces of equipment were production prototypes. Design capacity of the pilot plant was 50-lb of guanidine nitrate per hour.

Operation of the pilot plant demonstrated the process concept and verified the predicted productivity using Houdry CP-532 macroporous silica bead catalyst. Two other catalysts, noted below, also were employed. Mechanically, the plant ran well and produced about one ton of GN with 96+% purity over a period of about three months. A representative sample of the product was converted to nitroguanidine (NQ) at Cyanamid of Canada. The resulting NQ was equivalent to that from a control sample of Cyanamid's normal production GN. Production of the contractual 40,500 lb of GN was not realized because of technical problems. Two major problems were encountered: (1) loss of reactor productivity and (2) catalyst decrepitation. The results of a continuing program to resolve these two problems are summarized below.

The primary reason for catalyst deactivation was traced to a crystal habit modifier (primarily phosphate) that is normally added to prilled ammonium nitrate to prevent prill breakage. Via a logic diagram and utilizing a 1-inch-diameter downflow reactor, a combination of reagent grade AN prills (no additives) and Olin commercial urea prills was found to be satisfactory. A literature review generally reinforced the

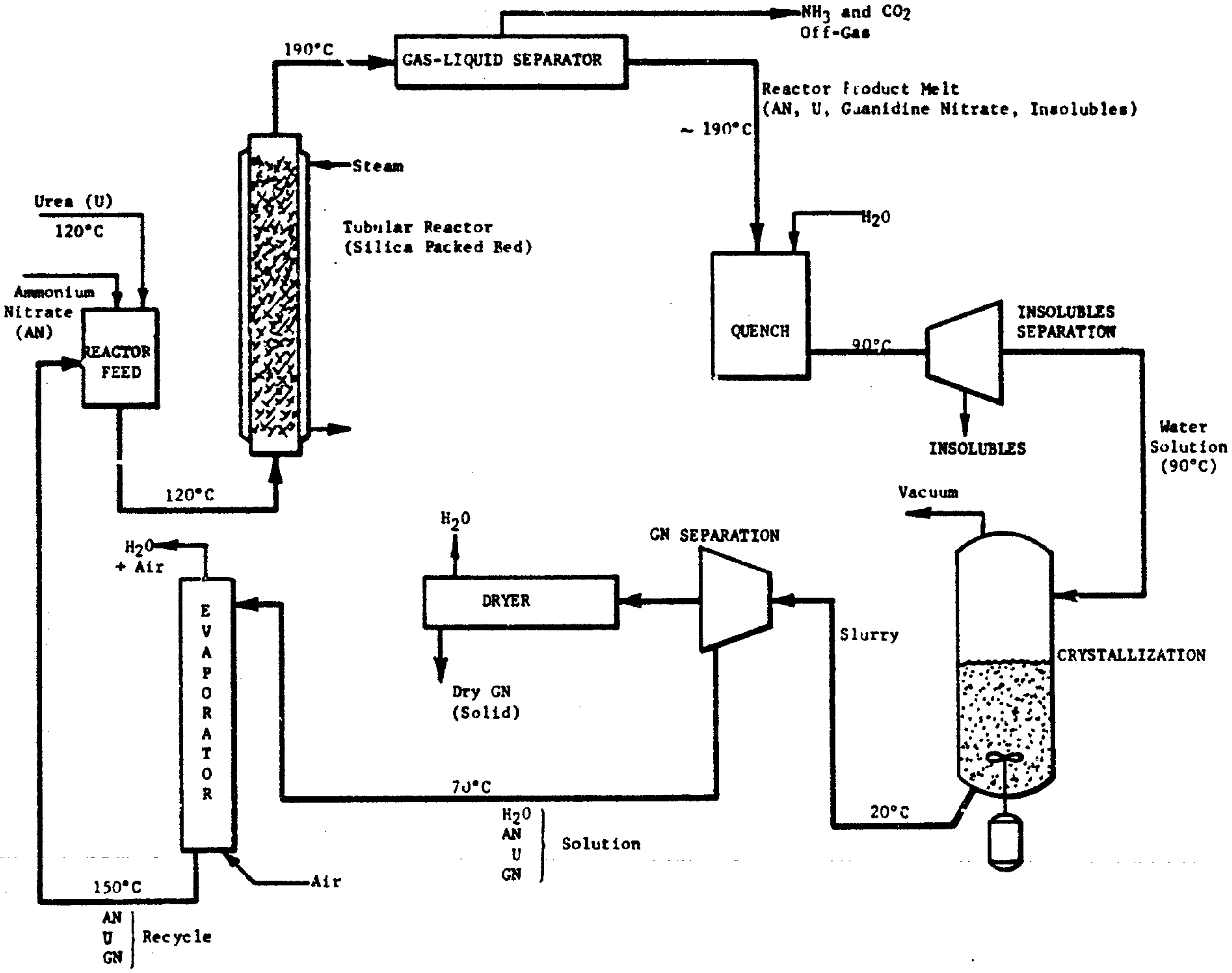


Figure 1-A. U/AN/Silica Process Schematic for Manufacturing Guanidine Nitrate

conclusion that impurities in the AN were the key problem to catalyst deactivation. Evaluation of the three silica gel-type catalyst candidates in a tubular reactor showed that Grace 59 silica gel poisoned most rapidly. Houdry CP-532 silica beads poisoned at a lower rate. Mobil Sorbeads showed the least effect but broke down physically. Testing of Grace 59 silica gel in a 1-inch-diameter reactor employing a reagent grade AN/commercial urea feed mixture containing 1 wt. % water resulted in catalyst poisoning in about 60 hours of operation (15 gm GN/gm catalyst mileage) and decrepitation of the catalyst. Similar conditions employing Houdry CP-532 silica beads did not result in physical breakdown of the beads. A minimum mileage of 65 gm GN/gm catalyst was demonstrated when the experiment was voluntarily terminated after 335 hours of continuous operation. Continued testing of Houdry beads in one of the pilot plant 4-inch-diameter reactors with reagent grade AN, commercial urea and 1 wt. % water showed a minimum mileage of 38 lb GN/lb catalyst in 115 hours of operation. Maximum mileage was not demonstrated. In this experiment without recycle, the GN yield based on urea consumed was 95% (based on 2-mole stoichiometry) and the productivity rate was the same as predicted from the packed-bed, tubular-reactor mathematical model. The above results confirmed that Houdry CP-532 silica bead catalyst was the only known catalyst suitable for packed bed reactors to manufacture guanidine nitrate via the U/AN route, consistent with high conversion and yield.

C. OBJECTIVES OF FOLLOW-ON PROGRAM (FEBRUARY-SEPTEMBER 1973)

Throughout the course of this program, the advantages of the U/AN/silica process for producing guanidine nitrate had been recognized, i.e., simplicity of operations, availability of raw materials, good conversions and yields, safety, good quality product, etc. Results obtained during the latter phases of the original program, following resolution of the catalyst poisoning program, instilled confidence in the future of the U/AN process. A proposal was then submitted to Picatinny Arsenal for continuation of the program. The follow-on program, outlined below, was authorized and completed during the third quarter of 1973. This report presents in detail the results of this program continuation.

Proposed and Contracted Follow-On Program

1. Modify the pilot plant in preparation for a demonstration of Houdry CP-532 silica bead catalyst activity with recycled AN and urea and a guanidine nitrate production run. Proposed modifications and additions were the result of recent investigations and experience gained in operation of the pilot plant. Examples of changes were

complete revisions of melt transfer lines and tank elevations to minimize line plugging, installation of a feed control system on each reactor, supply of high-pressure plant steam to the pilot plant to reduce the load on the on-site electric boilers, improved crystallizer vacuum system, relocation of water insolubles centrifuge, etc.

2. Operate the pilot plant as an integral unit as outlined in the original Phase III of the program to obtain design data, to determine the effects of process variables, and to produce a quantity of guanidine nitrate for conversion to nitroguanidine at Cyanamid of Canada facilities. The quantity of guanidine nitrate produced would be determined by limitations of time, cost and catalyst availability.

3. Place the pilot plant in a layaway condition for either future operations or dismantling at a future date. The preparedness program would consist of the following major items: a) removal of catalyst from reactors, b) inspection of reactors and other equipment for corrosion and scale, c) removal of all in-process materials from the system, d) cleaning all equipment with sodium carbonate solution, e) removal of water from all equipment jackets, lines, etc., f) painting motor and equipment frames, and g) tagging equipment as to condition, status, purchase order numbers, etc.

4. Perform an economic study for manufacturing nitroguanidine via both the urea/ammonium nitrate and British Aqueous Fusion processes. Prior to an overall economic analysis comparison, it was proposed that an analysis be made to determine the most suitable means for utilizing U/AN reactor off-gases (NH_3 and CO_2). The evaluation would include, in addition to costs, the best utilization of NH_3 and CO_2 to make the most desirable products either for in-house use or for sale foreign to the process. After selecting the best off-gas disposal system, an overall economic study would be performed to permit a logical and timely decision between the BAF and U/AN processes as an intermediate in the manufacture of nitroguanidine. The manufacturing costs would be developed in the format reported for the BAF process to NQ under a separate PDCM contract (Corps of Engineers DACA45-71-C-0121). Investment costs for the total U/AN-to-NQ facility would be developed in the format reported for the BAF process in the P-15 estimate for an AMC project (COE DACA45-72-C-0015). Costs would be updated to mid-1973 and projected to mid-1975.

IV. DISCUSSION

A. GUANIDINE NITRATE PILOT PLANT MODIFICATIONS

The guanidine nitrate pilot plant equipment was designed and installed in an existing building (Bldg. 2204) at Hercules/Kenvil. At the time of construction, it was believed that the system could be operated relatively free of mechanical problems. There were, however, areas of uncertainty due to limited space and restrictions imposed on the design in selecting a steam generating system. Operation of the pilot plant as an integral unit for about three months in 1972 defined a number of both required and desired design changes before proceeding with another sustained run. The proposed changes were based on improving operating continuity, minimizing operating downtime, minimizing introduction of water to the reactor feed (via steam sparging), improved reactor feed rate control, safety, etc. Modifications were made in certain areas of the pilot plant from February 15 through May 15, 1973. The major changes and/or improvements are summarized below. The pilot plant flow diagram has been altered to reflect some of the changes and is presented in Figure 1.

1. Urea/Ammonium Nitrate Melt System

The original AN/U melt and transfer system was unsatisfactory in that it was difficult and time-consuming to transfer hot material from the melters to the virgin and recycle feed tanks. Some improvements were made during the 1972 operational campaign by installing a degassing chamber on the suction side of the transfer pump. Subsequently, the degassing chamber was removed and the pump was modified to improve its performance, i.e., speed increased from 1750 rpm to 3500 rpm, impeller diameter decreased from 4-7/8 inch to 4 inch, and installation of a permanent vent line on the pump casing. Suction lines were changed from 1/2 inch to 1 inch diameter and the heat transfer steam tracings were improved. These changes resulted in excellent pump performance.

2. AN/U Melt Lines and Proportioning Pump

Ammonium nitrate/urea (AN/U) melt lines must be maintained hot and with no cold spots. The original pilot plant lines were 0.5-inch diameter and, in several instances, located close to the floor which was sometimes cool and damp. These conditions resulted in numerous instances of plugged lines, causing downtime and introduction of water to the system (via line steaming). Considerable improvement in the operation was achieved through the following changes: 4-inch elevation of all melt and feed tanks, as well as the Hills-McCanna blend pump; installation of one-inch diameter lines from melt tanks, feed tanks and evaporator bottom; and installation of improved steam tracing and line insulation. The Hills-McCanna pump was overhauled, and the stroke proportion control system was modified for manual setting of the recycle to virgin feed ratio. The intermediate virgin feed makeup tank (T-102) for the recycle system (T-104) was eliminated. The above line modifications minimized plugging as a result of solidified AN/U melts.

3. Reactor Feed System

Control of composition and mass rate to the reactors is important from the standpoint of both productivity and process design data. The system, as operated during the 1972 campaign, consisted of a densitometer for controlling the feed composition and one integral differential pressure controller plus valve system for controlling the total feed rate to all reactors. This system was unsatisfactory for both composition control and control of feed to the individual reactors. Consequently, the entire system was modified by eliminating the densitometer (relying upon independent adjustment of the two Hills-McCanna pump heads) and installation of a differential pressure cell, a controller, and a control valve for each of the eight reactors. Associated piping was also replaced.

4. Water Insolubles Recovery

The solid bowl centrifuge (S-300) employed for removing water insolubles from the aqueous quenched reactor product was initially installed at an elevation of about 6 feet for gravity feed of the effluent to the crystallizer feed tank (T-106). This installation proved to be unsatisfactory for bowl removal and cleaning and periodic vibration of the total reactor feed system. The centrifuge was relocated, on a separate stand, to a lower elevation. The effluent was gravity discharged to a collection tank (T-102), equipped with a float level switch, and then pumped to T-106. Operations and safety were improved.

5. Guanidine Nitrate Crystallization

Crystallization of guanidine nitrate (GN) from an aqueous solution had not been a particular problem. However, some downtime had been experienced because of decreased evaporative cooling rate as a result of poor vacuum and plugging of the single crystallizer-feed polishing filter. Modifications consisted of the following items: installation of a third vacuum pump, fabrication and installation of a dry ice trap for collecting entrained condensables, locating and fixing vacuum line leaks and installation of a second polishing filter.

6. Guanidine Nitrate Recovery

The DeLaval basket centrifuge (S-600) for recovering GN crystals from the crystallizer slurry had been virtually trouble-free. To facilitate materials handling, the bottom solids discharge was equipped with a chute to direct wet GN directly to a pre-positioned cloth bag. Previously, wet GN was collected in a tub and then manually transferred to bags. This process improvement resulted in more time for the operator to monitor operations.

7. GN Drying

The Strong-Scott rotary dryer installed originally in the pilot plant for drying GN proved to be unsatisfactory. There was insufficient time to evaluate, design, procure and install a substitute dryer for start-up of the pilot plant in May. Therefore, the dryer and feeder system was removed from the GN process building. Drying was accomplished by spreading partially filled cloth bags of GN (filled directly from the centrifuge) onto drying trays in a forced air dry house.

8. Steam Supply and Alarms

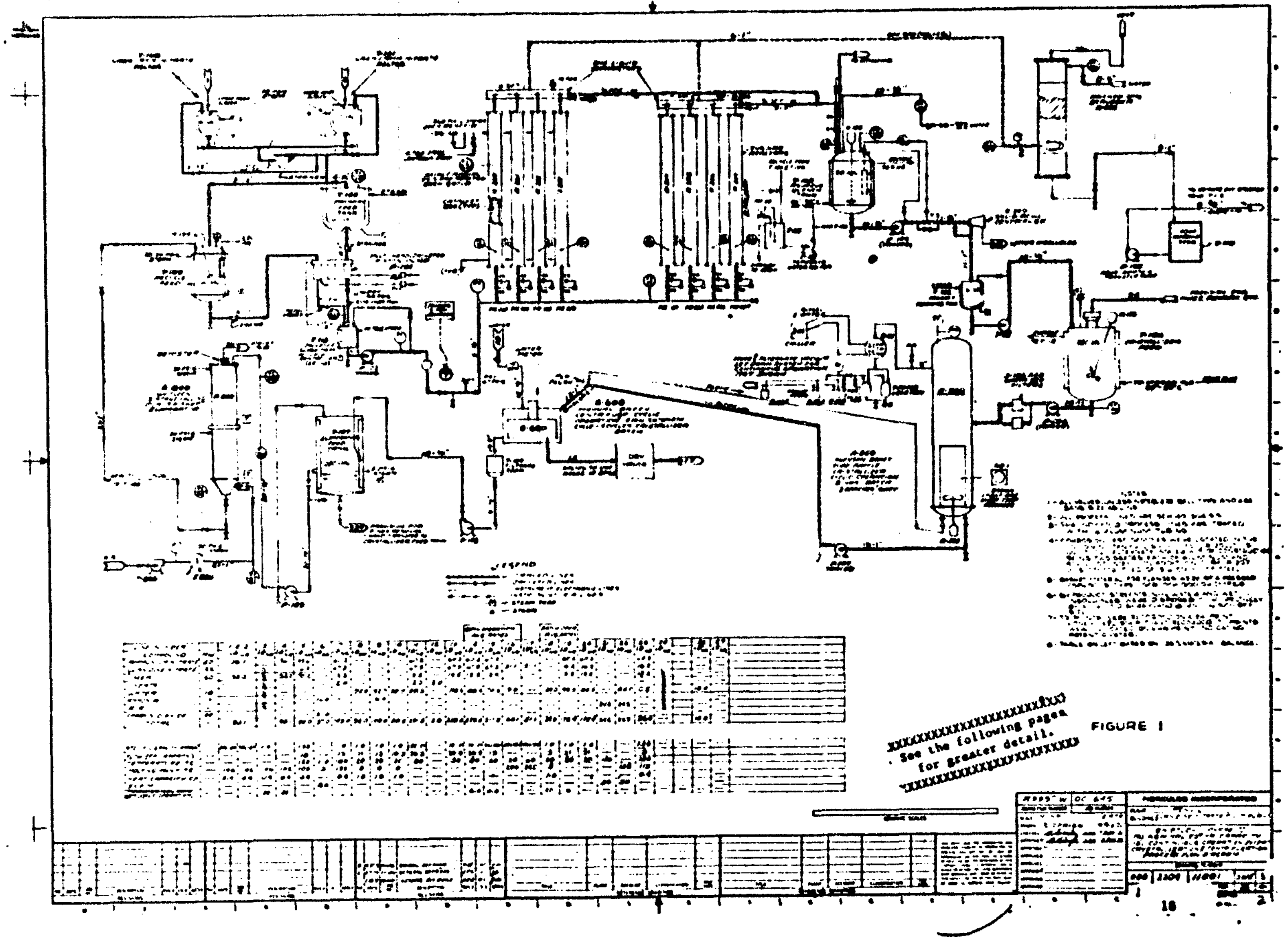
The electric Ebcor boilers would produce sufficient steam to operate the pilot plant; however, they were very susceptible to demand load changes. To minimize plant downtime and upset conditions resulting from the lack of steam, 150-psig steam was brought into the pilot plant from the main plant boilers. This action consisted of installing several steam-reducing stations in the main plant steam line and a complete revamping of the GN pilot plant steam system. In addition to the above, the Ebcor electric boilers were completely refurbished. Electric low pressure alarms were installed in the primary steam supply and air lines to warn operators of potential problems. Subsequent operation of the pilot plant justified these changes.

9. Steam Condensate Utilization

Steam condensate from the pilot plant was used as feed water to the electric boilers. Since catalyst decay problems had been traced to the presence of contaminants (particularly phosphate) in ammonium nitrate, the possibility of catalyst poisoning by introducing minerals via service water for reactor product quenching was questioned. To minimize this effect, a pump system was installed to utilize steam condensate for reactor product quenching and GN centrifuge cake washing.

B. GUANIDINE NITRATE SHIPMENTS AND SAMPLE CONVERSION

A total of 20,067 lb of guanidine nitrate was shipped to Cyanamid of Canada for conversion to nitroguanidine. This included a 10-lb increment for laboratory conversion and analysis. The material was sent as two partial shipments. The first partial shipment, made on August 8, 1973, consisted of 16,352 lb (GBL H-2096152). The second and final partial shipment, made on August 23, 1973, consisted of 3,715 lb (GBL H-2096603). Of this latter amount, 827 lb was material produced during the 1972 operating period.



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. See the following pages
for greater detail.
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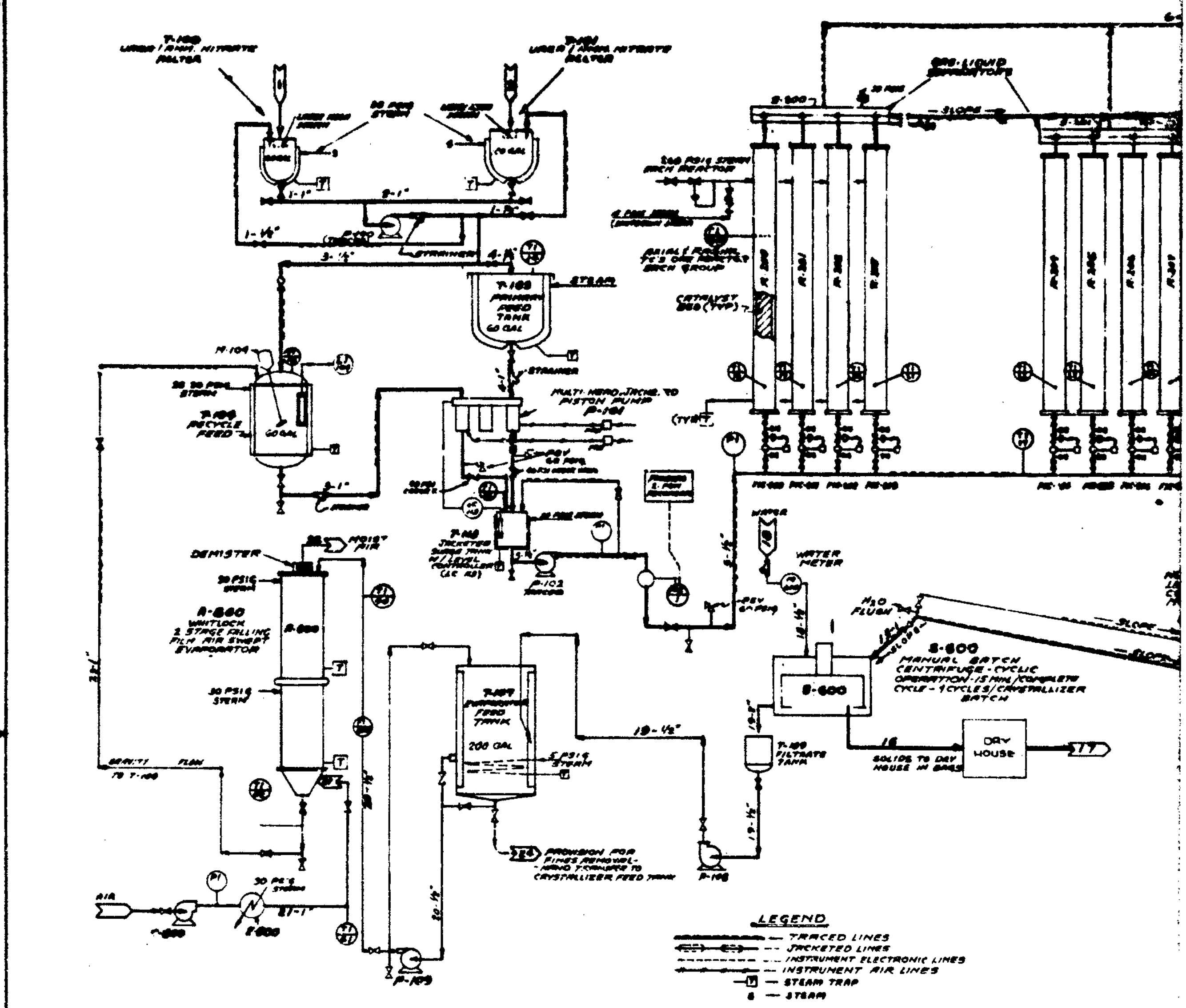
FIGURE 1

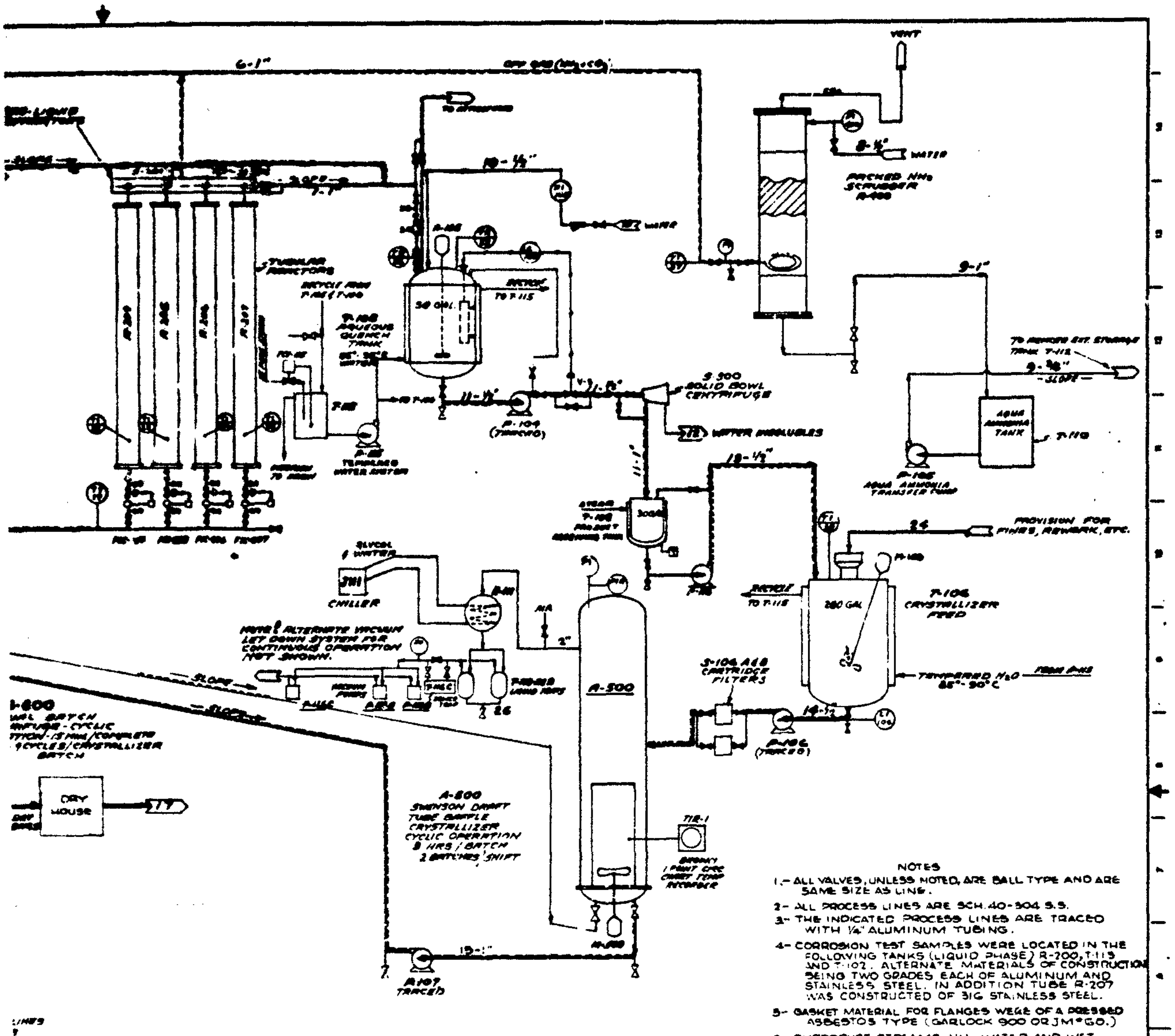


													BATCH OPERATION AVG. RATES				BATCH OPER AVG. RATES									
LINE NUMBER		1 & 2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18	19	20	21	22	23	24		26	27
COMPOUND	MW	LB/HR	LB/HR	LB/HR	LB/HR	LB/HR	LB/HR	LB/HR	LB/HR	LB/HR	LB/HR	LB/HR	LB/HR	LB/HR	LB/HR	LB/HR	LB/HR	LB/HR	LB/HR	LB/HR	LB/HR	LB/HR	LB/HR	LB/HR		LB/HR	LB/HR
AMMONIUM NITRATE	80	36.1	AS REQUIRED	36.1	93.6	—	60.2	—	—	—	60.2	—	60.2	60.2	60.2	—	—	—	60.2	60.2	—	—	60.2			—	—
GUANIDINE NITRATE	122	—		—	16.2	—	67.3	—	—	—	67.3	—	67.3	67.3	67.3	51.1	51.1	—	16.2	16.2	—	—	16.2			—	—
UREA	60	54.0		54.0	70.2	—	13.5	—	—	—	19.6	—	13.5	13.5	13.5	—	—	—	13.5	13.5	—	—	13.5			—	—
INERTS	—	-		—	—	—	2.0	—	—	—	2.0	2.0	—	—	—	—	—	—	—	—	—	—	—			—	—
WATER	18	—		—	—	—	—	92.0	92.0	88.5	88.5	—	88.5	88.5	70.5	9.0	—	34.0	85.5	85.5	—	85.0	0.5			18.0	—
AMMONIA	17	—		—	—	16.0	—	—	16.0	—	—	—	—	—	—	—	—	—	—	—	—	—	—			—	—
AIR	-	—		—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	242	242	—			—	—
CARBON DIOXIDE	44	—		—	—	21.0	—	—		—	—	—	—	—	—	—	—	—	—	—	—	—	—			—	—
TOTAL	-	90.1		90.1	180.0	37.0	143.0	92.0	108.0	88.5	231.5	2.0	229.5	229.5	211.5	60.1	51.1	34.0	175.4	175.4	242	327	90.4	—		18.0	—

SPECIFIC GRAVITY (APPROX)	—	SOLIDS	SOLIDS	1.4	1.1	1.85	—	1.9	1.0	1.0	1.0	[illegible]	—	LIQ/SOL	[illegible]	SLURRY	SOLIDS	SOLIDS	1.0	1.0	1.0	—	—	1.38	—	—	—	—
FLOW GPM (APPROX.)	—	—	—	—	—	18	—	13	11	12	10.5	10.5	—	20.4	20.4	19	—	—	3	21	21	—	—	3				
TEMPERATURE °C	—	—	—	—	—	120	130	180	20	20	20	80	—	80	80	20	20	60	20	20	20	[illegible]	—	130				
MELTING POINT °C	—	106	102	106	102	105	3	135	0	0	0	—	—	—	—	—	270	225	0	—	—	—	138	115				
HEAT CAPACITY CP	—	0.4	0.4	0.4	0.4	0.4	—	0.4	1.0	1.0	1.0	—	—	—	—	—	—	—	1.0	—	—	—	—	0.4				—
SCFM		—	—	—	—	—	8.0	—	—	—	—	—	—	—	—	—	—	—	—	—	—	50	80	—				—
TRANSFER ... (PER CYCLE OPERATION)	—	—	—	10	10	—	—	—	—	—	—	—	—	—	6.0	8.0	—	—	10	6	—	—	—	—	—	—	—	—

NO. DATE DESCRIPTION REVISIONS
TITLE PLANT ACCOUNT CLASSIFICATION REFERENCE DRAWING

U T S R Q P O N M L K



NOTES

1.- ALL VALVES, UNLESS NOTED, ARE BALL TYPE AND ARE SAME SIZE AS LINE.

2- ALL PROCESS LINES ARE SCH. 40-304 S.S.

3- THE INDICATED PROCESS LINES ARE TRACED WITH ¼" ALUMINUM TUBING.

4- CORROSION TEST SAMPLES WERE LOCATED IN THE FOLLOWING TANKS (LIQUID PHASE) R-200, T-113 AND T-102. ALTERNATE MATERIALS OF CONSTRUCTION BEING TWO GRADES EACH OF ALUMINUM AND STAINLESS STEEL. IN ADDITION TUBE R-207 WAS CONSTRUCTED OF 316 STAINLESS STEEL.

5- GASKET MATERIAL FOR FLANGES WERE OF A PRESSED ASBESTOS TYPE (GARLOCK 900 OR JM #60.)

6- BYPRODUCT STREAMS, NH3-WATER AND WET INSOLUBLES WERE DISPOSED OF RESPECTIVELY BY A GROUND SPRAY, AND BURNING, BOTH OFF SITE.

7- TR POINTS WERE RECORDED ON A 24-POINT HONEYWELL STRIP CHART RECORDER. TI POINTS WERE INDICATED ON A 48 POINT INDICATING POTENTIOMETER.

8- TABLE ON LEFT BASED ON 100% MATERIAL BALANCE.

21	22	23	24		26	27
[illegible]	lb/hr	lb/hr	lb/hr		lb/hr	lb/hr
—	—	60.2			—	—
—	—	[illegible]			—	—
—	—	13.5			—	—
—	—	—			—	—
—	85.0	0.5			10.0	—
—	—	—			—	—
42	242	—			—	—
—	—	—			—	—
42	327	964	—		10.0	—

—	—	138	—	—	—	—
—	—	3				
[illegible]	—	35				
—	138	115				
—	—	0.4				—
58	80	—				—
—	—	—	—	—	—	—

FIGURE 1

GRAPHIC SCALES

REFERENCE DRAWINGS — TITLE — PLANT — ACCOUNT — CLASSIFICATION — FILE CODE

A9997 W | 001645
MICRO FILM NUMBER | JOB NUMBER

SCALE NONE — DATE
DRAWN R. CRAVEN 18 Aug. 71
CHECKED J. A. Doyle 7 Sept. 71
APPROVED J. A. Doyle 2 Sept. 72
APPROVED
APPROVED
APPROVED
APPROVED
APPROVED
APPROVED

HERCULES INCORPORATED
PLANT KENVIL
BUILDING GUANIDINE NITRATE PILOT PLANT

GN STUDY PHASE II
(a) NOMINAL 50#/HR CAPACITY
(b) CONTINUOUS OPERATION EXCEPT CRYSTALLIZATION & CENTRIFUGATION
PROCESS FLOW DIAGRAM

DRAWING NUMBER
080 | 2204 | 11001 | SWE | 3
T.SH | FILE CODE | REV

2

B

An additional 878 lb of off-grade guanidine nitrate was forwarded to Picatinny Arsenal on August 28, 1973 (GBL H-2096025). This material was produced during the 1972 period and was considered off-grade because either the insolubles or the ammonium nitrate content was too high. If the level of insolubles was greater than 1.25% or the level of ammonium nitrate was greater than 4.0%, the material was rejected for shipment to Cyanamid of Canada. It should be noted that none of the 1973 material had to be rejected for these reasons.

The average laboratory analysis of the material shipped to Cyanamid of Canada was as follows:

Guanidine nitrate	95.9%
Ammonium nitrate	2.4%
Urea	0.4%
Insolubles	0.5%
Water	0.8%
	100.0%

On the basis of the analyses performed on the many batches of guanidine nitrate produced and envisioned production plant operations, the GN product produced in a production plant should at least meet the following analyses. Higher GN contents are preferred.

Guanidine nitrate	>94.0%
Ammonium nitrate	<4.0%
Urea	<1.0%
Insolubles	<1.25%
Water	<2.0%

An effective way of determining the approximate composition of a sample of guanidine nitrate is to determine its melting point. The melting point of 100% guanidine nitrate reported in the literature is 214°C. If a significant amount of ammonium nitrate is present, the measured melting point will decrease. A plot of melting point versus ammonium nitrate in GN is shown in Figure 2. There was scatter in the data, probably due to the presence of insolubles and urea and, in some instances, not completely dry material. However, if a melting point were ≥208°C, it was assumed, pending complete analytical results, that the recovery end of the process was in control.

A lot of GN was defined as the amount of material which filled a dry-house bay. After the bay was filled, the GN was dried and later removed as a lot unit. This amounted to between 2065 and 2888 lb as shown in Table 1.

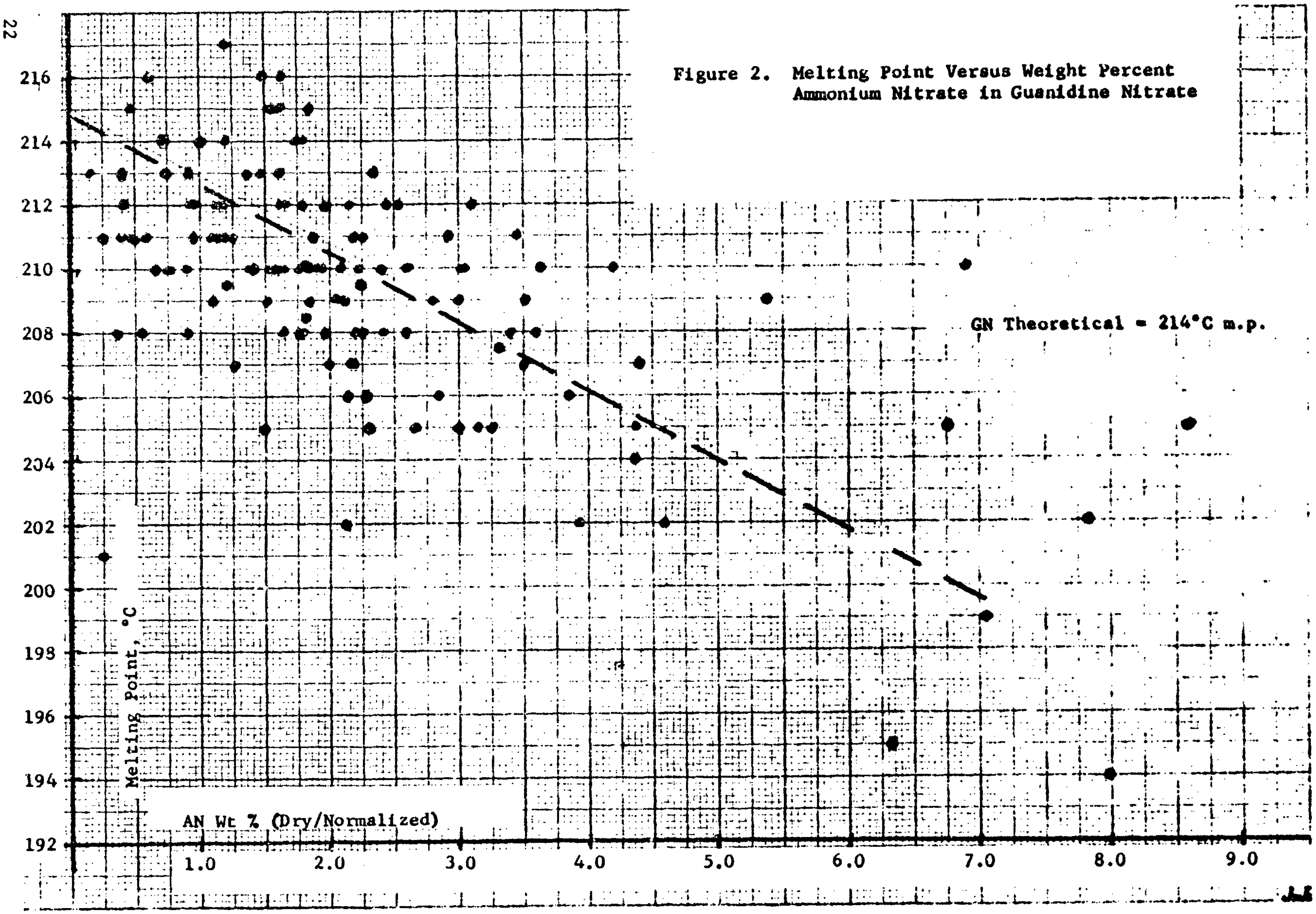


Figure 2. Melting Point Versus Weight Percent Ammonium Nitrate in Guanidine Nitrate

GN Theoretical = 214°C m.p.

TABLE 1

GUANIDINE NITRATE SHIPMENT LOTS - SUMMARY

LOT NO.	NET WEIGHT (lbs)	AN %	U %	GN %	INSOL %	H_2O %	MP°C	COMMENTS
1	2,065	0.75	0.10	98.65	0.0	0.5	214	
2	2,142	2.03	0.78	95.31	0.57	1.31	209	
3	2,434	2.74	0.63	96.09	0.29	0.25	207	
4	2,489	2.95	0.0	94.80	0.37	1.88	210	
5	2,686	2.18	0.33	96.49	0.35	0.65	213	
6	2,120	3.67	0.92	94.36	0.66	0.39	210	
7	2,406	2.06	0.0	96.63	0.43	0.87	209	
8	2,888	3.06	0.42	94.95	0.90	0.67	-	
1972	827	1.30	0.07	97.93	0.73	-	-	Results on dry basis
Off Grade Picatinny	878	-	-	-	-	-	-	
Total	20,067*	-	-	-	-	-	-	Exclusive of Off-Grade Lot Shipped to Picatinny

* Includes 10 pounds shipped for laboratory work.

A lot represented about 18 to 20 crystallizer batches of about 120 lb each. The batch weights varied considerably during the operating period depending upon the operating conditions; however, during normal steady operating conditions the batches ran about 160 lb with an occasional one reaching 210 lb.

The initial 16,352-lb shipment consisted of Lots 1 through 7 and the 10-lb laboratory increment. The final or 3715-lb shipment consisted of Lot 8 (2888 lb) from the 1973 campaign and the 827 lb manufactured in 1972.

Tables 2 through 11 give pertinent information regarding the various lots. This information includes laboratory analysis and identifies the batches represented in each drum.

The 10-lb sample of guanidine nitrate from crystallizer batch number 222 (1973) was satisfactorily converted to nitroguanidine at Cyanamid of Canada under Hercules Purchase Order No. 980-12727-08. The product was essentially identical to a control sample utilizing Cyanamid production GN. A Cyanamid technical report, dated September 11, 1973, is presented in Table 12. The test procedure, analytical results and conclusions are included.

The ten tons of guanidine nitrate shipped to the Cyanamid Welland plant was converted to nitroguanidine in their production facilities under a separate Picatinny Arsenal contract. Losses were encountered, resulting in a net yield of about 9400 lb of NQ. The Picatinny Arsenal Project Officer noted that the product met all military specifications.

TABLE 2

GN FROM 1973 CAMPAIGN SHIPPED TO CYANAMID

LOT 1

DRUM No.	NET WEIGHT		CONTRIBUTING BATCHES	DRUM NO.	NET WEIGHT		CONTRIBUTING BATCHES
	lb	oz			lb	oz	
1	52	3	126, 127	21	52	13	115, 115
2	47	10	127, 127	22	39	3	114, 114
3	48	13	128, 129	23	55	11	114, 114
4	49	0	126, 126	24	55	8	114, 113
5	46	3	128, 128	25	54	8	113, 113
6	66	10	126, 125, 125	26	54	10	111, 113
7	54	14	125, 125	27	51	0	111, 111
8	53	2	124, 124	28	57	3	112, 112
9	56	0	124, 123	29	48	10	112, 112
10	56	10	123, 122, 122	30	65	0	110, 110, 107
11	43	13	121, 121	31	53	3	107, 107
12	56	3	121, 121	32	53	6	106, 106
13	62	6	119, 119	33	56	0	106, 106
14	58	6	118, 118, 119	34	52	13	105, 105
15	54	13	119, 118	35	53	13	105, 105
16	54	10	118, 118	36	44	0	104, 104
17	51	11	117, 117	37	57	3	104, 104
18	48	10	116, 116	38	54	10	101, 101
19	55	0	117, 116	39	43	6	101, 101
20	54	10	115, 115				

1. Net weight of Lot 1 = 2,065 lb
2. Analysis:

	Analytical Closure	Analyses Normalized to 100%				
		U	AN	GN	Insol	H_2O
a. Composite sample	99.3%	0.10	0.75	98.65	-	0.50
b. Calculated from individual batch analysis	-	0.29	1.56	97.48	0.17	0.50

TABLE 3

GN FROM 1973 CAMPAIGN SHIPPED TO CYANAMID

LOT 2

DRUM NO.	NET WEIGHT		CONTRIBUTING BATCHES	DRUM NO.	NET WEIGHT		CONTRIBUTING BATCHES
	lb	oz			lb	oz	
1	61	10	153, 153	22	41	13	153, 151
2	57	10	153, 151	23	53	10	147, 141
3	52	10	151, 151	24	46	6	147, 149, 148
4	54	14	153, 151	25	47	10	141, 141
5	57	0	154, 154	26	51	3	138, 137
6	56	14	154, 154	27	57	3	148, 138, 138
7	56	0	154, 147	28	55	3	139, 139
8	51	13	150, 150	29	55	8	144, 144, 144
9	51	10	147, 147	30	46	3	137, 137
10	51	2	150, 150	31	44	6	144
11	50	6	147, 149	32	49	11	142, 142
12	53	13	146, 146, 150	33	58	6	143, 144
13	49	0	148, 145	34	55	0	142, 142
14	51	14	149, 149	35	53	5	141, 143
15	53	8	146, 145	36	55	13	143, 143
16	51	13	149, 149	37	51	5	140, 136
17	53	0	146, 145	38	58	13	143, 136,140
18	45	0	145, 148	39	54	13	136, 135
19	53	8	145, 145	40	56	3	134, 135, 140
20	49	5	148, 148	41	45	0	133, 139, 132, 130
21	51	10	146, 146				

1. Net weight of Lot 2 = 2,142 lb
2. Analysis:

	Analytical Closure	Analyses Normalized to 100%				
		U	AN	GN	Insol	H_2O
a. Composite sample	101.8%	0.78	2.03	95.31	0.57	1.31
b. Calculated from individual batch analysis	-	1.23	4.01	92.93	0.54	1.31

TABLE 4

GN FROM 1973 CAMPAIGN SHIPPED TO CYANAMID

LOT 3

DRUM NO.	NET WEIGHT lb	NET WEIGHT oz	CONTRIBUTING BATCHES	DRUM NO.	NET WEIGHT lb	NET WEIGHT oz	CONTRIBUTING BATCHES
1	52	10	169, 170	23	53	5	161, 161
2	54	10	170, 168	24	55	8	161, 161
3	52	5	170, 169, 170	25	59	0	157, 157
4	50	14	169, 170	26	55	0	157, 157
5	52	3	169, 169	27	59	8	162, 162, 157
6	52	14	168, 169	28	54	3	162, 162
7	52	14	166, 166	29	63	3	158, 158
8	51	3	166, 166	30	51	0	158, 158
9	54	0	163, 163	31	51	10	158, 155, 156
10	54	3	163, 163	32	55	0	156, 156
11	56	6	163, 152, 152	33	55	6	156, 156
12	79	10	152, 152, 152	34	54	10	156, 159
13	54	5	162, 162	35	54	14	159, 159
14	43	4	170, 168	36	49	10	159, 159
15	52	10	170, 169	37	57	3	165, 165
16	55	10	167, 167	38	54	0	165, 165
17	62	14	167, 167	39	55	5	165, 164
18	57	0	167, 167	40	55	10	164, 164
19	58	14	166, 166, 166	41	61	3	164, 164
20	55	5	155, 155	42	60	10	160, 160
21	58	2	155, 155	43	53	3	163, 160
22	54	3	155, 161	44	58	3	160, 160

1. Net weight of Lot 3 = 2,434 lb
2. Analysis:

	Analytical Closure	Analyses Normalized to 100% U	AN	GN	Insol	H_2O
a. Composite sample	100.6%	0.63	2.74	96.09	0.29	0.25
b. Calculated from individual batch analysis	-	0.40	2.43	96.60	0.32	0.25

TABLE 5

GN FROM 1973 CAMPAIGN SHIPPED TO CYANAMID

LOT 4

DRUM	NET WEIGHT			DRUM	NET WEIGHT		
NO.	lb	oz	CONTRIBUTING BATCHES	NO.	lb	oz	CONTRIBUTING BATCHES
1	50	6	187, 187	25	53	11	177, 177
2	53	0	185, 186	26	54	3	174, 172
3	48	5	186, 185	27	51	6	172, 172
4	51	13	182, 182	28	55	6	174, 174
5	51	8	179, 179	29	50	13	174, 174
6	48	14	172, 172	30	52	8	178, 178
7	74	14	179, 179	31	51	6	174, 174
8	52	10	176, 177	32	51	6	179, 179
9	44	6	177, 177	33	53	8	180, 180
10	54	6	176, 176	34	52	6	181, 181
11	52	13	176, 168	35	51	6	182, 182
12	54	5	168, 168	36	49	5	185, 187
13	43	3	178, 177	37	32	14	186, 186
14	49	0	172, 172	38	42	2	174, 174
15	55	13	178, 178	39	52	6	184, 186
16	51	0	182, 181	40	52	10	175, 175
17	50	6	174, 174	41	42	13	181, 181
18	51	13	186, 184	42	51	3	184, 184
19	46	14	185, 185	43	53	11	180, 180
20	52	14	187, 187	44	41	11	184, 184
21	55	10	176, 176	45	51	6	175, 180
22	55	13	171, 168	46	42	0	183, 183
23	55	8	171, 171	47	53	2	183, 183
24	56	11	171, 171	48	44	3	175, 175
				49	51	3	184, 184

1. Net weight of Lot 4 = 2,489 lb
2. Analysis:

	Analytical	Analysis Normalized to 100%				
	Closure	U	AN	GN	Insol	H_2O
a. Composite sample	105.06%	0.00	2.95	94.80	0.37	1.88
b. Calculated from individual batch analysis	-	0.23	2.45	95.07	0.37	1.88

TABLE 6

GN FROM 1973 CAMPAIGN SHIPPED TO CYANAMID

LOT 5

DRUM NO.	NET WEIGHT lb	NET WEIGHT oz	CONTRIBUTING BATCHES	DRUM NO.	NET WEIGHT lb	NET WEIGHT oz	CONTRIBUTING BATCHES
1	47	8	199, 192, 200	24	77	3	190, 192, 192
2	52	3	198, 198	25	82	3	199, 201, 199
3	54	0	198, 198	26	81	0	190, 190, 189
4	52	10	196, 198	27	46	3	207, 207
5	46	3	197, 197	28	53	8	205, 205
6	66	11	203, 203, 203	29	45	6	207, 206
7	49	5	196, 204	30	53	10	200, 192
8	53	10	203, 203	31	52	8	200, 200
9	53	10	204, 204	32	57	5	205, 201
10	63	2	196, 201	33	51	13	207, 207
11	54	6	204, 204	34	52	11	195, 200
12	55	6	195, 195	35	67	13	206, 206, 206
13	54	0	203, 203	36	53	10	199, 192
14	61	6	192, 194, 194	37	56	6	192, 201
15	56	2	201, 192	38	57	6	206, 205
16	54	11	191, 191	39	55	11	188, 188
17	55	11	196, 197	40	53	3	199, 200
18	79	3	191, 191, 191	41	46	3	189, 188
19	53	10	195, 195	42	78	0	189, 189, 189
20	81	5	193, 193, 193	43	71	5	193, 193, 193
21	53	3	194, 195	44	46	3	190, 190
22	78	5	192, 192, 192	45	55	3	188, 188
23	68	6	194, 194, 194	46	55	0	196, 197

1. Net weight of Lot 5 = 2,686 lb
2. Analysis:

	Analytical Closure	Analysis Normalized to 100% U	AN	GN	Insol	H_2O
a. Composite sample	100.84%	0.33	2.18	96.49	0.35	0.65
b. Calculated from individual batch analysis	-	0.14	2.42	97.54	0.54	0.65

TABLE 7

GN FROM 1973 CAMPAIGN SHIPPED TO CYANAMID

LOT 6

DRUM NO.	NET WEIGHT		CONTRIBUTING BATCHES
	lb	oz	
1	53	0	210, 210
2	51	10	210, 210
3	47	2	217, 225
4	52	3	217, 209
5	52	14	215, 215
6	41	10	227, 221
7	51	11	212, 212, 213
8	52	10	213, 212
9	51	3	208, 209
10	51	0	209, 209
11	51	6	213, 210
12	50	2	208, 208
13	52	10	211, 212
14	48	0	227, 226
15	52	8	215, 214
16	55	3	215, 216
17	55	8	216, 216
18	52	3	218, 219
19	51	3	219, 219
20	51	13	219, 219
21	52	13	226, 226
22	47	0	211, 211
23	46	13	208, 208
24	54	13	218, 218
25	54	8	216, 217
26	57	0	223, 223
27	52	5	220, 220
28	54	0	226, 226
29	52	5	220, 220
30	47	2	225, 226
31	53	13	221, 225
32	52	5	218, 217
33	59	6	220, 227
34	60	0	215, 215, 216
35	47	5	219, 218, 215
36	55	2	214, 223
37	51	6	214, 223, 214
38	44	3	223, 227
39	52	8	217, 216
40	53	11	227, 217
41	53	8	225, 225

1. Net weight of Lot 6 = 2,120 lb
2. Analysis:

	Analytical Closure	Analysis Normalized to 100%				
		U	AN	GN	Insol.	H_2O
a. Composite sample	100.89%	0.92	3.67	94.36	0.66	0.39
b. Calculated from individual batch analysis	-	0.57	2.67	95.74	0.62	0.39

TABLE 8

GN FROM 1973 CAMPAIGN SHIPPED TO CYANAMID

LOT 7

DRUM NO.	NET WEIGHT lb	NET WEIGHT oz	CONTRIBUTING BATCHES	DRUM NO.	NET WEIGHT lb	NET WEIGHT oz	CONTRIBUTING BATCHES
1	54	6	244, 245	24	60	2	239, 239
2	54	8	244, 243	25	53	3	221, 221
3	55	0	243, 245	26	53	10	239, 239
4	53	6	224, 224	27	43	0	238, 238
5	43	8	222, 222	28	56	11	231, 230. 230
6	39	0	224, 247	29	56	13	240, 234
7	55	10	222, 224	30	58	3	234, 234
8	52	3	245, 244, 237	31	52	13	247, 241
9	51	5	240, 241	32	55	2	244, 245
10	49	14	229, 229	33	41	5	242, 247
11	54	3	233, 233	34	57	14	240, 241
12	57	13	233, 233	35	56	3	240, 241
13	55	0	245, 239	36	45	8	244, 243
14	53	10	238, 240	37	51	6	224, 222
15	50	13	236, 236	38	49	8	246, 246
16	57	10	238, 234, 240	39	53	10	242, 243
17	52	10	238, 238	40	53	13	246, 243
18	42	6	236, 236	41	55	0	246, 242
19	41	3	228, 228	42	56	6	222, 241
20	53	5	221, 221	43	52	10	232, 232
21	54	14	228, 239	44	53	10	228, 231
22	52	11	231, 231	45	54	3	235, 235
23	56	3	229, 241, 241	46	54	13	232, 232

1. Net weight of Lot 7 ≈ 2,406 lb

2. Analysis:

	Analytical Closure	Analysis Normalized to 100% U	AN	GN	Insol	H_2O
a. Composite sample	-	0.0	2.06	96.63	0.43	0.87
b. Calculated from individual batch analysis	-	0.39	2.77	95.68	0.29	0.87

TABLE 9

GN FROM 1973 CAMPAIGN SHIPPED TO CYANAMID

LOT 8

DRUM NO.	NET WEIGHT lb	CONTRIBUTING BATCHES	DRUM NO.	NET WEIGHT lb	CONTRIBUTING BATCHES
1	54.6	272, 273	31	55.6	280, 280
2	51.0	271, 272	32	53.3	279, 280
3	40.9	270, 271	33	56.2	263, 263
4	45.3	270, 271	34	53.2	279, 279
5	53.6	270, 271	35	57.7	263, 264
6	51.7	269, 271	36	54.3	253, 254
7	63.1	268, 269	37	55.2	261, 262
8	54.0	268, 269	38	53.1	254, 255
9	39.1	268, 266	39	33.3	260, 262
10	55.1	267, 266	40	53.3	261, 261
11	42.3	267, 267	41	54.8	257, 258
12	54.8	264, 266	42	38.9	257, 258
13	36.5	264, 264	43	52.5	249, 255
14	54.0	264, 276	44	54.5	248, 253
15	56.0	276, 276	45	49.9	253, 248
16	47.7	277, 277	46	53.3	252, 252
17	53.3	275, 277	47	55.6	248, 248
18	56.1	274, 274	48	41.5	254, 248
19	54.1	275, 275	49	52.1	249, 249
20	54.7	274, 274	50	32.0	253, 255
21	40.1	271, 273	51	32.9	248, 252
22	36.6	272, 275	52	39.3	252, 256
23	46.2	271, 273	53	42.5	250, 256
24	53.2	272, 271	54	53.1	250, 250
25	48.1	267, 273	55	45.7	251, 251
26	65.7	278	56	51.0	251, 250
27	35.0	266, 271	57	27.3	251
28	35.9	278, 278	58	45.8	237, 237
29	55.5	264, 264	59	43.0	237, 232
30	41.8	279, 280	60	21.6	235

1. New weight of Lot 8 = 2,888 lb

2. Analysis:

	Analytical Closure	Analysis Normalized to 100%				
		U	AN	GN	Insol	H_2O
a. Composite sample	101.12%	0.42	3.06	94.95	0.90	0.67
b. Calculated from individual batch analysis	-	0.19	2.18	96.96	0.67	0.67

TABLE 10

GN MANUFACTURED IN 1972 AND SHIPPED TO CYANAMID

	Normalized Wet Analysis (Wt. %)						Normalized Dry Basis (Wt. %)					
Drum No.	AN	U	GN	Insol.	H_2O	Closure	AN	U	GN	Insol.	MP °C	Wt. (lb)
1	1.17	0.0	90.0	0.38	8.3	96.51	1.28	0.0	98.26	0.41	211	24.3
2	0.57	0.0	89.08	0.89	9.47	100.36	0.63	0.0	98.40	0.98	211	25.9
3	0.14	0.0	94.33	1.01	4.52	95.48	0.15	0.0	98.8	1.06	220	47.3
4	2.94	0.0	95.32	0.85	0.88	99.12	2.97	0.0	96.16	0.86	230	36.1
5	0.65	0.0	83.97	0.31	15.07	84.93	0.77	0.0	98.87	0.37	210	39.5
6	1.20	0.0	92.00	0.65	6.16	93.84	1.28	0.0	98.04	0.69	198	38.7
7	1.06	0.0	97.09	1.19	0.66	99.34	1.07	0.0	97.74	0.66	219	33.5
8	1.08	0.0	97.15	1.19	0.58	99.42	1.09	0.0	97.72	1.20	219	37.4
9	0.53	0.0	98.84	0.36	0.27	99.73	0.53	0.0	99.11	0.36	214	19.4
10	0.62	0.0	98.02	0.53	0.82	99.18	0.63	0.0	98.83	0.53	210	50.0
11	1.62	0.44	95.77	1.34	0.84	99.16	1.63	0.44	96.58	0.85	211	21.3
12	1.90	0.52	95.27	1.23	1.07	98.93	1.92	0.53	96.30	1.24	216	64.1
13	2.46	0.0	92.9	-	4.64	-	2.58	0.0	97.2	-	208	19.5
14	1.97	0.0	97.8	-	0.26	-	1.98	0.0	98.1	-	213	55.0
15	1.19	0.33	93.8	0.47	4.21	104.15	1.24	0.34	97.8	0.49	215	51.2
16	1.89	0.0	97.38	0.54	0.19	100.53	1.89	0.0	97.99	0.54	213	63.4
17	0.74	0.0	97.8	0.99	0.48	100.31	0.74	0.0	98.3	0.99	216	71.8
18	1.47	0.0	97.8	0.59	0.15	101.75	1.47	0.0	98.0	0.59	216	58.7
19	0.85	0.0	98.4	0.63	0.14	98.6	0.85	0.0	98.5	0.63	210	72.7

Total Net Weight - 827 lb

TABLE 11

OFF-GRADE GN SHIPPED TO PICATINNY ARSENAL

	Normalized Wet Analysis (Wt. %)						Normalized Dry Basis (Wt. %)						
Drum No.	AN	U	GN	Insol.	H_2O	Closure	AN	U	GN	Insol.	MP °C	1973 Batch	Wt. (lb)
1	1.97	0.0	85.1	4.56	8.36	97.29	2.14	0.0	92.86	4.98	220	NA ↓	30.7
2	3.04	0.0	85.9	2.61	8.41	97.74	3.32	0.0	93.79	2.85	200		29.9
3	3.10	0.0	81.5	2.44	13.00	95.39	3.56	0.0	93.69	2.80	-		38.3
4	5.66	1.52	76.46	3.21	14.67	94.04	6.63	1.78	89.61	3.76	190		43.6
5	20.92	6.97	68.64	2.96	7.47	86.97	22.60	7.53	74.18	3.20	217		38.1
6	0.61	0.0	84.37	4.60	10.42	89.58	0.68	0.0	94.18	5.14	212		48.7
7	0.33	0.0	88.70	2.90	8.07	96.62	0.36	0.0	96.49	3.15	214		57.6
8	2.39	0.0	95.60	1.78	0.77	95.9	2.41	0.0	96.36	1.79	215		48.8
9	6.38	1.57	74.87	3.88	14.87	96.17	7.49	1.84	87.95	4.56	230		51.7
10	2.64	0.26	88.74	2.72	5.90	95.79	2.81	0.28	94.30	2.90	235		55.0
11	0.79	0.0	72.06	6.77	20.38	96.17	0.99	0.0	90.5	8.50	220		41.8
12	1.59	0.0	79.79	3.97	14.66	104.4	1.89	0.0	94.61	4.71	208		33.7
13	6.11	2.14	85.40	1.66	6.83	101.17	6.56	2.30	91.66	1.78	210		61.3
14	1.58	0.0	90.52	2.41	5.49	98.21	1.67	0.0	95.78	2.55	211		32.1
15	1.61	0.0	83.64	1.66	12.55	95.65	1.84	0.0	95.64	1.90	218		36.7
16	0.39	0.0	94.88	1.82	2.92	103.29	0.38	0.0	97.73	1.87	220		25.9
17	1.19	0.0	85.68	1.76	11.37	93.25	1.34	0.0	96.67	1.99	220		30.5
18	11.9	0.2	82.6	0.11	5.2	99.1	12.55	0.2	87.1	0.12	-	102	58.4
19	8.8	0.2	87.4	0.18	3.5	98.2	9.12	0.2	90.6	0.19	-	103	57.6
20	1.0	TR.	67.0	13.6	18.4	105.7	1.20	0.0	82.1	16.7	210	120	57.1

Total Net Weight - 878 lb

TABLE 12

LABORATORY CONVERSION OF AN/U GUANIDINE NITRATE AT CYANAMID OF CANADA

399 M 02:72 | September 11, 1973

CONVERSION OF HERCULES (U/AN) GUANIDINE NITRATE TO NITROGUANIDINE—LABORATORY SCALE TESTS		MANAGER - TECHNICAL DEPARTMENT TECHNICAL FILE
REQUESTED BY J. Doyle, Hercules Inc., Kenvil, N.J.	FOR	DATE September 11, 1973
REPORTED BY [illegible] C. Blodgett	WORK DONE BY O. C. Biggar	
NOTE BOOK REFERENCE 578		PROJECT REQUEST NO. Hercules P.O. 980 12727 08

INTRODUCTION:

Hercules Inc. requested laboratory scale conversion tests of a sample of their guanidine nitrate to nitro guanidine and a similar test using CCL guanidine nitrate for comparison purposes.

SUMMARY:

1. The laboratory work was completed using both CCL and Hercules Inc. guanidine nitrate.
2. The chemical analysis of each product was satisfactory and met specification Type II, Class I, MIL-N-494A AMdt 3, EO 45490-5, 7 Dec. 1966. Total volatiles were slightly off specification for both tests due to inadequate drying in the laboratory oven but this is not significant.
3. Particle size specifications were not met. This was expected prior to the test program because of the different crystallization method used in the laboratory.

CONCLUSION:

The laboratory work confirms that nitroguanidine can be made from Hercules (U/AN) guanidine nitrate and meet the required chemical specifications.

DESCRIPTION OF LABORATORY PROCEDURES

Six hundred and thirty-one (631) grams of guanidine nitrate was added to a stainless steel beaker containing fourteen hundred and thirty two (1,432) grams of concentrated sulfuric acid. Temperature of the acid was maintained at 38-42°C during the addition by adjusting temperature of the surrounding water bath. Continuous agitation was provided and addition time was approximately one-quarter hour. A 10 gram sample of the syrup was removed to confirm acid strength was in the range of 65 ± 1%.

After a half hour, the syrup was diluted with water to approximately 18% H_2SO_4 to precipitate the nitroguanidine. Temperature was maintained at approximately 40°C during the dilution. The slurry was then cooled to $<10°C$ prior to separation of the solids on a laboratory basket centrifuge. The cake was washed with water to reduce the sulfate content to about 0.25%.

TABLE 12 (CONTINUED)

Conversion of Hercules (U/AN) Guanidine
Nitrate to Nitroguanidine - Lab Scale Tests (Continued) Page 2

Description of Lab Procedures (Continued)

The wet crude cake from the centrifuge was slurried in water and heated by an immersion steam coil to the boiling point to dissolve the nitroguanidine solids. The pH of the hot solution was measured and sufficient 10% soda ash solution added to neutralize the acidity of the crude cake. The hot solution was then poured down an inclined trough (jacketed with cold brine at approximately 0°C) to provide rapid crystallization of the nitroguanidine solution. The material on the trough was transferred to a centrifuge, washed with water and prepared for drying.

Drying was done in a forced air Brabender type oven in two stages, 25 minutes at 65°C, and 20 to 30 minutes at 110°C. The latter time was varied to achieve essentially constant weight at this temperature. The dried product was then ready for chemical analysis.

The above procedures were followed for both the CCL and Hercules (U/AN) guanidine nitrate samples in order to compare reaction characteristics of the two materials. The only difference noted was in the nitration stage where same additional gas evolution was noted with the Hercules U/AN material. However, the amount was very small.

S. C. Blodgett

SCB:ch
Attach.

TABLE 12 (CONTINUED)

Conversion of Hercules (U/AN) Guanidine
Nitrate to Nitroguanidine - Lab Scale Tests (Continued)

TABLE I

CONVERSION OF GUANIDINE NITRATE TO NITROGUANIDINE
SUMMARY OF DATA

	CCL Guanidine Nitrate	Hercules (U/AN) Guanidine Nitrate	Specifications
Guanidine Nitrate (gm.)	631	631	
% Purity	90.5	95.4	
Sulfuric Acid (92.8%), gm.	1432	1451	
Nitrator Syrup, % H_2SO_4	64.0	64.7	
Dilution Slurry, % H_2SO_4	17.6	18.4	
Redissolving Solution	Water	Water	
Mother Liquor Analysis			
pH	6.3	3.3	
Melamine, %	0.20	0.015	
Na_2SO_4, %	0.16	0.07	
Yield, gm.	392	413	
Product Quality:			
Purity, %	99.6	99.6	99.0 (min.)
Ash, %	0.01	0.01	0.30 (max.)
pH	7.0	6.2	4.5 (min.), 7.0 (max.)
Volatiles, %	0.28	0.31	0.25 (max.)
Sulfates, %	< 0.01	< 0.01	0.20 (max.)
Water Insolubles, %	0.03	0.03	0.20 (max.)
Acidity	< 0.01	0.01	0.06 (max.)
Particle Diameter (microns)	8.7	8.8	3.4 - 6.0

TABLE 12 (CONTINUED)

Conversion of Hercules (U/AN) Guanidine
Nitrate to Nitroguanidine - Lab Scale Tests (Continued)

TABLE II

GUANIDINE NITRATE ANALYSES

	GUANIDINE NITRATE			
	CCL	Hercules (U/AN)		
	Analysis by CCL	Analysis by CCL		Analysis by Hercules
% Purity (G.N.)	90.5	95.4		95.6
% AN	7.7	3.0		2.3
			% Urea	0.3
% Melamine	1.2	0.2	% Insol.	0.6
% Total Volatiles	1.5	1.1	% H_2O	1.4
% Ash	0.02	0.004		
Temperature rise (°C)	25	24		
Chlorides (ppm)	9	4		
Iron (ppm)	20	10		

C. OPERATIONS

1. Raw Materials and Catalyst

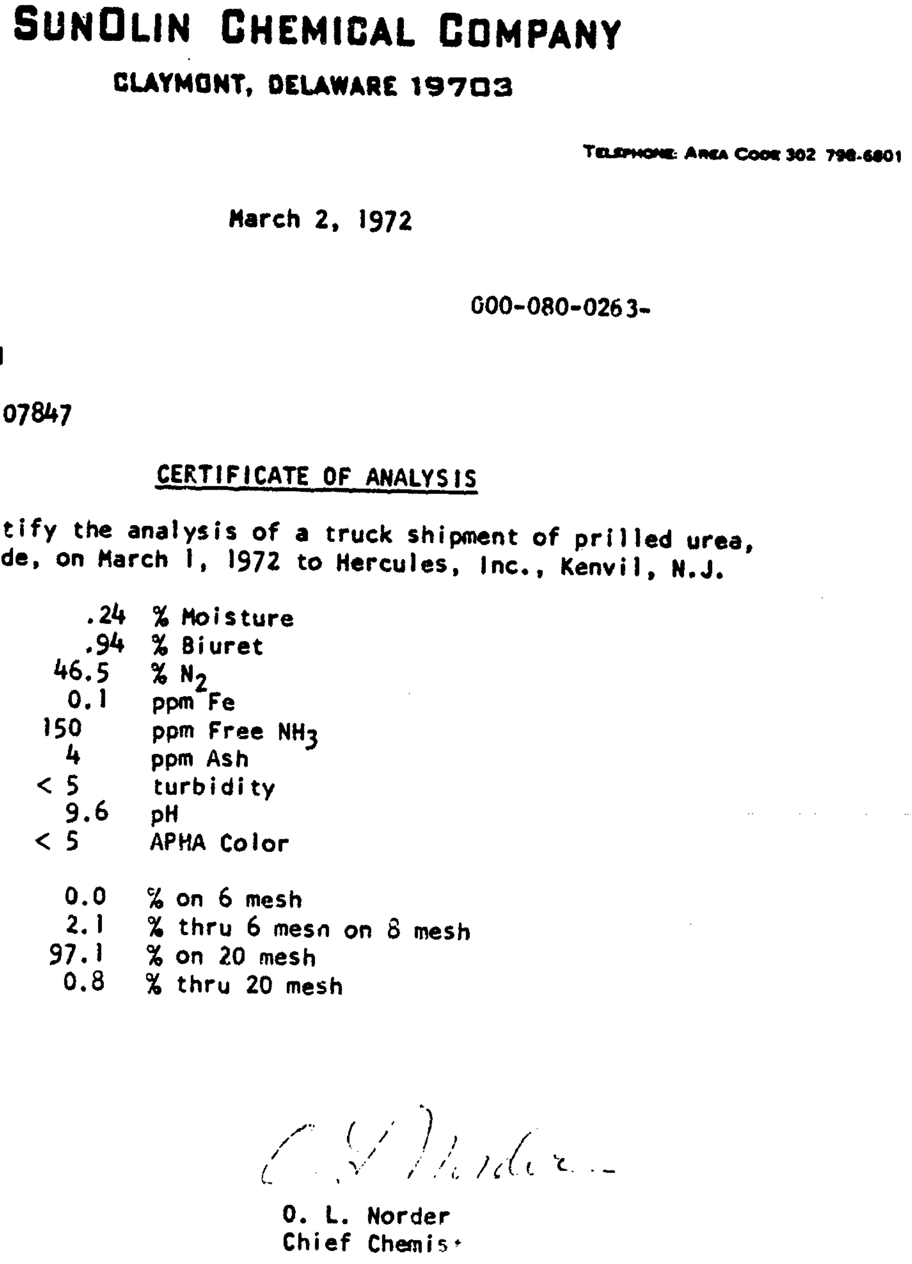
Ammonium nitrate (AN) and the urea (U) are the two material ingredients used in this process to manufacture guanidine nitrate (GN). For the 1972 pilot plant campaign, a large quantity of urea was purchased from the Olin Corporation and subsequent laboratory testing revealed that this material was not the cause of catalyst poisoning. Approximately 70,000 lb of urea remained from the previous operation and was utilized for the 1973 campaign. The certificate of analysis for this material is shown in Table 14. Laboratory and bench-scale experiments, reported in detail in Final Report - Volume I, confirmed that the crystal habit modifier (particularly diammonium phosphate), present in the Hercules MCW ammonium nitrate poisoned silica-gel type catalysts. It was concluded that reagent grade AN would be required to assure success of GN pilot plant operations.

J. T. Baker Chemicals Co., Phillipsburg, New Jersey, was contacted for a large supply of reagent grade AN. They sell the material in maximum container sizes of 25 lb and at a high price. The AN that Baker Chemicals markets is purchased in drum quantities from the Hercules Donoro, Pa. plant, and subsequently repackaged. Their experience in handling AN prills without either crystal habit modifiers or coating agents had been good. Therefore, arrangements were made to procure 53,000 lb of reagent grade AN from the Hercules Donora plant. The prills were loaded in drums directly from the conveyor system used to deliver prills from the screener to the coating blender. The AN was delivered to Kenvil in 100-, 300- and 400-lb drums, stored in a heated building, and then manually transferred to polyethylene-lined Kraft paper bags (50 lb/bag). The material was bagged in the middle of May; by August 10, 1973, there were some large lumps but they could be broken easily. The analysis of ammonium nitrate used for the 1973 pilot plant campaign is shown in Table 15.

It had been stated in Volume I of this Final Report that the only known suitable catalyst for packed bed reactors was Houdry CP-532 macroporous silica beads manufactured by Air Products and Chemicals, Inc. This conclusion was based primarily on the good activity of this product and its resistance to decrepitation. Approximately 275 lb of Houdry beads were on hand from previous operations; this amount was sufficient to load about eleven reactors; i.e., one complete pilot plant charge plus material for three additional reactor changes. Inquiries were made regarding the purchase of an additional quantity of Houdry beads. It was discovered that the catalyst manufacturing pilot plant had been dismantled and there was no available stock of catalyst. Air Products and Chemicals, Inc., personnel estimated that a modified pilot plant installation would cost approximately $50,000. They proposed to the government a set-up charge to help offset the cost of a

TABLE 14

ANALYTICAL RESULTS FOR INDUSTRIAL GRADE PRILLED UREA



SUNOLIN CHEMICAL COMPANY
CLAYMONT, DELAWARE 19703

TELEPHONE: AREA CODE 302 798-6801

March 2, 1972

000-080-0263-

Mr. Fred Fremd
Hercules, Inc.
Kenvil, N.J. 07847

CERTIFICATE OF ANALYSIS

This is to certify the analysis of a truck shipment of prilled urea, industrial grade, on March 1, 1972 to Hercules, Inc., Kenvil, N.J.

.24	% Moisture
.94	% Biuret
46.5	% N_2
0.1	ppm Fe
150	ppm Free NH_3
4	ppm Ash
< 5	turbidity
9.6	pH
< 5	APHA Color
0.0	% on 6 mesh
2.1	% thru 6 mesn on 8 mesh
97.1	% on 20 mesh
0.8	% thru 20 mesh

O. L. Norder
Chief Chemist

TABLE 15

ANALYSIS OF REAGENT GRADE AMMONIUM NITRATE PURCHASED FOR 1973 GN PILOT PLANT OPERATIONS

HERCULES INCORPORATED
DONORA WORKS
DONORA, PENNSYLVANIA

CHEMICAL ANAYLSIS - PRILLED AMMONIUM NITRATE

Characteristics	Specifications	
	Maximum	Found
Insoluble Matter	0.005%	0.003
Residue after Ignition	0.010%	0.002
pH of a 5% Solution	4.5-6.0 at 25°C	5.00
Chloride (Cl)	0.0005%	0.0001
Nitrite (NO_2)	0.0005%	N.D.
Phosphate (PO_4)	0.0005%	<0.0001
Sulfate (SO_4)	0.002%	0.0013
Heavy Metals (as Pb)	0.0005%	N.D.
Iron (Fe)	0.0002%	<0.0001
Moisture	-	-
Ammonium Nitrate	-	-

AMMONIUM NITRATE (NH_4NO_3) FORMULA WT. 80.04

REAGENT CHEMICALS SPECIFICATIONS

Comments:

/S/ John Fanala Date: 4/26/73

pilot plant and a guarantee to supply 1000 lb of Houdry CP-532 beads within 4-1/2 months of a negotiated contract. A description of this arrangement and the projected catalyst costs are presented in Table 16. Picatinny Arsenal subsequently issued a contract to Air Products and Chemicals, Inc., on May 4, 1973, with full understanding that the Kenvil pilot plant program, barring major problems, would be complete before the receipt of new catalyst. This action, however, assured the government of small-scale facilities for manufacturing Houdry beads through 1974. Perhaps of more importance, the contract contains an intent to negotiate construction of a captive catalyst manufacturing facility in the event that Air Products and Chemicals, Inc., does not wish to pursue manufacture of this particular catalyst in the future. The details of this option are not presented in this report. Catalyst manufacture was completed during October 1973.

The Houdry beads on hand were tray dried in a 130°F forced air oven for 72 hours, screened and then charged to the eight pilot plant tubular reactors. Steam was admitted to the jackets of the first bank of four reactors (R-200, 201, 202 and 203) initially and then to the second bank (R-204, 205, 206, and 207) when they were brought on stream. The reactors were not permitted to cool down until the mid-July total Kenvil plant shutdown.

2. Chronology of Operations

Pilot plant operations were started on May 21, 1973, on a three-shift, seven-day per week basis. Each shift was staffed with three operators and a shift supervisor. Two technicians were assigned to performing routine chemical analysis for process control. Two engineers were responsible for data correlation, maintenance, troubleshooting, directing pilot plant operations, etc. Consulting services were obtained periodically from the Corporate Engineering Department.

The GN pilot plant was operated continuously from May 21 until August 10, 1973, except for a planned 2-week shutdown and limited unscheduled downtimes due to mechanical problems. There was a 5-1/2 day period when all eight reactors were on stream and the plant was operating on a total recycle basis. During this period, operations were very smooth with the plant operating in complete balance, discounting material losses. As an overall assessment, the pilot plant functioned much better than during the 1972 period of performance. Resolution of the catalyst poisoning problem, increased personnel staffing, and better understanding of the process chemistry and unit operations enabled the process engineers to control the system. A chronology of plant operations is presented in Figure 3. Operations are summarized in the following paragraphs:

TABLE 16

PRODUCTION AND COSTS OF HOUDRY CP-532 BEADS

Air Products and Chemicals INC.
CHEMICALS GROUP
Five Executive Mall, Swedesford Road, Wayne, Pa. 19087

HOUDRY DIVISION

W. J. Cross, Jr., General Manager
R. G. Craig, Mkt. Mgr.
Tel: (215) 687-6150
Twx: 510-668-2034

December 21, 1972

Commanding Officer
Picatinny Arsenal
Dover, New Jersey 07801

Attention: SMUPA-MT-C
Mr. S. Wachtell

Gentlemen:

This confirms the telephone conversation that you, Mr. Nichols, and myself had on Wednesday, December 20, with regard to our supplying macroporous silica beads in the coming months.

As a result of our recent meeting on December 8, we in Air Products have reviewed the probable investment and manufacturing costs to produce the product in quantities up to 250,000 pounds per year on the assumption that you would be the sole customer. At the same time, you will recall that our former price schedules were based on projections of higher quantities to be produced.

As you are also aware, it is necessary for us to reinstall our pilot plant equipment as well as make some substantial improvements to it at a significant cost to ourselves. If this is done, however, it appears that we might have enough capacity to handle your potential requirements. Of course, a lot depends on the catalyst life when in use. Accordingly, we made the following proposal to yourselves:

1. For the immediate need of an additional 1,000 pounds of catalyst for pilot plant work, we propose a charge of $10,000 for set-up costs plus $2.25 per pound selling price, f.o.b. Paulsboro, NJ. We indicated that should your

TABLE 16 (CONT.)

process become commercial and you undertake to buy commercial quantities from us at a later date, we would work out a refunding arrangement for the $10,000 set-up charge in the form of a credit against the catalyst purchased.

