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**CONTINUOUS MANUFACTURING OF  
NITROCELLULOSE BY  
MAGNESIUM NITRATE METHOD - VOLUME I**

TRUMAN K. DANIEL, ET AL  
RADFORD ARMY AMMUNITION PLANT  
RADFORD, VIRGINIA

R. L. TRASK  
E. O. HANN  
C. W. LEWIS  
ARRADCOM, DOVER, NEW JERSEY

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20. ABSTRACT (Continue on reverse side if necessary and identify by block number) The objectives of this project were to design, procure, install and evaluate pilot and prototype equipment for the continuous production of nitrocellulose (NC) by the magnesium nitrate process. Laboratory and bench-scale studies were performed to establish methods, techniques, and basic design concepts for the continuous prototype process. Laboratory-scale studies evaluated the conditions for nitration and purification of NC. Bench-scale studies established nitration conditions, determined the use of attrition mills, and evaluated ion exchange as a means of recovering magnesium nitrate. →	17. DISTRIBUTION STATEMENT (of the abstract entered in Block 20, if different from Report)	

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20. ABSTRACT (Continued)

Corrosion studies were used in evaluating construction materials and selecting equipment for the continuous pilot plant. A nitration, purification, chemical recovery, and pollution abatement process flow was established. Equipment for the prototype plant was designed, procured, and installed.

Before the evaluation of the continuous pilot plant was begun, equipment was modified to permit evaluation of an acid recycle system proposed for use in the continuous mixed-acid NC manufacturing facility. Following this evaluation, the project was terminated.

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## SUMMARY

In the mid-1950's work was conducted by several agencies and plants on a process for nitrating cellulose utilizing magnesium nitrate in lieu of sulfuric acid as the dehydrating agent for the water of reaction. Several small lots of magnesium nitrate NC were prepared batchwise for evaluation. This NC was granular and harsh to the touch; however, the material did meet all of the requirements of the existing NC specification (JAN-N-244) with the exception of the 134.5°C Heat Test (German accelerated stability test).

The material was further processed into various propellants for evaluation of processing techniques and propellant characteristics. Reports published by Picatinny Arsenal stated that the NC failed to dehydrate properly in the Loomis presses and required an increase in solvent-to-ingredient ratio to manufacture the propellants. The reports stated that the magnesium nitrate NC yielded propellants which were ballistically equal to propellants manufactured with the conventional mixed acid NC.

Based on the fact that magnesium nitrate NC had been successfully tested in military propellants and that the use of sulfuric acid is eliminated in the manufacture of NC by this process, thus reducing the amount of stream pollution, Picatinny Arsenal recommended that a pilot plant study be funded to evaluate the process more extensively, but this recommendation was not approved. However, in the late 1960's emphasis on reducing stream pollutants resulted in a review of methods by which these reductions could be accomplished. The magnesium nitrate NC process was revived, with modifications, as a potential for effecting these reductions as associated with the manufacture of NC.

Bench-scale work showed that a continuous process for production of NC using magnesium nitrate instead of sulfuric acid as a dehydrating agent was technically feasible. The bench-scale studies investigated the nitration of cotton linters and wood pulp and the use of an attrition mill to initiate nitration, purification requirements for magnesium nitrate NC, various ion exchange resins and systems for the recovery of magnesium and nitrate ions from wastewater effluents, and corrosive effects on various candidate materials of construction to be used in the pilot plant. Blends of NC manufactured with the bench-scale equipment were provided US Army Armament Research and Development Command (ARRADCOM) for surveillance tests. Results of these tests indicated the NC was acceptable for military use and are included in Appendix F. A pilot plant was designed and built to evaluate this process. The pilot plant was designed as a scaled-down version of the continuous mixed acid nitration facility in order to permit the adaptation of the process developed in the pilot plant to the production-scale equipment. Additional features to be evaluated in the pilot plant included the initiation of nitration in attrition mills, countercurrent washing of the NC on a dewatering filter

and use of the filtrate as the initial countercurrent wash in the centrifuge, and the use of ion exchange resins to recover pollutants from process wastewater streams. A subpilot-scale magnesium nitrate/nitric acid concentrator was built to determine operating conditions for concentrating the spent nitrating mix for the pilot plant.

After a series of reviews with cognizant Government agencies, it was decided to modify the continuous mixed acid process to permit the recovery of excess acid from the NC with a dewatering filter and to recycle the filtrate to the initial wash stage of the continuous centrifuge as had been planned for the magnesium nitrate process. A decision was made to convert the magnesium nitrate pilot plant for evaluation of the modified mixed acid process. Work on the magnesium nitrate process was terminated by the Project Manager. After evaluation of the modified mixed acid process under another project, the pilot plant was placed in a protective storage status.

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## INTRODUCTION

### Background and Justification

Prior to the invention of the magnesium nitrate process for the manufacture of NC, the only commercial method for preparing nitric acid esters of cellulose employed nitrating mixtures containing essentially nitric acid, sulfuric acid, and water. In the mid-fifties, Hercules Incorporated obtained three United States patents on the magnesium nitrate process: (1) 2,776,964; (2) 2,776,965; and (3) 2,776,966. The first relates to a continuous system for the production of NC wherein a nitrating mixture essentially containing nitric acid, magnesium nitrate, and water is used. The second relates to new nitric acid esters of cellulose and their preparation. These esters are free of unstable sulfur compounds because they are manufactured utilizing mixtures containing essentially nitric acid, magnesium nitrate, and water. The third patent relates to a continuous method for the preparation of esters of cellulose in which these esters are manufactured from cellulose in sheet form wherein a nitrating mixture essentially containing nitric acid, magnesium nitrate, and water is the esterification medium.

Lewis and Hahn received United States Patent 3,714,143 relating to a continuous pollution-free process for manufacture of NC involving passing cellulose and nitric acid-magnesium nitrate, the nitrating agent, through an attrition mill, separating the spent nitrating agent from the NC, purifying the NC by a countercurrent water wash, treating the wash liquors with an ion exchange system to recover the magnesium and nitrate values therefrom, and recycling the latter and spent nitrating agent to the system.

The use of magnesium nitrate rather than sulfuric acid as a dehydrating agent in the manufacture of NC has a number of potential advantages. These include the elimination of the need for sulfuric acid manufacturing and concentrating facilities, reduced stream pollution, and decreased NC purification requirements.

Hercules Incorporated, at Radford Army Ammunition Plant (RAAP) was funded to perform laboratory and bench-scale investigations to confirm the original magnesium nitrate process for nitration of cellulose, and to investigate the initiation of nitration in attrition mills as a part of this process. Also, purification methods for this NC were investigated. Various ion exchange resins for recovery of magnesium nitrate and nitrate ions, and corrosion rates of various candidate materials of construction for pilot plant use were investigated. These studies verified the technical feasibility of the process and provided information for the design of equipment for a pilot scale evaluation of the process.

Additional funds were provided to design, install, and evaluate pilot or prototype equipment for the continuous manufacture of NC by the magnesium nitrate process, and to establish basic design criteria for the

utilization of this process in the modernized continuous nitration system to reduce stream pollution and reduce the time required for purification of the NC by eliminating the use of sulfuric acid. The scope of this project also included the production of limited quantities of NC and propellants for product evaluation.

## LABORATORY INVESTIGATION

The purpose of the laboratory investigation was to prepare mixes of magnesium nitrate, nitric acid and water, establish techniques for their analysis and with them produce a range of NC from cotton linters and wood pulp that would meet present NC specifications of MIL-N-244A, and establish nitrating parameters to be used in the bench-scale unit.

A literature search consisted of previously published work including Report 2502, "Product Development Study of Nitrate - 41 Nitrocellulose," by Picatinny Arsenal, which described successful nitrations of wood pulp but only minor work on linters. Data from Report 2502 contained specific mixes of magnesium nitrate, nitric acid, and water, and the nitrating parameters of time, temperature, and acid mix/cellulose ratios that had produced both high and low grade NC from wood pulp.

Before preparing one of these mixes, tests were conducted to determine the solubility of magnesium nitrate in nitric acid at various temperatures. The results obtained are shown in figure 1. Since the acid-salt mixes must be free of undissolved magnesium nitrate, this proved to be a critical area, particularly with high grade mixes, as the magnesium nitrate quickly fell out of solution with a drop of temperature.

The strongest nitric acid available was analyzed and weighed (the nitric acid should be 98%, minimum) and a theoretical mix calculated. The acid mix was calculated so that the theoretical mix was within the solubility range of the magnesium nitrate in the nitric acid at the temperature of the proposed nitration, and the acid mix was kept at, or above, the proposed nitration temperature. The indicated amount of magnesium nitrate hexahydrate was placed in a stainless steel beaker, weighed and then fused over a bunsen burner to the previously calculated weight. This fusing yielded a solution of up to 72 percent magnesium nitrate, and it was found impractical to fuse beyond this point. The molten magnesium nitrate while still hot, approximately 170°C, was carefully poured into the nitric acid with continuous stirring, and the temperature of the mix rose quickly to approximately 80°C. The acid mix was analyzed by pipetting 25-30 grams of the mix into a known weight of cold water, weighed, mixed, and this mixture was analyzed for nitric acid in the conventional manner. A disodium ethylene diamine tetraacetate dihydrate (EDTA) titration was used to determine the percentage of magnesium nitrate.

A statistical study was used to establish a test plan to determine the effects of time, temperature, acid mix-cellulose ratios and magnesium nitrate-nitric acid-water ratios on nitration of cellulose (figs. 2 through 4).

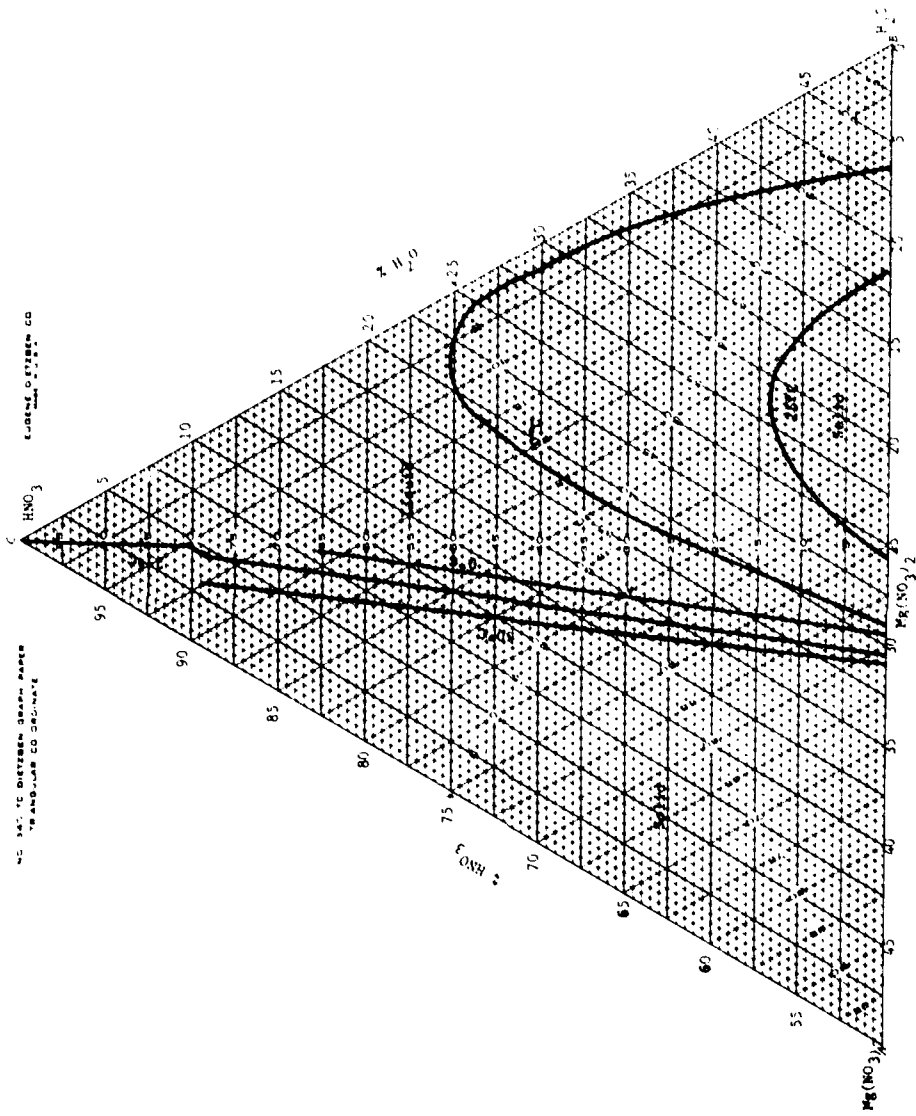


Figure 1. Mg(NO<sub>3</sub>)<sub>2</sub> - HNO<sub>3</sub> - H<sub>2</sub>O solubilities.

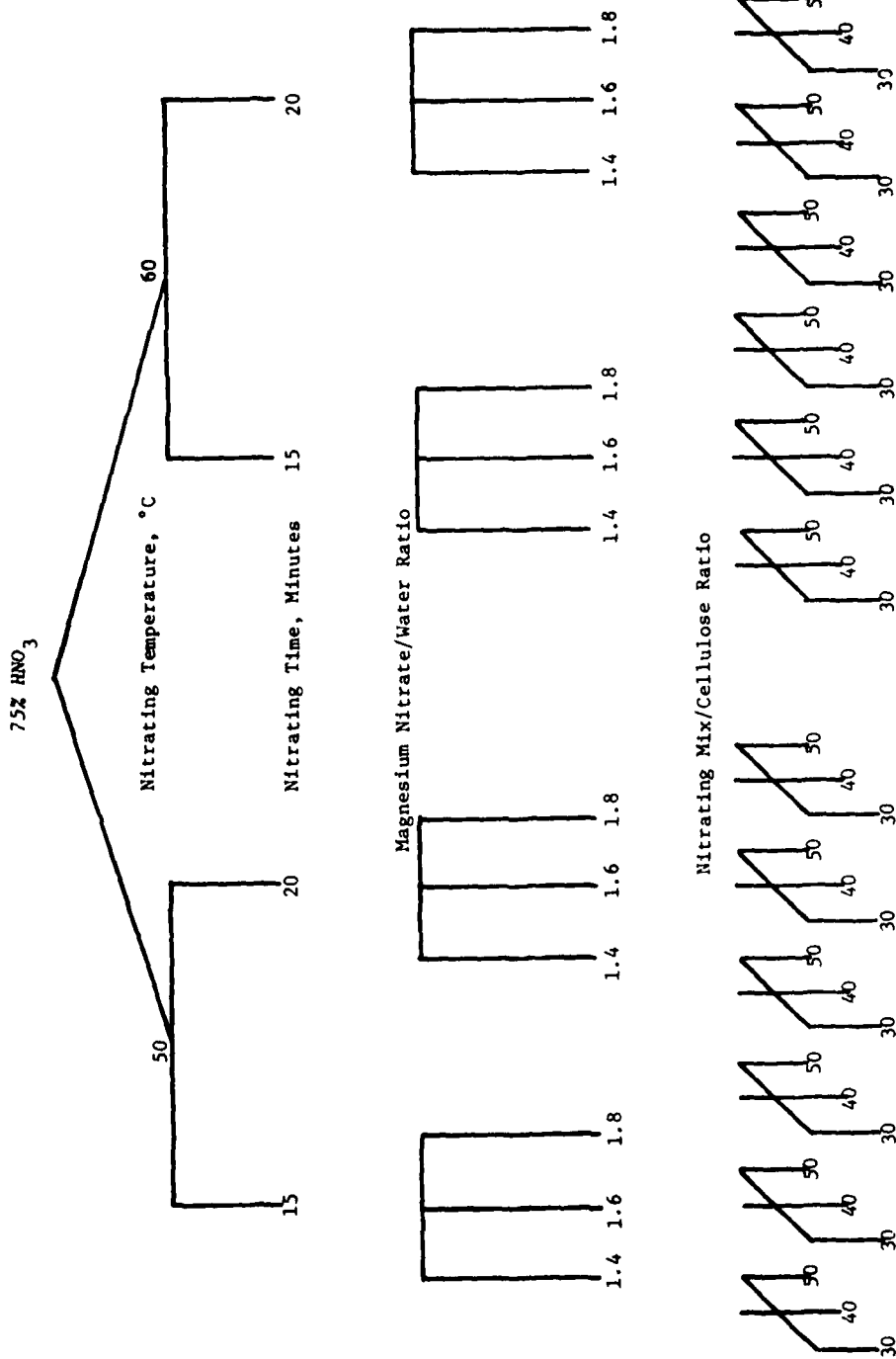


Figure 2. Test plan to determine effects of time, temperature, acid mix-cellulose ratios and magnesium nitrate-nitric acid-water ratios on the nitration of cellulose using 75% nitric acid.

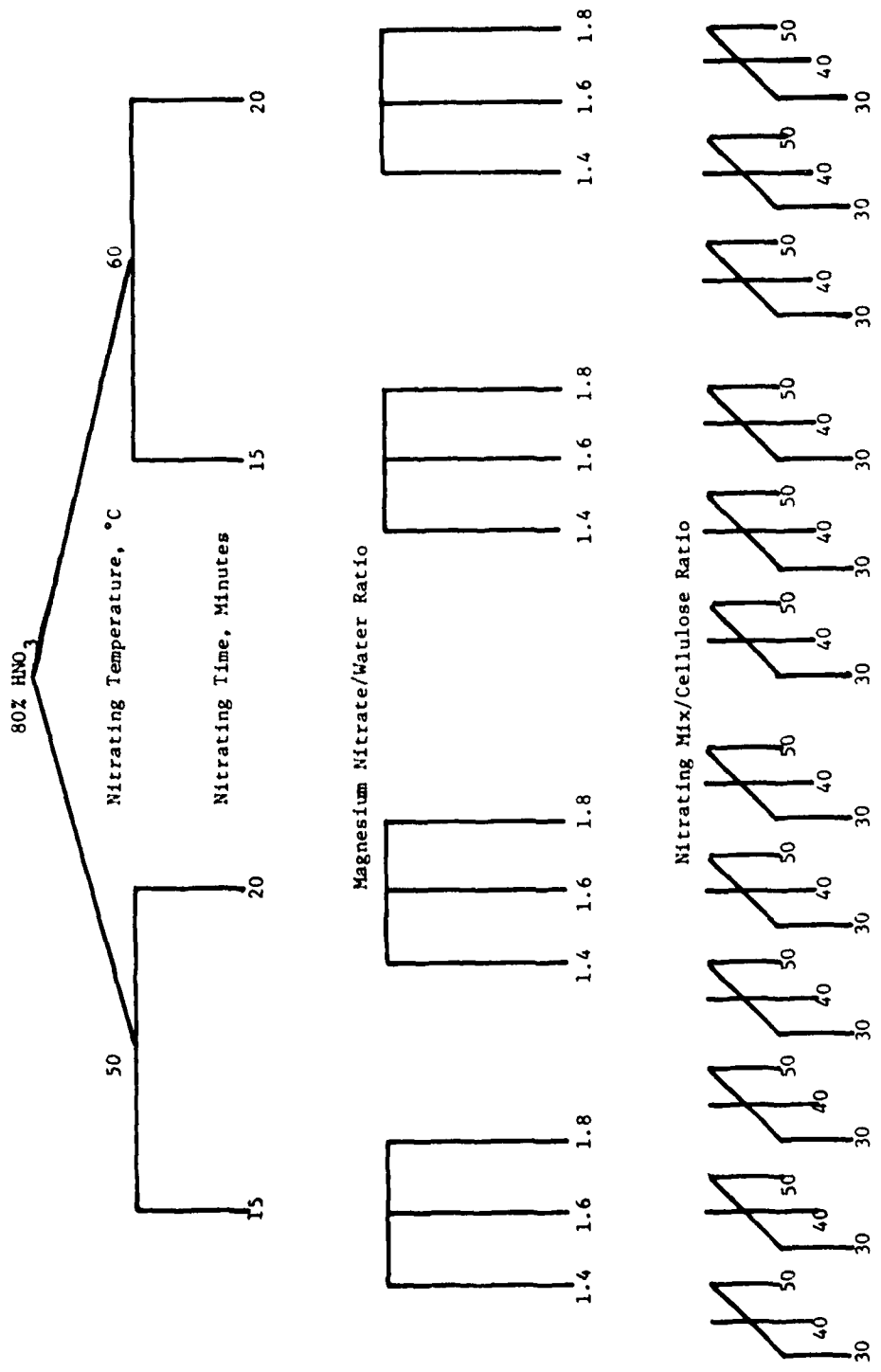


Figure 3. Test plan to determine effects of time, temperature, acid mix-cellulose ratios and magnesium nitrate-nitric acid-water ratios on the nitration of cellulose using 80% nitric acid.

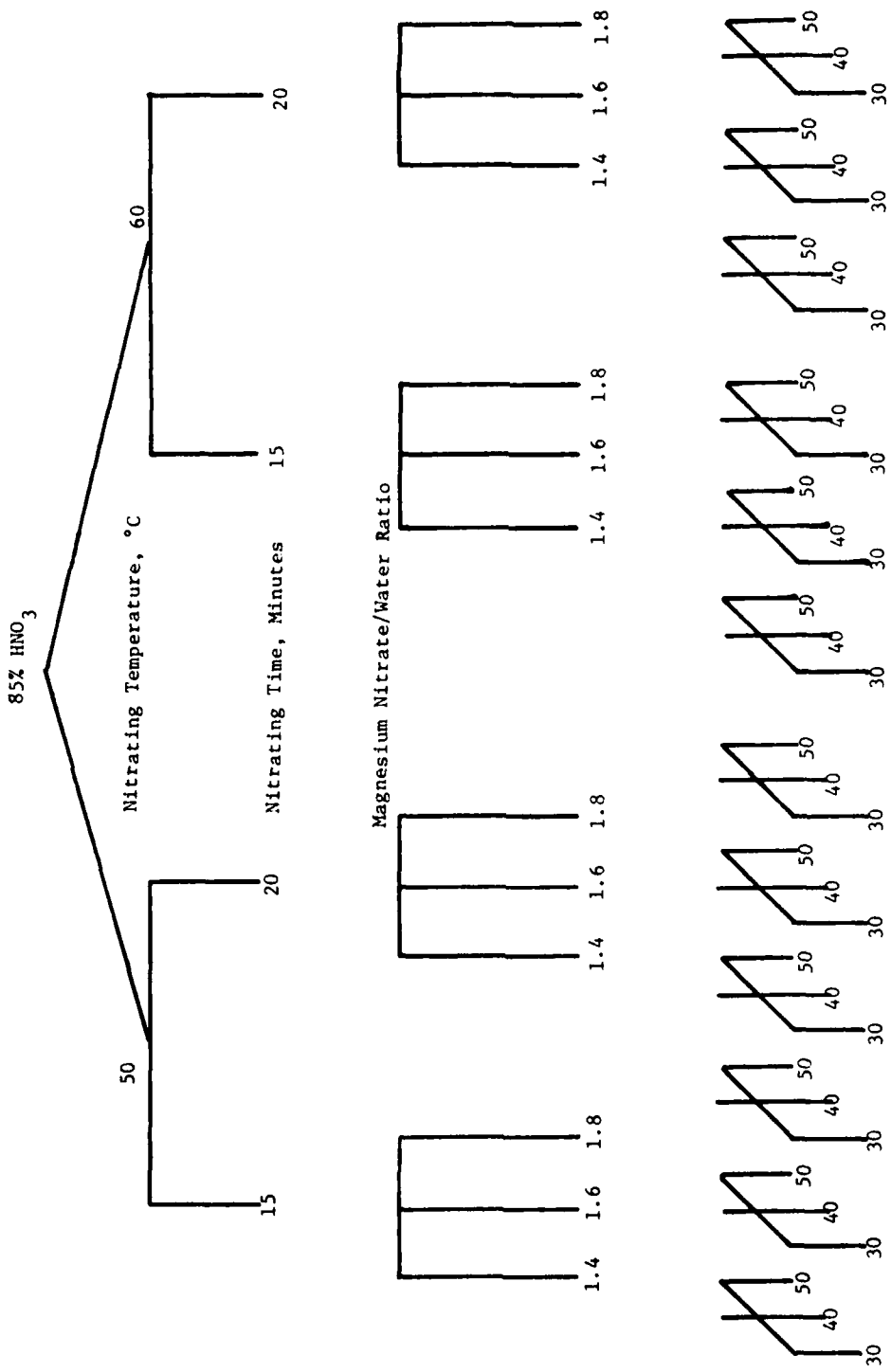


Figure 4. Test plan to determine effects of time, temperature, acid mix-cellulose ratios and magnesium nitrate-nitric acid-water ratios on the nitration of cellulose using 85% nitric acid.

An exploratory series of laboratory-scale nitrating mixes were made in an attempt to bracket the desired nitrating mixes for low grade (12.6 percent nitrogen) and high grade (13.35 percent nitrogen, minimum) NC. Nitrations were conducted using the equipment shown in figure 5, the acid mix being maintained above the nitration temperature in the balloon flask seated in a heating jacket and fitted with a condenser to prevent loss of nitric acid and water. Twenty grams of cellulose were placed in a one-liter stainless steel beaker with 600 grams of the acid mix, stirred continuously with an air-operated agitator, and the temperature held as required for specific nitrating periods. The nitration temperatures ranged from 40 to 60°C, and the time was 10, 15, or 20 minutes. The temperature of the nitration always rose approximately 4°C in the first few minutes of nitration. Following each nitration the NC was filtered on a regular Buchner funnel to remove spent acid, drowned in cold water and ground in cold water in a Waring Blender. The sample was then washed free of acid on a stainless steel Buchner funnel of five micron porosity, filtered, dried, and analyzed. The nitrations were performed on both regular plant linters and on plant shredded wood pulp, and in all cases the NC was hard and granular. The nitrogen results first obtained were low and erratic, although they improved as better control of the nitrations and the makeup and analysis of acid-salt mixes were developed. The results were still much lower on linters than those obtained under similar conditions as quoted in Report 2502. Three high grade nitrations (averaging 13.35 percent nitrogen) with good acetone solubility\* were obtained using wood pulp; whereas, three comparable nitrations of cotton linters yielded a nitrogen level of only 12.43 percent and bad acetone solubility (table 1). Denitration appeared to be a factor and evidence of this was revealed by microscopic examination.

Efforts were now made to control denitration and produce a high grade nitration of cotton linters. A series of nitrations were conducted using various forms of cotton linters (regular, cube cut and sheeted linters cut into squares of up to one-inch dimension). A concentrated high grade acid-salt mix was used in each case, the temperature held close to 50°C, the acid-cellulose ratio 30:1 and the time 10, 15, and 20 minutes. Immediately following nitration various after-treatments were used in an attempt to control denitration. These treatments included drowning the NC in an excess of cold 70 percent nitric acid in some tests and in cold five percent soda ash in others. After-treatment in an atmosphere of nitrogen was also used but little improvement was noted in all cases. Efforts had been made to procure a centrifuge, but since this was not available at the time and the unsatisfactory removal of spent acid from the NC immediately after nitration appeared to be a problem, one inch squares of sheeted cotton linters were nitrated, and after nitration these squares were hand pressed as dry as possible on a Buchner funnel and immediately ground in a Waring Blender containing five percent soda ash. The samples were washed free of alkali on a Buchner funnel, filtered, dried, and analyzed. This produced the first NC from linters that was close to

\*Acetone solubility as determined in accordance with MIL-N-244A.