Insofar as timing is concerned, it will take 90 days to acquire the needed equipment that we propose to add to the pilot unit, and we feel it reasonable to allow another 30 days beyond this for completion of installation. The actual production of the 1,000 pounds, once we are operating, should take only a very short time, perhaps no more then a week. Your Mr. Caggiano asked in one telephone conversation what the timing would be on 200 pounds. Actually, what we would do in such a case would be to take the first 200 pounds completed from the 1,000 pounds; thus, if you wanted 200 pounds completed from the 1,000 pounds ahead of the balance, we would gain a few days but not a great amount of time.

2. Looking ahead to a situation in which you will be purchasing commercial quantities of catalyst and again on the assumption that you would prove to be the only customer that we would have for the material, we estimate the following prices for the product:

Pounds Purchased Per Year	Dollars Per Pound
25,000	5.00
50,000	4.00
250,000	2.50

For intermediate levels of production, you can estimate prices by drawing a curve through the above three points. I am sure you appreciate that these figures are estimates at this time and not firm quotations. Also the situation could change if we are successful in developing additional markets for the beads. Should our annual sales exceed 250,000, then the price for quantities in the 25,000 pound range would obviously be lower.

TABLE 16 (CONT.)

Picatinny Arsenal
Page 3
December 21, 1972

Regarding the question of our assuring you of a supply of the material, I indicated upon receipt of your order for a 1,000 pounds under the terms of this proposal, we would initiate installation of the pilot plant equipment. Furthermore, we agree to maintain the equipment in operable condition till the end of 1974. This date will provide you ample time to make a decision on your commercial facility and indicate to us whether we will have to provide additional production capacity beyond the initial pilot plant stage.

I further indicated that we have discussed this proposal together with our potential financial commitments with our Profit Center's General Manager, who has given his agreement to this plan of action. At the same time, I am sure you are aware that for substantial expenditures for new equipment we always have to seek formal approval from our Board of Directors. Since we have provided in the above estimated costs to make this what we believe a viable project, we foresee no problem in this regard.

I hope that this letter summarizes all of the information that bears on your situation and which will permit you to make an early decision from your end. Certainly, we are most interested in working with you, and we want to cooperate with you in every way possible. If there are more questions, please do get in touch with us.

Yours very truly,

G. W. Higginson
Manager, Catalyst Sales

GWH:mef

cc: Mr. C. Nichols, Picatinny Arsenal
Mr. Norman Steel, Hercules, Kenvil, NJ

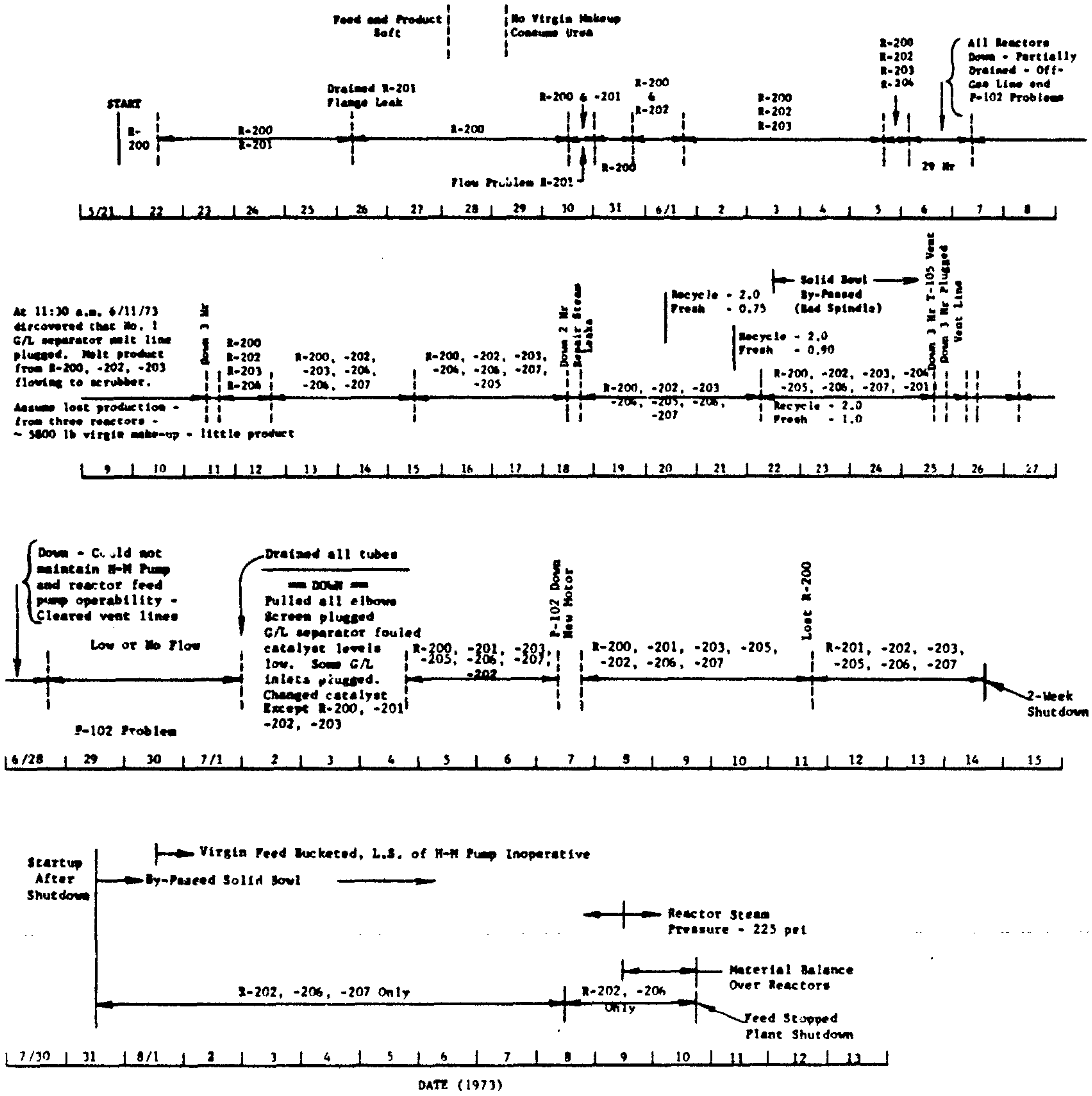


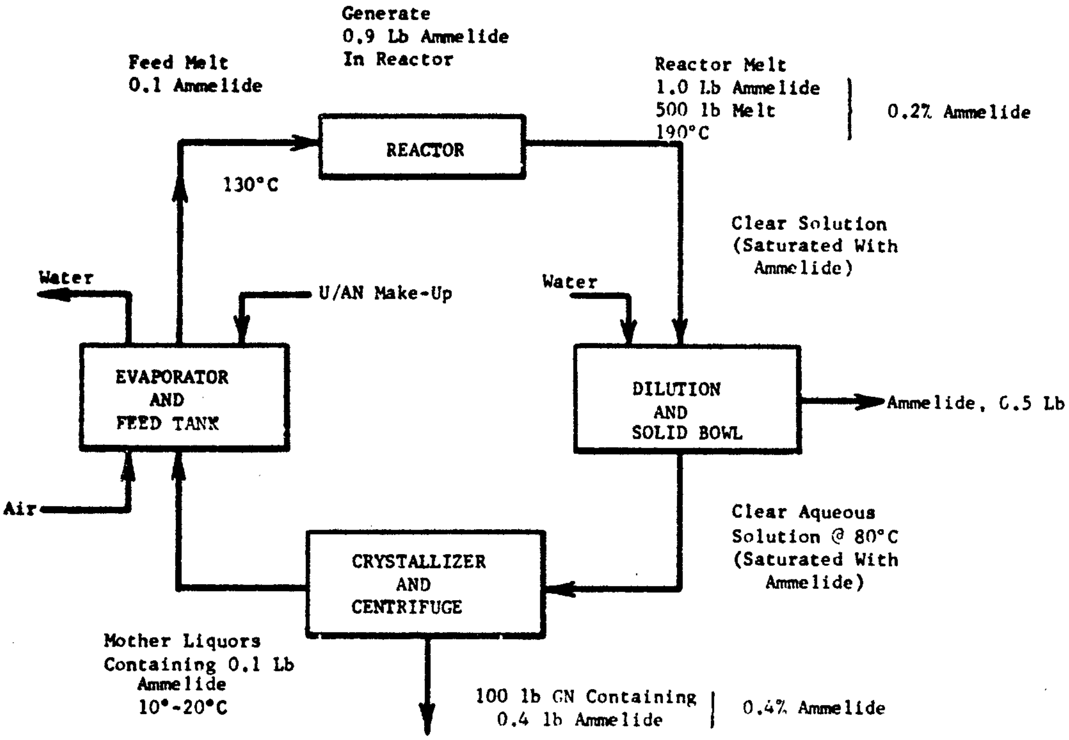
Figure 3. Chronology of Plant Operations

Melt was introduced to one reactor (R-200) on May 21 at a nominal reaction temperature of 190°C. Within one day, a second reactor (R-201) was initiated but it was shut down within 3-1/2 days because of a leak in the top flange. Operation of R-200 was continued. R-201 was drained, and the gasket was replaced. Repeated attempts to resume flow of feed to R-201 were unsuccessful until June 22. Recycle of AN, U and GN to the reactor feed system was begun three days after startup. Other reactors were brought on stream sequentially, and by June 5 (16 elapsed days), four reactors were functioning. The first pilot plant shutdown (29 hours) occurred on June 6 and was caused by a plugged reactor off-gas line and reactor feed pump problems. Four days later, with four reactors functioning (three in one bank and one in the other), it was noted that the recovered GN production rate had decreased (inconsistent with the calculated productivity) and that the make-up of virgin AN and U had increased. Since there had not been any problems with off-gas line plugging, it was theorized initially that recycling AN and U had introduced an unexpected process variable. A check of the off-gas scrubber water revealed a density of about 1.2 gm/cc, considerably higher than the $<$ 1.0 gm/cc value for ammonia water. Analysis revealed the presence of all three reactor product components in the NH_3-H_2O stream. The missing GN mystery was solved; 75% of the total reactor product melt stream was being diverted to the NH_3-H_2O storage tank. Only three hours were required to clear the reactor product melt line and improve the steam tracing. Operations were resumed on June 11, with four reactors, and by June 22, eight reactors were operating. During this time, minor problems were encountered; however, it was a period of smooth operation. Minor problems consisted of steam leaks, plugged vent lines, varying AN/U feed ratio, evaporator bottleneck, etc.

After 5-1/2 weeks of essentially continuous operation, operating problems started to mount; e.g., malfunctioning Hill-McCanna proportioning pump, occasional low discharge pressure on the reactor feed pump, and decreasing feed rates to all reactors. After three days of troubleshooting, the plant was shut down and the reactors were drained. Low pressure steam was maintained on the reactor jackets. The top discharge elbows on the eight reactors were removed, revealing heavy deposits of water insolubles (ammelide) plus reactor melt in the elbows and gas liquid separators. The catalyst retention screens were essentially plugged. The catalyst level in R-200, on stream for about 37 days, had decreased about 10 inches (ca. 7.5% of total depth). Catalyst fines were noted in the gas-liquid separator and in the aqueous quench tank (T-105), confirming the attrition of Houdry beads. R-200 was subsequently topped with 2.5 lb of fresh catalyst. While the plant was down, the remaining available catalyst was utilized to recharge reactors R-201, R-202 and R-203. The original catalyst in these reactors drained freely from the individual tubes.

The solid bowl centrifuge (S-300) used for separating insolubles from the reactor quench stream had been inoperable for nine days before the forced shutdown as a result of a bad bowl spindle. This resulted in a gradual build-up of insolubles in the reactor feed stream (via the recycle system) which finally surpassed its solubility level in both the feed and reactor product streams. Consequently, ammelide deposited in the tops of the reactors, gas-liquid separators, evaporator tubes, all melt process lines, Hill-McCanna pump checks and the reactor feed pump. Hot sodium carbonate solution flushing of equipment (exclusive of reactors) and lines removed most of the insolubles. The magnitude of this problem can be appreciated by noting the increase in the reactor product melt insolubles level; i.e., from a normal level of less than 1 wt. % to 2.5 wt. %, based on GN. Insolubles in the evaporator bottoms stream went from nil to 0.4 wt. %. This incident and its results stress the importance of removing insolubles from the system before crystallization of GN. It has been demonstrated that the GN cake will remove only a portion of the ammelide in the system, particularly at high levels. Therefore, it is imperative that insolubles be removed separately from the system and/or maintained at a low level of production by altering processing conditions, e.g., low urea feed concentration or low reactor temperature. It is now believed that the principal cause for reactor bed plugging was due to the presence of melt and water insolubles. There are perhaps other contributing factors such as catalyst attrition.

Figure 3A shows quantities of ammelide in the total U/AN/system and ammelide removed from the system under stable operating conditions. The values shown are approximations based on typical analysis. In Volume I of this Final Report, it was reported that the solubility of ammelide in reactor melt was > 0.2 wt.% but < 0.8 wt.%. Figure 3A shows that about 0.9 lb ammelide is formed per 500 lb of melt (0.2 wt.%). Below saturation levels, the reactor melt is clear. Following dilution with water (80°C), about 0.5 lb of ammelide is removed from the solution via the solid bowl centrifuge, leaving a clear crystallizer feed solution. Further cooling and some concentration in the crystallizer result in removal of another 0.4 lb ammelide with the guanidine nitrate cake. As a result, 0.1 lb of ammelide from the original one pound leaving the reactors is recycled to the reactors with the recycle and make-up feed stream. If the solid bowl centrifuge were by-passed, some additional ammelide would be removed with the GN cake, but the major portion of ammelide normally collected in the centrifuge would be recycled to the reactors. If one assumes that the ammelide content of the GN product remains constant, then after about four system turn-arounds, the reactor melt ammelide concentration would be about 0.6%, presumably the upper solubility limit. Continued recycle without any ammelide purge would exceed the solubility and, consequently, start deposition of ammelide in the catalyst bed.



AMMELIDE BALANCE:

Ammelide Generated in Reactor	0.9 lb	Ammelide Removed in Solid Bowl	0.5 lb
	___	Ammelide Removed With GN	0.4 lb
Ammelide Generated	0.9 lb	Ammelide Removed	0.9 lb

NOTE: Assuming reactor melt to be saturated at outlet conditions - then by-passing solid bowl will probably result in higher ammelide content in mother liquors. This would result in solids deposition in the reactor catalyst bed due to exceeding the solubility limit of ammelide in the reactor melt.

Figure 3-A. Example of Ammelide Balance Under Stable Operations

Following the 3-day shutdown to clear up the insolubles problem, feed was introduced to the reactors and within 3 days (July 7), seven reactors were operating. Feed to R-204 could not be sustained. On July 11, flow of feed to R-200 stopped, and on July 14 feed to the remaining reactors was stopped voluntarily for a scheduled 2-week shutdown. The reactors were flushed twice with reactor feed melt with 100 psig steam pressure in the jackets (below reaction temperature), drained, and then permitted to cool down.

Following the 2-week shutdown, all reactors were heated but the feed rate could be sustained to only three (R-202, 206, and 207) of the previously functional six reactors. Feed to R-207 was erratic, and after nine days (August 8) flow stopped. Operation of the remaining two reactors continued until August 10, 1973, when pilot plant operations were voluntarily stopped. During the final stages of pilot plant operations, considerable effort was expended in running material balances and determining the source of yield losses. These results are discussed in a separate section of the report.

The remaining in-process inventory was worked through the system to recover the available guanidine nitrate. Residual mother liquors and unused melts were discarded. Catalyst was removed, with the aid of a high-pressure water jet, from all reactors. The complete layaway of the pilot plant is described in a later section.

The operating time for each reactor is shown graphically in Figure 4. Total on-stream reactor tube time was 6,460 hours. Assuming a conservative 4 lb GN/hr/tube productivity (Figure 5), the total calculated GN production was 25,800 lb. The total accounted for or recovered GN was 19,300 lb (100% GN, dry basis), leaving an unaccounted for quantity of 6500 lb. An attempt was made to account for the missing material based on analytical results, observations, measured rates and assumptions. The results are shown in Table 17. These types of losses are to be expected in a pilot plant and would be minimal in a production plant. About one-third of the estimated losses resulted from the GN centrifuge operation during the charging portion of the cycle. It was purposely elected not to return this material to the crystallizer feed tank on a routine basis. This material was returned to the tank during the 1972 operations, and the procedure resulted in both crystallizer feed filter plugging and centrifuge cloth blinding. One solution would have been to adjust the quench water rate to dissolve these "slops," but this would have upset the total process and was not justified. A production plant, and perhaps a modified pilot plant, would have GN repulping provisions as well as a rework system for line purges, spills, etc.

The pilot plant was operated as an integrated system with recycle for a total time of about 62 days. "In-the-barrel" production totaled about 20,000 lb of dry product with a nominal 95 wt.% guanidine nitrate content. A small

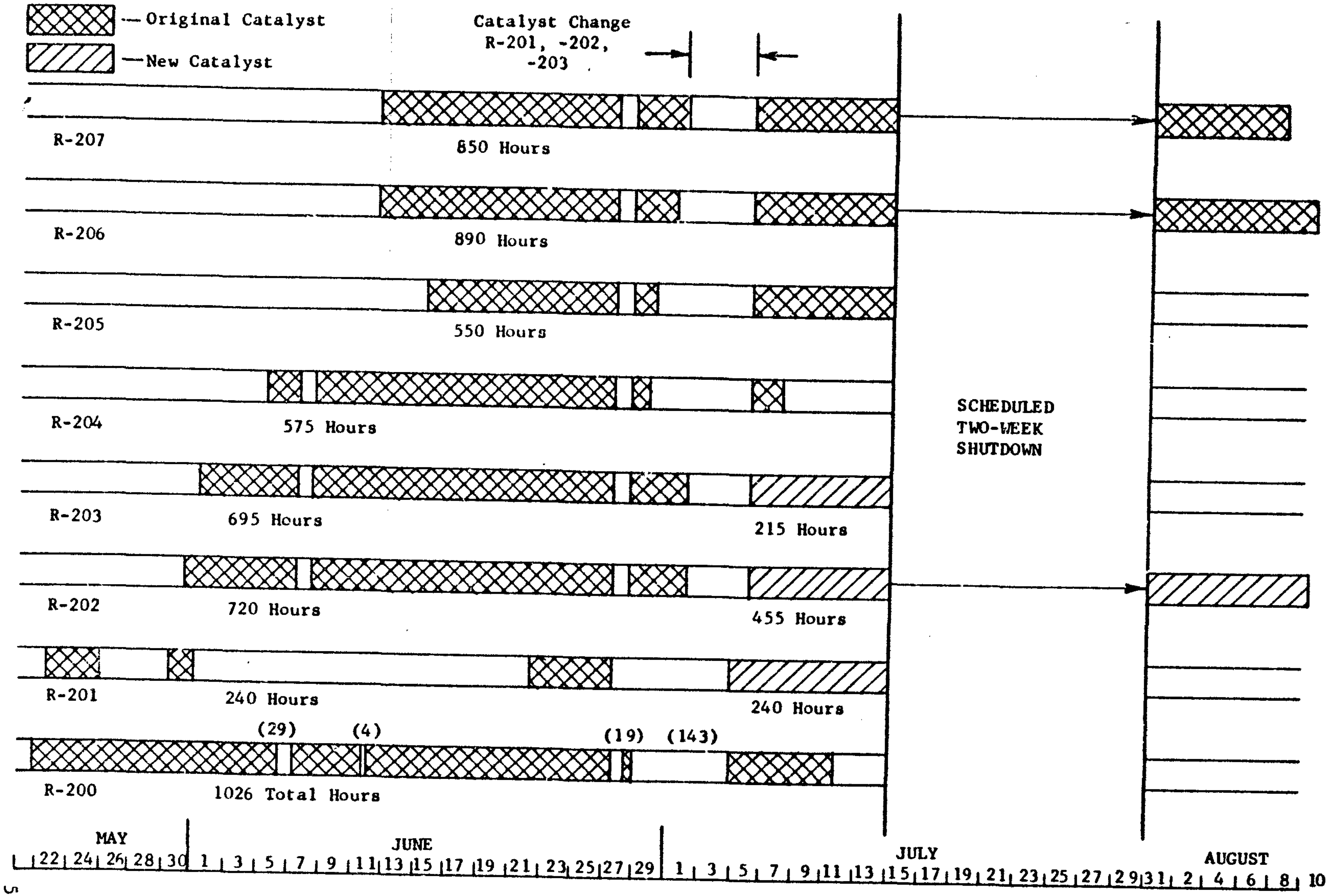


Figure 4. History of Reactor Operating Times

TABLE 17

GUANIDINE NITRATE ACCOUNTABILITY TABULATION

Basis: a) 100% guanidine nitrate, dry basis

b) Total reactor-tube hours (see Figure 4) - 6,460 hours

c) Total guanidine nitrate produced (4.0 lb/hr/tube) - 25,800 lb

d) "In-the-barrel" guanidine nitrate - 19,300 lb

e) Unrecovered guanidine nitrate - 6,500 lb

Accountability of Unrecovered Guanidine Nitrate

	Estimated Pounds
Loss from evaporator feed pump leak	620
Loss from solid bowl cleaning	630
Loss from cleaning polishing filters	180
Loss from crystallizer hang-up flush-outs	380
Loss from centrifuge cleanings	180
Loss from centrifuge charging slops	2,015
Loss from two evaporator feed tank dumps	135
Loss from reactor tube drainings	250
Loss from four-day period with product from 4 reactor T-112	1,150
Other losses including (1) start-up, (2) leaks, and (3) samples	960
Total	6,500

portion of GN produced was not considered acceptable for shipment to Cyanamid of Canada. Shipments and lot analyses are discussed in a separate section.

3. Catalyst Performance

Prior to the operations discussed in this report, the following minimum catalyst mileages had been demonstrated:

a) 1-inch diameter reactor, no recycle - 68 gm GN/gm catalyst

b) 4-inch diameter reactor, no recycle - 38 lb GN/lb catalyst

* Houdry CP-532 macroporous silica beads

These values represented significant improvements over any previously demonstrated catalyst mileage for the U/AN process for manufacturing guanidine nitrate. Preliminary cost estimates indicated that the catalyst mileage should be at least 200 lb GN/lb catalyst from the standpoint of both catalyst replacement cost and operating logistics. One of the objectives of the 1973 operations was to demonstrate this minimum mileage level with the pilot plant operating on a recycle basis. This level of catalyst mileage would represent about a 6-week reactor turn-around in a production plant which is practical from an operating standpoint.

Reactor R-200 served as the basis for demonstrating catalyst mileage. Twice during the period that R-200 was in operation (June 11 and July 6), feed to the other reactors was terminated for about two hours to determine if the catalyst in R-200 was still active. Analytical results indicated that the catalyst, with recycled AN and U complementing virgin feed makeup, was as active as at the beginning of operations. This reactor voluntarily stopped accepting feed after 1030 hours of operation (actual time of introducing feed). Figure 5 was graphically integrated for the operating period from May 21 through July 11 (R-200 operating time) to determine the pounds of guanidine nitrate produced and the resulting demonstrated catalyst mileage. The mileage obtained is considered to be a minimum value since the catalyst bed became plugged rather than the catalyst losing its activity. Productivity values plotted in Figure 5 are based on reactor product analyses with more than one reactor on stream, but the values were assigned to R-200 based on the above-mentioned activity check points. The calculated minimum mileage was 188 lb GN/lb catalyst. A conservative value of 200 lb GN/lb catalyst can be assumed for plant design. Data for determining this mileage are presented in Table 18.

Houdry silica bead attrition occurred as evidenced by the presence of sand in the reactor quench tank and loss of catalyst beads in the reactor, particularly the 10-inch depletion in R-200. This depletion took place over a period of 37 days and some of it may have been due to packing. If one assumes that the

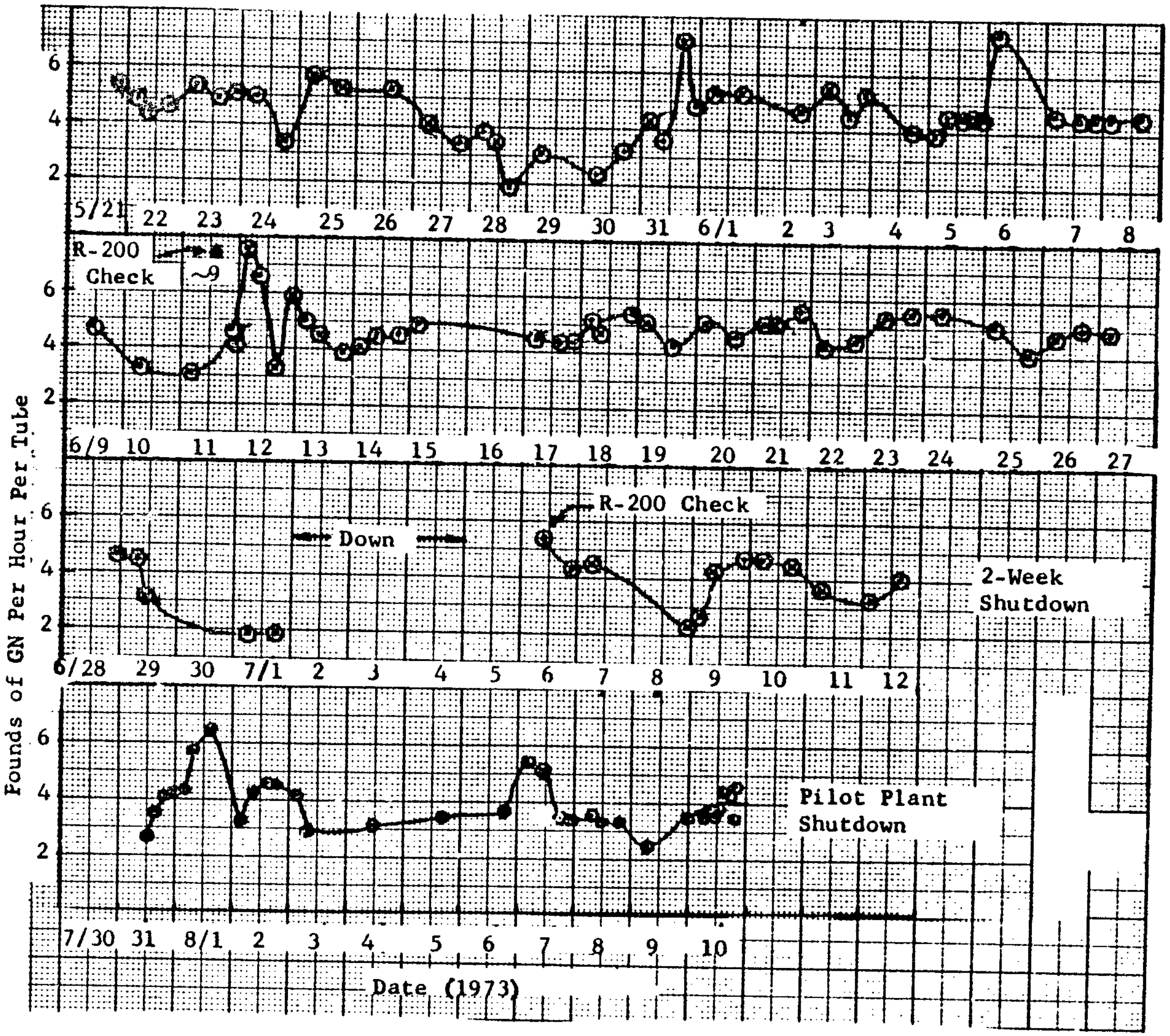


Figure 5. Calculated Productivity Per Tube

TABLE 18

ESTABLISHMENT OF HOUDRY CP-532 SILICA BEAD CATALYST MILEAGE

(REACTOR R-200)

Time Increment	Hours	GN lb/hr	GN lb
7:00 p.m., 5/21 - 8:00 a.m., 5/24	61	5.0	305
8:00 a.m., 5/24 - 8:00 a.m., 5/25	24	4.4	106
8:00 a.m., 5/25 - 12:00 Noon, 5/27	52	5.0	260
12:00 Noon, 5/27 - 12:00 Noon, 5/31	96	3.5	336
12:00 Noon, 5/31 - 12:00 Noon, 6/3	84	5.0	420
12:00 Noon, 6/3 - 4:00 a.m., 6/6	52	4.2	218
4:00 a.m., 6/6 - 9:00 a.m., 6/7	Down	-	-
9:00 a.m., 6/7 - 12:00 Noon, 6/9	51	4.7	240
12:00 Noon, 6/9 - 11:00 a.m., 6/11	47	4.0	188
11:00 a.m., 6/11 - 3:00 p.m., 6/11	Down	-	-
3:00 p.m., 6/11 - 12:00 Noon, 6/14	69	5.0	345
12:00 Noon, 6/14 - 12:00 Noon, 6/21	168	5.0	840
12:00 Noon, 6/21 - 9:00 a.m., 6/27	141	5.0	705
9:00 a.m., 6/27 - 4:00 a.m., 6/28	Down	-	-
4:00 a.m., 6/28 - 3:00 a.m., 6/29	23	4.5	104
3:00 a.m., 6/29 - 2:00 a.m., 7/5	Down	-	-
2:00 a.m., 7/5 - 4:00 p.m., 7/11	158	4.0	632
	1,026		4,699

$$\text{Catalyst Mileage} = \frac{4{,}699 \text{ lb/GN}}{25 \text{ lb Catalyst}} = \underline{\underline{188 \text{ lb/GN/lb/Catalyst}}}$$

2.5 lb of makeup catalyst was due to attrition, then the attrition or usage rate is about 0.6 lb catalyst/1000 lb GN produced in the reactor. On this basis, a reactor could produce about 17,000 lb of GN before requiring the addition of new catalyst. This value is based on a 40% catalyst depletion which should result in about a 20% urea conversion decrease (based on computer predictions, included in Volume I of the Final Report, for a 4-inch-diameter reactor). (This is equivalent to a mileage of 1750 if the bed is topped.)

4. Reactor Feed Systems

The pilot plant was originally installed with an in-line densitometer to measure the density of reactor feed and, consequently, automatically reset the individual stroke lengths of the Hills-McCanna pump to vary the ratio of virgin to recycle material. This system was not totally satisfactory because of the varying water and guanidine nitrate contents of the recycle stream. The densitometer was removed from the system and the Hills-McCanna pump was manually adjusted to control the feed AN/U molar ratio. Control of the AN/U ratio was not as good as desired, but operators were able to command the system and bring the ratio back to about the desired value. Pump settings were determined by the difference between actual and desired AN/U ratios plus the analyses of the recycle and virgin streams. Inventory of recycle material was controlled through small changes in the total reactor feed rate; i.e., higher feed rate for decreasing inventory and lower feed rate for increasing inventory. Occasionally, the Hills-McCanna pump would malfunction because of the presence of gases, dirt, etc., but the problems were not insurmountable. The major mechanical problem was the result of ammelide buildup in the ball checks. The pump heads had to be removed for physical cleaning of the balls and seats.

Feed rate to the reactor was initially controlled with a single-flow recorder controller, depending upon pressure drop through the individual reactors for even feed distribution. This system was not satisfactory. Consequently, a flow indicator controller was installed on each reactor. This installation proved satisfactory in that the ability of a reactor to sustain flow could be determined. Since installation of a flow controller for each reactor tube in a production plant will be prohibitive, the design of multi-tube reactors will have to be analyzed thoroughly. One suggested method for flow distribution would be to use orifices sized for a 20-30 psig pressure drop and no more than 100% flow in excess of the design value.

5. Gas-Liquid Separation and Reactor Product Quenching

The gas-liquid separators used in the pilot plant for separating reactor off-gases (NH_3 and CO_2) from the reactor melt functioned quite well. A small amount of reactor product was entrained in the gases and, subsequently, found in the water-scrubber effluent. Production plant gas-liquid separators equipped with entrainment devices should not present any design problems.

Quenching the hot reactor product melt with water presented no serious problems; however, subsequent ammelide removal, GN recovery and evaporator problems were lessened by monitoring the quench stream density (and crystallization point at times) and adjusting the water addition rate accordingly. A density of about 1.24 gm/cc at ca. 70° C favored good operations. Some problems were encountered in pumping the 90° C aqueous quench material because of the presence of NH_3 vapors and vapor pressure of the water. An increased pump suction head would have minimized this problem. To minimize ammonia in the quench system, it is imperative that the reactor product melt line contain a sufficiently designed liquid seal loop.

6. Insolubles Removal

Water insolubles (ammelide) produced in the reactor as a urea polymerization product must be removed from the system before GN crystallization and recovery. A laboratory model De Laval solid bowl centrifuge was employed for this operation. Considerable mechanical problems were encountered with this unit because of the 24-hour per day, seven-day week requirement. Consequently, the centrifuge was inoperative on several occasions. The solid bowl centrifuge, when operative, effectively removed insolubles from the system as evidenced by the quantities of cakes removed, analyses of the cakes (Table 19), levels of insolubles in the recovered GN, and constant level of insolubles in the reactor product melt. If the feed to the centrifuge was too high in total solids, there was a tendency to kick out guanidine nitrate. In any event, it was necessary to steam trace the curb of the centrifuge to prevent solids buildup in the overflow annulus and discharge nozzle. A similar unit with a continuous plow arrangement should be satisfactory for a production plant.

7. Mother Liquor Evaporation

The Whitlock air [illegible] falling-film evaporator employed for concentrating GN centrifuge mother li[illegible] a commercial prototype unit. High-pressure plant steam rather than steam [illegible] Ebcor electric boilers was supplied to the evaporator. Downtime resulting from lack of steam was zero, a considerable improvement over previous [illegible] ons. The evaporator functioned well with the unit operating at design capacity and normally producing a bottoms product with less than 1% water. Operation of the evaporator did become erratic following the buildup of ammelide in the system. Post-inspection of the unit disclosed a film of insoluble material on all four tubes. Removal of such a film in a commercial unit would not be a serious problem. However, elimination of insolubles in the feed stream would be desirable. Results of special tests revealed the loss of about 1 to 1-1/2% of the ammonium nitrate present in the feed in the exit air. A suitable entrainment separator or scrubber would be desirable on a commercial unit. Analyses of the evaporator bottoms are shown in Table 20. These data do not indicate any buildup of unknowns in the system. A programmed chemical purge from the overall system is not required based on these results.

TABLE 19

SOLID BOWL CENTRIFUGE MATERIAL ANALYSES

DATE (1973)	TIME	AN (%)	U (%)	GN (%)	H_2O (%)	INSOL (%)	TOTAL SOLIDS (%)	ASH (%)
5/25	5:00 a.m.	25.5	2.6	52.7	-	5.3	-	-
5/26	9:30 p.m.	12.6	7.0	43.8	-	13.5	-	-
5/30	4:30 a.m.	22.0	4.9	62.6	-	4.2	-	-
6/6	12:45 a.m.	-	-	-	4.28	24.0	-	-
6/7	9:30 p.m.	21.7	-	58.1	17.0	9.49	-	-
6/15	7:30 a.m.	28.5	10.5	57.9	22.3	36.8	-	-
6/18	4:30 a.m.	20.8	7.67	36.1	24.0	13.4	-	-
6/19	4:00 a.m.	13.6	2.4	72.9	13.61	1.6	-	-
6/20	5:00 a.m.	25.7	3.12	19.77	35.9	-	-	-
6/21	4:00 a.m.	21.3	3.6	45.2	-	20.4	-	-
7/1	-	-	-	-	-	92.14	90.0	0.10
7/2	-	-	-	-	-	-	-	0.23
7/7	8:00 a.m.	15.8	5.2	66.0	18.1	15.01	-	-
7/10	9:30 p.m.	26.3	7.83	19.2	-	2.83	-	-
7/11	3:30 a.m.	-	-	-	-	19.91	74.18	0.60
7/12	5:00 a.m.	-	-	-	-	23.06	74.46	0.16
7/12	9:30 a.m.	-	-	-	-	18.75	76.60	0.19

TABLE 20

1973 EVAPORATOR BOTTOMS ANALYSES

DATE	TIME	AN (%)	U (%)	GN (%)	H_2O (%)	INSOL (%)	CLOSURE (%)
5/25	3:30 p.m.	78.3	8.1	7.2	4.8	-	98.4
6/9	4:30 p.m.	60.7	10.8	27.9	0	-	99.4
6/10	1:10 p.m.	62.1	11.7	26.6	0	-	99.8
6/11	5:00 a.m.	65.1	14.1	21.6	2.04	-	103.2
6/11	8:30 p.m.	61.2	17.2	20.2	1.84	-	100.44
6/12	3:00 a.m.	62.9	14.7	20.7	-	.05	98.35
6/13	8:45 a.m.	-	-	-	.51	-	-
6/13	7:50 p.m.	66.8	16.8	12.3	.62	-	96.52
6/14	4:00 a.m.	65.1	17.9	13.3	2.18	-	98.48
6/15	3:00 a.m.	71.4	20.4	0.01	1.35	-	93.16
6/15	4:00 a.m.	-	-	-	1.49	-	-
6/16	4:00 a.m.	63.0	22.4	12.8	1.03	.002	99.242
6/17	4:00 a.m.	64.3	21.1	13.8	-	-	-
6/18	4:00 a.m.	67.1	20.9	8.52	.77	-	97.29
6/19	4:00 a.m.	69.9	16.3	13.6	.96	-	100.76
6/20	4:00 a.m.	75.8	10.9	10.5	.75	-	97.95
6/21	4:00 a.m.	77.9	10.3	11.0	.77	-	99.77
6/22	4:00 a.m.	80.4	8.47	10.5	.39	-	99.76
6/23	4:00 a.m.	75.9	11.7	11.9	.56	-	100.06
6/24	3:50 a.m.	73.8	11.7	11.7	1.29	-	98.49
6/25	4:40 a.m.	73.4	12.2	14.4	.54	-	100.54
6/26	4:05 a.m.	76.2	10.0	12.6	.72	-	99.52
6/27	4:00 a.m.	61.1	16.0	23.4	.61	-	101.11
6/29	3:45 a.m.	68.6	16.7	9.33	1.41	.40	96.08
7/1	3:30 a.m.	65.4	20.9	12.15	.42	-	98.91
7/7	3:00 a.m.	67.3	18.2	10.9	1.52	-	97.92
7/8	3:00 a.m.	-	17.0	-	2.79	-	-
7/9	4:00 a.m.	63.6	11.8	10.3	3.88	-	94.78
7/10	4:00 a.m.	43.6	11.8	5.7	31.8	-	92.90
7/11	11:45 a.m.	63.4	20.2	13.8	.88	-	98.28
7/12	4:00 a.m.	-	-	-	3.6	-	-
7/30	10:30 p.m.	81.0	7.65	7.04	2.22	-	97.91
8/1	11:30 a.m.	75.4	11.9	9.2	2.18	-	98.68
8/1	7:30 p.m.	69.0	17.5	11.1	0.73	-	98.33
8/3	4:00 a.m.	71.4	11.2	13.8	1.08	.45	97.93
8/3	8:00 p.m.	70.9	13.3	12.1	1.29	-	97.59
8/6	4:00 a.m.	69.8	17.2	8.93	1.01	-	96.94
8/7	4:00 a.m.	71.2	16.6	8.8	3.8	-	100.4
8/7	7:30 p.m.	69.6	19.2	8.79	0.76	-	98.35
8/8	4:00 a.m.	70.7	18.4	10.2	0.76	.13	100.19
8/9	3:30 p.m.	65.6	13.7	12.2	7.89	-	-

8. Guanidine Nitrate Crystallization

The vacuum crystallizer used in the pilot plant is a prototype commercial unit. The size of the unit was selected on the basis of a batch operation to produce 50 lb of guanidine nitrate per hour. The unit was also designed for continuous operation although no attempt was made to demonstrate this for crystallizing guanidine nitrate in the pilot plant.

The designed cycle time for the crystallizer was four hours. Three hours were allocated for charging and crystallizing, and one hour was allocated for discharging the slurry to the centrifuge in four increments of 15 minutes duration each.

Actual operation showed that charging the crystallizer required 20-25 minutes, crystallization took 1-1/2 to 2-1/2 hours, and charging the centrifuge one hour. At no time were the crystallization and centrifuge operations unable to keep up with the reactor-quench system.

There was no noticeable difference in the final product when the crystallization time was 1-1/2 rather than 2-1/2 hours. Apparently any difference in crystal size was small enough not to cause difficulty in handling the material in the centrifuge. No difficulty was encountered in pumping the crystal slurry through the pump-around-loop from which the centrifuge was fed. This unit operation can be successfully scaled up to plant size without difficulty.

Reduced pressure for evaporative cooling was created via three Stokes mechanical vacuum pumps. Introduction of procedures to drain condensed water from the oil reservoirs and air purging of the oil on a shift basis minimized crystallizer downtime. Maintenance of the vacuum pumps and attainments of good vacuum were augmented by a dry ice trap on the suction side of the vacuum pumps. Only minor problems were encountered with the Edwards chiller.

A total of 180 crystallizer batches was processed. Operating data are summarized in Table 21. Feed to the crystallizer averaged 64% total solids with a range of 44% to 74%.

9. Crystalline Guanidine Nitrate Recovery

The DeLaval, 22-inch-diameter, link suspended centrifuge was the least troublesome piece of process equipment installed and operated in the pilot plant. The hydraulic power system resulted in excellent speed control and rapid speed change response. The original polypropylene filter cloth was still in use at the end of the program.

TABLE 21

GUANIDINE NITRATE PILOT PLANT CRYSTALLIZATION

		Transfer to Crystallizer				Start of Crystallization			Finish of Crystallization			Centrifugation				
		Start		Finish												
Batch No./	Date	Time	T-106 Level (in.)*	Time	T-106 Level (in.)	Crystal. Temp. (°F) TIR-1	Chilled H_2O Temp. (°F)	Time Vac. Cracked	Crystal. Temp. (°F) TIR-1	Final Vac.	Time Finished	No. of Chgs.	Total Wash Water (Gal.)	Weight Wet Prod. (lb)	No. of Bags	Condensate Water (Gal.)
101	5/23	1:12 PM	34.5	1:55 PM	13.0	155	35	1:55 PM	70	29+	4:30 PM	4	7	109	4	9
102	5/24	7:35 AM	38.0	3:20 PM	14.5	175	28	8:25 AM	72	29+	10:54 AM	5	8	120	4	8
103	5/24	7:10 PM	32.5	7:40 PM	9.0	170	35		80		10:30 PM	4	4	136	5	
104	5/25	5:20 AM	24.0	7:50 AM	2.0	150	35	7:55 AM	75	28+	11:00 AM	3	3	108	4	5
105	5/25	9:10 PM	27.0	9:45 PM	3.5	166	30	9:45 PM	73	29+	1:00 AM	3	6	113	4	
106	5/26	8:20 PM	26.5	8:50 PM	MT	171	30	8:55 PM	75	29.5	11:00 PM	3	6	124	4	
107	5/28	4:10 PM	26.0	5:10 PM	5.25	170	50	5:10 PM	80	28.0	9:35 PM	5	10	88	3	5
108	5/29	10:25 AM	49.0	11:15 AM	26.0	170	50	11:20 AM	70	28.0	1:30 PM	0	0	0	0	
109	5/29	6:00 PM	36.0	6:30 PM	13.0	170	45	6:31 PM	73	28.0	7:55 PM	0	0	0	0	7
110	5/30	5:00 PM	33.0	5:20 PM	8.0	170	40	5:30 PM	76	28.0	7:05 PM	4	8	40	2	
111	5/31	4:25 AM	26.5	4:54 AM	4.0	183	60	5:00 PM	70	28.0	11:15 AM(1)	3	5	86	3	
112	5/31	4:25 PM	22.5	4:52 PM	0.0	180	40	4:55 PM	80	29.0	7:10 PM	5	9	112	4	6
113	6/1	9:40 AM	27.5	10:05 AM	3.0	180	40	10:05 AM	75	28.5	12:15 PM	3	6	121	4	
114	6/1	7:45 PM	25.0	8:10 PM	4.0	170	39	8:15 PM	80	29.0	12:45 PM	3	5	131	5	6
115	6/2	3:00 AM	27.0	4:00 AM	6.5	176	30	4:18 AM	75	28	8:10 AM	3	6	120	4	
116	6/2	10:00 AM	31.0	10:20 AM	12	160	35	10:20 AM	80	28	1:55 PM	3	7	85	3	
117	6/3	7:45 PM	31.0	8:20 PM	10	175	24	8:23 PM	80	29.5	1:00 AM	3	6	85	3	5
118	6/3	2:45 AM	27.5	3:18 AM	6.25	178	25	3:30 AM	75	27	8:00 AM	3	6	109	5	
119	6/3	10:15 AM	26.0	10:35 AM	3.0	170	25	10:35 AM	80	28	1:40 PM	3	6	128	4	5
120	6/3	6:25 PM	27.25	6:47 PM	3.5	175	24	6:50 PM	75	29.4	1:00 AM	3	6.75	102	3	4
121	6/4	3:05 AM	26.0	3:30 AM	4.25	175	25	3:50 AM	78	28.5	8:55 AM	5	8	107	4	
122	6/4	10:55 AM	29.0	11:25 AM	9.0	173	30	11:30 AM	100	27.5	2:20 PM	4	8	49	2	4
123	6/4	5:45 PM	27.5	6:15 PM	5.5	177	30	6:20 PM	105	28.5	9:00 PM	3	6	44	2	4
124	6/5	1:35 AM	27.0	2:10 AM	2.5	135	26	2:50 AM	69	28.8	3:30 AM	3	5	89	3	5.5

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(1) Chiller problems; restart 10:10 AM

*1 inch = 5 gallons

TABLE 21 (CONTINUED)

	Transfer to Crystallizer				Start of Crystallization			Finish of Crystallization			Centrifugation				
	Start		Finish												
Batch No./ Date	Time	T-106 Level (in.)	Time	T-106 Level (in.)	Crystal. Temp. (°F) TIR-1	Chilled H_2O Temp. (°F)	Time Vac. Cracked	Crystal. Temp. (°F) TIR-1	Final Vac.	Time Finished	No. of Chgs.	Total Wash Water (Gal.)	Weight Wet Prod. (lb)	No. of Bags	Condensate Water (Gal.)
125 6/5	8:22 AM	23.0	8:50 AM	0.5	160	29	8:51 AM	70	28.5	9:51 AM	5	8	105	4	6
126 6/5	5:25 PM	24.5	5:45 PM	2.0	145	35	5:50 PM	69	29	6:36 PM	4	8	116	4	6
127 6/6	2:40 PM	28.5	3:00 AM	5.0	175	30	3:00 AM	70	25	3:45 AM	3	6	84	3	
128 6/7	5:50 PM	24.5	6:15 PM	1.0	170	37	6:17 PM	68	29	7:15 PM	3	6	81	3	6
129 6/8	8:30 AM	23.0	8:55 AM	1.0	170	39	8:57 AM	70	29	9:28 AM	5	8.5	26	1	6
130 6/8	9:18 PM	24.0	9:40 PM	2.0	170	25	9:45 PM	55	29.3	10:30 PM	2	4	9	1	7
131 6/9	10:40 AM	23.5	11:10 AM	0.5	165	39	11:11 AM	67	29	11:47 AM	2	4	0	0	7
132 6/10	11:35 PM	20	12:05 AM	0	165	45	12:05 AM	60	29	1:10 AM	2	4	11	1	
133 6/11	7:40 PM	21.25	8:03 PM	0	160	35	9:15 PM	50	28.75	11:00 PM	4	7	23	1	
134 6/12	8:15 PM	27	8:38 PM	6.0	80	90	8:30 AM	72	27.5	8:55 AM	2	4	8	1	
135 6/12	10:35 AM	55.0	11:00 AM	30.5	155	74	11:00 AM	65	28.0	1:15 PM	3	6	55	2	
136 6/12	3:05 PM	47	3:28 PM	25.5	140	45	4:15 PM	56	28.3	4:48 PM	3	5.5	84	3	5.5
137 6/12	6:15 PM	42	6:35 PM	21.0	165	35	6:40 PM	59	28.8	8:05 PM	3	6	75	3	6.5
138 6/12	10:01 PM	39	10:48 PM	21.0	160	35	11:00 PM	59	29.0	11:25 PM	4	6	60	2	6
139 6/13	4:00 AM	52	4:40 AM	32.0	165	39	4:54 AM	65	28.5	5:15 AM	4	7.5	69	3	7
140 6/13	8:00 AM	52	8:30 AM	30.0	165	35	8:35 AM	45	29	9:00 AM	3	6	87	3	
141 6/13	11:00 AM	43	11:15 AM	22.0	150	35	11:20 AM	75	28.5	1:00 PM	3	6	109	4	
142 6/13	3:10 PM	40	3:30 PM	19.0	N/R[1]	38	3:54 PM	N/R	29.5	6:02 PM	4	8	113	4	7
143 6/13	8:08 PM	40	8:30 PM	18.5	N/R	35	8:30 PM	N/R	29.4	10:30 PM	5	8	134	5	
144 6/14	11:45 PM 6/13	32	12:12 AM	14.0	N/R	38	12:15 AM	N/R	29.0	2:15 AM	5	8	138	5	
145 6/14	6:15 AM	40	7:00 AM	17.5	N/R	28	7:30 AM	N/R	28.5	9:00 AM	4	8	172	6	6
146 6/14	1:00 PM	43	1:25 PM	21	150	30	1:30 PM	70	29.0	4:00 PM	3	6	164	6	
147 6/14	6:00 PM	45	6:20 PM	21	150	30	6:20 PM	75	29.0	8:50 PM	3	6	162	6	
148 6/14	10:30 PM	40	10:45 PM	18	150	30	10:55 PM	75	29.0	1:05 AM	3	6	159	6	7

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[1] No reading.

TABLE 21 (CONTINUED)

Batch No./ Date		Transfer to Crystallizer				Start of Crystallization			Finish of Crystallization			Centrifugation				Condensate Water (Gal.)
		Start		Finish												
		Time	T-106 Level (in.)	Time	T-106 Level (in.)	Crystal. Temp. (°F) TIR-1	Chilled H_2O Temp. (°F)	Time Vac. Cracked	Crystal. Temp. (°F) TR-1	Final Vac.	Time Finished	No. of Chgs.	Total Wash Water (Gal.)	Weight Wet Prod. (lb)	No. of Bags	
149	6/15	3:00 AM	40.0	3:30 AM	18.0	150	32	3:40 AM	75	29.0	5:42 AM	3	6	164	6	2
150	6/15	10:00 AM	47.0	10:30 AM	24.0	158	30	10:35 AM	87	28.7	12:10 PM	3	6	138	5	7
151	6/15	3:00 PM	48.0	3:20 PM	26.0	155	30	3:20 PM	80	27.0	6:20 PM	3	3	152	5	
152	6/15	7:45 PM	45.5	8:10 PM	23.0	150	25	8:10 PM	75	27.0	10:25 PM	3	3	144	5	
153	6/16	12:45 AM	42.0	1:08 AM	21.0	155	30	1:10 AM	75	29	2:35 AM	7	7	146	5	7
154	6/16	4:45 AM	39.0	5:10 AM	17.0	150	30	5:10 AM	72	29	6:57 AM	4	4	153	5	7
155	6/16	9:03 AM	34.0	9:30 AM	11.5	147	30	10:05 AM	85	29.3	12:35 PM	4	4	163	6	6
156	6/16	2:30 PM	37.5	3:00 PM	13.5	150	35	3:05 PM	85	28.5	6:02 PM	3	3	163	6	
157	6/16	7:30 PM	35.0	7:50 PM	11.0	150	30	7:50 PM	85	28.5	9:30 PM	3	3	154	5	
158	6/16	11:00 PM	26.0	11:24 PM	6.0	150	30	11:25 PM	85	28.5	1:10 AM 6/17	3	3	163	6	7
159	6/17	2:15 AM	22.0	2:36 AM	0.0	150	30	2:37 AM	85	28.5	3:45 AM	3	3	150	5	6.5
160	6/17	7:40 AM	22.5	8:20 AM	1.5	145	28	8:24 AM	76	28.8	10:30 AM	4	4	153	5	7
161	6/17	2:04 PM	2[illegible].5	2:30 PM	3.25	150	30	3:25 PM	80	29.0	5:00 PM	3	3	148	5	
162	6/17	7:40 PM	26.0	8:00 PM	4.0	150	25	8:05 PM	80	29.0	10:10 PM	4	4	157	6	
163	6/18	1:40 AM	27.0	2:15 AM	6.5	150	32	2:20 AM	74	28.7	3:35 AM	4	4	161	6	6
164	6/18	7:10 AM	24.0	7:32 AM	0	150	35	7:42 AM	75	28.8	9:32 AM	4	3	158	5	7.5
165	6/18	1:15 PM	25.0	1:42 PM	0.5	140	35	3:35 PM	75	27.0	5:00 PM	3	3	150	5	
166	6/19	11:10 PM 6/18	27	11:35 PM	5.5	150	37	11:47 PM	75	28.9	1:10 AM	4	4	188	7	7
167	6/19	4:55 AM	26.5	5:20 AM	1.5	150	32	5:25 AM	75	28.5	7:13 PM	5	4.5	188	6	6.5
168	6/19	11:35 AM	23.5	11:57 AM	0.5	154	33	12:05 PM	74	28.3	1:42 PM	4	4	198	6	8.5
169	6/19	6:05 PM	24	6:30 PM	4.0	155	35	6:40 PM	[illegible]	29	8:40 PM	4	4	210	7	
170	6/19	11:25 PM	27	11:50 PM	0.5	150	32	11:51 PM	75	29.0	1:15 AM 6/20	5	5	183	6	
171	6/20	6:05 AM	26	6:30 AM	4.5	146	38	6:30 AM	72	28.5	8:34 AM	5	5	151	5	7
172	6/20	12:00 Noon	27	12:25 PM	6	155	35	12:35 PM	75	29.0	3:45 PM	5	5	207	7	8.5

TABLE 21 (CONTINUED)

Batch No./ Date		Transfer to Crystallizer: Start: Time	Transfer to Crystallizer: Start: T-106 Level (in.)	Transfer to Crystallizer: Finish: Time	Transfer to Crystallizer: Finish: T-106 Level (in.)	Start of Crystallization: Crystal. Temp. (°F) TIR-1	Start of Crystallization: Chilled H_2O Temp. (°F)	Start of Crystallization: Time Vac. Cracked	Finish of Crystallization: Crystal. Temp. (°F) TIR-1	Finish of Crystallization: Final Vac.	Finish of Crystallization: Time Finished	Centrifugation: No. of Chgs.	Centrifugation: Total Wash Water (Gal.)	Centrifugation: Weight Wet Prod. (lb)	Centrifugation: No. of Bags	Condensate Water (Gal.)
173	6/20	5:30 PM	28.0	5:52 PM	6.0	150	35	5:53 PM	75	29.0	7:53 PM	5	5	146	5	9
174	6/20	9:35 PM	23.0	10:00 PM	0	150	36	10:01 PM	75	29.0	11:50 PM	4	4	169	6	7
175	6/21	4:45 AM	28.5	5:10 AM	5.0	150	35	5:12 AM	75	28.5	7:15 AM	4	4	131	5	7.5
176	6/21	10:32 AM	28.0	10:56 AM	5.5	154	33	11:00 AM	75	28.8	2:06 PM	5	5	163	6	8
177	6/21	4:00 PM	26.0	4:25 PM	5.0	155	35	4:26 PM	77	29.0	7:30 PM	4	4	178	6	8.5
178	6/21	8:50 PM	24	9:20 PM	2.0	150	35	9:22 PM	75	29.0	11:55 PM	4	4	154	5	6
179	6/22	3:00 AM	24.5	3:25 AM	3.0	150	35	3:27 AM	75	29.0	5:40 AM	4	4	162	6	
180	6/22	9:00 AM	24.0	9:20 AM	0	150	35	9:20 AM	90	28.0	12:25 PM	4	4	120	4	
181	6/22	2:50 PM	31.0	3:15 PM	8.0	150	35	3:16 PM	75	29.0	5:00 PM	5	5	140	5	
182	6/22	6:20 PM	25.0	6:55 PM	5.0	150	32	6:57 PM	76	29.0	8:30 PM	6	6	150	5	
183	6/22	10:40 PM	22.5	11:05 PM	1.0	145	36	11:06 PM	76	28.8	12:45 AM 6/23	4	4	139	5	
184	6/23	4:40 AM	26.5	5:05 AM	3.5	142	35	5:07 AM	75	29.0	7:00 AM	4	4	198	7	
185	6/23	10:35 AM	27.5	11:10 AM	4.5	150	35	11:35 AM	75	29.0	3:00 PM	5.5	5.5	170	6	
186	6/23	4:40 PM	27.0	5:05 PM	7.0	150	35	5:05 PM	76	29.0	7:00 PM	5.5	5.5	157	6	
187	6/23	9:20 PM	24.0	9:45 PM	7.0	150	35	9:50 PM	75	28.2	11:33 PM	5	5	134	5	
188	6/24	3:07 AM	30.0	3:31 AM	8.0	145	35	3:40 AM	72	28.0	5:12 AM	4	4	142	5	
189	6/24	8:00 AM	34.0	8:20 AM	12.0	150	35	8:30 AM	65	27.0	10:30 AM	5	5	147	5	
190	6/24	11:45 AM	30.0	12:20 PM	10.0	150	35	12:30 PM	70	29	2:20 PM	5	5	143	5	
191	6/24	3:15 PM	24.0	3:50 PM	7.0	150	35	4:00 PM	78	29.0	7:30 PM	5	5	154	5	
192	6/24	8:30 PM	24.0	8:55 PM	6.0	150	34	9:00 PM	76	27.9	11:35 PM	5	5	139	5	
193	6/25	2:45 AM	33.0	3:05 AM	12.0	145	35	3:22 AM	75	28.0	5:40 AM	5	5	170	6	
194	6/25	7:40 AM	35	8:00 AM	11.0	150	35	8:10 AM	70	29.0	9:20 AM	4	4	168	6	
195	6/25	11:00 AM	24.5	11:20 AM	4.0	150	35	11:30 AM	75	29.0	12:45 PM	4	4	152	5	
196	6/25	5:10 PM	20.0	5:30 PM	0.0	150	35	5:31 PM	76	29.0	7:30 PM	5	5	157	5	

TABLE 21 (CONTINUED)

	Transfer to Crystallizer				Start of Crystallization			Finish of Crystallization			Centrifugation				
	Start		Finish												
Batch No./ Date	Time	T-106 Level (in.)	Time	T-106 Level (in.)	Crystal. Temp. (°F) TIR-1	Chilled H_2O Temp. (°F)	Time Vac. Cracked	Crystal. Temp. (°F) TIR-1	Final Vac.	Time Finished	No. of Chgs.	Total Wash Water (Gal.)	Weight Wet Prod. (lb)	No. of Bags	Condensate Water (Gal.)
197 6/26	12:20 AM	20.5	12:50 AM	2.25	149	35	12:50 AM	70	28.5	1:57 AM	4.5	4.5	114	4	
198 6/26	5:31 AM	25.0	5:54 AM	3.0	147	39	6:00 AM	70	29.0	7:00 AM	4	4	150	5	
199 6/26	11:50 AM	25.0	12:15 PM	3.5	150	35	12:20 PM	70	29.0	2:05 PM	4	4	160	6	
200 6/26	4:45 PM	26.0	5:10 PM	4.0	157	35	5:15 PM	75	29.0	7:00 PM			164	6	11
201 6/26	9:20 PM	25.0	9:50 PM	5.5	160	37	10:00 PM	75	28.8	11:40 PM	5	5	158	5	8.5
202 6/27	2:24 AM	27.0	2:45 AM	5.0	148	35	2:50 AM	73	27.5	4:30 AM	5	5	160	6	
203 6/27	7:05 AM	26	7:45 AM	7.0	150	35	8:50 AM	75	29.0	9:30 AM	5	5	196	7	
204 6/27	11:30 AM	26	12:20 PM	6.0	150	35	12:20 PM	75	29.0	1:30 PM	4	4	150	5	
205 6/27	10:00 PM	24	10:30 PM	0	144	38	11:34 PM	75	28.5	1:35 AM 6/28	5	5	127	4	
206 6/28	10:25 PM	25	11:00 PM	1.0	145	40	11:20 PM	80	28.5	2:00 AM 6/29	5	4	117	5	5
207 6/29	4:42 AM	24.5	6:20 AM	6.0	149	35	6:20 AM	75	28.5	7:30 AM			145		
208 6/29													143		
209 6/29													122		
210 6/30													150		
211 6/30													85		
212 6/31 213 6/31													100/80		
214 7/5	11:40 PM	21.0	12:15 AM	0.0	150	30	12:15 AM	86	29.0	3:30 AM	5	5	100	4	6
215 7/6	8:40 AM	23.5	9:40 AM	0.0	160	32	9:50 AM	80	29.0	12:00 Noon	5	5	189	7	
216 7/6	5:45 PM	27.5	7:10 PM	11.0	162	32	7:10 PM	75	28.5	8:42 PM	6	6	180	6	
217 7/6	11:15 PM	35.0	12:00 MN	16.0	150	32	12:00 MN	75	29.0	1:05 AM	6	6	173	6	
218 7/7	2:35 PM	32.0	3:00 AM	11.0	155	32	3:05 AM	68	28.5	4:30 AM	6	6	133	5	
219 7/7	5:45 AM	27.0	6:15 AM	7.0	155	34	6:15 AM	70	28.5	7:40 AM	4	4	160	6	
220 7/7	9:15 AM	24.0	9:35 AM	4.0	140	30	9:40 AM	80	28.9	11:00 AM	5	5	158	5	
221 7/7	7:15 PM	23.75	7:40 PM	3.0	155	40	7:40 PM	79	28.5	9:35 PM	5	5	166	6	

TABLE 21 (CONTINUED)

Batch No./ Date	Transfer to Crystallizer				Start of Crystallization			Finish of Crystallization			Centrifugation				Condensate Water (Gal.)
	Start		Finish												
	Time	T-106 Level (in.)	Time	T-106 Level (in.)	Crystal. Temp. (°F) TIR-1	Chilled H_2O Temp. (°F)	Time Vac. Cracked	Crystal. Temp. (°F) TIR-1	Final Vac.	Time Finished	No. of Chgs.	Total Wash Water (Gal.)	Weight Wet Prod. (lb)	No. of Bags	
222 7/8	11:20 PM 7/7	23.0	11:40 PM	3.0	150	33	11:41 PM	78	29.0	12:41 AM	4	4	147	5	7
223 7/8	3:10 AM	23.0	3:32 AM	0.0	150	30	3:33 AM	77	29.0	[illegible]:32 AM	4	4	138	5	
224 7/8	7:07 AM	1.5	7:34 AM	0.25	135	32	7:36 AM	72	29.0	9:45 AM	5	5	134	5	9
225 7/8	11:30 AM	24.5	11:55 AM	1.5	150	40	12:05 PM	78	29.0	2:12 PM	4	4	142	5	
226 7/8	3:30 PM	22.0	4:00 PM	0.0	150	40	4:00 PM	75	29.0	5:30 PM	4	4	174	6	
227 7/8	6:45 PM	22.0	7:10 PM	3.0	150	40	7:10 PM	75	29.0	9:00 PM	4	4	139	5	
228 7/8	10:30 PM	25.0	11:01 PM	6.0	150	38	11:05 PM	75	29.0	12:35 AM 7/9	4	4	108	4	
229 7/9	1:30 AM	21.0	2:00 AM	3.0	145	37	2:01 AM	74	29.0	4:00 AM	3	3	78	3	
230 7/9	4:50 AM	21.0	5:16 AM	1.0	145	37	5:17 AM	68	29.0	6:55 AM	4	4	54	2	9
231 7/9	9:05 AM	24.0	9:45 AM	2.5	150	32	9:45 AM	83	28.7	11:40 AM	4	4	101	4	7.5
232 7/9	1:30 PM	26.0	1:52 PM	2.5	145	41	1:55 PM	80	29.0	4:00 PM	4	4	138	5	
233 7/9	6:00 PM	22.0	6:15 PM	0.0	145	40	6:15 PM	80	29.0	8:00 PM	4	4	125	4	
234 7/9	8:50 PM	24.0	9:20 PM	4.0	140	40	9:20 PM	80	29.0	11:00 PM	4	4	140	5	6
235 7/9	11:40 PM	22.0	12:10 AM	2.0	140	40	12:10 AM 7/10	76	29.0	1:05 [illegible]	3	3	53	3	6
236 7/10	3:00 AM	21.0	3:30 AM	0.0	145	37	3:30 AM	80	29.0	5:00 AM	3	3	104	4	
237 7/10	7:50 AM	22.0	8:24 AM	0.5	149	30	8:20 AM	75	29.0	9:45 AM	4	4	103	4	
238 7/10	12:45 PM	22.0	1:27 PM	4.0	154	35	1:48 PM	80	29.0	3:30 PM	4	4	169	6	
239 7/10	6:00 PM	25.0	6:45 PM	6.0	150	40	6:45 PM	80	29.0	8:40 PM	4	4	157	5	
240 7/10	9:50 PM	22.0	10:10 PM	0.0	150	40	10:11 PM	80	29.0	12:05 AM 7/11	4	4	135	5	
241 7/11	2:05 AM	21.0	2:40 AM	2.0	150	40	2:40 AM	80	29.0	4:40 AM	4	4	135	5	
242 7/11	5:45 AM	21.0	6:20 AM	0.0	150	35	6:21 AM	75	28.5	7:56 AM	4	4	112	112	6
243 7/11	10:18 AM	21.75	10:45 AM	0.0	149	30	10:48 AM	75	28.5	12:15 PM	4	4	138	5	8
244 7/11	3:25 PM	24.0	3:50 PM	2.0	150	35	3:50 PM	80	29.0	4:50 PM	4	4	167	6	
245 7/11	7:15 PM	22.0	7:40 PM	0.0	150	35	7:40 PM	80	29.0	8:45 PM	4	4	138	5	

TABLE 21 (CONTINUED)

Batch No./ Date	Transfer to Crystallizer				Start of Crystallization			Finish of Crystallization			Centrifugation				Condensate Water (Gal.)
	Start		Finish												
	Time	T-106 Level (in.)	Time	T-106 Level (in.)	Crystal. Temp. (°F) TIR-1	Chilled H_2O Temp. (°F)	Vac. Cracked	Crystal. Temp. (°F) TIR-1	Final Vac.	Time Finished	No. of Chgs.	Total Wash Water (Gal.)	Weight Wet Prod. (lb)	No. of Bags	
246 7/12	12:12 AM	22.5	12:37 AM	0.0	150	35	12:40 AM	75	28.6	1:50 AM	4	4	116	4	
247 7/12	5:33 AM	24.0	6:00 AM	3.5	140	35	6:05 AM	72	28.0	7:15 AM	4	4	112	4	
248 7/12	10:20 AM	24.5	10:42 AM	0.5	144	35	11:04 AM	74	29.0	12:55 PM	4		122	5	
249 7/12	3:45 PM	24.0	4:05 PM	2.0	150	40	4:05 PM	75	29.0	5:35 PM	4	4	124	4	
250 7/12	8:10 PM	22.0	8:30 PM	0.0	150	40	8:30 PM	75	29.0	10:00 PM	4	4	118	4	
251 7/13	1:05 AM	24.0	1:40 AM	0.5	145	45	1:42 AM	80	28.5	4:00 AM	4	4	112	4	
252 7/13	7:05 AM	25.5	7:29 AM	4.0	138	32	7:45 AM	81	28.0	10:01 AM	4	4	104	4	
253 7/13	1:50 PM	22.5	2:25 PM	5.0	150	32	2:25 PM	80	28.5	5:40 PM	4	4	110	4	
254 7/13	7:30 PM	22.0	7:50 PM	0.0	150	40	7:50 PM	80	28.5	11:40 PM	4	4	78	3	
255 7/14	1:00 AM	25.0	1:30 AM	2.0	150	32	1:32 AM	85	28.1	3:47 AM	4	4	78	3	
256 7/14	6:30 AM	27.0	7:20 AM	5.0	140	30	7:27 AM	90	28.0	9:30 AM	4	4	38	2	
257 7/14	11:25 AM	24.5	11:55 AM	2.0	155	40	12:00 Noon	90	28.5	4:00 PM	3	3	44	2	
258 7/14	4:50 PM	21.0	5:15 AM	1.0	150	38	5:15 PM	90	29.0	7:45 PM	3	3	60	2	
259 7/14	9:22 PM	21.0	9:45 PM	0.0	140	40	9:50 PM	90	28.5	2:00 AM					
260 7/31	8:05 PM	24	8:45 PM	4.0	170		8:52 PM	85	28.5	4:00 AM	3	3	20	1	2
261 8/1	6:35 AM	26.5	7:10 AM	3.0	160		7:10 AM	95	28.0	12:30 PM	4	4	88	3	
262 8/1	3:40 PM	25.5	4:05 PM	2.5	155	32	4:10 PM	90	28.5	5:30 PM	4	4	49	2	
263 8/2	5:55 AM	24.5	6:15 AM	3.0	155	40	6:17 AM	80	28.0	10:00 AM	5	5	93	3	6.5
264 8/2	3:40 PM	24	4:15 PM	2.0	150	40	4:15 PM	90	28.0	7:15 PM	3	3	94	3	
265 8/3	4:15 AM	25.5	4:50 AM	1.5	155	35	4:55 AM	80	28.5	7:55 AM	4	4	101	4	6
266 8/3	3:10 PM	22.0	4:15 PM	0.0	150	40	4:15 PM	80	28.0	5:30 PM	4	4	116	4	
267 8/4	2:30 AM	23.5	3:10 AM	0.0	157	40	3:12 AM	75	29	5:12 AM	4	4	107	4	
268 8/4	2:00 AM	22.5	2:20 PM	6.0	150	37	2:23 PM	92	28	7:00 PM	3	3	75	3	6.5
269 8/4	11:45 PM	22.0	12:00 MN	0.0	160	75	12:00 MN 8/5	75	29	2:30 AM	4	4	99	3	

TABLE 21 (CONCLUDED)

	Transf·r to Crystallizer				Start of Crystallization			Finish of Crystallization			Centrifugation				
	Start		Finish												
Batch No./ Date	Time	T-106 Level (in.)	Time	T-106 Level (in.)	Crystal. Temp. (°) TIR-1	Chilled	Time Vac. Cracked	Crystal. Temp. (°F) TIR-1	Final Vac.	Time Finished	No. of Chgs.	Total Wash Water (Gal.)	Weight Wet Prod. (lb)	Bags	Condensate Water (Gal.)
270 8/5	10:25 AM	24.5	10:50 AM	1.5	160		10:52 AM	90	28.7	1:40 PM	3	3	78	3	5.5
271 8/5	8:00 PM	24.5	8:30 PM	2.0	160	35	8:33 PM	80	28.9	12:45 AM	4	4	110	4	
272 8/6	6:45 PM	35.5	7:10 PM	11.5	160	30	8:15 PM	80	28.5	9:30 PM	4	4	129	5	6
273 8/7	12:00 MN	22.0	12:25 AM	0.0	160	30	12:25 AM	80	29.0	2:40 AM	4	4	103	4	
274 8/7	11:15 AM	24.0	11:40 AM	1.0	150	30	11:40 AM	85	28.0	2:05 PM	4	4	120	4	
275 8/7	8:40 PM	23.5	9:55 PM	1.5	145	32	10:10 PM	77	29.0	11:37 PM	4	4	102	4	
276 8/8	8:45 AM	24.0	9:30 AM	1.5	150	30	9:33 AM	80	28.0	11:20 AM	5	5	90	3	
277 8/8	7:00 PM	22.0	7:40 PM	0.0	150	30	7:40 PM	80	28.0	9:30 PM	4	4	83	3	
278 8/9	9:40 AM	25.0	10:20 AM	1.5	165	30	10:22 AM	85	28.0	1:00 PM	4	4	73	3	
279 8/9	9:40 PM	25.0	10:40 PM	2.5	160	35	10:45 PM	85	28.0	1:30 AM 8/10	4	4	112	4	
280 8/10	4:00 PM	25.0	4:35 PM	3.0	160	35	4:35 PM	85	28.0	4:10 AM	3	3	115	4	

Normally, four slurry charges were processed through the centrifuge for each crystallizer batch. The number of charges sometimes varied, depending upon the crystallizer feed GN concentration, etc. Total batch sizes approximated 160 lb or 80% of design. Batch sizes as large as 210 lb of wet GN resulted from a single crystallizer charge. Operation of the centrifuge developed into an art, particularly in regard to the rate of slurry charging. A too-rapid charging rate would result in excessive liquid spillage from the bottom chute. Excessively low charging rates would cause the centrifuge to vibrate due to an uneven cake with a low angle beach slope. Several times during the program, it was necessary to manually remove the residual 3/8-inch-thick cake because of low filtration rates. Moisture contents of the GN plowed from the centrifuge averaged 7%. Some individual batches contained as much as 15% water. Others were as low as 3% water.

The standard procedure for a centrifuge increment was as follows:

(a) Adjust basket speed to 700 rpm.

(b) Charge slurry to centrifuge as fast as possible without spillage from the bottom chute.

(c) Increase the basket speed to 1250 rpm and wring the cake for 2 minutes.

(d) Wash GN cake with 1 gallon of water (established as sufficient for nominal 95% GN product).

(e) Wring cake for an additional 2 minutes.

(f) Reduce basket speed to 100 rpm and plow cake into canvas bags.

Analytical data for each batch of GN produced are presented in Table 22. Analyses were performed on wet GN. To assess the consistency of product quality, the analyses were normalized to 100% and on a dry basis.

10. Guanidine Nitrate Drying

Because of the difficulties encountered in processing products through the Strong-Scott Solidaire indirect-heated dryer during the 1972 campaign, it was decided that all guanidine nitrate produced in 1973 would be dried on trays in a commercial smokeless dry house.

The wet GN produce, normally containing 5% to 15% water, was plowed from the centrifuge into 19 in. x 42 in. layflat duck cloth bags. A total of 30 lb was charged to each bag. Each bag was closed with a string tie close to the neck so the material could be distributed into a thin 3-inch layer. The bags were then

TABLE 22

ANALYSIS OF GUANIDINE NITRATE MADE IN 1973

Batch No.	Date	No. of Bags	Wet Wt. (lb)	Actual Wet Analysis (Wt. %)						Dry Normalized				M.P. (°C)	Comments
				AN	U	GN	Insol.	H_2O	Total	AN (%)	U (%)	GN (%)	Ins. (%)		
101	5/23	4	109	1.7	0.2	90.5	-	8.0	100.4	1.85	0.2	98.4	-	210	
102	5/24	4	120	11.8	0.2	81.9	0.11	5.1	99.1	12.55	0.2	87.1	0.12	-	Destroyed
103	5/24	5	136	8.6	0.2	85.8	0.18	3.4	98.2	9.12	0.2	90.6	0.19	-	Destroyed
104	5/25	4	108	2.0	0.2	92.8	0.23	4.9	100.1	2.10	0.2	97.6	0.24	-	
105	5/25	4	118	0.86	-	94.7	-	5.5	101.1	0.95	-	99.2	-	212	
106	5/26	4	124	0.56	0.1	85.7	0.12	14.2	100.7	0.70	0.1	99.1	0.14	-	
107	5/28	3	88	0.72	0.4	92.6	-	5.4	99.1	0.74	0.4	98.8	-	212	
108	5/29	0	0	-	-	-	-	-	-	-	-	-	-	-	
109	5/29	0	0	-	-	-	-	-	-	-	-	-	-	-	
110	5/30	2	40	0.89	Nil	92.6	-	7.1	100.6	0.97	Nil	99.1	-	212	
111	5/31	3	86	1.09	Nil	95.8	-	4.1	101.0	1.14	Nil	99.0	-	212	
112	5/31	4	112	3.15	Nil	91.3	-	7.5	102.0	3.31	1.2	95.6	-	207.5	
113	6/1	4	121	-	-	-	-	-	-	-	-	-	-	-	
114	6/1	5	131	1.25	0.27	91.5	-	6.8	99.8	1.40	0.3	98.3	-	210	
115	6/2	4	120	0.93	0.27	95.2	-	6.3	102.7	0.96	0.3	98.7	-	211	
116	6/2	3	85	3.86	0.73	95.6	-	9.1	109.3	3.85	0.8	95.4	-	206	
117	6/3	3	85	1.70	0.57	93.0	-	5.7	101.0	1.77	0.6	97.6	-	210	
118	6/3	4	109	1.13	0.32	95.8	-	5.8	103.1	1.15	0.3	98.4	-	211	
119	6/3	4	128	1.50	Nil	93.7	0.10	3.9	99.2	1.57	Nil	98.6	0.10	215	
120	6/3	3	102	1.10	Trace	70.8	14.4	19.4	105.7	1.20	0.0	82.1	16.7	209.5	Destroyed
121	6/4	4	107	-	Nil	-	2.7	-	-	-	-	-	-	-	
122	6/4	2	49	-	Nil	-	0.2	-	-	-	-	-	-	-	
123	6/4	2	44	-	-	-	0.4	-	-	-	-	-	-	-	
124	6/5	3	89	1.24	-	-	-	7.8	-	-	-	>95	-	211	
125	6/5	4	105	1.40	Nil	91.2	0.1	6.8	99.5	1.50	Nil	97.9	0.11	215	
126	6/5	4	116	1.50	-	-	-	-	-	-	-	>95	-	210	
127	6/6	3	84	1.61	Trace	65.1	0.13	13.0	79.8	2.4	Trace	97.5	0.19	210	
128	6/7	3	81	0.48	Nil	95.8	0.27	7.3	103.9	0.50	Nil	99.1	0.28	211	
129	6/8	1	26	-	-	-	-	-	-	-	-	-	-	-	
130	6/8	1	9	0.53	Nil	94.3	-	6.9	101.73	0.56	0.00	99.43	-	208	
131	6/9	0	0	-	-	-	-	-	-	-	-	-	-	-	
132	6/10	1	10.5	0.84	0.00	92.1	-	8.9	101.84	0.90	0.00	99.04	-	208	

TABLE 22 (CONT.)

				Actual Wet Analysis (Wt. %)						Dry Normalized					
Batch No.	Date	No. of Bags	Wet Wt. (lb)	AN	U	GN	Insol.	H_2O	Total	AN (%)	U (%)	GN (%)	Ins. (%)	M.P. (°C)	Comments
133	6/11	1	23	0.42	0.00	105.3	-	10.2	115.92	0.39	0.00	99.64	-	211	
134	6/12	1	8	2.29	0.00	92.7	0.37	7.08	102.44	2.41	0.00	97.21	0.39		
135	6/12	2	55	2.10	0.70	91.5	1.1	8.3	103.7	2.20	0.74	95.91	1.15	219	
136	6/12	3	84	0.51	0.50	95.8	1.4	8.0	106.21	0.52	0.51	97.54	1.43	219	
137	6/12	3	75	0.44	0.00	92.0	0.37	7.7	100.51	0.48	0.00	99.15	0.40	211	
138	6/12	2	60	1.08	0.50	87.4	0.60	13.5	103.08	1.20	0.56	97.56	0.67	217	
139	6/13	3	69	0.61	1.05	94.5	0.40	7.3	103.86	0.63	1.087	97.92	0.42	216	
140	6/13	3	87	2.91	1.15	90.2	0.80	9.5	104.56	3.06	1.21	94.85	0.84	210/ 237	
141	6/13	4	109	0.71	0.73	93.0	0.56	5.6	100.6	0.75	0.77	97.88	0.59	210	
142	6/13	4	113	0.64	0.00	94.1	0.30	4.9	99.94	0.67	0.00	98.95	0.32	210	
143	6/13	5	134	1.26	0.00	88.2	0.95	10.4	100.81	1.39	0.00	97.56	1.02	210	
144	6/14	5	138	1.03	0.00	91.5	0.45	9.0	101.98	1.11	0.00	98.41	0.48	209	
145	6/14	6	172	1.15	0.94	94.3	0.47	7.9	104.76	1.19	0.97	97.35	0.49	211	
146	6/14	6	164	1.73	0.75	94.1	0.95	6.2	103.73	1.78	0.77	96.47	0.98	212/ 225	
147	6/14	6	162	6.37	2.51	85.1	0.21	17.8	111.99	6.77	2.66	90.35	0.22	205	
148	6/14	6	159	11.08	2.94	72.6	0.18	11.07	97.87	12.76	3.38	83.65	0.21	189	
149	6/15	6	164	10.94	2.89	69.3	0.41	15.3	98.84	13.10	3.47	82.93	0.50	195	
150	6/15	5	138	8.54	2.47	76.8	0.28	13.6	101.69	9.70	2.80	87.18	0.32	198	
151	6/15	5	152	3.33	0.87	88.0	0.27	7.3	99.77	3.59	0.94	95.18	0.29	208	
152	6/15	5	144	3.09	0.60	85.5	0.21	8.7	98.10	3.46	0.67	95.64	0.23	211	
153	6/16	5	146	3.00	0.78	81.4	0.21	6.9	92.29	3.51	0.91	95.34	0.24	207	
154	6/16	5	152.5	0.80	-	96.4	-	-	-	-	-	-	-	209	
155	6/16	6	162.6	0.93	-	93.9	-	-	-	-	-	-	-	208	
156	6/16	6	163	2.08/ 0.49	0.00	94.2/ 96.1	0.27	3.08	99.63	2.16	0.00	97.55	0.28	206/ 210	
157	6/16	5	154	1.47/ 1.58	0.00	92.2/ 90.9	0.31	3.55	97.53	1.57	0.00	98.11	0.33	210/ 213	
158	6/16	6	163	1.43/ 0.91	0.00	93.3/ 91.8	0.42	4.13	99.28	1.50	0.00	98.06	0.44	205/ 213	
159	6/17	5	150	2.30	0.95	91.5	0.20	6.21	101.16	2.42	1.00	96.37	0.21	208	
160	6/17	5	152.8	1.24/ 1.18	0.00	95.5	0.41	2.35	99.5	1.28	0.00	98.30	0.42	206/ 211	
161	6/17	5	148	0.71	0.73	93.0	0.56	5.60	100.6	0.74	0.77	97.92	0.59	210	
162	6/17	6	157	3.18	0.00	89.6	0.37	4.27	97.42	3.41	0.00	96.19	0.40	210	
163	6/18	6	161	1.85	0.93	90.7	0.19	6.10	99.77	1.97	0.99	96.81	0.20	208	
164	6/18	5	158.25	1.69	0.00	92.4	0.35	3.25	97.69	1.79	0.00	97.84	0.37	210	

TABLE 22 (CONT.)

Batch No.	Date	No. of Bags	Wet Wt. (lb)	Actual Wet Analysis (Wt. %) AN	U	GN	Insol.	H_2O	Total	Dry Normalized AN (%)	U (%)	GN (%)	Ins. (%)	M.P. (°C)	Comments
165	6/18	5	150	1.38	-	94.1	-	-	-	-	-	-	-	-	
166	6/18	7	188	4.67	1.08	84.3	0.55	13.1	103.7	5.15	1.19	93.0	0.61	209	
167	6/19	6	187.75	1.22	-	95.6	0.21	3.18	100.21	1.25	-	98.53	0.22	211	
168	6/19	6	193	1.37	0.15	88.2	0.20	17.1	107.02	1.52	0.17	98.08	0.23	209	
169	6/19	7	210	4.02	0.37	86.3	0.10	15.1	105.89	4.42	0.40	95.0	0.10	207	
170	6/19	6	183	3.46	-	89.8	0.20	12.2	105.66	3.70	-	96.05	0.21	210	
171	6/20	5	151	0.46	-	97.0	0.05	4.41	101.92	0.47	-	99.48	0.05	215	
172	6/20	7	207	1.98	-	90.9	0.11	8.76	101.75	2.12	-	97.70	0.11	209	
173	6/20	5	146	0.23	-	94.6	0.11	8.74	103.67	0.24	-	99.67	0.11	201	
174	6/20	6	169	8.29	0.58	85.3	0.10	11.9	106.17	8.8	0.62	90.5	0.11	-	
175	6/21	5	131.5	3.56/ 1.66	0.0/ 0.0	89.5/ 94.2	0.96/ 0.26	3.94/ 3.60	97.46/ 99.72					215/ 206	
176	6/21	6	183	0.95	-	93.5	0.40	5.06	99.1	1.0	-	98.4	0.42	214	
177	6/21	6	178	0.66	0.0	92.6	0.11	5.56	98.93	0.71	0.0	99.17	0.12	214	
178	6/21	5	154	0.39	0.0	94.0	0.30	5.65	100.34	0.41	0.0	99.26	0.32	212	
179	6/22	6	162	1.30	0.0	82.6	0.37	12.9	97.17	1.55	0.0	98.01	0.44	215	
180	6/22	4	120	1.46	0.0	94.3	0.41	3.24	-	-	0.0	-	-	205	
181	6/22	5	140	5.67	1.38	82.1	0.37	8.52	98.04	6.33	1.54	91.71	0.42	195	
182	6/22	5	150	6.98	1.08	79.4	0.33	9.37	97.16	7.95	1.23	90.45	0.37	194	
183	6/22	5	139	2.68	0.0	91.3	0.37	5.70	100.15	2.84	0.0	96.77	0.39	206	
184	6/23	7	198	2.02	0.0	94.2	0.41	5.04	101.67	2.09	0.0	97.50	0.42	210	
185	6/23	6	177	1.78	0.0	92.4	0.31	7.85	102.34	1.88	0.0	97.78	0.32	211	
186	6/23	6	157	2.08	0.0	89.8	0.31	10.9	103.09	2.26	0.0	97.44	0.34	211	
187	6/23	5	134.5	0.24	0.0	84.8	0.31	4.48	100.83	0.25	0.0	98.39	1.36	211	
188	6/24	5	142	1.91	0.0	91.0	0.48	7.44	100.83	2.04	0.0	97.44	0.52	209	
189	6/24	5	147	2.08	0.0	90.2	-	4.83	97.11	2.25	0.0	97.74	-	208	
190	6/24	5	143	2.11	0.0	90.1	-	5.93	98.14	2.29	0.0	97.70	-	205	
191	6/24	5	154	2.56	0.0	90.0	-	8.95	101.51	2.67	0.0	97.24	-	205	
192	6/24	5	138.7	1.88	0.0	92.7	-	4.89	99.47	1.99	0.0	97.97	-	207	
193	6/25	6	170	1.75	0.0	94.2	-	3.88	99.83	1.82	0.0	98.18	-	208.5	
194	6/25	6	168	1.68	0.0	93.3	0.60	4.64	100.22	1.76	0.0	97.60	0.63	208	
195	6/25	5	152	1.32	0.0	91.4	0.54	5.04	98.30	1.41	0.0	98.00	0.58	210	
196	6/25	5	157	2.39	0.59	89.04	0.31	6.67	99.00	2.58	0.64	96.44	0.33	208	

TABLE 22 (CONT.)

				Actual Wet Analysis (Wt. %)						Dry Normalized					
Batch No.	Date	No. of Bags	Wet Wt. (lb)	AN	U	GN	Insol.	H_2O	Total	AN (%)	U (%)	GN (%)	Ins. (%)	M.P. (°C)	Comments
197	6/26	4	114	2.10	0.0	92.4	0.35	6.81	101.66	2.22	0.0	97.42	0.36	210	
198	6/26	-	150	2.08	0.0	89.9	0.42	7.75	100.15	2.25	0.0	97.3	0.45	209.5	
199	6/26	-	160	0.85	0.0	93.5	0.49	4.68	99.43	0.89	0.0	98.69	0.51	210	
200	6/26	-	164	1.55	0.0	92.1	0.49	4.58	98.72	1.64	0.0	97.83	0.52	208	
201	6/26	-	158	0.56	0.0	94.1	0.75	4.74	100.15	0.59	0.0	98.62	0.79	211	
202	6/27	-	160	-	0.0	-	-	-	-	-	0.0	-	-	-	
203	6/27	-	196	1.89	0.0	85.0	-	10.9	97.79	2.17	0.0	97.83	-	207	
204	6/27	-	150	3.96	0.43	86.2	-	5.42	96.01	4.37	0.47	95.16	-	205	
205	6/27	-	127	1.69	0.0	92.8	-	3.73	98.22	1.79	0.0	98.21	-	208	
206	6/28	-	117	1.49	0.0	93.8	0.83	5.29	101.41	1.57	0.0	97.59	0.88	210	
207	6/28	-	145	3.91	0.0	85.0	0.84	7.26	97.01	4.36	0.0	94.70	0.94	204	
208	6/29	5	143	2.09	0.0	88.8	0.61	6.47	97.97	2.28	0.0	97.06	0.66	206	
209	6/29	4	122	6.15	1.77	78.6	0.71	9.31	96.54	7.05	2.03	90.10	0.82	199	
210	6/30	5	150	1.96	0.0	89.3	0.64	5.28	97.18	2.13	0.0	97.17	0.70	202	
211	6/30	3	85	3.30	2.40	87.8	-	9.71	103.21	3.53	2.56	93.91	-	209	
212	7/1	4	100	0.40	0.0	97.6	0.70	6.25	104.95	0.40	0.0	98.90	0.71	213	
213	7/1	3	80	1.70	0.0	94.9	1.00	8.42	106.02	1.74	0.0	97.23	1.02	214	
214	7/5	4	100	2.84	0.41	87.5	1.10	5.60	97.45	3.09	0.45	95.27	1.20	212	
215	7/6	-	189	1.40	0.0	91.3	1.24	9.40	103.34	1.49	0.0	97.20	1.32	216	
216	7/6	-	180	1.13	0.0	94.1	0.42	3.60	99.75	1.18	0.0	98.38	0.44	212	
217	7/6	-	173	2.50	0.0	82.5	0.21	15.8	101.01	2.93	0.0	96.82	0.25	211	
218	7/7	-	133	1.55	0.39	92.7	0.41	5.09	100.14	1.63	0.41	97.03	0.43	210	
219	7/7	-	160	4.22	1.10	86.0	0.29	9.04	100.65	4.60	1.20	93.87	0.32	202	
220	7/7	-	158	3.00	0.93	90.1	0.79	5.83	100.65	3.16	0.98	95.57	0.84	205	
221	7/7	-	166	1.60	0.48	96.2	0.42	4.10	102.80	1.62	0.49	97.47	0.43	213	Sample to Cyamid for Conversion to Nitroguanidine
222	7/7	-	147	2.04	0.59	92.2	[illegible]	6.03	101.14	2.15	0.63	96.94	0.30	207	
223	7/8	-	138	2.14	0.62	94.5	0.30	5.13	102.69	2.19	0.63	96.87	0.31	208	
224	7/8	-	134	4.50	0.53	86.0	0.30	6.01	97.34	4.92	0.58	94.16	0.33	-	
225	7/8	-	142	7.13	2.03	81.4	0.67	8.83	100.06	7.82	2.23	89.28	0.73	202	
226	7/8	-	174	1.30	0.37	93.3	0.40	7.50	102.87	1.36	0.39	97.83	0.42	213	
227	7/8	-	139	1.33	0.0	92.0	0.26	5.08	98.67	1.42	0.0	98.30	0.27	210	
228	7/8	-	108	1.89	0.0	87.5	0.36	7.25	97.00	2.11	0.0	97.49	0.40	209	

TABLE 22 (CONT.)

Batch No.	Date	No. of Bags	Wet Wt. (lb)	Actual Wet Analysis (Wt. %) AN	U	GN	Insol.	H_2O	Total	Dry Normalized AN (%)	U (%)	GN (%)	Ins. (%)	M.P. (°C)	Comments
229	7/9	-	78	1.50	0.0	89.6	0.29	7.37	98.76	1.64	0.0	98.03	0.31	212	
230	7/9	2	54	2.20	0.0	92.4	0.25	4.11	98.96	2.34	0.0	98.44	0.26	213	
231	7/9	4	101	1.40	0.24	93.8	0.25	4.50	100.19	1.47	0.25	98.22	0.26	213	
232	7/9	5	138	1.28	0.0	93.7	0.32	5.33	100.63	1.34	0.0	98.32	0.34	210	
233	7/9	4	125	1.87	0.0	93.6	0.26	4.49	100.22	1.96	0.0	97.78	0.27	210	
234	7/9	5	140	-	-	-	-	-	-	-	-	-	-	-	
235	7/10	3	53	2.90	0.83	92.1	0.58	5.10	101.52	3.01	0.86	95.53	0.60	210	
236	7/10	4	104	6.10	0.41	84.5	-	8.99	100.00	6.70	0.45	92.85	-	-	
237	7/10	4	103	1.57	0.30	94.5	0.18	3.13	99.68	1.63	0.31	97.87	0.19	215	
238	7/10	6	169	2.30	0.0	91.3	0.06	4.95	98.61	2.45	0.00	97.48	0.06	212	
239	7/10	5	157	3.40	0.78	89.5	0.18	5.01	98.87	3.62	0.83	95.36	0.19	210	
240	7/10	5	135	10.90	1.30	83.5	0.31	2.58	98.59	11.36	1.36	86.97	0.32	202	
241	7/11	5	135	1.75	0.38	91.6	0.32	4.08	98.13	1.86	0.41	97.40	0.34	215	
242	7/11	4	112	1.74	0.92	93.4	0.29	4.30	100.65	1.81	0.95	96.94	0.30	214	
243	7/11	5	138	1.50	0.0	90.5	0.43	4.52	96.95	1.68	0.0	97.86	0.46	216	
244	7/11	6	167	2.28	0.59	-	0.67	14.4	-	-	-	-	-	208	
245	7/11	5	138	2.37	0.60	90.2	0.35	4.70	98.22	2.53	0.64	96.45	0.38	212	
246	7/12	4	116	2.10	1.10	92.4	0.45	4.60	100.65	2.19	1.14	96.20	0.47	211	
247	7/12	4	112	1.08	0.0	92.1	0.40	3.68	97.26	1.15	0.0	98.42	0.43	-	
248	7/12	5	121	3.81	1.29	91.1	0.76	2.29	99.25	3.93	1.33	93.95	0.79	202	
249	7/12	4	124	0.31	0.0	91.5	0.63	4.90	97.34	0.34	0.0	98.98	0.69	-	
250	7/12	4	118	-	-	-	-	-	-	-	-	-	-	-	
251	7/13	4	112	-	-	-	-	-	-	-	-	-	-	-	
252	7/13	4	104	0.35	0.0	96.1	0.50	4.06	101.01	0.36	0.0	99.11	0.52	208	
253	7/13	4	110	1.46	0.0	94.2	0.77	4.61	101.04	1.51	0.0	97.69	0.80	210	
254	7/13	3	78	-	-	-	-	-	-	-	-	-	-	-	
255	7/14	3	78	1.46	0.0	88.6	0.98	4.90	95.94	1.61	0.0	97.84	1.08	212	
256	7/14	2	38	-	-	-	-	-	-	-	-	-	-	-	
257	7/14	2	44	1.08	0.0	92.1	0.40	3.68	97.26	1.15	0.0	98.42	0.43	211	
258	7/14	2	60	6.75	0.0	87.2	1.48	2.86	98.03	7.08	0.0	91.37	1.55	-	
259	7/14	-	79	32.3	0.0	57.5	1.18	6.36	97.34	35.5	0.0	63.19	1.29	193	Destroyed

TABLE 22 (CONT.)

Batch No.	Date	No. of Bags	Wet Wt. (lb)	Actual Wet Analysis (Wt. %) AN	U	GN	Insol.	H_2O	Total	Dry Normalized AN (%)	U (%)	GN (%)	Ins. (%)	M.P. (°C)	Comments
260	7/31	1	20	0.14	0.00	94.0	2.25	3.46	99.85	0.15	0.00	97.52	2.37	213	
261	8/1	3	88	2.80	0.00	88.3	0.30	6.53	97.93	3.06	0.00	96.61	0.33	209	
262	8/1	2	49	6.53	2.50	84.6	0.83	4.32	98.78	6.91	2.65	89.55	0.88	210	
263	8/2	3	93	2.20	0.00	91.2	0.83	5.85	100.08	2.34	0.00	96.78	0.88	211	
264	8/2	3	94	-	-	-	-	-	-	-	-	-	-	-	
265	8/3	4	101	3.23	0.00	90.4	0.69	5.50	99.82	3.43	0.00	95.84	0.73	208	
266	8/3	4	116	2.59	0.00	89.8	0.28	6.58	99.25	2.80	0.00	96.90	0.30	209	
267	8/4	4	107	1.75	0.00	93.6	0.69	5.19	101.23	1.82	0.00	97.46	0.72	210	
268	8/4	3	75	1.77	0.00	92.5	0.50	3.19	97.96	1.87	0.00	97.61	0.53	210	
269	8/5	3	99	1.10	0.00	90.4	1.33	7.58	100.41	1.19	0.00	97.38	1.43	214	
270	8/5	3	78	0.85	0.00	92.0	0.90	4.89	98.64	0.90	0.00	98.13	0.96	213	
271	8/5	4	110	2.00	0.00	90.8	0.71	4.97	98.48	2.14	0.00	97.10	0.76	212	
272	8/6	4	129	0.90	0.00	93.9	1.21	6.10	102.11	0.94	0.00	97.80	1.25	212	
273	8/6	4	103	1.00	0.00	91.8	0.66	6.30	99.76	1.07	0.00	98.73	0.70	211	
274	8/7	4	120	2.93	0.50	93.7	0.72	4.82	102.67	2.99	0.51	95.75	0.73	205	
275	8/7	4	102	2.97	0.34	87.7	0.16	4.76	95.93	3.26	0.37	96.31	0.18	205	
276	8/8	3	90	1.74	0.00	92.6	0.17	4.18	98.69	1.84	0.00	97.97	0.18	209	
277	8/8	3	83	1.18	0.00	91.7	0.38	7.49	100.75	1.26	0.00	98.33	0.41	207	
278	8/9	3	73	1.80	0.00	92.6	0.37	5.30	100.07	1.90	0.00	97.72	0.39	210	
279	8/9	4	112	2.50	0.00	91.9	0.15	5.00	99.55	2.64	0.00	97.21	0.16	210	
280	8/10	4	115	8.20	0.00	87.0	0.00	4.40	99.60	8.58	0.00	91.39	0.00	205	

laid on smokeless powder-type wooden drying trays. The trays were stacked in criss-cross fashion in 10-high tiers. After the dry bay had been filled with 2200 lb to 2800 lb in this manner, the doors to the bay were closed and the temperature was brought up to 140°F with forced air. The bay was left on heat for 3 days. After this period the heat was turned off, allowed to cool for about 24 hours, and then samples were taken from each batch of GN in the bay and composited into a sample representing a lot. Each lot sample was analyzed for AN, U, GN, insolubles, melting point and water. Analyses of the different lots, presented in a previous section, showed that drying was very efficient. For reference, each dry house bay measured 9 ft x 22 ft x 22 ft for a total volume of 4356 cu. ft. With a blower capacity of 2500 gm, the number of air changes (100% fresh air) per minute was 0.57.

A Wolverine Jet Zone air dryer has been selected for drying wet GN in the BAF production plant design. This decision was based on laboratory tests performed at Kenvil on a jet zone module dryer. These results were presented in Volume I of this Final Report. To supplement the decision, drying tests were performed in a laboratory module of a Wyssmont Turbo Tray dryer at the vendor's laboratory. The test report, presented in Table 22A, notes that water-wet GN was dried readily to the desired moisture level of 1%. A maximum product temperature of 150°F was employed with no evidence of sticking, smearing or dusting. These results place confidence in selecting a GN dryer for a production plant. A preliminary price estimate for a production drying system is presented in Table 22B.

11. Monitoring of the Pilot Plant Operation

a. Reactor Performance

The methods discussed in the calculation section of this report were employed on a day by day basis to monitor the pilot plant operation. Table 23 presents calculated results based on daily feed (Table 24) and product analyses (Table 25) and calculation methods using the nitrate conservation and two-mole stoichiometry assumptions. These results were used as criteria to improve the reactor operation. If the AN/U feed ratio was not stable and/or was drifting to a urea-rich or very high ammonium nitrate-rich regime, changes were made in the relative pump stroke lengths on the Hills-McCanna Blend pump. If the production rate was lower than expected, the operation was adjusted to correct for this by adjusting temperature, feed rate or water content in the feed.

The major value of these calculations was that the catalyst activity was being evaluated daily. If poisoning had occurred in this production campaign, a definite decaying trend would have resulted. In Figure 5, GN productivities per hour per tube are plotted versus time. There are positive and negative swings in this curve, but the trend is not at all downward. In most cases, the individual swings in this curve can be explained by a process upset.

TABLE 22A

RESULTS OF WYSSMONT DRYING TESTS

WYSSMONT COMPANY, Inc.

September 18, 1973

Hercules, Inc.
Kenvil, New Jersey 07847

Attention: Mr. J. Doyle

Re: Drying Guanidine Nitrate in the Wyssmont
Drying System
Our Ref. No. 73109

Gentlemen:

We are pleased to submit our test report for the tests performed in our laboratory on your Guanidine Nitrate material on August 29 and 30, 1973.

You will note from the report that the material was readily dried to the desired moisture. These results show that the TURBO-Dryer will produce a superior quality product.

The final samples of all and intermediate samples of Test No. 3 and 4 have been submitted to you for your evaluations.

If you have any questions, please contact us.

Very truly yours,

WYSSMONT COMPANY, INC.

S. H. Shukla

SHS:pbo
Encl: Test Report
cc: J.Gardner
H.Zack
Wilmington, Del.

TABLE 22A (CONTINUED)

September 18, 1973

TEST REPORT

COMPANY: HERCULES INC. TEST NO: 73109-1, 2, 3, 4

MATERIAL: GUANIDINE NITRATE TEST DATE: 8-29, 30-73

DESIGN INITIAL MOISTURE	11% (wet basis)
DESIGN FINAL MOISTURE	0.5 - 1.0% (wet basis)
REPORTED MOISTURE COMPOSITION	Water
DRY DENSITY REPORTED	42 lbs/cu.ft.
TEST OBJECTIVE	To determine the drying and handling characteristics of Guanidine Nitrate in the TURBO-Dryer

	73109-1	73109-2	73109-3		73109-4	
FEED PREPARATION	Put thru #10 mesh screen to remove lumps	Final from 73109-1 rewetted with water	Dry material rewetted with distilled water			
TEST FEED	Guanidine Nitrate					
CHARACTER OF TEST FEED	Fluffy, white material agglomerates into small soft lumps					
CHARACTER OF TEST PRODUCT	Free-flowing, white material					
			Cenco By Wyss.	KF by Hercules	Cenco by Wyss.	KF by Hercules
MOISTURE OF TEST FEED (Wet Basis)	21.2%	11.0%	9.3%	9.09%	6.7%	5.66%
MOISTURE OF TEST PRODUCT (Wet Basis)	0.6%	0.9%	0.8%	0.51%	0.7%	0.34%
DENSITY OF TEST FEED, LOOSE (lbs/cu.ft)	31.0	31.2	32.5		29.0	
DENSITY OF TEST PRODUCT, LOOSE (lbs/cu.ft)	25.4	27.2	26.3		26.0	
VELOCITY OF DRYING ATMOSPHERE	150	200	200		200	
LAYER THICKNESS	1½"	1½"	1 - 1½"		1½"	
TRANSFER RATE	- - - - - - - - 3 Minutes - - - - - - - - - - - -					
WIPER MATERIAL	- - - - - - - - Teflon - - - - - - - - - - - -					
TRAY MATERIAL	- - - - - - - Stainless Steel - - - - - - - - - -					
TEST DRYING TIME	- - - See enclosed drying curves - - - - - - -					
DRYING ATMOSPHERE AND MATERIAL TEMPERATURE	- - - See enclosed temperature charts - - - -					
DRYING ATMOSPHERE COMPOSITION	- - - - - - - Air - - - - - - - - - - - -					

TABLE 22A (CONTINUED)

WYSSMONT COMPANY, INC.

COMPANY: HERCULES INC. - 2 - **TEST NO:** 73109-1, 2, 3, 4

MATERIAL: GUANIDINE NITRATE **TEST DATE:** 8-29, 30-73

OBSERVATIONS AND CONCLUSIONS

Four tests were run on the Guanidine Nitrate material. The first test, 73109-1, was run on material that had been rewetted by the customer. It was found that the moisture content of this material (21.2% wet basis) was almost double that of design feed moisture (10% wet basis).

Because of this, the dried material from Test 73109-1 was rewetted with distilled water to the proper moisture content (10% wet basis) and was used as the feed for Test 73109-2.

It was decided that the rewetting, drying and rewetting of the material might have affected the drying characteristics, so a fresh dry sample was brought by the customer and rewetted with distilled water to 10% wet basis. This sample was used for Test 73109-3.

The material used for Test 73109-4 was rewetted with distilled water to 6% wet basis. This was done because the customer indicated that some of the material during production might come to the TURBO-Dryer at that moisture content.

All four tests were begun at an air temperature of 200°F. and gradually lowered to keep the material temperature at 150°F. maximum. The material handled very well with no sticking, smearing or dusting and was easily dried to the final moisture specification in all of the tests.

Test 73109-3 using the freshly rewetted sample at 10% wet basis can be used for design purposes.

The TURBO-Dryer is well suited for drying the Guanidine Nitrate.

* **MOISTURE TEST** Cenco 70 setting, 250 watt bulb -
By Wyssmont
Karl Fischer - By Hercules

SAMPLES SUBMITTED TO CUSTOMER
73109-1 2 oz. Final 8-29-73
73109-2 2 oz. Final 8-29-73
73109-3 1 oz. Initial S_1, S_2, S_3, S_4
2 oz. Final 8-30-73
73109-4 1 oz. Initial, S_1, S_2, S_3, S_4
2 oz. Final 8-30-73

TEST WITNESSED BY:
Messrs. M. Whippen, J. Doyle, H. Zack
& J. Gardener of Hercules 8-29-73
Mr. M. Whippen 8-30-73

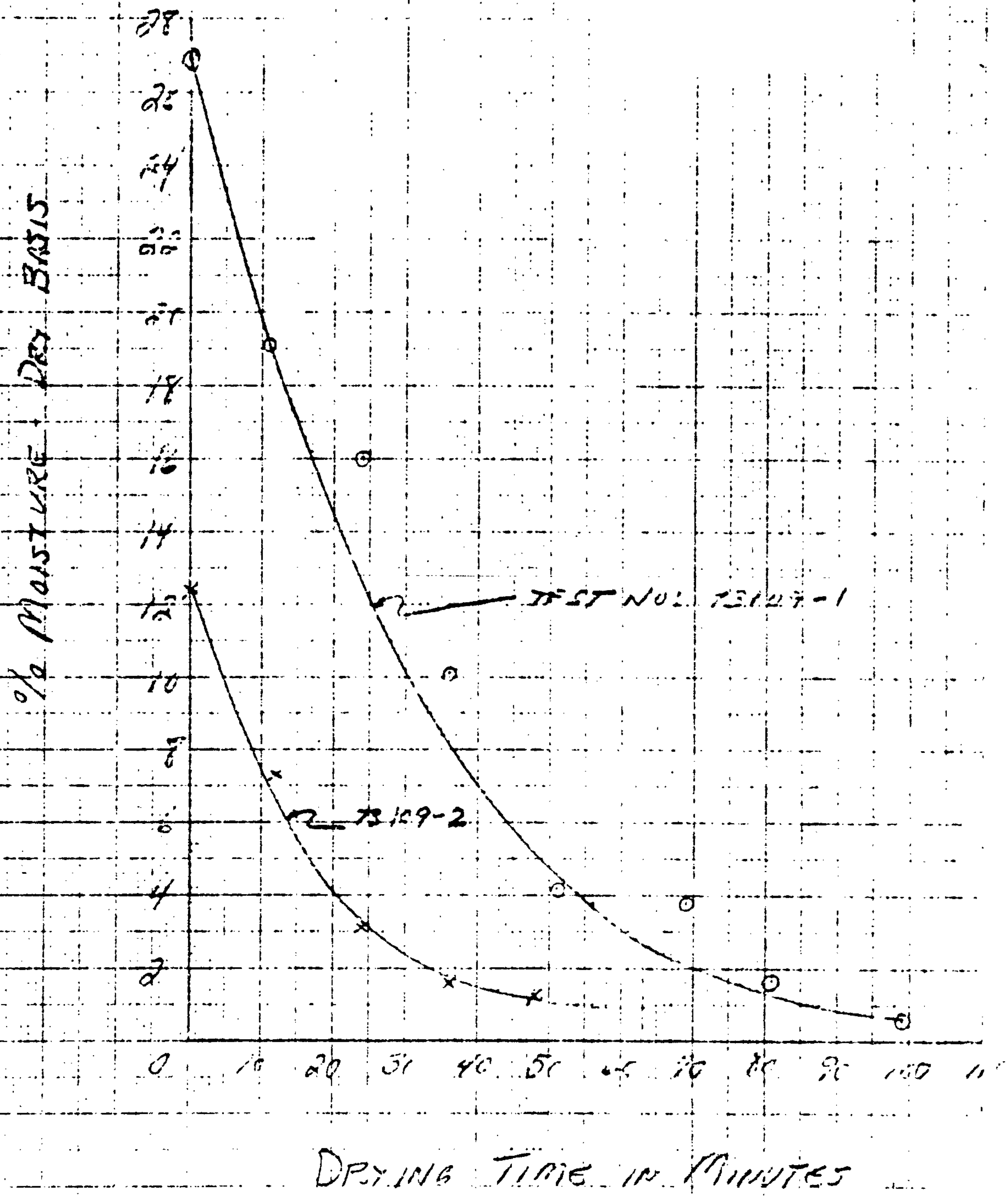
WYSSMONT COMPANY, INC.

J. Jacod

JJ:pbo
Encl: Drying Curve
Temperature Chart
cc: J.Gardner, H.Zack

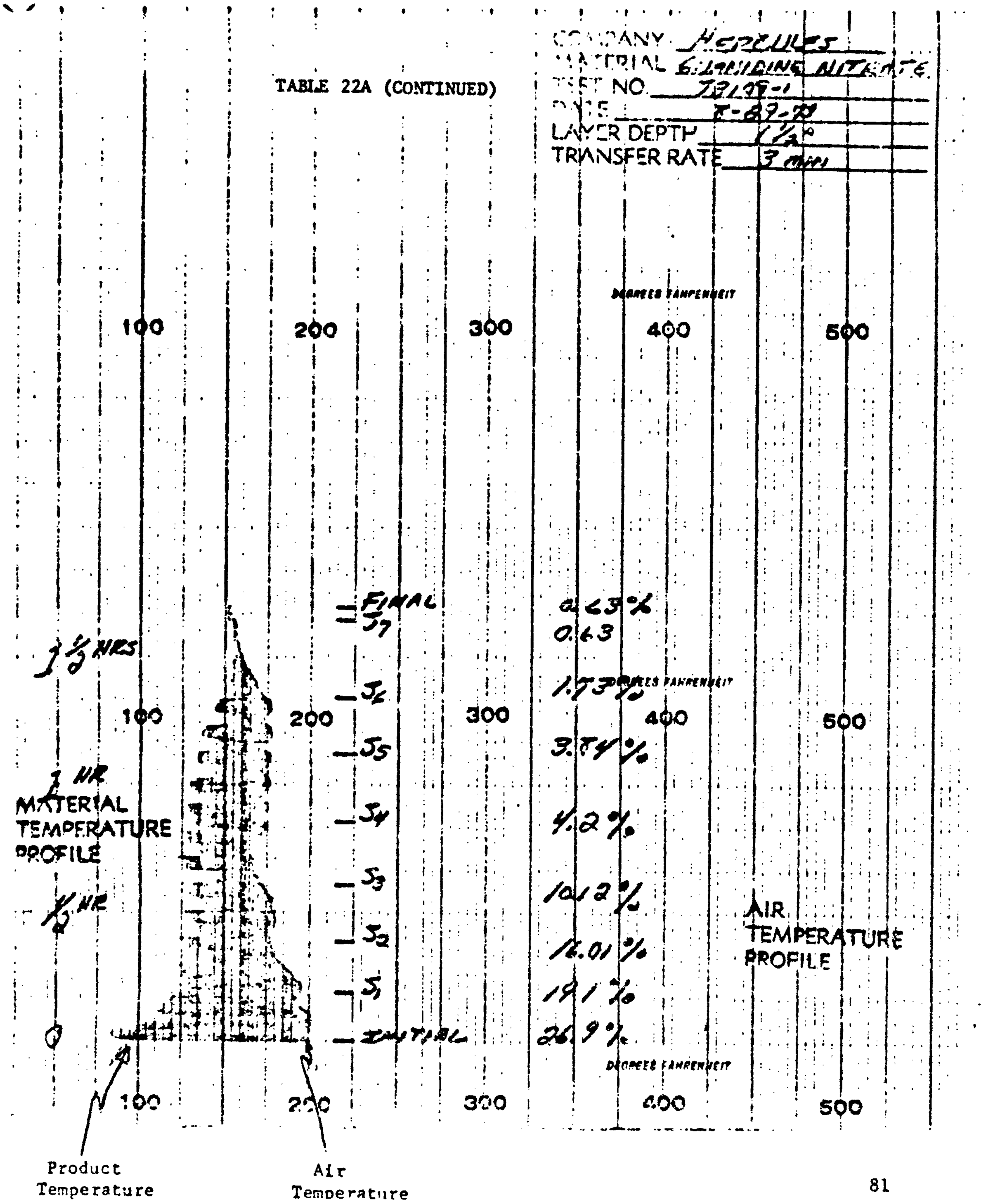
TABLE 22A (CONTINUED)

COMPANY Hercules
MATERIAL Guanidine Nitrate
TEST NO. 73109-1 73109-2
DATE 8-29-73
LAYER DEPTH — 1½" —
TRANSFER RATE — 3 min —



8-30-73

TABLE 22A (CONTINUED)



COMPANY HERCULES

MATERIAL GUANIDINE NITRATE

TEST NO. JBL98-1

DATE 8-29-78

LAYER DEPTH 1 1/2"

TRANSFER RATE 3 min

TABLE 22A (CONTINUED)

COMPANY Hercules
MATERIAL Guanidine Nitrate
TEST NO. 73109-2
DATE 5-29-73
LAYER DEPTH 1 1/2"
TRANSFER RATE 3 min

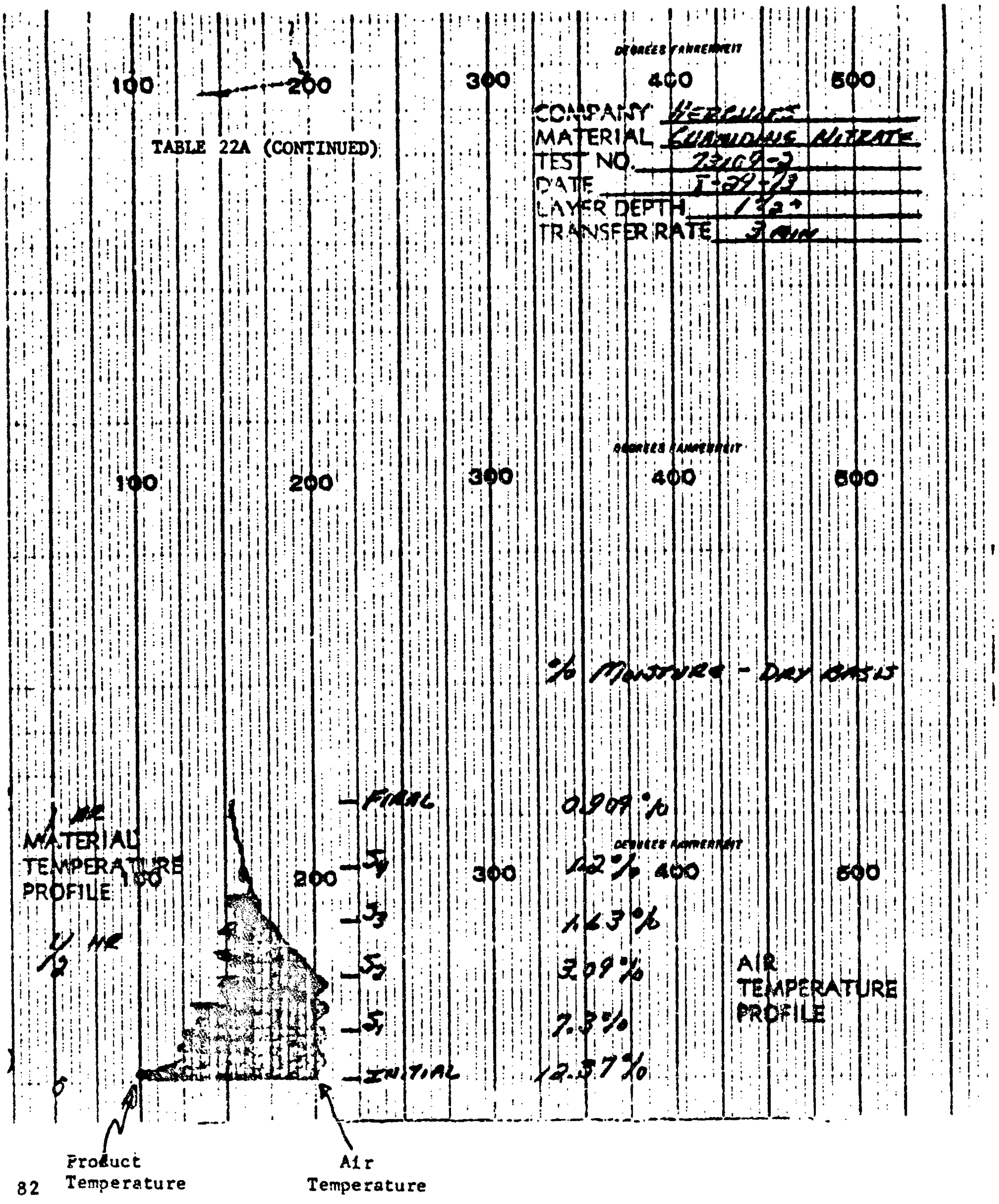


TABLE 22A (CONTINUED)

COMPANY HERCULES
MATERIAL AMMONIUM NITRATE
TEST NO. 73109-3 73109-4
DATE 7-30-73
LAYER DEPTH 1-1⅛" 1⅛"
TRANSFER RATE 3 MIN

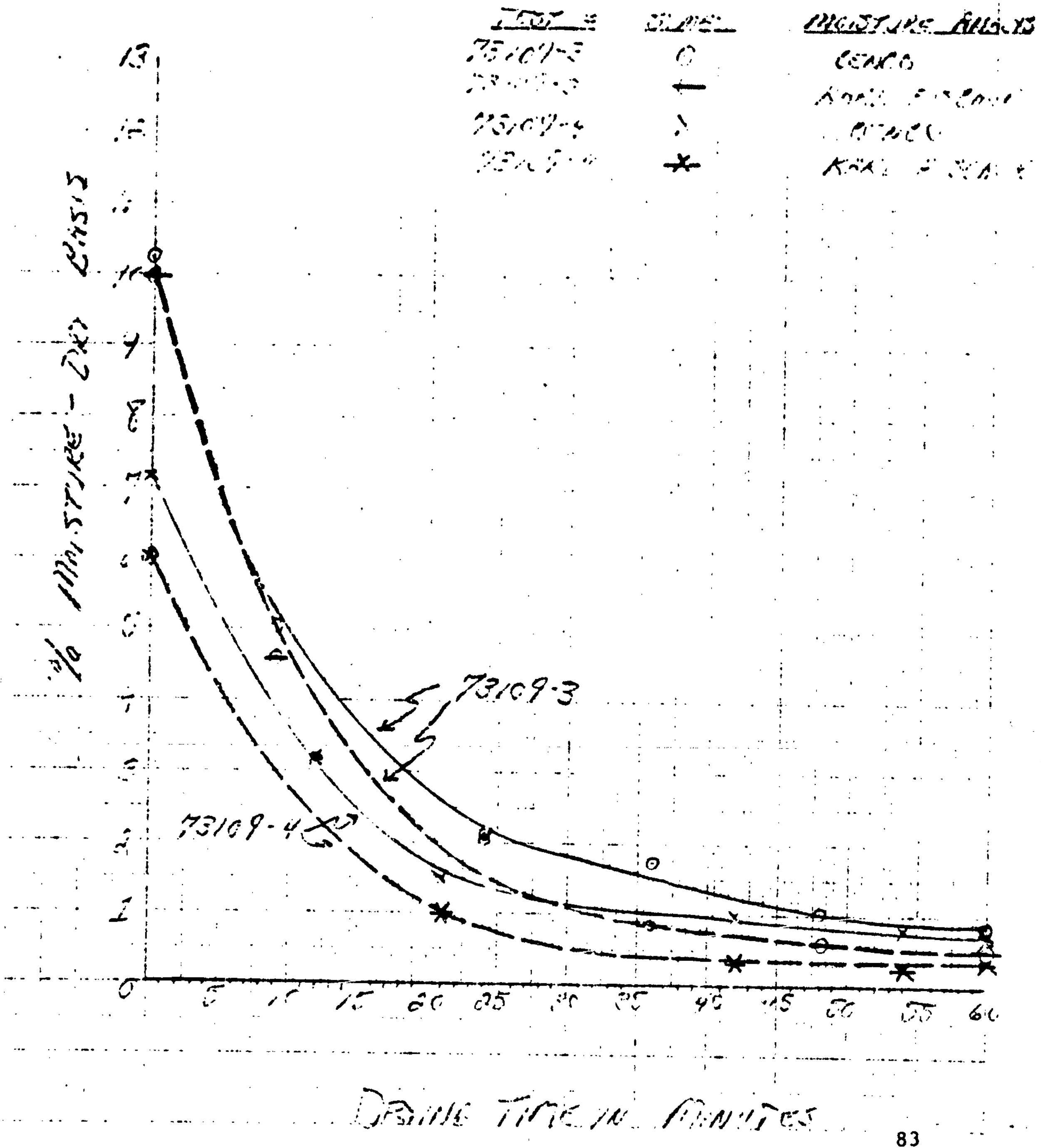
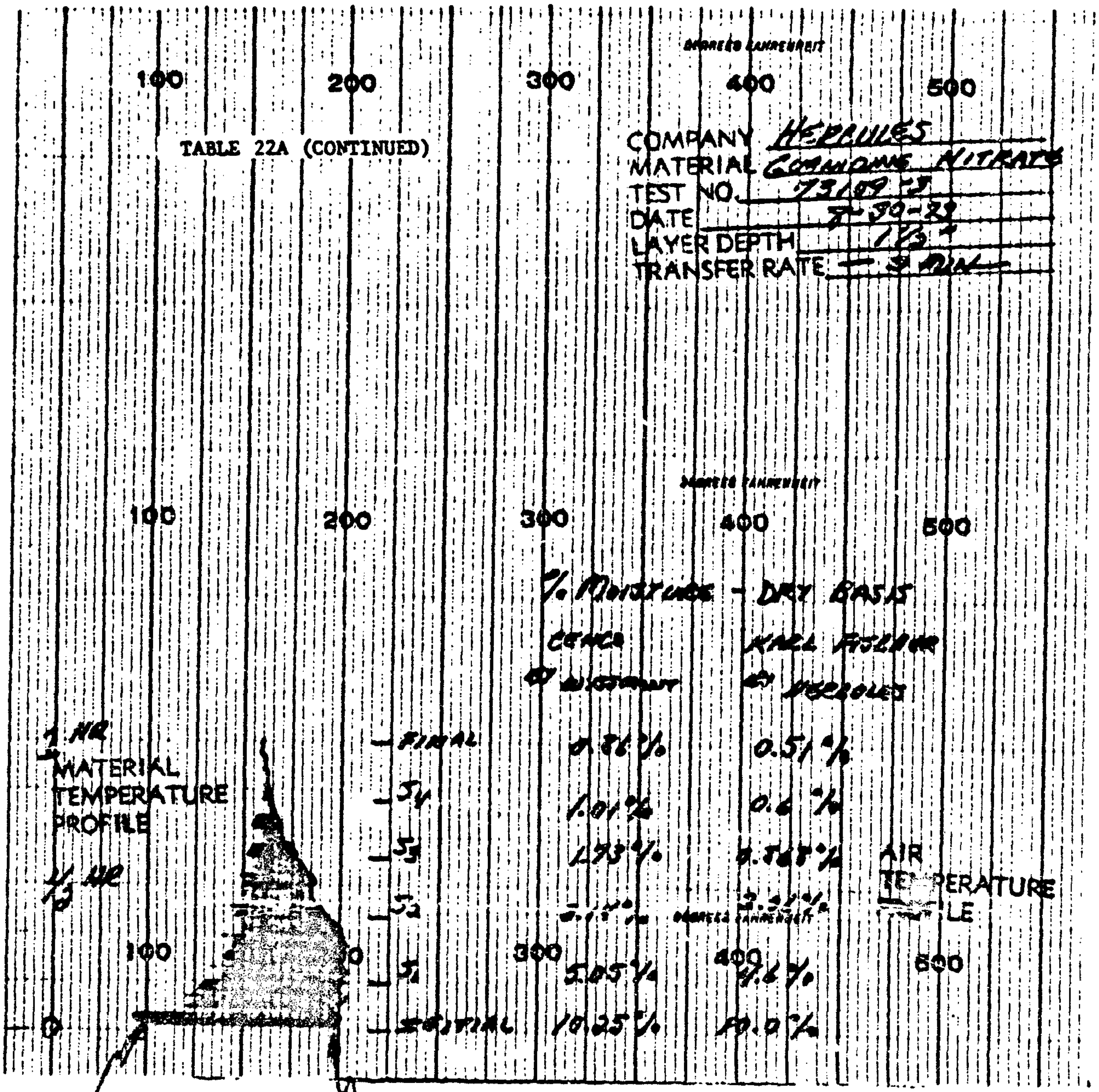


TABLE 22A (CONTINUED)

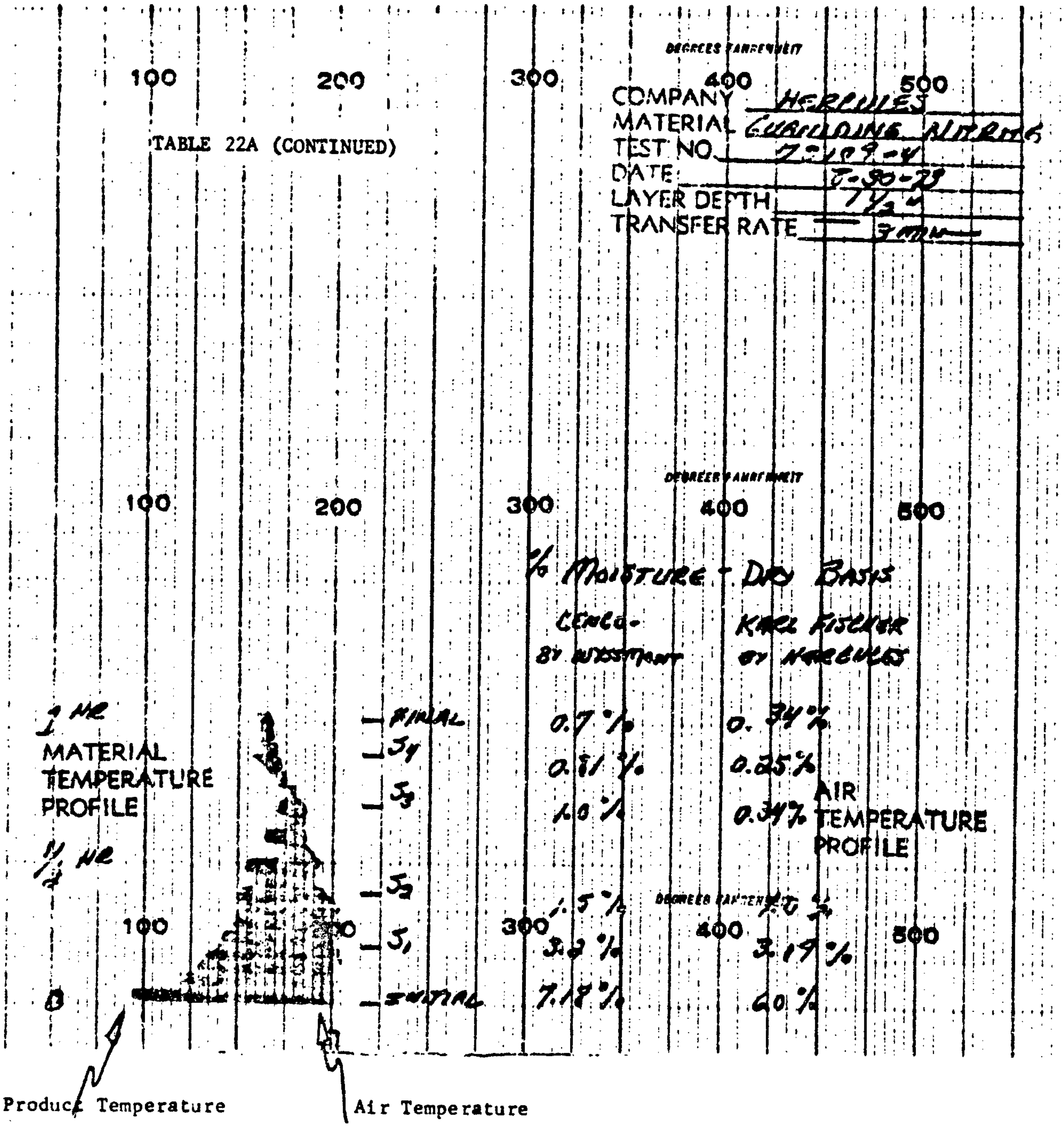
COMPANY HERCULES
MATERIAL GUANIDINE NITRATE
TEST NO. 73109-3
DATE 5-30-73
LAYER DEPTH 1½"
TRANSFER RATE — 3 MIN —



% MOISTURE - DRY BASIS

	CENCO BY [illegible]	KARL FISCHER BY HERCULES
FINAL	0.86%	0.51%
S4	1.01%	0.6%
S3	1.93%	[illegible]
S2	[illegible]	2.31%
S1	5.05%	4.6%
INITIAL	10.25%	10.0%

TABLE 22A (CONTINUED)



COMPANY HERCULES
MATERIAL GUANIDINE NITRATE
TEST NO. 7-109-4
DATE 7-30-73
LAYER DEPTH 1½"
TRANSFER RATE 7 min

% MOISTURE - DRY BASIS

	CALC. BY WYSSMONT	KARL FISCHER BY HERCULES
FINAL	0.7 %	0.34 %
S_4	0.81 %	0.25 %
S_3	1.0 %	0.34 %
S_2	1.5 %	1.0 %
S_1	3.2 %	3.19 %
INITIAL	7.18 %	6.0 %

TABLE 22B

PRELIMINARY PRICE ESTIMATE FOR PRODUCTION GN DRYING SYSTEM

WYSSMONT COMPANY, Inc.

October 9, 1973

Hercules Inc.
Kenvil, New Jersey 07847

Attention: Mr. N. Steele

Re: Drying Guanidine Nitrate in the Wyssmont
Drying System
Our Ref. No. 73109

Gentlemen:

We are pleased to submit our preliminary price estimate to dry Guanidine Nitrate in the Wyssmont drying system.

Our previous operating experience indicates that the TURBO-Dryer will produce a high quality product dried under controlled conditions. The TURBO-Dryer offers many advantages over other types of dryers for this application. The close temperature control feature of the TURBO-Dryer insures that the material is properly dried without overheating or degradation. This is of particular importance for this heat sensitive material. The TURBO-Dryer is being successfully used to dry heat sensitive materials at the temperature required for your application and as low as 95°F.

In the TURBO-Dryer small batches of the material are being processed continuously but separately. The individual tray segment receives a charge of the material and this charge is dried to the desired final moisture specification without mixing with any other material charges. This has proven to be a major advantage for drying calcium hypochlorite material.

The positive retention time feature and internal air recirculation insure that all the material is uniformly dried to the desired final moisture specification.

The internal TURBO-Fans recirculate the air in the TURBO-Dryer over the material for intimate contact between the air and material. As the material transfers from shelf to shelf, the material on each tray is mixed exposing new surfaces to the air stream. The low exhaust air velocity and vertical construction of the TURBO-Dryer keeps any fines carryover to a minimum.

WYSSMONT COMPANY, INC. TABLE 22B (CONTINUED)

Hercules Inc. - 2 - October 9, 1973
Kenvil, NJ. Ref. No. 73109

The TURBO-Dryer operates continuously and automatically without operator attention. Many chemical companies have indicated that the TURBO-Dryer installation resulted in a labor saving of one operator per shift as compared to the batch drying systems and other continuous drying systems previously installed.

The TURBO-Dryer has a successful history of extended periods of operation without shutdown for maintenance.

We are listing the following information for your requirements:

Material	Guanidine Nitrate
Production	4500 lbs/hr.
Initial Moisture	10% (wet basis)
Final Moisture	0.5 - 1%
TURBO-Dryer Size	Q-32
Diameter	15 ft.
Height	23 ft.
Horsepower Requirements:	
TURBO-Fan	7½ HP
Tray	1 HP
Steam required @ 50 psig	2070 lbs/hr.
Exhaust cfm @ 180°F.	4300 cfm
TURBO-Dryer Price *	$240,000. - $250,000.

* The TURBO-Dryer price is based on type 304 stainless steel material of construction for all parts and type 304L stainless steel material of construction for all welded parts (where available).

In addition to the TURBO-Dryer we have included an external heating system consisting of a fresh air fan, a steam heater, a vertical manifold with dampered inlets to introduce the hot drying medium into the dryer at several levels and interconnecting ductwork with temperature control instrumentation. Also included is the direct contact exhaust air scrubber and exhaust fan.

Please note that the TURBO-Dryer will be shipped in subassemblies for erection in the field by bolting only. Approximately 800 manhours are required for erection.

We have not included Class B tooling at this time. We will have to review these specifications as it relates to our design. We have however, included one-piece spun metal trays instead of the standard tray and ring construction for this size TURBO-Dryer.

We have not included any feeding and discharge equipment in this estimate. If you have any questions, please contact us.

Very truly yours,
WYSSMONT COMPANY, INC.

S. H. Shukla

SHS:pbo
cc: Messrs.J.A.Doyle,J.Gardner,
H.Zack, Wilmington,Del.

TABLE 23

SUMMARY OF CALCULATED RESULTS FOR REACTOR MONITORING

Date	Time	Samples	No. of Tubes	AN/U Feed	Y_{urea} %	Productivity lb GN/lb Feed	Production Rate lb GN/Hr	Tube Production Rate lb GN/Hr/Tube	Insoluble lb GN
5/21	11:30 p.m.	1S-1/2S-1	1	2.4	119.6	0.213	5.32	5.32	-
5/22	3:30 a.m.	1S-1/2S-1	1	2.4	116.2	0.192	4.80	4.80	-
5/22	7:30 a.m.	1S-2/2S-2	1	2.2	85.2	0.173	4.32	4.32	-
5/22	3:30 p.m.	1S-4/2S-4	2	2.1	79.5	0.181	9.04	4.52	-
5/22	7:30 p.m.	1S-5/2S-5	2	1.76	84.1	0.184	9.22	4.61	0.44
5/22	11:30 p.m.	1S-6/2S-6	2	2.0	122.8	0.224	11.20	5.60	-
5/23	7:30 a.m.	1S-2/2S-2	2	1.9	105.9	0.218	10.90	5.45	-
5/23	3:00 p.m.	1S-4/2S-4	2	2.02	91.6	0.199	9.96	4.98	0.54
5/23	11:30 p.m.	1S-6/2S-6	2	1.79	83.0	0.204	10.20	5.10	0.38
5/24	7:30 a.m.	1S-2/2S-2	2	2.02	102.2	0.201	10.04	5.02	0.43
5/24	11:30 p.m.	1S-6/2S-6	2	1.52	51.4	0.138	6.90	3.44	0.28
5/25	7:30 p.m.	1S-2/2S-2	2	1.46	99.6	0.233	11.63	5.82	0.78
5/25	11:30 p.m.	1S-6/2S-6	2	1.31	97.2	0.215	10.76	5.38	0.93
5/26	3:30 p.m.	1S-4/2S-4	1	1.46	123.4	0.214	5.35	5.35	1.17
5/27	7:30 a.m.	1S-1-2S-2	1	1.27	95.2	0.165	4.13	4.13	0.85
5/27	11:30 a.m.	1S-3/2S-3	1	1.36	104.3	0.137	3.43	3.43	1.03
5/28	3:30 a.m.	1S-1/2S-1	1	0.84	88.2	0.172	4.31	4.31	0.95
5/28	7:30 a.m.	1S-2/2S-2	1	0.84	83.9	0.157	3.94	3.94	0.65
5/28	11:30 a.m.	1S-3/2S-3	1	0.80	80.5	0.138	3.44	3.44	0.93
5/28	7:30 p.m.	1S-5/2S-5	1	0.86	44.4	0.076	1.91	1.91	0.86
5/29	9:30 a.m.	1S-2/2S-2	1	0.98	51.3	0.121	3.03	3.03	0.98
5/30	7:30 a.m.	1S-2/2S-2	1	3.2	114.7	0.097	2.40	2.40	1.1
5/30	10:30 a.m.	1S-3/	1	2.9	-	-	-	-	-
5/30	11:30 p.m.	1S-6/2S-6	1	1.9	84.4	0.125	3.12	3.12	0.44
5/31	3:30 a.m.	1S-1/2S-1	1	1.62	90.2	0.167	4.17	4.17	0.82
5/31	7:30 a.m.	1S-2/2S-2	1	1.48	88.4	0.144	3.60	3.60	1.14
5/31	11:30 p.m.	1S-6/2S-6	1	1.25	146.3	0.194	4.86	4.86	0.49
5/31	3:30 p.m.	1S-4/2S-4	1	1.60	141	0.270	7.18	7.18	1.29

TABLE 23 (CONT.)

Date	Time	Samples	No. of Tubes	AN/U Feed	Y_{urea} %	Productivity lb GN/lb Feed	Production Rate lb GN/Hr	Tube Production Rate lb GN/Hr/Tube	Insoluble lb GN
6/1	7:00 a.m.	1S-2/2S-2	2	1.60	89.4	0.170	8.50	4.25	0.81
6/1	7:30 p.m.	1S-5/2S-5	3	1.71	110.5	0.210	15.72	5.24	0.48
6/2	3:30 p.m.	1S-4/2S-4	3	1.61	104.6	0.194	14.55	4.85	0.59
6/3	7:30 a.m.	1S-2/2S-2	3	1.53	111.8	0.220	16.53	5.51	0.57
6/3	3:30 p.m.	1S-4/2S-4	3	1.49	94.1	0.183	13.71	4.57	0.55
6/3	11:30 p.m.	1S-6/2S-6	3	2.46	160.0	0.213	16.02	5.34	0.34
6/4	7:30 a.m.	1S-2/2S-2	3	2.6	107.6	0.107	8.01	2.67	0.41
6/4	7:30 p.m.	1S-5/2S-5	3	1.97	83.6	0.160	12.0	4.0	0.32
6/5	3:30 a.m.	1S-1/2S-1	3	1.4	71.8	0.158	11.88	3.96	0.54
6/5	7:30 a.m.	1S-2/2S-2	3	1.57	96.8	0.188	14.13	4.71	0.47
6/5	3:30 p.m.	1S-4/2S-4	3	1.92	99.4	0.173	12.96	4.32	0.50
6/5	7:30 p.m.	1S-5/2S-5	3	2.08	104	0.184	13.80	4.60	0.50
6/5	11:30 p.m.	1S-6/2S-6	4	1.96	91.4	0.178	18.40	4.46	0.46
6/6	3:30 p.m.	1S-1/2S-1	4	1.93	145.1	0.298	29.76	7.44	0.92
6/7	3:30 p.m.	1S-4/2S-4	4	2.0	95.8	0.187	18.68	4.67	0.27
6/7	7:30 p.m.	1S-5/2S-5	4	2.14	96.2	0.180	18.04	4.51	0.29
6/7	11:30 p.m.	1S-6/2S-6	4	1.97	103.2	0.181	18.12	4.53	0.58
6/8	3:30 p.m.	1S-1/2S-1	4	1.77	88.0	0.178	17.80	4.45	0.33
6/8	7:30 a.m.	1S-2/2S-2	4	1.89	113.8	0.168	16.80	4.2	0.30
6/9	11:30 a.m.	1S-3/2S-3	4	1.81	137.4	0.187	18.72	4.68	-
6/10	7:30 a.m.	1S-2/2S-2	4	1.57	73.9	0.130	13.12	3.28	-
6/11	7:30 a.m.	1S-2/2S-2	4	1.10	86.1	0.126	12.60	3.15	-
6/11	4:00 p.m.	1S-3/2S-3	4	1.50	164.7	0.368	36.84	4.21	0.48
6/11	7:30 p.m.	1S-5/2S-4	4	1.36	93.4	0.234	23.44	5.86	0.79
6/11	11:30 p.m.	1S-6/2S-5	6	1.61	115.4	0.206	30.90	5.15	1.50
6/12	3:30 a.m.	1S-1/2S-1	6	1.44	122	0.306	45.9	7.65	0.32
6/12	7:30 a.m.	1S-2/2S-2	6	1.42	116.2	0.265	39.66	6.61	0.93
6/12	3:30 p.m.	1S-4/2S-4	6	1.56	79.8	0.133	19.86	3.31	1.15
6/12	11:30 p.m.	1S-6/2S-6	6	1.25	124.6	0.239	35.94	5.99	1.15
6/13	7:30 a.m.	1S-2/2S-2	6	1.36	107.6	0.204	30.54	5.09	1.0
6/13	3:30 p.m.	1S-4/2S-4	6	1.39	99.3	0.179	26.88	4.48	0.72
6/13	11:30 p.m.	1S-6/2S-6	6	1.31	85.9	0.156	23.46	3.91	0.83
6/14	7:30 a.m.	1S-2/2S-2	6	1.43	103.8	0.161	24.18	4.03	1.13
6/14	11:30 a.m.	1S-3/2S-3	6	1.11	81.6	0.179	26.88	4.47	0.88

TABLE 23 (CONT.)

Date	Time	Samples	No. of Tubes	AN/U Feed	Y_{urea} %	Productivity lb GN/lb Feed	Production Rate lb GN/Hr	Tube Production Rate lb GN/Hr/Tube	Insoluble lb GN
6/14	7:30 p.m.	1S-5/2S-5	6	1.27	90.9	0.184	27.54	4.59	0.98
6/15	3:30 a.m.	1S-1/2S-1	6	1.20	94.2	0.202	30.24	5.04	1.06
6/16	7:30 a.m.	1S-2/2S-2	7	1.38	140.0	0.415	58.38	8.34	1.02
6/17	7:30 a.m.	1S-2/2S-2	7	1.33	123.7	0.227	31.78	4.54	0.59
6/17	3:30 a.m.	1S-4/2S-4	7	1.28	111.4	0.222	31.08	4.44	0.74
6/17	11:30 p.m.	1S-6/2S-6	7	1.11	84.2	0.217	30.31	4.33	0.74
6/18	7:30 a.m.	1S-2/2S-2	7	1.2	106.4	0.262	36.61	5.23	0.67
6/18	11:30 a.m.	1S-3/2S-3	7	1.34	107.8	0.216	33.25	4.75	0.77
6/18	7:30 p.m.	1S-4/2S-4	7	1.45	113.8	0.252	38.85	5.55	0.66
6/19	8:30 a.m.	1S-1/2S-1	7	1.31	87.6	0.224	34.58	4.94	0.67
6/19	3:30 p.m.	1S-4/2S-4	7	1.83	111.6	0.192	29.61	4.23	0.57
6/20	7:30 a.m.	1S-2/2S-2	7	1.86	115.4	0.235	36.26	5.10	0.41
6/20	11:30 p.m.	1S-6/2S-6	7	1.58	98.8	0.210	32.34	4.62	0.45
6/21	3:30 a.m.	1S-1/2S-1	7	1.62	103.7	0.233	35.91	5.13	0.83
6/21	7:30 a.m.	1S-2/2S-2	8	1.65	103.2	0.235	41.36	5.17	0.56
6/21	11:30 p.m.	1S-6/2S-6	8	1.62	106.6	0.260	45.76	5.72	-
6/22	7:30 a.m.	1S-2/2S-2	8	1.64	106.8	0.189	33.28	4.16	-
6/22	11:30 p.m.	1S-6/2S-6	8	1.33	83.8	0.202	30.17	4.43	0.63
6/23	7:30 a.m.	1S-2/2S-2	8	1.47	112.8	0.255	44.88	5.61	0.63
6/23	11:30 a.m.	1S-6/2S-6	8	1.66	108.6	0.214	37.76	4.72	0.47
6/24	7:30 a.m.	1S-2/2S-2	8	1.60	105.0	0.209	36.80	4.60	0.64
6/25	7:30 a.m.	1S-2/2S-2	8	1.57	113.8	0.231	40.56	5.07	0.47
6/25	11:30 p.m.	1S-6/2S-6	8	1.28	66.6	0.183	32.24	4.03	0.66
6/26	7:30 p.m.	1S-2/2S-2	8	2.16	165	0.304	53.52	6.69	1.03
6/26	11:30 p.m.	1S-6/2S-6	8	1.27	147	0.229	39.20	4.90	0.87
6/27	7:30 a.m.	1S-2/2S-2	8	1.27	124	0.244	39.04	4.88	1.2
6/28	11:30 p.m.	1S-6/2S-6	6	1.23	115.3	0.231	27.66	4.61	1.3
6/29	3:30 a.m.	1S-1/2S-1	6	1.01	85.5	0.221	26.46	4.41	1.36
6/29	7:30 a.m.	1S-2/2S-2	6	1.11	80.0	0.158	18.9	3.15	2.57
7/1	7:30 a.m.	1S-2/2S-2	3	1.47	30.6	0.098	5.85	1.95	-
7/1	3:30 p.m.	1S-4/2S-4	3	1.03	65.8	0.097	5.82	1.94	1.79
7/6	11:30 a.m.	1S-3/2S-3	8	0.97	79.4	0.217	43.44	5.43	1.57
7/6	11:30 p.m.	1S-6/2S-6	8	1.08	112.6	0.197	34.64	4.33	1.01

TABLE 23 (CONT.)

Date	Time	Samples	No. of Tubes	AN/U Feed	Y_{urea} %	Productivity lb GN/lb Feed	Production Rate lb GN/Hr	Tube Production Rate lb GN/Hr/Tube	Insoluble lb GN
7/7	7:30 a.m.	1S-2/2S-2	7	0.94	113.2	0.203	31.22	4.46	1.21
7/8	11:30 p.m.	1S-6/2S-6	7	2.12	75.6	0.202	15.68	2.24	1.31
7/9	3:30 a.m.	1S-1/2S-1	7	1.21	89.4	0.125	19.32	2.76	1.54
7/9	7:30 a.m.	1S-2/2S-2	7	1.35	161.0	0.198	30.45	4.35	1.20
7/9	11:30 p.m.	1S-6/2S-6	7	1.10	128	0.219	33.74	4.82	0.82
7/10	7:30 a.m.	1S-2/2S-2	7	1.10	128	0.219	33.74	4.82	0.82
7/10	5:30 p.m.	1S-5/2S-5	7	1.21	125.2	0.196	30.17	4.31	1.39
7/11	7:30 a.m.	1S-2/2S-2	7	1.12	125	0.169	26.04	3.72	1.76
7/12	3:30 a.m.	1S-1/2S-1	6	0.72	94.8	0.149	19.68	3.28	1.97
7/12	3:30 p.m.	1S-4/2S-4	6	0.87	70.9	0.182	24.06	4.01	1.83
7/31	1:30 p.m.	-	3	1.49	66.3	0.120	7.92	2.64	1.66
7/31	3:30 p.m.	-	3	1.44	78.8	0.161	10.62	3.54	1.07
7/31	7:30 p.m.	-	3	1.32	81.4	0.188	12.42	4.14	1.97
7/31	11:30 p.m.	-	3	1.13	79.2	0.192	12.66	4.22	2.16
8/1	3:30 a.m.	1S-1/2S-1	3	0.98	73.6	0.198	13.05	4.35	1.83
8/1	7:30 a.m.	1S-2/2S-2	3	1.01	115	0.264	17.40	5.80	2.03
8/2	3:30 a.m.	1S-1/2S-1	3	2.57	101.6	0.150	9.90	3.30	0.86
8/1	3:30 p.m.	1S-4/2S-4	3	1.36	200	0.335	22.17	7.39	2.09
8/2	8:00 a.m.	1S-2/2S-2	3	2.36	141	0.192	12.69	4.23	0.85
8/2	3:30 p.m.	1S-4/2S-4	3	1.53	87	0.182	13.80	4.60	-
8/2	7:30 p.m.	1S-5/2S-5	3	1.51	105.2	0.208	13.71	4.57	0.72
8/3	3:30 a.m.	1S-1/2S-1	3	1.62	111.8	0.189	12.48	4.16	1.15
8/3	7:30 a.m.	1S-2/2S-2	3	1.61	120.2	0.135	8.91	2.97	1.60
8/4	11:30 a.m.	1S-3/2S-3	3	2.07	116.2	0.136	9.33	3.11	1.02
8/5	3:30 p.m.	1S-4/2S-4	3	1.95	113.6	0.129	10.29	3.43	0.98
8/6	7:30 p.m.	1S-5/2S-5	3	1.73	89.4	0.132	10.86	3.62	1.16
8/7	7:30 a.m.	1S-2/2S-2	3	1.42	139.8	0.204	16.29	5.43	1.24
8/7	11:30 p.m.	1S-3/2S-3	3	1.64	119.2	0.194	15.48	5.16	1.08
8/7	7:30 p.m.	1S-5/2S-5	3	2.08	93.6	0.128	10.20	3.40	0.92
8/7	11:30 p.m.	1S-6/2S-6	3	1.83	79.4	0.127	10.14	3.38	0.83
8/8	7:30 p.m.	1S-2/2S-2	3	1.82	100.4	0.162	10.53	3.51	1.21
8/8	11:30 a.m.	1S-3/2S-3	3	1.94	101.8	0.151	10.84	3.28	1.29
8/8	7:30 a.m.	1S-5/2S-5	2	2.49	161.2	0.153	10.96	3.32	2.69
8/9	7:30 a.m.	1S-2/2S-2	2	2.89	111.2	0.126	5.02	2.51	2.89
8/9	11:00 p.m.	1S-6/2S-6	2	1.86	87.2	0.175	6.96	3.48	1.86
8/10	7:30 a.m.	1S-2/2S-2	2	1.58	79.4	0.177	7.04	3.52	1.58
8/10	9:00 a.m.	1S-3/2S-3	2	1.48	74.4	0.194	7.72	3.86	1.46

TABLE 24

RESULTS OF REACTOR FEED ANALYSIS

Date	Closure	Time	Reactor Feed Analyses (%) U	AN	GN	H_2O	Insol.	Comments
5/21	100.79	11:30 p.m.	24.0	76.2	0	0.59	-	AN_m/U_m - 2.4/1
5/22	98.11	3:30 a.m.	22.9	74.5	0	0.71	-	2.4/1
5/22	102.59	7:30 a.m.	25.8	74.3	1.8	0.69	-	2.2/1
5/22	99.11	7:30 p.m.	29.3	68.7	0.5	0.61	-	1.76/1
5/23	99.24	7:30 a.m.	27.6	70.1	1.1	0.44	-	1.9/1
5/23	105.26	3:00 p.m.	28.1	75.8	0.8	0.56	-	2.02/1
5/23	98.15	11:30 p.m.	28.8	68.6	0	0.75	-	1.79/1
5/24	98.83	7:30 a.m.	26.5	71.6	0	0.73	-	2.02/1
5/24	98.47	11:30 p.m.	32.1	64.9	0	1.47	-	1.52/1
5/25	97.32	7:30 a.m.	31.6	61.6	3.4	0.72	-	1.46/1
5/25	97.20	11:30 p.m.	33.7	59.0	3.9	0.60	-	1.31/1
5/26	97.44	3:30 a.m.	31.6	61.6	3.8	0.44	-	1.46/1
5/27	96.85	7:30 a.m.	34.4	58.0	3.8	0.65	-	1.27/1
5/27	97.13	11:30 a.m.	33.3	60.1	3.0	0.73	-	1.36/1
5/28	97.10	3:30 a.m.	44.3	49.8	1.6	1.4	-	0.84/1
5/28	98.30	7:30 a.m.	44.1	49.6	3.0	1.6	-	0.84/1
5/28	100.60	11:30 a.m.	44.7	47.9	5.5	2.5	-	0.8/1
5/28	99.60	7:30 p.m.	44.6	42.1	8.4	4.5	-	0.71/1
5/29	97.27	9:30 a.m.	44.9	49.4	2.8	0.17	-	0.66/1
5/29	95.90	11:30 p.m.	41.8	51.4	1.5	1.2	-	0.92/1
5/30	98.01	7:30 a.m.	16.9	71.6	9.0	0.51	-	3.2/1
5/30	96.20	10:30 a.m.	17.9	69.4	8.9	-	-	2.91/1
5/30	95.83	11:30 p.m.	24.0	61.2	9.9	0.73	-	1.91/1
5/31	97.60	3:30 a.m.	27.2	58.8	10.2	1.4	-	1.62/1
5/31	95.33	7:30 a.m.	28.7	56.4	9.4	0.83	-	1.48/1
5/31	97.96	3:30 p.m.	33.3	55.4	8.4	0.86	-	1.25/1
5/31	99.00	11:30 p.m.	23.2	65.8	9.4	0.6	-	2.13/1
6/1	101.39	7:00 a.m.	28.2	60.2	12.3	0.69	-	1.60/1
6/1	99.67	8:00 a.m.	16.3	70.5	12.1	0.77	-	3.24/1
6/1	97.15	7:30 p.m.	26.7	60.8	9.0	0.63	-	1.71/1
6/2	97.36	3:30 p.m.	26.1	60.4	8.0	0.46	-	1.61/1
6/3	96.54	7:30 a.m.	28.5	58.1	8.9	1.04	-	1.53/1
6/3	97.10	3:30 p.m.	30.2	59.9	8.6	1.17	-	1.49/1
6/3	99.12	11:30 p.m.	21.0	68.9	8.5	0.72	-	2.46/1
6/4	99.85	7:30 a.m.	19.8	69.6	9.8	0.65	-	2.6/1
6/4	102.17	7:30 p.m.	23.6	62.0	15.6	0.97	-	1.97/1
6/5	102.73	3:30 a.m.	29.2	54.7	16.1	2.73	-	1.4/1
6/5	101.05	7:30 a.m.	27.8	58.1	13.8	1.35	-	1.57/1
6/5	103.60	3:30 p.m.	24.3	62.2	15.3	1.8	-	1.92/1
6/5	101.70	7:30 p.m.	23.9	66.1	10.2	1.5	-	2.08/1
6/5	103.70	11:30 p.m.	25.5	66.6	10.5	1.1	-	1.96/1
6/6	100.00	3:30 a.m.	25.4	65.3	8.3	1.6	-	1.93/1
6/7	-	11:30 a.m.	27.2	-	-	-	-	-
6/7	101.80	3:30 p.m.	23.8	63.4	13.0	1.86	-	2.0/1
6/7	100.62	7:30 p.m.	22.8	65.0	10.8	2.02	-	2.14/1
6/7	96.92	11:30 p.m.	23.7	62.3	9.0	1.92	-	1.97/1

TABLE 24 (CONT.)

Date	Closure	Time	Reactor Feed Analyses (%)					Comments
			U	AN	GN	H_2O	Insol.	
6/8	99.28	3:30 a.m.	27.1	64.0	7.0	1.18	-	AN_m/U_m 1.77/1
6/8	100.50	7:30 a.m.	25.4	63.8	9.6	1.7	-	1.89/1
6/9	103.60	11:30 a.m.	27.6	66.4	9.6	-	-	1.81/1
6/10	97.20	7:30 a.m.	29.6	62.0	4.7	0.9	-	1.57/1
6/11	99.20	7:30 a.m.	37.6	55.6	5.4	0.6	-	1.10/1
6/11	101.30	4:00 p.m.	30.8	61.5	8.8	1.0	-	1.50/1
6/11	101.99	7:30 p.m.	33.9	61.6	4.5	1.99	-	1.36/1
6/11	101.0	11:30 p.m.	29.8	63.9	6.4	0.9	-	1.61/1
6/12	101.6	3:30 a.m.	32.4	62.3	3.1	3.8	-	1.44/1
6/12	100.0	7:30 a.m.	33.8	64.1	0.6	1.5	-	1.42/1
6/12	103.5	3:30 p.m.	28.8	60.1	13.5	1.1	-	1.56/1
6/12	104.3	11:30 p.m.	34.2	57.0	8.8	4.3	-	1.25/1
6/13	101.7	7:30 a.m.	31.6	57.3	9.5	3.3	-	1.36/1
6/13	97.8	3:30 p.m.	30.4	56.3	8.9	2.2	-	1.39/1
6/13	99.7	1:30 p.m.	33.1	58.0	7.0	1.6	-	1.31/1
6/14	99.8	7:30 a.m.	30.9	58.9	8.4	1.6	-	1.43/1
6/14	99.7	11:30 a.m.	37.1	54.6	7.0	1.0	-	1.11/1
6/14	101.8	7:30 p.m.	34.5	58.3	5.3	3.7	-	1.27/1
6/15	98.9	3:30 a.m.	36.2	57.5	4.1	1.1	-	1.20/1
6/16	96.2	7:30 a.m.	30.9	57.0	8.3	-	-	1.38/1
6/17	97.0	7:30 a.m.	32.6	57.7	6.7	-	-	1.33/1
6/17	98.91	3:30 a.m.	34.2	58.4	5.4	0.91	-	1.28/1
6/17	99.0	11:30 p.m.	36.7	54.2	7.2	0.90	-	1.11/1
6/18	99.53	7:30 a.m.	35.5	56.7	6.6	0.73	-	1.20/1
6/18	100.54	11:30 a.m.	32.5	58.0	9.3	0.74	-	1.34/1
6/18	97.76	7:30 p.m.	29.7	57.3	10.0	0.76	-	1.45/1
6/19	101.07	3:30 a.m.	31.9	55.8	12.0	1.37	-	1.31/1
6/19	99.05	3:30 p.m.	24.9	60.6	11.9	1.65	-	1.83/1
6/20	98.27	7:30 a.m.	26.1	64.8	6.2	1.17	-	1.86/1
6/20	99.5	11:30 p.m.	30.0	63.1	5.1	1.30	-	1.58/1
6/21	99.49	3:30 a.m.	29.1	63.0	6.6	0.79	-	1.62/1
6/21	98.48	7:30 a.m.	28.9	63.6	6.6	0.58	-	1.65/1
6/21	97.5	11:30 p.m.	30.1	64.9	2.5	-	-	1.62/1
6/22	99.57	7:30 a.m.	27.5	60.2	11.4	0.67	-	1.64/1
6/22	99.85	11:30 p.m.	32.7	58.0	8.4	0.75	-	1.33/1
6/23	97.0	7:30 a.m.	30.5	59.9	5.3	1.30	-	1.47/1
6/23	98.23	11:30 p.m.	27.5	61.9	7.8	1.03	-	1.66/1
6/24	96.98	7:30 a.m.	28.7	60.4	7.0	0.88	-	1.60/1
6/25	98.23	7:30 a.m.	29.4	61.6	6.4	0.83	-	1.57/1
6/25	96.71	11:30 p.m.	32.6	55.4	6.7	2.01	-	1.28/1
6/26	97.95	7:30 a.m.	30.7	66.4	0	0.85	-	2.16/1
6/26	99.57	11:30 p.m.	32.4	59.7	6.6	0.87	-	1.26/1
6/27	98.8	7:30 a.m.	33.3	56.4	6.3	2.80	-	1.27/1
6/28	99.14	11:30 p.m.	33.2	55.1	8.9	1.94	-	1.23/1
6/29	95.44	3:30 a.m.	37.3	50.5	6.0	1.64	-	1.01/1
6/29	117.4	7:30 a.m.	55.3	52.3	8.8	1.0	-	1.11/1

TABLE 24 (CONT.)