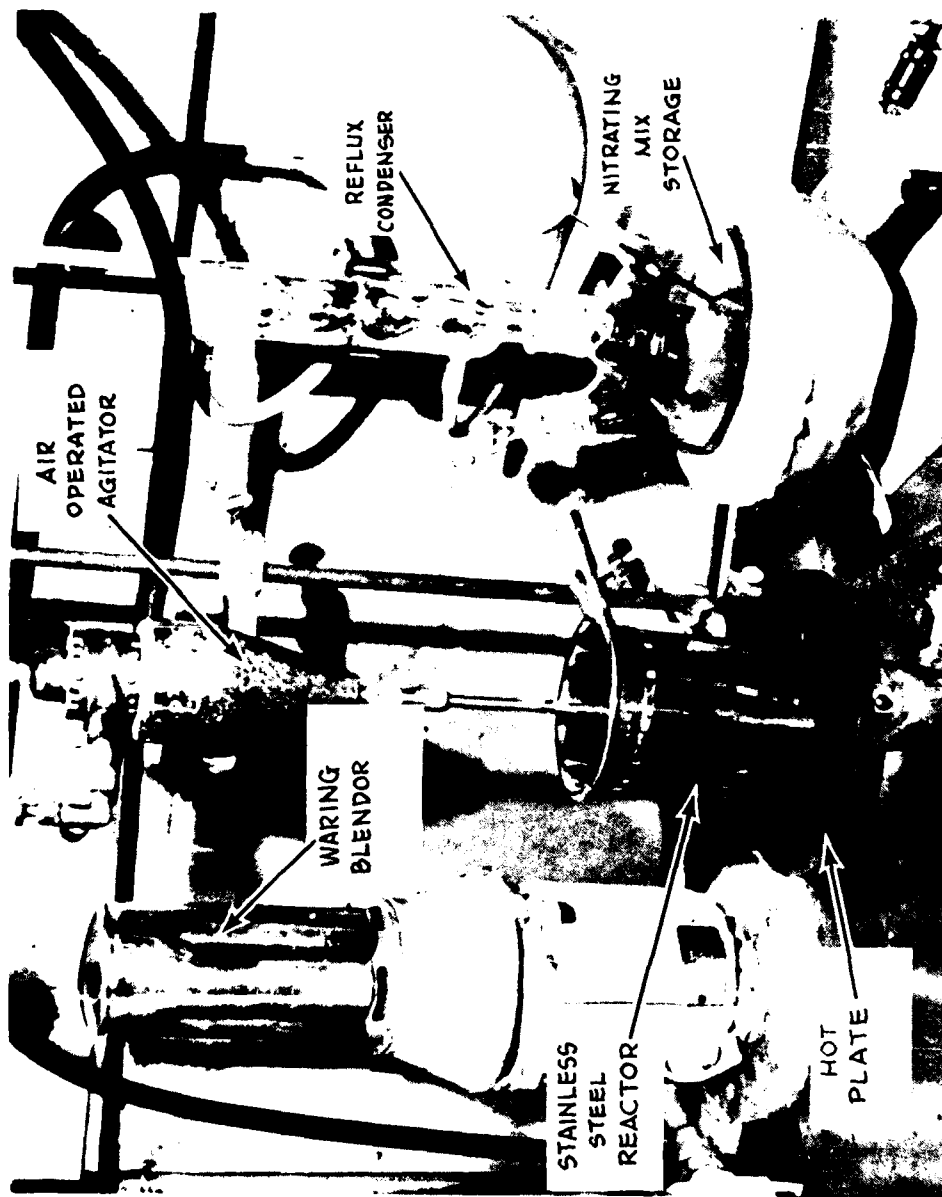


Figure 5. Equipment utilized in preparing laboratory-scale nitrating mixes.

Table 1

## Early laboratory nitrations

% $\text{HNO}_3$	Acid Mix		% $\text{H}_2\text{O}$	$\frac{\text{H}_2\text{O}}{\text{HNO}_3}$ Ratio	Time Minutes	Acid/Cell Ratio	N <sub>2</sub> %	AI	After-Treatment
	% $\text{H}_2\text{O}$	% $\text{HNO}_3$							
<u>Linters</u>									
82.08	10.83	7.09	1.53	12	40:1	12.12	Bad		Filtered quickly and dry
89.76	6.32	3.92	1.61	12	30:1	12.99	Bad		as possible in Buchner
82.56	10.96	6.48	1.69	15	30:1	12.35	Good		funnel. Washed with 3
86.34	8.94	4.72	1.89	20	30:1	12.25	Fair		liters cold water. Grou
85.10	8.95	5.95	1.50	10	40:1	12.05	Bad		cold water in Waring
85.10	8.95	5.95	1.50	15	40:1	12.11	Bad		Blendor.
85.10	8.95	5.95	1.50	20	40:1	12.33	Bad		
<u>Pulp</u>									
85.55	8.68	5.77	1.50	10	30:1	13.26	Good		Filtered quickly and dry
71.99	18.01	10.00	1.80	10	30:1	12.34	Fair		as possible in Buchner
86.14	8.95	4.91	1.82	10	30:1	13.47	Good		funnel. Washed with 3
85.10	8.95	5.95	1.50	20	30:1	13.33	Good		liters cold water. Grou
85.10	8.95	5.95	1.50	10	40:1	13.32	Good		cold water in Waring
85.10	8.95	5.95	1.50	10	40:1	13.33	Good		Blendor.

the high grade range of 13.35 percent nitrogen, minimum. The nitrogen value of this material was 13.28 percent with good acetone solubility. This test was repeated ten times; in some cases  $(\text{NH}_4)_2\text{CO}_3$  was substituted for the soda ash. The average nitrogen result was 13.44 percent, and the acetone solubility was good except for one test which gave a fair acetone solubility and a nitrogen result of 13.25 percent. This was the only result outside of the high grade range. Repeat tests were conducted using Parlin cube cut linters. The nitrogen results were higher than previously obtained on this cellulose but much lower than the desired 13.35 percent nitrogen minimum, indicating that the physical nature of the cube cut linters caused greater spent acid retention, and, therefore, denitration (table 2). This problem was overcome by use of a laboratory centrifuge.

The centrifuge was an International Stainless Steel Model 5-472 with a 127 mm (5 in.) center slung capable of 4,000 rev/min at 75 watts. This centrifuge was used to remove the spent acid immediately after nitration, and cold water was used in the basket to stop denitration. Improved nitrogen percentages and acetone solubility results were obtained using linters and some high grade NC was produced.

A series of nitrations were then conducted using linters and a high grade acid mix with the centrifuge providing rapid removal of the spent acid from the NC. In each test after the spent acid removal of the NC was washed with two liters of cold water and then divided into two parts. The first half of the sample was ground in cold five percent soda ash in the Waring Blendor while cold water was used for the second half of the sample. The samples were washed until neutral, then filtered, dried, and analyzed. The results shown in table 3 indicate that cold water washes, when correctly used, control denitration as satisfactorily as cold soda ash.

A nitration was next conducted on linters to compare nitric acid after-treatments with cold water. At intervals samples were removed from the nitrating bath, placed in the centrifuge to quickly remove the spent acid, and then washed with cold 70 percent nitric acid followed by a cold water wash and grinding in cold water. Other samples when removed were washed only with cold water. After grinding in the Waring Blendor, all samples were washed until neutral, then filtered, dried, and analyzed. The results (table 4) indicate that cold water washes, correctly used, control denitration as satisfactorily as cold 70 percent nitric acid. After 15 minutes, the time of nitration was not a factor as equilibrium had been reached in the reaction at or near this point.

The NC produced was always hard and granular and an investigation of a dual nitration was conducted to determine if an improved product could be obtained. Wood pulp was placed in a stainless steel basket and nitrated for five minutes with spent acid in this first stage as the nitrating mix, then quickly transferred to a fresh high grade mix for a

Table 2

## After-treatments to control denitration

Type Cellulose	HIO <sub>3</sub>		Mg(NO <sub>3</sub> ) <sub>2</sub>		Acid Mix		Nitration		AI	German* min	Solubility	After- treatment		
	%	%	%	%	H <sub>2</sub> O %	%G(NO <sub>3</sub> ) <sub>2</sub> H <sub>2</sub> O Ratio	Time minutes	Temp °C					Acid/Cell Ratio	N <sub>2</sub> %
Weyerhaeuser Pulp	70.94	17.94	11.12	1.61	1.61	1.61	10	50	30:1	11.84	Good	16	99 +	1
Weyerhaeuser Pulp	70.94	17.94	11.12	1.61	1.61	1.61	10	50	30:1	11.95	Good	17	99 +	1
Weyerhaeuser Pulp	70.94	17.94	11.12	1.61	1.61	1.61	10	50	30:1	11.87	Good	17	99 +	1
Weyerhaeuser Pulp	70.94	17.94	11.12	1.61	1.61	1.61	10	50	30:1	11.65	Good	15	99 +	2
Weyerhaeuser Pulp	70.94	17.94	11.12	1.61	1.61	1.61	10	50	30:1	11.55	Good	19	99 +	2
Weyerhaeuser Pulp	70.94	17.94	11.12	1.61	1.61	1.61	10	50	30:1	11.65	Good	19	99 +	2
Cotton Sheeted	86.88	8.63	4.49	1.92	1.92	1.92	10	50	30:1	12.98	Good	18	28.28	1
Linters														
Cotton Sheeted	86.88	8.63	4.49	1.92	1.92	1.92	10	50	30:1	12.96	Good	20	28.94	1
Linters														
Cotton Sheeted	86.88	8.63	4.49	1.92	1.92	1.92	10	50	30:1	12.74	Good	19	42.11	1
Linters														
Cotton Sheeted	86.88	8.63	4.49	1.92	1.92	1.92	10	50	30:1	12.85	Good	19	40.25	1
Linters														
Cotton Sheeted	86.88	8.63	4.49	1.92	1.92	1.92	10	50	30:1	12.83	Good	18	24.59	1
Linters														
Cotton Sheeted	86.88	8.63	4.49	1.92	1.92	1.92	20	50	30:1	11.59	Good	17	1.80	1
Linters														
Regular Linters	86.88	8.63	4.49	1.92	1.92	1.92	20	50	30:1	12.84	Bad	17	1.33	1
Squares CS	86.88	8.63	4.49	1.92	1.92	1.92	20	50	30:1	13.28	Good	18	3.96	4
Linters														
Regular Linters	86.88	8.63	4.49	1.92	1.92	1.92	20	50	30:1	11.91	Bad	20	1.47	5
Cotton Sheeted	84.75	9.49	5.76	1.65	1.65	1.65	5	50	30:1	10.75	Bad	30 +	57.05	6
Linters 1/2" SW														
Cotton Sheeted	84.75	9.49	5.76	1.65	1.65	1.65	10	50	30:1	11.81	Bad	30 +	49.99	6
Linters 1/2" SW														
Cotton Sheeted	84.75	9.49	5.76	1.65	1.65	1.65	15	50	30:1	12.33	Fair	23	1.65	6
Linters 1/2" SW														
Cotton Sheeted	84.75	9.49	5.76	1.65	1.65	1.65	15	50	30:1	12.93	Good	21	45.74	7
Linters 1/2" SW														
Cotton Sheeted	84.75	9.49	5.76	1.65	1.65	1.65	5	50	30:1	10.98	Bad	24	32.20	8
Linters 1/2" SW														

Table 2 (continued)

Type Cellulose	Acid Mix			Nitration			German* min	Solubility	After- treatment
	HNO <sub>3</sub> %	Mg(NO <sub>3</sub> ) <sub>2</sub> %	H <sub>2</sub> O %	Time minutes	Temp °C	Acid/Cell Ratio			
Cube Cut Linters	84.75	9.49	5.76	10	50	30:1	17	2.10	8
Cube Cut Linters	84.75	9.49	5.76	10	50	30:1	-	-	7
Cube Cut Linters	84.75	9.49	5.76	15	50	30:1	17	0.48	8
Cube Cut Linters	84.75	9.49	5.76	15	50	30:1	-	-	9
Cube Cut Linters	84.75	9.49	5.76	15	50	30:1	17	1.63	8
Cube Cut Linters	84.75	9.49	5.76	15	50	30:1	-	-	9
Cube Cut Linters	87.39	8.28	4.33	20	50	30:1	16	1.24	1
Cube Cut Linters	87.39	8.28	4.33	20	50	30:1	16	1.72	5
Cotton Sheeted Linters 1" Sq.	87.39	8.28	4.33	20	50	30:1	17	2.16	4
Wood Pulp	86.14	8.95	4.91	12	50	30:1	12	8.12	1
Wood Pulp	86.14	8.95	4.91	12	50	30:1	12	7.68	1
Wood Pulp	86.14	8.95	4.91	12	50	30:1	12	9.53	1
Wood Pulp	86.14	8.95	4.91	12	50	30:1	15	7.78	2
Wood Pulp	86.14	8.95	4.91	12	50	30:1	15	9.29	2
Wood Pulp	86.14	8.95	4.91	12	50	30:1	15	8.50	2
Cotton Sheeted Linters	86.34	8.94	4.72	10	50	30:1	21	62.91	1
Cotton Sheeted Linters	86.34	8.94	4.72	15	50	30:1	21	57.09	1
Cotton Sheeted Linters	86.34	8.94	4.72	20	50	30:1	16	51.30	1
Cotton Sheeted Linters	86.34	8.94	4.72	10	50	30:1	18	56.61	2
Cotton Sheeted Linters	86.34	8.94	4.72	15	50	30:1	19	56.15	2
Cotton Sheeted Linters	86.34	8.94	4.72	20	50	30:1	20	52.14	2

Table 2 (continued)

Type Cellulose	Acid Mix			Nitration			German* min	Solubility	After- treatment			
	HNO <sub>3</sub> %	Mg(NO <sub>3</sub> ) <sub>2</sub> %	H <sub>2</sub> O %	Mg(NO <sub>3</sub> ) <sub>2</sub> to H <sub>2</sub> O Ratio	Time minutes	Temp °C				Acid/Cell Ratio	AI %	
Regular Linters	86.34	8.94	4.72	1.89	10	50	30:1	12.77	Bad	16	1.90	1
Regular Linters	86.34	8.94	4.72	1.89	10	50	30:1	12.81	Bad	16	1.37	1
Regular Linters	86.34	8.94	4.72	1.89	10	50	30:1	12.78	Bad	16	1.56	1
Regular Linters	86.34	8.94	4.72	1.89	10	50	30:1	12.62	Bad	16	.76	2
Regular Linters	86.34	8.94	4.72	1.89	10	50	30:1	12.57	Bad	16	.75	2
Regular Linters	86.34	8.94	4.72	1.89	10	50	30:1	12.62	Bad	17	1.40	2
Regular Linters	86.34	8.94	4.72	1.89	15	50	30:1	11.88	Bad	17	1.58	3
Cotton Sheet Linters	86.34	8.94	4.72	1.89	15	50	30:1	11.99	Bad	17	.66	1
Cotton Sheet Linters	86.34	8.94	4.72	1.89	10	50	30:1	12.51	Good	17	43.37	1
Cotton Sheet Linters	86.34	8.94	4.72	1.89	15	50	30:1	12.91	Good	17	17.51	1
Cotton Sheet Linters	86.34	8.94	4.72	1.89	20	50	30:1	12.81	Good	17	2.93	1
1" Squares Cotton Sheeted Linters	87.44	8.29	4.27	1.94	20	50	37 1/2:1	13.50	Good	20	.99	4
Sheeted Linters	87.44	8.29	4.27	1.94	20	50	37 1/2:1	13.42	Good	-	.49	10
Sheeted Linters	87.44	8.29	4.27	1.94	20	50	37 1/2:1	13.52	Good	15	2.25	10
Sheeted Linters	87.44	8.29	4.27	1.94	20	50	37 1/2:1	13.39	Good	17	.09	4
Sheeted Linters	87.44	8.29	4.27	1.94	20	50	37 1/2:1	13.35	Good	16	3.24	4
Sheeted Linters	87.44	8.29	4.27	1.94	20	50	37 1/2:1	13.49	Fair	16	3.48	10
Sheeted Linters	87.44	8.29	4.27	1.94	10	50	37 1/2:1	13.55	Good	16	3.61	4
Sheeted Linters	87.44	8.29	4.27	1.94	10	50	37 1/2:1	13.45	Good	16	2.69	10
Sheeted Linters	87.44	8.29	4.27	1.94	10	50	37 1/2:1	13.25	Fair	16	4.32	10
Sheeted Linters	87.44	8.29	4.27	1.94	20	50	37 1/2:1	13.53	Good	18	2.26	4

Table 2 (continued)

\*The German test is the 134.5°C heat test as required by MIL-N-244A and conducted in accordance with MIL-STD-286, Method 409.1.

After-treatment code:

- 1 - Filtered quickly, drowned in 3 liters cold water, ground in Waring Blendor in cold water.
- 2 - Filtered quickly, drowned in 3 liters cold 70% HNO<sub>3</sub>, drowned in cold water, and ground in Waring Blendor in cold water.
- 3 - All nitration bath dumped into cold water, filtered, and drowned in cold H<sub>2</sub>O, 3 liters, and ground in Waring Blendor in cold water.
- 4 - Filtered and pressed as dry as possible by hand - direct to Waring Blendor, ground in 5% soda ash.
- 5 - Filtered, drowned in 3 liters 2 1/2% soda ash, ground in Waring Blendor in cold water.
- 6 - Filtered under nitrogen, drowned in 3 liters 5% soda ash and ground in Waring Blendor in 2 1/2% soda ash.
- 7 - Filtered under nitrogen direct to Waring Blendor, ground in 5% soda ash.
- 8 - Filtered, drowned in 3 liters 5% soda ash, ground in Waring Blendor in 2 1/2% soda ash.
- 9 - Filtered quickly direct to Waring Blendor, ground in 5% soda ash.
- 10 - Filtered and pressed as dry as possible by hand - direct to Waring Blendor, ground in 5% ammonium carbonate.

Table 3

Nitrations with centrifuge for comparing alkali versus cold water wash

<u>Linters</u>	<u>HNO<sub>3</sub> %</u>	<u>% (NO<sub>3</sub>)<sub>2</sub></u>	<u>H<sub>2</sub>O %</u>	<u>% (NO<sub>3</sub>)<sub>2</sub> to H<sub>2</sub>O Ratio</u>	<u>Temp. °C</u>	<u>Treatment</u>	<u>N<sub>2</sub> %</u>	<u>AI</u>
Hercules	85.14	10.02	4.81	2.08	60-70	Soda Ash Water	13.42 13.22	Good Good
Hercules	85.14	10.02	4.81	2.08	60-70	Soda Ash Water	13.40 13.33	Good Good
Buckeye	85.14	10.02	4.81	2.08	65-72	Soda Ash Water	13.46 13.47	Good Good
Buckeye	86.29	8.88	4.62	1.92	50-54	Soda Ash Water	13.30 13.26	Bad Good
Buckeye	86.29	8.88	4.62	1.92	50-58	Soda Ash Water	13.20 13.39	Good Good

Ratio Acid/Cell 30:1

Water and Soda Ash Cold

All washed with 1 liter ice water before grinding.



Table 4

Nitrations with centrifuge for comparing nitric acid versus cold water wash

<u>Time - minutes</u>	<u>Treatment</u>	<u>% 32</u>	<u>AI</u>
15	2°C 70% Nitric	13.64	Good
20	2°C 70% Nitric	13.68	Good
25	2°C Water	13.66	Good
38	2°C Water	13.76	Good

Acid - 85.34 HNO<sub>3</sub> - 9.88 Mg(ClO<sub>3</sub>)<sub>2</sub> - 4.74 H<sub>2</sub>O Ratio - 2.08

Buckeye Linters Ratio Acid/Cell - 48:1

Temp °C 58-62

further five minutes nitration in the second stage. The NC was centrifuged for two minutes, washed with three liters of cold water, ground in the Waring blender in cold water, washed until neutral then with hot water, filtered, dried, and analyzed. The nitrogen percentage result was comparable with the single-stage nitrations but failed to show any improvement in softness as the NC was still granular (table 5).

High and low grade NC could now be produced in the laboratory from either pulp or linters and denitration could be controlled by rapid and efficient removal of the spent acid in the centrifuge and subsequent cold water washes. A series of nitrations were conducted to optimize the work and define the nitrating parameters of time, temperature, and acid mix/cellulose ratio that would yield the most satisfactory results. The results of these nitrations are given in table 6, and from these results the following parameters, time 15 minutes, temperature 50-60°C, acid mix/cellulose ratio 37-1/2:1 were set to be used in future series of nitrations. The next series of nitrations using linters were conducted to determine the effect of the magnesium nitrate/water ratio on the nitrogen value of the NC. The nitric acid level was held as close as possible to 70 percent while the magnesium nitrate to water ranged from a 1.23 to a 1.46 ratio. This was designed to produce NC in the low grade range. The nitrogen value of the NC produced ranged from 12.24 to 12.72 percent and are shown in table 7.

A series of nitrations were then conducted using both pulp and linters and with the same parameters of time 15 minutes, temperature 50-60°C, acid mix/cellulose ratio 37-1/2:1, while the nitric acid value was held as close to 75 percent as possible and the magnesium nitrate to water ratio progressively increased. The series of nitrations were repeated while holding the nitric acid value as close as possible to 80 percent. The results shown in table 7 are averages of two or more nitrations in each case and show that the nitrogen value of the NC increases as the ratio of the magnesium nitrate to water increases.

A summary of work done in the laboratory to this point showed that the necessary mixes of nitric acid, magnesium nitrate, and water had been used to nitrate linters or pulp to produce low and high grade NC. This NC was tested and found in agreement with MIL-N-244A except for the 135°C heat test using methyl violet paper is not suitable for testing NC made by the magnesium nitrate process as the gases of decomposition are different from those found when nitric/sulfuric acid nitrated cellulose is tested. Further information and data of the purification and testing of NC produced by the magnesium nitrate process are included in a later section of this report entitled, "Viscosity Studies." A summary and analysis of laboratory data to date led to the recommendation that the parameters shown in table 8 should be used for initial nitration on the bench-scale unit.

Table 5

Wood pulp - single versus double nitrations

HNO <sub>3</sub> %		Acid Mix		Mg(NO <sub>3</sub> ) <sub>2</sub> /H <sub>2</sub> O		Nitration		AI	N <sub>2</sub> %	German min	Solubility
Mg(NO <sub>3</sub> ) <sub>2</sub> %	H <sub>2</sub> O %	Mg(NO <sub>3</sub> ) <sub>2</sub> Ratio	Time Minutes	Temp °C	Acid/Cell Ratio	Single Nitrations	Double Nitrations				
85.10	5.95	1.50	10	50	40:1			Good	13.32	13	5.31
85.10	5.95	1.50	10	50	40:1			Good	13.33	14	6.80
85.10	5.95	1.50	10	50	40:1			Good	13.32	14	8.75
86.14	4.91	1.82	12	50	30:1			Good	13.42	12	7.68
86.14	4.91	1.82	12	50	30:1			Good	13.47	12	9.53
<u>Double Nitrations</u>											
83.37	7.35	1.26	5	50	40:1			Good	13.39	14	1.66
85.61	5.38	1.67	5	50	40:1						
83.37	7.35	1.26	5	50	40:1			Good	13.41	14	1.17
85.61	5.38	1.67	5	50	40:1						
83.37	7.35	1.26	5	50	40:1			Good	13.18	15	2.89
85.61	5.38	1.67	5	50	40:1						
83.37	7.36	1.26	5	50	30:1			Bad	13.30	15	2.78
85.85	5.10	1.77	5	50	30:1						
83.37	7.36	1.26	5	50	30:1			Good	13.39	14	3.47
85.85	5.10	1.77	5	50	30:1						

Table 6

Nitration time versus percent nitrogen

HNO <sub>3</sub>	Acid Mix		Cellulose	Nitration			N <sub>2</sub> %	AI
	Mg(NO <sub>3</sub> ) <sub>2</sub>	H <sub>2</sub> O		Acid Mix/ Cell Ratio	Time minutes	Temp °C		
86.29	8.88	4.62	Buckeye Linters	30:1	10	50-60	13.34	Good
86.29	8.88	4.62	Buckeye Linters	30:1	15	50-60	13.58	Good
86.29	8.88	4.62	Buckeye Linters	30:1	20	50-60	13.44	Good
84.75	9.49	5.76	Cut Sheeted Linters	30:1	5	50	10.75	Bad
84.75	9.49	5.76	Cut Sheeted Linters	30:1	10	50	11.81	Fair
84.75	9.49	5.76	Cut Sheeted Linters	30:1	15	50	12.33	Fair
87.39	8.28	4.33	Parlin CC Linters	30:1	10	50	11.85	Bad
87.39	8.28	4.33	Parlin CC Linters	30:1	15	50	12.54	Good
87.39	8.28	4.33	Parlin CC Linters	30:1	20	50	12.97	Good
87.39	8.28	4.33	Parlin CC Linters	30:1	10	50	12.06	Fair
87.39	8.28	4.33	Parlin CC Linters	30:1	15	50	12.60	Fair
87.39	8.28	4.33	Parlin CC Linters	30:1	20	50	12.61	Bad
84.75	9.49	5.76	Cut Sheeted Linters	30:1	5	50	10.75	Bad
84.75	9.49	5.76	Cut Sheeted Linters	30:1	10	50	11.81	Bad
84.75	9.49	5.76	Cut Sheeted Linters	30:1	15	50	12.33	Fair
84.75	9.49	5.76	Cut Sheeted Linters	30:1	5	50	10.98	Bad
84.75	9.49	5.76	Parlin CC Linters	30:1	10	50	12.34	Good
84.75	9.49	5.76	Parlin CC Linters	30:1	10	50	12.63	Good
84.75	9.49	5.76	Parlin CC Linters	30:1	15	50	12.46	Good
84.75	9.49	5.76	Parlin CC Linters	30:1	15	50	12.44	Good
84.75	9.49	5.76	Parlin CC Linters	30:1	15	50	12.65	Good

Table 6 (continued)

HNO <sub>3</sub>	Acid Mix		Cellulose	Nitration			N <sub>2</sub> %	AI
	Mg(NO <sub>3</sub> ) <sub>2</sub>	H <sub>2</sub> O		Acid Mix/ Cell Ratio	Time minutes	Temp °C		
70.09	17.12	12.79	Buckeye Linters	30:1	15	39-48	12.46	Good
70.09	17.12	12.79	Buckeye Linters	37 1/2:1	15	39-48	12.69	Good
70.09	17.12	12.79	Buckeye Linters	45:1	15	33-39	12.72	Good
70.09	17.12	12.79	Buckeye Linters	30:1	15	42-52	12.55	Good
70.09	17.12	12.79	Buckeye Linters	37 1/2:1	15	44-53	12.58	Good
70.09	17.12	12.79	Buckeye Linters	47 1/2:1	15	43-53	12.74	Good
70.27	16.75	12.97	Buckeye Linters	30:1	15	40-45	12.10	Good
70.27	16.75	12.97	Buckeye Linters	37 1/2:1	15	48-52	12.46	Good
70.27	16.75	12.97	Buckeye Linters	45:1	15	45-50	12.46	Good
80.25	12.31	7.44	Buckeye Linters	30:1	15	48-55	13.47	Fair
80.25	12.31	7.44	Buckeye Linters	37 1/2:1	15	48-52	13.55	Fair
80.25	12.31	7.44	Buckeye Linters	45:1	15	49-52	13.54	Fair
80.25	12.31	7.44	Buckeye Linters	30:1	15	37-45	13.41	Fair
80.25	12.31	7.44	Buckeye Linters	37 1/2:1	15	38-45	13.59	Fair
80.25	12.31	7.44	Buckeye Linters	45:1	15	32-39	13.59	Fair

Table 6 (continued)

HNO <sub>3</sub>	Acid Mix		Cellulose	Nitration			N <sub>2</sub> %	AI
	ig(NO <sub>3</sub> ) <sub>2</sub>	H <sub>2</sub> O		Acid Mix/ Cell Ratio	Time minutes	Temp °C		
83.90	9.76	6.27	Parlin CC Linters	30:1	20	30-38	13.29	Good
83.90	9.76	6.27	Parlin CC Linters	30:1	20	50-54	13.15	Bad
83.90	9.76	6.27	Parlin CC Linters	30:1	20	50-58	13.20	Good
83.90	9.76	6.27	Parlin CC Linters	30:1	20	70-75	13.25	Good
83.90	9.76	6.27	Rayonier Pulp	30:1	20	30-38	13.26	Good
83.90	9.76	6.27	Rayonier Pulp	30:1	20	50-58	13.20	Good
83.90	9.76	6.27	Rayonier Pulp	30:1	20	70-74	13.41	Good
88.20	7.33	4.31	Parlin CC Linters	30:1	20	22-32	13.20	Good
88.20	7.33	4.31	Parlin CC Linters	30:1	20	36-46	13.39	Good
88.20	7.33	4.31	Parlin CC Linters	30:1	20	46-58	13.49	Good
85.14	10.02	4.81	Hercules Linters	30:1	20	54-66	13.38	Good
85.14	10.02	4.81	Hercules Linters	30:1	20	60-70	13.40	Good
85.14	10.02	4.81	Hercules Linters	30:1	20	65-72	13.46	Good
71.70	14.10	14.04	Buckeye Linters	45:1	15	35-42	11.23	Bad
71.70	14.10	14.04	Buckeye Linters	45:1	15	43-51	11.48	Good
71.70	14.10	14.04	Buckeye Linters	45:1	15	44-53	11.57	Good

Table 7

Magnesium nitrate/water ratio versus percent nitrogen in nitrocellulose

HNO <sub>3</sub>	ACID MIX			Cellulose	NITRATION				AI
	Mg(NO <sub>3</sub> ) <sub>2</sub>	H <sub>2</sub> O	Mg(NO <sub>3</sub> ) <sub>2</sub> to H <sub>2</sub> O Ratio		Acid Mix to Cellulose Ratio	Time min	Temp °C	N <sub>2</sub> %	
<b>70% Nitric Acid</b>									
69.91	16.64	13.43	1.23	Linters	30:1	15	43-53	12.24	Bad
70.27	16.75	12.97	1.29	Linters	37 1/2:1	15	45-52	12.48	Good
70.09	17.12	12.79	1.34	Linters	37 1/2:1	15	44-53	12.60	Good
70.56	17.33	12.11	1.43	Linters	37 1/2:1	15	44-65	12.62	Good
70.52	17.49	11.98	1.46	Linters	45:1	15	47-56	12.72	Good
<b>75% Nitric Acid</b>									
74.10	14.13	11.77	1.26	Linters	45:1	15	45-54	12.75	Bad
75.73	14.02	10.20	1.37	Linters	37 1/2:1	15	46-60	13.04	Good
75.06	14.50	10.44	1.39	Linters	37 1/2:1	15	43-50	13.07	Good
75.38	14.69	9.93	1.48	Linters	37 1/2:1	15	49-61	13.22	Good
74.48	15.51	9.93	1.56	Linters	37 1/2:1	15	49-61	13.27	Good
74.55	15.64	9.78	1.60	Linters	37 1/2:1	15	47-61	13.34	Good
74.92	15.32	9.21	1.72	Linters	37 1/2:1	15	42-58	13.37	Good
75.02	16.50	8.40	1.96	Linters	37 1/2:1	15	50-65	13.50	Good
75.73	14.02	10.20	1.37	Pulp	37 1/2:1	15	49-59	13.11	Good
75.38	14.69	9.93	1.48	Pulp	37 1/2:1	15	49-59	13.21	Good
74.55	15.64	9.78	1.60	Pulp	37 1/2:1	15	46-61	13.35	Good
74.92	15.82	9.21	1.72	Pulp	37 1/2:1	15	42-58	13.42	Good
75.02	16.50	8.40	1.96	Pulp	37 1/2:1	15	50-61	13.47	Good

Table 7 (continued)

ACID MIX				NITRATION					
HNO <sub>3</sub>	Mg(NO <sub>3</sub> ) <sub>2</sub>	H <sub>2</sub> O	Mg(NO <sub>3</sub> ) <sub>2</sub> to H <sub>2</sub> O Ratio	Cellulose	Acid Mix to Cellulose Ratio	Time min	Temp °C	N <sub>2</sub> %	AI
<b>80% Nitric Acid</b>									
79.65	11.18	9.11	1.22	Linters	37 1/2:1	15	49-58	12.96	Good
80.48	11.31	8.21	1.38	Linters	37 1/2:1	15	49-61	13.15	Bad
80.38	11.83	7.79	1.51	Linters	37 1/2:1	15	49-64	13.36	Good
80.07	12.99	7.84	1.61	Linters	37 1/2:1	15	47-57	13.42	Good
80.54	12.35	7.11	1.74	Linters	37 1/2:1	15	47-63	13.50	Good
80.41	12.70	6.89	1.84	Linters	37 1/2:1	15	46-59	13.60	Good
80.21	12.88	6.91	1.90	Linters	37 1/2:1	15	48-61	13.61	Good
79.65	11.18	9.11	1.22	Pulp	37 1/2:1	15	49-58	13.04	Good
80.48	11.31	8.21	1.38	Pulp	37 1/2:1	15	48-58	13.18	Good
80.38	11.83	7.79	1.51	Pulp	37 1/2:1	15	48-63	13.28	Good
80.07	12.99	7.84	1.61	Pulp	37 1/2:1	15	45-61	13.26	Good
80.54	12.35	7.11	1.74	Pulp	37 1/2:1	15	48-64	13.47	Good
80.41	12.70	6.89	1.84	Pulp	37 1/2:1	15	47-60	13.59	Good
80.21	12.88	6.91	1.90	Pulp	37 1/2:1	15	48-61	13.60	Good



Table 8

Bench-scale parameters

<u>% HNO<sub>3</sub></u>	<u>% Mg(NO<sub>3</sub>)<sub>2</sub></u>	<u>% H<sub>2</sub>O</u>	<u>Acid/Cell Ratio</u>	<u>Time min</u>	<u>Grade</u>
+0.0	+0.5	+0.0	30-40/1	15 - 20	High
85.00	10.00	5.00			13.35 N <sub>2</sub> min
-1.0	-0.0	-0.5			
+0.0	+0.5	+0.0	30-40/1	15 - 20	Low
70.00	17.50	12.50			12.60 N <sub>2</sub> ± .15
-1.0	-0.0	-0.5			

## BENCH-SCALE EVALUATION

### Bench-Scale Nitration Studies

This phase of the program provided a scale-up capability from nitrating 20 grams of cellulose in the laboratory to the nitration of up to 0.23 kg (0.5 lb) of cellulose.

The bench-scale process flow for manufacturing NC by the magnesium nitrate method is presented in figure 6. Nitrating acid mix is depicted in the top left box with dotted and solid lines to illustrate the flexibility of either delivering mixed acid directly to an attrition mill or to an equilibrium chamber. The term equilibrium chamber was selected to emphasize that the use of an attrition mill was for initiating nitration and not for completing the nitration. The cellulose was shown going into the attrition mill with the nitrating acid or being introduced directly into the equilibrium chamber (nitrating pot) for batch type nitrations. A remotely controlled pneumatic valve was used to drop the nitrated mix from the equilibrium chamber into the centrifuge, which in turn wrung out the spent acid. Various combinations of acid and water washes were introduced into the centrifuge for prescribed periods to remove excess spent acid and minimize denitration of the NC. Purification of the NC included boiling and washing to stabilize the material and provide for viscosity adjustment.

Figure 7 shows the equipment control panel which was separated from the operating area by a reinforced glass partition to allow observation of the equipment while safely controlling various operating sequences, especially grinding the acid/NC slurry in the attrition mill and subsequently wringing out the spent acid.

Figure 8 is a photograph of the bench-scale equipment and shows the method by which cellulose and nitrating acid could be simultaneously introduced into an attrition mill to initiate the nitration reaction. A variable speed conveyor was loaded with a weighed quantity of cellulose, and a weighed quantity of preheated nitrating acid was pumped from a mix tank to a jacketed hold tank directly above the attrition mill. The cellulose and nitrating acid were discharged into the attrition mill where the slurry was ground and then dropped into a chute passing into the equilibrium chamber.

After the nitration was completed, a pneumatic control valve at the bottom of the equilibrium chamber was opened and the slurry was discharged into a centrifuge for wringing and washing of the NC.

During bench-scale testing a large number of nitrations were conducted where acid lines could not be drained satisfactorily between tests of various nitrating acid concentrations. It was ascertained that more reproducible results would be achieved by manually adding the nitrating acid into a nitrating pot in lieu of pumping acid from a weigh tank.

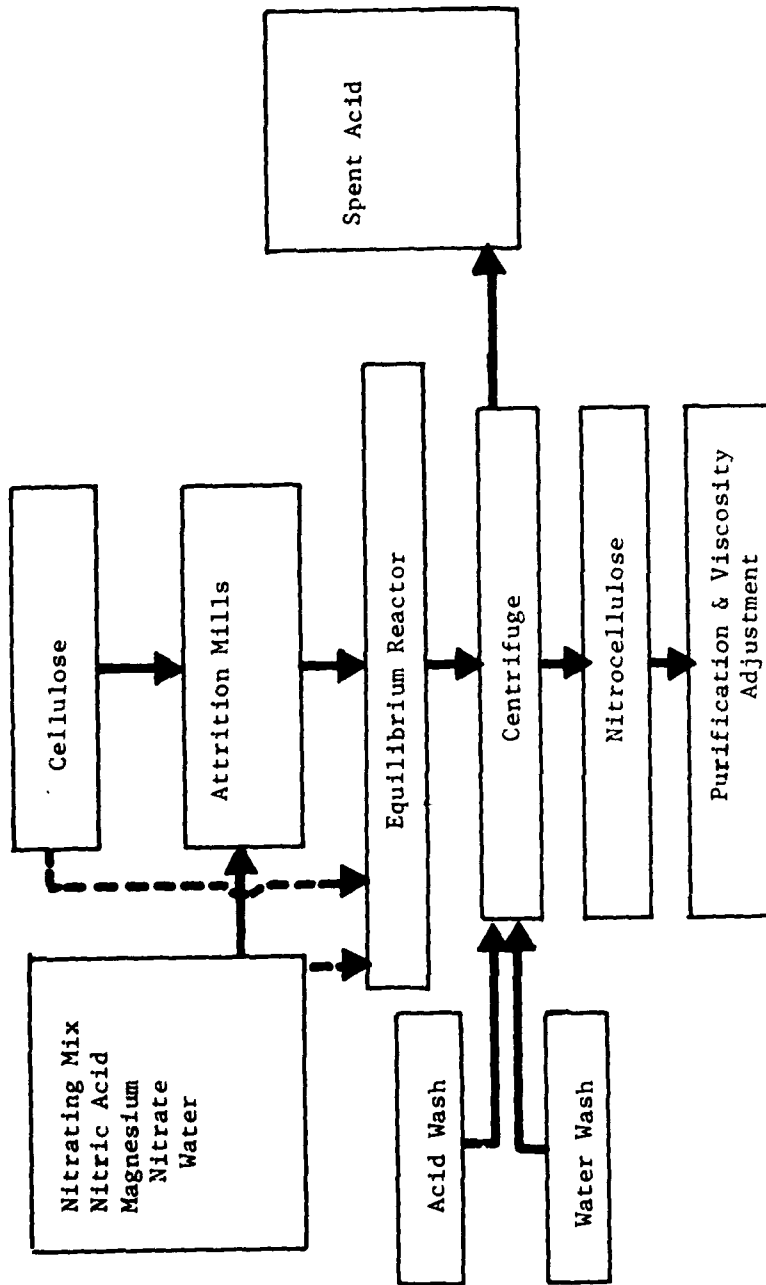


Figure 6. Bench-scale process flow.

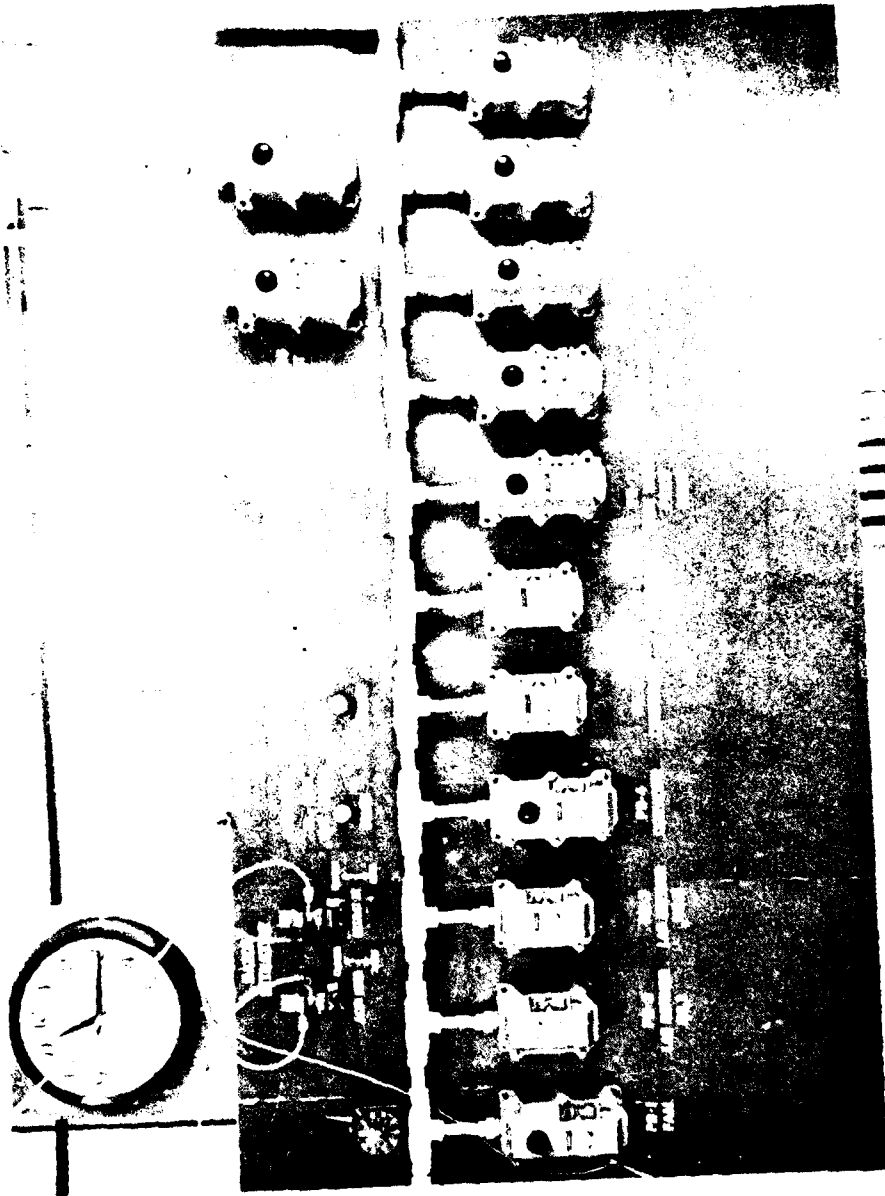


Figure 7. Control panel.

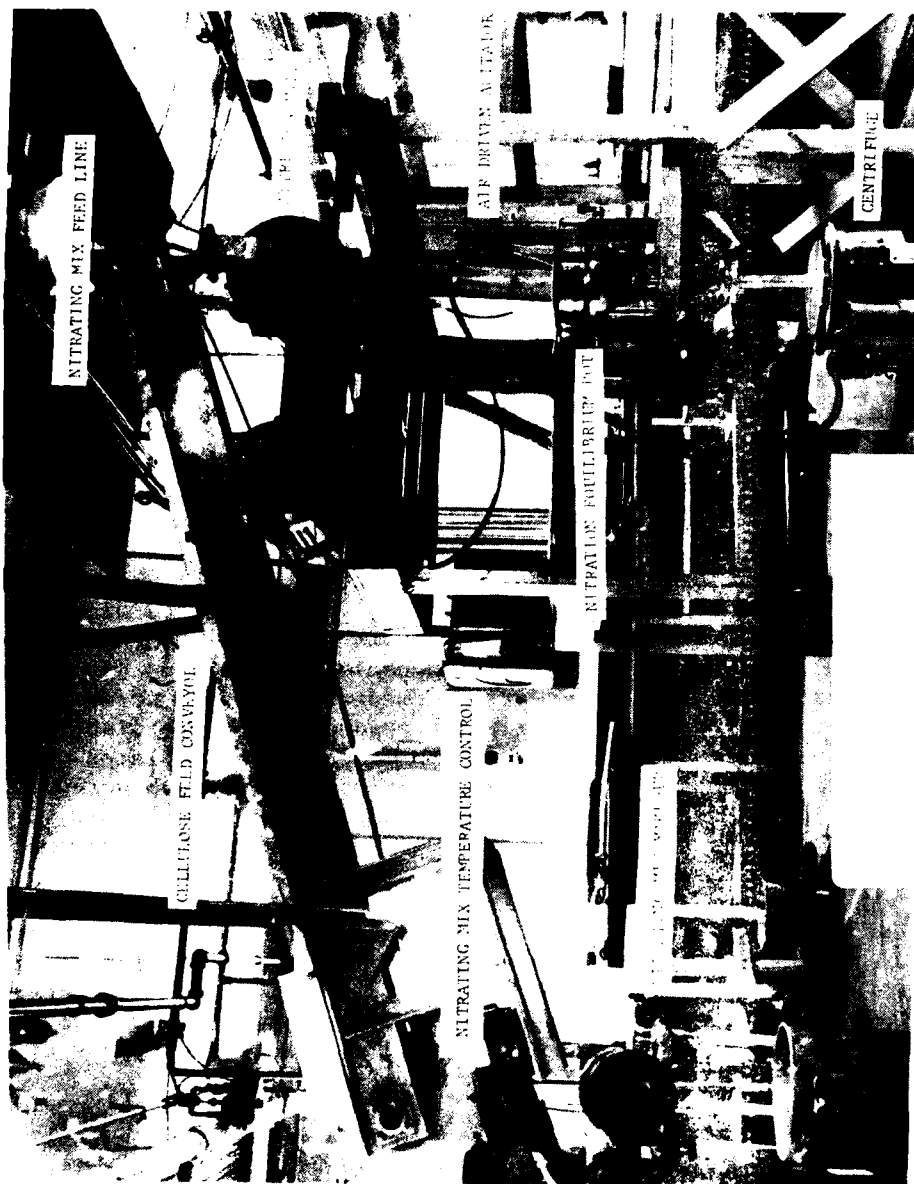


Figure 8. Magnesium nitrate bench-scale unit  
(only one attrition mill in use)

Figure 9 shows a nitrating pot, portable attrition mill and centrifuge in line with each other. This arrangement was used to evaluate the attrition mill in initiating the nitration reaction. Acid was weighed and poured into the nitrating pot and temperature conditioned. Then a weighed amount of cellulose was hand-fed into the mixed acid which was agitated. The slurry was discharged into the attrition mill and then directed to a centrifugal pump for circulation back to the nitrating pot. The grinding time of the attrition mill cycle was arranged to duplicate the conditions that would exist when passing through a series of several attrition mills. When the attrition mill cycle time had elapsed, a pneumatic control valve was closed to collect the nitrating slurry in the nitrating pot to complete the equilibrium reaction. Then the line from the attrition mill was redirected to the centrifuge for wringing and washing of the NC after reaction was completed.

A Sprout-Waldron 300 mm (12 in.) Laboratory Refiner (attrition mill) was evaluated in the bench-scale effort. All wetted parts, except the plates, were of 304 stainless steel where cast and 304L were welded. The shaft seal consisted of a single ring of Garlock 5861 asbestos braided Teflon filled packing. The mill was furnished with a 11.2 kW (15 hp), Class I-D, and Class II-G, 440 volt, 60 Hz, 3 Phase, 1800 rev/min motor. The "V" belt drive provided a mill speed of 1800 rev/min. The packing seal was separated from the bearing support to prevent NC from entering the bearings. The proper rotation of the refiner was clockwise when facing the feed end.

Normal operation of the refiner consisted of starting the machine with the plates not in contact with each other, then slowly turning the handwheel adjustment inward until the plates contacted as evidenced by a low rubbing sound. Without moving the handwheel, an indicator disc was turned until "zero" was directly under the pointer. A thumb screw was tightened to lock the indicator disc. Each graduation of the disc indicated one-thousandth inch movement of the rotor plate. The plates were set to desired clearance and locked into position with a thumb screw.

The single runner attrition mill passed material between two discs, one rotating and one stationary.

The acid/NC slurry was fed through the center of the stationary disc. Two plate designs were tested in evaluating the mill. The first style discs shown in figure 10 (12716, Style A) were cast of 440-C stainless steel and had staggered periphery dams which raised the NC into the cutting edge. This pattern with dams required a pick to clean many of the pockets free of NC between runs of different grades of NC.

The second type of plate design, C-2976A, shown in figure 11 had an open periphery (no dams) and was cast from Ni-Hard steel. Both types of plate are cast in segments, three sections to each side and are secured to each side by six bolts. The cutting action is similar for each style;

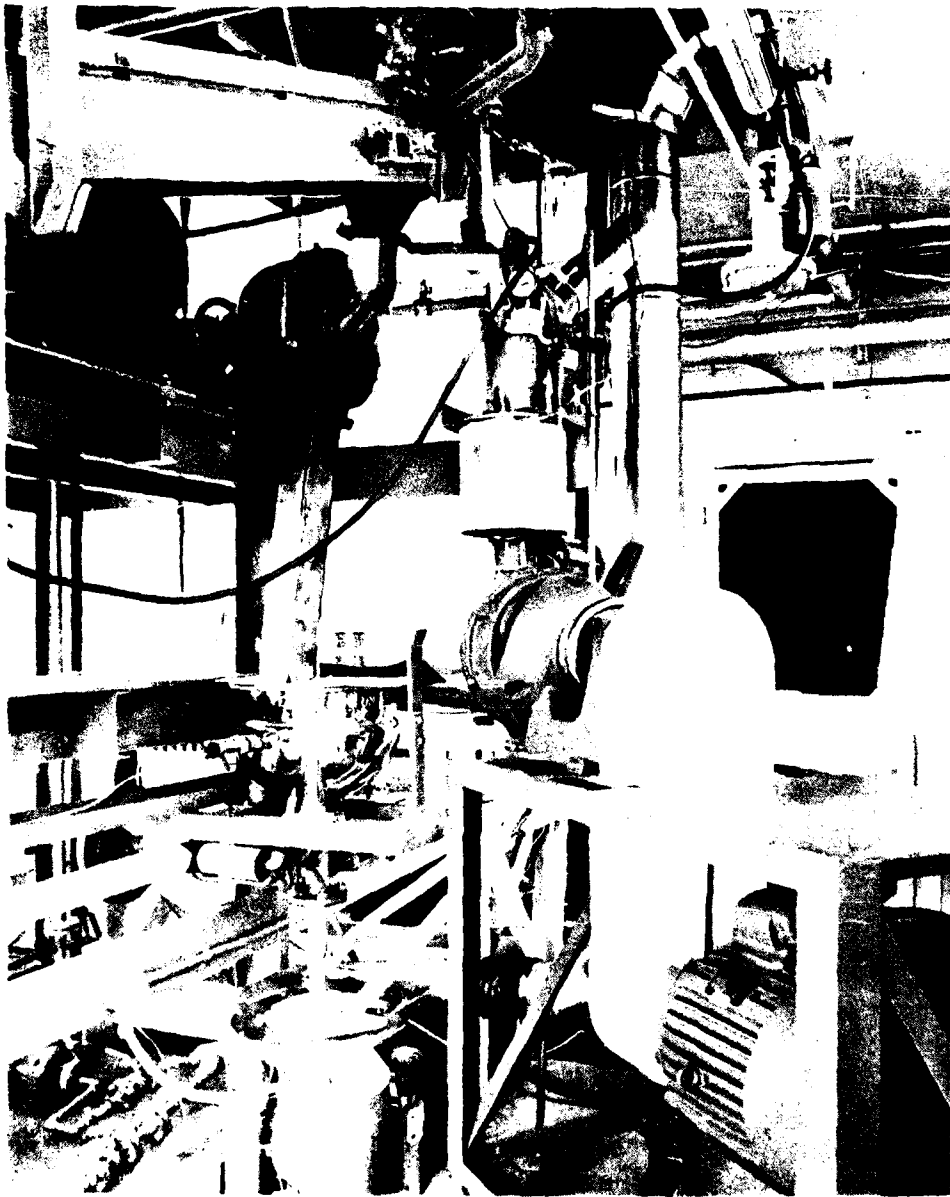


Figure 9. Nitrating pot, attrition mill, centrifuge.

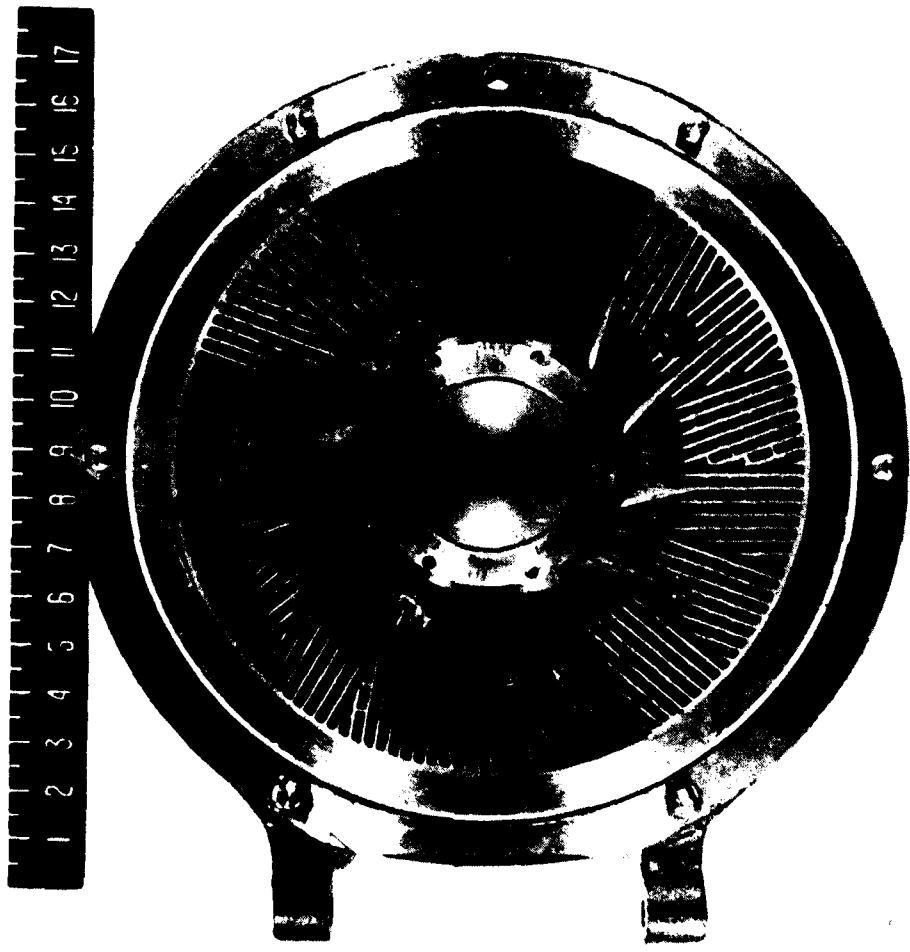


Figure 10. Sprout-Waldron attrition mill plate series 12716-A



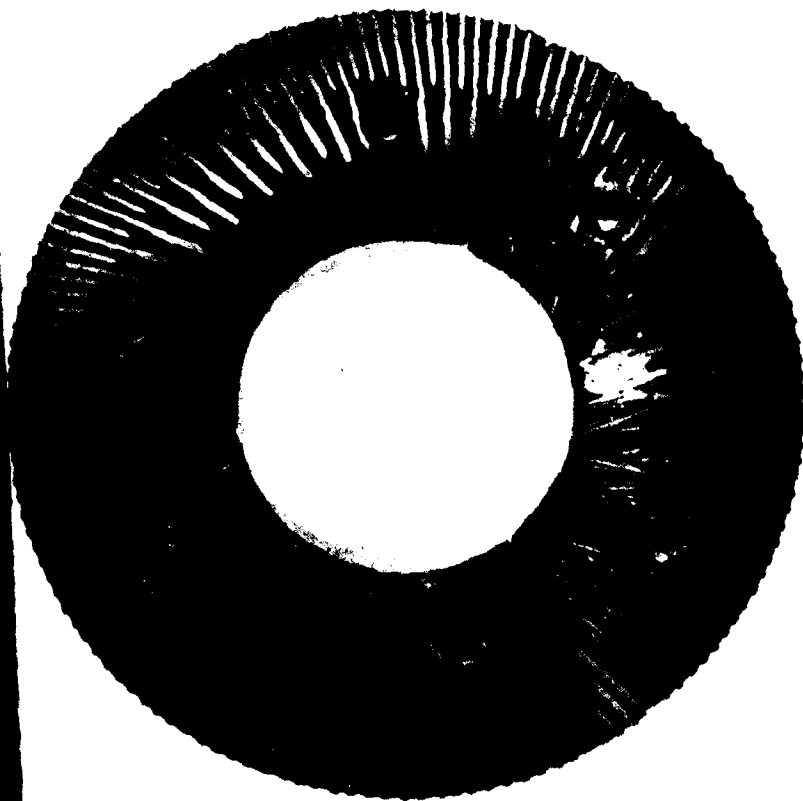


Figure 11. Sprout-Waldron attrition mill plate series C-2976A.

however, the pattern without dams facilitates cleaning after each run. A liquid wash with the mill operating is normally sufficient to clean the plates.