Date	Closure	Time	Reactor Feed Analyses (%)					Comments
			U	AN	GN	H_2O	Insol.	
7/1	99.45	7:30 a.m.	30.01	58.63	10.18	0.63	-	AN_m/U_m 1.47/1
7/1	97.97	3:30 p.m.	38.2	52.7	5.5	1.57	-	1.03/1
7/6	98.99	11:30 a.m.	41.3	53.1	3.0	1.59	-	0.97/1
7/6	104.93	11:30 p.m.	37.5	54.1	9.1	4.23	-	1.08/1
7/7	101.59	7:30 a.m.	41.1	51.4	6.3	2.79	-	0.94/1
7/8	95.82	11:30 p.m.	20.5	63.6	7.0	4.72	-	2.12/1
7/9	95.86	3:30 a.m.	35.9	57.5	0	2.46	-	1.21/1
7/9	93.16	7:30 a.m.	32.3	57.9	0	2.96	-	1.35/1
7/10	97.41	7:30 a.m.	36.4	53.6	5.6	1.81	-	1.10/1
7/10	96.74	5:30 p.m.	34.4	55.6	4.8	1.94	-	1.21/1
7/11	97.43	7:30 a.m.	36.7	54.6	4.4	1.73	-	1.12/1
7/12	98.52	3.30 a.m.	47.2	45.6	4.4	1.32	-	0.72/1
7/12	99.8	3:30 p.m.	42.1	48.9	7.2	1.6	-	0.87/1
7/31	98.7	1:30 p.m.	32.1	63.9	2.1	0.6	-	1.49/1
7/31	100.19	3:30 p.m.	32.6	62.5	4.1	1.0	-	1.44/1
7/31	99.78	7:30 p.m.	35.0	61.6	2.48	0.7	-	1.32/1
7/31	96.41	11:30 p.m.	37.8	57.2	0	1.41	-	1.13/1
8/1	100.37	3:30 a.m.	42.3	55.2	1.93	0.94	-	0.98/1
8/1	99.37	7:30 a.m.	41.9	56.3	0.55	0.62	-	1.01/1
8/1	96.75	3:30 p.m.	33.0	59.8	3.1	0.85	-	2.57/1
8/2	98.86	3:30 a.m.	20.0	67.8	10.2	0.86	-	1.36/1
8/2	99.13	8:00 a.m.	22.1	69.7	6.7	0.63	-	2.36/1
8/2	97.60	3:30 p.m.	29.1	59.2	7.4	1.9	-	1.53/1
8/2	98.00	7:30 p.m.	29.2	58.6	9.0	1.2	-	1.51/1
8/3	99.34	3:00 a.m.	27.8	60.1	10.1	1.34	-	1.62/1
8/3	92.32	7:30 a.m.	25.7	55.4	9.9	1.32	-	1.61/1
8/4	97.96	11:30 a.m.	24.0	66.1	7.0	0.86	-	2.07/1
8/5	99.57	3:30 p.m.	24.4	63.6	9.8	1.77	-	1.95/1
8/6	99.7	7:30 p.m.	27.2	62.6	6.1	3.8	-	1.73/1
8/7	96.77	7:30 a.m.	31.2	59.2	5.0	1.37	-	1.42/1
8/7	93.69	11:30 a.m.	28.2	61.6	2.8	1.09	-	1.64/1
8/7	99.29	7:30 p.m.	23.7	65.8	8.12	1.67	-	2.08/1
8/7	96.68	11:30 p.m.	26.0	63.5	5.1	2.08	-	1.83/1
8/8	98.94	7:30 a.m.	26.5	64.5	7.18	0.76	-	1.82/1
8/8	98.57	11:30 a.m.	25.4	65.7	6.6	0.87	-	1.94/1
8/8	97.19	7:30 p.m.	21.3	70.7	4.12	1.07	-	2.49/1
8/9	98.32	7:30 a.m.	18.7	72.1	6.10	1.42	-	2.89/1
8/9	100.2	11:00 p.m.	27.3	67.8	4.3	1.8	-	1.86/1
8/10	99.4	7:30 a.m.	30.4	64.1	2.5	2.4	-	1.58/1
8/10	99.9	9:00 a.m.	32.4	63.8	2.3	1.4	-	1.48/1
8/10	100.2	10:00 a.m.	32.0	64.6	2.4	1.2	-	1.52/1
8/10	102.6	12:00 Noon	31.9	64.7	4.8	1.2	-	1.52/1
8/10	98.9	1:00 p.m.	32.8	64.2	0.3	1.6	-	1.47/1

TABLE 25

REACTOR MELT ANALYSES

DATE	CLOSURE	TIME	U (%)	AN (%)	GN (%)	H_2O (%)	INSOL (%)
5/21	99.8	11:30 p.m.	6.2	69.8	23.8	-	-
5/22	101.2	3:30 a.m.	7.7	71.4	22.1	-	-
5/22	100.0	7:30 a.m.	6.7	71.7	21.6	-	-
5/22	99.7	3:30 p.m.	5.9	72.3	21.5	-	-
5/22	99.0	7:30 p.m.	9.2	67.1	22.6	-	0.10
5/22	99.1	11:30 p.m.	10.3	64.0	24.8	-	-
5/23	99.9	7:30 a.m.	8.9	64.9	26.1	-	-
5/23	99.02	3:00 p.m.	9.2	67.4	22.3	-	0.12
5/23	98.79	11:30 p.m.	9.5	65.2	24.0	-	0.09
5/24	98.31	7:30 a.m.	8.21	66.9	23.1	-	0.10
5/24	96.45	11:30 p.m.	7.4	71.4	17.6	-	0.05
5/25	98.45	7:30 a.m.	10.4	55.8	32.0	-	0.25
5/25	97.28	11:30 p.m.	14.1	52.9	30.0	-	0.28
5/26	98.23	3:30 p.m.	16.3	53.3	28.3	-	0.33
5/27	97.9	7:30 a.m.	20.0	54.3	23.4	-	0.20
5/27	96.99	11:30 a.m.	22.2	56.2	18.4	-	0.19
5/27	97.07	11:30 p.m.	31.4	43.4	22.0	-	0.27
5/28	96.61	3:30 a.m.	29.3	45.1	22.0	-	0.21
5/28	95.54	7:30 a.m.	29.2	44.7	21.5	-	0.14
5/28	96.8	11:30 a.m.	31.3	43.7	21.6	-	0.20
5/28	94.06	7:30 p.m.	32.2	43.0	18.7	-	0.16
5/29	98.01	9:30 a.m.	30.9	45.3	21.4	0.29	0.21
5/30	98.92	7:30 a.m.	9.2	69.6	19.9	-	0.22
5/30	98.11	11:30 p.m.	10.9	61.3	25.8	-	0.11
5/31	98.36	3:30 a.m.	10.5	56.0	31.6	-	0.26
5/31	98.62	7:30 a.m.	14.9	55.4	28.0	-	0.32
5/31	100.45	3:30 p.m.	15.2	42.0	42.7	-	0.55
5/31	103.5	11:30 p.m.	11.4	59.5	32.5	-	0.16
6/1	100.57	7:00 a.m.	10.8	56.1	33.4	-	0.27
6/1	100.27	7:30 p.m.	9.5	55.3	35.3	-	0.17
6/2	99.09	3:30 p.m.	11.4	55.3	32.0	-	0.19
6/3	100.21	7:30 a.m.	10.9	52.2	36.9	-	0.21
6/3	99.07	3:30 p.m.	12.7	55.2	31.0	-	0.17
6/3	100.81	11:30 p.m.	8.6	59.6	32.5	-	0.11
6/4	101.59	7:30 a.m.	10.9	68.4	22.2	-	0.09
6/4	103.12	7:30 p.m.	5.6	60.3	37.1	-	0.12
6/5	102.11	3:30 a.m.	9.2	53.9	38.8	-	0.21
6/5	101.68	7:30 a.m.	10.1	53.3	38.1	-	0.18
6/5	100.28	3:30 p.m.	8.0	56.2	35.9	-	0.18
6/5	101.56	7:30 p.m.	7.3	61.5	32.6	-	0.16
6/5	102.25	11:30 p.m.	7.2	62.6	32.3	-	0.15
6/6	103.2	3:30 a.m.	7.9	62.3	32.7	-	0.30
6/7	-	11:30 a.m.	9.4	-	-	-	-
6/7	101.7	3:30 p.m.	5.4	59.5	36.8	-	0.10

TABLE 25 (CONT.)

DATE	CLOSURE	TIME	U (%)	AN (%)	GN (%)	H_2O (%)	INSOL. (%)
6/7	102.2	7:30 p.m.	5.1	62.8	34.2	-	0.10
6/7	106.0	11:30 p.m.	8.1	63.5	34.2	-	0.20
6/8	101.4	3:30 a.m.	8.7	62.7	29.9	-	0.10
6/8	104.8	7:30 a.m.	12.6	61.4	30.7	-	0.10
6/9	100.9	11:30 a.m.	14.8	56.5	29.6	-	-
6/10	108.7	7:30 a.m.	14.5	46.8	47.4	-	-
6/11	100.1	7:30 a.m.	26.4	53.3	20.4	-	-
6/11	101.5	4:00 p.m.	8.0	51.9	41.6	-	0.20
6/11	98.16	7:30 p.m.	10.8	54.3	32.8	-	0.26
6/11	98.64	11:30 p.m.	13.5	55.3	29.4	-	0.44
6/12	102.0	3.30 a.m.	11.0	59.7	31.2	-	0.1
6/12	102.0	7:30 a.m.	13.6	55.8	32.3	-	0.3
6/12	100.54	3:30 p.m.	13.8	56.8	29.6	-	0.34
6/12	99.72	11:30 p.m.	17.0	45.9	36.4	-	0.42
6/13	99.24	7:30 a.m.	14.9	50.1	33.9	-	0.34
6/13	100.93	3:30 p.m.	15.2	53.5	32.0	-	0.23
6/13	97.42	11:30 a.m.	17.2	53.5	26.5	-	0.22
6/14	96.93	7:30 a.m.	18.5	49.0	29.1	-	0.33
6/14	98.96	11:30 a.m.	18.4	50.9	29.4	-	0.26
6/14	99.07	7:30 p.m.	17.1	54.1	27.6	-	0.27
6/15	97.9	3:30 a.m.	17.6	51.7	28.3	-	0.30
6/16	102.3	7:30 a.m.	15.3	47.3	39.3	-	0.40
6/17	100.69	7:30 a.m.	16.8	49.4	34.0	0.29	0.20
6/17	102.04	3:30 p.m.	17.3	51.9	32.6	-	0.24
6/17	97.66	11:30 p.m.	13.8	48.6	35.0	-	0.26
6/18	99.06	7:30 a.m.	13.4	46.7	38.7	-	0.26
6/18	99.87	11:30 a.m.	14.6	49.9	35.1	-	0.27
6/18	100.98	7:30 a.m.	9.5	48.9	42.3	-	0.28
6/19	100.08	3:30 a.m.	8.1	50.0	41.7	-	0.28
6/19	98.6	3:30 p.m.	9.0	54.3	35.1	-	0.20
6/20	99.44	7:30 a.m.	7.0	57.3	34.4	-	0.74
6/20	100.24	11:30 p.m.	10.8	58.3	31.0	-	0.14
6/21	102.1	3:30 a.m.	8.4	57.4	36.0	-	0.30
6/21	100.4	7:30 a.m.	7.7	56.9	35.6	-	0.20
6/21	99.1	11:30 p.m.	7.4	57.4	34.3	-	-
6/22	98.6	7:30 a.m.	11.2	53.6	33.8	-	-
6/22	101.22	11:30 p.m.	11.1	54.9	35.0	-	0.22
6/23	97.6	7:30 a.m.	9.8	51.3	36.5	-	0.23
6/23	99.96	11:30 p.m.	9.9	55.8	34.1	-	0.16
6/24	98.21	7:30 a.m.	10.7	54.7	32.6	-	0.21
6/25	98.65	7:30 a.m.	10.9	53.6	33.9	-	0.25
6/25	99.55	11:30 p.m.	7.5	58.3	33.5	-	0.25
6/26	97.34	7:30 a.m.	13.7	50.5	32.8	-	0.34
6/26	98.52	11:30 p.m.	12.1	49.3	36.8	-	0.32
6/27	97.52	7:30 a.m.	15.9	46.1	35.1	-	0.42
6/28	99.08	11:30 p.m.	15.7	46.1	36.8	-	0.48
6/29	95.88	3:30 a.m.	15.0	45.2	35.2	-	0.48
6/29	95.02	7:30 a.m.	18.2	48.1	28.0	-	0.72

TABLE 25 (CONT.)

DATE	CLOSURE	TIME	U (%)	AN (%)	GN (%)	H_2O (%)	INSOL. (%)
7/1	89.58	7:30 a.m.	17.03	52.6	19.9	-	-
7/1	94.7	3:30 p.m.	26.3	51.3	16.8	-	0.30
7/6	96.88	11:30 a.m.	17.8	48.1	30.5	-	0.48
7/6	97.76	11:30 p.m.	14.6	47.4	35.4	-	0.36
7/7	98.63	7:30 a.m.	15.8	46.9	35.5	-	0.43
7/8	96.15	11:30 p.m.	6.8	70.1	19.0	-	0.25
7/9	96.27	3:30 a.m.	16.4	62.1	17.5	-	0.27
7/9	96.03	7:30 a.m.	15.6	52.8	27.3	-	0.33
7/9	98.27	11:30 p.m.	14.9	50.9	32.1	-	0.37
7/10	98.30	7:30 a.m.	12.9	48.9	36.2	-	0.30
7/10	97.14	5:30 p.m.	13.4	51.8	31.5	-	0.44
7/11	96.39	7:30 a.m.	18.2	49.8	27.9	-	0.49
7/12	97.37	3:30 a.m.	21.8	46.1	28.9	-	0.57
7/12	98.83	3:30 p.m.	16.4	47.4	34.4	-	0.63
7/31	100.19	1:30 p.m.	13.2	69.3	17.4	-	0.29
7/31	100.16	3:30 p.m.	13.9	61.9	24.1	-	0.26
7/31	99.6	7:30 p.m.	14.7	59.0	25.4	-	0.50
7/31	96.01	11:30 p.m.	17.1	54.8	23.6	-	0.51
8/1	100.1	3:30 a.m.	19.8	52.6	27.2	-	0.50
8/1	100.74	7:30 a.m.	22.7	45.8	31.6	-	0.64
8/1	98.75	3:30 p.m.	19.1	43.0	35.8	-	0.85
8/2	101.35	3:30 a.m.	6.0	66.3	28.8	-	0.25
8/2	103.65	8:00 a.m.	9.8	64.3	29.3	-	0.25
8/2	99.6	3:30 p.m.	10.4	57.8	31.4	-	0.17
8/2	99.45	7:30 p.m.	11.5	52.7	35.0	-	0.25
8/3	99.28	3:00 a.m.	12.5	53.6	32.8	-	0.38
8/3	97.53	7:30 a.m.	16.8	53.8	26.9	-	0.43
8/4	97.5	11:30 a.m.	13.5	61.7	22.1	-	0.20
8/5	96.4	3:30 p.m.	14.0	58.2	23.9	-	0.26
8/6	99.56	7:30 p.m.	14.5	62.4	22.4	-	0.26
8/7	101.26	7:30 a.m.	19.3	52.6	29.0	-	0.36
8/7	100.4	11:30 a.m.	14.7	58.8	26.9	-	0.29
8/7	101.52	7:30 p.m.	11.7	65.7	23.9	-	0.22
8/7	94.97	11:30 p.m.	11.6	62.8	20.4	-	0.17
8/8	102.53	7:30 a.m.	12.3	62.6	27.3	-	0.33
8/8	98.31	11:30 a.m.	12.0	62.0	24.0	-	0.31
8/8	98.66	7:30 p.m.	12.7	64.6	20.8	-	0.56
8/9	100.36	7:30 a.m.	8.39	70.9	20.9	-	0.17
8/9	100.49	11:00 p.m.	8.8	65.9	25.6	-	0.19
8/10	99.53	7:30 a.m.	10.3	64.2	24.3	-	0.23
8/10	100.43	9:00 a.m.	8.5	64.2	27.4	-	0.33
8/10	99.53	10:00 a.m.	9.8	63.8	25.4	-	0.53
8/10	101.76	12:00 Noon	11.8	61.4	28.1	-	0.46
8/10	100.14	1:00 p.m.	11.1	60.5	28.1	-	0.44

b. Product Melt Disposition

On June 11, it was discovered that the melt product drain line on one of the gas liquid separators was plugged and that the melt from this separator has been leaving that separator via the gas line for 2 or 3 days. At the time, this was baffling since all previous melt flow diversion in the gas line incidents resulted in plugged gas lines and a resultant signal of a problem (gas backup). During this period of operation, four tubes (R-200, R-202, R-203 and R-204) were in operation. The plugged nozzle was in the left bank separator which serviced R-200 through R-203. This meant that during the plugged nozzle time period, three-fourths of the product made left the system via the off-gas system. To prevent recurrence of this event, additional readings and controls were instituted.

Product rate was measured once per shift after this event. Deviation from the expected melt rate (based on feed rate and number of active tubes) would indicate the diversion of product melt in another direction or a plugged product line. Table 26 shows the raw product rate data for the remainder of the operation.

c. Scrubber and Quenching Efficiency

In addition to measuring the product melt rate, hydrometer readings of the ammonia water and the quenched reactor product melt were taken periodically after the above incident. The ammonia-water density would fortify an improper melt flow conclusion. In addition to this, during the hydrometer sampling, if no ammonia smell was detected, a gas line plug was indicated; if a reduced ammonia-water flow was evidenced, a plugged scrubber line was suggested.

Proper control of the water to melt flow ratio to the quench tank is important (1) to ensure complete solution of the soluble portion of the melt and (2) to achieve the proper concentration of feed to the crystallizer so that a maximum GN crystallizer yield is obtained. Excess water would reduce the GN recovered per crystallizer batch and also increase the evaporator load. To obtain a quick reading on the quench solution concentration, its density also was periodically measured, (See Table 26). It was found that a quench solution having a specific gravity of 1.24 at 70° to 80°C satisfied the solution and maximum GN crystallizer yield requirements.

d. Evaporator Operations

Improper evaporator operation creates process problems either in the form of a shortage of recycle feed or in a "wet" recycle feed. There was a shortage of recycle feed when the evaporator-crystallizer system was not "balanced" with the feed system. This condition arose during the pilot plant operation when (1) the feed rate was changed (addition or loss of reactor tubes), (2) major process losses occurred (e.g., plugged G/L separator period referred to above), or (3)

TABLE 26

MISCELLANEOUS PROCESS CONTROL DATA

		Aqueous Quench Tank		Reactor Melt Product Rate		Ammonia Water Drum	
Date	Time	Sp. Grav.	Temp., °C	No. of Tubes	lb/hr	Sp. Grav.	Temp., °C
6/11	7:30 p.m.	1.202	70	4	90	1.000	
6/11	11:30 p.m.	1.220	70	4	93	1.010	
6/12	5:30 a.m.	1.225	70	4	102	1.000	
6/12	11:30 a.m.	1.232	64	4	90	1.002	
6/12	7:30 p.m.	1.200	72	6	130	1.000	
6/13	3:30 a.m.	1.201	70	6	120	1.000	
6/13	7:30 a.m.			6	135		
6/13	9:00 a.m.	1.184	69				
6/13	10:30 a.m.	1.202	73			1.000	
6/13	1:30 a.m.	1.232	77				
6/13	7:30 p.m.	1.250	75	6	125.25	1.010	
6/13	11:30 p.m.	1.255	75	6	117	1.000	
6/14	3:30 a.m.	1.250	72	6	135	1.005	47
6/14	11:30 a.m.	1.250	71	6	129	1.000	64
6/14	6:30 p.m.	1.247	75	6	135	1.006	50
6/15	12:30 a.m.	1.250	73				
6/15	3:30 a.m.	1.255	70	6	114	1.002	66
6/15	11:30 a.m.	1.222	68	7	192	1.001	67
6/15	12:30 p.m.			7	168		
6/15	11:30 p.m.			7	123(1)		
6/16	1:30 a.m.	1.260	75				
6/16	3:30 a.m.	1.255	65	7	126	1.000	65
6/16	4:30 a.m.	1.250	72				
6/16	7:30 a.m.	1.250	72			1.010	50
6/16	11:30 a.m.	1.260	74	7	127.12	1.010	
6/16	6:30 a.m.	1.252	75	7	132	1.003	54
6/16	9:30 p.m.			7	128.25		
6/16	11:30 p.m.			7	114.75		
6/17	3:30 a.m.	1.250	73	7	121.5	1.010	45
6/17	5:30 a.m.	1.255	75				
6/17	11:30 a.m.	1.242	73	7	144		
6/17	7:30 p.m.	1.232	72	6	116.25	1.005	50
6/18	3:30 a.m.	1.230	74	7	108	1.000	58
6/18	11:30 a.m.	1.220	73	7	92.75	1.000	45
6/18	8:30 p.m.	1.224	76	7	117.75	1.030	45
6/19	3:30 a.m.	1.238	75	7	121.5	1.012	67

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(1) Rates decreased on reactors.

TABLE 26 (CONTINUED)

Date	Time	Aqueous Quench Tank		Reactor Melt Product Rate		Ammonia Water Drum	
		Sp. Grav.	Temp., °C	No. of Tubes	lb/hr	Sp. Grav.	Temp., °C
6/19	11:30 a.m.	1.226	76	7	78	1.004	53
6/19	12:30 p.m.			7	98.653		
6/19	7:30 p.m.	1.212	69	7	85.5	1.010	50
6/19	8:30 p.m.			7	84		
6/19	9:30 p.m.					1.010	
6/20	2:30 a.m.	1.232	76	7	102	1.008	50
6/20	11:30 a.m.	1.244	77	7	115.5	1.010	49
6/20	3:30 p.m.	1.231	76	7	109.5	1.004	43
6/20	7:30 p.m.	1.240	75	7	97.5	1.004	57
6/20	10:30 p.m.	1.240	73			1.004	53
6/21	3:30 a.m.	1.232	74	7	110	1.005	53
6/21	11:30 p.m.	1.255	73	7	121.87		
6/21	3:30 p.m.					1.004	46
6/21	5:30 p.m.	1.240	71				
6/21	8:30 p.m.	1.235	71	7	132	1.004	52
6/22	2:30 a.m.	1.220	67	7	87	1.004	51
6/22	11:30 a.m.	1.260	78	8	137.75	1.036	48
6/22	4:30 p.m.	1.240	76			1.005	52
6/22	7:30 p.m.			8	114		
6/22	10:30 p.m.	1.235	73			1.006	55
6/23	2:30 a.m.	1.222	74	8	112.5	1.005	48
6/23	9:30 a.m.			8	97.5		
6/23	10:30 a.m.			8	115.5		
6/23	1:30 p.m.	1.198	75	8	75	1.006	47
6/23	5:30 p.m.			8	123	1.015	
6/23	7:00 p.m.	1.230	60	8	126	1.020	50
6/23	10:30 p.m.	1.230	68				
6/23	11:30 p.m.	1.235	70			1.025	50
6/24	3:30 a.m.	1.236	64	8	135.87	1.009	46
6/24	11:30 a.m.	1.224	71	8	114	1.006	40
6/24	7:30 p.m.	1.220	75	8	120	1.008	45
6/25	3:30 a.m.	1.224	70	8	121.25	1.004	56
6/25	1:30 p.m.	1.222	73			1.008	44
6/25	7:30 p.m.	1.150	73	8	72	1.100	
6/26	3:30 a.m.	1.226	70	8	110.22		
6/26	1:30 p.m.	1.220	77	7	122	1.006	55

TABLE 26 (CONTINUED)

Date	Time	Aqueous Quench Tank Sp. Grav.	Temp., °C	Reactor Melt Product Rate No. of Tubes	lb/hr	Ammonia Water Drum Sp. Grav.	Temp., °C
6/26	7:30 p.m.	1.232	73	8	129.75	1.003	56
6/27	4:30 a.m.	1.234	66	8	110.25	1.006	56
6/27	7:30 p.m.	1.220	68	7	93	1.004	66
6/28	8:30 p.m.	1.240	70	8	91.5	1.006	50
6/29	4:30 a.m.	1.202	63	8	110.37	1.006	46
6/29	8:30 a.m.	1.200	64			1.006	45
6/29	11:30 a.m.			8	66		
6/29	1:30 p.m.	1.200	65	8	75	1.006	45
6/30	6:30 p.m.	1.220	64	5	78	1.005	46
6/30	3:30 a.m.	1.224	63	5	63		
6/30	10:30 a.m.	1.200	69	4	57	1.002	42
6/30	6:30 p.m.	1.278	69				
6/30	7:30 p.m.			4	54.75	1.003	43
6/30	8:30 p.m.	1.270	70				
6/30	9:30 p.m.	1.248	66				
7/1	3:30 a.m.	1.251	60	3	36.75		
7/1	6:30 a.m.			3	42	1.000	55
7/1	8:30 a.m.	1.225	60				
7/1	11:30 a.m.			3	28		
7/1	3:30 a.m.	1.240	63			1.010	40
7/1	7:30 p.m.	1.280	54	2	10 oz	1.002	36
7/1	9:30 p.m.	1.222	74				
7/5	7:30 a.m.			2	34.25		
7/5	9:30 a.m.	1.160					
7/5	11:30 a.m.	1.240	86	2	19.5	1.008	47
7/5	7:30 p.m.	1.270	78	4	33.6	0.99	67
7/5	8:30 p.m.			8	75.0		
7/6	12:30 a.m.	1.275	81				
7/6	3:30 a.m.			4	84		
7/6	6:30 a.m.	1.270	81			1.005	60
7/6	11:30 a.m.	1.268	80	1	24.5	1.001	40
7/6	4:30 p.m.			8	115.25		
7/6	5:30 p.m.	1.230	69				
7/6	7:30 p.m.	1.238	73				
7/6	8:30 p.m.	1.210	65				
7/6	9:30 p.m.			8	143.62		

TABLE 26 (CONTINUED)

		Aqueous Quench Tank		Reactor Melt Product Rate		Ammonia Water Drum	
Date	Time	Sp. Grav.	Temp., °C	No. of Tubes	lb/hr	Sp. Grav.	Temp., °C
7/7	12:30 a.m.	1.220	68			1.010	60
7/7	2:30 a.m.			8	141		
7/7	5:30 a.m.	1.210	65			1.020	50
7/7	12:30 p.m.	1.200	65			< 1.000	65
7/7	7:30 p.m.	1.200	70	8	153	1.020	65
7/7	9:30 p.m.	1.22	66				
7/8	1:30 a.m.	1.220	65			1.020	53
7/8	3:30 a.m.			8	138		
7/8	6:30 a.m.	1.200	71			1.025	50
7/8	9:30 a.m.	1.200	78				
7/8	11:30 a.m.			7	123	1.022	55
7/8	12:30 p.m.	1.196	75				
7/8	9:30 p.m.	1.160	73	7	136 lb-12 oz		
7/9	1:30 a.m.			7	171		
7/9	2:30 a.m.	1.170	68			1.010	55
7/9	5:30 a.m.	1.230	74			1.010	58
7/9	10:30 a.m.	1.242	68				
7/9	11:30 a.m.			7	168		
7/9	12:30 p.m.	1.224	73				
7/9	7:30 p.m.	1.240	-	7	144	1.010	-
7/10	2:00 a.m.	1.200	-	7	147	1.010	-
7/10	6:30 a.m.	1.200	-			1.010	-
7/10	10:00 a.m.	1.216	-	7	145	1.010	-
7/10	7:30 p.m.	1.226	76	7	135	1.000	62
7/11	12:30 a.m.	1.200	75	7	134	1.000	
7/11	5:30 a.m.	1.235	62			1.012	55
7/11	11:00 a.m.	1.234	66	7	162	1.014	65
7/11	8:30 p.m.	1.220	67	6	120 lb-12 oz	1.006	-
7/12	3:30 a.m.	1.232	64	6	115 lb-8 oz	1.012	49
7/12	12:30 p.m.	1.242	65	6	120 lb-8 oz	1.006	50
7/12	7:30 p.m.	1.220	68	6	111	1.010	
7/13	3:30 a.m.	1.214	74	6	78	1.004	66
7/13	8:30 a.m.	1.237	74	6	123 lb-12 oz	1.016	67
7/14	3:30 a.m.	1.380	76	6	122 lb-4 oz	1.016	60
7/14	10:30 a.m.	1.244	72			1.000	57

TABLE 26 (CONTINUED)

		Aqueous Quench Tank		Reactor Melt Product Rate		Ammonia Water Drum	
Date	Time	Sp. Grav.	Temp., °C	No. of Tubes	lb/Hr	Sp. Grav.	Temp., °C
8/1	12:36 a.m.	1.250	70			1.006	55
8/1	3:30 a.m.			3	27		
8/1	7:30 a.m.	1.280	78			1.000	60
8/1	12:30 p.m.	1.250	60	3	63	1.006	58
8/1	7:30 p.m.	1.200	69	3	39	1.006	52
8/2	1:30 a.m.	1.200	69	3	32-4	1.012	42
8/2	10:30 a.m.	1.225	75	2	27	0.995	63
8/2	7:30 p.m.	1.204	72	3	63	0.997	30
8/3	9:30 a.m.	1.130	72	3	51	1.010	48
8/3	7:30 p.m.	1.238	74	3	45	1.010	46
8/4	4:30 a.m.	1.220	68	3	48	1.006	45
8/4	10:30 a.m.	1.231	74	3	45	1.003	43
8/4	8:30	1.220	72	3	79-8	1.009	44
8/5	4:3	1.260	70	3	72	1.006	60
8/5	11:30	1.242	77	3	45	1.003	49
8/5	8:30 p.m.	1.262	78	3	82-8	1.006	49
8/6	2:30 a.m.	1.268	82	3	70-3	1.009	-
8/6	11:30 a.m.	1.260	80	3	63	1.003	-
8/6	8:30 p.m.	1.250	80	3	72	1.010	-
8/7	2:30 a.m.	1.266	80	3	67-3	1.012	-
8/7	12:30 p.m.	1.250	81	3	81	1.019	-
8/7	9:00 a.m.	1.210	75	3	51	1.005	-
8/8	5:30 a.m.	1.236	77	3	54	1.006	-
8/8	11:30 a.m.	1.224	78	3	54	1.003	-
8/8	5:30 p.m.	1.240	76	3	57	1.005	46
8/8	11:00 p.m.	1.225	74	3	48	1.006	42
8/9	3:30 a.m.	1.220	74	3	33	1.009	-
8/9	8:30 a.m.			3	60		
8/9	1:30 p.m.	1.240	81			1.005	

crystallizer vacuum problems created an evaporator feed shortage. To achieve a "balanced" pilot plant recycle operation, it was necessary to occasionally add artificial "recycle" (fresh makeup of the AN/U ratio or actual recycle), slowing down the reactor and/or evaporator feed rates, or eliminate fresh feed makeup. The last procedure was necessary if there were an excess system inventory or if the system became urea-rich. During periods of smooth operation, a "balanced" pilot plant operation was frequently demonstrated by controlling the blended feed and evaporator feed tank levels at constant levels and by controlling the weight of fresh makeup added to the system to balance the products (GN and insolubles) and losses. The evaporator feed tank level fluctuated constantly because of the batch addition and continuous depletion, but its level swings between batch additions should not change.

"Wet" evaporator bottoms is a process problem because this water is ultimately fed into the reactor and its effect is increased urea hydrolysis and lower productivity and urea yield. It was found that by maintaining a minimum evaporator bottoms temperature of 295°F, a satisfactory minimum water level (ca. 1%) in the evaporator bottoms could be attained. It was later necessary to increase this minimum temperature to overcome ammelide fouling of the evaporator. An experiment was conducted before tube fouling occurred to determine this minimum bottoms temperature. The results of that experiment, made at constant feed rate, are shown below.

Evaporator Bottoms Temperature, °F	% H_2O in Product
295	0.51
284	5.27

Since ammelide fouling will be minimized in a commercial plant evaporator, surveillance of the bottoms temperature and bottoms product "fudge point" will provide two rapid monitors of evaporator performance. In the "fudge point" method, a sample of the bottoms product is placed on a heat sink (aluminum ladle) and the temperature at which this material becomes "fudgy" is noted. It was known that for the U/AN/GN evaporation, a fudge point of "165°C or greater" would ensure a satisfactory "dry" bottoms product.

e. Product Assay

Since numerous crystallizer batches result from the continuous operation, a quick indication of the GN assay was needed before all these batches were blended in the drying operation. As noted in a previous section, the melting point of the pilot plant product provided a "quick" assessment of the product quality (see Figure 2). A low melting point indicated high AN and U in the product; a high nondistinct melting point indicated a higher than normal insolubles level. It was found that a distinct melting point in the range of 208 to 214°C would, on later complete analyses, prove to be a good product.

D. PROCESS CALCULATIONS AND RESULTS

1. Determination of Reactor Feed Rate

As noted in previous sections, feed rates to the reactors were controlled by individual flow control loops. The sensitivity of the control systems resulted in fluctuating feed rates around the set point. This was due to the extremely small flow rate of 2 gal/hr/tube, relatively large 1-in.-diameter lines, and pulsating downstream pressure resulting from the gas-liquid foaming in the reactor. Attempts to tune the flow controllers were unsuccessful. Typically, at a setpoint of 22 lb/hr, a flow of 15-25 lb/hr would result. Day-to-day reactor productivities were calculated using nominal reactor feed rates. An alternate calculation method was devised in which the feed rate was determined from the reactor feed analysis, product analysis, and the product melt rate. As shown in the following paragraphs, methods using these data, providing proper assumptions are made, are suitable for accurately determining feed rate and measuring reactor performance. The validity of the assumptions was later checked in a one-day material balance in which all the reactor feed and product were weighed.

a. Nitrate Balance

If there are no entrainment or vaporization losses of nitrates, and if the chemistry of the reaction system is as assumed, all the nitrates entering the reactors as either ammonium nitrate or guanidine nitrate also leave the reactor as nitrates (as AN or GN but in a different composition).

$$\text{Total Nitrates In} = \text{Total Nitrates Out} \qquad (1)$$

$$(\text{Feed Rate In}) \times (\%\ NO_3^-)\ \text{In} = (\text{Product Rate Out}) \times (\%\ NO_3)\ \text{Out} \qquad (2)$$

$$\frac{\text{Product Rate Out}}{\text{Feed Rate In}} = (P/F) = \frac{\%\ NO_3\ \text{In}}{\%\ NO_3\ \text{Out}} \qquad (3)$$

Using the above expressions, the feed rate can be calculated if the feed analysis, product analysis, and the product melt rate are known.

b. NO_3 Conservation with Entrainment Losses

If there is entrainment loss, the same expression can be used for its calculation as was used for the nitrate balance as long as the feed analysis is adjusted to account for this added loss. An assumption has to be made for the composition of the entrainment stream. Feed loss rather than product melt loss was assumed in those calculations utilizing this method of calculation.

c. Blended Feed Using Graphical Methods

The reactor feed rate can be determined by using the compositions of the two streams making up this feed (evaporator bottoms and fresh make-up feed) and knowing at least one of the rates of these two streams. The assumptions of nitrate and urea conservations in the blending step are inherent in this approach. The appropriate mathematical expressions are as follows:

Make-up feed rate + Recycle feed rate = Total feed rate (4)

(Make-up feed rate x % urea) + (recycle feed rate x % urea)

= (Total feed rate x % urea) (5)

Expressions similar to equation (5) exist for ammonium nitrate and guanidine nitrate.

The solution of the resultant expressions can best be done using triangular graphical methods. Figure 6 shows an example of a hypothetical case. In this figure, if the compositions of the recycle and make-up streams are plotted and then a straight line drawn between them, the resultant line represents the combination of all possible blends that can be made with these two mixes. If the actual resultant blend is known, then

(1) It will fall on this line, and

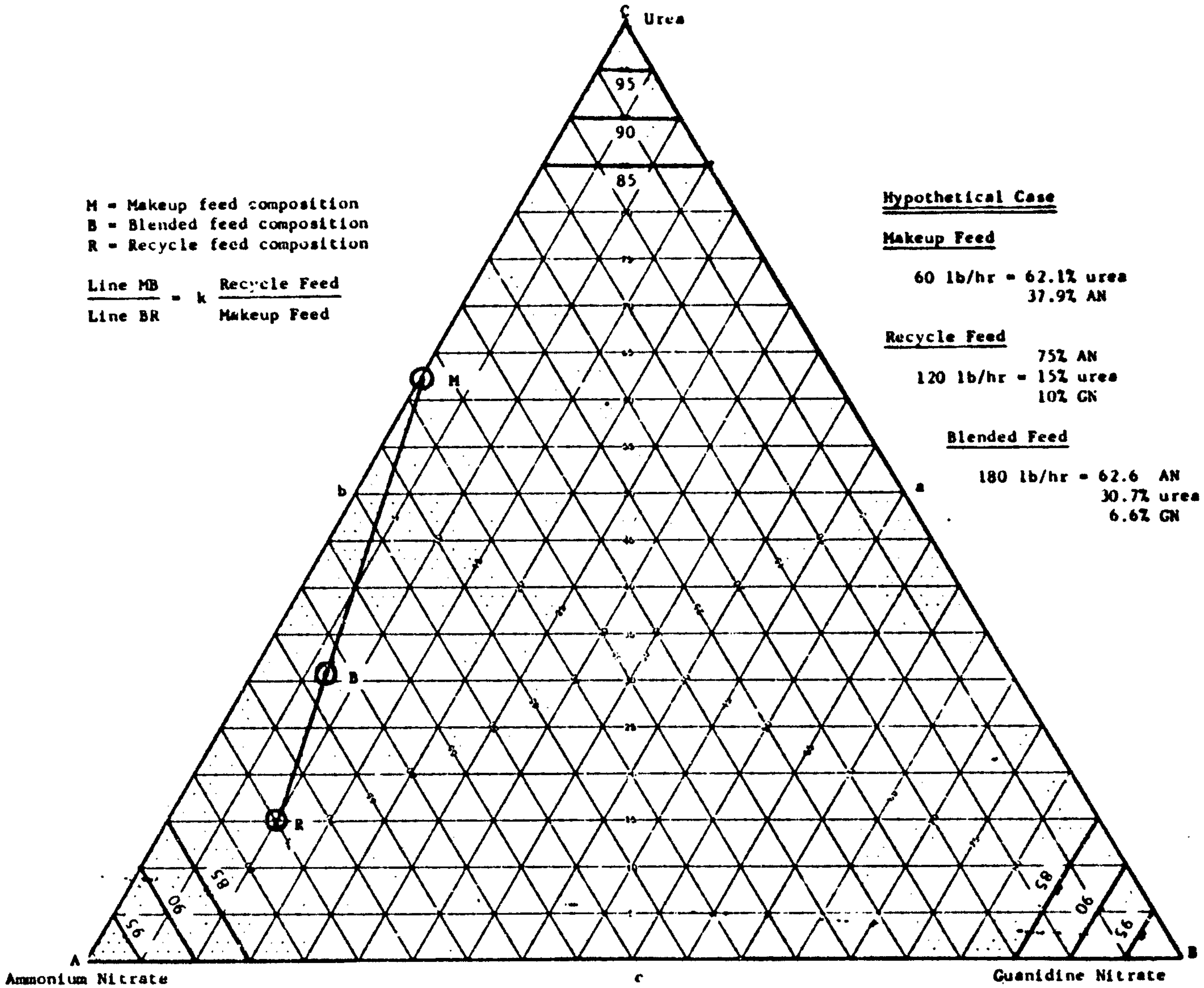
(2) The lengths of the two lines are proportional to the weights of the two materials blended.

Therefore, if all three analyses (recycle, make-up and blended compositions) and at least one of the two feed-rates (make-up or recycle) are known, then the other feed-rate can be determined. Since the total feed rate is the value of interest and it is the sum of the individual two feed rates, it can then be calculated. Figure 7 shows an example of data used in this manner. As can be seen from this diagram, the analyzed compositions evidently were not precise since the resultant measured blended feed composition did not fall precisely on the predicted line. This lack of precision is attributed to the presence of other components in these samples (primarily water).

The calculated total reactor feed rate was 109.8 lb/hr. If the nitrate conservation method of (a) above is used, a value of 109.4 lb/hr is achieved. The agreement is very good, and the difference in these values is within experimental error.

M = Makeup feed composition
B = Blended feed composition
R = Recycle feed composition

$$\frac{\text{Line MB}}{\text{Line BR}} = k\,\frac{\text{Recycle Feed}}{\text{Makeup Feed}}$$



Hypothetical Case

Makeup Feed

60 lb/hr = 62.1% urea
37.9% AN

Recycle Feed

75% AN
120 lb/hr = 15% urea
10% GN

Blended Feed

180 lb/hr = 62.6 AN
30.7% urea
6.6% GN

Figure 6. Principle of Using Triangular Coordinates to Determine Reactor Feed Rate

A - Data

Time - 6/27/73, 7:00 p.m.

1. Makeup Feed

46.5 lb/hr = 47.9% AN
52.1% urea

2. Recycle Feed

X lb/hr = 74.2% AN
10.8% urea
15.0% GN

3. Blended Feed

Y lb/hr = 63.5% AN
29.3% urea
7.2% GN

B - Calculations

Length_{MB} = 1-7/8 inch
Length_{BR} = 1-3/8 inch

$$\frac{\text{Recycle Feed Rate}}{\text{Makeup Feed Rate}} = \frac{1\text{-}7/8}{1\text{-}3/8}$$

Recycle Feed Rate

= 1.36 x 46.5 lb/hr = 63.3 lb/hr

Total blended feed

= 63.3 + 46.5

= 109.8 lb/hr

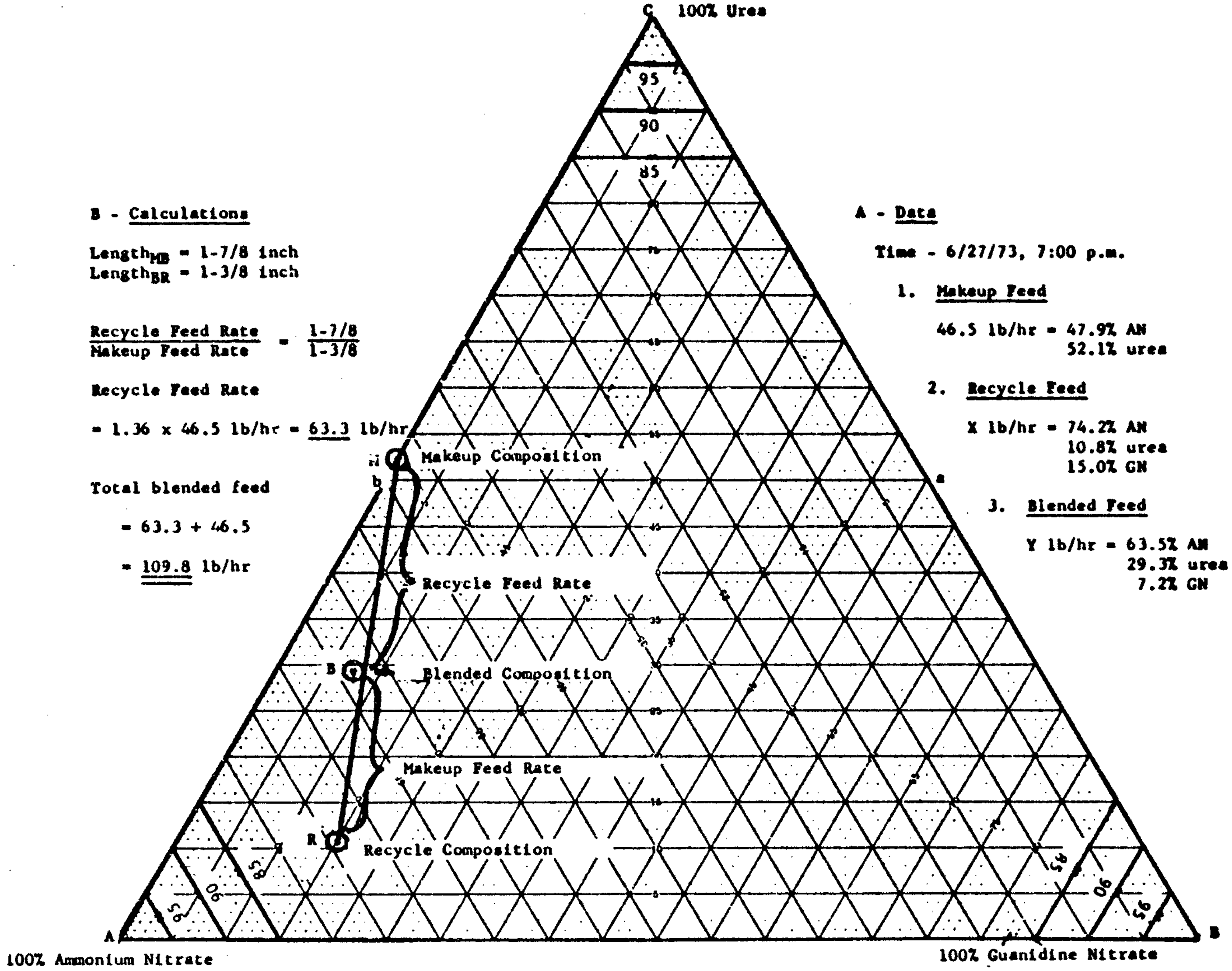


Figure 7. Use of Triangular Coordinates to Solve for Blended Reactor Feed Rate

2. Reactor Performance

With the procedures described in the previous section to calculate feed rates, with the analyses completed and product melt rates determined, all the input data were on hand to calculate the necessary parameters to indicate reactor performance. Sets of data from various typical operating periods were selected, and these data and the developed calculations are shown in Table 27.

Definitions of some of the columns in Table 27 are presented below:

Column No.	Definition
A	Moles AN, U, GN, H_2O/100 lb feed - These values are determined by dividing the analytical result of each component by its respective molecular weight and then normalizing to 100 lb.
B	Moles AN, U, GN, Insolubles/100 lb reactor product - Same as above except reactor product analysis is employed.
C	AN/U Reactor Feed Molar Ratio - This ratio value is determined by dividing the moles AN by the moles U per 100 lb feed.
D	Product/Feed Weight Ratio (P/F) - This value is used, as shown below, to place the reactor product analysis on a reactor feed basis. The assumption of nitrate conservation is used; i.e., no loss of nitrates from the feed stream due to decomposition. Solving the expression: $\frac{\text{Moles } NO_3}{\text{100 lb Feed}} = \frac{\text{Pweight}}{\text{Fweight}} \times \frac{\text{Moles } NO_3}{\text{100 lb Product}}$ results in the desired P/F ratio of (lb Product/lb Feed)
E	Moles Product Components/100 lb Feed - These values are calculated to put the reactor product analysis on a feed rate basis and employs the above assumed nitrate conservation P/F ratio. The calculation is as follows: $(P/F) \text{ or } \frac{\text{lb Product}}{\text{lb Feed}} \times \frac{\text{Moles Product AN, etc.}}{\text{100 lb Product}} = \frac{\text{Moles Product AN, etc.}}{\text{100 lb Feed}}$

TABLE 27

CALCULATED FEED RATES, YIELDS, ETC., UTILIZING REACTOR PRODUCT ANALYSIS AND RATE - PART I

		Feed								Reactor Product								Calculated From Analyses					
		Analyses, %				Ⓐ Moles/100 Lb				Analyses, %				Ⓑ Moles/100 Lb				Ⓒ AN/U In Feed	Ⓓ P/F	Ⓔ P/F × Moles/100 Lb Product			
Date	Time	AN	U	GN	H_2O	AN	U	GN	H_2O	AN	U	GN	Insol.	AN	U	GN	Insol.			AN	U	GN	Insol.
6/14	11:30 p.m.	54.6	37.1	7.02	1.02	0.6843	0.6199	0.0577	0.0568	50.9	18.6	29.4	0.26	0.6429	0.3099	0.2435	0.0021	1.10	0.8371	0.5382	0.2594	0.2038	0.0017
6/15	1:30 p.m.	58.6	31.8	8.54	1.04	0.7330	0.5298	0.0700	0.0582	46.2	15.0	38.4	0.39	0.5780	0.2493	0.3149	0.0031	1.38	0.8991	0.5196	0.2242	0.2832	0.0028
6/18	7:30 a.m.	56.7	35.5	6.6	0.70	0.7123	0.5946	0.0544	0.0391	46.7	13.4	38.9	0.30	0.5879	0.2249	0.3211	0.0024	1.20	0.8435	0.4958	0.1897	0.2708	0.0020
6/19	3:30 a.m.	60.6	24.9	11.90	1.65	0.7648	0.4190	0.0985	0.0926	54.3	9.04	35.1	0.20	0.6881	0.1527	0.2917	0.0016	1.83	0.8811	0.6062	0.1345	0.2570	0.0014
6/20	7:30 a.m.	65.9	26.55	6.31	1.22	0.8240	0.4425	0.0517	0.0678	57.6	7.04	34.6	0.74	0.7203	0.1173	0.2836	0.0058	1.86	0.8723	0.6283	0.1023	0.2474	0.0051
6/24	7:30 a.m.	62.3	29.6	7.42	0.88	0.7784	0.4931	0.0593	0.0504	69.6	18.2	27.2	0.21	0.6962	0.1816	0.2721	0.0017	1.58	0.8651	0.6023	0.1571	0.2354	0.0015
6/26	11:30 p.m.	59.7	32.4	6.57	0.87	0.7497	0.5425	0.0541	0.0486	49.3	12.1	36.8	0.32	0.6255	0.2049	0.3062	0.0025	1.38	0.8627	0.5396	0.1768	0.2642	0.0022
7/9	11:30 p.m.	59.34	37.2	2.76	0.73	0.7418	0.6196	0.0226	0.0403	51.83	15.17	32.7	0.31	0.6479	0.2529	0.2679	0.0024	1.38	0.8347	0.5408	0.2108	0.2236	0.0020
7/10	7:30 a.m.	55.0	37.4	5.76	1.85	0.6879	0.6229	0.0471	0.1027	49.74	13.1	36.8	0.31	0.6218	0.2187	0.3018	0.0024	1.10	0.7958	0.4998	0.1740	0.2402	0.0019
7/10	5:30 p.m.	57.5	35.6	4.98	2.00	0.7183	0.5925	0.0408	0.1114	56.04	13.0	30.5	0.42	0.7005	0.2165	0.2503	0.0033	1.21	0.7984	0.5593	0.1728	0.1998	0.0026
7/11	7:40 a.m.	56.0	37.7	4.51	1.77	0.7005	0.6278	0.0370	0.0986	52.0	18.9	29.0	0.51	0.6498	0.3147	0.2373	0.0040	1.12	0.8351	0.5430	0.2628	0.1982	0.0033
7/12	3:00 a.m.	46.3	47.9	4.47	1.34	0.5786	0.7985	0.0366	0.0744	47.3	22.4	29.7	0.59	0.5918	0.3731	0.2433	0.0046	0.73	0.7367	0.4360	0.2749	0.1792	0.0034
7/12	3:00 p.m.	49.0	42.2	7.21	1.60	0.6125	0.7031	0.0591	0.0891	48.0	16.7	34.8	0.64	0.5995	0.2776	0.2853	0.0050	0.87	0.7590	0.4550	0.2107	0.2165	0.0038
7/31	4:00 a.m.	68.7	27.3	2.35	1.67	0.8590	0.4593	0.0193	0.0926	77.4	13.2	8.8	0.58	0.9677	0.2203	0.0721	0.0045	1.89	0.8477	0.8203	0.1867	0.0611	0.0038
7/31	1:30 p.m.	64.8	32.5	2.1	0.61	0.8095	0.5422	0.0172	0.0338	69.2	13.2	17.4	0.29	0.8646	0.2196	0.1424	0.0023	1.49	0.8210	0.7098	0.1803	0.1169	0.0019
7/31	7:30 p.m.	61.7	35.1	2.5	0.70	0.7717	0.5846	0.0204	0.0390	59.3	14.8	25.5	0.38	0.7415	0.2463	0.2093	0.0030	1.32	0.8331	0.6177	0.2052	0.1744	0.0025
8/1	3:30 a.m.	55.0	42.1	1.93	0.94	0.6875	0.7024	0.0158	0.0520	52.5	19.8	27.2	0.50	0.6568	0.3297	0.2227	0.0039	0.98	0.7997	0.5252	0.2637	0.1781	0.0031
8/1	7:30 a.m.	56.7	42.2	0.55	0.62	0.7082	0.7027	0.0045	0.0347	45.5	22.5	31.4	0.63	0.5752	0.3756	0.2571	0.0049	1.00	0.8635	0.4967	0.3243	0.2220	0.0042
8/2	3:30 a.m.	68.6	20.2	10.3	0.87	0.8573	0.3372	0.0846	0.0483	65.4	5.9	28.4	0.26	0.8177	0.0987	0.2329	0.0020	2.54	0.8965	0.7331	0.0885	0.2088	0.0018
8/2	8:00 a.m.	70.3	19.9	6.76	0.64	0.8789	0.3716	0.0555	0.0353	62.0	9.5	28.3	0.24	0.7754	0.1576	0.2317	0.0019	2.37	0.9277	0.7193	0.1462	0.2149	0.0018
8/2	7:30 p.m.	59.8	29.8	9.19	1.22	0.7474	0.4966	0.0753	0.0680	53.0	11.6	35.2	0.24	0.6624	0.1927	0.2885	0.0020	1.50	0.8652	0.5731	0.1667	0.2496	0.0017
8/3	3:00 a.m.	60.5	28.0	10.2	1.35	0.7562	0.4664	0.0833	0.0749	54.0	12.6	33.0	0.38	0.6749	0.2098	0.2708	0.0030	1.62	0.8577	0.5791	0.1802	0.2404	0.0027
8/4	11:30 a.m.	67.5	24.5	7.15	0.88	0.8435	0.4083	0.0586	0.0488	63.3	13.8	22.7	0.20	0.7910	0.2308	0.1858	0.0016	2.07	0.9235	0.7305	0.2131	0.1716	0.0015
8/5	3:30 p.m.	63.9	24.5	9.85	1.78	0.7984	0.4084	0.0807	0.0988	60.4	14.5	24.8	0.28	0.7550	0.2422	0.2033	0.0022	1.95	0.9174	0.6926	0.2222	0.1865	0.0020
8/6	7:30 a.m.	70.5	23.4	5.22	0.91	0.8806	0.3902	0.0428	0.0505	64.3	17.3	18.2	0.23	0.8036	0.2888	0.1488	0.0018	2.26	0.9676	0.7776	0.2794	0.1440	0.0017
8/6	7:30 p.m.	63.3	27.5	6.17	3.03	0.7912	0.4584	0.0506	0.1685	62.7	14.6	22.3	0.26	0.7835	0.2427	[illegible]	0.0020	1.72	0.8698	0.6814	0.2111	0.1604	0.0017
8/7	7:30 p.m.	66.3	31.8	8.17	1.48	0.8284	0.3979	0.0670	0.0934	64.7	11.5	23.5	0.22	0.8088	0.1920	0.1929	0.0017	2.08	0.8939	0.7230	0.1716	0.1724	0.0015
8/8	7:30 a.m.	65.2	26.8	7.26	0.77	0.8149	0.4464	0.0595	0.0427	61.1	12.0	26.7	0.32	0.7632	0.2006	0.2185	0.0025	1.83	0.8907	0.6798	0.1781	0.1946	0.0022

NOTES:

AN	Ammonium Nitrate	P/F	Reactor Product to Feed Wt. Ratio (moles NO_3^- per 100 lb feed/moles NO_3^- per 100 lb Prod)	Y_{U-H_2O}	Urea Yield to Feed H_2O Hydrolysis
U	Urea	% Urea Equiv.	Urea consumption to hydrolysis, vaporization and other losses	Y_{U-UUP}	Urea Yield in Excess of Y_{U-GN} + Y_{U-A} + $Y_{U-Vap.}$
GN	Guanidine Nitrate	Y_{U-GN}	Urea Yield to GN (2-mole stoichiometry)	F/W	% Moles Urea/Total Water Moles (Free + Reaction)
Insol.	Insolubles (Ammelide)	Y_{U-A}	Urea Yield to Ammelide		
AN/U	Molar Ratio Reactor Feed				
A	Ammelide (3 urea equivalents)				

TABLE 27 (CONTINUED)

PART II

Date	Time	Ⓕ Urea Equivalents, Moles								Ⓖ Moles GN Made	Ⓗ Urea Yields, %				Ⓘ Urea Hydrolysis Efficiency E_{UV}	Ⓙ Lb Urea Used / LB GN Made	Lb AN Used / Lb GN Made	Meas. Melt Rate, Lb/Hr	Ⓚ Calc. Feed Rate, Lb/Hr	Ⓛ Plant Productivity lb/Day
		Feed			Product				Δ Urea Equiv.		Y_{U-GN} to GN	Y_{U-A} to Insol.	Y_{U-H_2O} to Hydrol.	Y_{U-VAP}, 100%-U_{GN}-U_A-U_{H_2O}						
		As U	As GN	Total	As U	As GN	As A(3U)	Total												
6/14	11:30 a.m.	0.6199	0.0577	0.6776	0.2594	0.2038	0.0052	0.4684	0.2092	0.1461	81.1	1.9	15.8	1.2	102.0	1.214	0.656	129	154	651
6/15	1:30 p.m.	0.5298	0.0700	0.5998	0.2242	0.2832	0.0083	0.5157	0.0841	0.2132	139.5	3.6	19.0	-61.2	30.6	0.705	↓	168	187	1165
6/18	7:30 a.m.	0.5946	0.0544	0.6490	0.1897	0.2708	0.0060	0.4665	0.1825	0.2164	106.9	2.0	9.7	-18.1	71.0	0.920		100	119	745
6/19	3:30 a.m.	0.4190	0.0985	0.5175	0.1345	0.2570	0.0041	0.3956	0.1219	0.1585	111.4	1.9	32.5	-45.3	48.3	0.883		100	114	517
6/20	7:30 a.m.	0.4425	0.0517	0.4942	0.1023	0.2474	0.0152	0.3649	0.1293	0.1957	115.0	6.0	19.9	-39.4	48.2	0.855		110	126	723
6/24	7:30 a.m.	0.4931	0.0593	0.5524	0.1571	0.2354	0.0043	0.3968	0.1556	0.1761	104.8	1.7	15.0	-21.1	68.3	0.938		125	145	558
6/26	11:30 p.m.	0.5425	0.0541	0.5966	0.1768	0.2642	0.0066	0.4476	0.1490	0.2101	115.0	2.4	13.3	-30.1	57.2	0.856		122	141	855
7/9	11:30 p.m.	0.6196	0.0226	0.6422	0.2111	0.2236	0.0060	0.4407	0.2015	0.2010	98.4	2.0	9.9	-9.8	82.8	1.00	0.656	168	201	1185
7/10	7:30 a.m.	0.6229	0.0471	0.6700	0.1740	0.2402	0.0057	0.4199	0.2501	0.1931	86.0	1.7	22.9	-10.2	84.0	1.14	↓	146	183	1036
7/10	5:30 p.m.	0.5925	0.0408	0.6333	0.1728	0.1998	0.0080	0.3806	0.2527	0.1590	75.8	2.5	26.5	-4.2	92.6	1.30		135	169	786
7/11	7:40 a.m.	0.6278	0.0370	0.6648	0.2628	0.1982	0.0100	0.4710	0.1938	0.1612	88.3	3.6	27.0	-18.0	73.9	1.11		148	177	839
7/12	3:00 a.m.	0.7935	0.0366	0.8351	0.2749	0.1792	0.0101	0.4642	0.3709	0.1426	54.5	2.5	14.4	29.2	168.2	1.79		116	157	658
7/12	3:00 p.m.	0.7031	0.0591	0.7622	0.2100	0.2166	0.0113	0.4379	0.3243	0.1575	63.9	3.1	18.1	15.7	129.4	1.54		120	158	724
7/31	4:00 a.m.	0.4543	0.0193	0.4736	0.1861	0.0609	0.0113	0.2583	0.2153	0.0416	31.0	5.6	34.5	30.3	155.6	3.17	0.656	-	-	-
7/31	1:30 p.m.	0.5422	0.0172	0.5594	0.1803	0.1169	0.0056	0.3028	0.2566	0.0997	55.1	2.0	9.3	34.1	189.3	1.79	↓	-	-	-
7/31	7:30 p.m.	0.5846	0.0204	0.6050	0.2052	0.1744	0.0074	0.3870	0.2180	0.1540	81.2	2.7	10.3	6.5	111.5	1.22		-	-	-
8/1	3:30 a.m.	0.7024	0.0158	0.7182	0.2637	0.1781	0.0094	0.4512	0.2670	0.1623	74.0	2.8	11.9	12.0	122.2	1.33		27	34	160
8/1	7:30 a.m.	0.7027	0.0045	0.7072	0.3243	0.2220	0.0126	0.5589	0.1483	0.2175	115.0	4.4	9.2	-27.5	57.8	0.856		63	73	465
8/2	3:30 a.m.	0.3372	0.0846	0.4218	0.0885	0.2088	0.0055	0.3028	0.1190	0.1242	99.9	2.9	19.4	-21.5	90.7	0.985		33	37	133
8/2	8:00 a.m.	0.3716	0.0554	0.4270	0.1462	0.2149	0.0052	0.3663	0.0807	0.1595	141.5	3.1	15.7	-59.5	30.9	0.695		63	68	318
8/2	7:30 p.m.	0.4966	0.0753	0.5719	0.1667	0.2496	0.0051	0.4214	0.1505	0.1743	105.7	2.0	20.6	-27.8	62.1	0.931		63	73	371
8/3	3:00 a.m.	0.4664	0.0833	0.5497	0.1862	0.2404	0.0080	0.4346	0.1151	0.1571	112.1	4.0	26.7	-41.7	49.0	0.877		51	57	264
8/4	11:30 a.m.	0.4083	0.0586	0.4669	0.2131	0.1716	0.0044	0.3891	0.0778	0.1130	115.8	3.1	25.0	-43.1	47.6	0.850		48	52	172
8/5	3:30 p.m.	0.4084	0.0807	0.4891	0.2222	0.1865	0.0061	0.4148	0.0743	0.1058	113.6	4.4	53.1	-70.0	36.0	0.866		72	78	244
8/6	7:30 a.m.	0.3902	0.0428	0.4330	0.2800	0.1443	0.0053	0.4296	0.0034	0.1015	184.2	6.4	45.8	-134.8	2.2	0.534		68	70	210
8/6	7:30 p.m.	0.4584	0.0506	0.5090	0.2111	0.1604	0.0053	0.3768	0.1322	0.1098	88.8	2.8	68.1	-59.0	47.2	1.108		72	83	266
8/7	7:30 p.m.	0.3978	0.0670	0.4648	0.1716	0.1724	0.0046	0.3486	0.1162	0.1054	93.2	2.7	41.3	-36.5	57.9	1.172		80	89	277
8/8	7:30 a.m.	0.4464	0.0595	0.5059	0.1281	0.1946	0.0067	0.3794	0.1265	0.1351	100.7	3.3	15.9	-19.1	70.3	0.977		54	60	241

F Moles Urea Equivalents on Normalized Feed Basis - All components present either as urea or derived from urea in both the feed and the product are converted to urea equivalents for determining the disposition of urea, either as urea, product (GN), ammelide, hydrolyzed urea losses. Guanidine nitrate is assigned a urea equivalent value of one mole urea per mole GN. Ammelide is assigned a value of three moles of urea per mole of ammelide. Urea can "vaporize," i.e., leave the melt, by hydrolysis to ammonia and carbon dioxide or by thermal decomposition to ammonia and cyanic acid. The difference (Δ-urea equivalent) represents these losses.

G Moles GN produced/100 lb Feed - Guanidine nitrate produced per 100 lb feed results from determining the difference between the moles GN/100 lb product (adjusted to moles GN/100 lb feed by the P/F ratio) and the moles GN/100 lb actual feed.

H Urea Yields - Urea yields are calculated for four different categories as shown below:

1. $$Y_U \text{ to GN} = \frac{\text{Moles GN Produced} \times 2}{\text{Moles U Consumed}} \times 100$$

2. $$Y_U \text{ to A} = \frac{\text{Moles Ammelide Produced} \times 4}{\text{Moles U Consumed}} \times 100$$

Note: Factor of four accounts for 3 moles urea to ammelide, plus 1 mole water from ammelide formation to hydrolyze 1 mole urea.

3. $$Y_U \text{ to Hydrolysis} = \frac{\text{Moles Water in Feed}}{\text{Moles Urea Consumed}} \times 100$$

4. $$Y_U \text{ to Vaporization} = 100\% - (Y_U \text{ to GN} + Y_U \text{ to A} + Y_U \text{ to Hydrolysis})$$

The above urea yields are accurate and legitimate only within a specified AN/U feed mole ratio range as discussed in a subsequent section. These expressions assume (1) that the 2-mole urea to GN stoichiometry is valid for all cases, (2) that all water present hydrolyzes urea, and (3) urea is consumed only by the reactions noted.

I <u>Urea Hydrolysis Efficiency (E_{UV})</u> - The Δ urea equivalent noted above (F) represents the urea equivalents that have left the product melt. The two routes by which this loss can occur are hydrolysis to ammonia and carbon dioxide and loss to vaporization. The moles of Δ urea equivalents divided by the moles of water available (initial water in the feed, water from GN formation, and water from ammelide formation) represents the efficiency of that water for hydrolyzing urea. Subsequent results will show that the accuracy of the two-mole urea to GN stoichiometry is limited to a specific AN/U feed mole ratio range.

J <u>Pounds Urea Used/Pound GN Produced</u> - "In the barrel" requirements were calculated based on the urea usage and guanidine nitrate produced. If the 2-mole urea equation is valid and if a 100% urea to GN yield is achieved, then the theoretical "in-the-barrel" urea requirements would be 0.984 lb U/lb GN.

K <u>Calculated Reactor Feed Rate</u> - On the basis of nitrate conservation, the total reactor feed rate was calculated by dividing the measured reactor product rate by the P/F ratio.

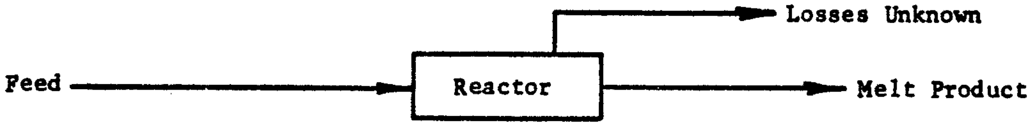
L <u>Plant Productivity</u> - The theoretical plant productivity was calculated by determining the difference between the pounds of GN in the reactor product and reactor feed on a 24-hour basis; i.e.,

$$\text{GN Produced} = \frac{\text{lb reactor melt}}{\text{hr}} \times \frac{\%\ \text{GN}}{100} \times 24\ \text{hrs}$$

$$- \frac{\text{lb calc. feed}}{\text{hr}} \times \frac{\%\ \text{GN}}{100} \times 24\ \text{hrs}$$

Sample calculations for one set of data are presented below:

Sample Data: 11:30 a.m., 6/14/73



Feed:

a) Composition (Wt %):

AN - 54.60
U - 37.10
GN - 7.02
H_2O - 1.02

b) Rate - Unknown

Melt Product:

a) Composition (Wt %):

AN - 50.90
U - 18.40
GN - 29.40
Ammelide - 0.26

b) Rate = 129/hr

A. Moles Components/100 lb Feed:

$$AN = \frac{54.60}{80} = 0.6843$$

$$U = \frac{37.10}{60} = 0.6199$$

$$GN = \frac{7.02}{122} = 0.0577$$

$$H_2O = \frac{1.02}{18} = 0.0560$$

B. Moles Components/100 lb Product:

$$AN = \frac{50.90}{80} = 0.6429$$

$$U = \frac{18.40}{60} = 0.3099$$

$$GN = \frac{29.40}{122} = 0.2435$$

$$\text{Ammelide} = \frac{0.26}{124} = 0.0021$$

C. AN/U Feed Mole Ratio

0.6843/0.6199 = 1.104 moles AN/mole U

D. Product/Feed Weight Ratio (P/F)

$$P/F = \frac{\text{Moles Nitrates}}{\text{100 lb Feed}} \; / \; \frac{\text{Moles Nitrates}}{\text{100 lb Product}}$$

$$= (0.6843 + 0.0577)/(0.6429 + 0.2435)$$

$$= 0.7420/0.8864$$

$$= 0.8371$$

E. Moles Product Components/100 lb Feed

$$P/F \times \frac{\text{Moles AN, etc.}}{\text{100 lb Product}}$$

1. AN = 0.8371 x 0.6429 = 0.5382
2. U = 0.8371 x 0.3099 = 0.2594
3. GN = 0.8371 x 0.2435 = 0.2038
4. Ammelide = 0.8371 x 0.0021 = 0.0017

F. Moles Urea Equivalent

1. Feed = U + GN
   = 0.6199 + 0.0577
   = 0.6776 moles urea equivalents

2. Product (P/F corrected) = U + GN + 3 ammelide
   = 0.2594 + 0.2038 + (3)(0.0017)
   = 0.4684 moles urea equivalents

G. Moles GN Produced/100 lb Feed

GN Produced = (Moles GN/100 lb Product)(P/F)-(Moles GN/100 lb feed)
= (0.2435)(0.8371) - 0.0577
= 0.1461 moles GN/100 lb Feed

H. Urea Yields

1. $$Y_U \text{ to GN} = \frac{\text{0.1461 moles GN Produced}}{(0.6199 - 0.2594)/2} \times 100 = 81.1\%$$

2. $$Y_U \text{ to A} = \frac{\text{0.0017 moles Ammelide}}{(0.6199 - 0.2594)/4} \times 100 = 1.9\%$$

3. $$Y_U \text{ to Hydrolysis} = \frac{\text{0.0568 moles H2O in Feed}}{0.3605} \times 100 = 15.8\%$$

4. $$Y_U \text{ to Vaporization} = 100\% - (81.1\% + 1.9\% + 15.8\%) = 1.2\%$$

I. Urea Hydrolysis Efficiency (E_{UV})

$$E_{UV} = \frac{\Delta \text{ Urea Equivalents (Excess of U, GN, Ammelide)}}{\text{Total H2O (Feed + GN Reaction + Ammelide Reaction)}} \times 100$$

$$= \frac{0.6776 - 0.4684}{0.0568 + 0.1461 + 0.0017} \times 100$$

$$= 102\%$$

J. Pounds Urea Used/Pound GN Produced

lb U/lb GN =

$$\frac{(0.6199 \text{ moles U in feed} - 0.2594 \text{ mole U Product}) \times 60 \text{ lb U/mole}}{0.1461 \text{ moles GN produced} \times 122 \text{ lb GN/Mole}}$$

$$= 1.214 \text{ lb Urea consumed/lb GN produced}$$

K. Calculated Reactor Feed Rate (R_F)

$$R_F = \frac{\text{Reactor Product Rate}}{\text{P/F ratio}}$$

$$= \frac{129 \text{ lb/hr}}{0.8371}$$

$$= 154 \text{ lb Feed/hr}$$

L. Calculated Plant Productivity

$$\text{Productivity} = \left[\left(\text{Reactor Melt Rate} \times \frac{\%\text{ GN}}{100}\right) - \left(\text{Calc. Feed Rate} \times \frac{\%\text{ GN}}{100}\right)\right] 24$$

$$= \left[(129 \text{ lb/hr} \times 0.294) - (154 \text{ lb/hr} \times 0.0702)\right] 24$$

$$= 651 \text{ lb GN/day}$$

Examination of the results in Table 27 plus plots of some of the calculated results points to the following general conclusions:

- Urea yield to GN can exceed 100% when based on the 2-mole urea to GN stoichiometry.

The basis of the 2-mole stoichiometry is that one mole of urea is converted to GN while the remaining mole of urea is hydrolyzed to NH_3 and CO_2. Water for this hydrolysis is a product of the GN formation, i.e., one mole of water per mole of GN. If conditions are such that less than 2 moles of urea are consumed per mole of GN produced, then the urea yield to GN can be greater than 100%. This apparently is the case in some instances. Urea hydrolysis efficiency (E_{UH}) is plotted versus the AN/U feed mole ratio in Figure 8. As can be seen from this graph, the urea hydrolysis efficiency is less than 100% at AN/U values > 1.5. This means that less than the theoretical amount of urea is consumed for the corresponding GN production.

At AN/U values < 1.0, the calculated hydrolysis efficiency is greater than 100%. Values greater than 100% (urea consumed greater than theoretical) are possible if there is complete urea hydrolysis plus an additional urea loss, e.g., urea vaporization. Interpretation of Figure 8 leads one to the following conclusions:

- AN-rich reactor feeds result in incomplete hydrolysis of urea.
- Urea-rich reactor feeds result in complete hydrolysis of urea plus additional urea losses.

The urea yield to GN (Y_U - GN) is plotted versus the AN/U feed ratio in Figure 9. The resulting curve supports the above conclusions. At low AN/U ratios, or conversely in the urea-rich regime, urea yields as low as 50% result. At AN/U ratios of 1.0-1.5, yields of 80% to 95% result and at AN/U ratios > 1.5, yields of 100% or greater are possible. Figures 8, 9 and 10 contain the data from Table 27 as well as data points from prior experiments, i.e., 1-in., 2-in., and 4-in. single-tube runs presented in Final Report, Volume I.

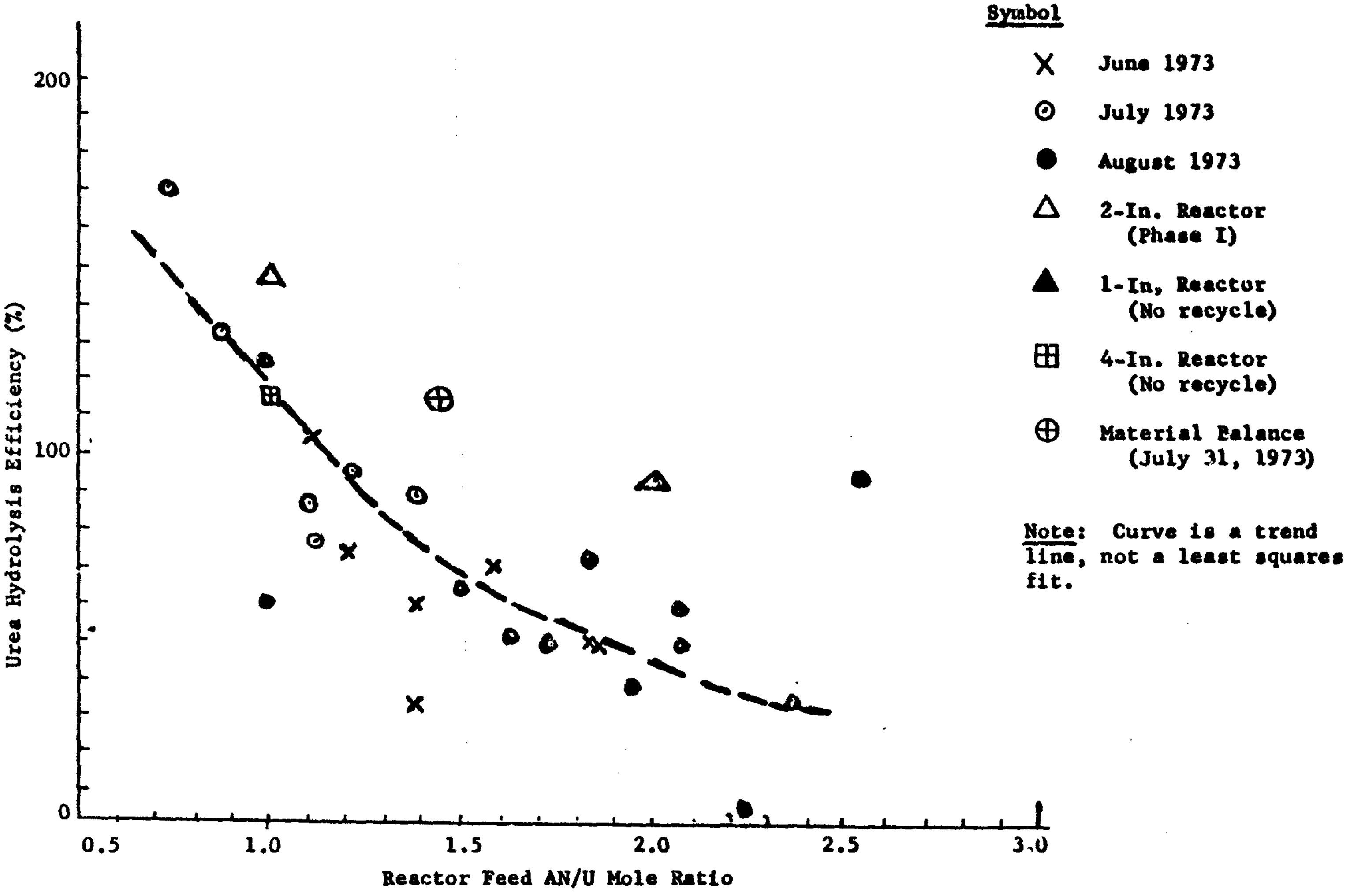
In Figure 10, the pounds of urea consumed per pound of GN made is plotted versus AN/U feed ratio. The trend of these data shows that less urea is required to make guanidine nitrate as the AN/U ratio increases. This conclusion is consistent with an increasing yield at higher AN/U ratios.

It should be noted that the urea vaporization at low AN/U ratio agrees with the stability studies of Mackay. He showed that as much as 57% urea would be lost to vaporization at an AN/U ratio of 0.5. This loss percentage decreases rapidly as the mixture becomes richer in AN.

3. Material Balances

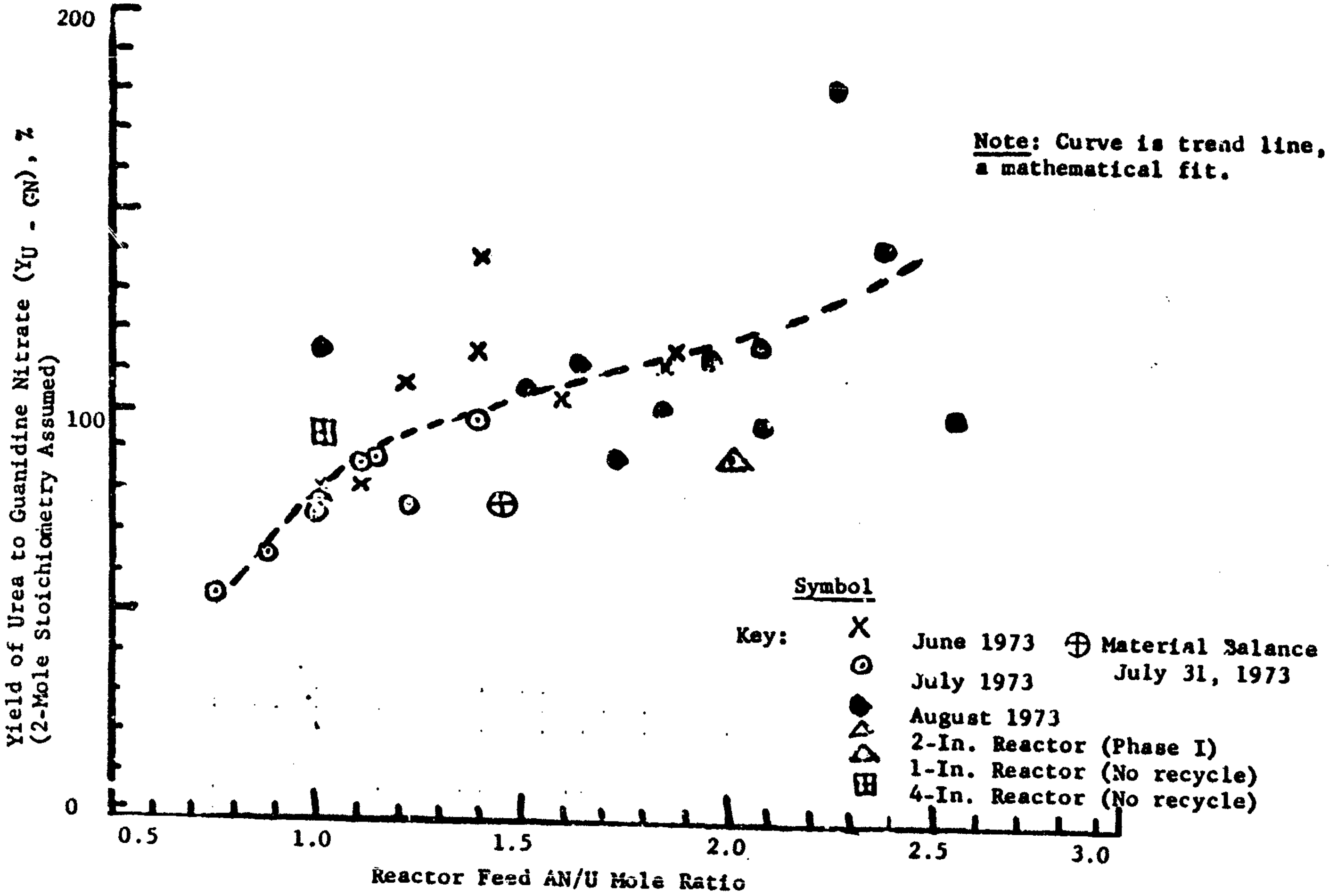
As shown in the previous section, the reactor performance can be quickly estimated using the product melt rate, feed and reactor product analyses, and the nitrate conservation assumption. To rely on this method, it had to be shown that the same reactor performance is achieved when a detailed total material balance is performed. In addition to being a check on the quick calculation procedures, the material balance is helpful in determining the sources of process losses, and the potential "in-the-barrel" yield.

In this regard, a number of material balances were conducted during the production campaign. The most pertinent ones were the following:



Note: Curve is a trend line, not a least squares fit.

Figure 8. Urea Hydrolysis Efficiency Versus Ammonium Nitrate/Urea Ratio



Note: Curve is trend line, not a mathematical fit.

Figure 9. Urea Yield to Guanidine Nitrate Versus Feed Ammonium Nitrate/Urea Ratio

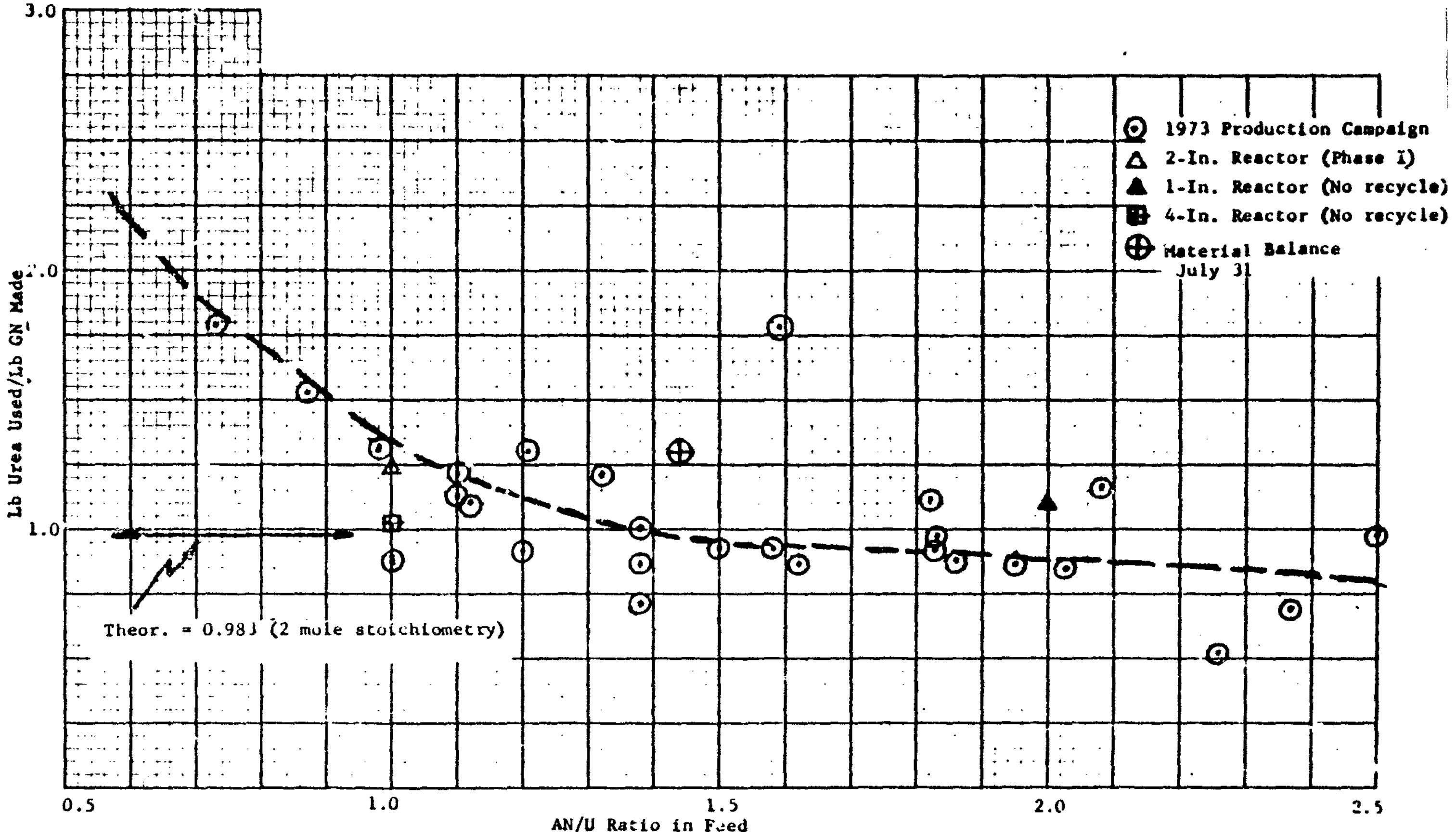


Figure 10. Pounds Urea Used/Pounds Guanidine Nitrate Made Versus Feed Ammonium Nitrate/Urea Ratio

a) Total product and total reagents for the entire run ("in-the-barrel" yields)

b) Total and individual process step balance for July 10-11, 1973 (7 operating tubes and total recycle)

c) Reactor-scrubber balance of July 31, 1973 (3 operating tubes)

d) Total weight balance of reactors and scrubber on August 10, 1973 (2 operating tubes)

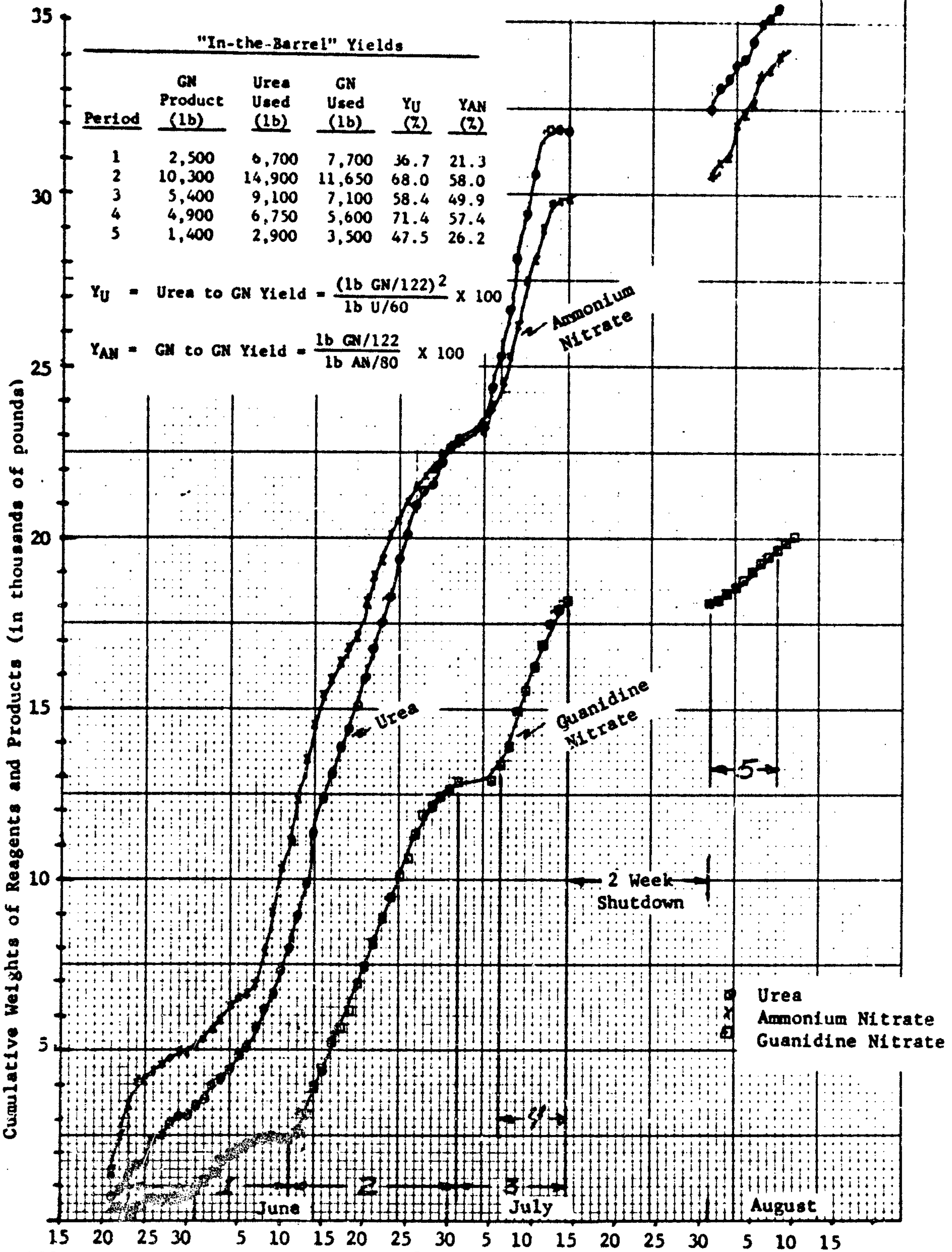
In addition to the above, other balances were attempted to duplicate some of the above data, but either process or analytical problems limited their value. In these runs, however, useful confirming data were obtained.

a. "In-the-Barrel" Yields

Cumulative weights of urea and ammonium nitrate charged to the pilot plant system and guanidine nitrate recovered (on an "as-analyzed" basis) are plotted in Figure 11 as a function of date. "In-the-barrel" yields of GN from urea and ammonium nitrate were calculated for five different time periods as follows:

Time Period	Time Interval	Remarks
1	5/23-6/11/73	Followed start-up of plant, one to four reactors on stream, included periods of high feed water and high urea product from three reactors lost to NH_3-H_2O tank for three days.
2	6/11-7/1/73	Period of fairly stable operations, four to eight reactors onstream, reactors shut down as a result of ammelide build-up problem.
3	7/1-7/14/73	Four to seven reactors operating. Fairly steady operations. Period of high urea in feed. Plant down three days because of the above ammelide problem.
4	7/6-7/14/73	Same as Period No. 3 except elimination of three-day downtime.
5	7/31-8/8/73	Operating period following two-week scheduled shutdown. Two and three reactors on stream.

Figure 11. Cumulative Plot of Reactants Used and Guanidine Nitrate Produced Versus Date



"In-the-Barrel" Yields

Period	GN Product (lb)	Urea Used (lb)	GN Used (lb)	Y_U (%)	Y_{AN} (%)
1	2,500	6,700	7,700	36.7	21.3
2	10,300	14,900	11,650	68.0	58.0
3	5,400	9,100	7,100	58.4	49.9
4	4,900	6,750	5,600	71.4	57.4
5	1,400	2,900	3,500	47.5	26.2

$$Y_U = \text{Urea to GN Yield} = \frac{(\text{lb GN}/122)^2}{\text{lb U}/60} \times 100$$

$$Y_{AN} = \text{GN to GN Yield} = \frac{\text{lb GN}/122}{\text{lb AN}/80} \times 100$$

Calculated "in-the-barrel" yields for the above time periods are tabulated below:

Time Period	Urea to GN[1] Yield (%)	AN to GN Yield (%)
1	36.7	21.3
2	68.0	58.0
3	58.4	49.9
4	71.4	57.4
5	47.5	26.2

(1) Based on 2-mole stoichiometry

These "in-the-barrel" yields, particularly those for ammonium nitrate, are disappointingly low. Several sources of material losses (discussed below) were uncovered which would account for some of the yield losses. The highest yields were obtained during periods of stable operations with most of the reactors operating and when the feed urea concentration was not excessive (i.e., AN/U ratio > 1.0). With fixed or constant process losses, this effect would be magnified with fewer reactors on stream. Process material losses were occurring in the following pilot plant locations, and attempts to eliminate these losses were not totally satisfactory: (1) evaporator vent, (2) evaporator feed pump packing gland, (3) reactor feed pump packing gland, (4) crystallizer polishing filter changes, (5) entrainment from the gas-liquid separator to the off-gas scrubber, (6) spillage from the GN centrifuge, and (7) GN and equivalents in the ammelide cake.

The major source of material losses was the evaporator feed pump packing gland. A measured leak rate showed a loss of about 1 gal liquid per hour which is equivalent to about 11 lb of 60% solids (typical evaporator feed) per hour or 158 lb of total solids lost from the system per day. "In-the-barrel" AN and U yields of about 70% are equivalent to a daily loss of about 250 lb of combined feed make-up. Consequently, the evaporator feed pump loss represented about 60% of the total missing material or about a 14% AN yield loss. The high concentration of ammonium nitrate in the evaporator feed stream explains why "in-the-barrel" AN yields were lower than urea yields. With a fixed loss, there is a significant and detrimental effect on pilot plant yield as the number of on-stream reactors decreases.

During a two-day pilot plant material balance (discussed in detail in a subsequent section) where all leaks, etc., were collected and returned to the system, a total material balance closure of 101% was achieved. Acceptable AN and U yields to GN were also demonstrated.

Two tests were conducted to determine if material was being lost from the top of the evaporator. The details of these tests and the resultant loss calculation will be discussed in a later section of this report. In summary, it was found that about 25-30 lb/day of AN and U (1-1/2% of the recycle feed) were volatilized in the evaporator off-gas. This type of loss is perhaps typical for a pilot plant and would be less, percentage-wise, in a commercial-size evaporator. The pilot plant evaporator, because of its size, contained two air-swept stages, whereas a two-stage commercial unit would have only one air-swept stage. In addition to this, the pilot plant evaporator during the test periods was being operated at higher temperatures to overcome tube fouling. The resulting hotter air would have increased the volatile loss.

The entrainment loss of reactor melt to the scrubber was approximately 25-30 lb/day which will be discussed in a later section. The other losses noted were minor t their discovery and accountability contributed to the tightening of the system.

b. Two-Day Total and Individual Process Step Material Balance - July 10-11, 1973

After the losses noted above had been discovered and either corrected or accounted for, a two-day material balance was made with the plant operating at 85% capacity and with full recycle. The balance covered a 46-hour period and resulted in a weight closure of 100.7%, an ammonium nitrate yield of 97-105% and a urea yield of 79%. Details of the calculations and results are presented in Table 28. Raw data for the material balance are presented in Table 29.

This balance was not optimum in that it was necessary to make some assumptions. The holdup in the system was larger than the material fed into the system, and the material balance period was not one of particularly smooth operation. There was approximately a 5000 lb solids holdup in the process vessels during the material balance period while only about 3542 lb of reagents were fed in. This was less than a one volume turn-around so that a changing composition and/or a changing level in a large tank was a significant term in the material balance accounting. Theoretically, the material balance should have been conducted over many residence times so that the only significant terms would have been the reagent and product weights (both accurate measurements). The pilot plant operation could not be maintained in a smooth, trouble-free fashion long enough to guarantee this. In the actual 46-hour run, there were mechanical problems with the Hills-McCanna feed blend pump, the evaporator and the scrubber. The blend pump was skipping on one head, allowing the AN/U ratio feed to the reactors to float. Accurate analyses were necessary to determine the accumulation of feed reagents in the system. The evaporator was not operating at full efficiency because of suspected heat transfer surface fouling. The result was a variable water content in the feed that would contribute to a changing product composition.

The scrubber was frequently plugged and had to be prodded continuously to maintain reactor gas flow to the scrubber. It was later discovered that the scrubber had a cake buildup (comprising ~75% of the cross-sectional area) which was the source of the plugging. On analysis, the

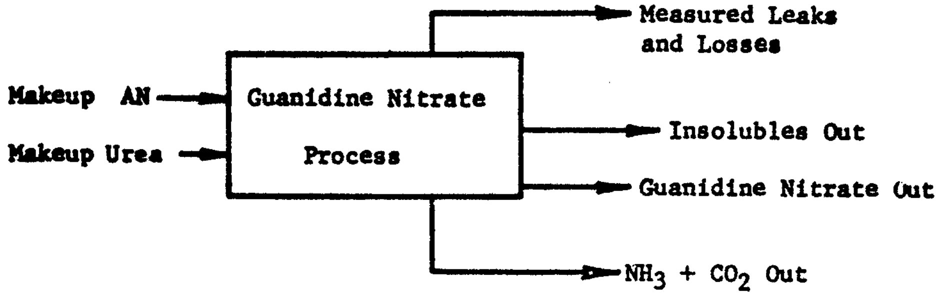
TABLE 28

CALCULATIONS FOR TWO-DAY MATERIAL BALANCE - JULY 10-12, 1973

Period: 5:30 p.m., 7/10 - 3:30 p.m., 7/12/73

Elapsed Time: 46 hours

I. TOTAL BALANCE SUMMARY



Input - Output = Accumulation

Input = Accumulation + Output

1. Input

AN	1,342 lb
Urea	2,200 lb
Total Input	3,542 lb

2. Output

Insolubles	55 lb
Guanidine Nitrate Product	1,247 lb *
$NH_3 + CO_2$	1,185 lb
Measured Leaks	24 lb
Assumed Losses:	
Volatiles from Evap.	30 lb
Entrainment Into Scrubber	50 lb
	2,589 lb

*1,212 lb. GN (100% Basis)

TABLE 28 (cont.)

3. **Accumulation**

As Level and Concentration Changes:

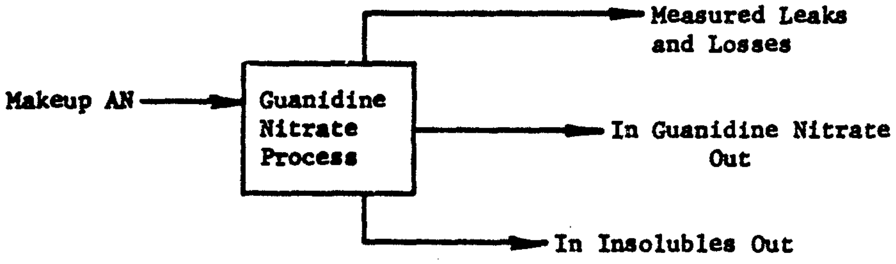
T103	-88 lb
T104	+178 lb
T105	+13 lb
T106	+11 lb
T107	+836 lb
T113	+28 lb
	+978 lb

4. **Total Balance**

$$\text{Closure} = \frac{\text{Output} + \text{Accumulation} \times 100}{\text{Input}} = \frac{2589 + 978 \times 100}{3542} = \frac{3567}{3542} \times 100$$

Closure = 100.7%

II. AMMONIUM NITRATE BALANCE



Input = Accumulation + Output

1. Input

AN = 1,342 lb.

2. Output

As AN in GN	25 lb
As GN $(1212 \times \frac{80}{122})$	795 lb
As AN in Insolubles	14 lb
As Measured Leaks	13 lb
As Assumed Losses	
Volatiles from Evap.	25 lb
Entrainment	30 lb
	902 lb

TABLE 28 (cont.)

3. Accumulation

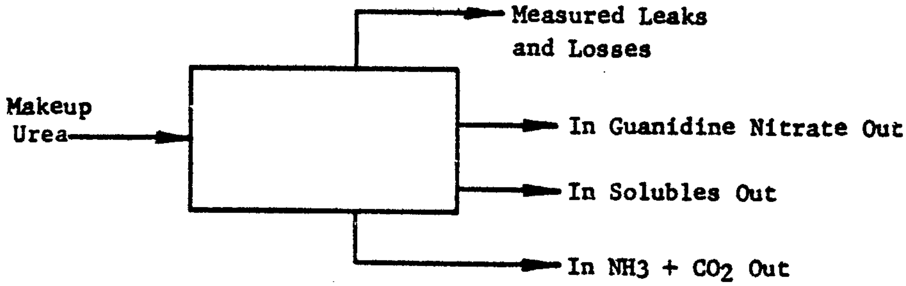
As Level and Composition Changes:

An equivalent of GN in all Tanks $(137 \times \frac{80}{122})$	+90 lb
T103	-33 lb
T104	+90 lb
T105	- 3 lb
T106	-123 lb
T107	+462 lb
T113	-3 lb
	+480 lb

4. $$\text{AN Closure} = \frac{\text{Output + Accumulation}}{\text{Input}} \times 100$$

$$\text{AN Closure} = \frac{90.2 + 480}{1342} \times 100 = \frac{1382}{1342} \times 100 = \underline{\underline{103.0\%}}$$

III. UREA BALANCE



1. Input

Urea = 2,200 lb

TABLE 28 (cont.)

2. Output

As Urea in GN	+10 lb
As GN (1212 x 60/122)	+596 lb
As $NH_3 + CO_2$	+1,185 lb
As Measured Leaks	+7 lb
As Urea in Insolubles	+4 lb
As Insolubles (11 x 180/128)	+15 lb
As Assumed Losses	
Volatiles from Evap.	+5 lb
Entrainment Into Scrubber	+10 lb
	+1,832 lb

3. Accumulation

As Level and Composition Changes:

GN Levels (137 x 60/122)	+67 lb
T103	-55 lb
T104	+71 lb
T105	+8 lb
T106	+131 lb
T107	+300 lb
T113	+31 lb
	+553 lb.

4. Urea Closure

$$\text{Urea Closure} = \frac{\text{Output + Accumulation}}{\text{Input}} \times 100 =$$

$$\frac{1832 + 553}{2200} \times 100 = \underline{\underline{108.4\%}}$$

TABLE 28 (cont.)

IV. YIELDS

1. Guanidine Nitrate Made

GN Made = Output + Accumulation

a.

Output as GN	1,212 lb
As GN in Insoluble Cake	30 lb
	1,242 lb

b. Accumulation

As level changes =

T104	+17 lb
T105	+8 lb
T106	+3 lb
T107	+74 lb
T113	+1 lb
As Measured Leaks:	+4 lb
As Assumed Losses:	+10 lb
	+117 lb

c. GN Made = 1242 + 117 lb. = 1359 lb

2. Urea Yields

a. Urea Usage

Urea Usage = Input - Output - Accumulation

(1) Input = 2,200 lb

(2) Output

As urea in GN	10 lb
As Measured Leaks	7 lb
As urea in Insolubles	4 lb
As Assumed Losses	
Volatiles from Evap.	5 lb
Entrainment	10 lb
	+36 lb

(3) Accumulation +486 lb

Urea Usage = 2,200 - 36 - 486 = 1678 lb

TABLE 28 (cont.)

b. Urea Yield to GN

$$Y_{U1}^{(1)} = \frac{\text{Mole GN Made}}{\text{Moles U Used}} \times 100 = \frac{1359\ \text{lb}\ /122}{1678\ \text{lb}\ /60} \times 100 + \frac{11.14}{27.97} \times 100$$

$$= 39.8\%$$

(1)No credit assumed for NH_3 and CO_2

$$Y_{U_2}^{(2)} = 39.5\% \times 2 = \underline{\underline{79.6\%}}$$

(2)Assumed 2-mole urea stoichiometry

3. AN Yield

a. AN Usage

AN Usage = Input - Output - Accumulation

(1) Input	1,342 lb
(2) Output	
As AN in GN	25 lb
As AN in Insolubles	14 lb
As Measured Leaks	13 lb
As Assumed Losses:	
Volatiles from Evap.	25 lb
Entrainment Into Scrubber	30 lb
	107 lb
(3) Accumulation	390 lb

AN Usage = 1,342 - 107 - 309 lb. = $\underline{\underline{845}}$ lb

b. AN Yield to GN (Credit to Loss Sources)

$$Y_{AN} = \frac{\text{Moles GN Made}}{\text{Moles AN Used}} \times 100 = \frac{1359\ \text{lb}\ /122}{845\ \text{lb}\ /80} \times 100 = \frac{11.14}{10.56} \times 100$$

Y_{AN} = $\underline{105.5\%}$ (loss credit)

TABLE 28 (cont.)

c. AN Yield to GN (No credit to loss sources)

(1) Input	1,342 lb
(2) Output	
As AN in GN	25 lb
As AN in Insolubles	14 lb
	39 lb
(3) Accumulation	390 lb

AN Usage = 1,342 - 39 - 390 = 913 lb

$$Y_{AN} \text{ (no loss credit)} = \frac{11.14 \text{ moles}}{913/80} = \frac{11.14}{11.41} = \underline{\underline{97.6\%}}$$

4. Yield Losses

a. Yield Loss to Insolubles

(1) Measured Insolubles Made

11 lb /128 = 0.086 mole

(2) Measured GN Made

1359 lb /122 = 11.14 moles

(3) % Loss to Insolubles

$$\frac{0.086 \text{ Mole Insoluble}}{11.14 \text{ Moles GN}} \times 100 = \underline{\underline{0.77\%}}$$

(4) Urea Yield Loss to Insolubles

$$Y_I(\text{Urea}) = \frac{\text{Insolubles Equivalents}}{\text{Urea Usage}} \times 100$$

$$= \frac{0.086 \text{ Mole} \times 3 \text{ Moles U/Insol}}{27.97 \text{ Moles Urea Used}} \times 100$$

$$= \frac{0.258}{27.97} \times 100 = \underline{\underline{0.93\%}}$$

TABLE 28 (CONT.)

b. Urea Yield Loss to Hydrolysis from Feed Water

(1) Measured Water in Feed

$$\text{Moles } H_2O \text{ In Feed} = \frac{(1.5\%/100) \times (140 \text{ lb/hr feed}) \times (46 \text{ hr})}{18 \text{ lb/mole}}$$

$$= 5.37 \text{ moles}$$

(2) Moles Urea Hydrolyzed from Feed Water

From Figure 8, E_{UH} = 100%

$$Y_U \text{ to Hydrolysis} = \frac{5.37 \times 100\%}{27.97 \text{ moles Urea Used}}$$

Y_U to Hydrolysis = $\underline{\underline{19.2\%}}$

V. INTERMEDIATE CALCULATIONS

1. Off-Gas Generation

Assuming 2 mole equation + complete Urea hydrolysis

a. By Reaction

$$\text{Moles Ammonium Carbamate (AC) Formed} = \left(\frac{1359}{122}\text{Moles GN}\right) \times \frac{1 \text{ Mole AC}}{\text{Mole GN}}$$

$$= 11.13 \text{ Moles AC}$$

Wt. AC Formed = $\underline{\underline{868.0 \text{ lb}}}$

b. By Feed H_2O

Avg. % Water In Feed = 1.5%
Avg. Feed Rate to 7 Tubes = 140 lb/hr
Avg. lb/hr of H_2O in Feed = 2.1 lb/hr
Total lb Water in Feed = 96.0
Total Moles of H_2O Fed = 5.33

Total Wt. of Urea Potentially Hydrolyzed = 320 lb

Maximum Urea Loss as $NH_3 + CO_2$ = 868 + 320 = 1188 lb

Urea Hydrolysis Efficiency (at AN/U = 1.0) = 100%
(See Figure 8)

Assumed urea Off-Gas Loss = $\underline{\underline{1188 \text{ lb}}}$

TABLE 28 (CONT.)

2. Productivity

a. Per Day

$$PN = \frac{\text{lb GN Made}}{\text{Total Hr}} \times 24$$

$$PN = \frac{1359 \text{ lb} \times 24}{46} = 709 \text{ lb GN/Day}$$

b. Per Hour/Tube

$$\text{Prod./Hr/Tube} = \frac{709 \text{ lb/Day}}{24 \text{ hr/Day} \times 7 \text{ Tubes}} = 4.22 \text{ lb GN/Hr/Tube}$$

VI. SUMMARY OF RAW DATA FOR MATERIAL BALANCE

(See Table 29)

INVENTORY CHANGES

Vessel	Total Change of AN, U, GN	UREA	AN	GN
T103	-88	-55	-33	--
T104	+178	+71	+90	+17
T113	+29	+31	-3	+0.6
T105	+13	+8	-3	+8
T106	+11	+131	-123	+3
T107	+836	+300	+462	+74
Solid Bowl	+48	+4	+14	+30
P102 Seal	+14	+5	+9	--
Product Out	+1247	+10	+25	+1212
Feed In	-3542	-2200	-1342	--
Spills	+10	+2	+4	+4

TABLE 29

RAW DATA FOR JULY 10-12, 1973, MATERIAL BALANCE -1

Equipment	Period	Level	Calibration Gal/in.	Analyses, % AN	U	GN	H_2O	Insol.	Notes	
T100	Start	Heel + 1 Charge	---	37.9	62.11	---	---	---	+2200 lb U	
	Finish	Heel + 1 Charge		37.9	62.11	---	---	---	+1342 lb AN (Fresh Additions)	
T101	Start	Heel + 1 Charge	---	80.0	20.0	---	---	---	-----	
	Finish	Heel + 1 Charge		80.0	20.0	---	---	---	-----	
T103	Start	10-7/8 inches	2.0	37.9	62.1	---	---	---	-----	
	Finish	7 inches		37.9	62.1	---	---	---	-----	
T104	Start	19-1/4 inches	2.0	69.8	19.0	13.5	3.9	---	-----	
	Finish	26-3/4 inches		64.8	25.3	8.0	1.9	---	-----	
T105	Start	8 inches	---	37.05	8.45	19.5	--- *	---	-----	
	Finish	8 inches		31.73	15.52	20.25	--- *	---	-----	
									ρ(g/cc)	T.S.%
T106	Start	25 inches	5.0	37.05	8.45	19.5	--- *	---	1.23	65
	Finish	25 inches		31.73	15.52	20.25	--- *	---	1.24	67.5
T107	Start	18 inches	5.0	39.9	11.9	8.2	40.0	---	1.22	60
	Finish	41 inches		38.8	19.2	7.0	35.0	---	1.246	65.0
T113	Start	12.5 inches		57.5	35.5	5.0	1.9	---	-----	
	Finish	15 inches	1.0	46.3	47.9	4.5	1.1	---	-----	
Solid Bowl	-----	-----	-----	18.9	5.4	40.5	20.3	14.9	74 lb removed	
GN Product	Batches 239-248	----	-----	91.0	1.9	0.7	6.4	----	1332 lb Wet GN out	

*Based on melt analyses and density changes

TABLE 29 (Cont.)

RAW DATA FOR JULY 10/11 MATERIAL BALANCE -2

Equipment	Quantity	Analyses, %	Notes
Feed Pump Packing Leak	43 Gal	2.4% AN, 1.4% U, 0.08% GN	-----
Spills Measured			
(a) Crystallizer Feed	9 lb	-----	Same Analyses as T106
(b) Wet GN	2 lb	-----	Same Analyses as Product
(c) T-103	1 lb	-----	Same Analyses as T103
(d) Reactor Vent	2 lb	-----	Same Analyses as Melt
Scrubber Water	6146 lb	-----	Assumed AN = 1.3%
Reactors R200-203 R205 - 207 Evaporator Scrubber	No Change During Balance	-----	In operation
Crystallizer T102 T109	Empty at Start and Finish of Balance	-----	-----

cake was determined to be primarily ammonium carbonate which presumably had built up gradually. The effect of the erratic scrubber operation was that the composition of the ammonia-water made from the off-gas fluctuated and could not be determined precisely during the material balance.

Assumptions that had to be made to calculate the material balance were the following:

a) Urea hydrolysis efficiency
b) Composition of the crystallizer feed tank contents
c) Losses from the system through entrainment and volatilization.

As discussed in a previous section, the efficiency of urea hydrolysis is apparently a function of the AN/U feed mole ratio (see Figure 8). For the two-day material balance, the average AN/U feed mole ratio to the reactors was about 1/1. At this ratio, all the water in the reactor (both from feed input and from the reaction) was assumed to have hydrolyzed urea.

As noted in an earlier paragraph, the closure of this material balance was strongly dependent on tank analyses. The largest contributors were the crystallizer feed tank (T-106) and the evaporator feed tank (T-107). Unfortunately, the analyses of T-106 were the least accurate. Samples taken hot would crystallize before analysis. Obtaining a uniform sample of the resultant slurry proved to be difficult. This situation was corrected for subsequent balances by making a known dilution of the samples from T-106 while hot. For the two-day balance, the T-106 analyses were derived using product melt analyses and the water content of the tank via both a density measurement and by the water/melt ratio in the quench tank (T-105). This is an indirect method but is rigorous.

As will be shown in a future section of this report, there were losses of melt to the scrubber by entrainment and losses of AN and U by volatilization from the evaporator. For the two-day balance, weight losses of these streams were assumed to be consistent with the percentages measured in individual tests.

As calculated in Table 28, the urea yield to guanidine nitrate, assuming the two-mole stoichiometry, was 79%. The missing 21% is attributed to losses to insolubles (1-2%) and to hydrolysis of urea from water in the feed (18-20%). Since the water in the feed was higher than designed (due to evaporator problems during the balance), it is reasonable to expect that the loss of urea due to feed water hydrolysis would be less in a production plant. If the feed water level were 0.5% instead of 1.5%, a urea yield to GN of about 92% would have been achieved. This yield value and water level were demonstrated (based on spot analyses) during previous periods of operations.

c. Reactor-Scrubber Balance of July 31, 1973

In the 2-day material balance discussed above, assumptions were made regarding materials lost as off-gas and liquid entrainment from the reactors. To show the validity of these assumptions, balances were made over the reactor-scrubber system on two different occasions. The discussion which follows pertains to a balance conducted on August 2, 1973 for one hour. Since both the reactor and scrubber are continuous operations and with constant steady state holdups, a balance measuring only flow rates should be sufficient. Figure 12 shows the process conditions during this balance and Table 30 shows the calculations and results.

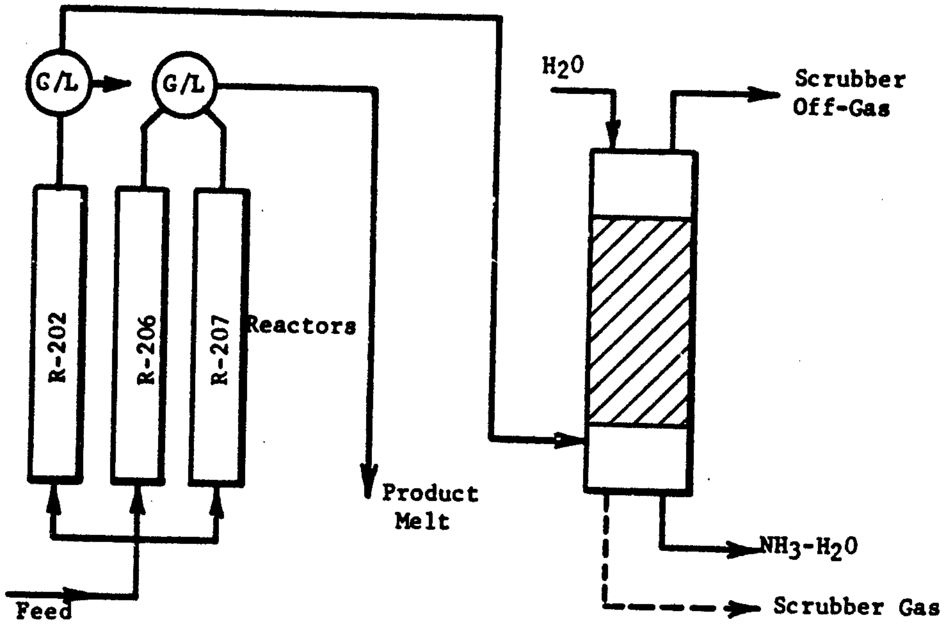
The results in Table 30 were encouraging in that essentially all of the ammonium nitrate and urea fed to the reactor-scrubber system was accounted for, i.e., 100.0% AN closure and 94.9% urea closure. Ammonia and carbon dioxide closures were calculated on the basis of predicted quantities versus actual measured quantities of the two materials. The CO_2 closure was 102.1% while the NH_3 closure was 98.1%. These results confirm the assumption that one mole of ammonium carbamate (decomposed to free $2NH_3$ and CO_2) is produced for each mole of guanidine nitrate produced, particularly at the experimental AN/U molar ratio of 1.44.

Ammonium nitrate and urea yields to GN are also shown in Table 30 for the reactor-scrubber system. The actual calculated AN-to-GN yield was 93.3% while the AN yield corrected for entrainment loss was 100%. This represents a 6.7% AN yield loss to entrainment from the gas-liquid separator. The actual urea-to-GN yield was 75.6%; however, yield losses due to water-in-feed hydrolysis, ammelide formation and entrainment were 16.4%, 1.97% and 1.79%, respectively. Total urea yield accounted for was 95.76%. Through proper design of equipment and control of operating variables, AN and urea yields of 100% and 90%, respectively, are envisioned for a production plant. It should be noted that the economic analysis (Section I) used conservative values.

d. Total Weight Balance Over Reactor-Scrubber System for August 9 and 10

In the reactor-scrubber balance of July 31 discussed above, the nitrate balance was again assumed to determine the feed rate to the reactors. To confirm these assumptions, a balance was made on August 9 and 10 using two operating reactors. All of the feed and product were weighed. Table 31 shows the details of this balance. The analytical data for this run consisted of a series of eight feed and product samples, from which averages were derived. Table 32 shows the multiple sets of analyses. The average AN/U feed mole ratio for this run was 1.4/1.

As Table 31 indicates, the accuracy of the feed rate calculated using the nitrate conservation assumption was within 1% of that measured.



<u>Time of Balance</u>: 3-4 p.m., 7/31/73

<u>Measured Rates</u>

Product Melt = 55 lb/hr
NH_3-H_2O = 117 lb/hr

Scrubber Off-Gas:
Top = 9.33 liters/minute
Bottom = 8.40 liters/minute*

<u>Measured Analyses</u>

Feed (1S-3, 3:30 p.m.) - 62.5% AN, 32.6% U, 4.1% GN, 0.99% H_2O
Product (2S-3, 3:30 p.m.) - 61.9% AN, 13.9% U, 24.1% GN, 0.26% Insol.
NH_3-H_2O - 1.81% $(NH_4)_2CO_3$, 2.94% NH_3, 0.42% NO_3 as AN
Scrubber Off-Gas - 96.4% CO_2, < 0.1% Argon, 0.5% O_2, 2.2% CO + N_2, 0.8% H_2O, 0.1% max as N_2O

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*Estimated from separate experiment.

Figure 12. Reactor-Scrubber Balance of 7/31/73
(Refer to Table 30 for Calculations)

TABLE 30

CALCULATIONS FOR REACTOR-SCRUBBER BALANCE - 7/13/73
(Refer to Figure 12)

I. CALCULATION OF REACTOR FEED RATE

Assum.: 1) Approximate 65 lb Feed/hr to calculate entrainment
2) Reactor entrainment is feed
3) Nitrates in NH_3-H_2O are NH_4NO_3

1. Feed and Product Analysis as Sampled

	Feed		Product		
Component	Wt. %	Moles 100 gm	Wt. %	Moles 100 gm	
AN	62.50	0.7812	61.9	0.7737	
U	32.60	0.5433	13.9	0.2317	
GN	4.10	0.0336	24.1	0.1975	
H_2O	0.99	0.0550	-	-	
Insol.	-	-	0.26	0.0082	(four Urea Equiv.)
	100.19%		100.16%		

2. Feed AN/U Molar Ratio $= \dfrac{0.7812}{0.5433} =$ 1.44 moles AN/mole U

3. Approximation of Reactor Entrainment:

$$\text{Entrainment} = \frac{117\ \text{lb}\ NH_3\text{-}H_2O}{\text{hr}} \Big/ \frac{0.0042\ \text{lb AN}}{1\ \text{lb}\ NH_3\text{-}H_2O} \Big/ \frac{1\ \text{lb Feed}\ *}{0.666\ \text{lb AN}} \Big/ \frac{\text{hr}}{\sim 65\ \text{lb Feed}}$$

$$= 0.0114 \times 100 = \underline{1.14\%}\ \text{Entrainment Loss}$$

4. Determine P/F Ratio

Feed Nitrate Moles Corrected for Entrainment (GN + AN)

$$= \frac{(0.9886)(4.1\ \text{lb GN}/100\ \text{lb Feed})}{122\ \text{lb GN/mole}} + \frac{(0.9886)(62.5\ \text{lb AN}/100\ \text{lb Feed})}{80\ \text{lb AN/mole}}$$

$= 0.0330 + 0.7723 = 0.8053$ moles NO_3/100 parts Feed

Product Nitrates - $0.1975 + 0.7737 = 0.9712$ moles NO_3/100 parts Product

$$P/F = \frac{\text{moles}\ NO_3/100\ \text{parts Feed}}{\text{moles}\ NO_3/100\ \text{parts Product}} = \frac{0.8053}{0.9712} = \underline{\underline{0.8292}}$$

* Assumed GN in feed as AN

TABLE 30 (CONT.)

5. Calculated Feed Rate

$$= \frac{\text{Product Rate}}{P/F}$$

$$= \frac{55 \text{ lb Product/hr}}{0.8292}$$

$$= 66.3 \text{ lb Feed/hr}$$

6. Correct Reactor Feed and Product Moles/100 gm for Entrainment Loss and P/F Ratio, Respectively:

	Feed (moles)		Product (moles)	
	In	Corrected for Entrainment	Out	Corrected for P/F
AN	0.7812	0.7723	0.7737	0.6417
U	0.5433	0.5371	0.2317	0.1922
GN	0.0336	0.0332	0.1975	0.1638
H_2O	0.0550	0.0550	-	-
Insol.	-	-	0.0062 (as U)	0.0068

II. REACTOR YIELD CALCULATIONS

1. Ammonium Nitrate to Guanidine Nitrate

a.

$$Y_{AN}(\text{actual}) = \frac{\Delta\ GN}{\Delta\ AN} \times 100 = \frac{0.1638 - 0.0336}{0.7812 - 0.6417} \times 100$$

$$= \frac{0.1302}{0.1395} \times 100$$

$$= \underline{\underline{93.3\%}}$$

b.

$$Y_{AN}(\text{Entrainment corrected}) = \frac{\Delta\ GN}{\Delta\ AN} \times 100 = \frac{0.1638 - 0.0332}{0.7723 - 0.6417} \times 100$$

$$= \frac{0.1306}{0.1306} \times 100$$

$$= \underline{\underline{100\%}}$$

$\therefore$ AN yield loss to Entrainment = $\underline{\underline{6.7\%}}$

TABLE 30 (CONT.)

2. Urea to Guanidine Nitrate Assuming the Two-Mole Stoichiometry

$$Y_{Urea} = \frac{\Delta GN}{\frac{\Delta U}{2}} \times 100 = \frac{0.1638 - 0.0332}{(0.5371 - 0.1922)/2} \times 100 = \frac{0.1306}{0.1724} \times 100$$

$$Y_{U/GN} = \underline{\underline{75.6\%}}$$

3. Urea Loss to Hydrolysis and Insolubles

a. Y_U to Hydrolysis $= \dfrac{0.0550 + 0.0068/4}{(0.5371 - 0.1922)} \times 100 = \dfrac{0.0567}{0.3449} \times 100$

Y_U to Hydrolysis $= \underline{\underline{16.4\%}}$

b. Y_U to Insol. $= \dfrac{0.0068 \text{ (as U)}}{0.3449} \times 100 = \underline{\underline{1.97\%}}$

4. Urea Loss to Entrainment

Y_U to Entrainment $= \dfrac{0.5433 - 0.5371}{0.3449} \times 100 = \dfrac{0.0062}{0.3449} \times 100$

$= \underline{\underline{1.79\%}}$

III. MATERIAL BALANCE

1. Ammonium Nitrate

a. In $= 66.3 \text{ lb Feed/hr} \times \dfrac{0.625 \text{ lb AN}}{\text{lb Feed}} = 41.44$ lb AN/hr

b. Out:

(1) As AN $= \dfrac{55 \text{ lb Product}}{\text{hr}} \times \dfrac{0.619 \text{ lb AN}}{\text{lb Product}} = 34.05$ lb AN/hr

(2) As GN $= \Delta \text{ GN} \times \dfrac{\text{MW AN}}{\text{MW GN}}$

$= [(55 \times 0.241) - (66.3 \times 0.041)] \dfrac{80}{122}$

$= 6.91$ lb AN/hr

TABLE 30 (CONT.)

(3) As Entrainment $= \frac{117 \text{ lb } NH_3 - H_2O}{\text{hr}} \times \frac{0.0042 \text{ lb AN}}{\text{lb } HN_3 - H_2O}$

$= 0.4914$ lb AN/hr

(4) Total AN Out $= 34.05 + 6.91 + 0.4914$

$= 41.45$

c. Closure $= \frac{\text{AN Accounted For}}{\text{AN In}} \times 100$

$= \frac{41.45}{41.44} \times 100$

$= \underline{\underline{100.0\%}}$

2. Urea

a. In $= 66.3$ lb Feed/hr $\times \frac{0.326 \text{ lb U}}{\text{lb Feed}}$

$= 21.61$ lb U/hr

b. Out

(1) As Urea $= \frac{55 \text{ lb Product}}{\text{hr}} \times \frac{0.139 \text{ lb U}}{\text{lb Prod}}$

$= 7.65$ lb U/hr

(2) As GN $=$ (Product GN - Feed GN) $\frac{\text{MW U}}{\text{MW GN}}$

$= [(55 \times 0.241) - 66.3 \times 0.41)] \frac{60}{122}$

$= 5.19$ lb U/hr

(3) As Insolubles $= \frac{55 \text{ lb Prod}}{\text{hr}} \times \frac{0.0026 \text{ lb Insol.}}{\text{lb Product}} \times \frac{240^*}{128}$

$= 0.268$ lb Urea per hour

* Ammelide = 4 Urea equivalents or 4 x 60.

TABLE 30 (CONT.)

(4) As NH_3 in Scrubber H_2O

$$= \frac{117 \text{ lb } NH_3\text{- } H_2O}{\text{hr}} \times \frac{0.0294 \text{ lb } NH_3}{\text{lb } NH_3\text{- } H_2O} \times \frac{60 \text{ (MW U)}}{34 \text{ (MW } NH_3\text{)}}$$

= 6.07 lb U/hr

(5) As $(NH_4)_2\ CO_3$ In Scrubber Water

$$= 117 \times \frac{0.0181 \text{ lb } (NH_4)_2\ CO_3}{\text{lb } NH_3 \text{ - } H_2O} \times \frac{60}{90}$$

= 1.32 lb U/hr

Total Urea Out - 20.50 lb U/hr

c. Closure - 20.50/21.6 x 100 = 94.86%

IV GAS BALANCE

1. Ammonia (NH_3)

a. Predicted NH_3 Production

(1) From GN Formation ($AN + 2U \rightarrow GN + 2\ NH_3 + CO_2$)

$$= \frac{\Delta \text{ GN formation}}{\text{MW GN}} \times 2 \text{ moles } NH_3\text{/mole GN} = \text{moles } NH_3$$

$$= \left[\frac{(55 \times 0.241) - (66.3 \times 0.041)}{122}\right] 2 = 0.1728 \text{ mole } NH_3\text{/hr}$$

(2) From Hydrolysis of U Due to H_2O in Feed

($U + H_2O \rightarrow 2\ NH_3 + CO_2$)

$$= \frac{\text{lb } H_2O \text{ in Feed}}{\text{MW } H_2O} \times 2 \text{ moles } NH_3\text{/mole } H_2O$$

$$= \frac{(66.3)(0.0099)}{18} \times 2 = 0.074 \text{ mole } NH_3\text{/hr}$$

(3) From Formation of Ammelide

($4U \rightarrow CO_2 + 4\ NH_3 + \text{Ammelide}$)

$$= \frac{\text{Ammelide Produced}}{\text{MW Ammelide}} \times 4 \text{ moles } NH_3\text{/mole Ammelide}$$

$$= \frac{55 \times 0.0026}{128} \times 4 = 0.00448 \text{ mole } NH_3\text{/hr}$$

TABLE 30 (CONT.)

(4) Total Predicted NH_3 = 0.1728 + 0.0740 + 0.0045

= 0.2513 mole NH_3/hr

b. Actual Ammonia Measured

(1) NH_3 In Scrubber Water

$$= \frac{117 \text{ lb } NH_3 - H_2O \;/\; 0.0294 \text{ lb } NH_3 \;/\; \text{mole}}{\text{hr} \;/\; \text{lb } NH_3 - H_2O \;/\; 17 \text{ lb } NH_3}$$

= 0.2024 moles NH_3/hr

(2) $(NH_4)_2CO_3$ In Scrubber Water

$$= \frac{117 \text{ lb } NH_3 - H_2O \;/\; 0.0181 \text{ lb}(NH_4)_2CO_3 \;/\; \text{mole} \;/\; 2 \text{ moles } NH_3}{\text{hr} \;/\; \text{lb } NH_3 - H_2O \;/\; 96 \text{ lb}(NH_4)_2CO_3 \;/\; \text{mole}(NH_4)_2CO_3}$$

= 0.0441 mole NH_3/hr

(3) Total Measured NH_3 = 0.2024 + 0.0441 = 0.2465 mole NH_3/hr

c. Ammonia Closure

$$= \frac{\text{moles } NH_3 \text{ measured}}{\text{moles } NH_3 \text{ predicted}} \times 100$$

$$= \frac{0.2465}{0.2513} \times 100$$

= 98.1%

2. Carbon Dioxide (CO_2)

a. Predicted CO_2 Production

(1) From GN Formation ($AN + 2\,U \rightarrow GN + 2\,NH_3 + CO_2$)

$$= \frac{\text{moles } NH_3}{2}$$

$$= \frac{0.1728}{2}$$

= 0.084 moles CO_2/hr

TABLE 30 (CONT.)

(2) From Hydrolysis of Urea Due to Feed Water

$$= \frac{\text{moles } NH_3}{2}$$

$$= \frac{0.074}{2}$$

$= 0.037$ moles CO_2/hr

(3) From Formation of Ammelide

$$= \frac{\text{moles } NH_3}{4}$$

$$= \frac{0.00448}{4}$$

$= 0.00114$ moles CO_2/hr

(4) Total Moles CO_2 Predicted

$= 0.0864 + 0.637 + 0.00114$

$= 0.1245$ moles CO_2/hr

b. <u>Actual CO_2 Measured</u>

(1) $(NH_4)_2\ CO_3$ in Scrubber Water

$$= \frac{117\ \text{lb}\ NH_3\text{-}H_2O / 0.0181\ \text{lb}(NH_4)_2\ CO_3 / \quad \text{mole} \quad / 1\ \text{mole}\ CO_2}{\text{hr} \quad / \quad \text{lb}\ NH_3\text{-}H_2O \quad /96\ \text{lb}(NH_4)_2CO_3/\ \text{mole}\ (NH_4)_2\ CO_3}$$

$= 0.0221$ moles CO_2/hr

(2) CO_2 in Scrubber Off-Gas

$$= \frac{9.33\ \text{liter gas} \ / \quad \text{mole} \ / \quad \text{lb} \quad / \ 60\ \text{min}}{\text{min.} \qquad / \ 22.4\ \text{l.} / \ 454\ \text{g} / \quad \text{hr}}$$

$= 0.055$ lb-moles CO_2/hr

(3) CO_2 Gas from Bottom of Scrubber

$$= \frac{8.40\ \text{liter gas} / \quad 60 \ /}{\text{min.} \qquad / \ 22.4 / \ 454}$$

$= 0.050$ moles CO_2/hr

TABLE 30 (CONT.)

(4) Total CO_2 Measured

$= 0.0221 + 0.0550 + 0.0500$

$= 0.1271$ moles CO_2/hr

c. <u>CO_2 Closure</u>

$$= \frac{\text{moles } CO_2 \text{ measured}}{\text{moles } CO_2 \text{ predicted}} \times 100$$

$$= \frac{0.1271}{0.1245} \times 100$$

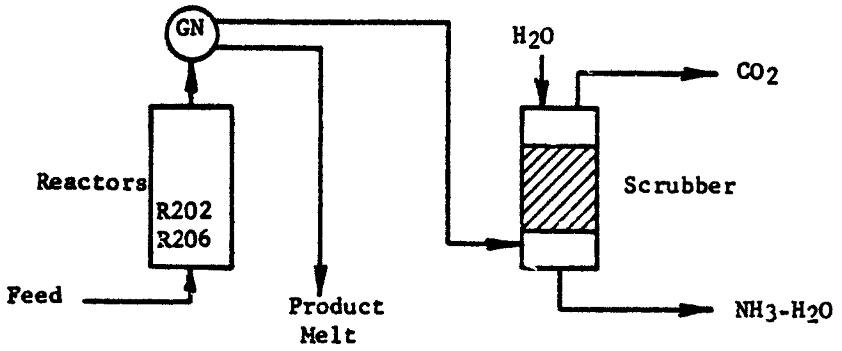
$$= \underline{\underline{102.1\%}}$$

TABLE 31

REACTOR-SCRUBBER WEIGHT BALANCE
AUGUST 9-10, 1973

Time Period: August 9, 1973, 9:00 p.m. Aubust 10, 1973, 6:00 p.m.

Elapsed Time: 21 Hours



Measured Rates

	Feed	Product Melt	NH_3-H_2O
Total, Lb	1029.26	836.55	2466
Hourly Rate, lb/hr	48.9	39.84	117.43

Analysis

	% AN	% U	% GN	% H_2O	% Insol.
1. Feed (Avg.)	62.2	33.1	2.3	2.4	-
2. Product (Avg.)	60.9	10.4	28.4	-	0.36
3. NH_3-H_2O	0.08% AN, 1.74% NH_3, 3.79% $(NH_4)_2CO_3$				

I. Measured P/F Ratio $= \frac{836.55}{1029.26} = \underline{\underline{0.812}}$

TABLE 31 (CONT.)

II. Assumption of Nitrate Conservation

	Feed		Product	
	%	Moles/100 lb	%	Moles/100 lb
AN	62.2	0.7775	60.9	0.7612
U	33.1	0.5517	10.4	0.1750
GN	2.3	0.0188	28.4	0.2326
H_2O	2.4	0.1333	-	-
Insol.	-	-	0.36	0.0029

$$\text{Nitrates In} = \text{Nitrates Out}$$

$$(\%\ NO_3)(F_{lb}) = (\%\ NO_3)(P_{lb})$$

$$\frac{\text{Moles } NO_3}{100\ \text{lb Feed}} = \frac{P_{lb}}{F_{lb}} \times \frac{\text{Moles } NO_3}{100\ \text{lb Prod.}}$$

$$\left(\frac{P}{F}\right)_{\substack{NO_3\\ \text{Conservation}}} = \frac{\text{Moles } NO_{3\,In+}}{\text{Moles } NO_{3\,Out}} = \frac{0.7775 + 0.0188}{0.7612 + 0.2326}$$

$$= \frac{0.7963}{0.9939} = 0.802$$

III. Validity of Nitrate Conservation

$$\text{P/F Closure} = \frac{\text{P/F Calculated}}{\text{P/F Measured}} = \frac{0.802}{0.812} = \underline{\underline{98.8\%}}$$

TABLE 32

ANALYSES FOR WEIGHT BALANCE OVER REACTOR-SCRUBBER ON AUGUST 9/10, 1973

			Individual Analysis						Average Analysis				
Date	Time	Sample	AN	U	GN	H_2O	Insol.	Closure	AN	U	GN	H_2O	Insol.
8/10	9:00 am	Feed	63.8	32.4	2.3	1.4	---	99.9					
8/10	10:00 am	Feed	64.6	32.0	2.4	1.2	---	100.2	62.9	33.5	2.3	2.4	---
8/10	12:00 am	Feed	64.7	31.9	4.8	1.2	---	102.6					
8/10	1:00 pm	Feed	64.2	32.8	0.3	1.6	---	98.9	Normalized				
8/10	2:00 pm	Feed	61.4	34.0	1.7	2.5	---	99.6					
8/10	3:00 pm	Feed	63.6	32.3	1.2	2.2	---	99.3	62.2	33.1	2.3	2.4	---
8/10	3:30 pm	Feed	59.8	33.5	2.7	4.1	---	100.1					
8/10	6:00 pm	Feed	61.1	39.9	2.7	4.9	---	108.6					
8/10	9:00 am	Product	64.2	8.5	27.4	---	0.33	100.43					
8/10	10:00 am	Product	63.8	9.8	25.4	---	0.53	99.53					
8/10	12:00 am	Product	61.4	11.8	28.1	---	0.46	101.76	61.7	10.5	28.8	---	0.36
8/10	1:00 pm	Product	60.5	11.1	28.1	---	0.44	100.16					
8/10	2:00 pm	Product	59.9	10.4	33.8	---	0.38	104.48	Normalized				
8/10	3:00 pm	Product	60.5	11.4	28.0	---	0.23	100.13					
8/10	3:30 pm	Product	61.8	13.6	25.8	---	0.28	101.48	60.9	10.4	28.4	---	0.36
8/10	6:00 pm	Product	61.3	7.2	33.7	---	0.19	102.33					

This lends further credence to the assumption of nitrate conservation and the interpretation of Figure 7 in a previous section of this report.

4. Special Experiments and Analyses

To determine loss sources for the material balances and to provide design data for specific operations, the following special experiments, analyses, and analytical requests were conducted:

(a) Evaporator off-gas tests
(b) Analysis of ammonia-water
(c) Back-up material balance data
(d) Centrifuge washing efficiency
(e) Ammelide repulping experiment
(f) Nonroutine analytical requests.

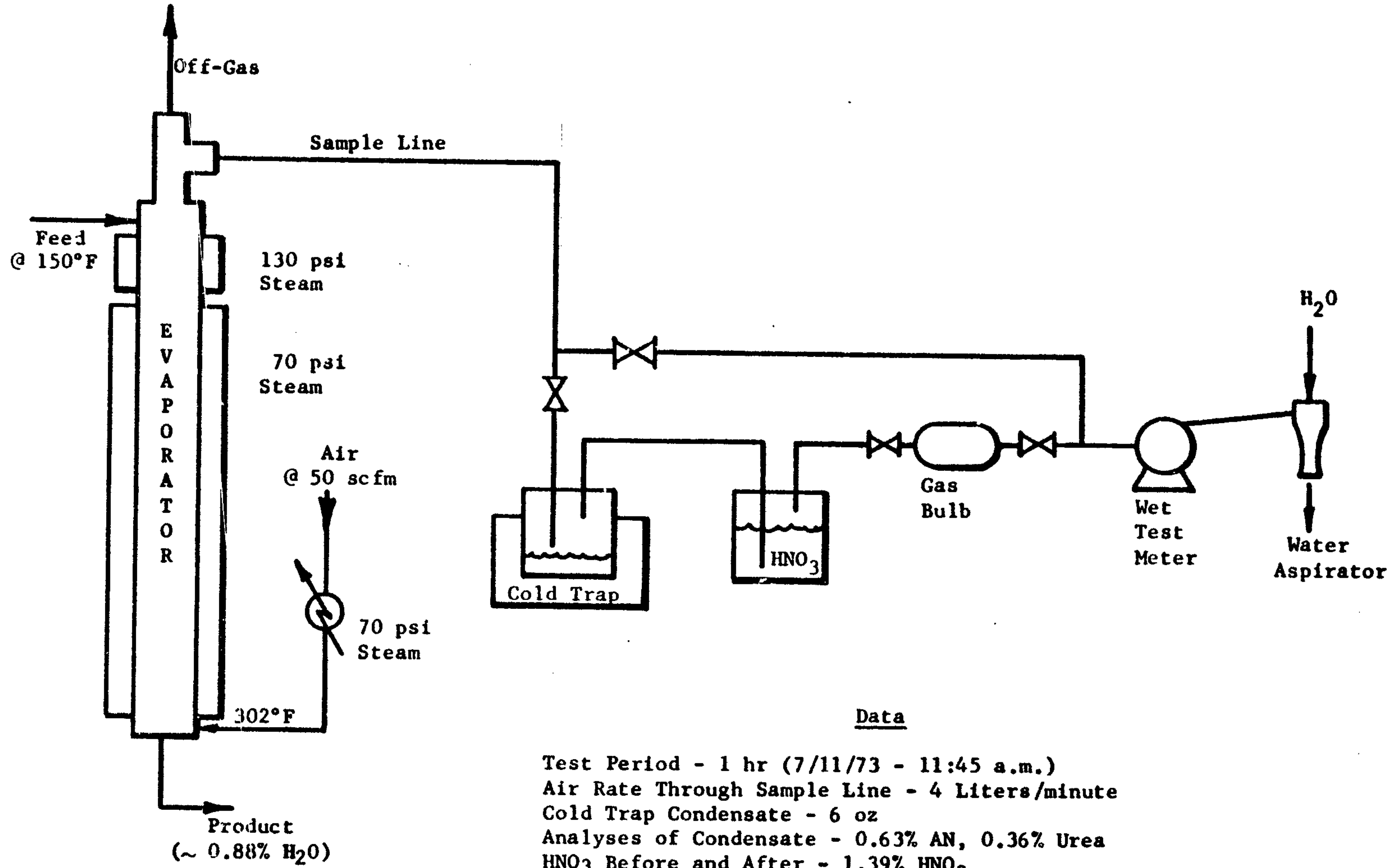
Other long term special experiments, e.g., (1) corrosion studies and (2) off-site special experiments such as NQ conversion and Wyssmont dryer tests, are discussed in other sections of the report.

a. Evaporator Off-Gas Tests

In an effort to determine the volatile losses of nitrates and urea in the vent gas of the evaporator, two tests were conducted in which a pilot stream of the evaporator off-gas was collected and analyzed. Figure 13 shows a schematic of the experimental set-up and the major analytical results from the first of the two tests. Table 33 presents the calculated results. The test showed that approximately 2% of the AN and 6.6% of urea in the evaporator feed stream were lost to the off-gas. This test was considered extreme since the steam pressures to the two evaporator stages and the air heater were higher than designed (design was 90 psig for the upper stage and 30 psig for the lower stage and air heater). The reason for the higher steam pressures was the suspected ammelide fouling in the evaporator tubes and a necessarily higher temperature drop.

This test was repeated at design steam pressures on August 8, 1973. The results in this run showed a 1.3% AN loss (based on feed) and no loss of urea (based on collected condensate). The feed composition of the stream in the second test was lower in urea concentration (8%). The lower urea value, in addition to the lower temperatures, reduced the urea loss.

It is believed that these two tests bracket the extreme conditions at which the evaporator would be operated. When it is realized that a commercial size evaporator would have but one air-swept stage, it can be



Data

Test Period - 1 hr (7/11/73 - 11:45 a.m.)
Air Rate Through Sample Line - 4 Liters/minute
Cold Trap Condensate - 6 oz
Analyses of Condensate - 0.63% AN, 0.36% Urea
HNO_3 Before and After - 1.39% HNO_3
Gas Sample Analyses - 0.11% mole CO_2, 19.3% mole O_2, 1.7% mole H_2O
77.8% N_2, 1.1% Argon

Feed

100 lb/hr -
41% AN, 13.2% Urea
6% GN, 39% H_2O

Figure 13. Experimental Test Setup to Measure Evaporator Off-Gas Volatiles

TABLE 33

EVAPORATOR OFF-GAS TEST NO. 1
(REFER TO FIGURE 13 FOR DATA AND CONDITIONS)

1. AN and U in Condensate

AN - 6 oz/16 x 0.0063 = 0.00236 lb AN/Hr

Urea - 6 oz/16 x 0.0036 = 0.00135 lb Urea/Hr

2. Extrapolated AN and Urea From Stack

$$\frac{4\ \ell\text{/min}}{50\ \text{scfm}} \times \frac{1}{28.32\ \ell\text{/ft}^3} = 0.0028 = \text{fraction of stack gas out test apparatus}$$

$$\text{AN Vapors in Stack Gas} = \frac{0.00236}{0.0028} = \underline{0.84\ \text{lb AN/hr}}$$

$$\text{U Vapors in Stack Gas} = \frac{0.00135}{0.0028} = 0.48\ \text{lb Urea/hr}$$

3. Percentage Loss of AN and Urea

$$\text{AN}_{\text{In}} = 100\ \text{lb Feed/Hr} \times 0.41 = 41\ \text{lb AN/Hr}$$

$$\text{AN}_{\text{Vaporized}} = \frac{0.84}{41} \times 100 = \underline{\underline{2.05\%}}$$

$$\text{Urea}_{\text{In}} = 100\ \text{lb feed/hr} \times 0.132 = 13.2\ \text{lb urea/hr}$$

$$\text{Urea}_{\text{Vaporized}} = \frac{0.48}{13.2} \times 100 = \underline{\underline{3.64\%}}$$

4. Calculated Decomposed CO_2

$$\frac{50\ \text{scfm}}{359\ \text{scf/lb-mole}} = 0.1393\ \text{lb-mole evaporator gas/min}$$

CO_2 found = 0.11 mole %

CO_2 in inlet air = 0.03 mole %

$$\text{lb mole } CO_2 \text{ made} = \frac{(0.11-0.03)}{100} \times 0.1393\ \frac{\text{lb-mole gas}}{\text{min}}$$

$$= 0.00011\ \frac{\text{lb-mole } CO_2}{\text{min.}} = 0.0048\ \text{lb } CO_2\text{/min}$$

TABLE 33 (CONT.)

5. Urea Equivalent of Decomposed Gas

$$\text{Urea Decomposed to CO}_2 = \frac{0.00011 \text{ moles CO}_2}{\text{min.}} \times \frac{1 \text{ mole U}}{\text{mole CO}_2} \times \frac{60 \text{ lb U}}{\text{Mole U}}$$

$$\times \frac{60 \text{ min}}{\text{hr}} = 0.396 \text{ lb Urea decomposed/hr}$$

$$\text{Feed Urea Decomposed to CO}_2 = \frac{0.396 \text{ lb Urea Decomposed}}{13.2 \text{ lb Urea to Evap.}} \times 100 = \underline{\underline{3\%}}$$

6. Total AN and U Losses From Evaporator

AN = 2.05% of Feed

Urea = 6.64% of Feed

projected that scaled-up operations would have even lower volatile losses. For design purposes, it can be projected that volatile losses of 2 to 3% AN and 1 to 2% urea, based on the evaporator feed, might be expected in a production evaporator. Since the amount of AN being fed to the evaporator is a substantial percentage (50-60%) of the total AN reactor feed, this type of loss would affect the total process AN yield by about 1.5%. The urea fed to the evaporator is less than a fifth of the virgin make-up. Its effect on the total urea yield loss would be a fraction of a percent.

b. Analyses of Ammonia-Water from Scrubber

The water leaving the bottom of the scrubber in the pilot plant was not deemed an important process stream since it was not a prototype of an envisioned commercial process. However, for the sake of the material balance closure and for determining the reactor entrainment loss, a method for complete analyses had to be instituted. The selected method and an example of its typical usage are shown in Table 34.

A scrubber material balance based on these analyses for the scrubber water and an off-gas analysis defined the gas split (NH_3 and CO_2) in the scrubber. This balance, discussed in the previous section (Reactor-Scrubber Balance of July 31, 1973), showed that essentially all of the ammonia produced is scrubbed by the water, i.e., 83% of the ammonia was found as free ammonia while the remaining was tied up as ammonium carbonate and 17% of the CO_2 was absorbed by the scrubber water which was neutralized to the carbonate by the ammonia-rich water. The remaining carbon dioxide left the system as a gas. The pressure drop characteristics of the pilot plant scrubber created a split of about 50% gaseous CO_2 from the top of the scrubber and about 50% out the bottom of the scrubber with the ammonia-water stream.

As the calculations in Table 34 show, nitrates were found in the ammonia-water. Tables 35 and 36, which will be discussed later, contain additional data that support this finding. There are three possible ways that the nitrates can appear in the ammonia-water; that is, (1) a reactor melt flow upset, (2) vaporization of ammonium nitrate, and (3) reactor melt entrainment in the off-gas. The fact that nitrates appeared in a number of the water samples which were taken at different periods of time and which are at approximately the same composition (0.1-0.5% nitrate), suggests that a melt flow upset condition is not the likely source of the water nitrates. To differentiate between the other two sources, it was necessary to determine if the nitrates found in the ammonia-water are all ammonium nitrate (by vaporization) or a split between guanidine and ammonium nitrate (by entrainment). Any urea that vents to the scrubber is believed to be hydrolyzed because of the scrubber feed temperature and water concentration.

TABLE 34

ANALYSIS OF AMMONIA-WATER FROM REACTOR OFF-GAS SCRUBBER

I. METHOD OF ANALYSIS

1. Total NO_3 as NH_4NO_3

a. Dilute 10 to 15 gm NH_3-H_2O to 100 ml.
b. Determine absorbance at 302 and 355 nanometers
c. Calculate as follows:

(1) $\frac{A_{302} - A_{335}}{0.0865}$ = mg. NO_3 as NH_4NO_3/ml

(2) $\frac{(\text{mg. } NO_3 \text{ as } NH_4NO_3/\text{ml})(100)(0.1)}{\text{gm Sample}}$ = ____ % NO_3 as NH_4NO_3

2. Free NH_3 (Includes NH_4 From $(NH_4)_2CO_3$)

a. Titrate 25 ml aliquot diluted NO_3 sample to methyl purple and point with 0.1 N HCl
b. Calculate as follows:

(1) $\frac{(\text{ml HCl})(\text{N HCl})(1.7)}{\text{gm Sample x } 25/100}$ = ____ % Free NH_3

(2) $\frac{(\text{ml HCl})(\text{N HCl})(8.0)}{\text{gm Sample x } 25/100}$ = ____ % Free NH_3 as NH_4NO_3

3. $CO_3^=$ as $(NH_4)_2\ CO_3$

a. Weigh 10 gm sample into 100 ml of saturated $Ba(OH)_2$ solution in a 150 ml beaker. Stir while adding.

NOTE: Prepare saturated $Ba(OH)_2$ solution by placing an excess of $Ba(OH)_2$ in water in a one-liter beaker. Filter the solution through a No. 41 filter paper.

b. Filter the $Ba(CO_3)_2$ precipitate through a glass filter. Wash the precipitate three times with 10-ml portions of distilled water. Air dry the precipitate by drawing air through the filter (3-5 minutes).

c. Quantitatively transfer the precipitate to a 400 ml beaker and add 70 to 90 ml of 0.1 N HCl. Boil solution for 3 to 5 minutes, cool and titrate to methyl purple and point with 0.1 N NaOH.

d. Calculate as follows:

$$\frac{(\text{ml HCl})(\underline{N}\ \text{HCl}) - (\text{ml NaOH})(\underline{N}\ \text{NaOH})}{\text{gm Sample}} \times 4.8$$

$$= \quad \%\ (NH_4)_2CO_3$$

II. REPORTING

1. % Free NH_3 Corrected for $(NH_4)_2CO_3$
   [% Free NH_3 - (% NH_3 as $(NH_4)_2CO_3$) x $\frac{34}{76}$]

2. % CO_3 as $(NH_4)_2CO_3$ from 3.

3. % NO_3 as NH_4NO_3 from 1.

III. SAMPLE CALCULATION

H_2O sample No. 1

1. Weight of Sample

108.2158	Gross
97.9225	Net
10.2933	Gm Sample

2. Volume of Standard Solutions

50 ml Sat'd $Ba(OH)_2$
70 ml 0.0982 N HCl
28.43 ml 0.1054 N NaOH

3. Percent $(NH_4)_2 CO_3$

$$\frac{[(70.00)(0.0982) - (28.43)(0.1054)] \times 4.8}{10.2933} = 1.81\%\ (NH_4)_2CO_3$$

4. Weight of 2nd Aliquot

69.9537
60.2962

9.6575 gms/100 ml

TABLE 34 (CONT.)