A Tolhurst 300 mm (12 in.) center-slung (1B15) centrifuge was used to wring out the acid-wet NC and to remove retained nitrating acid by washing. All wetted parts were 316 stainless steel and the unit had a removable basket. The unit was provided with a Class I, Group D motor, 0.56 kW (0.75 hp), 440 V, 3 Phase, 60 Hz, and 3450 rev/min variable speed "V" belt drive. Normal operation was at 1800 rev/min.

Twenty bench-scale nitrations using conventional cotton linters were run in the evaluation of attrition mills to initiate nitration, and the results are presented in table 9. Ten runs were conducted by circulating the acid/cellulose slurry through one attrition mill to simulate passes through several mills in series, and ten runs were made as straight pot nitrations for comparison. Duration of the circulating cycle through the mills was 30 seconds for each run, followed by a hold time of 20 minutes in the pot nitrator. Clearance of the attrition mill plates was set at 0.13 mm (0.005 in.). The pot nitrations were all of 20-minute duration to reach equilibrium and provide comparison data for the attrition mill tests.\* Temperature, acid/cellulose ratio, and nitrating mix concentration were held constant during the twenty runs. Acid analysis was maintained at 85 percent nitric, 10 percent magnesium nitrate, and 5 percent water. Temperature and acid/cellulose ratio were held at 60°C and 35 to 1, respectively. The average nitrogen level of the NC produced was 13.58 percent for the attrition mill tests and 13.56 percent for the pot nitrations with ranges of 13.44 to 13.67 percent and 13.43 to 13.82 percent, respectively. There was no apparent difference in the degree of NC harshness between the two sets of tests. However, all of the NC produced by initiating nitration in the attrition mill had a grayish coloration, whereas that from pot nitrations was white. This coloration reverted to a reddish brown during the subsequent viscosity boils, indicating corrosion and/or erosion of the attrition mill plates. Also, the initial viscosity of the NC circulated in the attrition mill was in the range of 300 to 550 seconds, with that of the pot nitrations being 67 to 128 seconds. The higher viscosities were probably associated with the heat sink in the attrition mill or short runs which reduced the nitrating temperature and increased the viscosities of the NC.

Further evaluation of the attrition mill was conducted during the nitration tests of 6.4 mm (0.25 in.) square pieces (diced cut) of sheeted wood pulp. Table 10 compares pot nitrated pulp that was ground in an attrition mill before wringing and washing with diced cut material

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\*Initial bench-scale tests showed that nitrations in general had not reached equilibrium in 10 minutes; however, after 20-minute nitrations no further increase in nitrogen level was apparent. For this reason, bench-scale testing was set at 20 minutes.

that was treated similarly but without grinding to smaller particle size to facilitate the removal of spent acid. The unground larger chips retained sufficient spent acid after the wringing sequence to slow down the effect of subsequent wash water treatments and introduce significant denitration.

The unground NC averaged less than 12.70 percent nitrogen, whereas the ground material was all high grade and averaged 13.55 percent nitrogen. Also, the texture of the diced cut nitrated material, although tough on the surface, produced soft material which was comparable to conventional NC. High grade lint nitrations were conducted and results are presented in table 11. The nitrating acid composition was similar to that used for high grade pulp. In this case, high grade lint was achieved without the use of an attrition mill.

Table 9

Attrition mill versus pot nitrations

<u>Test No.</u>	<u>Lint</u>	
	<u>Attrition Mill - NC</u> <u>Percent Nitrogen</u>	<u>Pot - NC</u> <u>Percent Nitrogen</u>
1	13.58	13.67
2	13.62	13.43
3	13.62	13.51
4	13.67	13.38
5	13.66	13.50
6	13.59	13.57
7	13.53	13.61
8	13.50	13.82
9	13.44	13.82
10	13.60	13.48
$\bar{X}$	13.58	13.56
Range	13.44 - 13.67	13.43 - 13.82

Table 10

Denitration of unground nitrated chip versus ground chip

Unground Dice Cut Pulp

Run No.	Type Cellulose	Nitration		AI Appearance	N <sub>2</sub> %	Acid-Wet NC Treatment	HNO <sub>3</sub> %	Acid/Mix	
		Time, min	Temp °C					Mg(NO <sub>3</sub> ) <sub>2</sub> %	H <sub>2</sub> O %
<u>Unground High Grade Nitrocellulose</u>									
573	Dice Cut Pulp	20	52-56	Bad	12.75	Large centrifuge - chilled H <sub>2</sub> O wash - ground cold H <sub>2</sub> O	83.26	10.78	5.96
574	Dice Cut Pulp	20	54-59	Bad	13.02	Large centrifuge - chilled H <sub>2</sub> O wash - ground cold H <sub>2</sub> O	85.81	9.58	4.61
575	Dice Cut Pulp	20	52-59	Bad	13.09	Large centrifuge - chilled H <sub>2</sub> O wash - ground cold H <sub>2</sub> O	85.81	9.58	4.61
576	Dice Cut Pulp	10	54-57	Bad	12.02	Small centrifuge - chilled H <sub>2</sub> O wash - ground in soda ash	85.09	9.89	5.02
578A	Dice Cut Pulp	20	57-61	Good	12.52	Large centrifuge - chilled H <sub>2</sub> O wash - samples separated - ground in cold H <sub>2</sub> O	85.04	9.89	5.06

Attrition Mill Grinding High Grade Nitrocellulose

580	Dice Cut Pulp	20	55-46	Good	13.37	Attrition mill setting 0.13 mm (0.005 in.) used in nitration - centrifuged cold H <sub>2</sub> O wash - ground cold H <sub>2</sub> O	85.82	8.83	5.34
581	Dice Cut Pulp	20	43-55	Good	13.41	Attrition mill setting 0.02 mm (0.001 in.) used in nitration - centrifuged cold H <sub>2</sub> O wash - ground cold H <sub>2</sub> O	83.68	10.12	6.20

Table 10 (continued)

Run No.	Type Cellulose	Nitration		AI Appearance	N <sub>2</sub> %	Acid-Wet NC Treatment	Acid/Mix		
		Time, min	Temp °C				HNO <sub>3</sub> %	Mg(NO <sub>3</sub> ) <sub>2</sub> %	H <sub>2</sub> O %
586	Dice Cut Pulp	20	57-61	Good	13.46	Large centrifuge - ground in attrition mill [0.13 mm (0.005 in.) setting] chilled H <sub>2</sub> O wash - ground cold H <sub>2</sub> O	85.13	9.71	5.10
584	Dice Cut Pulp	20	55-64	Good	13.45	Attrition mill setting 0.04 mm (0.0015 in.) at end of nitration - centri- fuged - cold H <sub>2</sub> O wash - ground cold H <sub>2</sub> O	84.95	9.36	5.65
585	Dice Cut Pulp	20	54-64	Good	13.51	Attrition mill setting 0.3 mm (0.010 in.) at end of nitration - centrifuged - cold H <sub>2</sub> O wash - ground cold H <sub>2</sub> O	84.95	9.36	5.65

Table 11

## High grade lint bench-scale nitrations

Run No.	Type Cellulose	Nitration		Acetone Insol.	N %	Acid-Wet NC Treatment	Acid/Mix	
		Acid/Cell Ratio	Time, Temp, min °C				HNO <sub>3</sub> %	Mg(NO <sub>3</sub> ) <sub>2</sub> H <sub>2</sub> O %
547	Buckeye Linters	40:1	20 61-66	Good	13.52	70% HNO <sub>3</sub> , -18°C (0°F), plus cold water wash, ground in cold H <sub>2</sub> O	84.61	10.31 5.08
548	Buckeye Linters	40:1	20 70-72	Good	13.46	70% HNO <sub>3</sub> , 0°C (32°F), plus cold water wash, ground in cold H <sub>2</sub> O	85.09	10.18 4.73
549	Buckeye Linters	40:1	20 58-62	Good	13.68	70% HNO <sub>3</sub> , -18°C (0°F), plus cold water wash, ground in cold H <sub>2</sub> O	85.34	9.88 4.74
550	Buckeye Linters	40:1	20 60-65	Good	13.76	Water, 1°C (38°F), plus cold water wash, ground in cold H <sub>2</sub> O	85.22	10.25 4.53
551	Buckeye Linters	40:1	20 60-65	Good	13.59	Water, 1°C (38°F), plus cold water wash, ground in cold H <sub>2</sub> O	85.22	10.25 4.53

Tables 12 and 13 present the acid mixes and conditions that produced satisfactory low grade lint and pulp, respectively, from conventional lint or pulp types of cellulose.

The spent acids from bench-scale tests increased approximately 1.4 percent in water and 0.6 percent in magnesium nitrate, with a decrease of 2.0 percent in nitric acid concentration. The comparison of high and low grade spent acids is presented in tables 14 and 15. Five samples of spent acid were taken directly from the nitrating pot to prevent contamination from acid lines and tanks.

Nitrocellulose retention of spent nitrating acids was determined by weighing NC after the bulk of nitrating acid was wrung out of it but prior to the NC receiving any water or acid wash treatments. The difference between the dried and acid wet weights was reported in pounds of retained acid per pound of NC. These results are in table 16.

Retention of magnesium nitrate in NC was also investigated, and tests showed an average retention of 287 parts per million with a range of 53 to 690 ppm. The results are in table 17.

Two significant observations of magnesium nitrate/nitric acid manufactured NC were: (1) that it does not ignite when foreign organic material is placed on acid-wet NC; (2) it does not disintegrate on several hours of standing without agitation in the salt/acid mix.

In summary, high and low grade bench-scale nitrations have resulted in establishing nominal acid mix compositions for pilot plant operations. The nominal conditions for preparing high grade NC will consist of 79 percent nitric acid, 14 percent magnesium nitrate and 7 percent water with the ratio of salt to water being 2 to 1. The nitrating temperature will be a nominal 65°C, and the cellulose to acid ratio will be 35 to 1. Low grade nitrations will be optimized from nominal 70 percent nitric acid, 18 percent magnesium nitrate and 12 percent water with a salt to water ratio of 1.5 to 1. A 55°C nitrating temperature and 35 to 1 acid to cellulose ratio will be the nominal operating parameters.

#### Viscosity Adjustment

##### Equipment

To initiate viscosity studies, laboratory equipment was installed to simulate actual production boiling tub processing conditions (fig. 12). This equipment consists of Glas-Col electrical heating units with voltage regulator controls, 5,000 ml flasks, water cooled condensers, and automatic electrical timers. NC samples used for these viscosity studies were obtained from the bench-scale nitrating unit.

Table 12

## Low grade lint bench-scale nitrations

Run No.	Type Cellulose	Acid/ Cellulose Ratio	Nitration		Acetone Insol.	N %	Acid-Wet NC Treatment	Acid/Mix		
			Time, min	Temp, °C				HNO <sub>3</sub> %	Mg(NO <sub>3</sub> ) <sub>2</sub> H <sub>2</sub> O %	
554	Buckeye Linters	40:1	20	51-54	Good	12.67	Cold water wash, ground in cold H <sub>2</sub> O	70.18	17.70	12.10
556	Buckeye Linters	40:1	20	52-56	Good	12.60	Cold water wash, ground in cold H <sub>2</sub> O	74.94	14.74	10.30
557	Buckeye Linters	40:1	20	60-67	Good	12.62	Cold water wash, ground in cold H <sub>2</sub> O	70.52	17.49	11.99
563	Buckeye Linters	40:1	20	50-60	Good	12.50	Cold water wash, ground in cold H <sub>2</sub> O	71.06	17.44	12.46
564	Buckeye Linters	40:1	20	52-56	Good	12.51	Cold water wash, ground in cold H <sub>2</sub> O	70.01	17.33	12.66



Table 13

## Low grade pulp bench-scale nitrations

Run No.	Type Cellulose	Nitration		AI Appearance	N <sub>2</sub> %	Acid-Wet NC Treatment	Acid/Mix		
		Time, min	Temp, °C				HNO <sub>3</sub> %	Mg(NO <sub>3</sub> ) <sub>2</sub> %	H <sub>2</sub> O %
566	Shredded Pulp	20	54-59	Good	12.58	Chilled H <sub>2</sub> O wash - ground cold H <sub>2</sub> O	75.50	14.36	10.11
570	Shredded Pulp	20	50-56	Good	12.62	Chilled H <sub>2</sub> O wash - ground cold H <sub>2</sub> O	75.46	13.81	10.73
614	Shredded Pulp	20	53-56	Good	12.52	Chilled H <sub>2</sub> O wash - ground cold H <sub>2</sub> O	74.77	14.81	10.42
615	Shredded Pulp	20	53-59	Good	12.65	Chilled H <sub>2</sub> O wash - ground cold H <sub>2</sub> O	74.98	14.38	10.60
616	Shredded Pulp	20	52-56	Good	12.69	Chilled H <sub>2</sub> O wash - ground cold H <sub>2</sub> O	74.98	14.38	10.60

Table 14

## Low grade acid comparisons

	<u>Nitrating Acid, %</u>			<u>Spent Acid, %</u>		
	<u>Nitric</u>	<u>Magnesium Nitrate</u>	<u>Water</u>	<u>Nitric</u>	<u>Magnesium Nitrate</u>	<u>Water</u>
	68.60	17.60	13.80	66.61	18.31	15.08
	74.98	14.38	10.60	73.12	14.40	12.48
	74.98	14.38	10.60	73.10	14.49	12.39
	70.04	17.25	12.71	68.33	18.02	13.63
	75.24	14.78	9.98	72.83	16.17	10.98
Avg	72.76	15.68	11.54	70.80	16.28	12.91
Change				- 1.96	+ .60	+ 1.37

Table 15

## High grade acid comparisons

	<u>Nitrating Acid, %</u>			<u>Spent Acid, %</u>		
	<u>Nitric</u>	<u>Magnesium Nitrate</u>	<u>Water</u>	<u>Nitric</u>	<u>Magnesium Nitrate</u>	<u>Water</u>
	85.13	9.77	5.07	83.26	10.42	6.30
	84.00	10.99	4.99	82.64	10.67	6.65
	87.70	7.82	4.48	85.49	8.11	6.40
	85.70	9.59	4.71	83.72	10.00	6.28
	84.13	9.03	6.78	82.11	9.67	8.22
Avg	85.37	9.44	5.21	83.44	9.78	6.77
Change				- 1.93	+ .34	+ 1.56

Table 16

Nitrocellulose retention\*of spent acid  
(lb acid/lb NC)

<u>Sample No.</u>	<u>Lint</u>	<u>Pulp</u>
1	1.69	1.63
2	1.69	1.61
3	1.95	1.52
4	1.70	1.69
5	1.74	1.70
Average	1.75	1.63

\*After nitration

Table 17

Retention\*of magnesium nitrate in nitrocellulose

<u>Sample No.</u>	<u>Amount Retained, ppm</u>
1	690
2	350
3	53
4	143
5	200
Average	287

\*After nitration

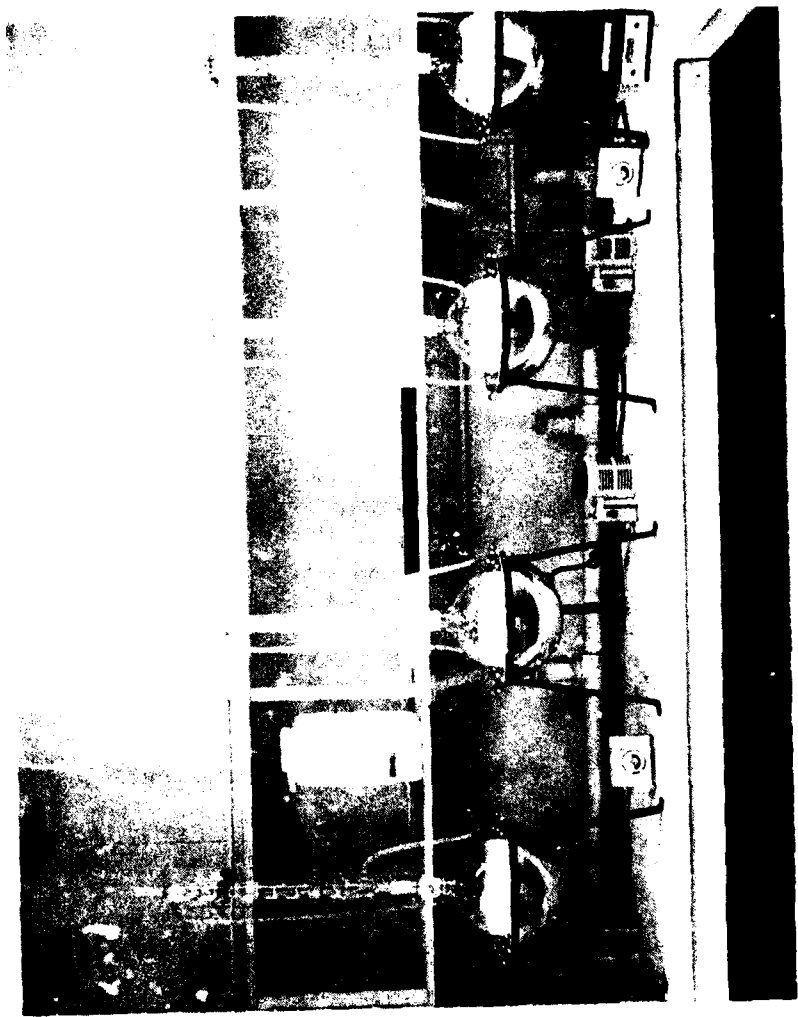


Figure 12. Laboratory equipment for viscosity studies.

Viscosity Boiling Curves

a. High Grade Nitrocellulose

Initial viscosity boils were performed on six samples of high grade type magnesium nitrate NC to correlate the effect of acid viscosity boiling on this material with that of normal production NC.

Two samples were boiled eight hours, two 16 hours and two 24 hours. The data indicated that the material would be near the present NC requirement for viscosity after approximately 24 hours boiling time.

It was decided that in order to establish a valid viscosity curve, a series of high grade type samples would be boiled in increments of eight hours over a 40-hour range. The data generated are shown in table 18 below.

Table 18

High grade nitrocellulose effect of acid boil time on viscosity (seconds)

Sample No.	Boiling Time, hours					
	0	8	16	24	32	40
1	127	70	18	16	8	5
2	66	34	33	10	6	4
3	74	11	4	2	1	2
4	144	51	28	18	10	8
5	134	55	28	17	12	7
Average	108	44	22	10	7	5

The average viscosity curve established from the data is shown in figure 13.

Three of these samples were selected for further purification treatment after 40 hours boiling time. This treatment included a four-hour soda boil, a two-hour neutral boil, and a one-hour neutral boil with three hot water washes after each boil. The laboratory analyses of these three samples yielded German test values of 20, 20, and 23 minutes. These values were in the range expected but did not meet the MIL-N-244A

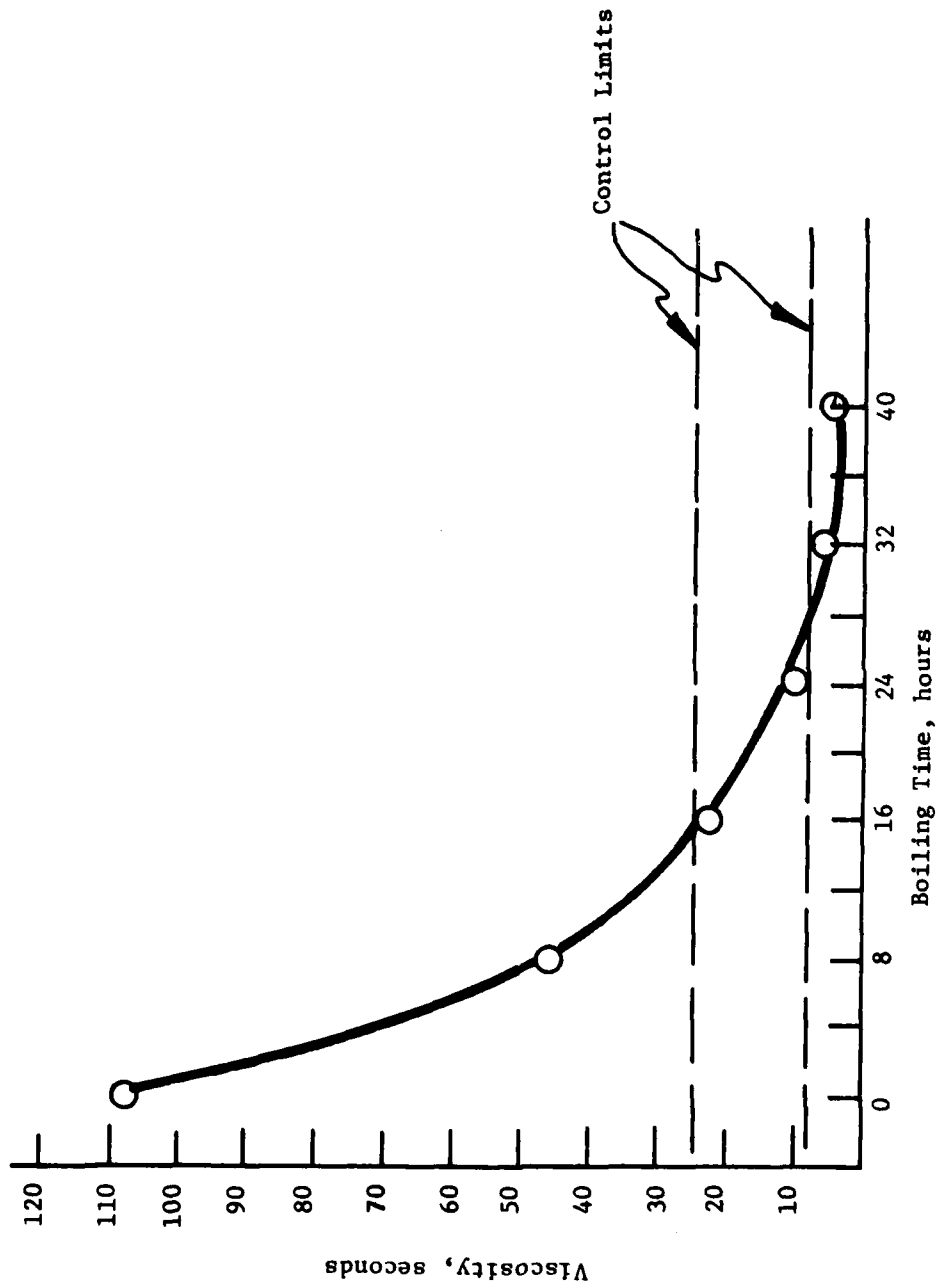


Figure 13. Effect of acid boil time on viscosity - high grade nitrocellulose.

specification requirement of 26 minutes minimum. The KI\* values were all 45+ minutes and were acceptable as specified in MIL-N-244A. To verify the data obtained on high grade NC other samples were processed using a viscosity boiling time of 24 hours and a purification treatment as outlined previously. The results of the laboratory analyses were similar to the data obtained on the material from which the high grade viscosity boiling curve was generated.

b. Low Grade Nitrocellulose

The first samples of low grade NC produced by the bench-scale nitrating process were small in volume. Because of this it was decided to boil three samples 24 hours, which was near the optimum time established for high grade NC, to indicate the effect of boiling on viscosity of low grade NC. The samples were analyzed in the laboratory. Following are the results:

Viscosity, seconds	3	2	3
German, minutes	23	23	23
KI, minutes	45+	45+	45+

These data showed that a boiling time of less than 24 hours was needed for low grade NC since the viscosities were very low. The German test values were in the range to be expected, and the KI values met MIL-N-244A specification requirements.

Based on the above data, four low grade NC samples were selected and boiled in increments of four hours over a 16-hour period. The data generated are shown in table 19 below.

Table 19

Low grade nitrocellulose - effect of acid boil time on viscosity (seconds)

Sample No.	Boiling Time, hours				
	0	4	8	12	16
1	105	44	28	16	13
2	61	32	22	21	9
3	83	45	32	21	12
4	188	90	70	35	18
Average	109	52	38	23	13

\*The KI test is the 65.5°C heat test (with potassium iodide, KI, starch paper) conducted as specified by MIL-N-244A.

The average viscosity versus boiling time curve established from the data is shown in figure 14.

After the low grade samples had completed 16 hours of boiling, they were given further purification treatment. It was decided to eliminate the four-hour soda boil from the treatment and give the samples a two-hour neutral boil, a one-hour neutral boil with two hot water washes after each boil plus two cold water washes after the last neutral boil. The samples were analyzed with the following results:

	Sample No.			
	1	2	3	4
Viscosity, seconds	13	9	12	18
German, minutes	25	18	19	19
KI, minutes	45+	45+	45+	45+

These data indicate that the four-hour soda boil can be eliminated since the German and KI values were comparable with the data obtained using the four-hour soda boil. To verify the data obtained on low grade NC, other samples were processed using a viscosity boiling time of 12 hours and a purification treatment as outlined previously. The results of the laboratory analyses were similar to the data obtained on the material from which the low grade viscosity boiling curve was generated.

#### c. Blended Nitrocellulose

Various combinations of viscosity boiling and purification treatments were pursued in an effort to optimize a minimum magnesium nitrate NC purification treatment procedure. After a theoretical treatment procedure was selected, 14 bench-scale samples were blended to allow a working volume of material that was uniform. Although the nitrogen content varied widely this material was all of a high grade type. Two samples (A and B) of this material were processed, at different times, in order to verify the data. Viscosity boils were conducted at 8, 16, 24 and 32 hours. Sample A, after the 32-hour viscosity boil, was treated further by giving a one-hour neutral boil, and three cold washes, using tap water. Sample B was treated further by giving the 24-hour and 32-hour viscosity boiled samples a one-hour neutral boil and three cold water washes using tap water. To show the need for a neutral boil after the viscosity boil, the 24-hour viscosity boiled material was filtered and washed with cold tap water until methyl red indicated the wash water to be above a pH of 6.2. A sample of the NC was analyzed and showed a KI of 35 minutes which is the minimum acceptable time specified in MIL-N-244A. The sample was then given a one-hour neutral boil and filtered. The filtered water was checked with methyl red and indicated the water to be acid. This showed that acid particles were entrapped in the NC fibers and were released



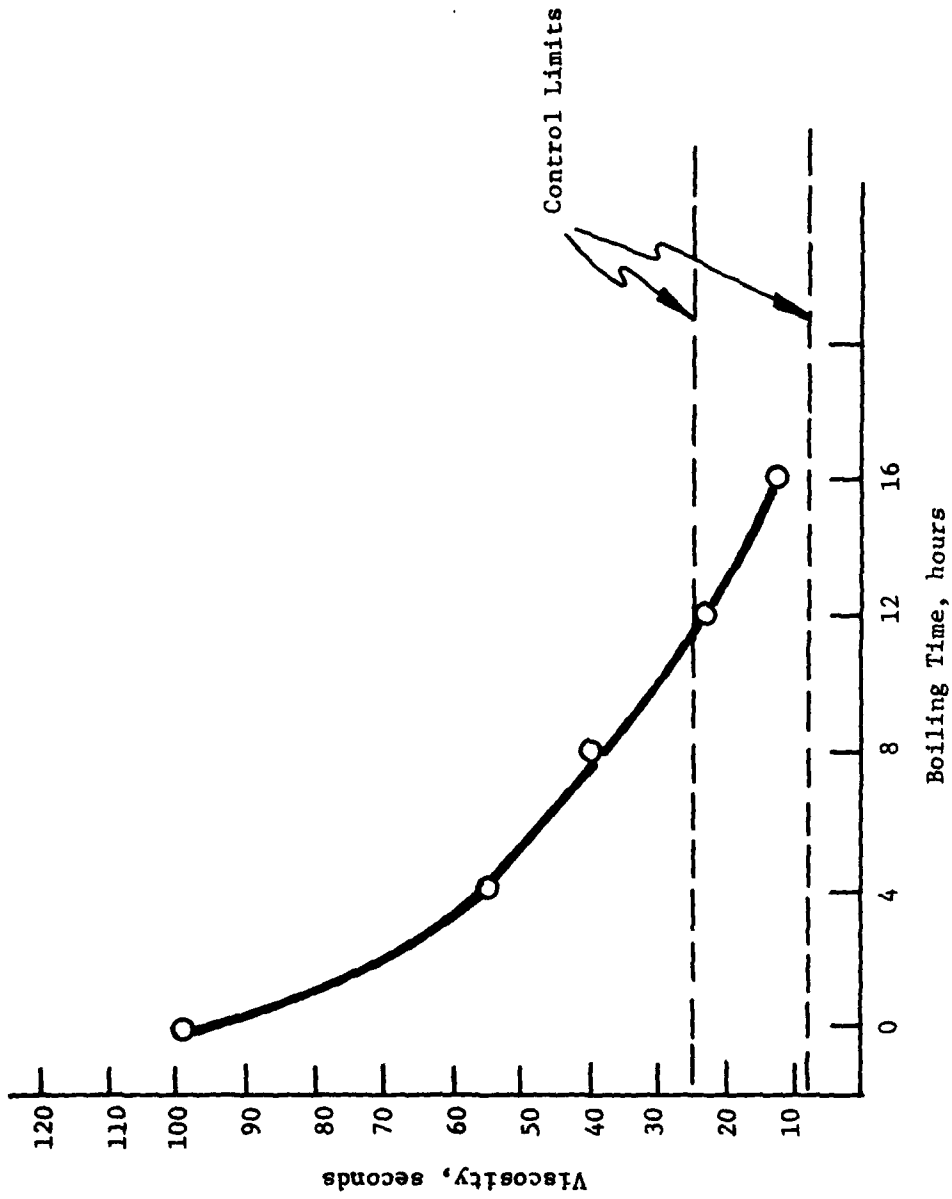


Figure 14. Effect of acid boil time on viscosity - low grade nitrocellulose.

during the neutral boil. The material was then washed with cold tap water until methyl red indicated the water was above a pH of 6.2. Laboratory analyses of a sample of this material gave a KI value of 45+ minutes which met the MIL-N-244A specification requirement. The data from these tests verify the viscosity versus boiling time curve shown in figure 13.

#### Viscosity Adjustment Summary

A summary of work done on viscosity adjustment to this point showed that the high grade type magnesium nitrate NC could be reduced to an acceptable viscosity based on NC specification MIL-N-244A by boiling 16 to 24 hours in a nitric acid solution of approximately 0.5 percent, provided the initial viscosity was between 60 and 140 seconds.

Also shown was that low grade type magnesium nitrate NC could be reduced to an acceptable viscosity by boiling 12 to 16 hours in a nitric acid solution of approximately 0.5 percent, provided the initial viscosity was between 60 and 160 seconds.

Both high and low grade magnesium nitrate NC given an acid boil and neutral boils with tap water washes after each boil would produce NC that met the requirements of NC specification MIL-N-244A except for the 134.5°C German stability test.

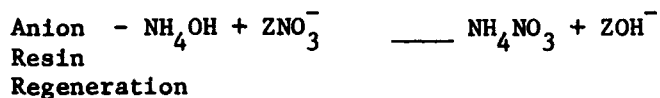
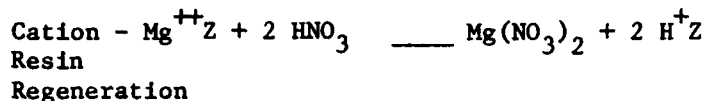
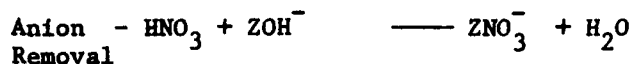
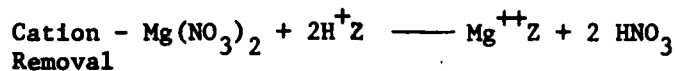
#### Ion Exchange Studies

##### Principle of Operation

The ion exchange system investigated in this study consisted of a two-bed, regenerative-type system for removing and reclaiming low concentrations of nitric acid and magnesium nitrate from wash water from the purification system.

The operation of the ion exchange system consisted of four major steps. These were exhaustion, backwashing, regeneration, and rinsing. In the exhaustion step the ions which were to be reclaimed were removed from the process stream by an appropriate anion or cation resin. When the supply of ions desired for exchange was almost fully depleted from the ion exchange resin, the resin was said to be exhausted. In the backwashing step, water was passed upflow through the ion exchange resin to remove foreign material from the resin and to reduce compaction of the resin by loosening and re-classifying the resin particles. This step assured maximum operating efficiency during the subsequent regeneration and exhaustion steps. In the regeneration step, the ions which were removed from the process stream by the resin were displaced from the ion exchange resin by passing through the resin a fairly concentrated solution of the ion which was desired to be on the resin. In the rinsing step, water was passed through the resin to flush out excess regenerant present within the interstices of the resin particles prior to placing the system back in the exhaustion cycle.

A typical ion exchange removal and regeneration reaction was as follows:



Concentration of the regenerants,  $\text{NH}_4\text{OH}$  and  $\text{HNO}_3$  were selected to give the required exchange capacities to each resin. The concentrations of the ammonium nitrate and nitric acid solutions obtained in the regeneration reactions were approximately the same as the concentrations of the regenerant chemicals that contact the resins. Rinsing the excess regenerant chemicals out of the resins diluted the ammonium nitrate and magnesium nitrate to some concentration lower than those of the regenerant chemicals. The resins investigated in this study could be regenerated stoichiometrically, but more reproducible results were obtained when over 100 percent of the quantity of regenerants chemically equivalent to the operating capacities of the resins were utilized.

#### Selection of Resins

Reports in the literature and prior work indicated that there were several ion exchange resins suitable for the removal of nitrates and magnesium from process solutions. Vendors were contacted for recommendations as to what combinations of resins to use for the removal of a nitric acid solution containing magnesium nitrate. The following four resins were then selected for use in this evaluation:

##### a. Cation Resins

- (1) Amberlite 200 - Rohm and Haas Company
- (2) Amberlite IR-120 Plus - Rohm and Haas Company

##### b. Anion Resins

- (1) Amberlite IRA-47 - Rohm and Haas Company
- (2) Duolite A-102D - Diamond Shamrock Chemical Company

#### Ion Exchange Unit

A Barnstead Two-Bed Demineralizer (Catalog No. D0823), rated capacity of  $1.054 \times 10^{-4} \text{ m}^3/\text{s}$  (100 gal/h), was used in the bench-scale phase of

this project for ion exchange studies (fig. 15). One 150 mm (6 in.) diameter Lucite column containing approximately  $1.7 \times 10^{-2} \text{ m}^3$  ( $0.6 \text{ ft}^3$ ) of resin, can be seen behind each of the two regenerant tanks. Control knobs for the proper flow of solutions through the resin columns and instruments for observing certain characteristics of the solutions as they flow through the system may be seen on the control console. A small demineralizer (Dow Model LD2A) may be seen in upper left corner of figure 15. This unit was used to make demineralized water for rinsing the resin columns and for make-up of simulated feed solutions for evaluation.

#### Ion Exchange System Storage Tanks

The stainless steel storage tanks used with the Barnstead unit to complete the ion exchange system are depicted in figure 16. The tank shown on the left has a  $0.39 \text{ m}^3$  (100 gal) capacity and is used as a feed tank. The tank on the right is a  $1.25 \text{ m}^3$  (330 gal) storage tank for demineralized water. This tank is equipped with a "Ventgard" to prevent carbon dioxide and organic matter from entering the storage tank from the atmosphere. Two tanks of  $0.08 \text{ m}^3$  (22 gal) capacity each may be seen in the center. These two tanks were used for effluent receiver and/or regenerant receiver tanks. The piping arrangement also permitted these two tanks to be used as feed tanks.

At the top center of figure 16 is a pressure reducing valve which was used to control the pressure of the feed solution to the limits required for the operation being carried out. Feed rate was controlled by a micrometer stroke adjustment on the Model C, Yarway Cyclophram metering pump located on the floor behind the two tanks shown in the center of figure 16.

The pressure reducing valve, which used air pressure for regulation, was installed in the ion exchange system feed line between the feed pump and the ion exchange unit. This valve, in conjunction with a surge line that was placed above the feed pump, dampened the pump pulsations and provided a smooth flow to the ion exchange unit.

#### Ion Exchange System Process Flow

Shown in figure 17 is the first of two process flow arrangements that were investigated. The feed water was fed into the exchange unit in a manner that accomplished removal of nitrate ions prior to removal of magnesium ions. Subsequent regeneration with ammonium hydroxide and nitric acid recovered the nitrates as ammonium nitrate and magnesium as magnesium nitrate.

This arrangement was discontinued because a conductivity end-point could not be readily established for the system. A third resin column would be required for use of this arrangement of the process flow.

The process flow arrangement that was established for this evaluation is shown in figure 18. The feed water was fed into the exchange

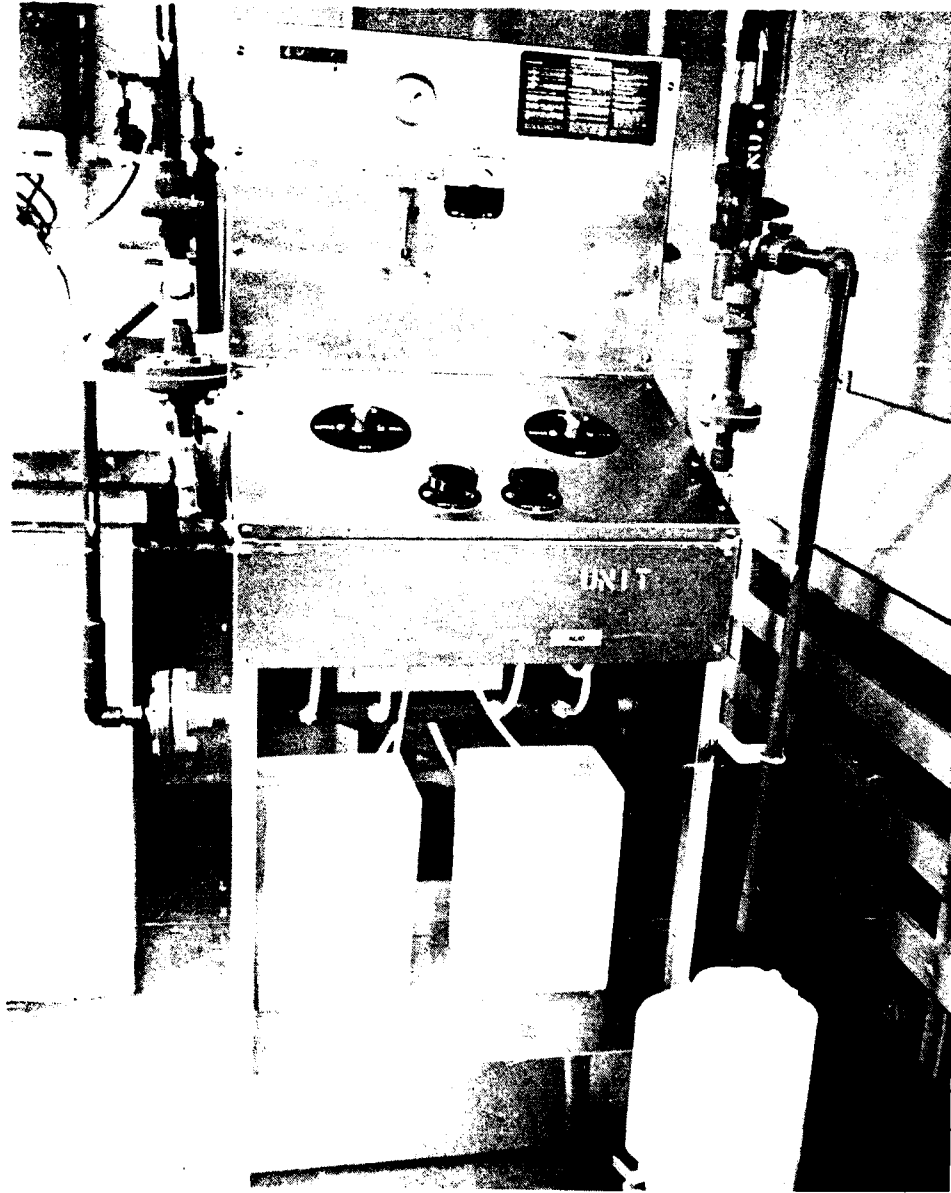


Figure 15. Barnstead two-bed demineralizer.

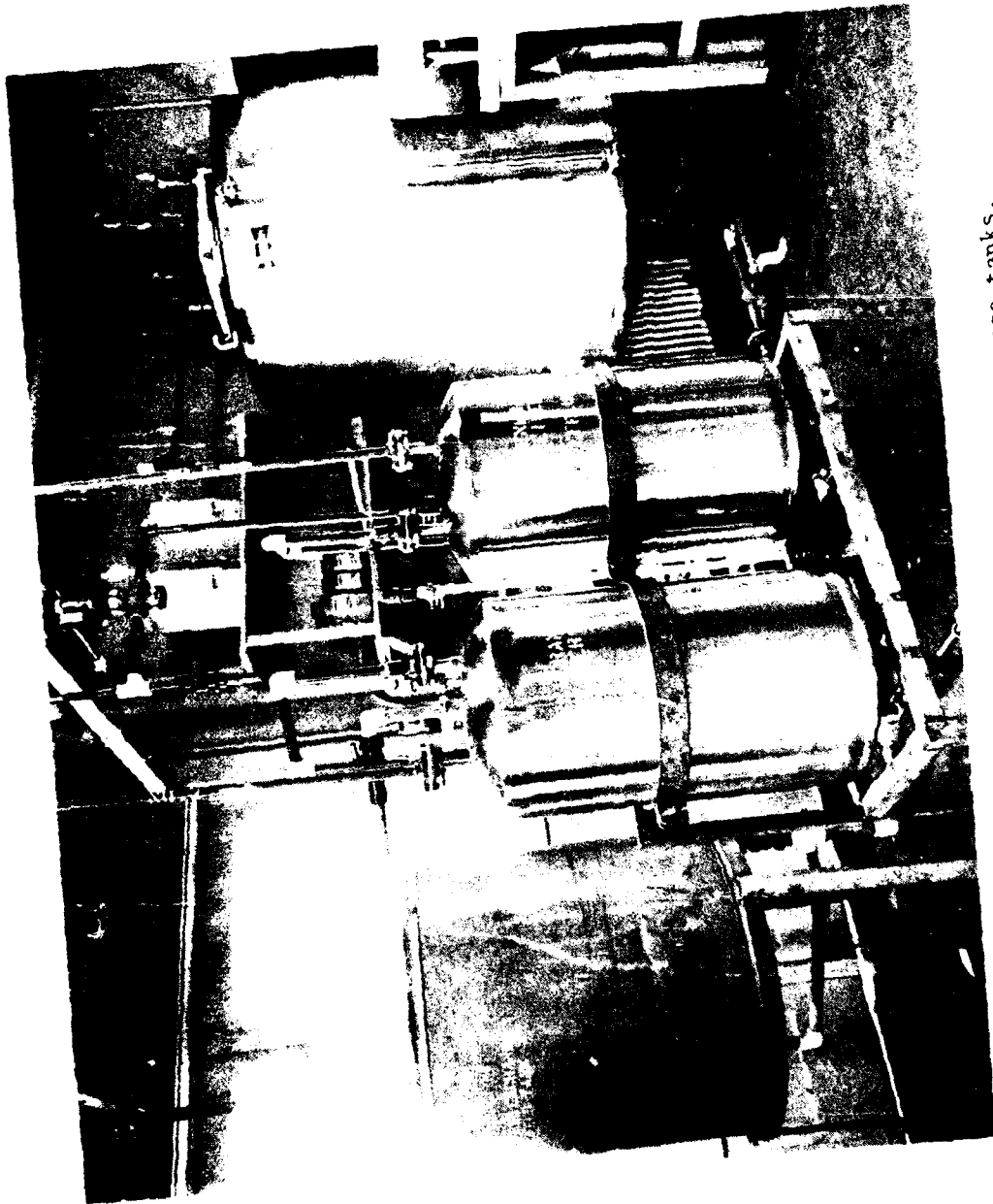


Figure 16. Ion exchange system storage tanks.

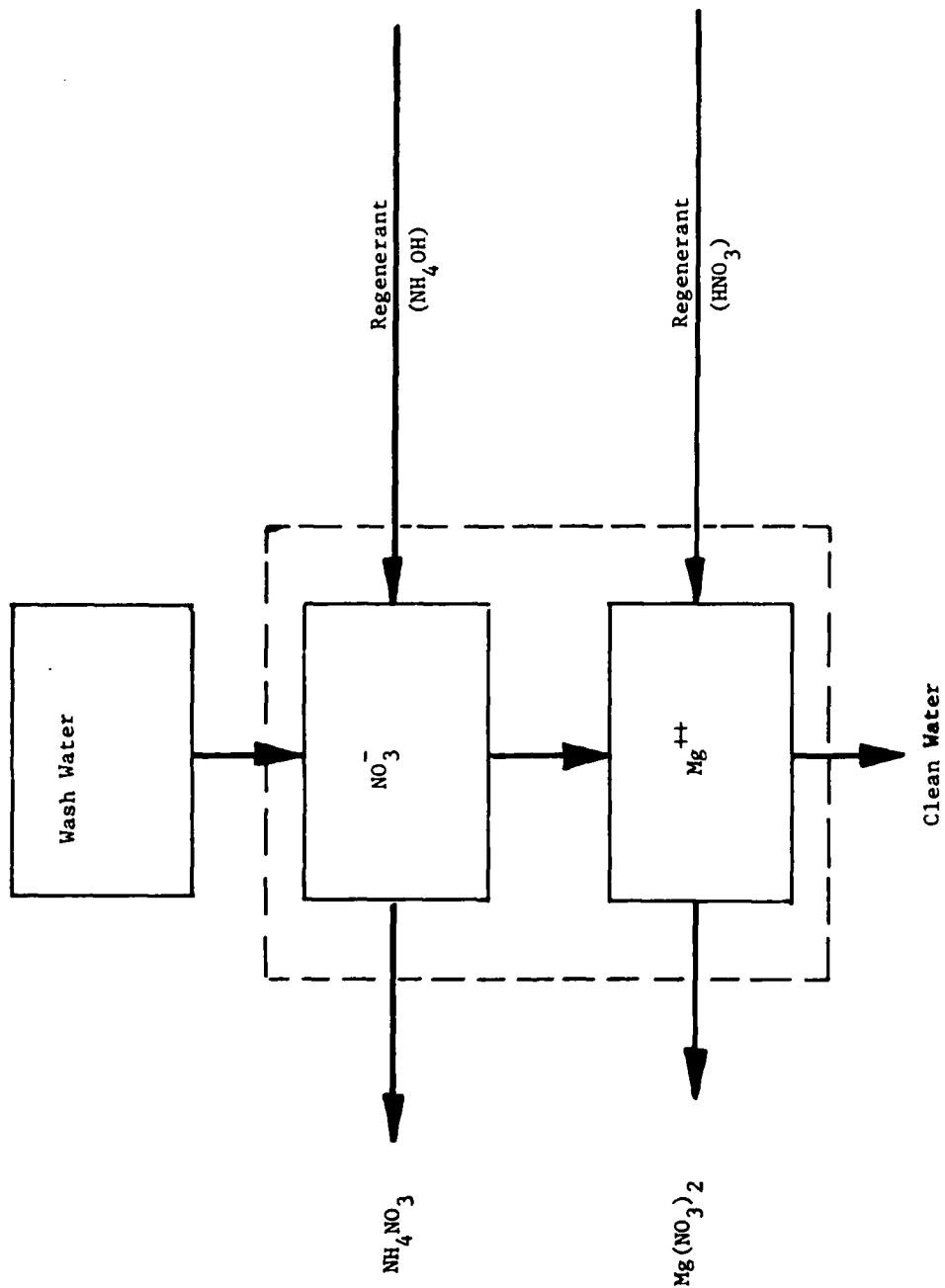


Figure 17. Ion exchange system process flow no. 1.

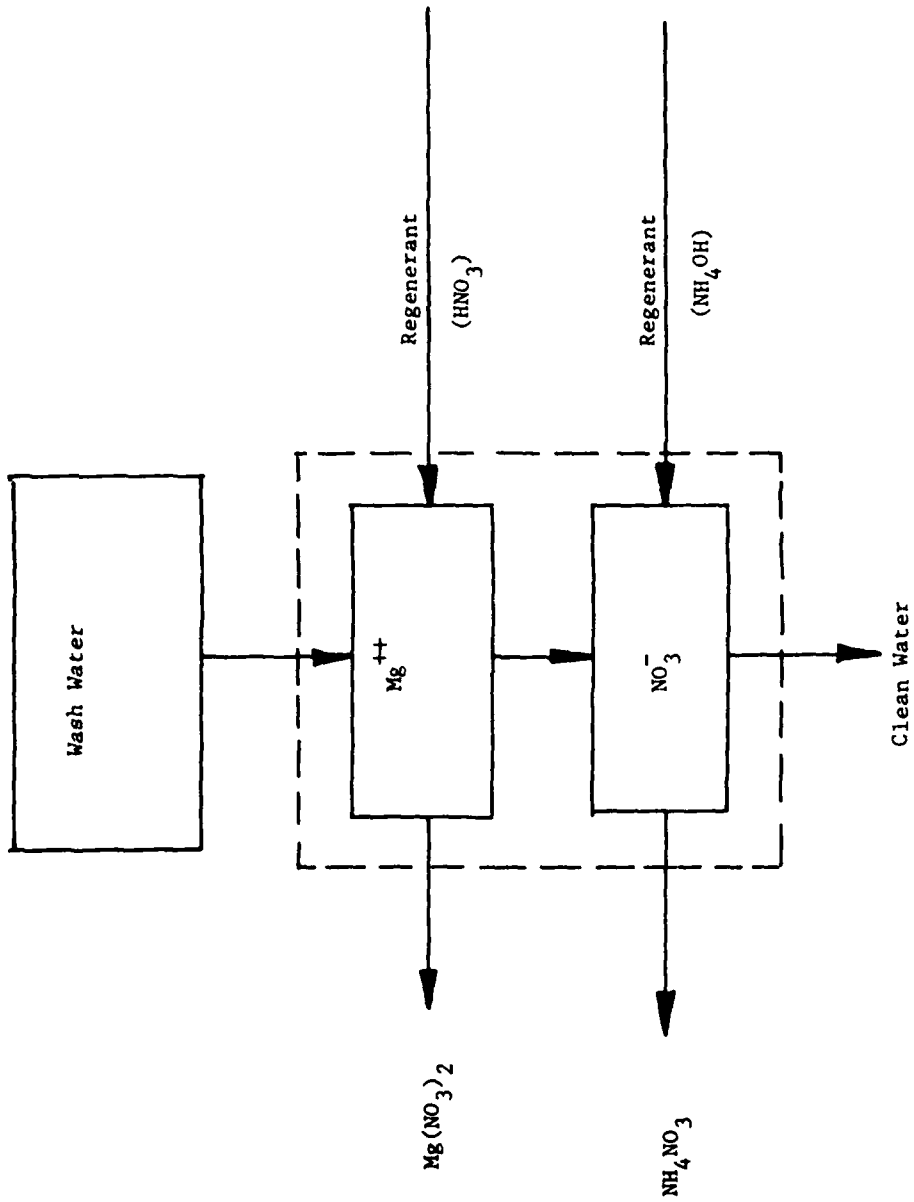


Figure 18. Ion exchange system process flow no. 2.



unit in a manner that accomplished removal of magnesium ions prior to the removal of nitrate ions. Subsequent regeneration with nitric acid and ammonium hydroxide recovered magnesium nitrate and ammonium nitrate. This arrangement gave a satisfactory effluent endpoint and was used for the majority of runs to obtain data on the resins studied.

### Investigation

Feed solutions were run through the ion exchange system with an endpoint of 20,000 ohms/cm resistance. Resins studied were Amberlite IR-120 Plus and Amberlite 200 for the removal of magnesium ions, and Amberlite IRA-47 for removal of nitrate ions. Laboratory analyses indicated that the nitrates found in the effluent varied between 8 and 199 ppm, depending upon the flow rate and the time of sampling during the exhaustion run. The magnesium content of the effluent varied from less than 0.1 to 13 ppm, depending upon the flow rate and the time of sampling. Laboratory analyses for magnesium indicated that the magnesium in the ammonium nitrate obtained during regeneration of the Amberlite IRA-47 resin column varied between 0.1 and 0.5 ppm.

Four kilograms (8 lb) of 30 percent ammonium hydroxide used for regeneration of the Amberlite IRA-47 resin column yielded a resin capacity of 0.20 m<sup>3</sup> (53 gal) of deionized water from a feed solution of approximately one percent nitric acid and 0.1 percent magnesium nitrate or 0.45 m<sup>3</sup> (120 gal) from a feed solution of 0.4 percent nitric acid with 0.1 percent magnesium nitrate, prior to the exhaustion of the column. Whereas, 1.8 kg (4 lb) of 30 percent ammonium hydroxide used to regenerate the Amberlite IRA-47 resin yielded only 0.16 and 0.20 m<sup>3</sup> (42 and 53 gal), respectively, of deionized water from the same influents prior to exhaustion of the column.

Data obtained for five runs using Amberlite IRA-47 resin for the removal of nitrates are listed in table 20. An endpoint of 20,000 ohms/cm was used throughout the runs. The nitrate level for a solution containing 0.5 percent HNO<sub>3</sub> (comparable to that to be drained from the boiling tubs) was consistently reduced to approximately 60 ppm nitrates. Regeneration with a very large excess of ammonium hydroxide did not effectively change the capacity of the resin column as can be seen when comparing data listed for runs A and B.

Flow rate affects the nitrate level in the effluent as can be seen when comparing data listed for runs D and E. The capacity of the resin for removal of nitrates is listed as kilograms of equivalent CaCO<sub>3</sub> per cubic foot of resin in the standard manner of expression of resin capacities used by most vendors of ion exchange systems.

Data obtained for Duolite A-102D resin were omitted from this report because reproducible results could not be obtained using ammonium hydroxide for regeneration of this resin. This is a quaternary ammonium, strong base type resin. The resulting small capacity of the resin (5.2 kgr CaCO<sub>3</sub>), as well as nonreproducible performance, resulted in the abandonment of further investigation of the resin.

Table 20

Ion exchange data

Run	Capacity as		Approximate Influent Comp % HNO <sub>3</sub>	Regenerant 30% NH <sub>4</sub> OH kg	Effluent at 20k ohm/cm ppm NO <sub>3</sub>	Flow (m <sup>3</sup> /s) / m <sup>3</sup> Resin	Influent ppm NO <sub>3</sub>
	m <sup>3</sup> Resin	ft <sup>3</sup> Resin					
A	0.93	33.0	0.5	3.6	8	0.44	6978
B	0.90	31.8	0.5	1.8	4	0.44	7753
C	1.13	39.9	0.4	3.6	8	0.44	4209
D	1.13	39.9	1.0	3.6	8	0.44	9525
E	0.89	31.6	1.0	1.8	4	0.06	9525

RESIN: Amberlite IRA-47      USE: Nitrate Removal

Data obtained for Amberlite 200 and IR-120 (the two cation resins which were recommended for removal of magnesium from acid solutions) are listed in table 21.

The magnesium effluent level shown as 22 ppm for the Amberlite 200 resin was obtained from a solution which was considerably higher in concentration than that expected in the pilot line. Capacity of this resin is comparable to the 22-25 kgr capacities of resins cited in the literature.

The value of 190 ppm  $Mg^{++}$  was obtained at an endpoint slightly lower than 20,000 ohms/cm used in run A. Note that the capacity for run B was larger also because capacity is based on throughput.

As can be seen in table 21, the Amberlite IR-120 Plus resin capacity made it unfeasible economically for the pilot line.

#### Process (Filtered) Water Versus Drinking Water

A question was raised as to the suitability of the selected resins and regenerant chemicals for the manufacture of deionized water to be used in the pilot plant for rinsing of resin columns and dilution of feed solutions.

Using the Barnstead unit and columns of Amberlite 200 and Amberlite IRA-47 resins, approximately 11.7 m<sup>3</sup> (3,100 gal) of drinking water and 13.2 m<sup>3</sup> (3,500 gal) of plant process (filtered) water were each deionized to an endpoint of 50,000 ohms/cm. Four kilograms (8 lb) of 30 percent ammonium hydroxide and 3.2 kg (7 lb) of 70 percent nitric acid were used in the regeneration of the Amberlite 200 and Amberlite IRA-47 resin columns, respectively.