5. Absorbance

$\alpha = 0.0865, \quad A = 0.035$

$A = (\alpha)(C)$

$C = A/\alpha = \frac{0.035}{0.0865} = 0.405$ gm/l

$\frac{0.0405(100)}{9.6575} = 0.42\%$ as NH_4NO_3

6. % Alkalinity

Titration using 62.15 ml of 0.0982 N HCl

Total alkalinity $= \frac{62.15\ (0.0982)(1.7)}{9.6575(30/100)} - 3.58\%$ Alk. as NH_4NO_3

% NH_3 corrected for $CO_3 = \left[3.58 - \left(\frac{34}{96}\right)(1.81)\right] = 2.94\%$

7. Results

% CO3 as $(NH_4)CO_3$ = 1.81%

% NO_3 as NH_4NO_3 = 0.42%

% Alk. as NH_3 = 2.94%

TABLE 35

RESULTS OF SPECIAL ANALYTICAL REQUESTS

Date	Sample	Method	Analyses	Notes
7/3	Scrubber Off-Gas	Mass. Spect.	97% CO_2, 0.14% O_2, 0.97% N_2 & CO, 1.6% H_2O, 0.15-0.45% N_2O	X2126-85-1
7/9	Volatile Portion of Ammonia-Water	Mass. Spect.	99% NH_3 & H_2O, 0.42% CO_2, 0.05-0.24% NO + N_2O	X2126-95-1
7/11	Ammonia-Water	Kjeldahl N_2	3.7% $CO_3^=$, 6.85% NH_4^+, 0.003% NO_3^-	X2126-95-1
7/16	Ammonia-Water	Kjeldahl N_2	4.7% $CO_3^=$, 6.69% NH_4^+, 0.012% NO_3^-	X2126-34-1
7/31	Scrubber Off-Gas	Mass. Spec.	96.4% CO_2, < 0.1% A, 0.5% O_2, 2.2% N_2 + CO, 0.8% H_2O, 0.1% N_2O (max)	2:15 p.m., 7/31
7/31	Scrubber Feed Gas	Mass. Spec.	17.0% CO_2, 0.9% A, 16.2% O_2, 65.4% N_2 + CO, 0.6% H_2O	2.15 p.m., 7/31
7/24	Evap. Bottoms	Elemental Analyses	20 ppm Ash, 2.5 ppm Fe, 0.07 ppm Ni, < 0.06 ppm Cr	7:30 p.m., 7/11
7/24	Evap. Bottoms	Elemental Analyses	40 ppm Ash, 0.34 ppm Fe, < 0.03 ppm Ni, < 0.03 ppm Cr	7:30 p.m., 7/13
5/24	Centrifuge Cake	-	12.1% AN, 1.5% Urea, 80.6% GN, 5.2% H_2O, M.P. = 170°C	No Wash
5/24	Centrifuge Cake	-	5.0% AN, 0.19% Urea, 84.8% GN, 8.5% H_2O, M.P. = 206°C	1 Gal Wash
5/24	Centrifuge Cake	-	1.7% AN, 0.13% Urea, 89.1% GN, 9.4% H_2O, M.P. = 210°C	2 Gal Wash
7/18	Repulped Insolubles	-	41.84% T.S., 39.07% Insol., 0.86% Ash, -	10/1 Wash
8/15	Ammonia-Water	DSC	3 Peaks, 115°, 150°, 175°C; Pure AN = 110, Pure GN = 210	X2126-47-1
8/15	Ammonia-Water	DSC	1 Peak @ 210°C	X2126-51-2

TABLE 36

RESULTS OF MISCELLANEOUS ANALYSES

Date	Sample	AN %	U %	GN %	H_2O %	Insol. %	Total Solids %	Ash %	Nitrates %	NH_3 %	$4NO_3$ %	pH	Conductivity Micromhos/sec	Comments
5/23	Quench Tank	-	-	-	-	-	87.2	-	-	-	-	-	-	
6/5	GN Boiler Water	-	-	-	-	-	0.016	-	-	-	-	8.1	312	
6/8	T-106 Contents	23.3	2.68	7.50	-	-	37.9	-	-	-	-	-	-	
6/11	Quench Tank	-	-	-	-	-	63.1	-	-	-	-	-	-	
6/13	Quench Tank	-	-	-	-	-	55.5	-	-	-	-	-	-	
6/28	T-103 Contents	30.4	62.4	-	2.00	-	-	-	-	-	-	-	-	
6/28	T-113 Contents	62.5	21.4	-	1.87	-	-	-		-	-	-	-	
6/28	T-104 Contents	62.4	21.6	-	1.46	-	-	-	-	-	-	-	-	
6/29	Residue in Reactor Elbows	-	-	-	-	4.51	97.7	1.4	-	-	-	-	-	
7/5	Material Bridging Product	27.3	2.84	58.4	7.60	0.31	-	-	-	-	-	-	-	
7/10	Ammonia Water		-	-	-	-	-	-	0.0012	-	-	-	-	
7/10	T-107 Contents	39.9	11.9	6.0	-	-	58.8	-	-	-	-	-	-	
7/10	T-104 Contents	65.3	17.8	6.75	3.6	-	-	-	-	-	-	-	-	
7/10-	NH_4OH for Material Balance	4.59	-	-	-	-	-	-	-	-	-	-	-	
7/12		14.5	0	0	-	-	-	-	-	-	-	-	-	
7/11	Sample from Vent	-	-	-	-	-	-	-	-	0.09	-	-	-	
7/11	Scrubber	-	-	-	-	-	-	-	-	8.45	-	-	-	
7/11	Ammonia Water from Scrubber	6.18	-	-	-	-	-	-	-	-	-	-	-	
7/11	NH_4OH From Scrubber	16.2	0	0	-	-	0.13	-	-	-	-	-	-	
7/12	T-104 Contents	61.9	24.2	7.60	1.84	0.16	-	-	-	-	-	-	-	
7/12	T-106 Contents	32.5	17.8	59.6	22.9	-	-	-	-	-	-	-	-	
7/12	T-107 Contents	38.8	19.2	57.7	30.37	-	-	-	-	-	-	-	-	
7/12	T-104 Contents	59.2	23.9	13.5	3.30	-	-	-	-	-	-	-	-	
7/12	T-106 Contents	32.0	16.8	15.4	21.0	-	-	-	-	-	-	-	-	
7/12	T-107 Contents	39.5	18.4	4.51	26.1	-	-	-	-	-	-	-	-	
7/13	H_2O From Cryst. Trap Batch 239	-	-	5.77	-	-	0.082	-	-	-	-	-	-	
7/13	P-102 Pump Seal	-	-	-	-	-	3.88	-	-	-	-	-	-	
7/13	267-106-4 Before	-	-	-	-	-	-	-	-	-	1.39	-	-	
7/13	267-107-2 After	-	-	-	-	-	-	-	-	-	1.42	-	-	
7/13	Scrubber Solid	-	-	-	-	41.2	51.5	30.7	-	-	-	-	-	
7/18	Cake From Filter Slurry 10% Solid	-	-	-	-	39.07	41.84	0.86	-	-	-	-	-	

TABLE 36 (CONCLUDED)

Date	Sample	AN %	U %	GN %	H_2O %	Insol. %	Total Solids %	Ash %	Nitrates %	NH_3 %	$4NO_3$ %	pH	Conductivity Micromhos/sec	Comments
7/31	Cryst. Feed - No. 260	38.3	7.81	14.1	-	-	42.7	-	-	-	-	-	-	-
8/1	Cryst. Feed - No. 261	40.2	11.8	16.9	-	-	66.9	-	-	-	-	-	-	-
8/1	Diluted T-105	11.2	6.64	9.01	-	-	75.6	-	-	0.064	-	-	-	2:00 p.m.
8/1	Diluted T-106	15.7	7.33	7.46	-	-	25.0	-	-	0.178	-	-	-	2:00 p.m.
8/1	Diluted T-107	20.0	5.69	1.94	-	-	28.9	-	-	-	-	-	-	2:00 p.m.
8/1	T-104	67.3	22.7	8.25	1.82	0.32	-	-	-	-	-	-	-	2:30 p.m.
8/7	Diluted T-105	16.5	5.9	8.8	57.8	-	-	-	-	-	-	-	-	9:00 a.m.
8/7	Diluted T-106	18.5	5.8	8.98	60.1	-	-	-	-	-	-	-	-	9:00 a.m.
8/7	Diluted T-107	22.8	5.6	3.5	82.6	-	-	-	-	-	-	-	-	9:00 a.m.
8/9	T-105	18.8	2.7	7.3	-	-	-	-	-	-	-	-	-	8:15 a.m.
8/9	Diluted T-106	13.1	2.5	4.6	-	-	-	-	-	-	-	-	-	8:15 a.m.
8/9	Diluted T-107	27.7	6.4	4.5	-	-	-	-	-	-	-	-	-	8:00 a.m.
8/9	T-104	74.0	11.5	15.5	0.70	-	-	-	-	-	-	-	-	-
8/8	Diluted T-106	18.5	3.7	9.0	-	-	-	-	-	-	-	-	-	-
8/11	Solids in T-105	-	-	-	-	7.32	-	1.8	-	-	-	-	-	-
7/11	Evap. Test Condensate	0.63	0.36	-	-	-	-	-	-	-	-	-	-	-
8/10	P102 Packing	-	-	-	-	-	5.5	-	-	-	-	-	-	-
8/10	NH_3-H_2O-8/9 p.m.	0.17	-	-	-	-	-	-	0.17	1.37	-	-	-	4.18$(NH_4)_2$ CO_3
8/10	Material Balance No. 2	0.80	-	-	-	-	-	-	0.80	0.95	-	-	-	2.95$(NH_4)_2$ CO_3
8/10	NH_3-H_2O	0.08	-	-	-	-	-	-	0.08	1.74	-	-	-	3.79$(NH_4)_2$ CO_3
8/13	NH_3-H_2O Storage	1.73	-	-	-	-	-	-	1.73	2.18	-	-	-	4.31$(NH_4)_2$ CO_3
8/1	NH_3-H_2O Sample	0.11	-	-	-	-	-	-	0.11	3.48	-	-	-	1.22$(NH_4)_2$ CO_3
7/18	Scrubber Plug	-	-	-	-	2.9	82.5	-	-	-	-	-	-	72.5% $(NH_4)_2$ CO_3
7/11	Solid Bowl - 4S-1	-	-	-	-	19.91	74.18	0.60	-	-	-	-	-	-
7/12	Solid Bowl - 4S-2	-	-	-	-	23.06	74.46	0.16	-	-	-	-	-	-
7/12	Solid Bowl - 4S-3	-	-	-	-	18.75	76.60	0.19	-	-	-	-	-	-

Two of the ammonia-water samples were examined for guanidine nitrate content in the following manner. The samples were dried by heating and analyzed using the differential scanning calorimeter (DSC). Control samples in this instrument indicated peaks of 110°C for pure ammonium nitrate and 210°C for pure guanidine nitrate. Since AN and GN form a eutectic, peaks can be expected between these values for blends of the two compounds. The two analyzed samples were found to have DSC peaks at 150 and 175°C for the first sample and 210°C for the second sample. The conclusion from this work is that the GN does exist in the ammonia-water sample; and therefore, the source of the nitrates in the process stream is entrainment from the reactor gas-liquid separators.

c. Backup Material Balance Data

Tables 35 and 36 contain nonroutine analytical requests and miscellaneous analyses. Some data in these tables were requested in support of the material balance efforts. In Table 35, the analyses by mass spectroscopy, Kjeldahl nitrogen, and DSC were all in support of the material balance effort. One of these analyses, the scrubber feed gas, was determined to be a poor sample due to air leakage. The other data in this table represent two special experiments (centrifuge washing and ammelide repulping) and a design data request for trace metal buildup in the recycle stream.

d. Centrifuge Washing Efficiency

During one of the early centrifuge batches (Batch 101), an experiment was conducted to determine the effectiveness of water washing a GN cake on the centrifuge. The results (complete analyses in Table 35) show that as the wash water was increased from 0 to 1 to 2 gallons per centrifuge batch, the residual AN in the wet GN cake decreased from 12.1 to 5.0 to 1.7% and the melting point of the product increased from 170° to 206° to 210°C, respectively. A similar decreasing trend was noted for urea (1.5 to 0.19 to 0.13%) and an increasing trend for GN (80.6 to 84.8 to 89.1%). The basket centrifuge during this experiment contained approximately 40 to 50 lb of wet product. Based as the results of this experiment, the standard GN cake was subjected to a 1 to 2 gallon water wash. Examination of the analytical results for the individual batches of GN showed that this was adequate.

e. Ammelide Repulping

The solid bowl centrifuge employed in the pilot plant, when operable, effectively removed insolubles from the water-quenched reactor product. Unfortunately, the solid bowl centrifuge employed was a laboratory machine and not suited for full-time service. The result was periods of down-time.

Projection to a commercial design for the insolubles separation suggests that a continuous solid bowl machine followed by a repulping tank and then a filtration step would provide an efficient and operable system. An experiment was conducted to explore the feasibility of repulping the solid bowl cake. Typical cakes (75-80% total solids, 20-25% insolubles), were repulped with 500% and 1000% water washes in two experiments. Both the feed cake and the filtered product from the 5/1 repulp experiment were tacky, presumably from residual AN. The 10/1 filtered product was a light tan, very fine solid with no tackiness. Analyses (see Table 35) of the latter cake showed that it was 94% insolubles (dry basis), 60% water, and 0.86% ash. The high water content was typical of the laboratory filter paper method used.

f. Other Analyses

To determine if there was a buildup of trace metals in the system, samples of evaporator bottoms were submitted for elemental analyses. Table 35 shows the results. Two samples showed an average of 1.5 ppm iron, 0.05 ppm nickel, < 0.05 ppm chromium and 30 ppm ash. Both samples represented material that could have been in the system as recycle for many residence times. The operation at this point in time had been on recycle for six weeks (one day at full capacity represents one turn-over of recycle material). These data suggest that metal contaminants were not building up in the system.

Table 36 shows analyses of miscellaneous samples submitted during the operation. Some of the data in this table deserve special comment. On June 29, a sample of the material plugging the upper reactor elbows was analyzed. This sticky, tan material had led to high reactor back pressures and a no-flow condition, resulting in shutdown of all of the reactors. The analyses of this material showed that it was reactor melt but with very high insolubles (4.5%) and ash (1.4%) content.

A number of material balance support data points are contained in Table 36. A considerable number of samples (tank contents, pump seal leakage, etc.), were analyzed during the July 11-13 period. After it was learned that more accurate tank composition samples were necessary for the material balances, diluted samples of T-105, T-106, and T-107 were taken (60% water/40% sample). A number of the samples in Table 36 represent these types of samples. Examination of these analyses shows more consistent results than obtained for earlier tank content samples.

E. PROCESS UPSETS AND EQUIPMENT VARIABLES

To determine the sensitivity of the process to upsets and system changes, it had been proposed that a series of experiments would be conducted after the production phase was completed. Since the full production goal was not achieved, these deliberate process upset experiments were not conducted. Natural perturbations and equipment problems during the normal production operation created system changes equivalent to the variations proposed. The following process variables and upsets will be discussed in the following paragraphs:

(1) Reactor productivity per tube versus AN/U feed ratio.

(2) Reactor productivity per tube versus reactor feed temperature.

(3) Reactor productivity per tube versus percent water in feed.

(4) Insolubles formation versus the AN/U feed ratio.

(5) Insolubles in product versus batch number.

(6) Multiple reactor shutdowns, startups and cooldowns.

(7) Loss of reactor melt and off-gas flows.

(8) High and low solids content of quench tank.

(9) Upsets in operation of centrifuge.

(10) Loss of insolubles separation operation.

(11) Catalyst fouling, poisoning, and attrition.

The conclusion based on the system's response to the above upsets is that the U/AN integrated process is a stable operation that can accept natural process upsets and variations without deleterious effects on long-term operation or product quality. The highest order of failure is the loss of catalyst activity. The state of the technology of the U/AN process used for the pilot plant is well understood.

1. Variables Affecting GN Productivity Per Reactor Tube

The most important step in the process is, of course, the reaction step in which the GN is produced. The parameters that affect the GN productivity per tube are the reactor geometry, catalyst activity, reaction temperature, feed rate, and feed composition (AN/U ratio, percent water in the feed). The reactor geometry is, of course, set by the process design and does not change during operation. It is significant to note, however, that actual versus theoretical conversions from the reactor design were demonstrated. No loss of catalyst activity was detected in this work as was noted in the catalyst mileage discussion in the operation section of this report.

Based on earlier work on this contact conducted with 2-inch and 4-inch-diameter reactors, it was theorized that, within a 50%-200% feed rate (or residence time) variation range, the productivity per tube does not vary appreciably (conversion X feed rate). Residence time is defined as reactor void volume divided by volumetric feed rate. For example, residence time in a packed 4 inch diameter x 10 foot tall reactor (0.42 void fraction) and a feed rate of 27 lb/hr is 60 to 70 minutes. This theory was neither confirmed nor disputed during the pilot plant campaign because of inaccurate feed rate measurements and a common product melt manifold. The general conclusion reached was that the production per tube is not very sensitive to feed rate due to the normal operating feed rate swings encountered.

The other parameters affecting productivity per tube noted above, i.e., temperature, AN/U ratio, and feed water, are important variables.

a. Reactor Productivity Per Tube Versus Time

Figure 14 presents data for the reactor productivity per tube versus time and shows how this value varied during the operation. Most of the swings in this curve are explained by variations in any of the three parameters noted above or by startup or shutdown periods. A downward pulse in the curve at point A represents a period of high water content in the feed (1.7% vs. 0.5%), resulting in excess urea hydrolysis and thus increasing the effective AN/U ratio. The lowered productivities in the periods labeled B, C and D on Figure 14 are from low AN/U ratios blended in with high water feed contents. Points B and C show the effect of reducing the water in the feed from 4.0% to 1.7%. Point E on this curve is a value calculated at an extremely high AN/U ratio (3/1) in which the productivity calculation is not as accurate (See Calculation section) and with incomplete urea hydrolysis (141% yield). Point F shows a lower productivity due to a 4°C drop in temperature for a short duration.

The areas in Figure 14 labeled G and H represent periods of operations in which process problems occurred. In area G, a plugged gas-liquid separator melt take-off line allowed three-fourths of the product flow to be diverted to the off-gas scrubber. The collected samples in this period were nonrepresentative of the total melt. Period H was a startup period after a shutdown (necessary to correct the above problem), and the high productivity represents a flushing of the tubes on startup. Point I was another temperature drop. Area J represents a period of smooth operation. The cycle in the curve follows the cycle recorded for the AN/U ratio during this time period.

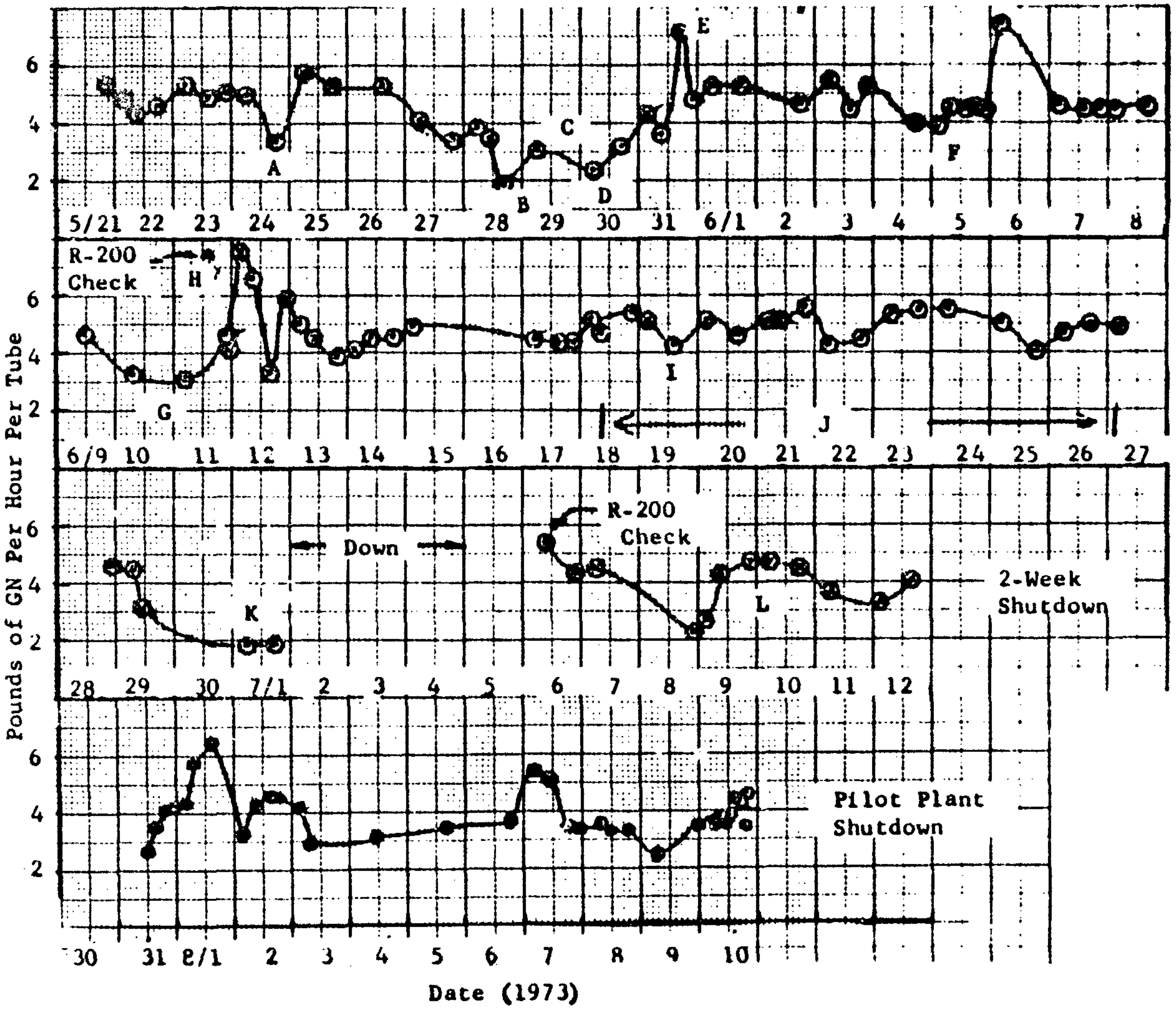


Figure 14. Calculated Productivity Per Tube

The portions of the curve for the periods of June 28 to July 1 (K) and from July 5 to July 12 (L) represent unstable operations due to compounded high feed water and ammelide fouling. In this period, a decrease in the feed water content would have resulted in an increase in the productivity (Figure 14).

The above conclusions related to the swings in Figure 14 were derived by simultaneous comparison of Figure 3 (plant operating chronology), Figure 15 (feed AN/U ratio versus time), Figure 16 (feed water versus time), and Figure 17 (reactor temperature versus time). The productivity graph (Figure 14) was previously presented in the operation but repeated in this section (with notations) to aid in following the above discussion.

b. Reactor Productivity Per Tube Versus AN/U Feed Ratio

Figure 18 presents the data for reactor productivity versus the reactor feed AN/U ratio. A trend line is drawn through these data. This line does not represent a regression fit but merely splits the available points. The trend of increased production with increasing AN/U ratio appears on the surface to be inconsistent with previous conclusions of increased conversions with decreasing AN/U ratio. The explanation for this departure is that the ordinate of Figure 18, GN productivity per tube, is derived by using both the conversion and yield. The urea yield decreases as the AN/U feed ratio decreases. A trend line of the slope of the curve in Figure 18 would result if the urea yield decreased faster than the conversion increased as the AN/U feed ratio decreased.

c. Reactor Productivity Per Tube Versus Reactor Temperature

Reference is made to Figures 14 and 17, with curves showing productivity per tube versus time and temperature of R-200 (78 inch height) versus time, respectively. As discussed in Section E.1.a., above, there are definite pulses in the curve of Figure 14 which can be explained only by pulses in temperature. In addition to those referred to in that discussion, additional low productivity - low temperature points occurred on June 3, 4, and 25 and high productivity - high temperature points occurred on June 19 and 21.

In an attempt to show a step in the curves for both GN productivity and insolubles formation, an intentional upset was introduced into the system on August 9. The steam pressure on the reactor jackets was increased from 190 to 225 psig and left at this new pressure until the end of operation (2 days).

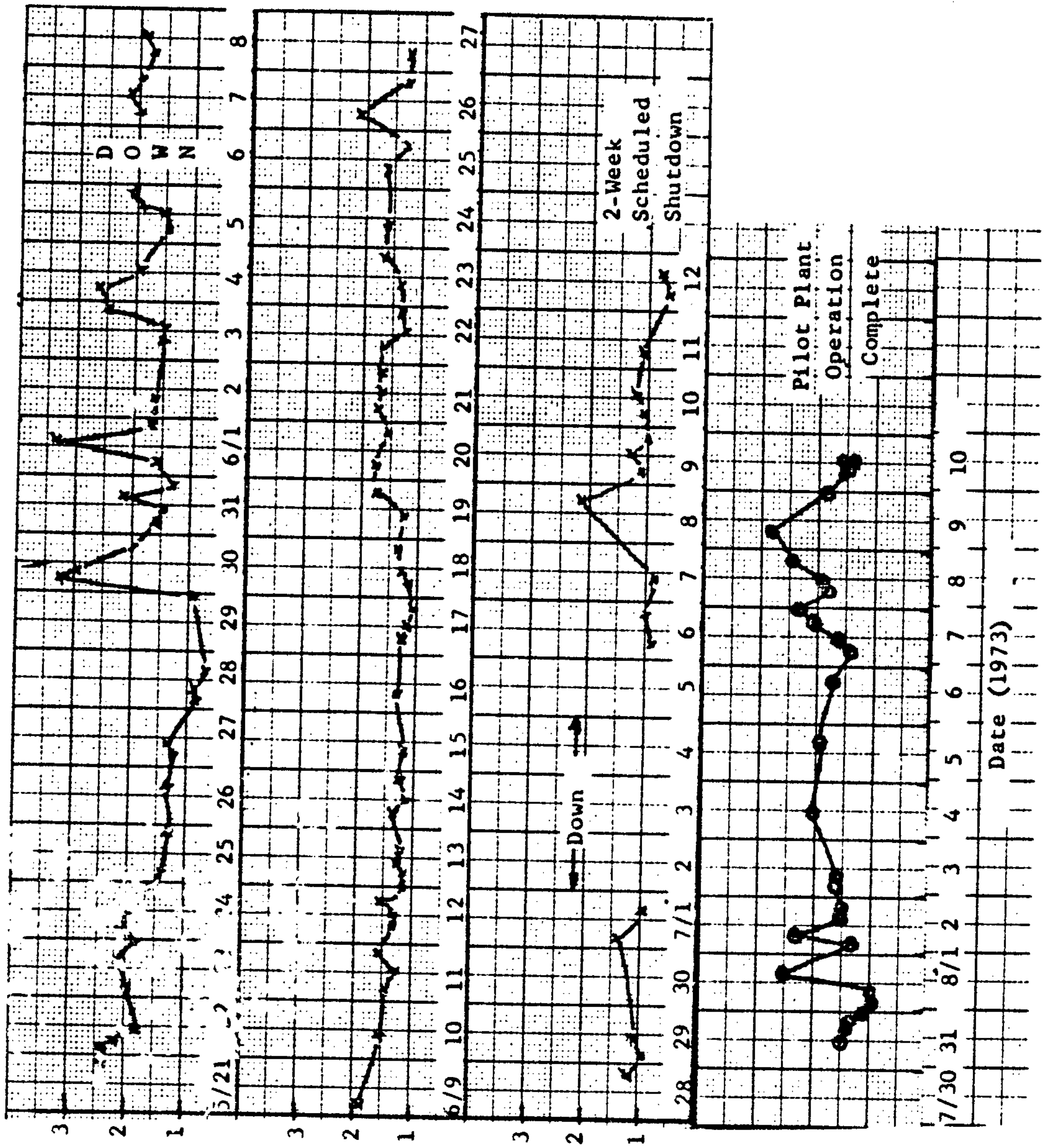


Figure 15. Reactor Feed AN/U Molar Ratio Versus Time

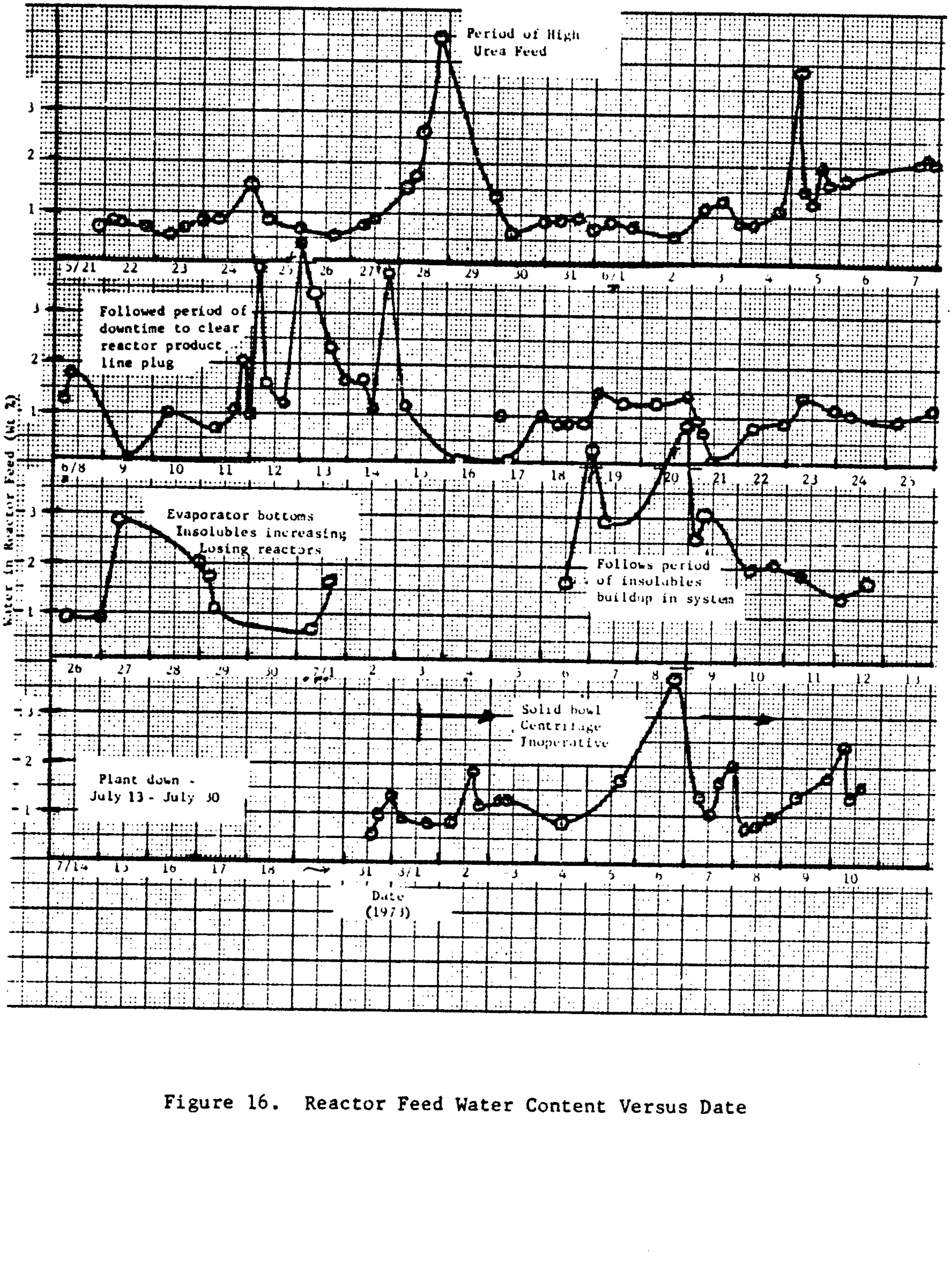


Figure 16. Reactor Feed Water Content Versus Date

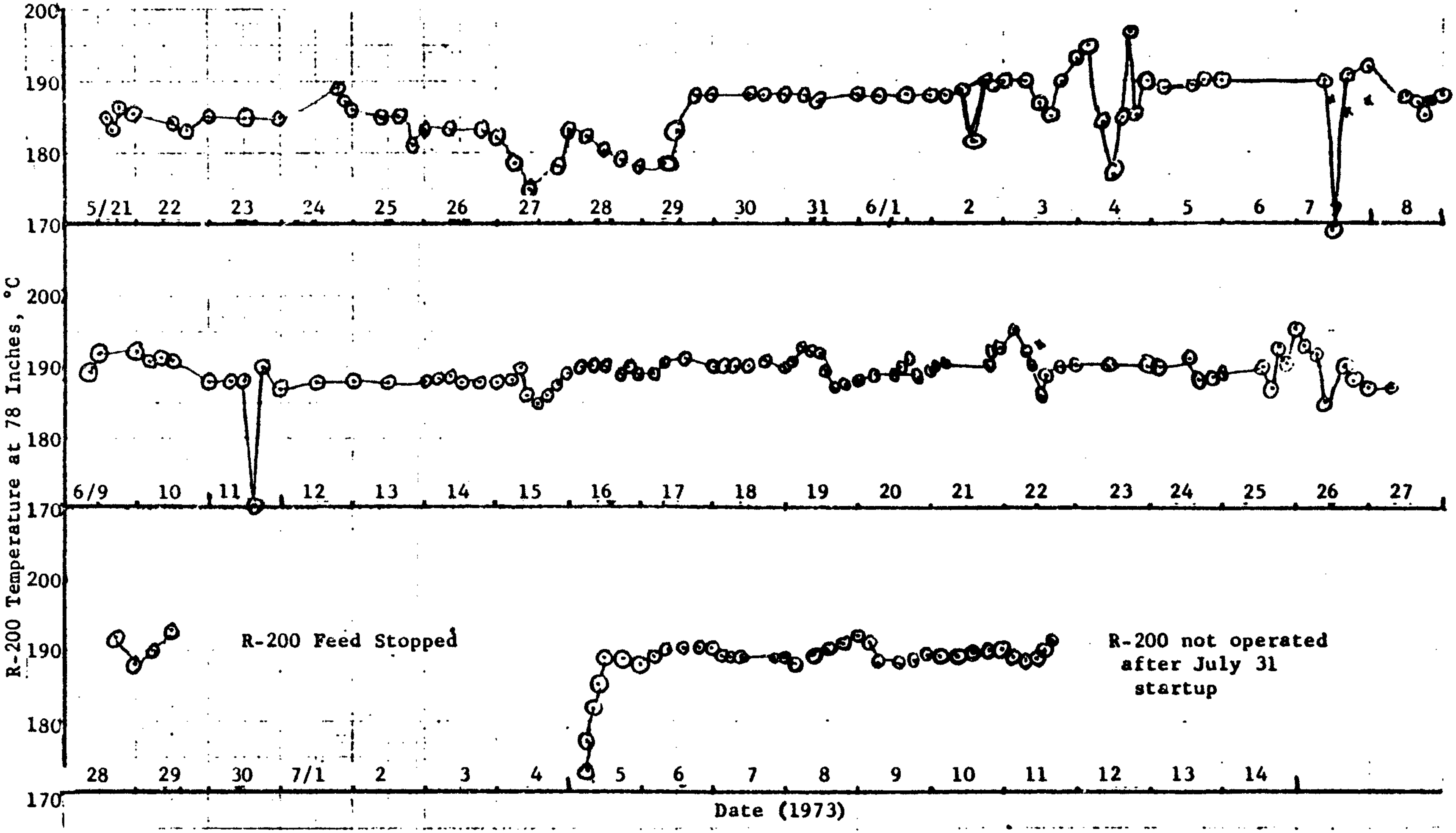


Figure 17. Temperature of R-200 at 78-Inch Height as a Function of Date

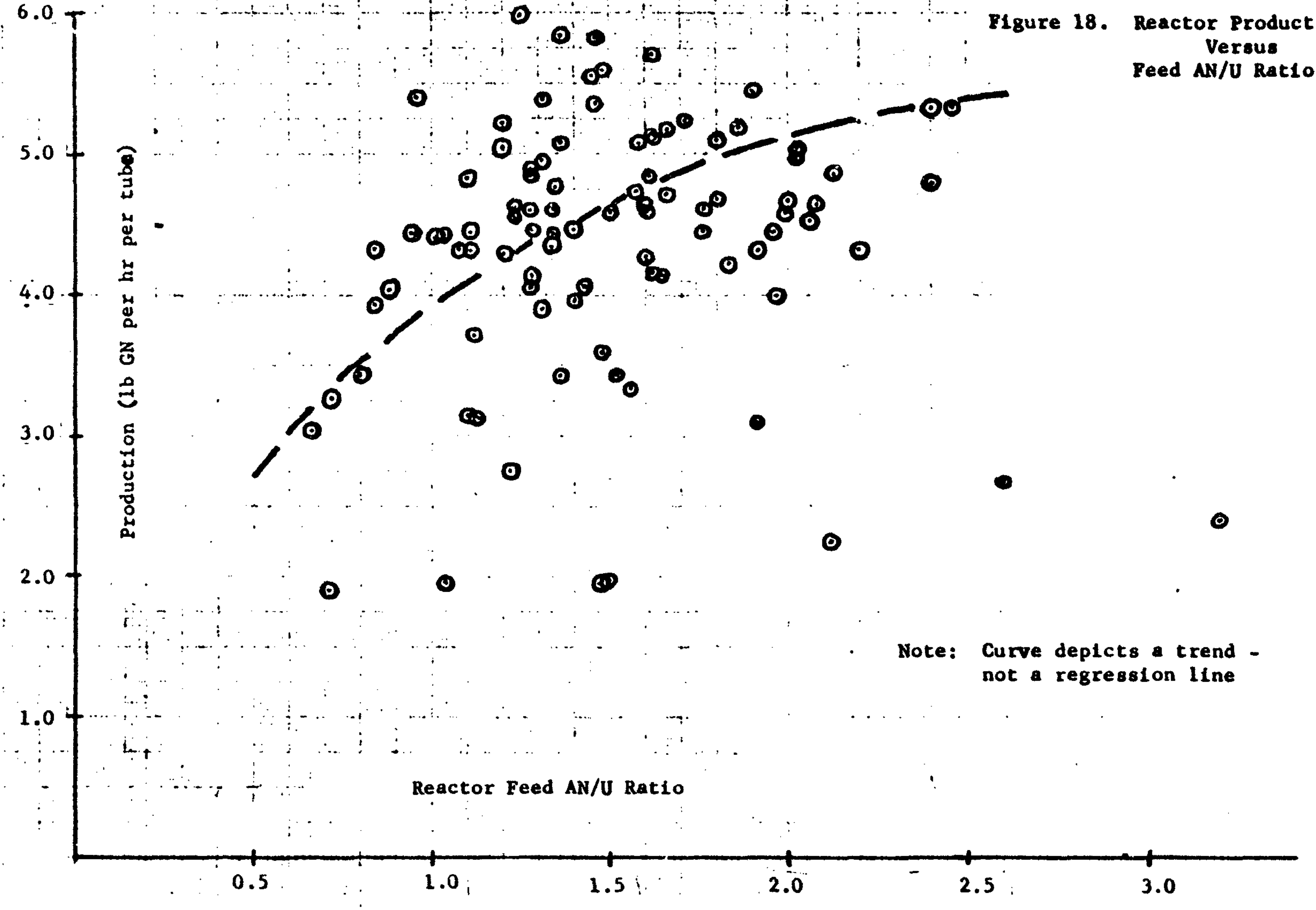


Figure 18. Reactor Productivity Versus Feed AN/U Ratio

Note: Curve depicts a trend - not a regression line

Unfortunately, the reactor tubes with the programmed temperature profile probes (R-200 and R-204) were inoperative by this date, so actual data on the resulting temperatures were unavailable. Past experience and data on such changes suggest that the jacket temperature would have increased from 198°C to 205°C. The resulting centerline temperature at the 78-inch axial position would have increased from about 189°C to about 195°C.

Examination of Figures 14 and 19 shows that sharp increases in both GN productivity per tube and insolubles occurred with this temperature increase. This is in complete agreement with the theory of the chemistry of the U/AN process. A quantitative assessment of this resultant change was not attempted since the other parameters in the system (AN/U ratio, water in the feed, number of tubes, and feed rate) were not held constant during the two-day period.

d. Reactor Productivity Per Tube Versus Percent Water in the Feed

Reference is made to Figures 14 and 16, with curves depicting GN productivity per tube versus time and water in reactor feed versus time, respectively. In the discussion above regarding Figure 14, a number of the productivity drops were attributed to high water content in the reactor feed. Excess water in the feed results in increased hydrolysis, thereby decreasing the urea available for the GN reaction. This increases the effective AN/U ratio fed to the reaction and results in a lower productivity.

Examination of Figure 16 shows periods of very high water level for May 28-29, June 5, June 12-14, June 27-28, July 7-8, and August 6. Figure 14 shows that for every one of these periods, there was a decrease in the reactor tube productivity.

The sources of water for these upsets were the reactor feed pump and melt transfer packing purges, the recycle stream from the evaporator, and hydroscopic pickup in the melt tanks. Maintaining a steady reactor feed flow ensured minimum residence time in the melters and minimized the packing purges. Proper control of the operating conditions on the evaporator minimized this water input source. Most of the upsets experienced in this production campaign can be attributed to the latter source. The frequency of these upsets increased after the apparent ammelide fouling. Figure 20 shows the evaporator bottoms water content versus date. Moisture content of the bottoms stream was erratic following the ammelide fouling problem.

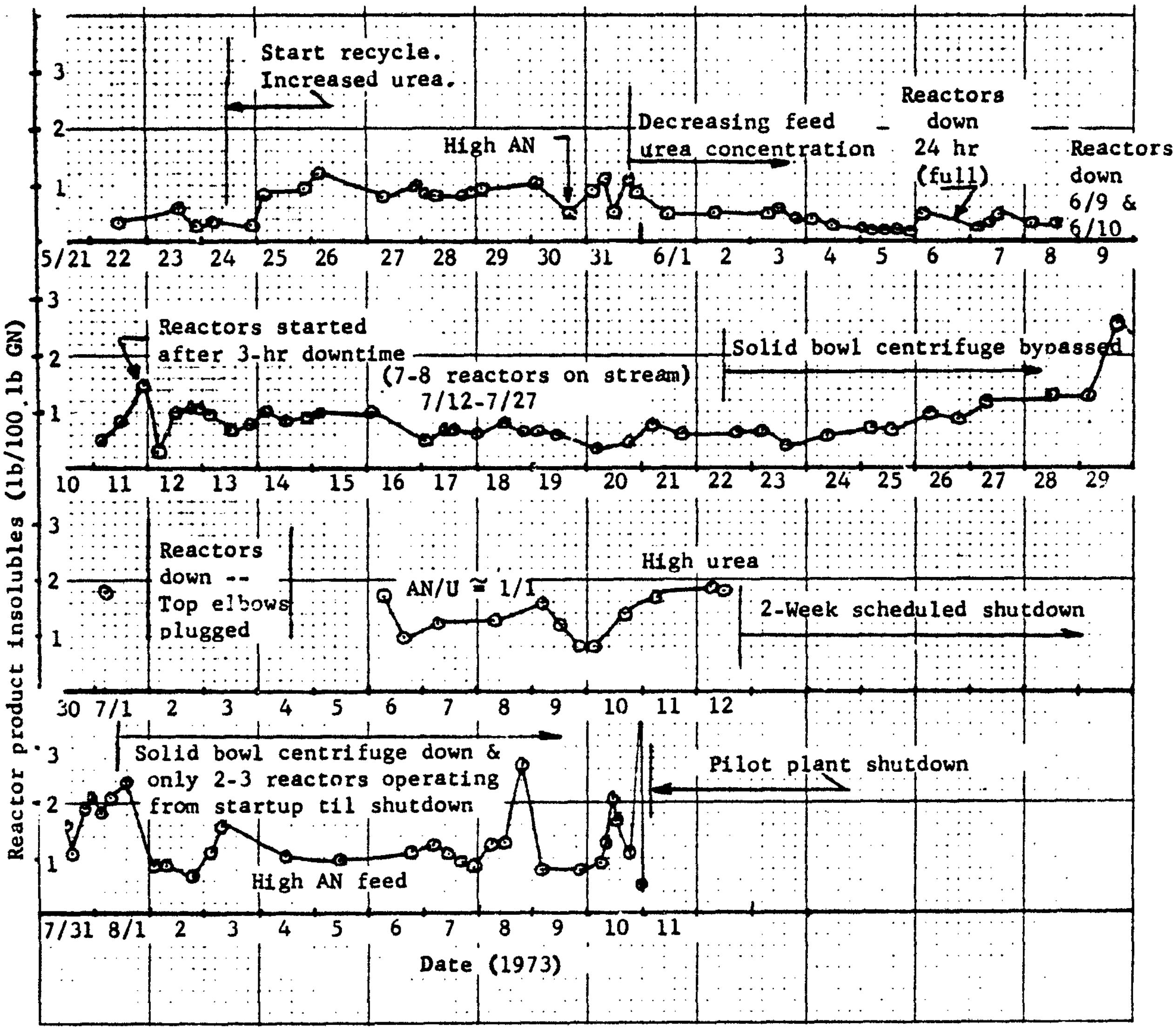


Figure 19. Reactor Melt Insolubles Versus Time

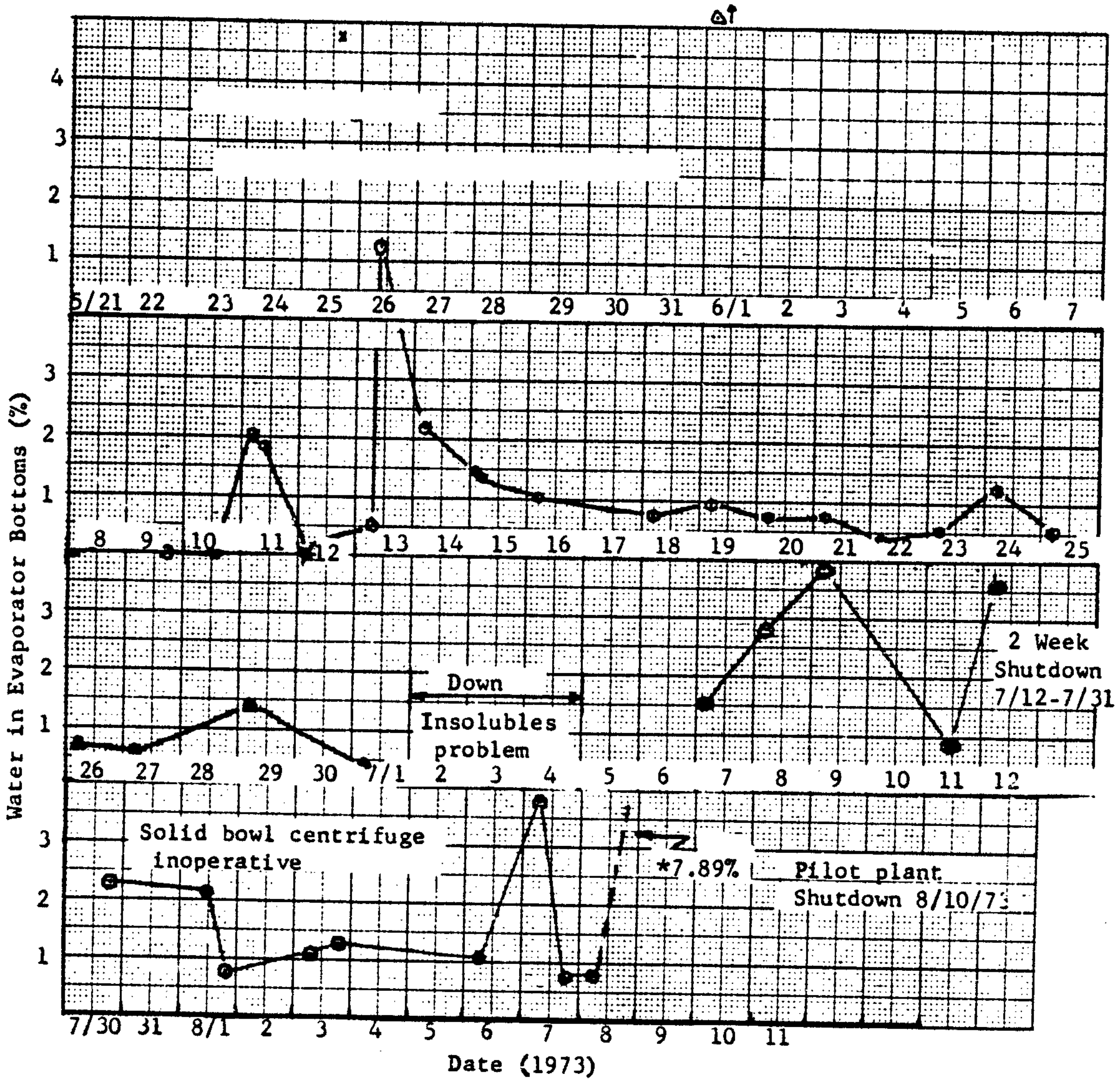


Figure 20. Water in Evaporator Bottoms

e. Insolubles Formation Versus AN/U Feed Mole Ratio

Figure 21 presents data for reactor melt insolubles versus the reactor feed AN/U ratio. These data were derived from the time plots of these parameters in Figures 19 and 15. A trend line is drawn through selected data in Figure 21. Data not considered in drawing the trend line represent either periods of operation during which the solid bowl centrifuge was inoperable or periods when the reactor feed water content was high. With our solid bowl centrifuge inoperable, the melt would be higher in insolubles because of the insolubles recycled in addition to those produced. High water contents would result in a lower AN/U ratio than was actually fed to the reactor, thereby skewing the data to the right of the curve. Examination of several of these points shows that correction of the data to account for the high water moves the points very close to the trend line.

The finding of an increasing insolubles formation with lower AN/U ratio is in agreement with theory. The fact that the trend line is a hyperbola is consistent with the theorized kinetics of insolubles formation in relation to GN formation. The insolubles formation kinetics is believed to be multiple-order in urea concentration (3U$\rightarrow$1A) whereas the GN reaction kinetics has a first-order urea concentration term.

f. Insolubles in Product Versus Batch Number

Figure 22 presents GN product insolubles versus batch number data. The peaks and cycles in this graph are representative of operating problems in the total workup system (solid bowl centrifuge, crystallizer polishing filters, centrifuge washing). When the workup system was functioning properly, the final GN product was low in insolubles regardless of the reaction conditions or product. One conclusion from this work is that, with a workup system functioning as designed, the product assay is essentially independent of the reactor operational history. If the workup system is not functioning in some aspect (e.g., solid bowl centrifuge), the product does reflect reactor performance. The most troublesome operation was the insolubles separation. This is indeed reflected in the product insolubles level. During the steady-state and mechanically smooth operating period represented by Batches 151 through 186 (Figure 22), the GN product insolubles level was almost consistently less than 0.5%. Examination of Table I shows a few batches of GN in which the AN was high. These batches also reflect less than optimum operating performance of the workup system. In this case, either the melt/water quench ratio, the crystallizer cooling cycle, or centrifuge washing step had not functioned as designed.

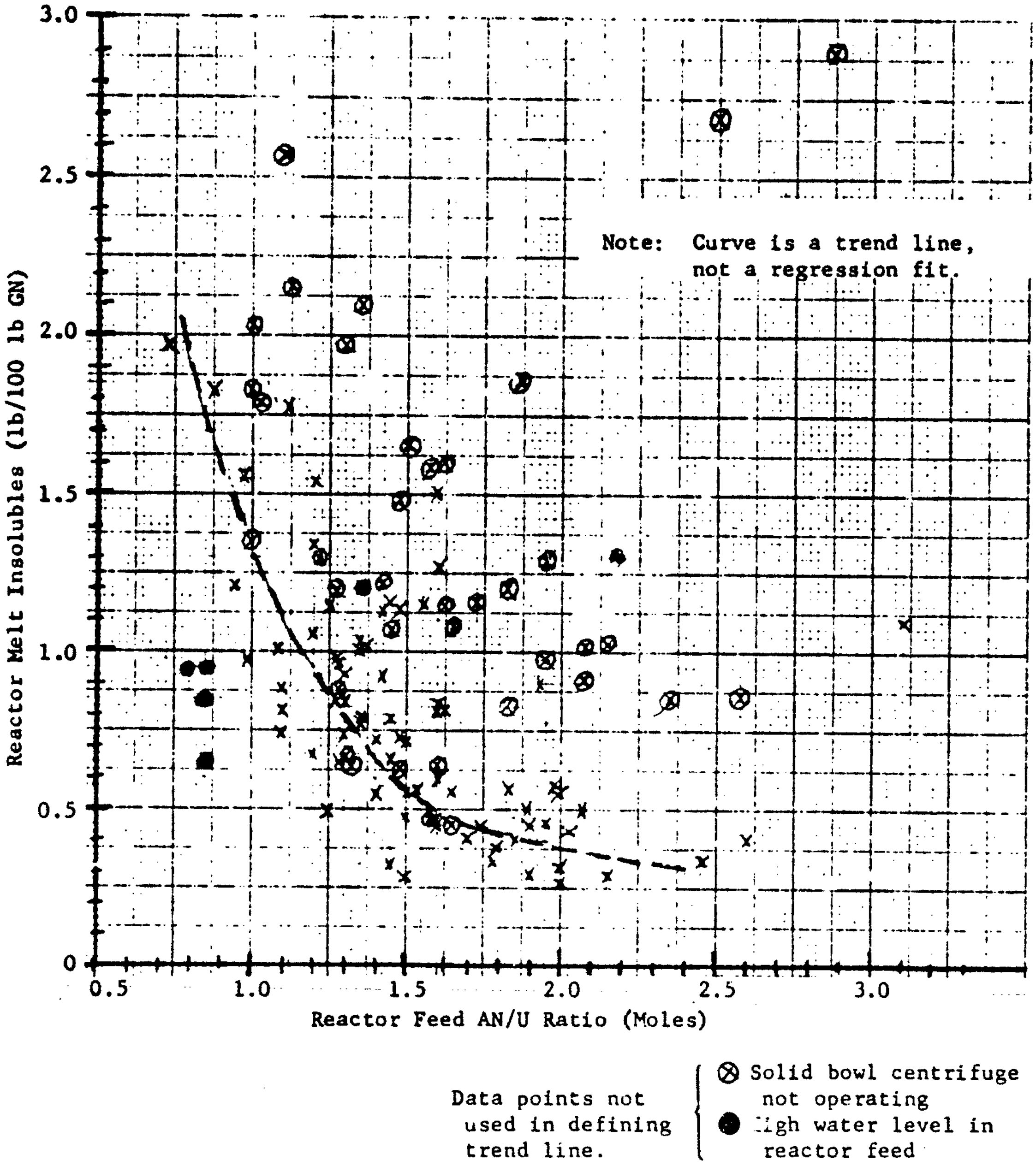


Figure 21. Effect of Reactor Feed AN/U Ratio on Insolubles Formation

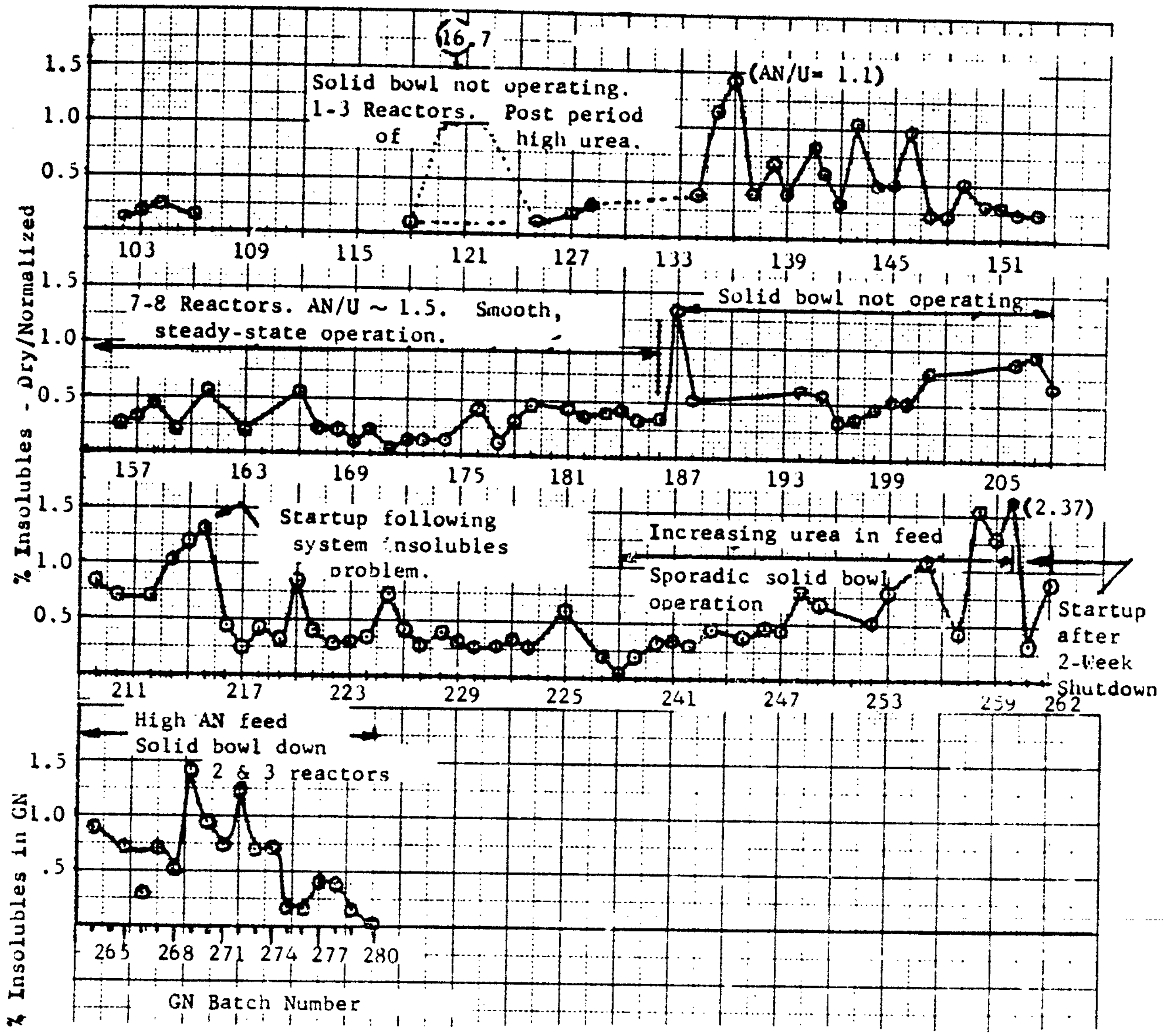


Figure 22. Guanidine Nitrate Insolubles Versus Batch Number

g. Multiple Reactor Shutdowns, Startups, and Cooldowns

Examination of Figure 3, the chronology chart, shows that the reactor tubes were frequently started up and shut down for various reasons. When a tube was shut down, except for the period of July 14 to July 30, the following procedures were employed:

(1) If the loss of flow was considered to be short-term (less than 1 hour), the reactor temperature was held at its operating value.

(2) If the loss of flow was to be temporary (1 hour), the reactor temperature was lowered to a nonreactive condition (165°C) and held until flow was again available.

(3) If the loss of flow was for a longer term than four hours, the melt in the tube was drained and the temperature reduced to about 165°C.

In all incidents in which loss of flow to a tube occurred, if flow could be resumed, the GN productivity was not affected. In fact, the first product from a resumed tube usually showed higher than normal conversion, suggesting that the longer hold-up was not completely reaction free. In cases where flow could not be resumed, it was found on later examination that the catalyst or upper screens and elbows were laden with insolubles.

On July 14, the pilot plant was shut down for 2 weeks because of a scheduled loss of steam. The Kenvil plant boilers were shut down for a yearly inspection and maintenance overhaul. During this period, no heat was provided to the reactors. Before shutdown, the reactors were flushed and drained two times with melt at low temperatures. After the two-week layoff, attempts were made to start these tubes. Only three of the six tubes operating before shutdown could be restarted. The reason for the inability to pump melt through the other three tubes is unknown. Whether it was due to the shutdown, to buildup of insolubles just before shutdown, to fines settling on draining, or to a combination of the above could not be determined. It is assumed that a combination effect was the cause, since two other tubes had became inoperable due to insolubles and the remaining tubes had long service, allowing catalyst fines to sift through the beds upon draining. This would increase the resultant pressure drop required for melt flows. In all cases, when the reactor tubes could not be restarted, the loss of the tubes was due to a higher required flowing pressure drop than that available from the feed pump. In every attempt to restart these tubes, flow could be sustained up into the bed to an estimated 9 to 10 feet before flow was lost.

In summary, the following conclusions can be made about flow through the catalyst beds:

(1) Flow can be restarted through the beds if the exposure of the bed to the following environments is minimized:

Extreme insolubles level

Numerous drainings

High temperature for long no-flow periods.

(2) Resumption of flow is questionable if the beds are cooled with melt remaining in the bed.

The latter event probably results in crushed catalyst, presumably due to crystallization of the ammonium nitrate. In any event, reactor tubes shut down under these circumstances and then cooled were difficult to dump.

h. Loss of Reactor Melt and Off-gas Flows

Two of the process upsets experienced on occasion during the 1973 campaign were loss of reactor product melt flow and loss of off-gas flow. The former would occur if a reactor product line became plugged. The latter occurred whenever either the off-gas line was plugged or the scrubber bottom outled line was plugged (resulting in a high water level in the scrubber).

The effects of the product melt flow loss upset were as follows: (1) The water/melt ratio to the quench tank became too high, resulting in a low crystallization yield. (2) The melt would have to be diverted to another path (e.g., off-gas line), resulting in a material balance loss. Either one of these events is unfortunate but not disastrous. Correcting the problem source (plugged line) immediately corrects the resultant event. Product flow to the proper path is resumed, and the crystallizer yield for the next batch is regained.

The result of an off-gas line plug is that the off-gases leave the reactor system with the melt. The effects of this were (1) leakage of the ammonia to the process bay, creating an unpleasant working atmosphere, and (2) centrifugal pumping problems due to the resultant gas-liquid quench mixture. Again, once the upset source (plugged line) was corrected, the system effects disappeared.

The conclusion that can be drawn from the above discussion is that process upsets attributed to loss of flow result in only a temporary inconvenience and do not have a long-term effect on the process operation.

i. Solids Content of the Quench Tank, GN Centrifuge Upset and Inoperative Solid Bowl Centrifuge

Three other process upsets that occurred during the pilot plant operation were (1) variation of the solids content in the quench tank, (2) upsets in the operation of the GN centrifuge, and (3) loss of the solid bowl centrifuge.

A high solids level in the quench tank created a process upset because the excess solids would crystallize out of solution in the lines or solid bowl centrifuge, plugging the lines, reducing the yield and causing the solid bowl centrifuge to malfunction. Low solids level in the quench tank would result in a smooth workup system but would reduce the crystallizer GN yield and increase evaporator load. Operation control of the water/melt ratio to the quench tank minimizes the above effects. Implementation of a periodic quench tank density reading improved the control of this important parameter.

The GN centrifuge operation was upset during the campaign by (1) a high charging rate, (2) wash water/solids ratio, or (3) filtration efficiency due to cloth blinding or fines buildup in the residual heel. A high feed rate had the effect of flooding the inlet core of the centrifuge and would result in inadequate filtration through the cake and loss of feed liquor out the solids discharge end (bottom discharge). If the wash water volume was too low in proportion to the solids level in the basket, the product assay would reflect an increase in the mother liquor constituents (AN and urea). If the wash water level was too high, the result was a yield loss to the mother liquor stream (eventually recovered via recycle).

On a few occasions, the filtration efficiency of the centrifuge was severely reduced. It was determined that the cause of this reduction in efficiency was (1) blinding of the filter cloth with ammelide and/or GN fines, (2) a fines-laden batch of GN caused by rapid crystallization, or (3) a hard cake or heel due to high-speed operation during charging of the GN slurry. Occasional removal of the heel followed by a soda ash flush would correct the first upset source. Proper control of the crystallizer cooling during this campaign apparently prevented the second difficulty (high-fines AN). The minimum cooldown period used in this work was 1-1/2 hours. Experience in feeding the centrifuge eliminated the third problem (hard cakes) after the first few batches. None of the noted upsets created more than a process inconvenience or a minor yield loss.

The most frequent process upset was loss of the solid bowl centrifuge. When the solid bowl centrifuge was bypassed, the insolubles flowed to the crystallizer feed tank. This meant that they had to be removed by the crystallizer feed polishing filters, crystallized out of the system with final product, or recycled back to the feed system. In the first instance, use of the polishing filters, an operational bottleneck was created since the feed rate to the crystallizer became severely restricted. Depositing the insolubles out of the system with the final GN product affects the final product assay. Both of these results are trivial when compared with the stability of the total operation. The recycling of the insolubles back to the feed system was a surprising occurrence and turned out to be a significant process upset. In the pilot plant, allowing the insolubles to get into the recycle system fouled the evaporator (with subsequent reduced water-stripping efficiency of the evaporator), and the ball check valves of the Hills-McCanna blend pump (with subsequent erratic performance of the feed blending step), and filled up the catalyst beds with insolubles (with subsequently higher required pressure drop and eventual loss of flow). The loss of reactor performance from loss of flow caused by insolubles fouling of the catalyst bed is a longer term failure in terms of time for the system to respond after the upset so that it is of major significance. Complete, or at least consistent, insolubles removal is essential in the full-scale plant if the catalyst mileage is to be optimized.

j. Catalyst Fouling, Poisoning, and Attrition

As noted above, catalyst fouling due to insolubles buildup in the bed is a major process upset. It can be minimized by proper control of the reactor conditions (so that only a small amount of insolubles is manufactured), and by complete removal of insolubles before the aqueous crystallizer. It is believed that a small amount of insolubles can be pumped through the reactors with the product melt so that buildup of insolubles in the bed under these conditions would not be appreciable. The effects of catalyst poisoning as a process upset were clearly demonstrated in 1972, when diammonium phosphate poisoning occurred. Efforts to control this upset in the 1973 operation by controlling the purity of the ingredients entering the system (ammonium nitrate, urea, process water) were very successful.

The third type of catalyst failure, attrition, can lead to problems of high-pressure drop and to fines being carried over into the quench system. This failure cannot be eliminated because one of the parameters controlling catalyst breakage is the physical properties of the catalyst itself. It can be minimized, however, by not subjecting the catalyst beds to thermal cycling and by keeping the water content of the feed to an absolute minimum. The effects of the latter condition on attrition have not been determined. Experience in 1972 on other forms of silica gel suggested that water does affect the catalyst breakage rate. The level of attrition due to water on Grace silica gel and Mobil

Sorbeads was extremely high. In 1972, the limited exposure of Houdry beads to water suggested little if any attrition. Based on the 1973 run and the fines found in the quench tank, attrition apparently does occur with Houdry beads but at a low level.

F. PILOT PLANT CORROSION TESTING

To assess corrosion potential in future pilot plant or commercial plant equipment, a brief corrosion study was performed during Phase I. In that work, samples of 5052 aluminum and 304 and 316 stainless steel were exposed in the 2-inch-diameter columnar reactor and a feed tank at Kenvil for a short period of time. A preliminary estimate was made of the corrosive potential of the process. The results of that work were presented in Table 19 of the Final Report, Volume I. This table is reproduced in this report as Table 37.

To expand on this preliminary work, corrosion coupons were made up for evaluation in the pilot plant. Hours of exposure for this Phase III effort were ca. 1680 hours, whereas the Phase I exposure was limited to 50-100 hours. The corrosion coupons for the pilot plant study consisted of samples of types 1100, 3003, 5052, and 5986 aluminum and type 304 and 316 stainless steel. Three sample stacks were placed in the system. One stock was placed in the top of reactor R-200 above the catalyst bed (gas-melt zone at 360°F). One stack was placed in the feed blend tank T-113 (in the liquid at 260°F), and the remaining sample stack was placed in the aqueous solution effluent from the solid bowl centrifuge (Tank T-102).

The results from the pilot plant corrosion coupons are presented in Table 38. The overall conclusion from both sets of tests is the same: 304 and 316 stainless steel performed equally well and either should be satisfactory for this service. The four aluminum alloys do corrode in these services and should be used only for selective equipment items. Of the aluminum alloys, aluminum 5086 had the lowest corrosion rate with pitting to 8 mils deep (about 40 mils/year).

It should be noted that part of the corrosion seen on the aluminum alloys could have been caused by the in situ soda ash washes. These washes were conducted on occasion to clear the lines of fouling (suspected ammelide). A similar operation will also be necessary in the commercial plant. A water solution containing 10% soda ash at 80-90°C was used to flush the pilot plant equipment. The soda ash contact was estimated to cover about 2 of the 70 operating days.

A 10% soda ash solution at 90°C will corrode aluminum at rates greater than 50 mils/year. The effect of these flushes alone could theoretically account for 50 mils/year x 2 days/70 days = 1.4 mils/year. Comparison of this value with those in Table 37 indicates that these flushes may have been a major contributor to the measured corrosion.

TABLE 37

CORROSION OF POSSIBLE MATERIALS OF CONSTRUCTION

Specimen Design	Weight (gms)	Weight Loss (mg)	(a) mg/dm^2	(b) mdd	(c) mpy	Evaluation
4.16 Day's Results or 100 hours @ 120°C.		Specimen Exposed in Feed Tank				
16C48 AP (316 SS)	6.2345	.2	1.31	.314	.06	No evidence of corrosion.
35D8 B (5052 AL)	2.1062	.3	1.93	.465	.25	General even corrosion - very light
15C26 CP (304 SS)	4.7898	.1	.68	.16	.03	No evidence of corrosion
2.08 Day's Results or 50 hours (Reactor Operation)		Specimen Exposed in Reactor Effluent				
16C48 AO (316 SS)	6.4544	.2	1.31	.63	.11	No evidence of corrosion.
35D8 A (5052 AL)	1.8269	.8	5.16	2.48	1.34	General even corrosion - very light.
15C26 CO (304 SS)	4.8717	.2	1.35	.65	.12	No evidence of corrosion

Note: Weighing accuracy with our Mettler Balance is ± 0.2 mg
(a) mg/dm^2 = milligram per decimeter squared
(b) mdd = milligram per decimeter per day
(c) mpy = mils per year

TABLE 38

PILOT PLANT CORROSION TEST RESULTS
(TOTAL EXPOSURE Ca. 1680 HOURS)

Sample Location	Alloy, Code and Specimen	Mils Per Year	Evaluation
Feed Tank (T-113)	316SS 16C43M	Nil	No evidence of corrosion
	3003 Al 35C14Q	6.7	Severe pitting and localized corrosion. Pits to 4 mils deep and varying widths.
	5052 Al 35D8E	5.3	About same as above
	304SS 15C26EY	Nil	No evidence of corrosion
	1100 Al 35B8AE	8.76	Severe pitting and localized corrosion. Pits to 6 mils deep and varying widths.
Reactor (between catalyst and top support screen)	316SS 16C43P	0.03	No evidence of corrosion
	5086 Al 35D11B	5.5	Localized corrosion. Remainder of coupon is general corrosion. Several shallow pits.
	3003 Al 35C14R	11.1	More general even corrosion. Shallow type pits. 3 or 4 pits to 7 mils deep.
	304SS 15C26FB	0.05	No evidence of corrosion
	1100 Al 35B8AF	11.5	Localized corrosion. Some pitting to 5 mils deep.

TABLE 38 (CONT.)

Sample Location	Alloy, Code and Specimen	Mils Per Year	Evaluation
Solid Bowl Effluent Tank (T-102)	316SS 16C43Q	Nil	No evidence of corrosion
	5086 Al 35D11C	0.35	Localized corrosion. Numerous pits. Largest is 8 mils deep and 18 mils wide.
	5052 Al 35D8F	0.95	Localized corrosion and pitting.
	3003 Al 35C14S	2.28	Same type corrosion as above except worse.
	304SS 15C26FC	Nil	No evidence of corrosion
	1100 Al 35E8AG	2.17	About same as 3003 Al.

G. PRODUCTION PLANT DESIGN

A pilot plant is constructed and operated for many reasons; however, its primary objective is to obtain process design data. This objective was achieved in operation of the guanidine nitrate pilot plant located at Hercules/Kenvil. The plant was operated on total recycle, and all of the basic unit operations were demonstrated. Prototypes of commercial processing equipment were evaluated; e.g., reactors, Whitlock evaporator, solid bowl centrifuge for insolubles recovery, Swenson crystallizer, DeLaval basket centrifuge for GN recovery, indirect heated Wyssmont Turbo Tray dryer, etc. Experienced process and project engineers can design a production plant by reviewing the data, results, conclusions, etc., presented in this Final Report, Volume II and Final Report, Volume I dated August 1973. A few design suggestions, which may be overlooked by a designer, are presented below. These suggestions are based on first-hand experience as a result of operating the pilot plant.

1. Positive gasketing should be provided for reactors, particularly for the top heads. In these pilot plant operations, two or three instances of leaking gaskets were experienced which resulted in smoldering of the reactor melt on the hot reactor jackets. Suitable smoke and/or fire detection devices should be considered.

2. High melting point AN/U/GN mixtures must be processed in jacketed lines. Where jacketing is not practical, a good tracing system coupled with heat transfer cement must be employed. Crosses and/or tees should be installed at strategic locations to facilitate line cleanout.

3. Reactor design should be based on use of the total internal volume for catalyst; i.e., no freeboard space between the catalyst and the catalyst retention screens. This could minimize catalyst attrition problems if attrition is attributed to physical movement of the Houdry beads.

4. Although separation of reactor off-gases from the reactor product melt is rapid, a small amount of liquid entrainment in the off-gas can be expected. Ammonia odors were always present in the GN recovery section of the pilot plant. Two design suggestions are (a) a demister should be installed in the gas-liquid separator gas line, amd (b) the melt should be sparged with nitrogen before quenching to minimize ammonia in the workup end of the process. A liquid seal loop in the gas-liquid separator melt drain line is a must. Catalyst fines will inevitably enter the separator. Provisions should be made to remove these fines without resorting to a reactor shutdown.

5. A means of measuring the mass flow rates of melt and off-gas from each reactor should be provided. Such measurements would be useful in monitoring reactor performance. The melt flow measurement could be cascaded to control the rate of quench water, thus controlling the workup end of the process.

6. Control of feed rate to each tube in a reactor bundle would be prohibitive in terms of costs. A multi-orifice feed distribution system should be designed for a 20-30 psig pressure drop and for no more than 100% excess flow.

7. The reactors should be heated with steam (or a compatible organic vapor system) for minimum temperature differential and there must be absolute provisions for eliminating an exotherm. Jackets should be designed so that the heating medium cannot enter the process; i.e., no welds where subjected to steam. This philosophy also applies to AN/U melt lines, evaporator, etc., upstream of the reactor system.

8. Mechanical seals on all pumps and an appropriate selection of seal fluid are recommended. Positive displacement pumps with ball check assemblies should not be considered.

9. A continuous solid bowl centrifuge should be selected for removal of insolubles from the crystallizer feed stream. Polishing filters in the evaporator feed line are also recommended. Provisions must be made for sodium carbonate washing of recycle equipment and lines and for manual cleaning of evaporator tubes.

10. Catalyst will not necessarily flow freely from the reactors during the dumping operation. A high-pressure (300-500 psig) water jet was used successfully in the pilot plant to remove caked catalyst from the reactors. Such equipment should be considered for a production plant.

11. Rework systems should be provided in a production plant: (a) an aqueous system for slops, spills, etc., to be fed to the evaporator, and (b) an aqueous repulper for reworking off-grade GN.

12. Phosphate-free water must be provided for reactor product quenching and washing of GN centrifuge cakes.

H. PILOT PLANT LAYAWAY

Following completion of the pilot plant guanidine nitrate production operation, the pilot plant was placed in a layaway condition for either possible future operation or dismantling for shipment at the U. S. Government's request. All eight reactor tubes were emptied of catalyst by using a high-pressure water jet. Some catalyst which could be removed without the use of a water jet was collected in polyethylene bags, retained, and identified by reactor number. Removal of the catalyst support screens was difficult, and in some instances the screens were punctured to facilitate removal of the assembly. Sleeve assemblies were left in two of the reactors, and these must be removed if the reactors are to be used again. This problem of stuck catalyst support screens can be resolved in future designs.

The primary concern in placing the pilot plant in a standby condition was to make certain that all equipment was clean. All process lines and equipment were initially drained, flushed with a hot sodium carbonate solution, and rinsed several times with hot water. This treatment removed all water soluble materials such as AN, GN and urea and a major portion of the insolubles (ammelide). Post-inspection of randomly selected process lines showed the presence of some internal ammelide scaling. The tops of all tanks, except those for T-104 and T-105, were removed for internal cleaning of the tanks. The top and bottom closures of the evaporator were removed, and a thin uniform coating of ammelide was observed on each of the four tubes. It will be necessary to either physically clean the tubes or heat the evaporator while flooded with soda ash before placing it into service. Quantities of ammelide remain in the system, but its presence should not present any problems in equipment dismantling and shipment. Insulation was removed from all tanks. Tank exteriors, building walls, etc., were washed down with a high-pressure water jet.

Drain lines on all equipment jackets were disconnected to prevent ruptures from freezing of residual water in the event of building steam failure. The steam boilers, vacuum pumps, chiller, high-pressure steam line, tempered water system, process steam lines, etc., were all drained. Air supply to the building was turned off, and the sprinkler system was deactivated. Oil reservoirs for the Hills-McCanna pump, vacuum pumps, chiller compressor and DeLaval centrifuge hydraulic drive system were drained, flushed with light oil, and drained again.

All of the pumps were disconnected from their respective process piping and electrical connections and subsequently painted. After being painted, the pumps were spotted at their operating positions but not reconnected permanently. Process lines, however, were temporarily installed by connecting a few threads or fastening with one or two bolts per flange assembly. A few other pieces of equipment and supports were also painted. The Strong-Scott dryer, demineralizer, bench-scale reactors, etc., were returned to the pilot plant building.

All major pieces of equipment and instrumentation items were identified with respect to equipment numbers and Hercules/Kenvil purchase order numbers. The tagged equipment is listed in Table 39, depicting item description, purchase order number, service exposure, method of cleaning and assessment of condition. This list was placed in a file box, along with other information listed below, located in the pilot plant building.

1. Copy of purchase order for each item listed in Table 38.
2. Drawings and manufacturers' installation/operation/maintenance booklets for the identified pieces of equipment.
3. Miscellaneous documents which may be helpful in the future; e.g., process flow sheet, electrical substation drawings and wiring diagrams.

Control samples, spare parts, and 1000 lb of Houdry CP-532 silica beads (manufactured after terminating pilot plant operation) were stored in the pilot plant area. Doors to the utility house and the process building were locked for security. Scrap process materials were disposed of at the Kenvil plant site. Good quality guanidine nitrate was shipped to Cyanamid of Canada. Off-grade GN was shipped to Picatinny Arsenal to the attention of Mr. C. H. Nichols.

TABLE 39

GUANIDINE NITRATE MAJOR PROCESS AND INSTRUMENTATION EQUIPMENT

Item No.	Item Description	P.O. No.	Purchase Price	Service Exposure	Cleaning	Condition
1	300 KVA Transformer	11978-06	$ 3,900	NA	NA	Good
2	Mark 99 Whitlock 2-Stage Evaporator Unit	12175-10	8,771	GN, H_2O, AN, U	Internal soda ash soln. flush, head removed, light tube scale	Good
3	Swenson Draft Tube Crystallizer Unit	12181-11	12,575	GN, H_2O, AN, U	Internal soda ash soln. flush, not disassembled	Good
4^{A}_{B}	2-Model 6-445-4 Ebcor Steam Generator Unit	12187-11	8,793	NA	NA	Poor(a)
5	DeLaval/ATM Model 12 Basket Centrifuge	12189-11	3,845	GN, H_2O, AN, U, Ammelide	Soda ash soln. flush, some ammelide scale	Good
6	Strong-Scott SJS-8-4 Solidaire Dryer Unit	12193-11	9,560	GN, H_2O, AN, U	Soda ash soln. flush, not disassembled	Good
7	8-Lee Metal S.S. Tanks, 30-200 gals, Jacketed	12195-11 A-H	9,673	GN, H_2O, AN, U, Ammelide	Soda ash soln. flush	Good
8	22 x 16 x 3½ In. Mark III Centrifuge Unit	12195-11	18,230	GN, H_2O, AN, U	Soda ash soln. flush	Good
9	Model K2M-3 Hills-McCanna Metering Pump	12201-11	4,587	GN, AN, U	Heads given soda ash soln. flush, not disassembled	Good
10	Edwards Model CC10A Air Cooled Chiller	12209-11	5,052	NA	Brine drained, compressor crankcase drained	Good
11	Type CL-10HYT Dynatrol Density Cell	12230-12	2,625	GN, AN, U	Soda ash soln. flush	Fair
12	Thermoelectric Digital Indicator and 24 Pt Switch	12229-12	854 A-B	NA	NA	Good
13	Foxboro Electronic Controls	12231-12 A-F	2,904	NA	NA	Fair
14	2-Model N-33-33 Lightning Mixers	12254-12	924	GN, U, AN	Not removed from operat-position	Good

(a) The Ebcor boilers should be thoroughly inspected and overhauled before being used again. The water feed should be controlled and conditioned to reduce corrosion and fouling.

TABLE 39 (CONCLUDED)

Item No.	Item Description	P.O. No.	Purchase Price	Service Exposure	Cleaning	Condition
15	2-Reactor Assemblies (Each Consisting of 4 tubes, 1 feed header, 1 outlet header)	12276-01	$ 8,247	GN, U, AN	Feed headers flushed, reactor tubes emptied, outlet headers soda ash soln. flushed	Good
16	3-Research Control Diaphragm Motor Valves	12301-01	416	GN, U, AN	Flushed	Good
17	No. 08648 SSCF American Standard Heat Exchanger	12323-01	1,277	H_2O, Ethylene Glycol	Drained	Good
18	4-Foxboro Level Control Systems	12326-02 A-E	2,818	GN, U, AN, H_2O	NA	Fair
19	UM-IF Hills-McCanna Pump Unit	12315-02	553	Unused	None	Good
20	Vibra Screw Model BF-75 Belt Feeder Unit	12320-01	2,032	GN, H_2O, AN, U	Hot water flush	Good
21	3-S.S. Tanks, Unjacketed 30-55 gal	12328-02	866	GN, H_2O, AN, U	Hot soda ash soln. flush	Good
22	8 Gould Model 3199-1 x 1-5 Centrifugal Pump Units	12333-02	4,262	GN, H_2O, AN, U	Hot soda ash soln. flush, packings removed	Good
23	8 In. Dia. x 36 In. Jacketed S.S. Tank	12360-02	615	Unused	None	Good
24	Model DA-50-S Culligan Demineralizer	12447-05	2,189	H_2O	Acid, caustic tanks cleaned, resin beds are fouled	Good
25	8-Research Control ½ In. Diaphragm Motor Valves	12579	1,420	GN, U, AN	Flushed	Good
26	8 Each, Foxboro dp Cells and Receiver-Controllers	12518 - A-B	5,818	A - GN, U, AN B - NA	A - Flushed	Good
27	2-Fisher Governor 1 In. Back Pressure Control Systems	12488	1,054	NA	NA	Good
28	Substation-Interrupter Switch - Fuse Mountings	12206-11	2,328	NA	NA	Good
29	1 In. Reactor	-	-	AN, U, GN	Water flushed	Good
			TOTAL $125,686			

I. ECONOMIC STUDY COMPARING TOTAL COSTS FOR MANUFACTURE OF NITROGUANIDINE UTILIZING GUANIDINE NITRATE MANUFACTURED BY THE BRITISH AQUEOUS FUSION - GUANIDINE NITRATE AND UREA/AMMONIUM NITRATE - GUANIDINE NITRATE PROCESSES

ECONOMIC STUDY

COMPARING TOTAL COSTS FOR MANUFACTURE OF NQ
UTILIZING GN MANUFACTURED
VIA PAF-GN PROCESS
VIA U/AN-GN PROCESS

PREPARED UNDER: CONTRACT DAAA 21-71-C-0193

PREPARED FOR: PICATINNY ARSENAL
DOVER, N. J.