It was concluded that either drinking water or filtered water can be used successfully as feed material to the resin beds for make-up water in the pilot line.

#### Summary of Ion Exchange Studies

A summary of ion exchange studies at this point indicated that:

- a. The resins selected for use in the pilot line ion exchange system are Amberlite 200 for the removal of magnesium ions and Amberlite IRA-47 for the removal of nitrate ions.
- b. These resins produce a satisfactory effluent for a closed loop recycle process.
- c. Magnesium is easily removed from Amberlite 200 resin with nitric acid and nitrates are easily removed from the Amberlite IRA-47 resin with ammonium hydroxide.

Table 21

Ion exchange data

Run	Capacity as		Approximate Influent Comp % HNO <sub>3</sub>	Regenerant 70% HNO <sub>3</sub> kg	Effluent at 20k ohm/cm ppm Mg++	USE: Magnesium Removal		Influent ppm Mg++
	m <sup>3</sup> Resin	kg CaCO <sub>3</sub> / ft <sup>3</sup> Resin				m <sup>3</sup> Resin	Flow (gal/min)/ ft <sup>3</sup> Resin	
A	0.64	22.5	0.25	3.2	22	0.26	1.0	1280
B	0.66	23.4	0.25	3.2	190	0.26	1.0	1280
RESIN: Amberlite 200								
RESIN: Amberlite IR-120 Plus								
A	0.18	6.2	0.4	3.2	0.7	0.44	1.67	127
B	0.12	4.3	1.0	3.2	13	0.06	0.67	202
USE: Magnesium Removal								

- d. Capacities of resins used in the magnesium nitrate/nitric acid system are comparable to those in the literature for other systems.

Based on information in the literature data obtained during this investigation and calculations for the pilot plant, the following recommendations are made for the design of the pilot plant ion exchange system:

- (1) The ion exchange unit for removing magnesium nitrate and nitric acid from the purification process stream should be a two-bed automatically regenerated system.
- (2) The anion resin should be Rohm and Haas Amberlite IRA-47, and the cation resin should be Rohm and Haas Amberlite 200.
- (3) Exchange vessels should be of such height as to provide adequate freeboard for backwashing.
- (4) Polyvinyl chloride should be suitable for use in contact with the exchange resins and regenerant chemicals.
- (5) Polyvinyl chloride piping and valves should be suitable for piping the ion exchange system.
- (6) Metal which comes into contact with regenerant chemicals and/or the exchange process stream should be made of stainless steel.
- (7) A filter should be used to prevent resins from getting into deionized water lines.
- (8) Sample lines and valves should be made available on inlet and outlet lines of each exchange vessel to provide a means of sampling the water streams of each resin column.
- (9) A means should be available for measuring the inlet and outlet pressures of each exchange vessel during each process step.
- (10) A means should be provided for measuring the flow of influent to the unit.
- (11) The regenerant chemicals should have a concentration between four and eight percent when in contact with the

resin beds - ammonium hydroxide for the anion exchanger and nitric acid for the cation exchanger.

- (12) The ion exchange unit should be taken out of service and regenerated on excess conductivity of the effluent.

### Corrosion and Materials Selection

Review of the literature on materials of construction for nitric acid service and magnesium nitrate/nitric acid service led to the conclusion that some corrosion tests should be conducted on the materials being considered under this project. Corrosion tests were conducted on various materials of construction as outlined in the following paragraphs. Results of these tests are given in tables 22 through 25.

#### Selection of Corrosion Tests

##### a. Boiling Nitric Acid Test

The boiling nitric acid test was selected as a preliminary test to be used for acceptance of the quality of various materials resistant to service in the magnesium nitrate/nitric acid system. A modification of the ASTM Method A262, Practice C was used. This test revealed the effects of poorly balanced alloy composition and improper heat treatment. This test was selected because it provided a means of direct comparison of the corrosion rates of candidate materials in a known medium of 65 percent nitric acid with the corrosion rates obtained in high grade and low grade nitric acid-magnesium nitrate nitrating mixes.

The corrosion test setup for the boiling nitric acid test was used as illustrated in figure 19. Three Huey-type apparatuses are shown on the left. The 65 percent boiling nitric acid tests and the high grade and low grade nitrating acid tests were conducted as shown here. The nitric acid solution was maintained at a boiling temperature utilizing a one-liter Erlenmeyer flask provided with a 750 mm (30 in.) reflux condenser (with ground glass joint) to prevent loss of acid. The condenser joint was fitted with glass hooks for supporting the test specimens. Ordinary low wattage laboratory hot plates were used for heating. For suspension of specimens Teflon pipe joint string was used. The solution used for the 65 percent boiling nitric acid tests was C. P. reagent grade nitric acid adjusted with distilled water to  $65 \pm 0.2$  percent by weight as determined by analysis. The nominal high grade nitrating acid used gave an analysis of 84.48 percent nitric acid, 9.90 percent magnesium nitrate, 5.39 percent water, and 0.23 percent oxides of nitrogen. The nominal low grade nitrating acid used gave an analysis of 75.50 percent nitric acid, 14.36 percent magnesium nitrate, 10.11 percent water, and 0.03 percent oxides of nitrogen. The measured temperatures of the boiling solutions were: 121°C (250°F) for the 65 percent nitric acid; 77°C (170°F) for the high grade nitrating acid mix; and 88°C (190°F) for the low grade nitrating acid mix.

Table 22

Corrosion tests in boiling 65 percent nitric acid

Material Identity	Test Period										Remarks				
	1		2		3		4		5			Cumulative			
	Rate	Hour	Rate	Hour	Rate	Hour	Rate	Hour	Rate	Hour	Rate	Hour			
304	8.4	48	4.5	48	7.0	48	8.0	48	12.7	48	8.0	240	Slight Uniform Corrosion		
304 L	5.6	48	4.3	48	7.0	48	5.6	48	6.0	48	5.4	240	Slight Uniform Corrosion		
316	+11.6	48	26	48	11.4	48	12.4	48	20.7	48	11.9	240	Slight Uniform Corrosion		
316 L	10	48	18.5	48	8.6	48	24.7	48	41.7	48	20.6	240	Slight Uniform Corrosion		
Hastelloy C (Wrought)	711	48	Discontinued because of excessive corrosion.												
Multimet Alloy (Welded)	+77.3	48	21.5	48	18.4	48	18.8	48			+4.6	192	Slight Uniform Corrosion		
304 W	11.5	72	8.4	48	8.9	48	8.2	72			9.4	240	Slight Uniform Corrosion		
304 L W	9.1	72	5.3	48	6.2	48	5.2	72			6.6	240	Slight Uniform Corrosion		
440 C	1343	72	1569	48	Discontinued because of excessive corrosion.								1425	120	Attrition Mill Plates are made of either 440 C or Ni-Hard Material.
Ni-Hard	13,183	48	Discontinued because of excessive corrosion.										13,183	48	Attrition Mill Plates are made of either 440 C or Ni-Hard Material.
Alu	Dissolved											<24			

Note: All corrosion rate data are expressed in mils per year.  
+ Gained weight.

Temperature: 121°C (250°F)

Table 23

Results of corrosion tests in high-grade magnesium nitrate nitriding mix at 77°C (170°F)

Material Identity	Test Period												Cumulative		Remarks	
	1		2		3		4		5		6		Rate	Hour		
	Rate	Hour	Rate	Hour	Rate	Hour	Rate	Hour	Rate	Hour	Rate	Hour	Rate	Hour		
304	103	94											103	94	94	Each specimen shows uniform corrosion over all surfaces with exception of weld beads where applicable.
304 L	29	94	81	48	88	48	109	48	124	48	107	72	87	358	358	
316	82	94											82	94	94	
316 L	226	94	473	48	48	Discontinued because of excessive corrosion.							344	142	142	
316	102	48	102	93	392	45							179	186	186	Recheck of order of corrosion rates using samples from different sources.
316 L	57	48	172	93	392	45							330	186	186	
Multimet Alloy (welded)	84	94											84	94	94	Discontinued because of excessive corrosion near weld bead
304 L W	33	48	60	48	87	48	108	48	127	72			87	264	264	Discolored blue-black-brownish weld bead looks as if it is a different material.
316 W	67	48	113	48	222	48	267	72					179	216	216	
Alu	44	45	50	45	30	70							39	160	160	
Alu W	58	45	109	45	53	70							70	160	160	
Carpenter 20	84	45	223	45	274	70							206	160	160	
Durimet 20	135	45	300	95	388	72							300	212	212	
410	49	45	118	95	95	72							97	212	212	
430	54	45	56	95	54	72							56	212	212	
17-4 PH	78	70	98	48	117	72	152	48					109	238	238	

Note: All corrosion rate data is expressed in mils per year.

Nominal High Grade Nitriding Mix Analysis	
Ingredient	Percent
HNO <sub>3</sub>	84.48
Mg(NO <sub>3</sub> ) <sub>2</sub>	9.90
Oxides	0.23
H <sub>2</sub> O	5.39



Table 24

Results of corrosion tests in low grade magnesium nitrate  
nitrating mix at 88°C (190°F)

Material Identity	Test Period				Cumulative	
	1		2		Rate	Hours
	Rate	Hours	Rate	Hours		
Durimet 20	84	96			84	96
Carpenter 20	38	96	8.6	119	21	215
316LW	21	96			21	96
304L	5	119			5	119
304LW	6	119			6	119

NOTE: All corrosion rate data is expressed in mils per year.

Nominal Low Grade Nitrating Mix Analysis

<u>Ingredient</u>	<u>Percent</u>
HNO <sub>3</sub>	75.50
Mg(NO <sub>3</sub> ) <sub>2</sub>	14.36
Oxides	0.03
H <sub>2</sub> O	10.11

Table 25

Elastomer chemical resistance test data (metric units)

Elastomer Type and Number	Sample Number	Original L <sub>0</sub>	Length, mm				A <sub>2</sub> Durometer Readings				Remarks*			
			3 Day Immersion		28 Day Immersion		Original D <sub>0</sub>	3 Day Immersion		28 Day Immersion				
			L <sub>1</sub>	L <sub>2</sub>	L <sub>1</sub>	L <sub>2</sub>		D <sub>1</sub>	D <sub>2</sub>	D <sub>1</sub>		D <sub>2</sub>		
DAL	1	114.66	114.68	0.002	114.86	0.020	0.18	69	71	+2	71	+2	+2	
	2	115.19	115.14	0.050	115.06	-0.013	-0.11							
NEO	3	115.06	115.44	0.038	117.68	0.262	2.2	72	71	-1	65	-7	-7	Edges dete- riorated on both NEO samples
	4	115.19	115.57	0.038	118.06	0.287	2.5							
EPT 8020	5	115.32	115.11	-0.021	115.27	-0.005	-0.044	59	61	+2	58	-1	-1	
	6	115.09	115.09	0.000	115.16	0.005	0.066							
EPDM (5207)	7	115.01	115.14	0.013	115.87	0.086	0.75	59	64	+5	58	-1	-1	
	8	115.32	115.32	0.000	116.21	0.089	0.77							

Chemical Composition of Liquid Medium

Ingredient, %	At Start	At End
HNO <sub>3</sub>	11.04	11.14
Mg(NO <sub>3</sub> ) <sub>2</sub>	0.60	0.68
H <sub>2</sub> O	88.36	88.18

\*Temperature Range 20°C to 29°C (68°F to 85°F); average temperature during 28 days exposure 26°C (80°F).

Table 25

## Elastomer chemical resistance test data

Elastomer Type and Number	Sample Number	Length, inches				A <sub>2</sub> Durometer Readings				Remarks*			
		Original L <sub>0</sub>	3 day L <sub>1</sub>	28 day L <sub>2</sub>	28 day L <sub>2</sub>	Original D <sub>0</sub>	3 day D <sub>1</sub>	28 day D <sub>2</sub>	28 day D <sub>2</sub>				
DAL	1	4.514	4.515	0.001	4.522	0.008	0.18	69	71	+2	71	+2	
	2	4.535	4.533	-0.002	4.530	-0.005	-0.11						
NEO	3	4.530	4.545	0.015	4.633	0.103	2.2	72	71	-1	65	-7	Edges deteriorated on both NEO samples
	4	4.535	4.550	0.015	4.648	0.113	2.5						
EPT 8020	5	4.540	4.532	-0.008	4.538	0.002	-0.044	59	61	+2	58	-1	
	6	4.531	4.531	0.000	4.534	0.003	0.066						
EPDM (5207)	7	4.528	4.533	0.005	4.562	0.034	0.75	59	64	+5	58	-1	
	8	4.540	4.540	0.000	4.575	0.035	0.77						

## Chemical Composition of Liquid Medium

Ingredient, %	At Start	At End
HNO <sub>3</sub>	11.04	11.14
Mg(NO <sub>3</sub> ) <sub>2</sub>	0.60	0.68
H <sub>2</sub> O	88.36	88.18

\*Temperature Range 20°C to 29°C (68°F to 85°F); average temperature during 28 days exposure 26°C (80°F).

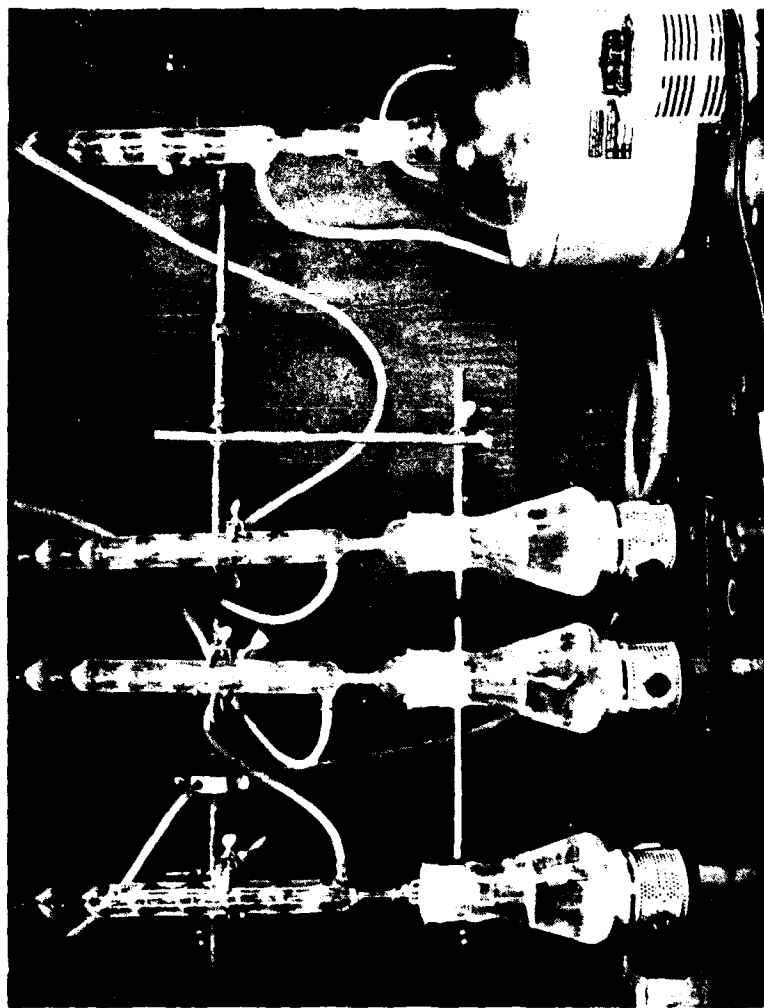


Figure 19. Corrosion test set-up for boiling nitric acid tests.

The surface of all specimens was finished with No. 320 grit abrasive paper. The specimens were then measured and the total surface area of each was calculated. They were then degreased in acetone, dried, and weighed. Each specimen was completely immersed in a quantity of the appropriate nitric acid solution to provide a minimum of 125 milliliters of solution per square inch of surface area.

The solutions were heated to boiling and maintained at boiling for a period of 48 hours (several other periods were used in some cases). At the end of 48 hours the specimens were removed, rinsed with tap water, scrubbed with a nylon brush under tap water to remove adherent corrosion products, dried, degreased in acetone, dried and weighed. The tests were repeated; weight losses were determined after each test period; and fresh acid solution was provided for each test period.

b. Other Corrosion Tests

Other corrosion tests consisted of exposure of materials to various concentrations of magnesium nitrate/nitric acid solutions at ambient and elevated temperatures; circulation of nitrating mixes through specially constructed pipe lines; and exposure of various materials to acid vapors that they could normally be expected to see in service.

Tests on plastic materials such as O-rings were conducted in the five-liter flask shown in the right side of figure 19. This flask was supported by a Glas-Col heater for maintaining temperature. Subject specimens were suspended into the fumes above the liquid in the flask by means of Teflon pipe joint string.

The EIMCO Processing Machinery Division of Envirotech Corporation furnished samples of four elastomer materials for testing in liquid simulating the composition of the flume water. These elastomer materials were candidates for the dewatering filter belt and filter cloth for the pilot plant dewatering filter.

Corrosion Specimens

Materials selected for corrosion specimens included some of both the 300 and 400 series stainless steels, Durimet 20, Carpenter 20, and aluminum, plus materials that were candidates for attrition mill grinding plates. Sixteen of these corrosion specimens are shown in figure 20. In general, the specimens shown on the left of the numerals are shown prior to exposure to an acid solution, and the specimens shown on the right of the numerals are specimens that have been exposed to high grade nitrating acid.

PE-274/293  
CORROSION SPECIMENS

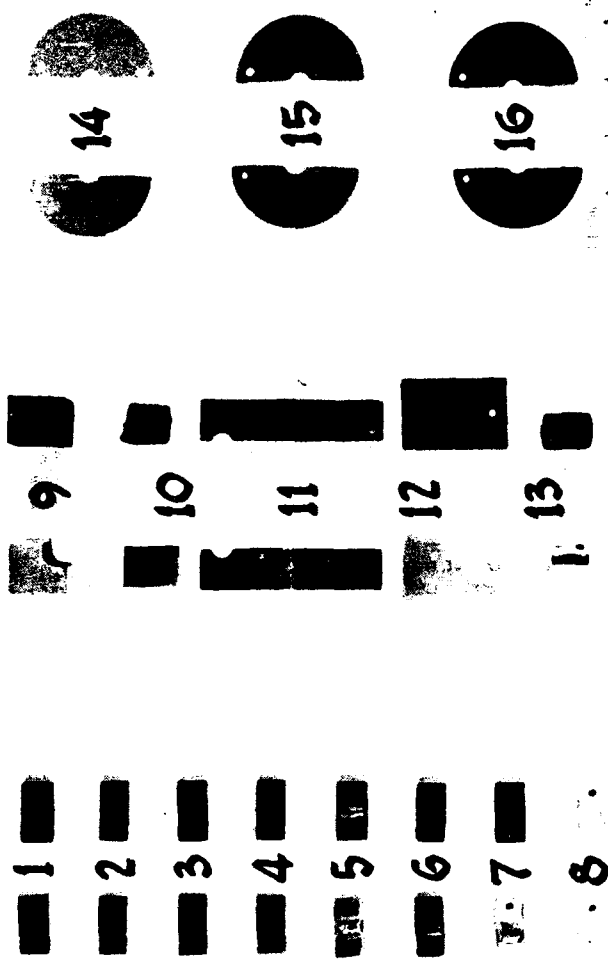


Figure 20. Corrosion specimens.

## Test Results

### a. Boiling Nitric Acid

Results of corrosion tests in 65 percent boiling nitric acid are listed in table 22. Hastelloy C was tested as a comparison for other materials. Note the negative corrosion rate (expressed with a plus sign) for the Multimet Alloy specimen. Instead of losing weight this specimen gained in weight over the first 48-hour test period. In the three subsequent 48-hour test periods, the specimen lost weight with slight uniform corrosion noticed on examination. The initial gain in weight was probably due to a buildup of hard corrosion products that were not washed away upon scrubbing with a nylon brush. Testing of the 440C and NiHard specimens was discontinued because of excessive corrosion after one and two test periods, respectively. Aluminum dissolved in the 65 percent acid as was expected. The attrition mill plates used in the bench-scale phase of this project were made of 440C in one set and NiHard material in the second set. As shown by the results in table 22, neither of these materials is suitable for service in concentrated nitric acid and will not be used in the pilot plant attrition mills. The attrition mill manufacturer could not economically furnish grinding plates of other materials, e.g., Type 304 or 316 stainless steel, with the 300 mm (12 in.) mills used in the bench-scale tests.

### b. High Grade Nitrating Acid Mix

Results of tests in 65 percent nitric acid were reviewed, and a number of materials were selected for exposure to magnesium nitrate process nitrating acids.

Results of corrosion tests in high grade magnesium nitrate/nitric acid nitrating mix are listed in table 23. As noted in the table, a recheck of the order of corrosion rates was made on specimens of Types 316 and 316L stainless steel using samples from different sources. Type 316L stainless steel corrodes at a much higher rate than does Type 316 in this medium and in 65 percent nitric acid. An example of the higher corrosion rates obtained for materials in the high grade nitrating mix than in the normal 65 percent boiling nitric acid test was shown by the cumulative corrosion rate of 87 mils per year for Type 304L stainless steel after an exposure of 358 hours in the high grade nitrating mix compared to the 5.4 mils per year for the same material in 65 percent nitric acid (approximately 15 times the rate in the weaker acid).

The testing of the Multimet Alloy specimen was discontinued even though it showed a fairly low comparative corrosion rate because of excessive corrosion near the weld bead. This weld bead decay zone is shown by the specimen on the left of numeral 11 in figure 20.

As found in the literature these tests confirmed the fact that aluminum exhibits good corrosion resistance to higher concentrations of nitric acid.

Note the low corrosion rate of Type 430 stainless steel and the relatively high rates for Carpenter 20 and Durimet 20 specimens in this medium. Type 17-4 PH material was tested as a candidate material for attrition mill plates; however, the attrition mill vendor could not supply grinding plates made of 17-4 PH material, Type 304, nor 316 stainless steel. Type 430 stainless steel has been selected for use in the pilot plant attrition mills.

c. Low Grade Nitrating Acid Mix

Several materials were selected for testing in low grade nitrating acid mix. Results of these tests are listed in table 24. The corrosion rates of specimens exposed to this medium were as expected because of the lower nitric acid content of the low grade nitrating mix. Note that the rate for Type 304L stainless steel is practically the same in this medium as in 65 percent nitric acid.

d. Dewatering Filter Belt Resistance Tests

Two samples each of four elastomer materials were furnished by EIMCO for testing in liquid simulating the composition of the flume water. The length, thickness, and A<sub>2</sub> Durometer hardness were measured for the four materials prior to immersion and after 3 and 28 days immersion in the liquid. The liquid was analyzed before and after these tests. The elastomer chemical resistance test data are shown in table 25. The material identified as EPT 8020 (an ethylene propylene terpolymer) exhibited the best resistance and was selected as the belt material for the pilot plant dewatering filter.

e. Other Corrosion Tests

Several other corrosion tests were run. Four Viton compounds in the form of O-rings were placed in boiling high grade nitrating mix for 24 hours. One compound dissolved, two distorted completely into shapeless masses, and one sample distorted very slightly but maintained its elasticity and shape. This semi-resistant compound is Viton Compound V747-75 from the Parker Seal Company.

It is important to note that one of the compounds which distorted completely in the high grade nitrating mix showed excellent resistance to flume water composition when a polypropylene bellows-type metering pump containing identical Viton O-rings operated continuously for over four months without deteriorating. Flume water composition used was 11 percent nitric acid and 0.6 percent magnesium nitrate. The flume water operating temperature averaged 26°C (80°F).



Small stainless steel pumps were used without success in an attempt to circulate nitrating mix through pipe lines made of fittings, valves, and lines such as those expected to be used in the pilot plant equipment. Failure of these tests was due to difficulties encountered in preventing excessive temperature rise in the circulating solutions because of friction. A reservoir of five gallons capacity was the largest that could be utilized in the available working space.

Samples of plastic drain lines and fiberglass tank materials that were suitable for use in 20-30 percent nitric acid were found to disintegrate on exposure to nitrating mixes at room temperature. This condition eliminated the consideration of these materials for use in the pilot plant because a nitrating or spent acid spill could cause replacement of the drain lines and/or tanks exposed to the acids.

Several samples of polyvinyl chloride (PVC Type 1) pipe and fittings were immersed in beakers of 20 percent and 11 percent nitric acid for a period of several weeks without any noticeable deterioration or other effects on the samples.

#### Design Recommendations for Pilot Plant Construction Materials

In summary, corrosion tests were conducted on materials of construction being considered for nitric acid and magnesium nitrate/nitric acid service utilizing the boiling nitric acid test for preliminary acceptance of the quality of materials in the system. After preliminary screening the corrosion rates of the candidate materials were obtained in high grade and low grade nitric acid/magnesium nitrate nitrating mixes. Some materials were also exposed at ambient and/or elevated temperatures to various concentrations of nitric acid/magnesium nitrate solutions and/or vapors that they could normally be expected to see in service. These tests provided the data used in selecting the fabrication materials for the pilot plant. The following materials are recommended for use as indicated in the pilot plant:

<u>Service</u>	<u>Material</u>
Strong Nitric Storage Tank	Aluminum
Attrition Mill Grinding Plates	Type 430 Stainless Steel
All Other Metal Equipment	Type 304L or Type 316 Stainless Steel
Dewatering Filter Belt	EPT 8020
Dewatering Filter Cloth	Polypropylene
Ion Exchange System Lines	PVC, Type 1
Fines Filters O-Rings	Viton Compound V747-75 from Parker Seal Company
Gaskets	Teflon
Filter Bags	Teflon

## PROCESS FLOW

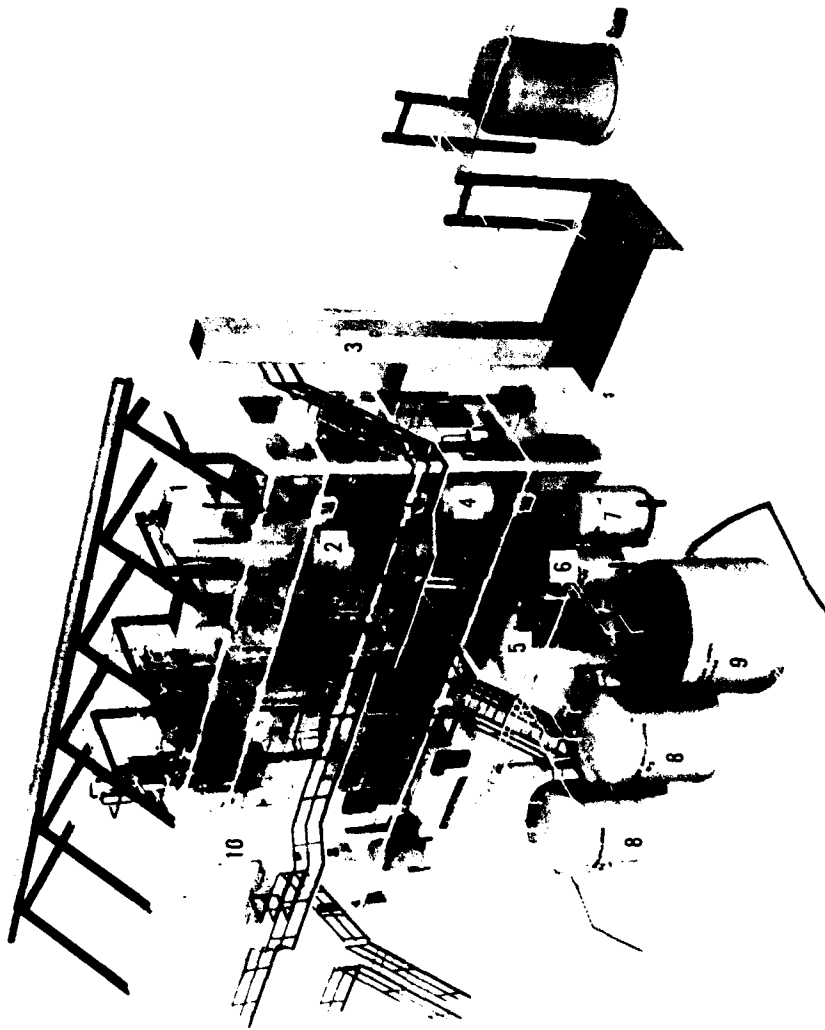
Subsequent to completion of the bench-scale evaluations under the scope of this project, design criteria were developed for a 68 kilograms per hour (150 pound per hour) NC pilot plant to be constructed and evaluated. A model of the general pilot plant layout is shown in figures 21 and 22. In the equipment identified on figure 21, magnesium carbonate is reacted with nitric acid and partially dehydrated in the magnesium carbonate reactor tank (2) and stored as magnesium nitrate in molten form for acid mix preparation (4). Strong nitric acid, spent acid, and magnesium nitrate are blended to the desired nitrating mix composition in the salt acid mix tank (5) and transferred to salt acid storage (6) for metering into the process.

In figure 22 dried cellulose is weighed across the Merrick Scale feeder (1) and mixed with the nitrating solution either in the attrition mills (2) or on entry into the nitrating vessels (3). Spent acid is wrung from the nitrated cellulose and the NC washed in the centrifuge (4). On discharge from the centrifuge the NC is slurried in water (5) and pumped to the dewatering filter (6). The slurry water is removed and two fresh washes are applied on the dewatering filter after which the NC is reslurried in water and flows by gravity to one of the viscosity adjustment boiling tubs (7).

Upon completion of boiling to adjust stability and viscosity and washing to remove residual acid and magnesium nitrate, the NC is blended (8), if necessary, to obtain the desired nitrogen and solubility parameters and discharged from the pilot unit for disposition (10). NC fines are removed from the spent acid and dewatering filter effluents and returned to the process.

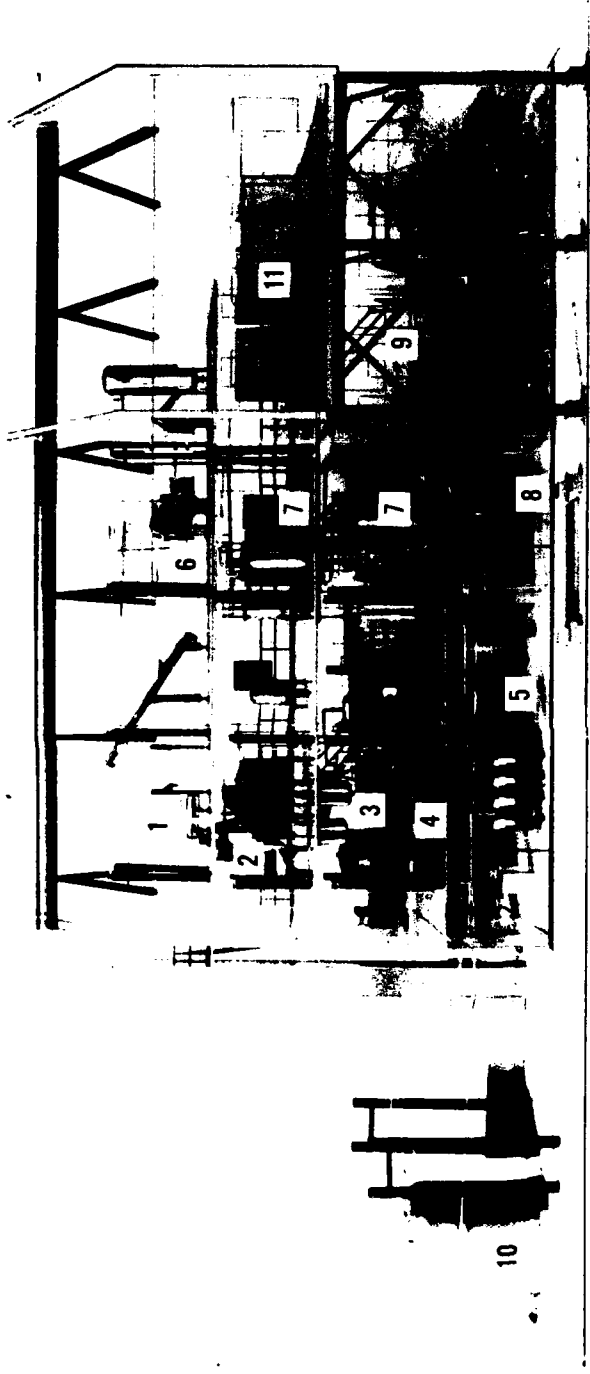
Water effluent from the dewatering filter is returned to the centrifuge for countercurrent wash makeup and reuse as slurry water. Recovered water from the boiling and washing operations is partially used as dewatering filter make-up wash and to reslurry NC as it is discharged from the dewatering filter. The excess water from this operation is passed through an ion exchange unit (9) for recovery of the magnesium and nitrate ions.

These figures represent the general layout of the pilot plant. The control consoles (10) are located here for ready access to the operations being performed.



- 1-CELLULOSE WEIGH FEEDER
- 2-MAGNESIUM CARBONATE REACTOR
- 3-INGREDIENT ELEVATOR
- 4-MAGNESIUM NITRATE STORAGE
- 5-SALT ACID MAKE UP
- 6-SALT ACID FEED
- 7-SPENT ACID TANK
- 8-ION EXCHANGE FEED WATER
- 9-DEIONIZED WATER
- 10-CONTROL CONSOLES

Figure 21. Magnesium nitrate - NC pilot plant model.



- 1-CELLULOSE WEIGH FEEDER
- 2-ATTRITION MILLS
- 3-NITRATORS
- 4-CENTRIFUGE
- 5-SLURRY TANK
- 6-DEWATERING FILTER
- 7-BOIL TUBS
- 8-NC BLEND TANK
- 9-ION EXCHANGE
- 10-NC SLURRY STORAGE
- 11-CONTROL CONSOLES

Figure 22. Magnesium nitrate - NC pilot plant model.

## PILOT PLANT DESIGN AND CONSTRUCTION

### General Comments

The pilot plant was designed to use equipment scaled down from the production-scale continuous nitration facility. This was done so that information collected in the pilot plant evaluation could be scaled up directly to convert the production-scale mixed acid nitration facilities to the magnesium nitrate process. The magnesium nitrate NC pilot plant included provisions for evaluating initiation of nitration in attrition mills prior to feeding the salt-acid-cellulose mixture to the nitrators. The pilot plant was designed for evaluation of the process either with or without this feature in order to determine its feasibility. This process incorporated washing of the NC before sending it to the purification area and recycling of the filtrate as the initial countercurrent wash in the centrifuge to improve recovery of the nitrating mix and greatly reduce nitrate stream pollution.

The pilot plant design included equipment for removing low concentrations of nitric acid and magnesium nitrate contained in wash water from the purification area. This system is a two-bed ion exchange resin system selected on the basis of previous bench-scale tests.

The design, procurement, installation, and evaluation of a subpilot-scale magnesium nitrate/nitric acid concentrator was included in order to process the spent nitrating mix from the pilot plant for reuse in the process and to provide design criteria information for the conversion of existing nitric acid/sulfuric acid concentrators to the magnesium nitrate process.

Construction materials as recommended and selected in the bench-scale studies were utilized in the pilot plant with the exception of process piping. Because of delivery schedules and cost escalations, substitutions of several types of 300 series stainless steel were made in noncritical areas. A list of process piping as to schedule, size, and type steel is on file at RAAP for all of the process areas.

## Equipment Layouts

Equipment in the pilot plant was arranged as shown in figures 21 and 22. At the beginning of the project, only one-half of the building was made available for use as a pilot plant. Therefore, equipment was arranged to fit available space on drawings and placed in position on arrival at RAAP. The remainder of the building was made available at a later date and utilized for planning the installation of the subpilot nitric acid concentrator. The concentrator equipment was arranged as shown in figure 23.

## Electrical Components

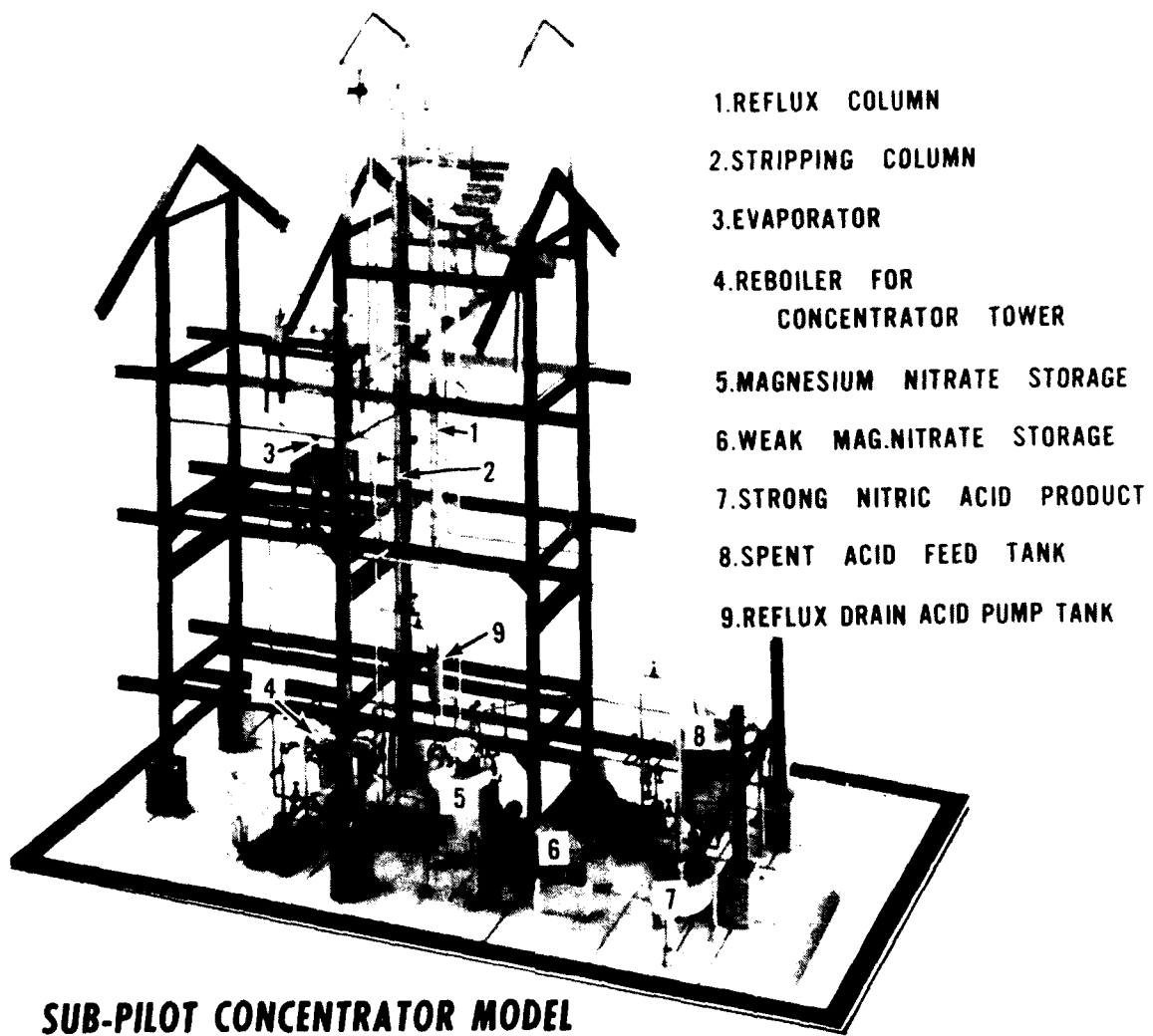
The electrical classifications of equipment in the pilot plant are shown in figure 24. The same requirements were used for the concentrator end of the building. Not shown in the figure is the electrical classification of equipment in the control room located on the third floor. Equipment in the control room was permitted to be NEMA 1 since the control room was ventilated by outside forced air and the conduit and fixtures through walls were sealed.

Breakers and starters for the pilot plant were ordered as one unit. Additional breakers and starters were ordered for the concentrator. These units were installed in one room adjacent to the control room. Relays were placed in the breaker room near the motor breakers and controlled from pushbuttons on the control console in the adjacent control room.

All electrical conduit and connectors were plastic coated, "Korkap," for use in areas exposed to acid fumes. All electrical conduit joints were sealed with "Korkap" paint where necessary.

Motors utilized in the pilot plant are generally 460 volt, 60 hertz, three phase motors with the exceptions of several small 115 volt, 60 hertz, single phase motors. Motor cases were ordered to suit requirements of the layout shown in figure 24.

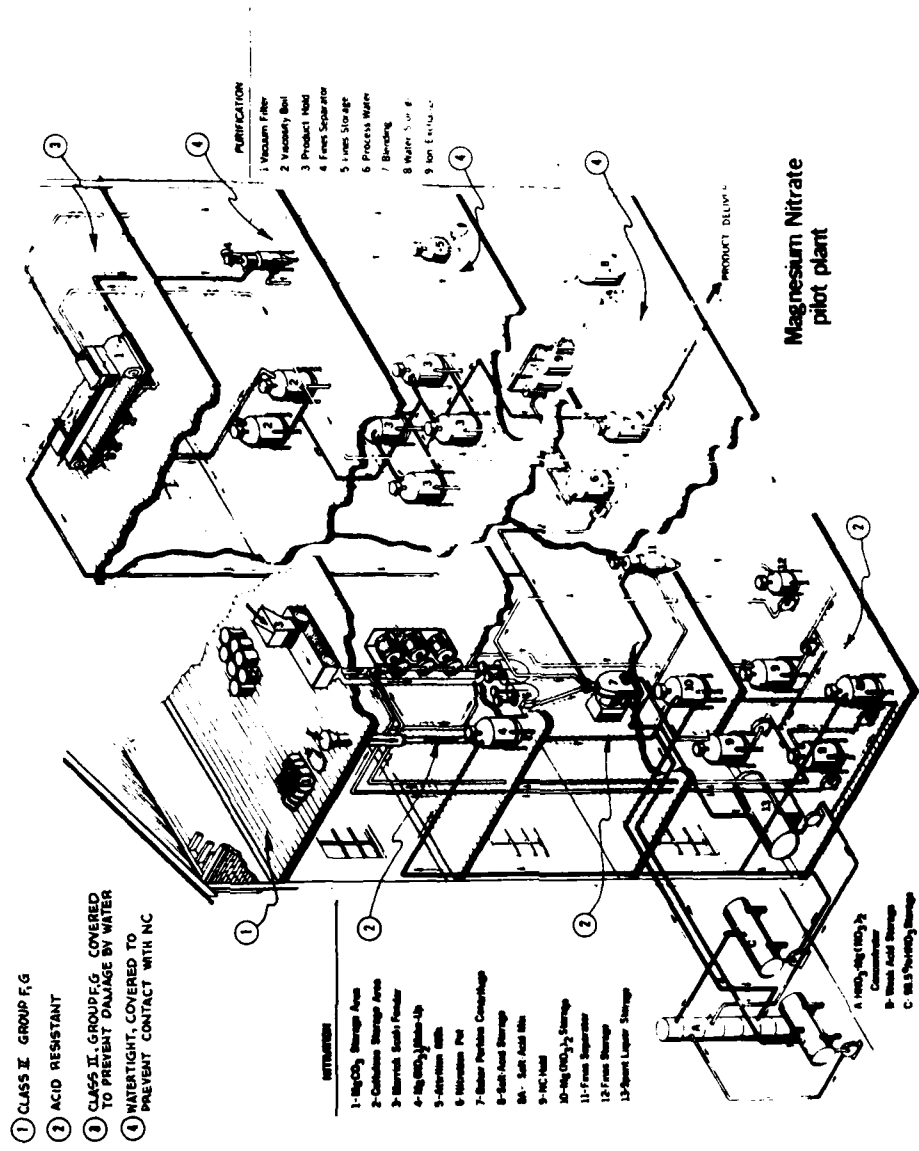
Function switches and start/stop switches were installed at each pump and/or agitator location throughout the pilot plant. The function switches are manual/auto. When in the "manual" position, the "start/stop" switch at the same location is activated and can be used to either start or stop the functioning of the applicable piece of equipment. With the switch in "manual" position, the "run/stop" switch on the applicable control room console is deactivated.



**SUB-PILOT CONCENTRATOR MODEL**

***Nitric Acid-Magnesium Nitrate***

Figure 23. Subpilot concentrator model.



- ① CLASS II GROUP F,G
- ② ACID RESISTANT
- ③ CLASS II GROUP F,G COVERED TO PREVENT DAMAGE BY WATER
- ④ WATERTIGHT COVERED TO PREVENT CONTACT WITH HCl

- INTRUSION**
- 1- MgCO<sub>3</sub> Storage Area
  - 2- Calcium Storage Area
  - 3- Blended Salt Powder
  - 4- Mg(NO<sub>3</sub>)<sub>2</sub> Storage
  - 5- Ammonium Salt
  - 6- Nitric Acid
  - 7- Water Purification
  - 8- Salt Acid Storage
  - 9- HCl
  - 10- HNO<sub>3</sub> Storage
  - 11- Feed Separator
  - 12- Feed Storage
  - 13- Spent Layer Storage

- A- Class II Group F,G Composite
- B- Acid Resistant
- C- HCl Resistant

Figure 24. Electrical classification of equipment.



When the function switch is in the "auto" position the "start/stop" switch at the same location is deactivated and the applicable equipment can be started and stopped from the control room console. If necessary the equipment can be stopped at the field location by changing the function switch from the "auto" to the "manual" position. This action interrupts the control circuit and cuts off the applicable relay, thereby stopping the power to the motor. When reasons for stopping the equipment have been eliminated the operator can return control of the equipment back to the control room by changing the function switch to the "auto" position.

Fluorescent lighting was installed throughout the building for use during normal operations. Each floor was provided with sufficient emergency lights to permit orderly emergency operations. These units are battery powered.

A diesel powered generator was provided for supplying emergency power to the control room instrument consoles and the pilot plant communications system. Included is a manual switch-over system. Automatic switching was deemed unnecessary.

A communication system was installed on all four floors and the loading dock of the pilot plant. An operator of each station is capable of talking with any other station or all stations at the same time or over the public address system at all locations at the same time. The system selected for the pilot plant is a C.W. Thomson Company system utilizing their TC-1485, TC-1085, TC-1015, TC-1015X, TC-1095, and TC-1090 units in a unitized audio network.

#### Building Modifications

The pilot plant was installed in and around an existing batch type NC nitrating house. Several structural steel members of the existing building were removed and/or rearranged to permit installation of the equipment as shown in the model depicted in figures 21, 22, and 23. Where needed to support the heavier equipment, additional reinforcing steel beams were installed. Headspace between floors required moving of several supports in order to provide room for processing equipment. Drawings for these modifications are on file at RAAP and listed in Appendix A.

#### Equipment Procurement Problems

##### General

As previously discussed, equipment procurement problems occurred during the construction of the pilot plant. Nearly all of these problems were associated with shortages, price escalations, and indefinite delivery schedules of stainless steel items to the equipment vendors.

## Redesign and Specification Changes

A much earlier delivery date was obtained for the process tanks mounted with flush bottom valves by accepting valves operated by 414-621 kPa (60-90 lbf/in.<sup>2</sup>) air pressure rather than the prescribed 21-103 kPa (3-15 lbf/in.<sup>2</sup>) design intended for operation of all equipment. However, this action resulted in the additional costs for solenoid operated air control valves placed between the control room consoles and the valves. Also, additional electrical conduit and air piping was required to make the valves function as intended.

## Utilization of On-Hand Items

An effort was made to make use of all suitable tanks and equipment on hand at RAAP that had been excessed from other projects and from production facilities. Three 22.7 m<sup>3</sup> (6000 gal) aluminum tanks and one 37.9 m<sup>3</sup> (10,000 gal) stainless steel tank that had been excessed from the TNT production facilities were cleaned and installed at the pilot plant. The three aluminum tanks were to be used for accumulation of ion exchange feed waters (containing magnesium nitrate, nitric acid, and water in very low concentrations) and storage of processed NC. Note: Prior to use these tanks were to be coated on the inside with a polyvinyl chloride coating.

The 37.9 m<sup>3</sup> (10,000 gal) stainless steel tank is to be used for storage of deionized water.

A 2.4 m x 9.2 m (8 ft x 30 ft) aluminum tank, excessed from another project, was obtained and installed as the strong nitric storage tank.

The boiling tub water hold tank pump, the belt wash accumulator tank pump, the brine makeup pump, the brine circulating pump, and the brine makeup tank were all obtained and used from on-hand equipment.

The fire suppression system that was installed to suppress fires if they occurred in the cellulose weigh and feed system was borrowed from a RAAP production facility which had been placed in a protective storage status.

## Individual Equipment Design

### Cellulose Feed System

Information pertaining to the cellulose feed system is proprietary, Hercules Incorporated information and can be found in Volume II of this report.

### Nitration

Information pertaining to nitration is proprietary Hercules Incorporated information and can be found in Volume II of this report.

## Tanks

Figure 25 shows the salt/acid makeup and storage tanks that are located just outside the ground floor of the southeast end of the pilot plant. Salt/acid mix of the required composition for the type NC being manufactured is pumped from the appropriate storage tank into the salt/acid feed tank. From this tank the acid is pumped into the nitrator at a controlled rate through the acid feed distributor. A preset quantity of flow is controlled by an in-line magnetic flow control instrument.

Spent acid effluent from the centrifuge flows into the spent acid tank for temporary storage and weighing. The quantity of spent acid is weighed continuously and readout is indicated on a meter in the control room, as shown later in figure 39.

All tanks, pumps, and lines associated with the processing of concentrated magnesium nitrate were steam jacketed or steam traced where required to prevent solidification of the magnesium nitrate solutions.

The ammonium hydroxide makeup tank shown in figure 25 is used as described later under the Ion Exchange System.

## Centrifuge and Slug Feeder

Information pertaining to centrifuging and the slug feeder is proprietary, Hercules Incorporated information and can be found in Volume II of this report.

## Wash Acid Chilling System

Information pertaining to the wash acid chilling system is proprietary, Hercules Incorporated information and can be found in Volume II of this report.

## Dewatering Filter

The Eimco dewatering filter is shown in figures 26 and 27 with the associated vacuum pump in figure 28. Detailed operation of the filter and the pollution control and vacuum system for the Eimco filter are shown in figures 29 and 30, respectively. The Eimco filter system reduces the acid content of the centrifuged NC to a level acceptable for viscosity boiling. NC/acid/water slurry (approximately five percent NC) is pumped from NC hold tank No. 1 to a stainless steel feed box at the head end of the filter (fig. 26).

The feed box distributes the slurry uniformly across the width of a porous polypropylene conveyor belt (filter cloth). Beneath the polypropylene belt is an ethylene-propylene terpolymer (EPT) belt with channels molded into it, which moves parallel to and supports the upper belt. The channels in the EPT belt run from near the sides of the

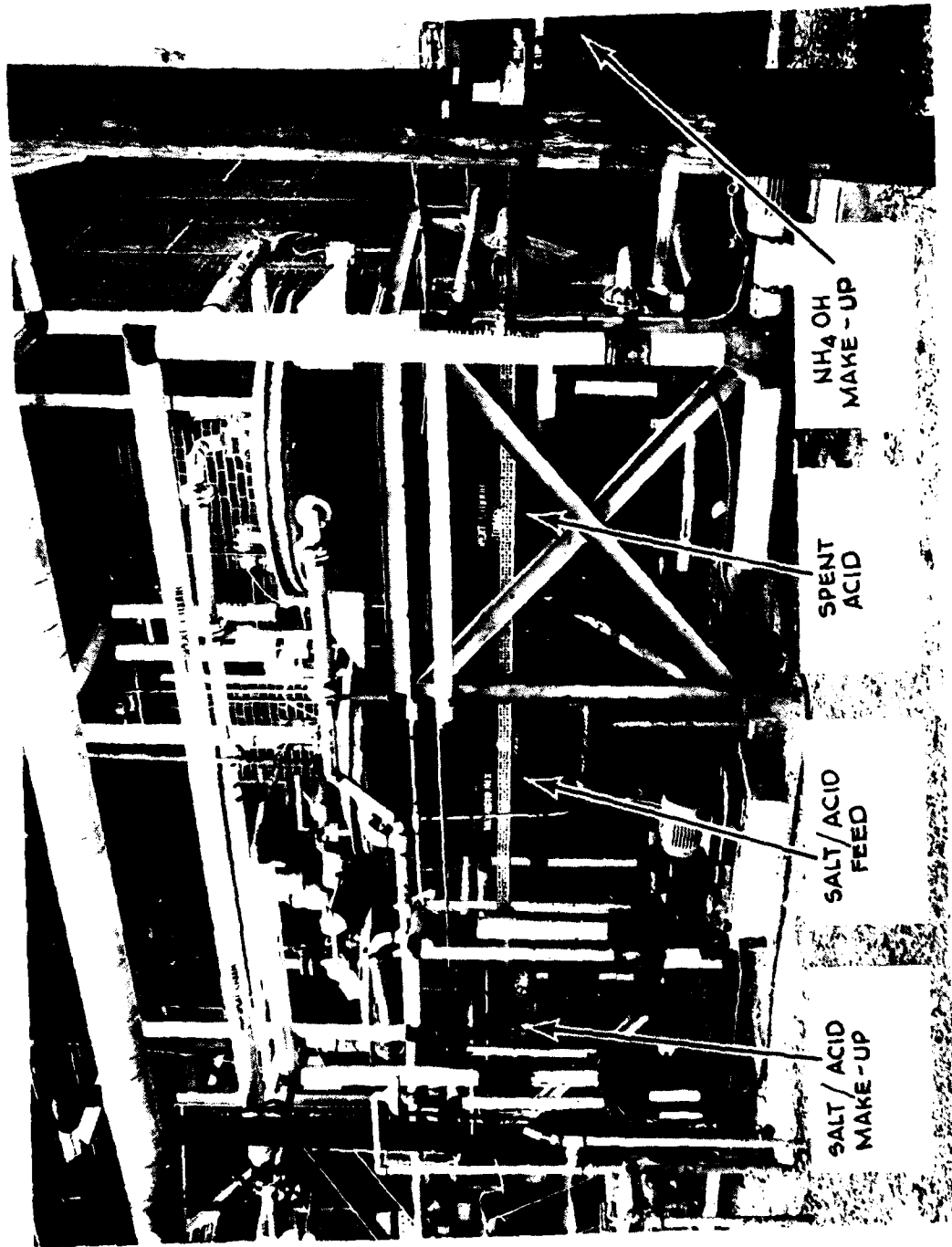


Figure 25. Acid make-up and storage tank.