SUBMITTED BY: HERCULES INCORPORATED
INDUSTRIAL SYSTEMS DEPARTMENT
910 MARKET STREET
WILMINGTON, DELAWARE 19899

OCTOBER 10, 1973

Table of Contents

Page No.

LIST OF FIGURES

FOREWORD

This economic study was prepared and submitted in accordance with Contract DAAA 21-71-C-0193 between the U.S. Army; Picatinny Arsenal; Dover, N.J., and Hercules Incorporated; Industrial Systems Department; Wilmington, Delaware.

The objective of the study was to perform an economic analysis comparing the total cost, non-recurring and recurring, for the manufacture of nitroguanidine (NQ) via two (2) alternatives:

1. utilizing guanidine nitrate (GN) manufactured via the British Aqueous Fusion (BAF-GN) process
2. utilizing guanidine nitrate (GN) manufactured via the Urea/Ammonium Nitrate (U/AN-GN) process.

The designation BAF-GN and U/AN-GN are used throughout the report to emphasize that the two (2) different processes for the manufacture of GN are the crux of the analysis.

The designation BAF-GN Process is used when referring to any aspect of the process for manufacture of NQ utilizing the BAF alternative. The designation U/AN-GN is used in a comparable manner.

A brief description of only the GN manufacturing process is provided in Section 7.0 Exhibit 7.1. The material balance for the manufacture of GN via each of the processes is provided in Section 7.0 Exhibit 7.2.

The economic study is summarized in Section 1.0, while the remaining Sections 2.0 through 7.0 provide the supporting documentation and data.

ECONOMIC STUDY OF THE MANUFACTURE
OF NITROGUANIDINE
VIA THE BAF-GN AND U/AN-GN PROCESSES
CONTRACT DAAA 21-71-C-0193
PICATINNY ARSENAL
DOVER, N. J.

1.0 SUMMARY OF ECONOMIC STUDY

1.1 OBJECTIVE

The objective of the study was to perform an economic analysis comparing the total cost, non-recurring and recurring, for the manufacture of nitroguanidine (NQ) via two (2) alternatives;

1. utilizing guanidine nitrate (GN) manufactured via the British Aqueous Fusion (BAF-GN) process.
2. utilizing guanidine nitrate (GN) manufactured via the Urea/Ammonium Nitrate (U/AN-GN) process.

1.2 APPLICABLE CRITERIA

1.2.1 AR 37-13 - Economic Analysis and Program Evaluation of Resource Management, effective 1 June 1973, dated April 1973; specifically Format A

1.2.2 FY 74 Inflation Guidance, AMCCP-ER, dated 4 June 1973; specifically inflation factors in Table 1A for Military Construction and Family Housing

1.2.3 The primary analysis was to be performed on the basis of constant (mid year 1973) dollars and the secondary analysis on the basis of current (inflated) dollars.

1.2.4 Economic life is ten years, (with sensitivity analysis)

1.2.5 Analysis was to be performed for operating rates of 100% and 25% of capacity.

1.2.6 The design criteria and design costs for the BAF-GN process were considered as sunk cost and were not to be included in the analysis.

1.2.7 No cost was to be included in the U/AN-GN analysis for purchase of NQ from Cyanamid of Canada.

1.2.8 Working capital requirements were to be based on 4 weeks of raw materials and returned at end of economic life.

1.2.9 No terminal value was to be included for the investment.

1.2.10 U/AN-GN catalyst costs were to be in accordance with Picatinny Arsenal letter, Mr. C. H. Nichols, dated August 16, 1973.

1.2.11 Timing of cash flows was to be in accordance with the NQ Project Milestone Schedule, received from Picatinny Arsenal, Mr. C. H. Nichols during August 2, 1973 meeting BAF-GN - Column 4, U/AN-GN - Column 1 - Phase 1, Column 3 - Phase 2.

1.2.12 Plot of uniform annual costs as a function of operating rates of 100% and 25% was to be provided.

1.2.13 Plot of uniform annual costs as a function of economic life was to be provided.

1.3 APPROACH

The criteria set forth in Section 1.2 established the framework for performing the economic analysis. Within this framework, documentation of the analysis for each alternative was accomplished by utilizing Format A of AR 37-13. In all, eight alternatives were considered.

The primary analysis was based on constant (mid year 1973) dollars and considered four alternatives as follows. The BAF-GN process at operating rates of 100% and 25% of plant capacity and the U/AN-GN process at operating rates of 100% and 25% of plant capacity. The secondary analysis was based on current (inflated) dollars and considered the same four alternatives.

The baseline for developing both the non-recurring investment and recurring operations costs was the cost estimates prepared under contracts with the Corps of Engineers, Omaha District. These contracts covered the Preparation of a Process Design Criteria Memorandum (PDCM), Contract DACA 45-71-C-0121, and Architect-Engineering (A-E) Services for Design of a

Facility for the Manufacture of NQ Utilizing GN Produced via the BAF-GN Process, Contract DACA 45 73 C0015. These cost estimates were based on the Modified Concept Design (MCD) for the BAF-GN process, and were used for preparing the P-15 Estimate submitted during June 1973. Therefore, this information is the best available for establishing the baseline costs for the economic study.

For the BAF-GN process the baseline costs were deflated to establish mid year 1973 costs. For the U/AN-GN process the deflated baseline costs were adjusted for discrete identifiable changes whenever possible. Otherwise factoring techniques such as ratios of facility costs and manpower requirements were employed.

Throughout the analysis the integrity of the comparison between the two processes was maintained. This was accomplished not only by employing the same baseline cost, but also by striving to employ analytical techniques and procedures that would provide for consistency between the costs developed for each process.

By approaching the analysis in this manner, the cost differential or variance provides a valid basis for concluding which process is more economical.

1.4 SUMMARY AND CONCLUSIONS

The total annual costs, both undiscounted and discounted, are summarized in Figure 1-1, while the discounted uniform annual costs are summarized in Figure 1-2.

The variance between the two processes is expressed in terms of total dollars and the percentage of the total dollar variance to the total annual cost for the U/AN-GN process.

It should be noted that the total dollar variance is increased when the constant dollars are adjusted for inflation.

The inflation factor used is approximately 4.7% compounded per annum, as specified by Section 1.2, Criteria 1.2.2. This factor appears to be lower than the actual inflation experienced during the past several years, which would indicate that the effects of inflation represented by this analysis are conservative.

In any event, the analysis shows that the U/AN-GN process is more economical than the BAF-GN process. Over the economic life of ten (10) years the difference at the operating rate of 100% is $11,000,000 to $13,000,000 or 13.5% to 15.6%.

Considering the comparative nature of this analysis, the depth and refinement of the cost details are judged to be consistent and compatible with the depth and refinement of the other factors such as the definitions for the scope of the project activities, the milestone schedule, the Applicable Criteria in Section 1.2, and the time available to perform the analysis. Additional time to refine the cost in greater detail would probably not in itself materially change the results of the analysis.

FIGURE 1-1

SUMMARY OF TOTAL ANNUAL COSTS - UNDISCOUNTED

IN THOUSANDS OF DOLLARS

	BAF-GN Process	U/AN-GN Process	Variance	
			Dollar	% of U/AN
1. Constant Dollars				
Operating Rate				
100%	$157,093	$142,286	$14,807	10.4
25%	$119,012	$108,676	$10,336	9.5
2. Current Dollars (Inflated)				
Operating Rate				
100%	$225,294	$210,779	$14,515	6.9
25%	$165,111	$155,188	$9,923	6.4

SUMMARY OF TOTAL ANNUAL COSTS - DISCOUNTED

IN THOUSANDS OF DOLLARS

	BAF Process	U/AN Process	Variance	
			Dollar	% of U/AN
1. Constant Dollars				
Operating Rate				
100%	$80,859	$69,912	$10,947	15.6
25%	$65,679	$57,722	$7,957	13.8
2. Current Dollars (Inflated)				
Operating Rate				
100%	$108,071	$95,200	$12,871	13.5
25%	$84,927	$75,754	$9,173	12.1

FIGURE 1-2

SUMMARY OF UNIFORM ANNUAL COSTS - DISCOUNTED

IN THOUSANDS OF DOLLARS

	BAF Process	U/AN Process	Variance Dollar/Year
1. Constant Dollars			
Operating Rate			
100%	19,111	18,178	933
25%	15,523	15,008	515
2. Current Dollars (Inflated)			
Operating Rate			
100%	25,543	24,753	790
25%	20,073	19,697	376

2.0 BAF-GN PROCESS - CONSTANT DOLLAR ANALYSIS

The annual costs shown in the preceeding Section 1.4 for the BAF-GN constant dollar analysis are documented in this section. The documentation, in accordance with Section 1.2, Criteria 1.2.1, is presented by Format A.

2.1 FORMAT A

Format A for both operating rates of 100% and 25% are shown on Figure 2-1. The total non-recurring and recurring costs are shown as cash flows for the project year in which they are scheduled to occur. The annual costs are the total of the non-recurring and recurring costs. The discount factors are from AR 37-13, refer Section 1.2, Criteria 1.2.1. The total annual costs and the total discounted annual costs are those which are summarized in Figure 1-1.

The uniform annual cost is calculated in accordance with the definition specified in AR 37-13.

The source derivation of the cost estimates is provided in subsequent Sections 2.5, 2.6 and 2.7.

2.2 SUMMARY OF CASH FLOWS

The cash flows are summarized on Figure 2-2. The non-recurring costs for both operating rates are identical except for the working capital which is defined as four (4) weeks of raw materials. As such it is a function of the operating rate.

FIGURE 2-1 - FORMAT A - CONSTANT DOLLAR ANALYSIS

ECONOMIC ANALYSIS - DOD INVESTMENTS
SUMMARY OF PROJECT COSTS

1. Submitting DoD Component: Picatinny Arsenal - Dover, N. J.

2. Date of Submission: October 3, 1973

3. Project Title: Nitroguanidine Facility

4. Description of Project Objective: Comparison BAF-GN Process with U/AN-GN Process

5. Alternative: BAF-GN Oper. Rate 100% 6. Economic Life: 10 Years

8. Project Costs (000)

7. Project Year	a. Nonrecurring Investment	b. Recurring Operations	Annual Costs	d. Discount Factor	e. Discounted Annual Cost
1	-	-	-	.954	-
2	4,964	-	4,964	.867	4,304
3	19,857	-	19,857	.788	15,647
4	19,857	-	19,857	.717	14,237
5	7,298	-	7,298	.652	4,758
6	673	9,592	10,265	.592	6,007
7	-	10,465	10,465	.538	5,630
8	-	10,465	10,465	.489	5,117
9	-	10,465	10,465	.445	4,657
10	-	10,465	10,465	.405	4,238
11	-	10,465	10,465	.368	3,851
12	-	10,465	10,465	.334	3,495
13	-	10,465	10,465	.304	3,181
14	-	10,465	10,465	.276	2,888
15	-	10,465	10,465	.251	2,627
16	(206)	873	667	.228	152
9. TOTALS	$32,443	$104,650	$157,093	-	$80,859

10a. Total Project Cost (discounted) $80,859

10b. Uniform Annual Cost (without terminal value) $19,111

11. Less Terminal Value (discounted) N/A

12a. Net Total Project Cost (discounted) $80,850

12b. Uniform Annual Cost (with terminal value) $19,111

13. Source Derivation of Cost Estimates: (use as much space as required)

a. Nonrecurring Costs:
1) Research & Development: N/A
2) Investment: Refer to Section 2.5

b. Recurring Cost: Refer to Section 2.6

c. Net Terminal Value: N/A

d. Other Considerations: Applicable Criteria - Section 1.2

14. Name and Title of Principal Action Officer:
Hercules Incorporated, Wilmington, Delaware
Prepared under Contract DAAA 21-71-C-0193
For SARPA-MT-C, Mr. C. H. Nichols, Project Manager
Date: October 3, 1973

ECONOMIC ANALYSIS - DOD INVESTMENTS
SUMMARY OF PROJECT COSTS

1. Submitting DoD Component: Picatinny Arsenal - Dover, N. J.

2. Date of Submission: October 3, 1973

3. Project Title: Nitroguanidine Facility

4. Description of Project Objective: Comparison BAF-GN Process with U/AN-GN Process

5. Alternative: BAF-GN Oper. Rate 25% 6. Economic Life: 10 Years

8. Project Costs (000)

7. Project Year	Nonrecurring Investment	Recurring Operations	Annual Costs	Discount Factor	Discounted Annual Cost
1	-	-	-	.954	-
2	4,964	-	4,964	.867	4,304
3	19,867	-	19,857	.788	15,647
4	19,857	-	19,857	.717	14,237
5	7,298	-	7,298	.652	4,758
6	519	6,101	6,620	.592	3,919
7	-	6,657	6,657	.538	3,581
8	-	6,657	6,657	.489	3,255
9	-	6,657	6,657	.445	2,962
10	-	6,657	6,657	.405	2,696
11	-	6,657	6,657	.368	2,450
12	-	6,657	6,657	.334	2,223
13	-	6,657	6,557	.304	2,024
14	-	6,657	6,657	.276	1,837
15	-	6,657	6,657	.251	1,671
16	(52)	555	503	.228	115
9. TOTALS	$52,443	$66,569	$119,012	-	$65,679

10a. Total Project Cost (discounted) $65,679

10b. Uniform Annual Cost (without terminal value) $15,523

11. Less Terminal Value (discounted) N/A

12a. Net Total Project Cost (discounted) $65,679

12b. Uniform Annual Cost (with terminal value) $15,523

13. Source Derivation of Cost Estimates: (use as much space as required)

a. Nonrecurring Costs:
1) Research & Development: N/A
2) Investment: Refer to Section 2.5

b. Recurring Cost: Refer to Section 2.7

c. Net Terminal Value: N/A

d. Other Considerations: Applicable Criteria - Section 1.2

14. Name and Title of Principal Action Officer:
Hercules Incorporated, Wilmington, Delaware
Prepared under Contract DAAA 21-71-C-0193
For SARPA-MT-C, Mr. C. H. Nichols, Project Manager
Date: October 3, 1973

FIGURE 2-2

SUMMARY OF CASH FLOWS
FOR RAP-GN PROCESS
MID YEAR 1973 COSTS IN THOUSANDS OF DOLLARS

NON-RECURRING COSTS							RECURRING COSTS			
OPERATING RATE - 100% and 25%							OPERATING RATE-100%		OPERATING RATE-25%	
Project Year	Design Criteria	Design	Plant Investment	Plant Start Up & Commissioning	Working Capital	Total Investment	Operations	Annual Costs	Operations	Annual Costs
1	N/A	N/A	--	--	--	--	--	--	--	--
2	N/A	N/A	4,964	--	--	4,964	--	4,964	--	4,964
3	N/A	N/A	19,857	--	--	19,857	--	19,857	--	19,857
4	N/A	N/A	19,857	--	--	19,857	--	19,857	--	19,857
5	N/A	N/A	4,964	2,334	--	7,298	--	7,298	--	7,298
6	N/A	N/A	--	467	206*	673	9,592	10,265	6,101	6,620
7	N/A	N/A	--	--	--	--	10,465	10,465	6,657	6,657
8	N/A	N/A	--	--	--	--	10,465	10,465	6,657	6,657
9	N/A	N/A	--	--	--	--	10,465	10,465	6,657	6,657
10	N/A	N/A	--	--	--	--	10,465	10,465	6,657	6,657
11	N/A	N/A	--	--	--	--	10,465	10,465	6,657	6,657
12	N/A	N/A	--	--	--	--	10,465	10,465	6,657	6,657
13	N/A	N/A	--	--	--	--	10,465	10,465	6,657	6,657
14	N/A	N/A	--	--	--	--	10,465	10,465	6,657	6,657
15	N/A	N/A	--	--	--	--	10,465	10,465	6,657	6,657
16	N/A	N/A	--	--	(206)*	(206)	873	667	555	503
			49,642	2,801	--	52,443	104,650	157,093	66,569	119,012

*NOTE: Working Capital of 206 and (206) apply for operating rate of 100%. For operating rate of 25% the working capital is 52 and (52) which is reflected in the Annual Cost column for the 25% operating rate.

The total costs are shown as cash flows for the project year in which the expenditures will occur. The timing of the cash flows is shown on Figure 2-3.

2.3 SCHEDULE FOR CASH FLOWS

Figure 2-3 presents a bar chart schedule for the BAF-GN and U/AN-GN processes at the 100% operating rate to determine the cash flows for each project year. The cash flow totals, both horizontal and vertical, for the BAF-GN process are transcribed to Figure 2-2 and shown by project year.

2.4 NQ PROJECT MILESTONE SCHEDULE

The Schedule of Cash Flows is based on the NQ Project Milestone Schedule, Figure 2-4. This schedule was furnished by Picatinny Arsenal, refer to Section 1.2, Criteria 1.2.11.

The BAF-GN cash flows are scheduled on the basis of the fourth column of dates.

2.5 SOURCE DERIVATION OF NON-RECURRING COSTS

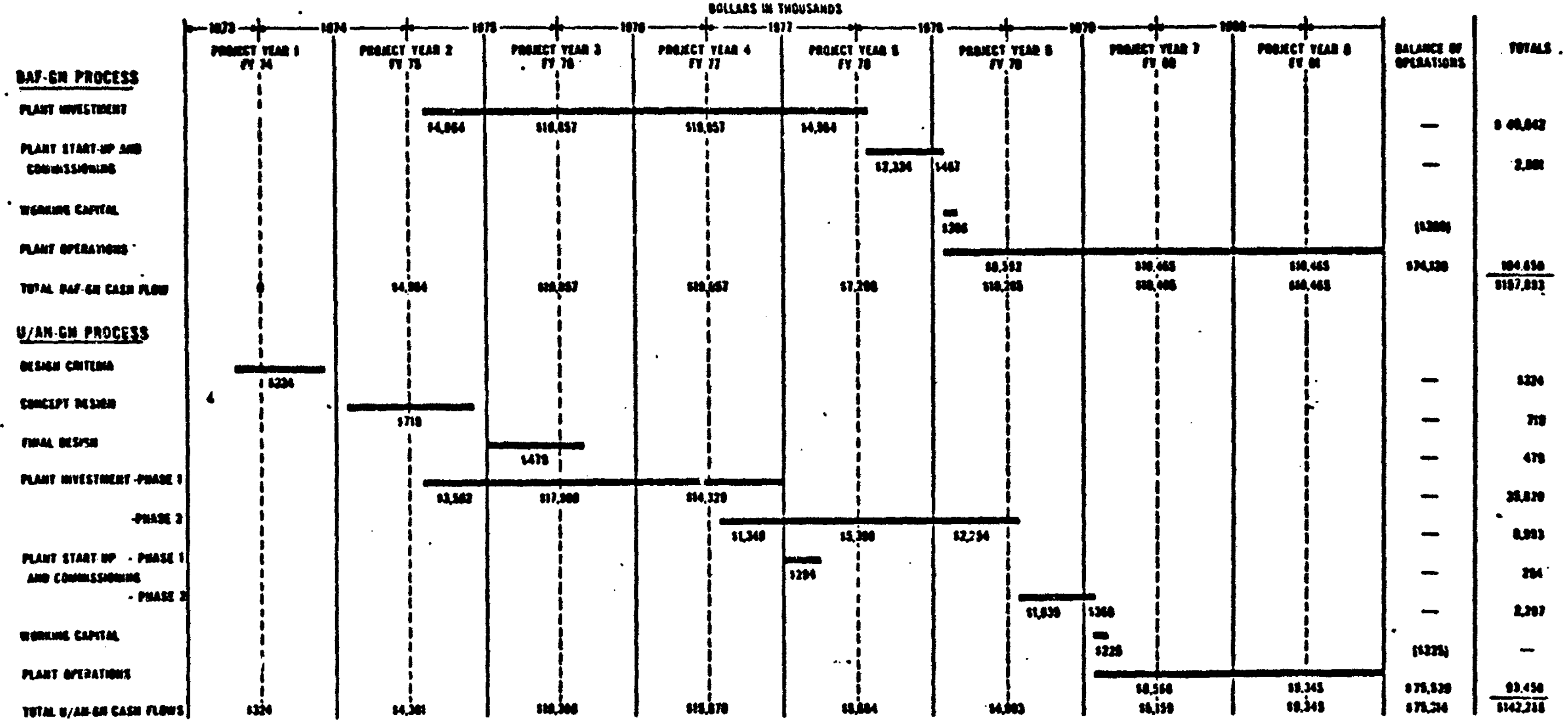
2.5.1 TOTAL NON-RECURRING COSTS

The summary of the non-recurring costs in Figure 2-5 provides the major components of the total non-recurring investment shown on Figure 2-1, Format A, Item 8, Column a.

FIGURE 2-3

SCHEDULE OF CASH FLOWS

BAF-GN PROCESS AND U/AN-GN PROCESS AT OPERATING RATE OF 100%
FROM JULY 1, 1973 THROUGH ECONOMIC LIFE OF PROJECT - 10 YEARS
DOLLARS IN THOUSANDS



	PROJECT YEAR 1 FY 74	PROJECT YEAR 2 FY 75	PROJECT YEAR 3 FY 76	PROJECT YEAR 4 FY 77	PROJECT YEAR 5 FY 78	PROJECT YEAR 6 FY 79	PROJECT YEAR 7 FY 80	PROJECT YEAR 8 FY 81	BALANCE OF OPERATIONS	TOTALS
BAF-GN PROCESS										
PLANT INVESTMENT		$4,864	$15,657	$15,657	$4,664				—	$ 40,842
PLANT START-UP AND COMMISSIONING					$2,334	$467			—	2,801
WORKING CAPITAL						$1,386			($1,386)	
PLANT OPERATIONS						$8,552	$10,465	$10,465	$74,128	104,630
TOTAL BAF-GN CASH FLOW	0	$4,864	$15,657	$15,657	$7,298	$10,285	$10,465	$10,465		$157,833
U/AN-GN PROCESS										
DESIGN CRITERIA	$224								—	$224
CONCEPT DESIGN		$719							—	719
FINAL DESIGN			$479						—	479
PLANT INVESTMENT - PHASE 1		$3,582	$17,300	$14,320					—	35,820
- PHASE 2				$1,340	$5,300	$2,294			—	8,933
PLANT START-UP AND COMMISSIONING - PHASE 1					$294				—	294
- PHASE 2						$1,839	$368		—	2,207
WORKING CAPITAL							$225		($225)	—
PLANT OPERATIONS							$8,566	$9,345	$75,539	93,450
TOTAL U/AN-GN CASH FLOWS	$224	$4,301	$18,308	$15,870	$5,594	$4,063	$9,159	$9,345	$75,314	$142,238

NOTE: THIS SCHEDULE BASED ON PROJECT MILESTONE DATES
FURNISHED ON AUGUST 2, 1973 BY PICATINNY ARSENAL, MR. C.H. NICHOLS.
BAF-GN REFLECTS DATES SHOWN IN COLUMN 4.
U/AN-GN REFLECTS DATES SHOWN IN COLUMN 1 - PHASE 1 AND COLUMN 3 - PHASE 2.

FIGURE 2-4

NQ PROJECT MILESTONE SCHEDULE
FURNISHED BY PICATINNY ARSENAL - DOVER, N. J.

	Proj 5752632 Phase 1 **	Proj 5762632 BAF Phase 2 **	Proj 5772632 U/AN Phase 2 **	Proj 5752632 Total BAF	Proj 577263 Total U/AN
1. Completion of Design Criteria	Feb 72	Feb 72	June 74	Feb 72	June 74
2. Design Criteria Released to CE	May 72	May 72	Aug 74	May 72	Aug 74
3. Concept Design Complete	Mar 73	Mar 73	June 75	Mar 73	June 75
4. Equipment Design Complete	Jan 74	Jan 74	Mar 76	Jan 74	Mar 76
5. Final Design Complete	Apr 74*	Feb 74	Mar 76	Feb 74	Mar 76
6. Construction Contract Award	Feb 75	Feb 76	Feb 77	Feb 75	Feb 77
7. Construction Complete	Jul 77	Feb 78	Feb 79	Feb 78	Feb 80
8. Complete Debugging	Oct 77	Aug 78	Aug 79	Aug 78	Aug 80

* Includes time to condense final design drawings complete for total BAF design into bid package for Phase 1 only.

** NOTE ADDED FOR CLARIFICATION:

Phase 1 covers all NQ facilities common to BAF-GN and U/AN-GN.
Phase 2 covers NQ facilities unique to respective GN process.

FIGURE 2-5

SUMMARY OF NON-RECURRING COSTS FOR THE MANUFACTURE OF NQ VIA THE BAF-GN AND U/AN-GN PROCESSES

	BAF-GN Process	U/AN-GN Process
Construction Costs		
Facilities Common to Both Processes	$26,986,540	$26,986,540
Facilities for Mfg. of Wet GN	11,030,282	6,775,003
Subtotal (1)	$38,016,822	$33,761,543
Escalation	0	0
Contingency - 10% of Subtotal (1)	3,801,682	3,376,154
Subtotal (2)	$41,818,504	$37,137,697
S & A - 5% of Subtotal (2)	2,090,925	1,856,885
Subtotal (3)	$43,909,427	$38,994,582
Construction Support - 1.5% of Subtotal (3)	658,641	584,919
Subtotal (4)	$44,568,070	$39,579,501
Booker Associates - Less Escalation	2,633,136	2,633,136
Extension Telephone Trunk Lines	70,000	70,000
Subtotal (5) - Construction Costs	$47,271,206	$42,282,637
Equipment Procurement Support Costs (Hazards Analysis and Safety Review)	769,688	486,584
Total Plant Construction Costs	$48,040,894	$42,679,221
Design Criteria	0	324,000
Concept and Final Design - Wet GN	0	714,037
Design Charges for Incorporating U/AN-GN into BAF-GN Design	0	484,405
Spare Parts	205,700	182,674
Operating Contractor Support During Construction	1,395,194	1,860,259
Total Plant Investment	$49,641,788	$46,334,596
Plant Start-Up and Commissioning	2,801,195	2,500,256
Total Non-Recurring Costs	$52,442,983	$48,834,852
Working Capital		
Operating Rate - 100%	$206,904	$224,818
- 25%	$51,726	$60,935

The costs for the BAF-GN process are based on the MCD Estimate submitted in March 1973 and the P-15 Estimate submitted in June 1973. Since these estimates were prepared as funding documents the costs were deflated to mid year 1973 for this analysis.

The Cost Data Summary from the P-15 Estimate is shown on Figure 2-6.

2.5.2 PLANT CONSTRUCTION COSTS

The total plant construction costs shown on Figure 2-7 for the BAF-GN Process consist of the Construction Costs shown by Subtotal (5) under the BAF-GN process, plus the equipment procurement support costs.

The source of the construction costs is the MCD Estimate submitted to the Corps of Engineers for the BAF-GN process under Contract DACA 45-73-C0015. The summary shown in Figure 2-7 is from the MCD Estimate with the escalation removed to provide mid year 1973 costs. The estimate is based on the design for the BAF-GN process which was approximately 60% complete at the time the estimate was prepared.

FIGURE 2-6

COST DATA SUMMARY
FROM P-15 ESTIMATE SUBMITTED TO
COMMANDING OFFICER SUNFLOWER ARMY AMMUNITION PLANT
ON JUNE 5, 1973

13. Cost Data Summary:

		AMC	CE	TOTAL
a.	Purchase of Land	-0-	-0-	-0-
b.	New Construction	-0-	18,532.8	18,532.8
c.	Facility Rehabilitation	-0-	96.0	96.0
d.	Purchase of Industrial Plant Equipment (IPE)	-0-	10,808.0	10,808.0
e.	Installation of IPE	-0-	3,539.0	3,539.0
f.	Rehabilitation of IPE	-0-	-0-	-0-
g.	Purchase of Non-Industrial Plant Equipment	-0-	6,033.3	6,033.3
h.	Installation of Non-Industrial Plant Equipment	13.0	1,212.6	1,225.6
i.	Roads, Walks, Parking Lots and Exterior Utilities	-0-	8,067.0	8,067.0
j.	Other			
	Operating Contractor Support During Construction	1,750.0*	-0-	1,750.0*
	Equipment Procurement Support-Safety Review and Hazard Analysis	-0-	762.0	762.0
	Plant Start Up & Commissioning	3,562.0	-0-	3,562.0
	Spare Parts	252.6	-0-	252.6
	Extension of S.W. Bell Trunks	70.0	-0-	70.0
	Misc. (refer to inclosure (3))	325.8	863.0	1,188.8
k.	Subtotal (Items A through J)	5,973.4	49,913.7	55,887.1
l.	Final Design	-0-	-0-	-0-
m.	SIOH	-0-	2,495.7	2,495.7
n.	Contingencies	-0-	4,991.4	4,991.4
o.	Construction Support	-0-	748.7	748.7
p.	Total Cost	5,973.4	58,149.5	64,122.9
q.	Fiscal Year Funding Required		FY 75	

*NOTE: These costs were modified to read $1,653,000 by the Sunflower plant after this Cost Data Summary was prepared and submitted.

FIGURE 2-7
(PAGE 1 OF 2)

SUMMARY OF PLANT CONSTRUCTION COSTS FOR THE MANUFACTURE OF NQ VIA THE BAF-GN AND THE U/AN-GN PROCESSES

	BAF-GN Process	U/AN-GN Process
Office	158,946	158,946
Change House	285,701	285,701
Lunch Room & Survival Shelter	221,095	221,095
Badge Alley	50,188	50,188
Gate House	16,328	16,328
Smoking Points	25,505	25,505
Boiler House	5,898,000	5,898,000
Area Maintenance Shop	220,001	220,001
Laboratory	199,507	199,507
Sample Magazine	38,078	38,078
Fire Extinguishers	12,000	12,000
Alterations to Warehouses	76,610	76,610
Cooling Tower & Control House	300,208	300,208
CC Railroad Unloading Station	740,035	---
Calcium Cyanamide Mfg.	2,505,000	---
Calciner & Accessories	1,274,631	---
Calciner (Building)	498,373	---
Wet Guanidine Nitrate (GN)	5,120,062	6,775,003*
Dry Guanidine Nitrate (GN)	1,252,193	1,252,193
Guanidine Nitrate Rest House	124,204	124,204
Wet Nitroguanidine (NQ)	4,582,514	4,582,514
Dry Nitroguanidine (NQ)	1,331,145	1,331,145
Nitroguanidine Pack House	509,129	509,129
Ammonium Sulfate Storage	827,047	827,047
Sulfuric Acid Concentrator	3,652,571	3,652,571
Ammonium Sulfate Mfg.	250,000	250,000
Nitrogen Plant	385,000	---
Waste Treatment	333,568	333,568
Fuel Oil Storage	186,762	186,762
Tank Farm Area	471,416	471,416
CO2 Storage	63,888	---
O.S. Process Lines	645,776	645,776
O.S. Utility Lines (Above Ground)	693,129	693,129
O.S. Utility Lines (Below Ground)	841,243	841,243
O.S. Fire Lines	1,181,655	1,181,655
O.S. Electric Lines	721,750	721,750
O.S. Telephone	142,417	142,417
O.S. Conveyors - H-20	227,929	---
O.S. Conveyors - H-500	107,681	107,681
O.S. Conveyors - H-504 A & B	145,370	145,370
O.S. Conveyors - H-585	76,895	76,895
O.S. Conveyors - H-218 & 219	215,364	---

FIGURE 2-7
(PAGE 2 OF 2)

	RAF-GN Process	U/AN-GN Process
Clearing & Grading	107,181	107,181
Roads & Drainage	1,006,203	1,006,203
Walks	19,464	19,464
Stone Blanket	19,965	19,965
Temporary Construction Facilities	63,720	63,720
Temporary Construction Utilities	49,912	49,912
Temporary Protection Service	114,213	114,213
Final Clean Up	26,620	26,620
Subtotal (1)	38,016,822	33,761,543
Escalation	0	0
Contingency - 10% of Subtotal (1)	3,801,682	3,376,154
Subtotal (2)	41,818,504	37,137,697
S & A - 5% of Subtotal (2)	2,090,925	1,856,885
Subtotal (3)	43,909,429	38,994,582
Construction Support - 1.5% of Subtotal (3)	658,641	584,919
Subtotal (4)	44,568,070	39,579,501
Booker Work - Less Escalation	2,633,136	2,633,136
Extend Telephone Trunk Lines	70,000	70,000
Subtotal (5) - Construction Costs	47,271,206	42,282,637
Equipment Procurement Support Costs - (Hazards Analysis and Safety Review)	769,688	486,584
Total Plant Construction Costs	$48,040,894	$42,769,221

*Note - Wet GN Includes the following for U/AN:

Prills Unloading & Melter Bldg.		416,281
Prills Storage Building		1,805,650
Reactor Building		1,742,716
Wet GN Building		2,410,952
Addtnl. O.S. Process Lines		279,467
Addtnl. O.S. Service Lines		119,937
		6,775,003

The equipment procurement support provides for performing hazards analysis and safety review of the equipment and the five (5) package plants, i.e. calcium cyanamide, calciner, nitrogen, ammonium sulphate and sulphuric acid concentrator, at the time the vendors are selected. This is to insure that the proposed designs comply with the hazards analysis and safety requirements.

The equipment procurement costs shown in the P-15 Estimate, in paragraph 13 j, were deflated to mid year 1973 costs. The S & A, contingencies and construction support costs percentages were then added as shown in the P-15 Estimate.

The inflation factor for the Hazards Analysis is 0.6% per month for 27 months, as specified by the Corps of Engineers for the MCD estimate. The inflation factors for the Safety Review are the FY 74 Inflation Guidance, Section 1.2, Criteria 1.2.1.

A summary of the equipment procurement support cost follows:

	P-15 Estimate	Inflation Factor	Total Costs
Hazards Analysis	227,500	1.162	$196,000
Safety Review			
1975	119,274	1.1079	107,658
1976	318,760	1.1600	274,793
1977	96,312	1.2145	79,302
Subtotal	$761,846		$657,853
Subtotal (1)			$657,853
Contingency - 10% of Subtotal (1)			65,785
Subtotal (2)			723,638
S & A - 5% of Subtotal (2)			36,182
Subtotal (3)			759,820
Construction Support - 1.5% of Subtotal (1)			9,868
Total Equipment Procurement Support			769,688

2.5.3 SPARE PARTS

At the time the P-15 estimate was prepared the allowance for spare parts was determined as 1.5% of the cost of the industrial plant equipment (IPE) plus the cost of the non-IPE. The costs for spare parts included in the P-15 Estimate, paragraph 13 j, is $252,660, includes 22.8%, 0.6% per month for 38 months, for inflation. Therefore, the mid year costs are $252.600 divided by 1.228 or $205,700.

2.5.4 OPERATING CONTRACTOR SUPPORT DURING CONSTRUCTION

The costs submitted by the Sunflower Plant in support of the P-15 Estimate were deflated to mid year 1973 costs by using the inflation factors from the FY 74 Inflation Guidance. Since these costs are scheduled to occur over a three year period it was necessary to divide by the inflation factor for the year in which the cash flow occurs as shown below.

Project Year	P-15 Estimate	Inflation Factor	Mid Year 1973
1974	24,725	1.0582	23,365
1975	286,044	1.1079	258,186
1976	487,838	1.1600	420,550
1977	578,435	1.2145	476,274
1978	275,707	1.2716	216,819
Total	$1,652,749		
Total Operating Contractor Support During Construction			$1,395,194

2.5.5 PLANT START UP AND COMMISSIONING

The costs submitted by the Sunflower Plant in support of the P-15 Estimate were deflated to mid year 1973 costs by using the inflation factors from the FY 74 Inflation Guidance. These costs, scheduled to occur in 1978, are $3,562,000 which when deflated by the inflation factor of 1.2716 results in $2,801,195.

2.5.6 WORKING CAPITAL

The working capital requirements were defined as four (4) weeks of raw material costs. These costs are as follows.

Operating Rate	Raw Material Cost/Year	Multiplier	Working Capital
100%	$2,689,758	$\frac{4}{52}$	$206,904
25%	$672,439	$\frac{4}{52}$	$51,726

2.6 SOURCE DERIVATION OF RECURRING COSTS AT AN OPERATING RATE OF 100%

2.6.1 SUMMARY OF OPERATIONS COSTS

The operations costs shown on Figure 2-8 for the operating rate of 100% are the annual recurring costs for each year of the project's ten (10) year economic life. The total recurring costs are shown on Figure 2-1, Format A, Item 8, Column b.

These costs are based on the design initiated during the preparation of the PDCM and developing during the Modified Concept Design for the BAF-GN Process.

2.6.2 DIRECT LABOR COSTS

The source of the direct labor requirements is Section 6.0 of the PDCM prepared for the BAF-GN process. The analysis of the operating

FIGURE 2-8

OPERATIONS COSTS FOR THE MANUFACTURE OF NQ
VIA THE BAF-GN PROCESS
OPERATING RATE 100% - 14,600 TONS/YR

1.0 Manufacturing Cost - Dollars Per Ton NQ

		Quantity	Cost Per Unit		Cost Per Ton NQ		Cost Per Year	
1.1	Processing Cost							
1.1.1	Direct Labor							
	A. Operating Labor	192 Men	$8,966/MAN-YEAR		$117.90		$1,721,340	
	B. Chemical Control	8 Men	$9,944/MAN-YEAR		5.45		79,570	
	C. Supervision	10 Men	$15,611/MAN-YEAR		10.70		156,220	
				Total Direct Labor	134.05		1,957,130	
1.1.2	Repairs Labor	2.18% of Plant Construction Cost ($48.0 Million)			71.67		1,046,382	
				Total Labor	205.72		3,003,512	
1.1.3	Repairs Material	2.0% of Plant Construction Cost ($48.0 Million)			65.75		959,950	
1.1.4	Electricity	3,000 KWH/TNQ	$0.01/KWH		36.00		525,600	
1.1.5	Steam	96,000 LBS/TNQ	$1.00/1000 LBS		96.00		1,401,600	
1.1.6	Water (Well)	8,000 GALS/TNQ	$0.42/1000 GALS		3.36		49,056	
				Processing Cost		$406.83		$5,939,718
1.2	Raw Materials Cost							
1.2.1	Calcium Carbide (80%)	0.97 T/TNQ	$128.80		$124.94		$1,824,124	
1.2.2	Fluorspar (70%)	0.02 T/TNQ	$ 95.20		1.90		27,740	
1.2.3	Nitrogen	0.89 T/TNQ	-0-		0.00		000	
1.2.4	Ammonium Nitrate (83%)	0.37 T 100%/TNQ	$ 50.00		18.50		270,100	
1.2.5	Nitric Acid (60%)	0.28 T 100%/TNQ	$ 52.00		14.56		212,576	
1.2.6	Nitric Acid (99%)	0.10 T 100%/TNQ	$131.00		13.10		191,260	
1.2.7	Oleum (40%)	0.16 T/TNQ	$ 28.00		4.48		65,408	
1.2.8	Anhydrous Ammonia (100%)	0.09 T/TNQ	$ 75.00		6.75		98,550	
				Total Raw Materials Cost		184.23		2,689,758
1.3	Fringe Benefits 37.34% of Total Labor			Fringe Benefits		76.82		1,121,572
1.4	G. and A. Overhead 26.64% of the Total of Direct Labor Plus Fringe Benefits			G. & A. Overhead		56.18		820,228
				Total Manufacturing Cost (Ex By-Product Credits)		$724.06		$10,571,276
1.5	By-Product Credit							
	1. Ammonium Sulfate	0.1-0.2 T/TNQ	$15/TON		$2.25		$32,850	
	2. Lime	0.84 T/TNQ	$6/TON		5.04		73,854	
						(7.29)		(106,434)
				Total Manufacturing Cost (Incl. By-Product Credit)		$716.77		$10,464,842

2.0 Working Capital

2.1 Four (4) Weeks Raw Materials @ Operating Rate $206,904

$$\frac{\$184.23/\text{Ton} \times 14{,}600\ \text{Tons/Yr} \times 4\ \text{Weeks}}{52\ \text{Weeks}} = \$206{,}904$$

manpower requirements for both processes and operating rates is shown on Figure 2-9.

2.6.3 REPAIRS LABOR AND REPAIRS MATERIALS COSTS

The total repairs cost is based on experience factors which indicate that for a plant of this type the annual repairs is expected to average 5% of the plant construction costs. This amount is in turn estimated to be split on the basis of 60% labor and 40% materials.

The labor costs of 2.18% of plant construction costs represents 60% of the 5% total costs which in turn is reduced by the fringe benefit rate of 37,33%, since the total fringe benefits are included in Item 1.3 of the operations costs.

2.6.4 ELECTRICITY COSTS

The electricity costs are calculated on the basis that the normal operating load equals eighty percent of the total

222

FIGURE 2-9

SUMMARY OF THE DIRECT LABOR REQUIREMENTS COMPARING THE BAF-GN AND U/AN-GN PROCESSES

	Operating Rate - 100%					Operating Rate - 25%			
	Total BAF-GN O.R.-100%	Reduction As Unique To BAF-GN	Common To Both Processes	Addition As Unique To U/AN-GN	Total U/AN-GN O.R.-100%	BAF-GN Reduction For O.R.-25%	BAF-GN Total For O.R.-25%	U/AN-GN Reductions For O.R.-25%	U/AN-GN Total For O.R.-25%
1. Operating Labor									
Car Unloading	6	(6)	-	6	6	(3)	3	(3)	3
Calcium Cyanamide	24	(24)	-	-	-	(12)	12	-	-
Wet GN	40	(40)	-	40	40	(3)	37	(3)	37
Dry GN	12	-	12	-	12	-	12	-	12
Wet NQ	40	-	40	-	40	-	40	-	40
Dry NQ	12	-	12	-	12	-	12	-	12
Pack House	16	-	16	-	16	(8)	8	(8)	8
Calciner	12	(12)	-	-	-	(4)	8	-	-
Sulphuric Acid Concentrator	8	-	8	-	8	-	8	-	8
Ammonium Sulphate	4	-	4	-	4	-	4	-	4
Lime/Ammonium Sulphate Loading	6	(3)	3	-	3	(3)	3	(2)	1
Industrial Waste and Cooling Tower	4	-	4	-	4	-	4	-	4
Yard Men	8	-	8	-	8	(4)	4	(5)	3
Total Operating Labor	192	(85)	107	46	153	(37)	155	(21)	132
2. Chemical Control	8	-	8	-	8	(4)	4	(4)	4
3. Supervision	10	(1)	9	-	9	(6)	4	(5)	4
Total Direct Labor Requirements	210	(86)	124	46	170	(47)	163	(30)	140

installed load. The total installed load based on the BAF-GN design for the NQ facilities is 8477 KW at an operating rate of 100%. Eighty percent (80%) of this load operated twenty-four hours a day to produce forty-five (45) tons of NQ per day represents an average electrical energy consumption of 3600 KWH/ton NQ. The unit cost of $0.01 per KWH is based on the Sunflower plant experience.

2.6.5 STEAM COST

The steam costs are based on the estimated steam consumption of 180,000 per hour or 96,000 pounds per ton for the production of forty-five (45) tons of NQ per day as determined from the BAF-GN design. The unit cost of $1.00 per thousand pounds of steam is an average cost based on experience.

2.6.6 WELL WATER COSTS

The well water costs are based on an estimated flow of 250 gallons per minute or 8,000 gallons per ton, for the production of forty-five (45) tons of NQ per day, as determined from the BAF-GN design. The unit cost of $0.42/per 1,000 gallons is based on the Sunflower Plant experience.

2.6.7 RAW MATERIAL COSTS

The raw material costs are based on the quantities of materials taken from the material balances for the BAF-GN process. The unit costs, including freight costs, are current costs obtained from the Hercules' Purchasing Department.

The costs are based on material as received except for the ammonium nitrate and the nitric acid where the quantity shown specifies 100% material.

2.6.8 BY-PRODUCT CREDITS

The by-product credits are based on the quantities taken from the material balances for the BAF-GN process. The unit costs are estimated selling prices based on current costs obtained from the Hercules Purchasing Department.

2.6.9 LABOR, FRINGE BENEFIT, AND GENERAL AND ADMINISTRATIVE RATES

The source derivation of the accounting rates is provided in Section 7.0, Exhibit 7.3.

2.7 SOURCE DERIVATION OF RECURRING COSTS AT OPERATING RATE OF 25%

2.7.1 SUMMARY OF OPERATIONS COSTS

The operations costs shown on Figure 2-10 for the operating rate of 25% are the annual recurring costs for each year of the project's ten (10) year economic life. The total recurring costs are shown on Figure 2-1, Format A, Item 8, Column b.

These costs are based on the costs for the operating rate of 100% shown on Figure 2-8 and discussed in Section 2.6. The following discussion is based on the acceptance of all previous data and assumptions. Therefore only the modifications and additions to the previous discussion are provided. The reduction from 14,600 tons per year, 100% operating rate, to 3,650 tons per year, 25% operating rate, must be carefully considered for those quantities and costs expressed on a per ton basis.

FIGURE 2-10

OPERATIONS COSTS FOR THE MANUFACTURE OF NQ
VIA THE BAF-CN PROCESS
OPERATING RATE 25% - 3,650 TONS/YR

	Quantity	Cost Per Unit		Cost Per Ton NQ		Cost Per Year	
1.0 Manufacturing Cost - Dollars Per Ton NQ							
1.1 Processing Cost							
1.1.1 Direct Labor							
A. Operating Labor	155 Men	$8,966/MAN-YEAR		$380.75		$1,389,737	
B. Chemical Control	4 Men	$9,944/MAN-YEAR		10.90		39,785	
C. Supervision	4 Men	$15,611/MAN-YEAR		17.11		62,451	
			Total Direct Labor	408.76		1,491,974	
1.1.2 Repairs Labor	2.18% of Plant Construction Cost ($48.0 Million)			286.68		1,046,382	
			Total Labor	695.44		2,538,356	
1.1.3 Repairs Material	2.0% of Plant Construction Cost ($48.0 Million)			263.01		959,986	
1.1.4 Electricity	9,000 KWH/TNQ	$0.01/KQH		90.00		328,500	
1.1.5 Steam	128,000 LBS/TNQ	$1.00/1000 LBS		128.00		467,200	
1.1.6 Water (Well)	8,000 GALS/TNQ	$0.42/1000 GALS		3.36		12,264	
			Processing Cost		$1,179.81		$4,306,306
1.2 Raw Materials Cost							
1.2.1 Calcium Carbide (80%)	0.97 T/TNQ	$128.80		$124.94		$456,031	
1.2.2 Fluorspar (70%)	0.02 T/TNQ	$ 95.20		1.90		6,935	
1.2.3 Nitrogen	0.89 T/TNQ	-0-		0.00		000	
1.2.4 Ammonium Nitrate (83%)	0.37 T 100%/TNQ	$ 50.00		18.50		67,525	
1.2.5 Nitric Acid (60%)	0.28 T 100%/TNQ	$ 52.00		14.56		53,144	
1.2.6 Nitric Acid (99%)	0.10 T 100%/TNQ	$131.00		13.10		47,815	
1.2.7 Oleum (40%)	0.16 T/TNQ	$ 28.00		4.48		16,352	
1.2.8 Anhydrous Ammonia (100%)	0.09 T/TNQ	$ 75.00		6.75		24,637	
			Total Raw Materials Cost		184.23		672,439
1.3 Fringe Benefits 37.34% of Total Labor			Fringe Benefits		259.68		947,832
1.4 G. and A. Overhead 31.01% of the Total of Direct Labor Plus Fringe Benefits			G. & A. Overhead		207.28		756,572
			Total Manufacturing Cost (Ex By-Product Credits)		1,831.00		6,683,150
1.5 By-Product Credit							
1. Ammonium Sulfate	0.1-0.2 T/TNQ	$15/TON		$2.25		$8,212	
2. Lime	0.84 T/TNQ	$6/TON		5.04		18,396	
					(7.29)		(26,608
			Total Manufacturing Cost (Incl. By-Product Credit)		$1,823.71		$6,656,541

2.0 Working Capital

2.1 Four (4) Weeks Raw Materials @ Operating Rate	$51,726

$$\frac{\$184.23/\text{Ton} \times 3{,}650\ \text{Tons/Yr} \times 4\ \text{Weeks}}{52\ \text{Weeks}} = \$51{,}726$$

2.7.2 DIRECT LABOR

Refer to Section 2.6.2 and Figure 2-9.

2.7.3 REPAIRS LABOR AND REPAIRS MATERIALS COSTS

Refer to Section 2.6.3.

The repairs cost is based on the assumption that at the 25% operating rate essentially all the equipment is in use. Consequently, the yearly costs will not change in changing from the 100% operating rate to the 25% operating rate, and the cost per ton NQ will be four (4) times the cost at the 100% rate.

2.7.4 ELECTRICITY COSTS

Refer to Section 2.6.4.

The electricity costs at the 25% rate is based on the estimate that half the electric load is independent of operating rate while the other half varies directly as the operating rate. Consequently, the yearly load at 25% operating rate is five-eights the load at the 100% operating rate, and the KWH/ton NQ value at the lower rate is 2.5 times the value at the higher rate, or 9,000 KWH/ton NQ.

2.7.5 STEAM COSTS

Refer to Section 2.6.5.

The steam cost at the 25% operating rate is based on the estimate that most of the steam consumption is directly proportional to the operating rate, but that some, e.g. building heating, is independent of operating rate. It is estimated that the steam consumption per ton NQ would increase approximately one-third as the operating rate dropped from 100% to 25% yielding a steam consumption of 128,000 lbs. per ton NQ.

2.7.6 WELL WATER COSTS

Refer to Section 2.6.6.

The well water costs are directly proportional to the production rate and hence does not change for each ton of NQ produced.

2.7.7 RAW MATERIAL COSTS

Refer to Section 2.6.7.

The raw material costs are assumed to be constant for each ton of NQ produced.

2.7.8 BY-PRODUCT CREDITS

Refer to Section 2.6.8.

The by-product credit is assumed to be constant for each ton of NQ produced.

2.7.9 LABOR, FRINGE BENEFIT, AND GENERAL AND ADMINISTRATIVE RATES

The source derivation of the accounting rates is provided in Section 7.0, Exhibit 7.3.

230

3.0 U/AN-GN PROCESS - CONSTANT DOLLAR ANALYSIS

The annual costs shown in the preceeding Section 1.4 for the U/AN-GN constant dollar analysis are documented in this section. The documentation, in accordance with Section 1.2, Criteria 1.2.1, is presented by Format A.

3.1 FORMAT A

Format A for both operating rates of 100% and 25% are shown on Figure 3-1. The total non-recurring and recurring costs are shown as cash flows for the project year in which they are scheduled to occur. The annual costs are the total of the non-recurring and recurring costs. The discount factors are from AR 37-13, refer Section 1.2, Criteria 1.2.1. The total annual costs and the total discounted annual costs are those which are summarized in Figure 1-1.

The uniform annual cost is calculated in accordance with the definition specified in AR 37-13.

The source derivation of the cost estimates is provided in subsequent Sections 3.5, 3.6 and 3.7.

3.2 SUMMARY OF CASH FLOWS

The cash flows are summarized on Figure 3-2. The non-recurring costs for both operating rates are identical except for the working capital which is defined as four (4) weeks of raw materials. As such it is a function of the operating rate.

ECONOMIC ANALYSIS - DOD INVESTMENTS
SUMMARY OF PROJECT COSTS

1. Submitting DoD Component: Picatinny Arsenal - Dover, N.J.

2. Date of Submission: October 3, 1973

3. Project Title: Nitroguanidine Facility

4. Description of Project Objective: Comparison BAF-GN Process with U/AN-GN Process

5. Alternative: U/AN-GN Oper. Rate 100%

6. Economic Life: 10 Years

8. Project Costs (000)

7. Project Year	a. Nonrecurring Investment	b. Recurring Operations	c. Annual Costs	d. Discount Factor	e. Discounted Annual Cost
1	324	-	324	.954	309
2	4,301	-	4,301	.867	3,729
3	18,388	-	18,388	.788	14,490
4	15,678	-	15,678	.717	11,241
5	5,684	-	5,684	.652	3,706
6	4,093	-	4,093	.592	2,423
7	593	8,566	9,345	.538	4,928
8	-	9,345	9,345	.489	4,570
9	-	9,345	9,345	.445	4,159
10	-	9,345	9,345	.405	3,785
11	-	9,345	9,345	.368	3,439
12	-	9,345	9,345	.334	3,121
13	-	9,345	9,345	.304	2,841
14	-	9,345	9,345	.276	2,579
15	-	9,345	9,345	.251	2,346
16	-	9,345	9,345	.228	2,131
17	(225)	779	554	.208	115
9. TOTALS	$48,836	$93,450	$142,286	-	$69,912

10a. Total Project Cost (discounted) $69,912

10b. Uniform Annual Cost (without terminal value) $18,178

11. Less Terminal Value (discounted) N/A

12a. Net Total Project Cost (discounted) $69,912

12b. Uniform Annual Cost (with terminal Value) $18,178

13. Source Derivation of Cost Estimates: (use as much space as required)

a. Nonrecurring Costs:
   1) Research & Development: N/A
   2) Investment: Refer to Section 3.5

b. Recurring Cost: Refer to Section 3.6

c. Net Terminal Value: N/A

d. Other Considerations: Applicable Criteria - Section 1.2

14. Name and Title of Principal Action Officer:
Hercules Incorporated, Wilmington, Delaware
Prepared under Contract DAAA 21-71-C-0193
For SARPA-MT-C, Mr. C. H. Nichols, Project Manager
Date: October 3, 1973

ECONOMIC ANALYSIS - DOD INVESTMENTS
SUMMARY OF PROJECT COSTS

1. Submitting DoD Component: Picatinny Arsenal - Dover, N. J.

2. Date of Submission: October 3, 1973

3. Project Title: Nitroguanidine Facility

4. Description of Project Objective: Comparison BAF-GN Process with U/AN-GN Process

5. Alternative: U/AN-GN Oper. Rate 25%

6. Economic Life: 10 Years

8. Project Costs (000)

7. Project Year	a. Nonrecurring Investment	b. Recurring Operations	c. Annual Costs	d. Discount Factor	e. Discounted Annual Cost
1	324	-	324	.954	309
2	4,301	-	4,301	.867	3,729
3	18,388	-	18,388	.788	14,490
4	15,678	-	15,678	.717	11,241
5	5,684	-	5,684	.652	3,706
6	4,093	-	4,093	.592	2,423
7	429	5,485	5,914	.538	3,182
8	-	5,984	5,984	.489	2,926
9	-	5,984	5,984	.445	2,663
10	-	5,984	5,984	.405	2,424
11	-	5,984	5,984	.368	2,202
12	-	5,984	5,984	.334	1,999
13	-	5,984	5,984	.304	1,819
14	-	5,984	5,984	.276	1,652
15	-	5,984	5,984	.251	1,502
16	-	5,984	5,984	.228	1,364
17	(61)	499	438	.208	91
9. TOTALS	$48,836	$59,840	$108,676	-	$57,722

10a. Total Project Cost (discounted) $57,722

10b. Uniform Annual Cost (without terminal value) $15,008

11. Less Terminal Value (discounted) N/A

12a. Net Total Project Cost (discounted) $69,912

12b. Uniform Annual Cost (with terminal value) $15,008

13. Source Derivation of Cost Estimates: (use as much space as required)

a. Nonrecurring Costs:
   1) Research & Development: N/A
   2) Investment: Refer to Section 3.5

b. Recurring Cost: Refer to Section 3.7

c. Net Terminal Value: N/A

d. Other Considerations: Applicable Criteria - Section 1.2

14. Name and Title of Principal Action Officer:
Hercules Incorporated, Wilmington, Delaware
Prepared under Contract DAAA 21-71-C-1093
For SARPA-MT-C, Mr. C. H. Nichols, Project Manager
Date: October 3, 1973

FIGURE 3-2

SUMMARY OF CASH FLOWS
FOR U/AN-CX PROCESS
MID YEAR 1973 COSTS IN THOUSANDS OF DOLLARS

NON-RECURRING COSTS							RECURRING COSTS			
OPERATING RATE - 100% AND 25%							OPERATING RATE-100%		OPERATING RATE-25%	
Project Year	Design Criteria	Design	Plant Investment	Plant Start Up Commissioning	Working Capital	Total Investment	Operations	Annual Costs	Operations	Annual Costs
1	324	--	--	--	--	324	--	324	--	324
2	--	719	3,582	--	--	4,301	--	4,301	--	4,301
3	--	479	17,909	--	--	18,388	--	18,388	--	18,388
4	--	--	15,678	--	--	15,678	--	15,678	--	15,678
5	--	--	5,390	294	--	5,684	--	5,684	--	5,684
6	--	--	2,254	1,839	--	4,093	--	4,093	--	4,093
7	--	--	--	368	225*	593	8,566	9,159	5,485	5,914
8	--	--	--	--	--	--	9,345	9,345	5,984	5,984
9	--	--	--	--	--	--	9,345	9,345	5,984	5,984
10	--	--	--	--	--	--	9,345	9,345	5,984	5,984
11	--	--	--	--	--	--	9,345	9,345	5,984	5,984
12	--	--	--	--	--	--	9,345	9,345	5,984	5,984
13	--	--	--	--	--	--	9,345	9,345	5,984	5,984
14	--	--	--	--	--	--	9,345	9,345	5,984	5,984
15	--	--	--	--	--	--	9,345	9,345	5,984	5,984
16	--	--	--	--	--	--	9,345	9,345	5,984	5,984
17	--	--	--	--	(225)*	(225)	779	554	499	438
	324	1,198	44,813	2,501	--	48,836	93,450	142,286	59,840	108,676

NOTE: Working capital of 225 and (225) apply for operating rate of 100%. For operating rate of 25% the working capital is 61 and (61) which is reflected in the Annual Cost column for the 25% operating rate.

The total costs are shown as cash flows for the project year in which the expenditures will occur. The timing of the cash flows is shown on Figure 2-3.

3.3 SCHEDULE OF CASH FLOWS

Figure 2-3 presents a bar chart schedule for the BAF-GN and U/AN-GN processes at the 100% operating rate to determine the cash flows for each project year. The cash flow totals, both horizontal and vertical, for the U/AN-GN process are transcribed to Figure 3-2 and shown by project year.

3.4 NQ PROJECT MILESTONE SCHEDULE

The Schedule of Cash Flows is based on the NQ Project Milestone Schedule Figure 2-4. This schedule was furnished by Picatinny Arsenal, refer to Section 1.2, Criteria 1.2.11.

The U/AN-GN cash flows are scheduled on the basis of the first and third column of dates.

3.5 SOURCE DERIVATION OF NON-RECURRING COSTS

3.5.1 TOTAL NON-RECURRING COSTS

The summary of the non-recurring costs in Figure 2-5 provides the major components of the total non-recurring investment shown on Figure 3-1, Format A, Item 8, Column a.

The costs for the U/AN-GN process is based on the deflated costs for the BAF-GN process, refer to Section 2.5.1. The specific items that were modified are noted on Figure 2-7 and discussed in the next Section 3.5.2.

3.5.2 PLANT CONSTRUCTION COSTS

The total plant construction costs shown in Figure 2-7 for the U/AN-GN process consist of the Construction Costs shown by Subtotal (5) under the U/AN-GN process, plus the equipment procurement support costs.

The source of the construction cost is the construction costs for the BAF-GN process, refer to Section 2.5.2. Wherever the facilities are common to both processes the same costs are used. Since both processes must produce an equal amount of dry GN for feed to the NQ plant, all process facilities downstream of the Wet GN building are exactly the same. For the purpose of this analysis only the major differences in the facilities were considered.

Utilities, (e.g. steam and water), for both GN processes are essentially the same, except for electrical power which is about 30% less for the U/AN-GN process. The primary substation would be reduced from about 9000 KW to about 6000 KW and some of power distribution lines would be smaller. It was judged that this reduction in cost would be less than 0.1% of the total project and so no change in the estimate was made for these items.

The Change House and the Lunch Room and Survival Shelter would also be somewhat smaller for a facility using the U/AN-GN process, 153 operating personnel compared to 192 for the BAF-GN process. A rough estimate of the reduction in square footage of these two buildings indicated that the reduction in cost would be about 0.2% of the total project, and, again, no change in the estimate was made for these items.

Since no design criteria or design drawings were available for the U/AN-GN process, the data from the Kenvil pilot plant operation were used to prepare a preliminary process flow sheet and material balance. The equipment was sized and estimated on the basis of general concepts from the pilot plant. The size and number of buildings were also determined on the basis of general concepts for the normal flow of materials in the process, hazards and safety considerations, and the form of raw materials required. The buildings, noted in Figure 2-6 for the U/AN-GN process were located approximately on the present approved Site Plan for the NQ facilities, in order to determine (1) that no more land was needed; (2) that their location would not affect the facilities common to both GN processes; and (3) that the requirements of AMCR 385-100 could be met.

In addition to the above, other criteria used for developing the cost of U/AN-GN facilities are:

1. Urea will be purchased as prills; ammonium nitrate will be purchased as 83% solution; and the silica catalyst will be purchased in suitable containers for warehouse storage and handling.

2. Urea will be fed to the reactor as a melt. Ammonium nitrate solution will be evaporated to 99+% and fed to the reactor as a melt.

3. Three reactors, each containing 300 tubes, will be required to produce the GN. A fourth reactor of equal size will be installed so that a reactor with spent catalyst can be discharged and re-charged with fresh catalyst without shutting down the operation.

4. The reactors will be installed in a separate building suitably designed and located for the hazards involved.

5. The rest of the GN process, after dilution of the reactor effluent, will be carried out in a separate building, from which the wet GN crystals will be pumped as a water slurry to the same Dry GN Building used for the BAF-GN process.

The equipment procurement support costs are based on the BAF-CN costs, refer to Section 2.5.2. For the U/AN-GN process three (3) of the five (5) package plants, i.e. the calcium cyanamide, calciner and nitrogen plants, are not required. Elimination of these plants reduces the estimated cost of the hazards analysis support from $196,000 to $123,908.

The safety review effort is also reduced by the reduction in the package plants. This reduction was considered proportional to the reduction in the hazards analysis effort. Therefore, the total costs for the U/AN-GN process was determined by multiplying the BAF-GN cost, of $769,688 by the ratio of $123,908 to $196,000 giving a cost of $486,584.

3.5.3 DESIGN CRITERIA COST

The source for the design criteria cost is the proposal submitted to Picatinny Arsenal on June 15, 1972. Rates for 1973 were applied to that portion of the proposal which covered the preparation of the design criteria.

3.5.4 CONCEPT AND FINAL DESIGN COSTS

The design costs are 6% of the total estimated cost of construction for the U/AN-GN facilities plus the hazards analysis and safety review costs. The total costs are summarized below.

Design	$440,812
Hazards Analysis	34,690
Safety Review	238,535
Total	$714,037

3.5.5 DESIGN CHANGES FOR INCORPORATING U/AN-GN DESIGN INTO BAF-GN DESIGN

The cost of this effort is extremely difficult to determine without having the benefit of any design effort for the U/AN-GN process. The estimate could be subject to considerable variation depending on the extent of the interaction between the designs of the two processes.

The estimate was approached by estimating the total number of drawings, approximately 495, that could be affected and an average cost for making changes to the drawings - $1,270 per drawing. Half of the drawings were assumed to require changes at the full $1,270 rate and half were assumed to require changes at half the rate or $635. The addition of project management costs of $12,600 resulted in the total cost of $484,405.

3.5.6 SPARE PARTS

Refer to Section 2.5.3.

The costs for the U/AN-GN process were determined by multiplying the spare parts costs for the BAF-GN process by the ratio of the construction costs before the add-ons were applied. Refer to Subtotal (1) of Figure 2-5.

$$\frac{\text{U/AN-GN} - \$33{,}761{,}543}{\text{BAF-GN} - \$38{,}016{,}822} \times \$205{,}700 = \$182{,}674$$

3.5.7 OPERATING CONTRACTOR SUPPORT DURING CONSTRUCTION

Refer to Section 2.5.4.

This schedule for construction of the NQ plant utilizing the U/AN-GN process is four (4) years as compared to the three (3) years for the BAF-GN process. Therefore the operating contractor support for the U/AN-GN process was determined by multiplying the costs for the BAF-GN process by the ratio of years.

$$\$1{,}395{,}194 \times \frac{4}{3} = 1{,}860{,}259$$

3.5.8 PLANT START-UP AND COMMISSIONING

Refer to Section 2.5.5.

The schedule for the U/AN-GN process shown on Figure 2-3, is based on a phase 1 - phase 2 approach. Since the mechanical check out of the plant is split under this schedule, it is assumed that the cost for both processes will be the

240

same even though the U/AN-GN process has less facilities.

For the process checkout, training of plant personnel and the live test run, the costs for the U/AN-GN process were reduced by the ratio of the personnel requirements at the various stages of the start-up and commissioning schedule. The ratio used 170:210.

3.5.9 WORKING CAPITAL

The working capital requirements are defined as four (4) weeks of raw material costs. These costs are as follows:

Operating Rate	Raw Material Cost/Year	Multiplier	Working Capital
100%	$2,922,628	4/52	$224,818
25%	$792,169	4/52	$60,935

3.6 SOURCE DERIVATION OF RECURRING COSTS AT OPERATING RATE OF 100%

3.6.1 SUMMARY OF OPERATIONS COSTS

The operations costs shown on Figure 3-3 for the operating rate of 100% are the annual recurring costs for each year of the project's ten (10) year economic life. The total recurring costs are shown on Figure 3-1, Format A, Item 8, Column b.

These costs are based on the BAF-GN design for the NQ facility to the extent the operations are common to both processes. For those operations which are not common, the costs are based on the data and experience obtained from the U/AN-GN pilot plant operation installed at Hercules' Kenvil, New Jersey plant.

3.6.2 DIRECT LABOR COSTS

Refer to Section 2.6.2 and Figure 2-9.

The direct labor requirements for the U/AN-GN process were determined by analyzing the BAF-GN requirements for each area of the plant and making the appropriate adjustments. There are estimated savings of thirty-nine (39) operating personnel and one (1) supervisor. The saving results primarily from the elimination of the calcium cyanamide plant, the calciner and the lime handling.

3.6.3 REPAIRS LABOR AND REPAIRS MATERIALS COSTS

Refer to Section 2.6.3.

3.6.4 ELECTRICITY COSTS

Refer to Section 2.6.4.

The total installed load for the U/AN-GN process is estimated to be 5477 KW, some 3000 KW less than the BAF-GN load. The primary reason is the elimination of the calcium cyanamide plant, the calciner and the lime handling. At 80% operating demand, the electrical energy consumption is 2,330 KWH/ton of NQ.

3.6.5 STEAM COSTS

Refer to Section 2.6.5.

The steam cost for the U/AN-GN process is probably less than that for the BAF-GN process, but due to uncertainty as to the optimum operating conditions for the U/AN-GN process, the material balance flow sheet for a U/AN ratio of 1.47 was selected in determining steam rates for this economic study.

Comparison of the flow sheets in Section 7.0, Exhibit 7.2 for U/AN ratios of 1.1 and 1.47 shows that the water evaporation rate from the evaporation step, and hence the steam load, is a strong function of the U/AN ratio. Therefore since it is expected that the U/AN-GN process will operate closer to a U/AN ratio of 1.1 than

FIGURE 3-3

OPERATIONS COSTS FOR THE MANUFACTURE OF NQ
VIA U/AN-GN PROCESS
OPERATING RATE 100% - 14,600 TONS/YR

.0 Manufacturing Cost - Dollars Per Ton NQ

		Quantity	Cost Per Unit		Cost Per Ton NQ		Cost Per Year	
1.1	Processing Cost							
1.1.1	Direct Labor							
	A. Operating Labor	153 Men	$8,966/MAN-YEAR		$93.96		$1,371,816	
	B. Chemical Control	8 Men	$9,944/MAN-YEAR		5.45		79,570	
	C. Supervision	9 Men	$15,611/MAN-YEAR		9.62		140,452	
				Total Direct Labor	109.03		1,591,838	
1.1.2	Repairs Labor	2.18% of Plant Construction Cost ($42.8 Million)			63.91		933,086	
				Total Labor	172.94		2,524,924	
1.1.3	Repairs Materials	2.0% of Plant Construction Cost ($42.8 Million)			58.63		855,998	
1.1.4	Electricity	2,330 KWH/TNQ	$0.01/KWH		23.30		340,180	
1.1.5	Steam	97,000 LBS/TNQ	$1.00/1000 LBS		97.00		1,416,200	
1.1.6	Water (Well)	8,000 GALS/TNQ	$0.42/1000 GALS		3.36		49,056	
				Processing Cost		$355.23		$5,186,358
1.2	Raw Materials Cost							
1.2.1	Urea (Prills)	1.39 T/TNQ	$75.00		$104.25		$1,522,050	
1.2.2	Ammonium Nitrate (83%)	0.81 T9100%)/TNQ	$50.00		42.00		613,200	
1.2.3	Catalyst	0.006 T/TNQ	$5,500.00		33.55		489,830	
1.2.4	Nitric Acid (99%)	0.10 T 100%/TNQ	$131.00		13.10		191,260	
1.2.5	Oleum (40%)	0.16 T/TNQ	$28.00		4.48		65,408	
1.2.6	Anhydrous Ammonia	0.04 T/TNQ	$70.00		2.80		40,880	
				Total Raw Materials Cost		200.18		2,922,628
1.3	Fringe Benefits 37.34% of Total Labor			Fringe Benefits		64.58		942,868
1.4	G. and A. Overhead 29.72% of the Total of Direct Labor Plus Fringe Benefits			G. & A. Overhead		51.60		753,360
				Total Manufacturing Cost (Ex By-Product Credits)		$671.59		$9,805,214
1.5	By-Product Credit							
	1. Ammonium Sulphate	0.1-0.2 T/TNQ	$15/TON		$2.25		$32,850	
	2. Ammonia	0.45 T/TNQ	$70/TON		31.50		459,900	
	3. Disposal of Ammelide	0.0067 T/TNQ	-		(2.25)		(32,850)	
						(31.50)		(459,900)
				Total Manufacturing Cost (Incl. By-Product Credit)		$640.09		$9,345,314

Working Capital

2.1 Four (4) Weeks Raw Materials @ Operating Rate $224,818

$$\frac{\$200.18/\text{Tons} \times 14{,}600\ \text{Tons/Yr} \times 4\ \text{Weeks}}{52\ \text{Weeks}} = \$224{,}818$$

to a ratio of 1.47 the approach is conservative. The steam consumption, calculated from the 1.47 ratio material balance flow sheet is 97,000 lbs/ton NQ, a value marginally higher than the 96,000 lbs./ton NQ for the BAF-GN process.

3.6.6 WELL WATER COSTS

Refer to Section 2.6.6.

The well water costs for the U/AN-NQ process are identical with those of the BAF-NQ process because well water is used only in the wet NQ building common to both processes.

3.6.7 RAW MATERIALS COSTS

The raw material costs are based on the quantities of materials taken from the material balances for the U/AN-GN process. A yield of GN from urea, by the two-mole equation, of 85% and a yield from ammonium nitrate of 97% were used. These values are considered to be conservative.

The costs are based on the materials as received except for the ammonium nitrate and the nitric acid where the quantity shown specifies 100% material.

The catalyst costs are based on a mileage of 200 pounds of GN per pound of catalyst. This value, in turn, is based on the pilot plant operation and is considered to be conservative.

Figure 3-4 shows a plot of unit catalyst price versus catalyst consumption as determined from the data furnished by Air Products and Chemicals Incorporated to Picatinny Arsenal, refer to letter of Higginson to Wachtell, Dec. 21, 1972, Section 7.0, Exhibit 7.4.

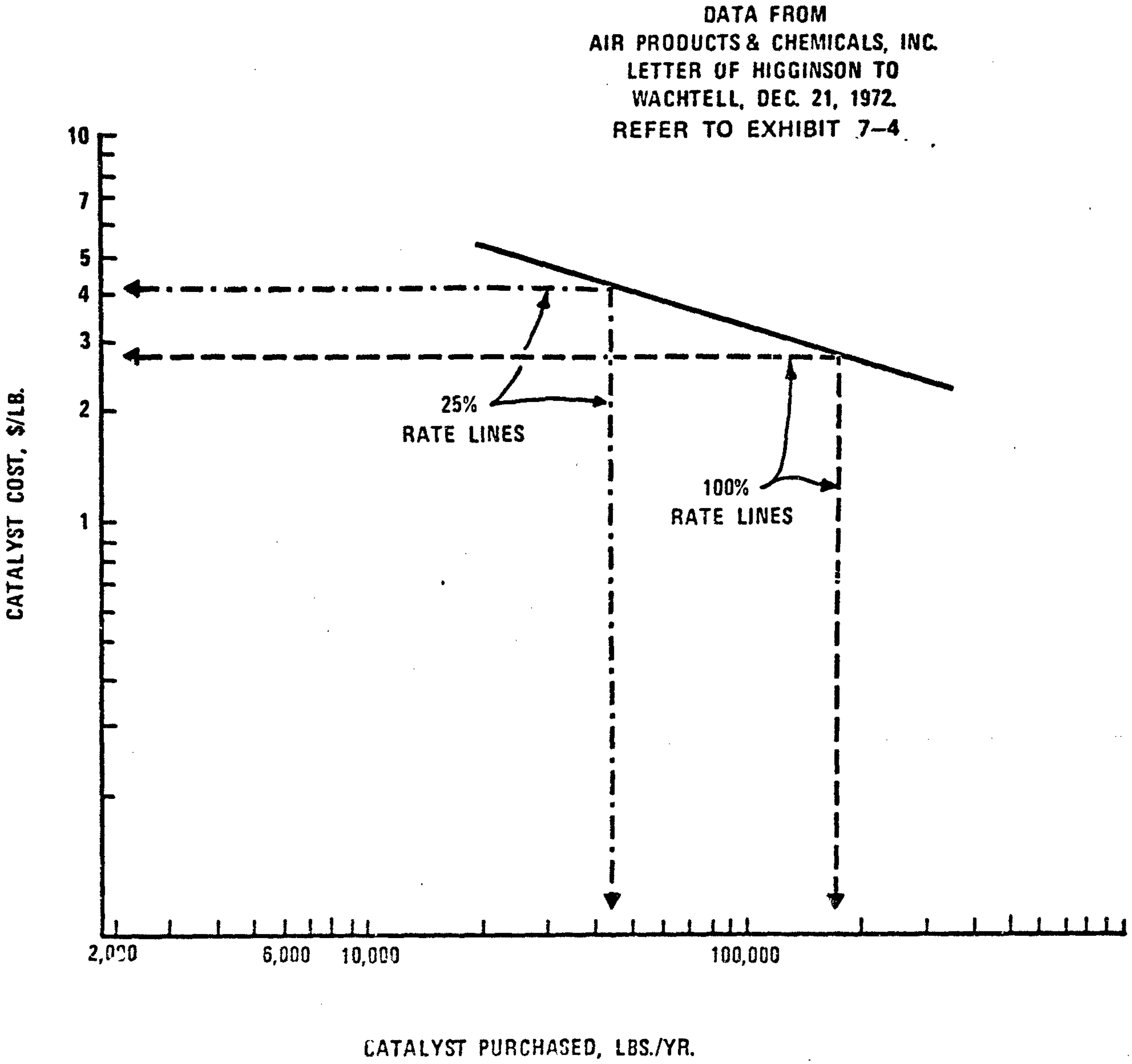
The catalyst consumption was calculated from the GN required and the mileage value of 200. A unit catalyst cost was obtained from Figure 3-4 and multiplied by the catalyst consumption to produce the total catalyst cost which was divided by the NQ production rate to yield the catalyst cost per ton of NQ.

3.6.8 BY-PRODUCT CREDITS

The by-product credits are based on the quantities from the material balances for the U/AN-GN process. The unit costs are estimated selling prices based on current costs obtained from the Hercules Purchasing Department.

FIGURE 3–4

COST OF CATALYST
AS FUNCTION OF CATALYST VOLUME



DATA FROM
AIR PRODUCTS & CHEMICALS, INC.
LETTER OF HIGGINSON TO
WACHTELL, DEC. 21, 1972.
REFER TO EXHIBIT 7–4

In the case of the Ammelide it is assumed that the costs for disposal will be approximately the same as the credit for the ammonium sulphate.

The total quantity of ammonia, 0.45 tons per ton of NQ, in the off-gas is shown as a by-product credit. An amount of 0.04 tons is charged to the process as raw materials for use in the ammonia sulphate plant. A selling price of $70.00 per ton was used to determine the by-product credit for the ammonia. This is $5.00 per ton less than the purchase price of $75.00 per ton used for the BAF-GN to allow for any cost incurred in selling the ammonia.

3.6.9 LABOR, FRINGE BENEFIT, AND GENERAL AND ADMINISTRATIVE RATES

The source derivation of the accounting rates is provided in Section 7.0, Exhibit 7.3.

3.7 SOURCE DERIVATION OF RECURRING COSTS AT OPERATING RATE OF 25%

3.7.1 SUMMARY OF OPERATIONS COSTS

The operations costs shown on Figure 3-5 for the operating rate of 25% are the annual recurring costs for each year of the project's ten (10) year economic life. The total recurring costs are shown on Figure 3-1, Format A, Item 8, Column b.

FIGURE 3-5

OPERATIONS COSTS FOR THE MANUFACTURE OF NQ
VIA U/AN-GN PROCESS
OPERATING RATE 25% - 3,650 TONS/YR

1.0 Manufacturing Cost - Dollars Per Ton NQ

	Quantity	Cost Per Unit		Cost Per Ton NQ		Cost Per Year	
1.1 Processing Cost							
1.1.1 Direct Labor							
A. Operating Labor	132 Men	$8,966/MAN-YEAR		$324.25		$1,183,512	
B. Chemical Control	4 Men	$9 [illegible]44/MAN-YEAR		10.90		39,785	
C. Supervision	4 Men	[illegible]/MAN-YEAR		17.11		62,451	
			Total Direct Labor	352.26		1,285,748	
1.1.2 Repairs Labor	2.18% of Pl[illegible] Cost ($42 [illegible]			255.63		933,049	
			Total Labor	607.89		2,218,798	
1.1.3 Repairs Material	2.0% of Plant [illegible] Cost ($42.8 Mill[illegible]			243.52		855,998	
1.1.4 Electricity	5,825 KWH/TNQ	$0.01/KWH		58.25		212,612	
1.1.5 Steam	129,300 LBS/TNQ	$1.00/1000 LBS		129.30		471,945	
1.1.6 Water (Well)	8,000 GALS/TNQ	$0.42/1000 GALS		3.36		12,264	
			Processing Cost		$1,033.32		$3,771,618
1.2 Raw Materials Cost							
1.2.1 Urea (Prills)	1.39 T/TNQ	$75.00		$104.25		380,512	
1.2.2 Ammonium Nitrate (83%)	0.81 T(100%)/TNQ	$50.00		42.00		163,300	
1.2.3 Catalyst	0.006 T/TNQ	$8,400.00		50.40		183,960	
1.2.4 Nitric Acid (99%)	0.10 T 100%/TNQ	$131.00		13.10		47,815	
1.2.5 Oleum (40%)	0.16 T/TNQ	$28.00		4.48		16,352	
1.2.6 Anhydrous Ammonia	0.04 T/TNQ	$70.00		2.80		10,220	
			Total Raw Materials Cost		217.03		792,169
1.3 Fringe Benefits 37.34% of Total Labor			Fringe Benefits		226.99		828,513
1.4 G. and A. Overhead 33.44% of the Total of Direct Labor Plus Fringe Benefits			G. & A. Overhead		193.70		707,005
			Total Manufacturing Cost (Ex By-Product Credits)		$1,671.04		$6,099,296
1.5 By-Product Credit							
1. Ammonium Sulphate	0.1-0.2 T/TNQ	$15/TON		$2.25		8,212	
2. Ammonia	0.45 T/TNQ	$70/TON		31.50		114,975	
3. Disposal of Ammelide	0.0067 T/TNQ	-		(2.25)		(8,212)	
					(31.50)		114,975
			Total Manufacturing Cost (Incl. By-Product Credit)		$1,639.54		$5,984,321

2.0 Working Capital

2.1 Four (4) Weeks Raw Materials @ Operating Rate $60,935

$$\frac{\$217.03/\text{Tons} \times 3{,}650\ \text{Tons/Yr.}}{52\ \text{Weeks}} = \$60{,}935$$

These costs are based on the costs for the operating rate of 100% shown in Figure 3-3 and discussed in Section 3.6. The following discussion is based on the acceptance of all previous data and assumptions. Therefore only the modifications and additions to the previous discussion are provided. The reduction from 14,600 tons per year, 100% operating rate, to 3,650 tons per year, 25% operating rate, must be carefully considered for those quantities and costs expressed on a per ton basis.

3.7.2 DIRECT LABOR

Refer to Section 2.6.2 and Figure 2-9.

3.7.3 REPAIRS LABOR AND REPAIRS MATERIALS COSTS

Refer to Section 2.6.3 and 2.7.3.

3.7.4 ELECTRICITY COSTS

Refer to Sections 2.6.4, 2.7.4 and 3.6.4.

The same procedure was used to determine the electricity requirements for the 25% rate for the U/AN-GN process.

3.7.5 STEAM COSTS

Refer to Sections 3.6.5 and 2.7.5.

The same procedure was used to determine the steam requirements for the 25% operating rate for the U/AN-GN process.

3.7.6 WELL WATER COSTS

Refer to Section 2.7.6 and 3.6.6.

3.7.7 RAW MATERIAL COSTS

Refer to Section 3.6.7. The raw material costs, except for the catalyst costs, are assumed to be constant for each ton of NQ produced.

The catalyst cost was determined by the same procedure discussed in Section 3.6.7 and indicated on Figure 3-4.

3.7.8 BY PRODUCT CREDITS

Refer to Sections 2.6.8 and 3.6.8.

3.7.9 LABOR, FRINGE BENEFIT, AND GENERAL AND ADMINISTRATIVE RATES

The source derivation of the accounting rates is provided in Section 7.0, Exhibit 7.3.

4.0 SENSITIVITY ANALYSIS

The sensitivity analysis criteria are defined in Section 1.2, Criteria 1.2.12 and 1.2.13. This analysis was performed for both the BAF-GN and the U/AN-GN process in terms of the constant dollar costs.

4.1 UNIFORM ANNUAL COSTS AS A FUNCTION OF OPERATING RATES

The plot for both the BAF-GN and the U/AN-GN process is shown on Figure 4-1. The uniform annual costs for the 25% and 100% operating rates were obtained from Format A, Item 10 b or 12 b for the respective processes.

Since only two (2) points were available for each plot, the function was assumed to be a straight line between the operating rates of 25% and 100%.

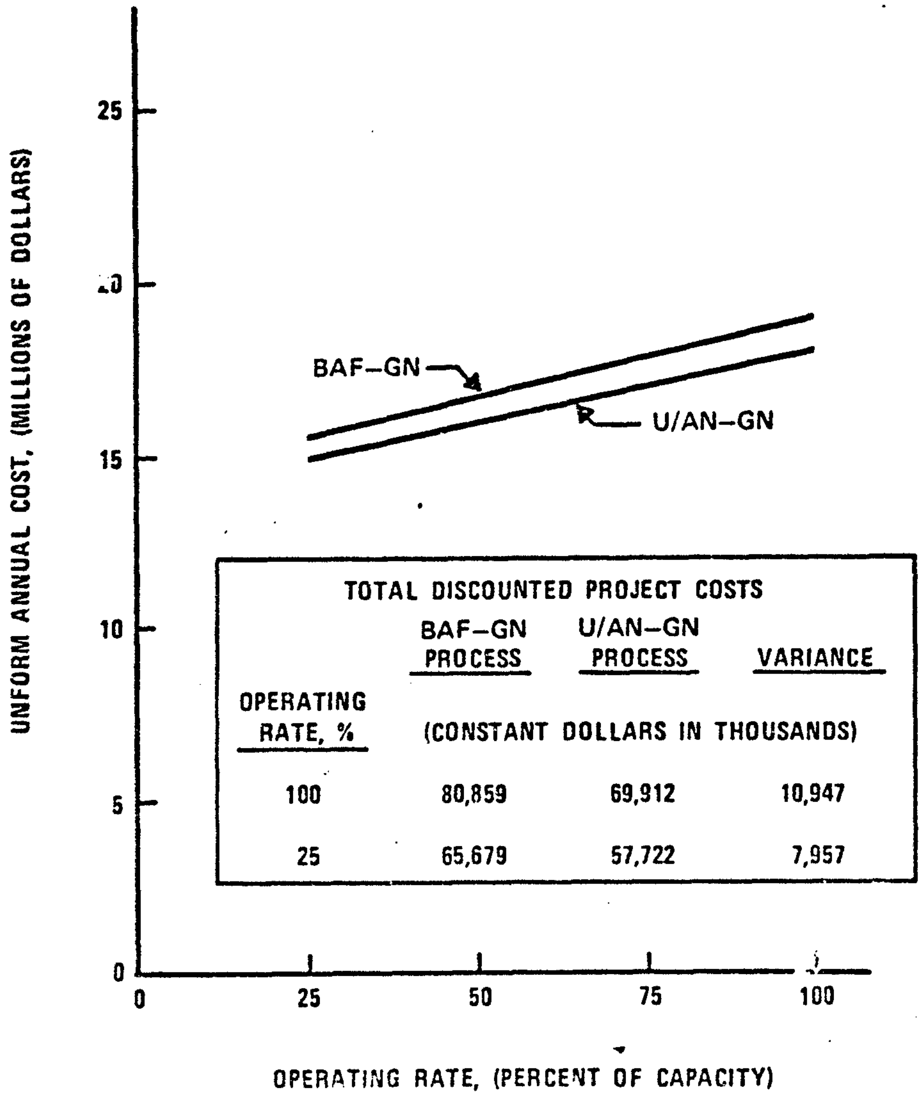
4.2 UNIFORM ANNUAL COSTS AS A FUNCTION OF THE ECONOMIC LIFE OF TEN (10) YEARS

The plot for the BAF-GN process is shown on Figure 4-2; while the plot for the U/AN-GN process is shown on Figure 4-3. The data for the two (2) processes is shown on Figure 4-4. This data was calculated from the Format A data for the respective processes. Since these data are essentially the same, separate plots were made to avoid the plots being superimposed on one another.

FIGURE 4–1

PROCESS COMPARISON – BAF–GN VS. U/AN–GN

UNIFORM ANNUAL COST TO PRODUCE NQ AS A FUNCTION OF OPERATING RATE FOR A 14,600 TPY CAPACITY NQ PLANT



TOTAL DISCOUNTED PROJECT COSTS

OPERATING RATE, %	BAF–GN PROCESS	U/AN–GN PROCESS	VARIANCE
	(CONSTANT DOLLARS IN THOUSANDS)		
100	80,859	69,912	10,947
25	65,679	57,722	7,957

FIGURE 4–2

UNIFORM ANNUAL COST AS A FUNCTION OF ECONOMIC LIFE (CONSTANT DOLLARS)

BAF–GN PROCESS

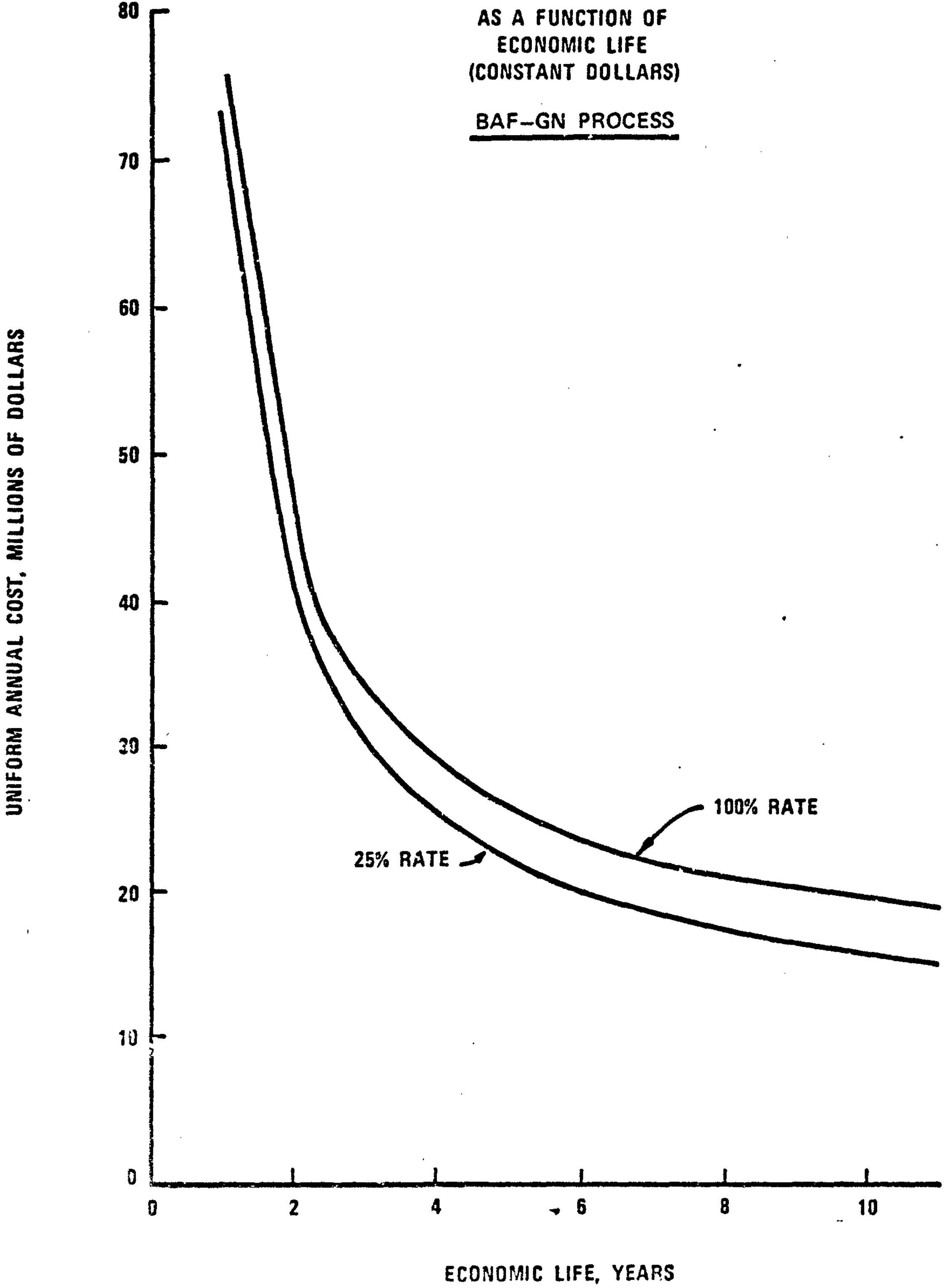


FIGURE 4–3

UNIFORM ANNUAL COST
AS A FUNCTION OF
ECONOMIC LIFE
(CONSTANT DOLLARS)

U/AN–GN PROCESS

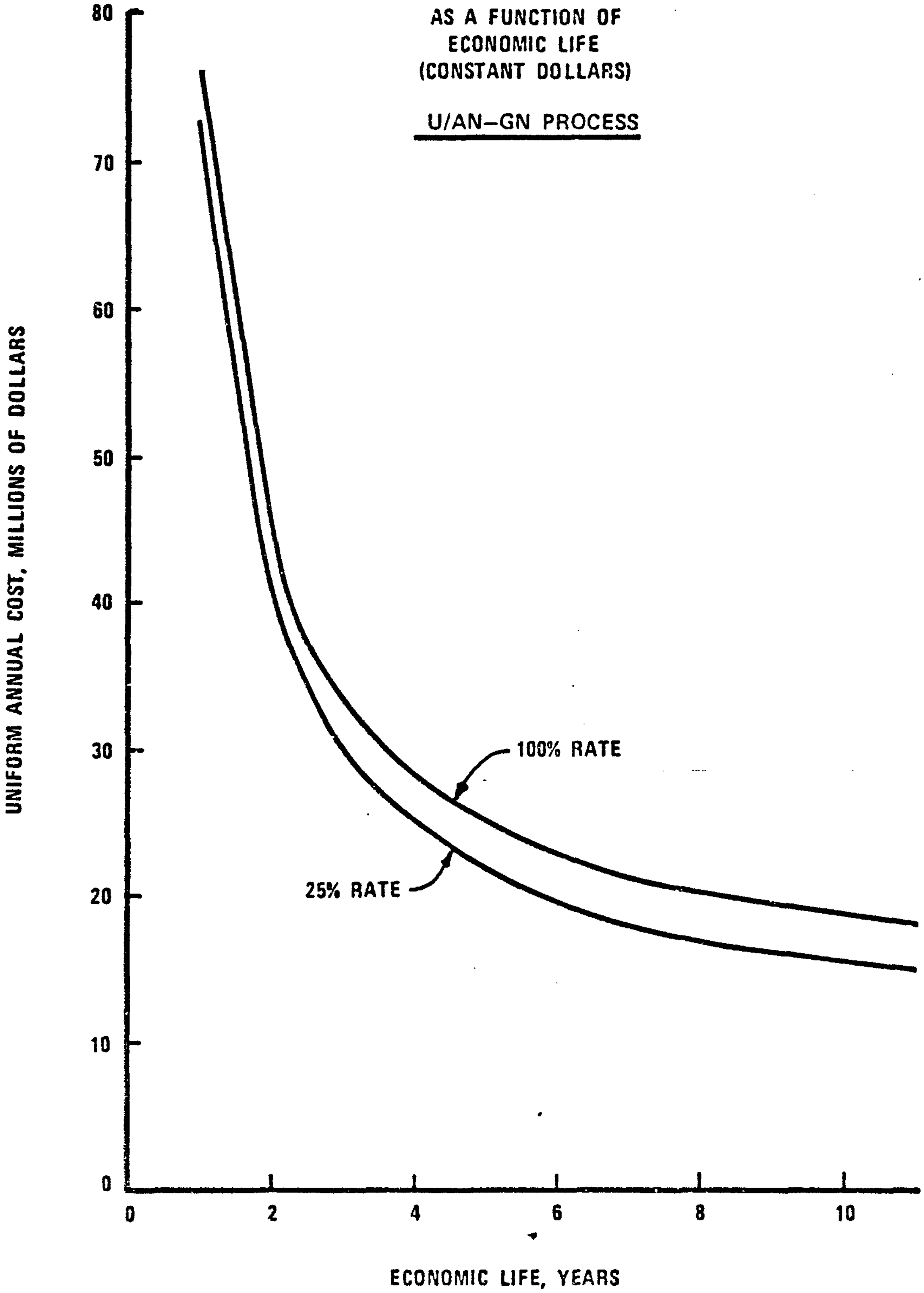


FIGURE 4-4

DATA FOR UNIFORM ANNUAL COST VS ECONOMIC LIFE

IN THOUSANDS OF DOLLARS

Year of Economic Life (1)	BAF-GN (2) 100% Capacity	U/AN-GN (2) 100% Capacity	BAF-GN (3) 25% Capacity	U/AN-GN (3) 25% Capacity
1	$75,845	$75,658	$72,407	$72,639
2	44,727	44,095	41,103	40,902
3	34,385	33,596	30,699	30,346
4	29,232	28,369	25,515	25,090
5	26,157	25,254	22,422	21,958
6	24,117	23,197	20,377	19,889
7	22,688	21,738	10,932	18,423
8	21,620	20,658	17,858	17,336
9	20,801	19,827	17,034	16,501
10	20,153	19,166	16,383	15,837
11	19,111	18,178	15,523	15,008

(1) The total economic life is ten (10) years, since the first and last years shown represent a split year the sum equals one (1) total year of the economic life.

(2) Adjusted each year for return of Working Capital.

(3) Not adjusted for return of Working Capital except in the last project year.

5.0 CURRENT (INFLATED) DOLLAR ANALYSIS

As a supplement to the primary analysis in terms of constant dollars, a secondary analysis in terms of current (inflated) dollars was made.

The results of this analysis are documented on Format A, Figure 5-1 for the BAF-GN process and Format A, Figure 5-2 for the U/AN-GN process.

The same non-recurring and recurring costs used for the constant dollar analysis were inflated by using the factors from the FY 74 Inflation Guidance, Section 1.0, Criteria 1.2.2. The inflated costs were then inserted in Format A, Item 8, Columns a, b, and c on Figures 5-1 and 5-2. The costs were then discounted by the same procedure used for the constant dollar analysis.

The discounted annual costs are summarized on Figure 1-1 for comparison of the two (2) processes.

SUMMARY OF PROJECT COSTS

1. Submitting DoD Component: Picatinny Arsenal - Dover, N.J.

2. Date of Submission: October 3, 1973

3. Project Title: Nitroguanidine Facility

4. Description of Project Objective: Comparison BAF-GN Process with U/AN-GN Process

5. Alternative: BAF-GN Oper. Rate 100% 6. Economic Life: 10 Years

8. Project Costs (000)

7. Project Year	a. Nonrecurring Investment	b. Recurring Operations	c. Annual Costs	d. Discount Factor	e. Discounted Annual Cost
1	-	-	-	.954	-
2	5,253	-	5,253	.867	4,554
3	22,000	-	22,000	.788	17,336
4	23,034	-	23,034	.717	16,515
5	8,863	-	8,863	.652	5,779
6	856	12,197	13,053	.592	7,727
7	-	13,933	13,933	.538	7,496
8	-	14,588	14,588	.469	7,134
9	-	15,274	15,274	.445	6,797
10	-	15,992	15,992	.405	6,477
11	-	16,743	16,743	.368	6,161
12	-	17,530	17,530	.334	5,855
13	-	18,354	18,354	.304	5,580
14	-	19,216	19,216	.276	5,304
15	-	20,119	20,119	.251	5,050
16	(415)	1,757	1,342	.228	306
9. TOTALS	$59,591	$165,703	$225,294	-	$108,071

10a. Total Project Cost (discounted) $108,071

10b. Uniform Annual Cost (without terminal value) $25,543

11. Less Terminal Value (discounted) N/A

12a. Net Total Project Cost (discounted) $108,071

12b. Uniform Annual Cost (with terminal value) $25,543

13. Source Derivation of Cost Estimates: (use as much space as required)

a. Nonrecurring Costs:
1) Research & Development: N/A
2) Investment: Figure 2-1 Costs Inflated

b. Recurring Cost: Figure 2-1 Costs Inflated

c. Net Terminal Value: N/A

d. Other Considerations: Applicable Criteria - Section 1.2

14. Name and Title of Principal Action Officer:
Hercules Incorporated, Wilmington, Delaware
Prepared under Contract DAAA 21-71-C-0193
For SARPA-MT-C, Mr. C. H. Nichols, Project Manager
Date: October 3, 1973

SUMMARY OF PROJECT COSTS

1. Submitting DoD Component: Picatinny Arsenal - Dover, N. J.

2. Date of Submittion: October 3, 1973

3. Project Title: Nitroguanidine Facility

4. Description of Project Objective: Comparison BAF-GN Process with U/AN-GN Process

5. Alternative: BAF-GN Oper. Rate 25% 6. Economic Life: 10 Years

8. Project Costs (000)

7. Project Year	a. Nonrecurring Investment	b. Recurring Operations	c. Annual Costs	d. Discount Factor	e. Discounted Annual Cost
1	-	-	-	.954	-
2	5,253	-	5,253	.867	4,554
3	22,000	-	22,000	.788	17,336
4	23,034	-	23,034	.717	16,515
5	8,863	-	8,863	.652	5,779
6	660	7,758	8,418	.592	4,983
7	-	8,863	8,863	.538	4,768
8	-	9,280	9,280	.489	4,538
9	-	9,716	9,716	.445	4,324
10	-	10,173	10,173	.405	4,120
11	-	10,651	10,651	.368	3,920
12	-	11,151	11,151	.334	3,724
13	-	11,675	11,675	.304	3,549
14	-	12,224	12,224	.276	3,374
15	-	12,798	12,798	.251	3,212
16	(105)	1,117	1,012	.228	231
9. TOTALS	$59,705	$105,406	$165,111	-	$84,927

10a. Total Project Cost (discounted) $84,927

10b. Uniform Annual Cost (without terminal value) $20,073

11. Less Terminal Value (discounted) N/A

12a. Net Total Project Cost (discounted) $84,927

12b. Uniform Annual Cost (with terminal value) $20,073

13. Source Derivation of Cost Estimates: (use as much space as required)

a. Nonrecurring Costs:
1) Research & Development: N/A
2) Investment: Figure 2-1 Costs Inflated

b. Recurring Cost: Figure 2-1 Costs Inflated

c. Net Terminal Value: N/A

d. Other Considerations: Applicable Criteria - Section 1.2

14. Name and Title of Principal Action Officer:
Hercules Incorporated, Wilmington, Delaware
Prepared under Contract DAAA 21-71-C-0193
For SARPA-MT-C, Mr. C. H. Nichols, Project Manager
Date: October 3, 1973

FIGURE 5-2 - FORMAT A - CURRENT (INFLATED) DOLLAR ANALYSIS

ECONOMIC ANALYSIS - DOD INVESTMENTS
SUMMARY OF PROJECT COSTS

1. Submitting DoD Component: Picatinny Arsenal - Dover, N.J.

2. Date of Submission: October 3, 1973

3. Project Title: Nitroguanidine Facility

4. Description of Project Objective: Comparison BAF-GN Process with U/AN-GN Process

5. Alternative: U/AN-GN Oper. Rate 100%   6. Economic Life: 10 Years

8. Project Costs (000)

7. Project Year	a. Nonrecurring Investment	b. Recurring Operations	Annual Costs	d. Discount Factor	e. Discounted Annual Cost
1	324	-	324	.954	309
2	4,551	-	4,551	.867	3,946
3	20,372	-	20,372	.788	16,053
4	18,186	-	19,346	.717	13,039
5	6,903	-	6,903	.652	4,501
6	5,205	-	5,205	.592	3,081
7	790	11,404	12,194	.538	6,560
8	-	13,027	13,027	.489	6,370
9	-	13,639	13,639	.445	6,069
10	-	14,280	14,280	.405	5,783
11	-	14,951	14,951	.368	5,502
12	-	15,654	15,654	.334	5,228
13	-	16,389	16,389	.304	4,982
14	-	17,159	17,159	.276	4,736
15	-	17,966	17,966	.251	4,509
16	-	18,811	18,811	.228	4,289
17	(474)	1,642	1,168	.208	243
9. TOTALS	$55,857	$154,922	$210,779	-	$95,200

10a. Total Project Cost (discounted) $95,200

10b. Uniform Annual Cost (without terminal value) $24,753

11. Less Terminal Value (discounted) N/A

12a. Net Total Project Cost (discounted) $95,200

12b. Uniform Annual Cost (with terminal value) $24,753

13. Source Derivation of Cost Estimates: (use as much space as required)

a. Nonrecurring Costs:
1) Research & Development: N/A
2) Investment: Figure 3-1 Costs Inflated

b. Recurring Cost: Figure 3-1 Costs Inflated

c. Net Terminal Value: N/A

d. Other Considerations: Applicable Criteria - Section 1.2

14. Name and Title of Principal Action Officer:
Hercules Incorporated, Wilmington, Delaware
Prepared under Contract DAAA 21-71-C-0193
For SARPA-MT-C, Mr. C. H. Nichols, Project Manager
Date: October 3, 1973

ECONOMIC ANALYSIS - DOD INVESTMENTS
SUMMARY OF PROJECT COSTS

1. Submitting DoD Component: Picatinny Arsenal - Dover, N.J.

2. Date of Submission: October 3, 1973

3. Project Title: Nitroguanidine Facility

4. Description of Project Objective: Comparison BAF-GN Process with U/AN-GN Process

5. Alternative: U/AN-GN Oper. Rate 25%   6. Economic Life: 10 Years

8. Project Costs (000)

7. Project Year	a. Nonrecurring Investment	b. Recurring Operations	Annual Costs	d. Discount Factor	e. Discounted Annual Cost
1	324	-	324	.954	309
2	4,551	-	4,551	.867	3,946
3	20,372	-	20,372	.788	16,053
4	18,186	-	18,186	.717	13,039
5	6,903	-	6,903	.652	4,501
6	5,205	-	5,205	.592	3,081
7	571	7,303	7,874	.538	4,236
8	-	8,342	8,342	.489	4,079
9	-	8,734	8,734	.445	3,887
10	-	9,144	9,144	.405	3,703
11	-	9,574	9,574	.368	3,523
12	-	10,024	10,024	.334	3,348
13	-	10,495	10,495	.304	3,190
14	-	10,988	10,988	.276	3,033
15	-	11,504	11,504	.251	2,888
16	-	12,045	12,045	.228	2,746
17	(129)	1,052	923	.208	192
9. TOTALS	$55,983	$99,205	$155,188	-	$75,754

10a. Total Project Cost (discounted) $75,754

10b. Uniform Annual Cost (without terminal value) $19,697

11. Less Terminal Value (discounted) N/A

12a. Net Total Project Cost (discounted) $75,754

12b. Uniform Annual Cost (with terminal value) $19,697

13. Source Derivation of Cost Estimates: (use as much space as required)

a. Nonrecurring Costs:
1) Research & Development: N/A
2) Investment: Figure 3-1 Costs Inflated

b. Recurring Cost: Figure 3-1 Costs Inflated

c. Net Terminal Value: N/A

d. Other Considerations: Applicable Criteria - Section 1.2

14. Name and Title of Principal Action Officer:
Hercules Incorporated, Wilmington, Delaware
Prepared under Contract DAAA 21-71-C-0193
For SARPA-MT-C, Mr. C. H. Nichols, Project Manager
Date: October 3, 1973

6.0 GN OFF-GAS UTILIZATION STUDY

6.1 OBJECTIVE

To perform an analysis of the alternatives for utilization of the ammonia and carbon dioxide off-gases from the U/AN process for manufacturing guanidine nitrate (GN).

The choice was to be based on (1) practicability; (2) ease and reliability of operation; (3) investment cost; (4) operating cost; and (5) ease in use of, or disposal of, the recovered material.

6.2 REASON FOR STUDY

The U/AN-GN process reacts urea and ammonium nitrate in the presence of a catalyst. For each molecular weight (mol) of Urea which reacts with ammonium nitrate under the reaction conditions, one mol of urea decomposes into two mols o[illegible]onia and one mol of carbon dioxide. These two mater[illegible]ave the reactor as a gas mixture and are gases at ambient temperatures and pressures. At the required production rate of 17,500 tons of GN per year, 6,600 tons of ammonia and 8,500 tons of carbon dioxide will be produced. Unless recovered and used or sold, they represent a loss of valuable material and the ammonia becomes an atmospheric pollertant.

6.3 CONCLUSION

The process finally selected for utilization of the off-gas mixture was the following:

Separate the ammonia and carbon dioxide in the off-gas. Vent the carbon dioxide to the atmosphere.

Liquefy the ammonia and provide seven days storage for internal use or for sale.

This case (identified as Case I below) more nearly met the criteria stated in the objective, than any other case studied. The selection of this case was approved by the Army on August 6, 1973, after the study had been presented to the Army on August 2, 1973.

6.4 DISCUSSION

Nearly all of the known means for using ammonia and carbon dioxide have been investigated by Hercules in the past, since we manufacture ammonia and obtain carbon dioxide as a by-product from several processes. In addition, Hercules has considerable experience in the manufacture of nitric acid, urea, ammonium nitrate, nitrogen fertilizers, and other nitrogen compounds.

From this knowledge, five cases, which appeared to meet all or some of the criteria in the objective, were formulated. The Army, during the meeting on August 2, 1973, suggested a sixth case and this was included in the study.

The cases studied are tabulated below:

Case I - Purchase ammonium nitrate and urea. Separate ammonia and carbon dioxide in the off-gas. Vent the carbon dioxide to the atmosphere. Liquefy the ammonia and store for use or sale. Provide 7 days ammonia storage.

Case II - Same as Case I, except provide 90 days ammonia storage.

Case III - Purchase urea and 56% nitric acid. Neutralize all ammonia in off-gas with nitric acid. Provide storage for use or sale of excess ammonium nitrate made from ammonia off-gas (over that required for the U/AN process).

Case IV - Purchase urea. Separate ammonia from carbon dioxide in off-gas. Provide nitric acid plant to convert part of ammonia to nitric acid. Use another part to neutralize nitric acid to provide ammonium nitrate for U/AN process. Provide storage for balance of ammonia not used above.

Case V - Purchase ammonium nitrate, ammonia and carbon dioxide. Provide total recycle urea plant. Recycle all off-gas to urea plant.

Case VI - Purchase urea and nitric acid. Provide neutralizer to make ammonium nitrate required for GN process. React sufficient off-gas and nitric to make the ammonium nitrate. Separate ammonia and carbon dioxide in excess off-gas. Liquefy ammonia and store. Vent carbon dioxide to atmosphere.

These cases are shown schematically in Figure 6-1. The comparison of the six cases is shown in Figure 6-2.

The comparison includes materials to be purchased; materials to be sold, used or disposed of; investment costs; operating costs; and advantages and disadvantages for each case.

The Capital Investment is further detailed in Figure 6-3. All dollar figures are mid-1973 prices and were estimated for this comparison from prices currently being quoted for similar equipment.

The Operating Costs are further detailed in Figure 6-4. Prices for raw materials were obtained from Hercules Purchasing Department, and represent mid-1973 delivered costs in Kansas. Labor, supervision and overhead costs on direct labor were based on costs supplied by Sunflower Army Ammunition Plant. Unit costs for utilities are an average of mid-1973 costs.

FIGURE 6-1
OFF GAS STUDY SCHEMATIC

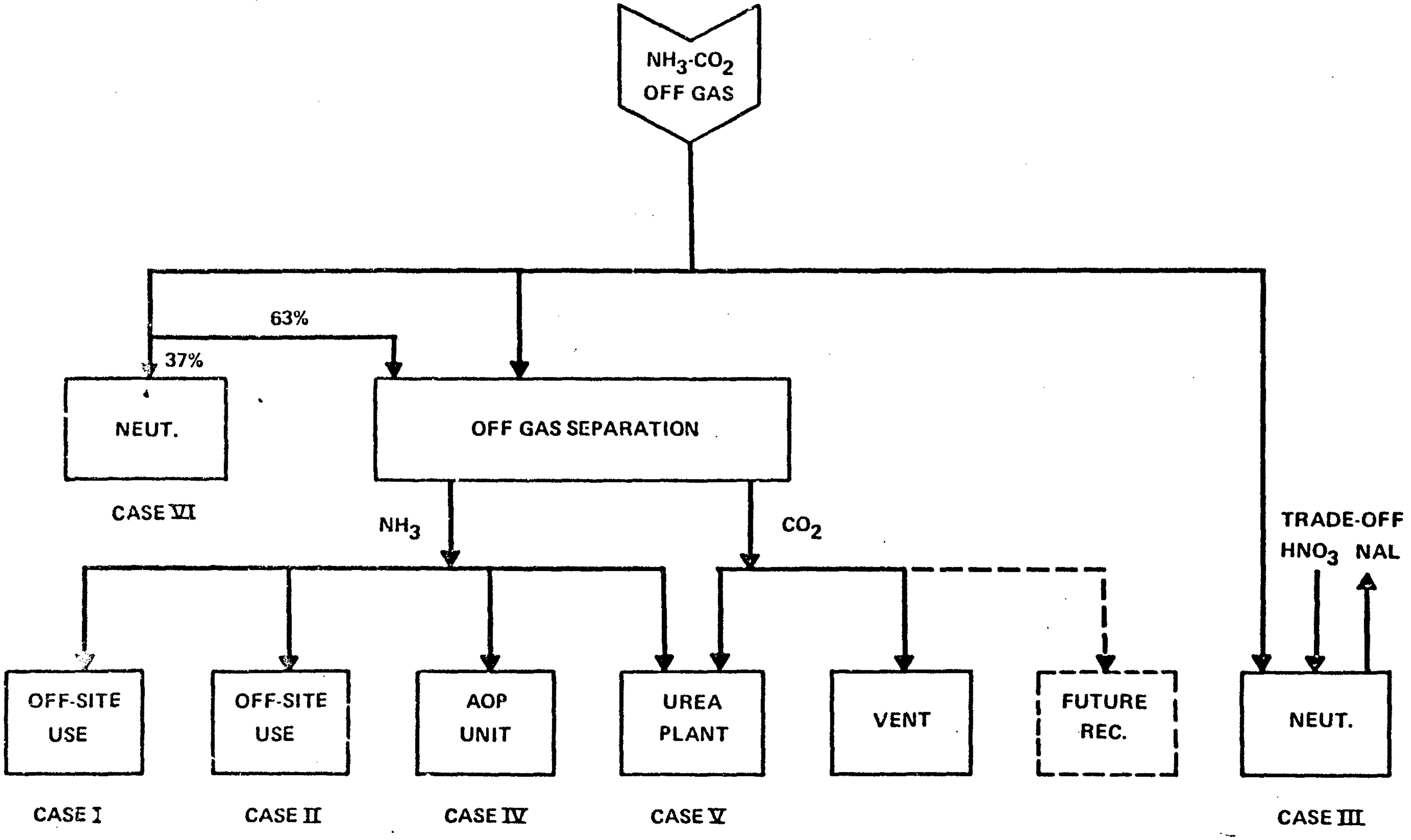
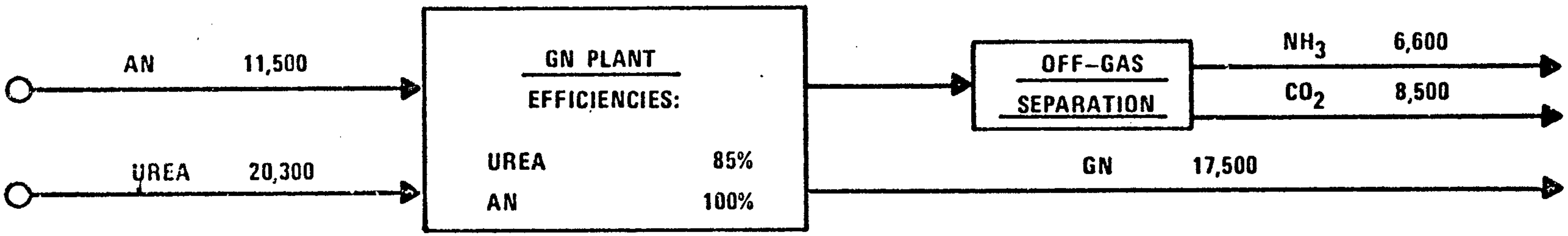


FIGURE 6-1-1
GN OFF-GAS STUDY

CASE I*: BUY UREA & AN/SELL NH_3

NEED OFF-GAS SEPARATION FACILITY



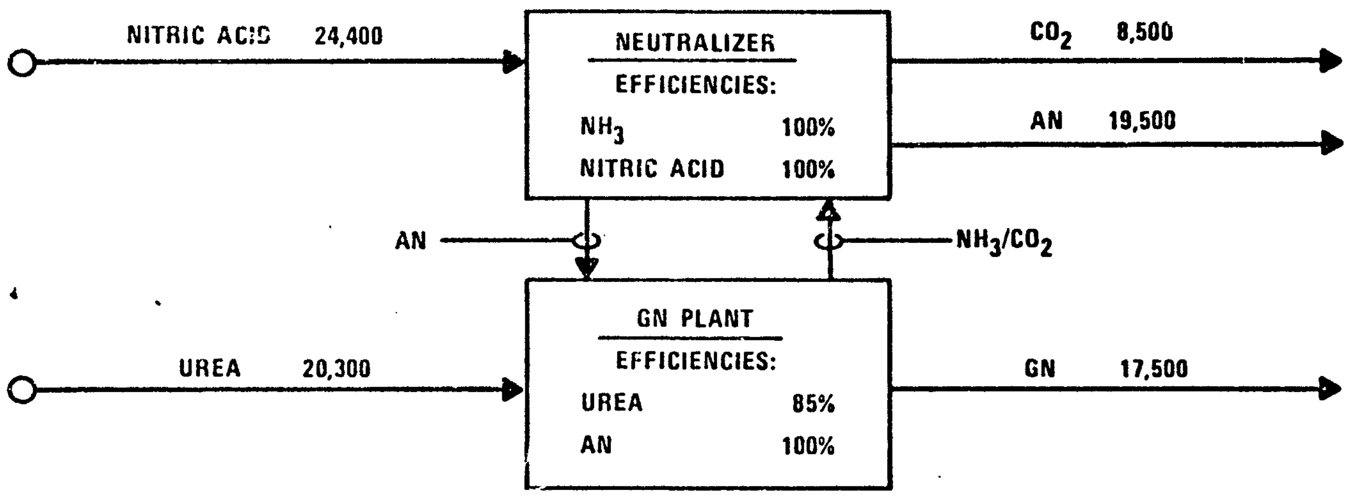
* CASE II IS BASICALLY THE SAME AS CASE I, BUT WITH ADDITIONAL AMMONIA STORAGE.

NOTES:

QUANTITIES IN TONS/YR.

FIGURE 6-1-2
GN OFF-GAS STUDY

CASE III: BUY UREA & NITRIC ACID/SELL AN
NEED NEUTRALIZER FACILITY

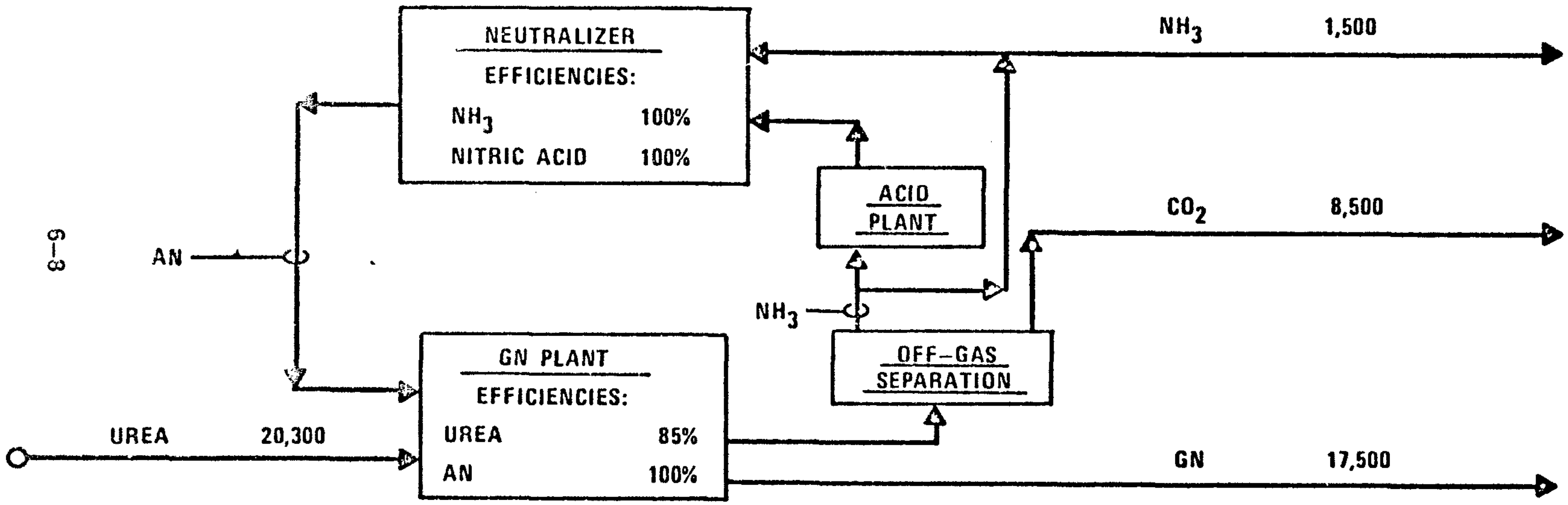


NOTES:
QUANTITIES IN TONS/YR.

FIGURE 6-1-3

GN OFF-GAS STUDY

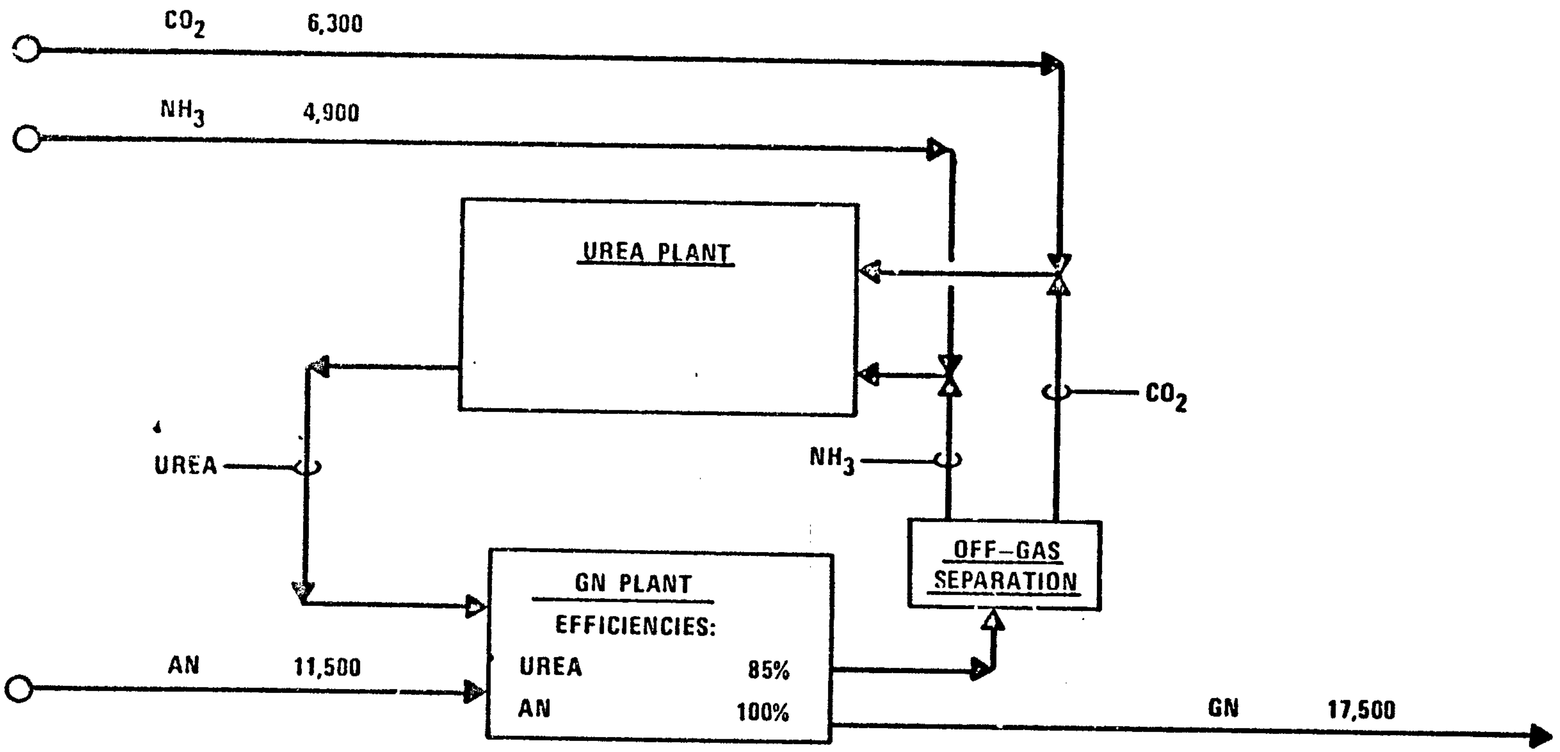
CASE IV: BUY UREA/SELL NH_3
NEED GAS SEPARATION, ACID PLANT, & NEUTRALIZER FACILITIES



NOTES:
QUANTITIES IN TONS/YR.

FIGURE 6-1-4
GN OFF-GAS STUDY

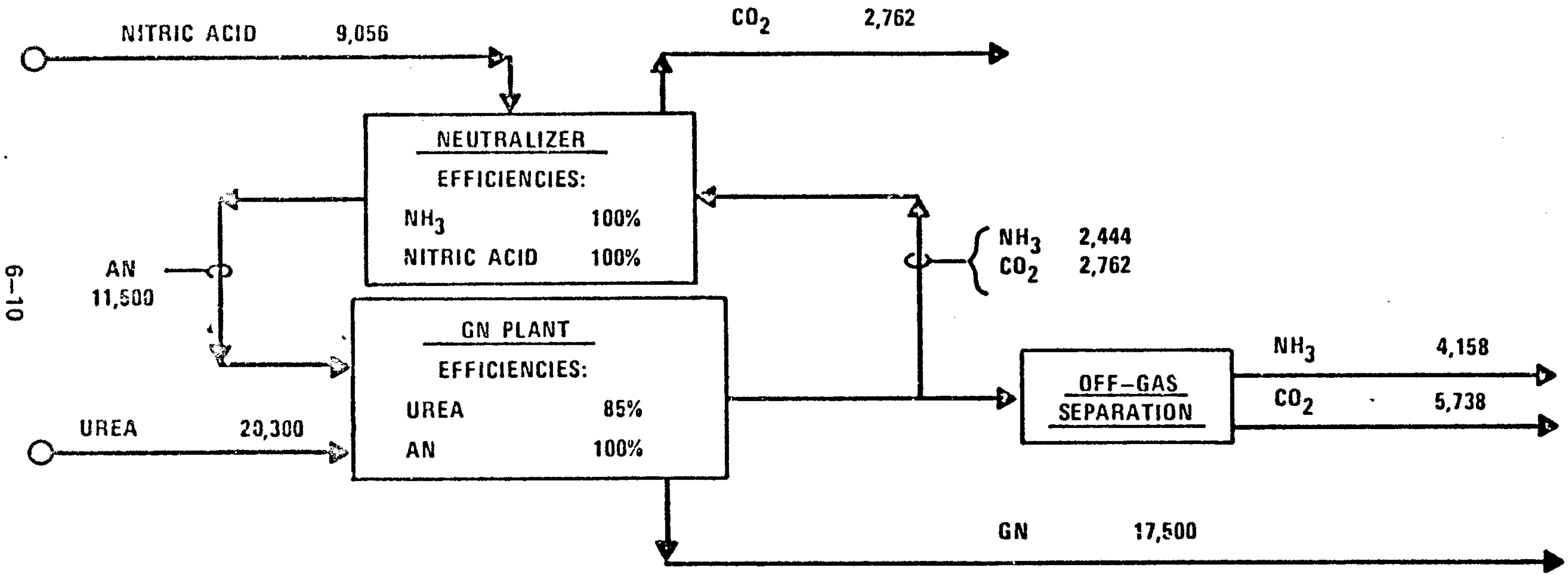
CASE V: BUY CO_2, NH_3, & AN/SELL NOTHING.
NEED UREA PLANT.



NOTES:
QUANTITIES IN TONS/YR.

FIGURE 6-1-5
GN OFF-GAS STUDY

CASE VI: BUY UREA & NITRIC ACID/SELL NH_3



QUANTITIES IN TONS/YR.

(PAGE 1 OF 2)

OFF GAS PROCESSING COMPARISONS

Case	I	II	III
Process Description	Purchase Ammonium Nitrate as an 81% solution & Prilled Urea in bulk. Separate Ammonia & CO_2 from off gas.	Same as Case I except additional ammonia storage facilities provided.	Facilities include a neutralizer. Purchase prilled urea in bulk & 53-58% nitric acid. Neutralize ammonia in off gas with acid to ammonium nitrate.
Buy Tons/Year			
a) Ammonium Nitrate Solution	11,500 tons as 100%	11,500 tons as 100%	--
b) Prilled Urea	20,300 tons	20,300 tons	20,300 tons
c) Ammonia	--	--	--
d) Carbon Dioxide	--	--	--
e) Nitric Acid	--	--	24,400 tons
Sell, Use or Dispose of			
a) Ammonium Nitrate Solution	--	--	19,500 tons
b) Ammonia	6,600 tons	6,600 tons	--
c) Carbon Dioxide	8,500 tons	8,500 tons	--
Capital Investment \$ Estimate Accuracy	1,078,000 ±25%	1,724,000 ±25%	821,500 ±25%
Operating Cost \$/year			
a) Raw Materials, Net	1,602,500	1,602,500	2,206,300
b) All other	530,900	530,900	403,200
Total Operating Cost \$/year Ex-Depreciation & Return	2,133,400	2,133,400	2,609,500
Ammonia Storage:	7 Days	90 Days	0
Advantages	1. Lowest Operating Cost	1. Same as I on Operating Cost. 2. Increase NH_3 storage so that Internal (Army) demand does not have to match GN plant operation and flexibility is provided in shipments.	1. Simple operation with processes. 2. Operating Cost excluding material is lowest. 3. O/L & R/L requirement minimum.
Disadvantages	1. CO_2 quality will not be suitable for commercial use and has no value. 2. Ammonia must be used internally (Sunflower) at same rate as produced from GN plant or disposed of elsewhere. 3. Sale of CO_2 to market is highly questionable.	1. CO_2 quality will not be suitable for commercial use and has no value. 2. Sale of CO_2 to market is highly questionable.	1. 19,500 tons/year of ammonium nitrate solution must be used internally, sold or traded. 2. CO_2 recovery is not practical. 3. Raw Material costs are maximum. 4. Possible Urea contamination of ammonium nitrate sol.

EXHIBIT 6-2
(PAGE 2 OF 2)

OFF GAS PROCESSING COMPARISONS

Case	IV	V	VI
Description	Facilities include an AOP and a neutralizer. Purchase prilled urea in bulk. Separate ammonia & CO2 in off gas - A portion of NH_3 is used as feed to the AOP and neutralizer.	Facilities include a total recycle urea plant. Purchase NH_3 & CO_2 for urea feed & amm. nitrate as an 81% solution. Recycle all off gas to urea plant.	Facilities include a neutralizer sized to meet GN requirements only. 53-58% nitric acid is purchased to meet AN production requirements. Urea is purchased and excess ammonia is separated from the off gas and sold.
Buy Tons/Year			
a) Ammonium Nitrate Solution	--	11,500 tons as 100%	--
b) Prilled Urea	20,300 tons		20,300 tons
c) Ammonia	--	4,900 tons	--
d) Carbon Dioxide	--	6,300 tons	--
e) Nitric Acid	--	--	9,056 tons
Sell, Use or Dispose of			
a) Ammonium Nitrate Solution	--	--	
b) Ammonia	1,500 tons	--	4,156 tons
c) Carbon Dioxide	8,500 tons	--	5,738 tons
Capital Investment $ Estimate Accuracy	2,790,000 ±25%	4,087,000 ±25%	1,135,400 ±25%
Operating Cost $/year			
a) Raw Materials, Net	1,410,000	1,131,500	1,681,800
b)*All other	1,298,500	1,389,900	547,300
Total Operating Cost $/year Ex-Depreciation & Return	2,708,500	2,521,400	2,229,100
Ammonia Storage (Days)	7 Days	7 Days	7 Days
Advantages	1. Use of proven processes. 2. Ammonia disposal outside facilities minimized. 3. Raw material costs are low.	1. There are no byproducts to dispose of. 2. Recovery of NH_3/CO_2 off gas from GN plant & recycle to urea appears practical.	1. Moderate Operating Cost. 2. Reduction in ammonia sales. 3. Inbuilt flexibility, i.e. in the event that separation system should fail, operations can continue, even if at a reduced level.
Disadvantages	1. Total facilities complicated by addition of a complex unit. 2. Ammonia/CO_2 separation reliability is questionable. 3. Some ammonia must be used outside GN facilities or sold. 4. Sale of CO_2 to market is highly questionable.	1. Total facilities complicated by addition of a highly complex unit. 2. Investment cost is maximum.	1. CO_2 quality will not be suitable for commercial use and has no value. 2. Need to purchase and handle nitric acid.

FIGURE 6-3

OFF-GAS CAPITAL INVESTMENT $

	Case I	Case II	Case III	Case IV	Case V	Case VI
MAJOR PLANT COMPONENTS						
1. Total Recycle Urea					2,730,000	
2. AOP				1,446,000		
3. Neutralizer			218,000	178,000		128,000
4. Gas Separation	561,000	561,000	--	561,000	561,000	466,000
5. Evaporation	91,500	91,500	91,500	91,500	200,000	91,500
6. Urea Melter	20,000	20,000	20,000	20,000		20,000
7. Amm. Liquefaction	92,000	92,000		92,000		76,500
TANKAGE & STORAGE						
1. Bulk Urea	110,000	110,000	110,000	110,000		110,000
2. Anhydrous Ammonia	108,000	749,000		57,000	108,000	108,000
3. Liquid CO_2	--				160,000	--
4. AN Solution	63,000	63,000	175,000	16,600	63,000	16,600
5. 58% HNO_3			175,000	21,000		90,000
6. Urea Surge					16,600	--
EXTRA UTILITIES						
1. Steam				7,200	25,200	--
2. Power				55,000	55,000	--
3. Cooling Water				35,000	44,800	--
4. Site Development	10,000	10,000	20,000	40,000	40,000	10,000
Total Installed Investment	1,055,500	1,696,500	809,500	2,730,300	4,003,600	1,116,600
SPARES & SUPPLIES						
2.25% of Major	17,200	17,200	7,400	55,200	78,100	15,400
1.0% of Tanks & Stores	5,300	10,300	4,500	4,500	3,600	3,400
1.25% of utilities			1,200		1,700	
TOTAL CAPITAL INVESTMENT	1,078,000	1,724,000	821,500	2,790,000	4,087,000	1,135,400

FIGURE 6-4

OFF-GAS OPERATING COSTS $/YEAR

		Case I	Case II	Case III	Case IV	Case V	Case VI
I.	Capital Investment	1,078,000	1,724,000	821,500	2,740,000	4,087,000	1,135,400
II.	Raw Materials						
	a) Urea @ $75	1,522,500	1,522,500	1,522,500	1,522,500	1,522,500	1,522,500
	b) Ammonia @ $75	-495,000	-495,000	--	-112,500	367,500	-311,700
	c) HNO_3 @ $52			1,268,800			471,000
	d) NAL SOL @ $50 (Buy)	575,000	575,000			575,000	--
	e) NAL SOL @ $30 (Sell)			-585,000			--
	f) CO_2 @ $30					189,000	--
	TOTAL MAT'L COST	1,602,500	1,602,500	2,206,300	1,410,000	1,131,500	1,681,800
III.	Operating Costs						
	a) Operating Labor	91,750	91,750	91,750	263,500	240,500	114,750
	b) Repairs Labor	10,400	10,400	10,400	61,350	56,150	15,600
	c) Direct Sup'v	33,200	33,200	33,200	92,800	76,200	49,800
	d) Overhead	135,350	135,350	135,350	417,650	372,850	130,350
	e) Maint. Mat'l	54,800	54,800	25,600	164,500	237,000	47,000
		325,500	325,500	296,300	999,800	982,710	357,500
IV.	Utilities						
	a) Steam @ $1.20/1000#	152,000	152,000	85,000	163,500	258,500	150,000
	b) Power @ 1.54/KW	17,800	17,800	10,000	69,900	75,000	17,800
	c) C.W. @ $0.05/1000 Gal.	35,600	35,600	11,900	65,300	73,700	22,000
		205,400	205,400	106,900	298,700	407,200	189,800
	TOTAL PLANT COST EX-DEP.	2,133,400	2,133,400	2,609,500	2,708,500	2,521,400	2,229,100

EXHIBIT 7.1
(PAGE 1 of 2)

DESCRIPTION OF BAF-GN PROCESS AND U/AN-GN PROCESS

The two (2) processes for the manufacture of guanidine nitrate (GN) included in this economic study are the British Aqueous Fusion (BAF-GN) Process and the Urea/Ammonium Nitrate (U/AN-GN) Process. The following is a brief description of the process flows for each of the processes.

7.1.1 BAF-GN Process

Calcium cyanamide is reacted with excess ammonium nitrate to yield calcium nitrate and guanidine nitrate. The reaction product is treated with ammonium carbonate to precipitate calcium carbonate and recover the nitrate values as ammonium nitrate. The slurry is settled in decanters, and the clear liquor is sent to vacuum crystallizers. The cool slurry from the crystallizers is centrifuged and the crystals are sent to a dryer. The mother liquor is concentrated for recycle to the reactors.

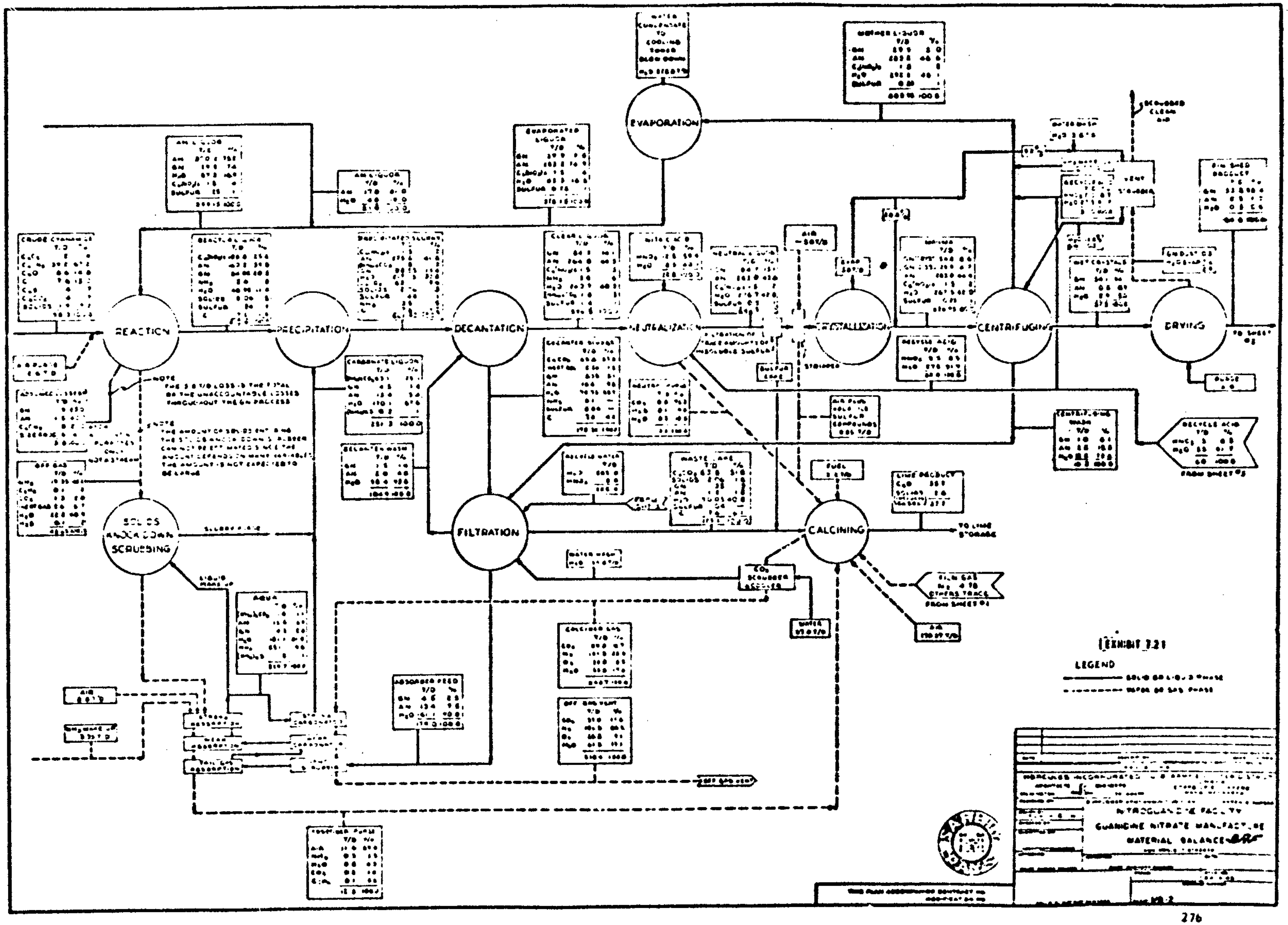
A by-product stream of ammonia from the reactors is absorbed in water to yield aqua ammonia. The slurry from the decanters is filtered and washed to yield a calcium carbonate which is calcined to yield lime and carbon dioxide. The carbon dioxide is absorbed in the aqua ammonia to yield ammonium carbonate which is used to precipitate the calcium from the reactor.

EXHIBIT 7.1
(PAGE 2 OF 2)

7.1.2 U/AN-GN Process

A solution of urea, ammonium nitrate, and guanidine nitrate composed of make-up urea and ammonium nitrate plus concentrated recycle mother liquor is passed over a silica catalyst at elevated temperature. The urea and ammonium nitrate react to yield guanidine nitrate and by-product ammonia and carbon dioxide. The gases pass to an absorber train to separate the ammonia from the carbon dioxide. The ammonia is sold, and the carbon dioxide is vented.

The reactor product is diluted with water to precipitate traces of ammelide which are spun out in a continuous solid bowl centrifuge. The clear liquid is cooled in a vacuum crystallizer. The slurry is centrifuged. The crystals are dried. The mother liquor is concentrated and is recycled to the reactor.

EVAPORATION
REACTION
PRECIPITATION
DECANTATION
NEUTRALIZATION
CRYSTALLIZATION
CENTRIFUGING
DRYING
FILTRATION
CALCINING
SOLIDS KNOCK DOWN SCRUBBING
MOTHER LIQUOR
CRUDE CYANAMIDE
OFF GAS
AQUA
TO LIME STORAGE
LIME PRODUCT
RECYCLE ACID
FUEL
WATER WASH
CO₂ SCRUBBER RECEIVER
EXHIBIT 7.21
LEGEND
SOLID OR LIQUID PHASE
VAPOR OR GAS PHASE
HERCULES INCORPORATED
NITROGUANIDINE FACILITY
GUANIDINE NITRATE MANUFACTURE
MATERIAL BALANCE
MB-2

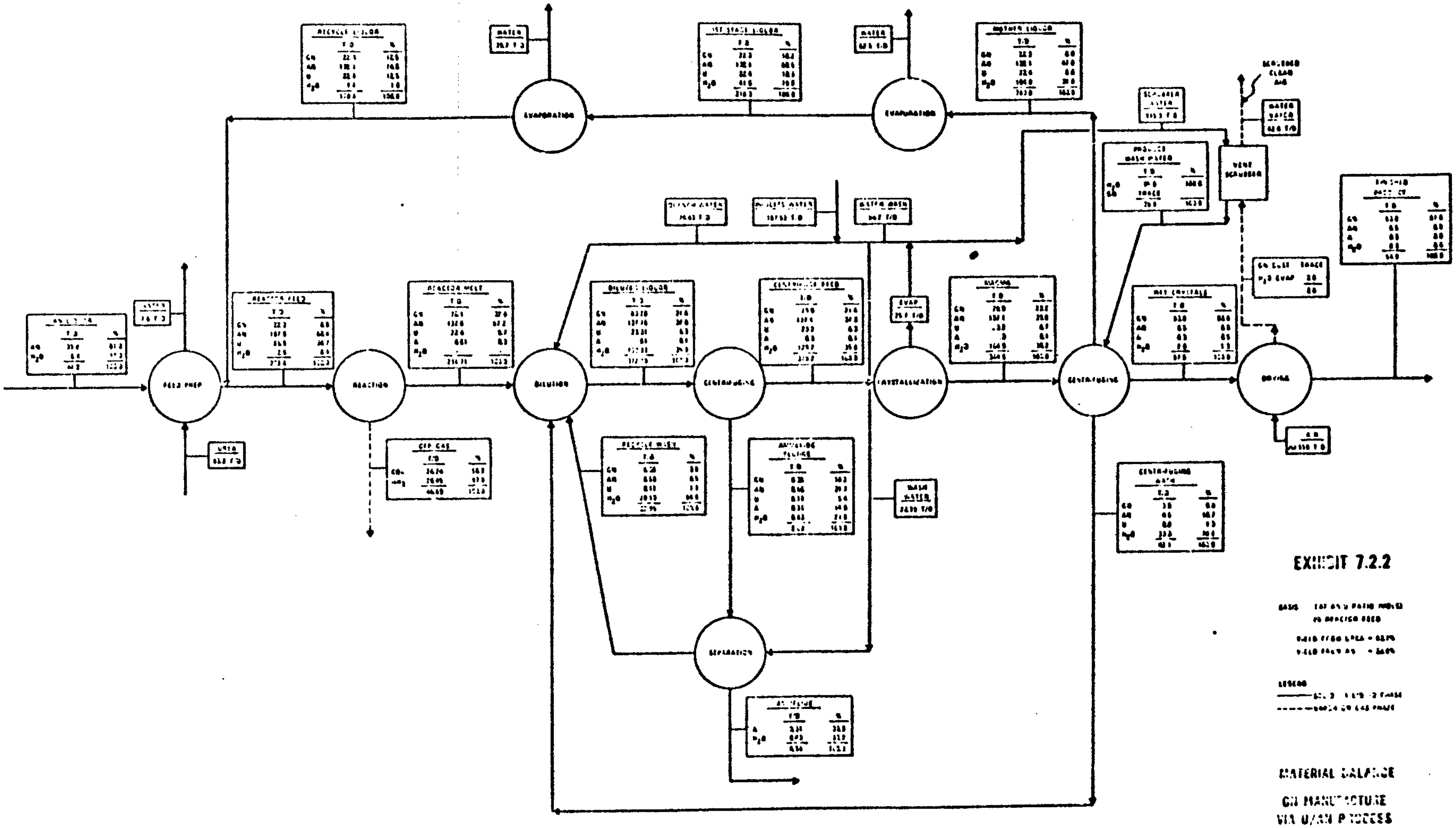


EXHIBIT 7.2.2

BASIS [illegible]

[illegible]

LEGEND

——— SOLID OR LIQUID PHASE

- - - - VAPOR OR GAS PHASE

MATERIAL BALANCE

CN MANUFACTURE
VIA U/AN PROCESS
HERCULES I.C. DAAA-21-71-C-0133

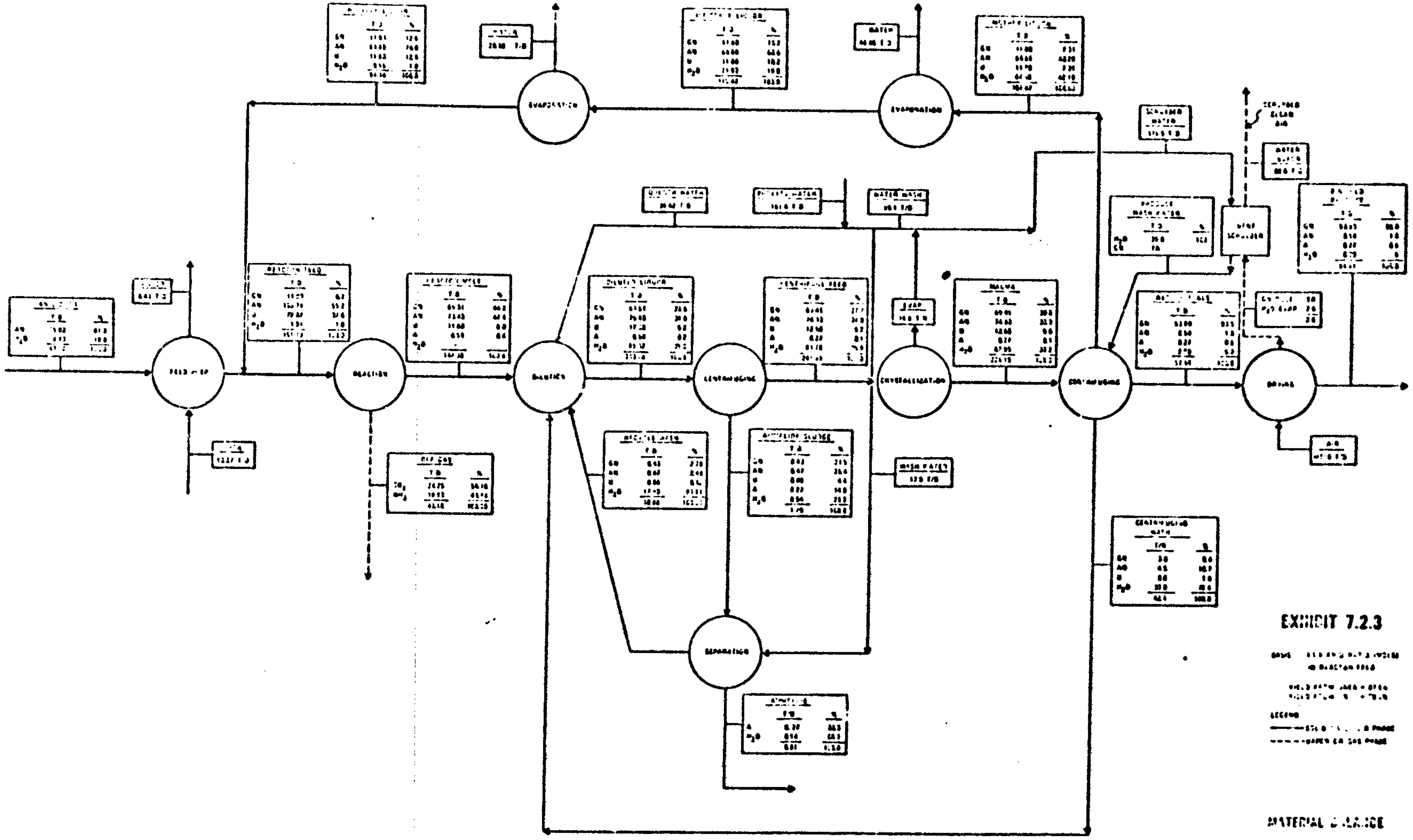


EXHIBIT 7.2.3

[illegible]

LEGEND

[illegible]

MATERIAL BALANCE

GN MANUFACTURE

VIA U, AN PROCESS

HERCULES INC. DAAA-21-73-C-[illegible]

EXHIBIT 7-3
(PAGE 1 OF 3)

SOURCE DERIVATION OF ACCOUNTING RATES

All rates used in the calculation of recurring operations costs were determined by the Sunflower Army Ammunition Plant financial management.

Salary and wage rates used are the actuals for June 1973.

Fringe Benefit and General and Administrative Overhead rates were calculated by annualizing the January through July 1973 actual experience which was then adjusted to include base and expense pool dollars for the indicated employment level for each process studied.

Rates used are summarized below:

A. Average Hourly Rates (June 1973 actual)

Wage	$4.80
Non-exempt salary	$5.36
Exempt salary	$8.47

B. Allowed Time Factor (Vacation, Holiday & Absenteeism)

Wage	10.20% of Total Hours
Non-exempt salary	10.81% of Total Hours
Exempt salary	11.39% of Total Hours

C. Direct Labor Calculation

	Hourly Rate	Yearly Pay*	Allowed %	Time %	Direct Labor
Wage	$4.80	$ 9,984	10.20%	$1,018	$ 8,966
Non-exempt salary	$5.36	$11,149	10.81%	$1,205	$ 9,944
Exempt salary	$8.47	$17,618	11.39%	$2,007	$15,611

*Based on 2080 Hours/year.

D. Fringe Benefit Rate

Fringe benefits are considered to be 100% variable with labor therefore, the same rate is used for both levels of production for each process.

EXHIBIT 7-3
(PAGE 3 OF 3)

E. General and Administrative Overhead cont'd.

	BAF 100%	BAF 25%	U/AN 100%	U/AN 25%
Pool Costs	$ 697,191	$ 697,191	$ 697,191	$ 697,191
Base	2,617,170	2,247,958	2,346,251	2,084,909
Rate	26.64%	31.01%	29.72%	33.44%

Elements of cost included in this pool are:

Maintenance Department (plant general maint.)
Engineering Department
Personnel, Plant Protection, Safety & Medical
General Services
Plant Administration
Relocated Costs
Allocated Fringe Benefit (Indirect Salaries)

Pg. 280

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EXHIBIT 7.4
(PAGE 1 OF 5)

DEPARTMENT OF THE ARMY
PICATINNY ARSENAL
DOVER, NEW JERSEY 07801

SARPA-MT-C

AUG 16 1973

Mr. Norman Steele
Hercules Inc
Kenvil, New Jersey 07847

Dear Mr. Steele:

Reference is made to Contract DAAA21-71-0193, Phase IV - Economic Study of Nitroguanidine Processes.

This Arsenal had indicated verbally that the Air Products and Chemical catalyst (532CP) cost input into the urea/ammonium nitrate process economic study would be based on the fixed capital and operating costs requirements to manufacture 200,000 lbs of catalyst per year. These estimates were to be supplied by this Arsenal.

Subsequent discussions with the catalyst supplier has resulted in an inability to supply all the economic data. Accordingly, the following procedure should be adopted in pursuance of the above referenced contract effort:

1. Hercules is to determine the annual Air Products and Chemicals silica bead, #532CP requirement based on operating experience in the pilot plant.

2. Hercules is to assume that this catalyst will be available for purchase from Air Products and Chemicals Inc in the quantities needed.

3. Hercules is to determine the estimated unit FOB cost of this catalyst based on the following estimated quantity/price quotation of the supplier.

Lbs. purchased/yr	FOB Unit Price
25,000	$5.00/lb
50,000	4.00/lb
250,000	2.50/lb

In order to determine the actual estimated price for intermediate quantities required, a curve should be plotted based on the above and the FOB estimated sales price taken from this curve using 25,000 lb increments. Freight cost should be added from the point of origin, Paulsboro, New Jersey to Sunflower Army Ammunition Plant, Kansas.

4. The estimated prices quoted in item 3 are to be assumed as of mid-1973. Price escalation due to inflation would be added to the 1973 price. However, predictions by the catalyst supplier indicate that other markets for this particular catalyst can be anticipated and that a rock bottom price of $1.50/lb can be foreseen. This latter price is for general information and not to be used in the formal economic report.

Sincerely yours,

C. H. NICHOLS
Contract Project Officer

Copy furnished:
Mr. Douglas Clarke
AMSAR-CPE, Mr. L. Guerrero

Air Products and Chemicals INC.
CHEMICALS GROUP
Five Executive Mall, Swedesford Road, Wayne, Pa. 19087

HOUDRY DIVISION

W. J. Cross, Jr., General Manager
R. G. Craig, Mkt. Mgr.
Tel: (215) 687-6150
Twx: 510-668-2034

EXHIBIT 7.4
(PAGE 3 OF 5)

December 21, 1972

Commanding Officer
Picatinny Arsenal
Dover, New Jersey 07801

Attention: SMUPA-MT-C
Mr. S. Wachtell

Gentlemen:

This confirms the telephone conversation that you, Mr. Nichols, and myself had on Wednesday, December 20, with regard to our supplying macroporous silica beads in the coming months.

As a result of our recent meeting on December 8, we in Air Products have reviewed the probable investment and manufacturing costs to produce the product in quantities up to 250,000 pounds per year on the assumption that you would be the sole customer. At the same time, you will recall that our former price schedules were based on projections of higher quantities to be produced.

As you are also aware, it is necessary for us to reinstall our pilot plant equipment as well as make some substantial improvements to it at a significant cost to ourselves. If this is done, however, it appears that we might have enough capacity to handle your potential requirements. Of course, a lot depends on the catalyst life when in use. Accordingly, we made the following proposal to yourselves:

1. For the immediate need of an additional 1,000 pounds of catalyst for pilot plant work, we propose a charge of $10,000 for set-up costs plus $2.25 per pound selling price, f.o.b. Paulsboro, NJ. We indicated that should your

EXHIBIT 7.4
(PAGE 4 OF 5)

process become commercial and you undertake to buy commercial quantities from us at a later date, we would work out a refunding arrangement for the $10 000 set-up charge in the form of a credit against the catalyst purchased.

Insofar as timing is concerned, it will take 90 days to acquire the needed equipment that we propose to add to the pilot unit, and we feel it reasonable to allow another 30 days beyond this for completion of installation. The actual production of the 1,000 pounds, once we are operating, should take only a very short time, perhaps no more then a week. Your Mr. Caggiano asked in one telephone conversation what the timing would be on 200 pounds. Actually, what we would do in such a case would be to take the first 200 pounds completed from the 1,000 pounds; thus, if you wanted 200 pounds completed from the 1,000 pounds ahead of the balance, we would gain a few days but not a great amount of time.

2. Looking ahead to a situation in which you will be purchasing commercial quantities of catalyst and again on the assumption that you would prove to be the only customer that we would have for the material, we estimate the following prices for the product:

Pounds Purchased Per Year	Dollars Per Pound
25,000	5.00
50,000	4.00
250,000	2.50

For intermediate levels of production, you can estimate prices by drawing a curve through the above three points. I am sure you appreciate that these figures are estimates at this time and not firm quotations. Also the situation could change if we are successful in developing additional markets for the beads. Should our annual sales exceed 250,000, then the price for quantities in the 25,000 pound range would obviously be lower.

EXHIBIT 7.4
(PAGE 5 OF 5)

Regarding the question of our assuring you of a supply of the material, I indicated upon receipt of your order for a 1,000 pounds under the terms of this proposal, we would initiate installation of the pilot plant equipment. Furthermore, we agree to maintain the equipment in operable condition till the end of 1974. This date will provide you ample time to make a decision on your commercial facility and indicate to us whether we will have to provide additional production capacity beyond the initial pilot plant stage.

I further indicated that we have discussed this proposal together with our potential financial commitments with our Profit Center's General Manager, who has given his agreement to this plan of action. At the same time, I am sure you are aware that for substantial expenditures for new equipment we always have to seek formal approval from our Board of Directors. Since we have provided in the above estimated costs to make this what we believe a viable project, we foresee no problem in this regard.

I hope that this letter summarizes all of the information that bears on your situation and which will permit you to make an early decision from your end. Certainly, we are most interested in working with you, and we want to cooperate with you in every way possible. If there are more questions, please do get in touch with us.

Yours very truly,

G. W. Higginson
Manager, Catalyst Sales

GWH:mef

cc: Mr. C. Nichols, Picatinny Arsenal
Mr. Norman Steel, Hercules, Kenvil, NJ

REFERENCES

1. Boatright, L. G. and Mackay, J. S., U.S. Patent 2,783,276, February 26, 1957, Assigned to American Cyanamid Company.

2. Steele, N. W., Doyle, J. A. and Whippen, M. G., Hercules Incorporated, Kenvil, New Jersey, "Process Engineering Design for Manufacture of Guanidine Nitrate," Final Report - Volume I, August 1973.

3. Mackay, J. S., U.S. Patent 2,949,484, August 16, 1960, Assigned to Pittsburgh Coke and Chemical Co.

4. Roberts, E. L. and Martin, T., U.S. Patent 3,043,878, July 10, 1962, Assigned to Minister of Aviation in Her Majesty's Government of the United Kingdom.

5. Hercules Incorporated, Kenvil Plant Research Record Books 2131, 2121, 2956 and 2126.