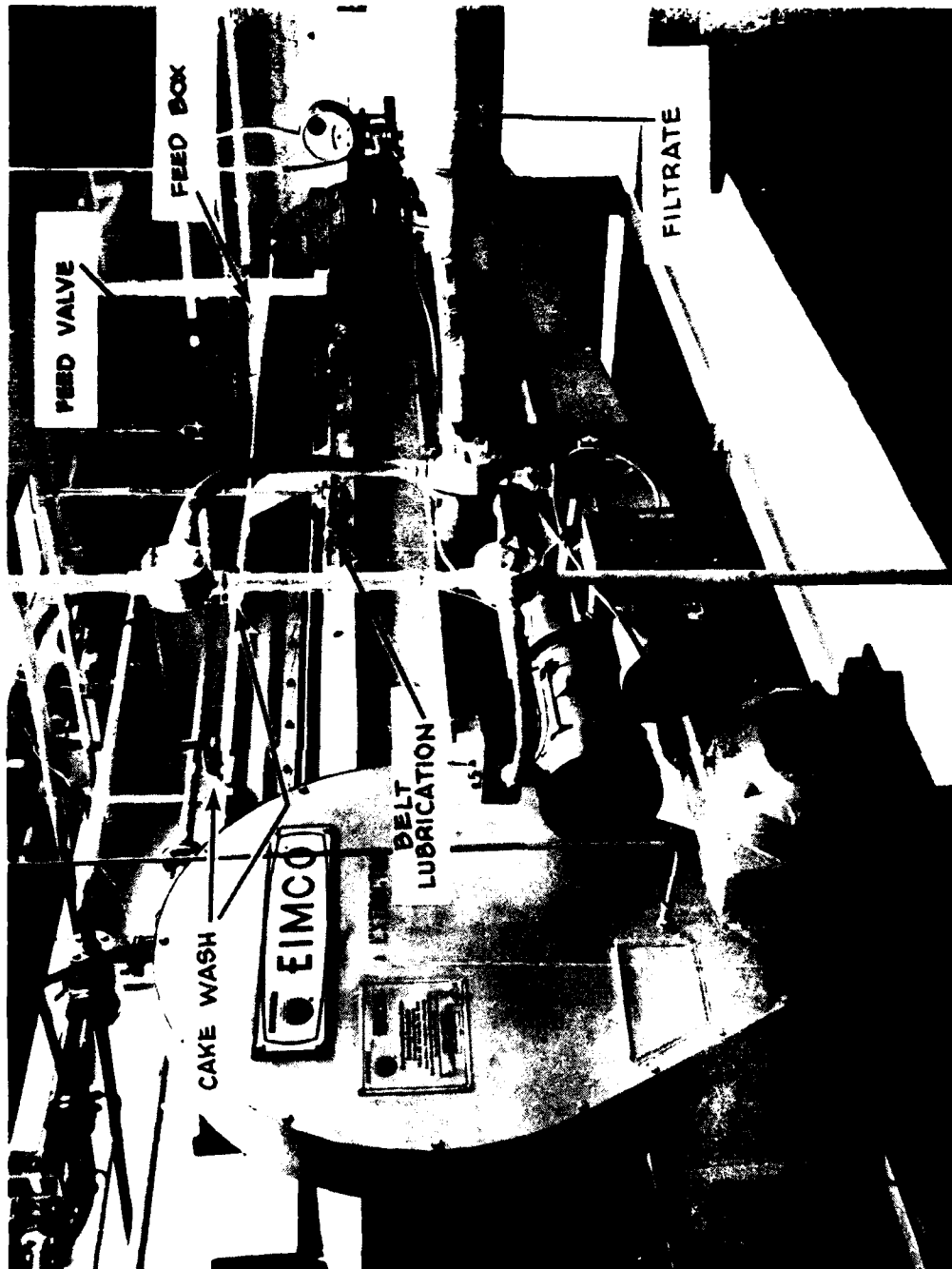


Figure 26. Dewatering filter.

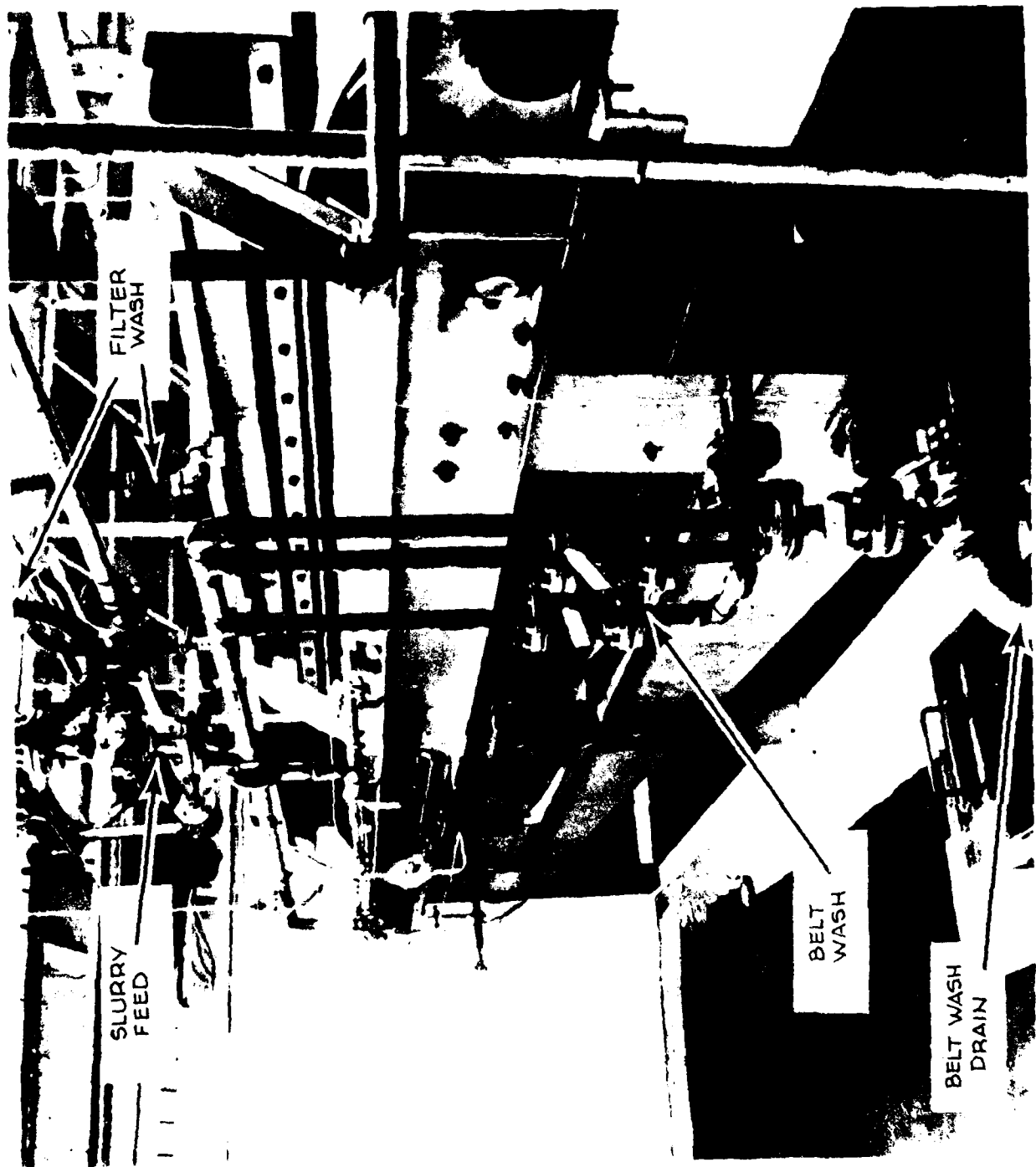


Figure 27. Dewatering filter - valve side.

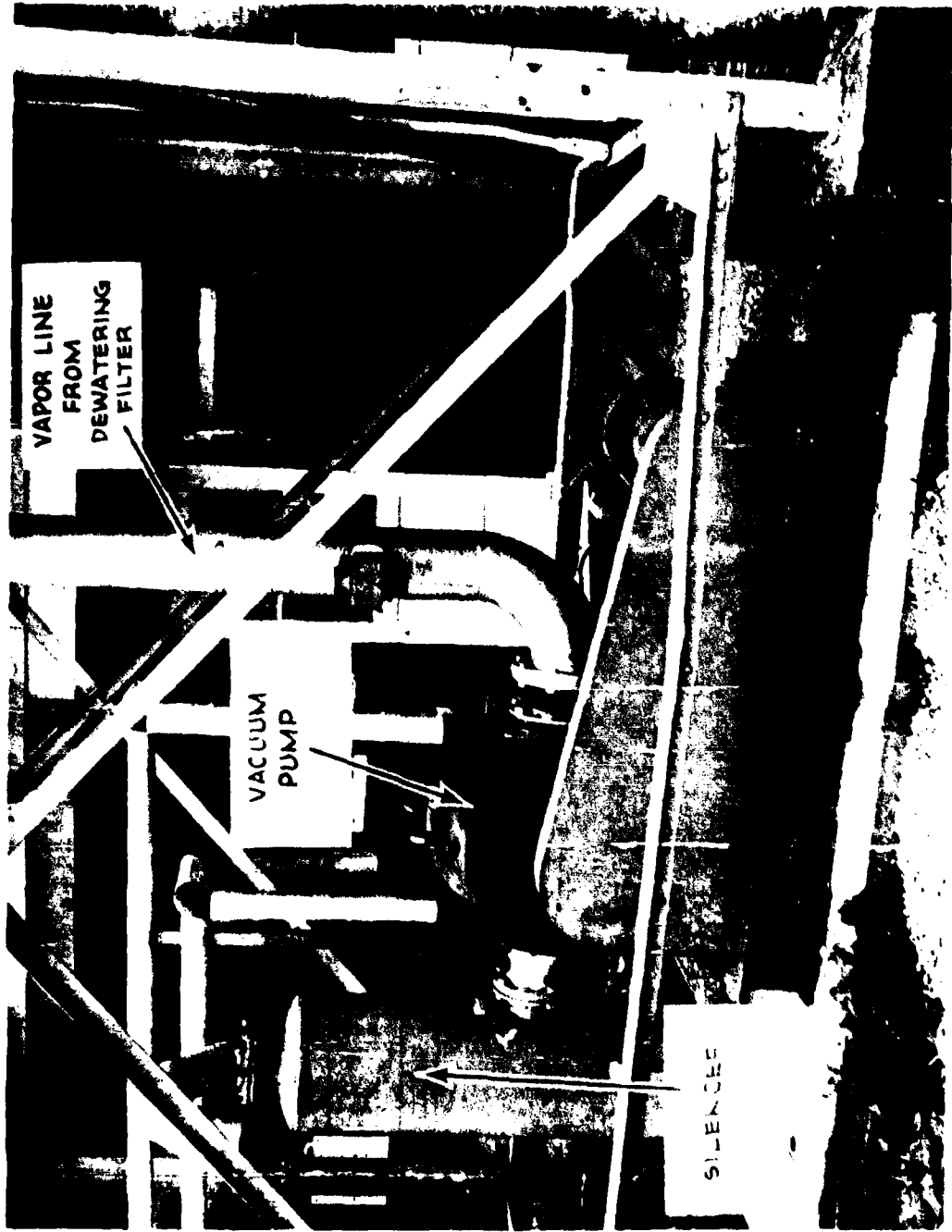


Figure 25. Hico vacuum pump.

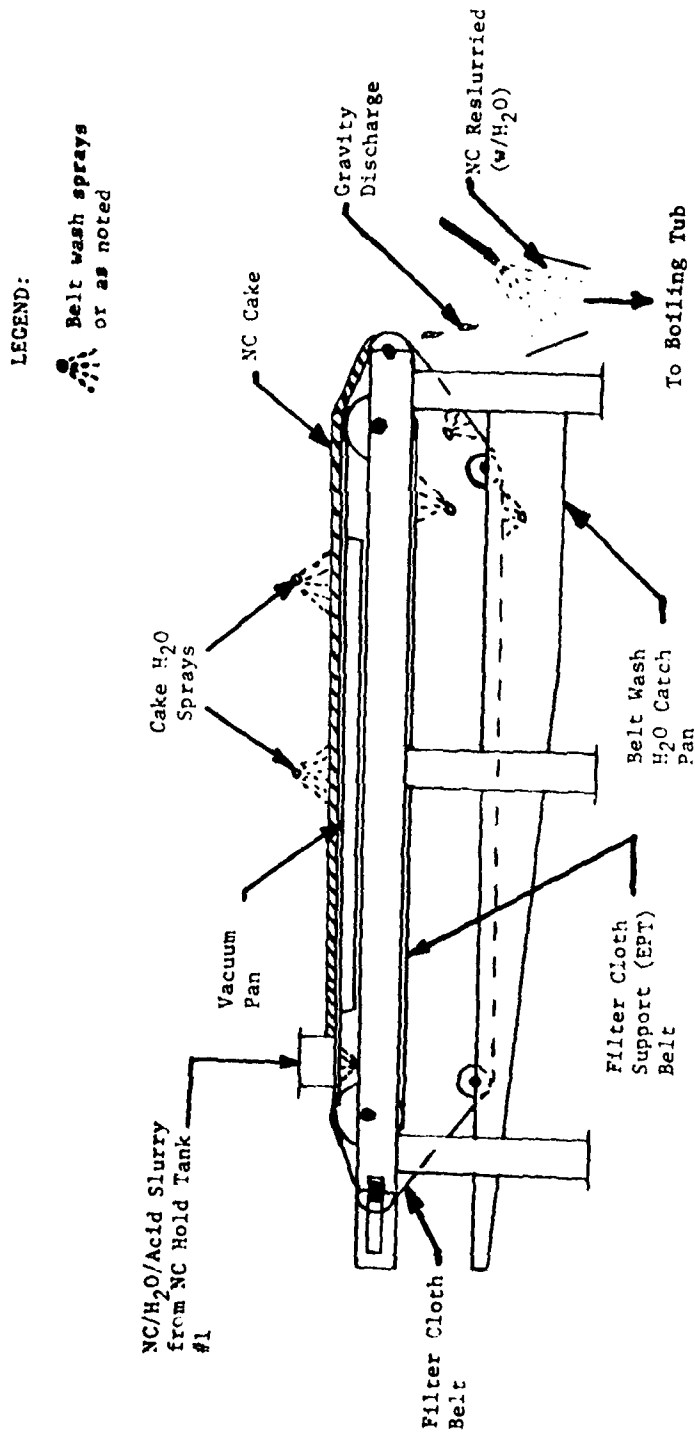


Figure 29. Eimco extractor horizontal belt filter.



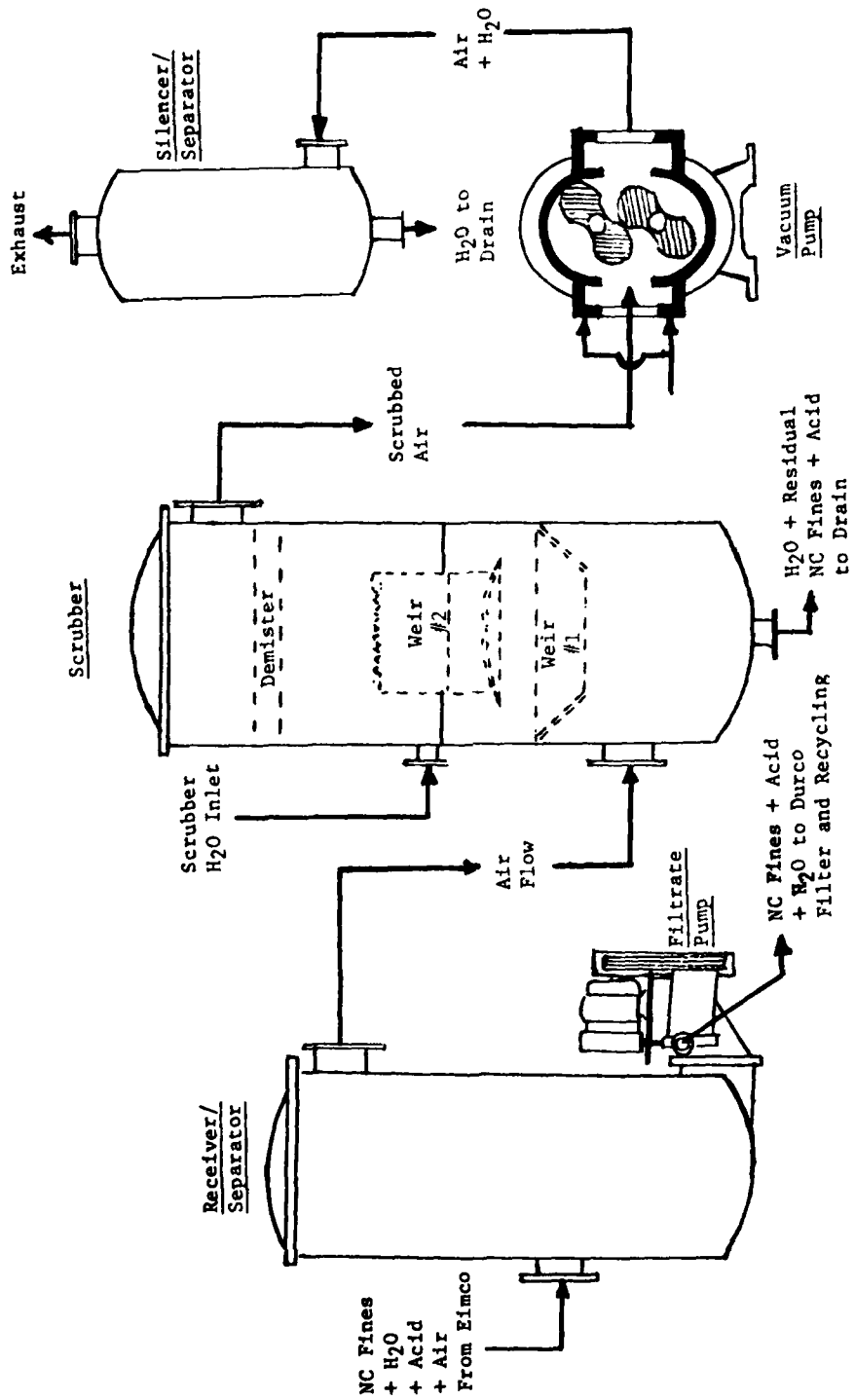


Figure 30. Eimco pollution control and vacuum system.

belt to the center of the belt, and terminate in holes through the belt. Directly beneath the center of the upper half of the EPT belt is a stationary vacuum pan which forms a seal with the underside of the moving EPT belt and is connected to a vacuum source and water/acid/NC fines collection system. Water is injected into the belt/pan interface to lubricate the sliding surfaces and improve the belt-to-pan seal. As the NC slurry contacts the filter cloth, excess acid/water are extracted from the NC by the vacuum (approximately five inches Hg) system and results in a NC cake on the filter cloth surface. As the NC cake is conveyed to the discharge end of the filter, it is sequentially washed by two banks of weak acid water sprays. This wash acid water is equal in volume and rate to the volume and rate of the feed to the first wash in the centrifuge. This wash acid water is also drawn through the NC cake by the vacuum system. The belt speed is controlled by a Reeves vari-speed motor drive and can be varied from 0.00015 to 0.121 m/s (0.03 to 23.8 ft/min). The speed is preset to conform to the rate of production of NC from the nitrator and centrifuge. At the discharge end of the filter, the NC cake is reslurried to approximately 95 percent water and gravity fed to one of five viscosity boil tanks.

The Eimco has belt separation and thorough washing on the belt return leg of its cycle. Washing prevents filter cloth binding and minimizes undesirable accumulations of NC on belt surfaces.

The acid/water/NC fines/wash water are drawn by the vacuum system through the filter cloth belt, along the channels and through the holes in the EPT belt, through the vacuum pan, and into the water/acid/NC fines collection system. The water/acid/NC fines are separated from the air (drawn through the NC cake) in a receiver tank per figure 30. The water/acid/NC fines fall to the bottom of the receiver where they are pumped by an Eimco-Krogh vane pump to a Durco filter (to remove NC fines) and recycled. The air exits from the top of the receiver and enters a scrubber. The scrubber removes entrained acid, water, and NC fines from the air before it enters a Roots water sealed (lobe type) vacuum pump and is exhausted to the atmosphere. The air enters the bottom of the scrubber and is forced (by vacuum) sequentially through two weir arrangements (which can be flooded with water) and a 100 mm (four in.) thick open pore polypropylene "demister". The water on the weirs removes entrained NC fines (by wetting) and acid fumes (by dissolution), and the demister provides extra assurance of NC fines removal from the air stream.

Safety interlocks included in the filter/collection system include: (1) a pressure switch located in the collection system which will shut off the NC hold tank No. 1 discharge valve if vacuum is lost; (2) a pressure switch in the vacuum pump will shut off the pump if oil pressure falls below 34.5 kPa (five lb /in.<sup>2</sup>); (3) an exit air temperature sensor in the vacuum pump discharge port will shut off the pump if the air temperature exceeds 64°C (148°F) (maximum pump design limitation); and (4) NC will not feed to the Eimco unless the (a) belt is running, (b) vacuum is on, and (c) wash water is on.

### Viscosity Boiling Tub

A typical viscosity boiling tub is shown in figure 31.

Construction of this tub is similar to the boiling tubs used in the production facilities with an addition of an agitator, remote operated flush bottom valve, level probe, and thermocouple probe. Nitrocellulose from the Eimco discharge enters the tank and settles on the false bottom so that the weak acid water used in slurring the NC can be drained through the bottom of the tub and pumped into the boiling tub water hold tank through a Merco and Durco filter combination for recycling to the Eimco discharge hopper. The NC fines are collected in the water/fines tank and pumped to the Eimco discharge hopper where they re-enter the process stream. After a boiling tub is loaded with the predetermined quantity of NC, the loading line is switched to another tub and the loaded tub is put on boil for the prescribed processing cycle. After the NC has received a full boiling tub cycle of treatment, a sample is taken and analyzed. If analysis results meet process criteria, the NC is pumped to either a storage tank or loaded into drums for transporting elsewhere. After treatment in the boiling tubs the NC can be pumped to the blend tank for blending prior to being pumped to a storage tank or loading into drums for shipment.

### Fines Separation and Recovery

Information pertaining to the Merco Rotary Strainer used in this operation is proprietary, Hercules Incorporated information and can be found in Volume II of this report.

The Durco filter separates NC fines from acid-water/NC fines mixtures at four different locations in the pilot plant. In addition to the three units mounted with the Merco rotary strainers mentioned earlier, a Durco unit is located in the line between the Eimco-Krogh filtrate pump on the vacuum receiver tank at the Eimco filter and the cake wash and belt wash nozzles of the Eimco filter. Acid-water filtrate is pumped to a storage facility and the NC fines are pumped back into the process at an appropriate processing stage. The Durco filter is a tubular-type, dual filtration system consisting of two complete and interconnected filtration units, as shown in figure 32. The units function alternately; while one unit is filtering NC fines the other one is automatically disposing of NC accumulations on the filter. Each unit consists of two basic stainless steel parts; the outer housing and a removable, reusable filter element. The filter element consists of a perforated stainless steel basket which is inserted into, and serves as a support for, the woven Teflon filter bag [septum-designed for the retention of particles of  $280 \times 10^{-6}$  m (280 microns) or greater; stainless steel screen bags are also used interchangeably with the Teflon bags]. The bag is held in place with stainless steel bands secured by a simple hinged tab and channel locking arrangement. The basket is manually inserted into the outer housing and is held in position by a flared lip at the top of the basket which rests upon a mating surface in the housing.

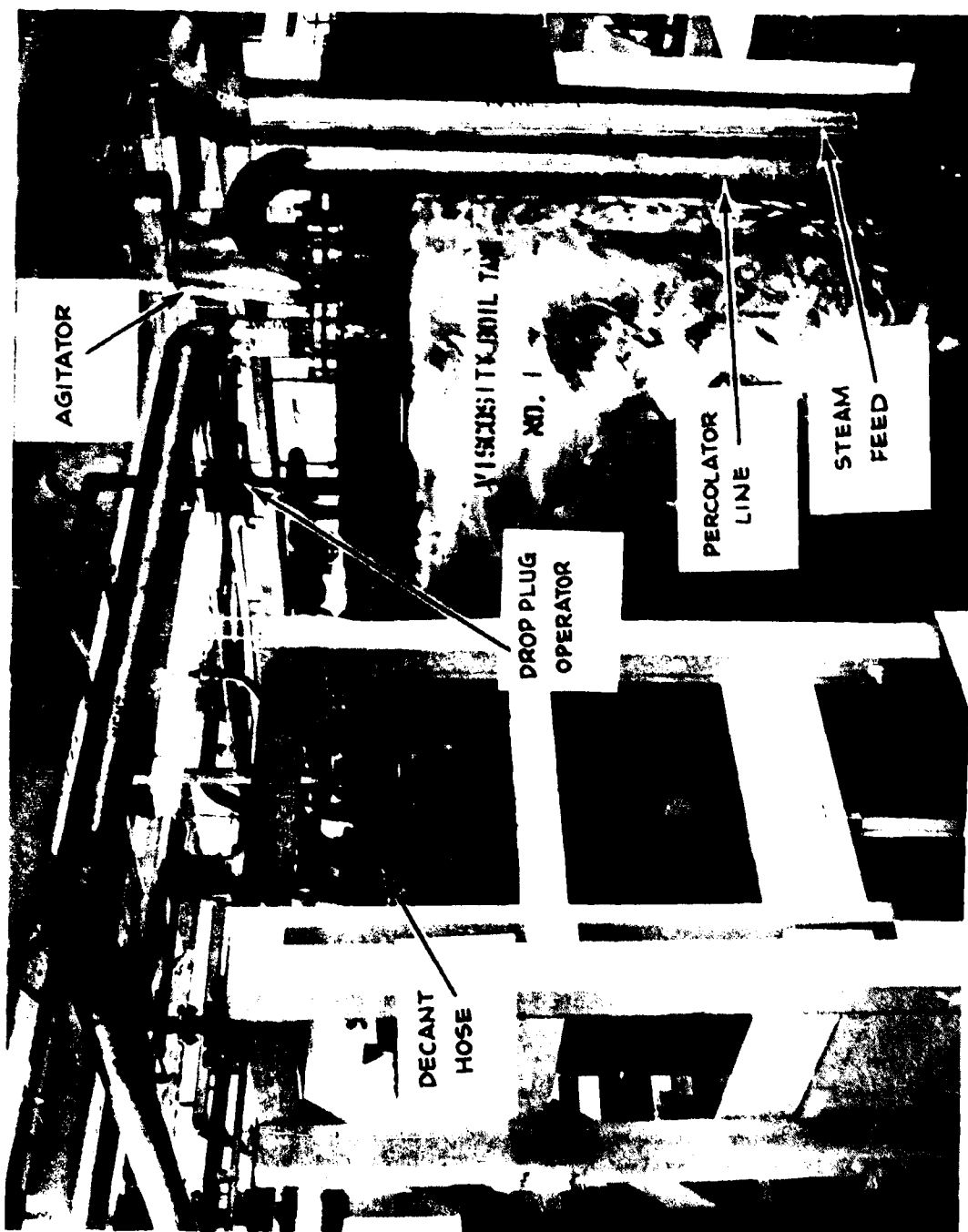


Figure 31. Viscosity boiler sub no. 1.

An O-ring (Teflon and Viton O-rings are used interchangeably) is provided between the basket and housing surfaces, which assures a leakproof seal to 1380 kPa (200 lb / in<sup>2</sup>) and forces all filtrate to pass through the septum to reach the outlet port. Pressure taps are located above and below the O-ring seal to measure the pressure differential across the septum. An increase in pressure differential indicates that an NC cake is building up on the filter and is increasing the resistance to acid-water flow (fig. 32 - Filter No. 1 schematic). When the pressure differential reaches a preset level, a pressure switch trips and automatically (1) diverts the incoming acid-water/NC fines to the other filtration unit, and (2) begins a series of two shock backwash cycles to remove NC cake from the septum surfaces and out of the top of the unit. Each shock backwash cycle consists of (a) permitting entry of filtrate into the top of the unit (through valve 2 shown in the figure) to pressurize the air trapped in the air space in the unit and above the filtrate outlet line; (b) further pressurizing the filtrate in the unit by introducing pressurized air into the air space; and (c) opening valve 3. When valve 3 is opened, the pressurized filtrate and air rapidly forces its way through the septum, dislodges NC cake from the septum surfaces, and carries the broken NC cake pieces from the unit to an acid/NC fines storage tank (fig. 33) and subsequent recycling (fig. 32 - Filter No. 2 schematic). After two backwash cycles, the cleaned filter is inactive until another entire cycle (filtering and cleaning) is repeated. The above description outlines the automatic operation of the Durco filter. Manual operation of the Durco filter can be utilized for removal of NC fines from the decanted water from the viscosity boiling tubs. The control panel for operation of this filter contains a hand and automatic switch and a manual discharge switch for each of the two filter tubes installed as one unit for continuous operation. The other Durco filter installations can also be operated either manually or automatically.

#### Attrition Mills and Screw Feeder

Cellulose mixed with salt/acid will be fed sequentially through two modified 610 mm (24 in.) diameter Sprout-Waldron attrition mills to (1) reduce the cellulose fiber length to desired levels, and (2) achieve good salt/acid-to-cellulose contact for the nitration reaction (nitration will begin in the attrition mills). NC/water slurry may also be pumped from NC hold tank No. 1 and fed (not concurrently with cellulose/salt/acid) through the mills when desired to achieve further reduction in NC fiber length. Each mill consists of a stationary and rotary set of stainless steel plates. Each set of plates consists of six ground and balanced plates which, when assembled, form an annulus 610 mm (24 in.) in diameter with a 356 mm (14 in.) diameter hole. Each set has open channels radially machined on the stationary-to-rotary sets mating surfaces which serve to grind the cellulose or NC as it passes through the mills. The gap between the grinding plates is both manually adjustable and spring-loaded to prevent plate damage in the event that a metallic (or hard) foreign object enters the gap. Entry of salt-acid-cellulose or NC/water slurry into the first (stage) attrition

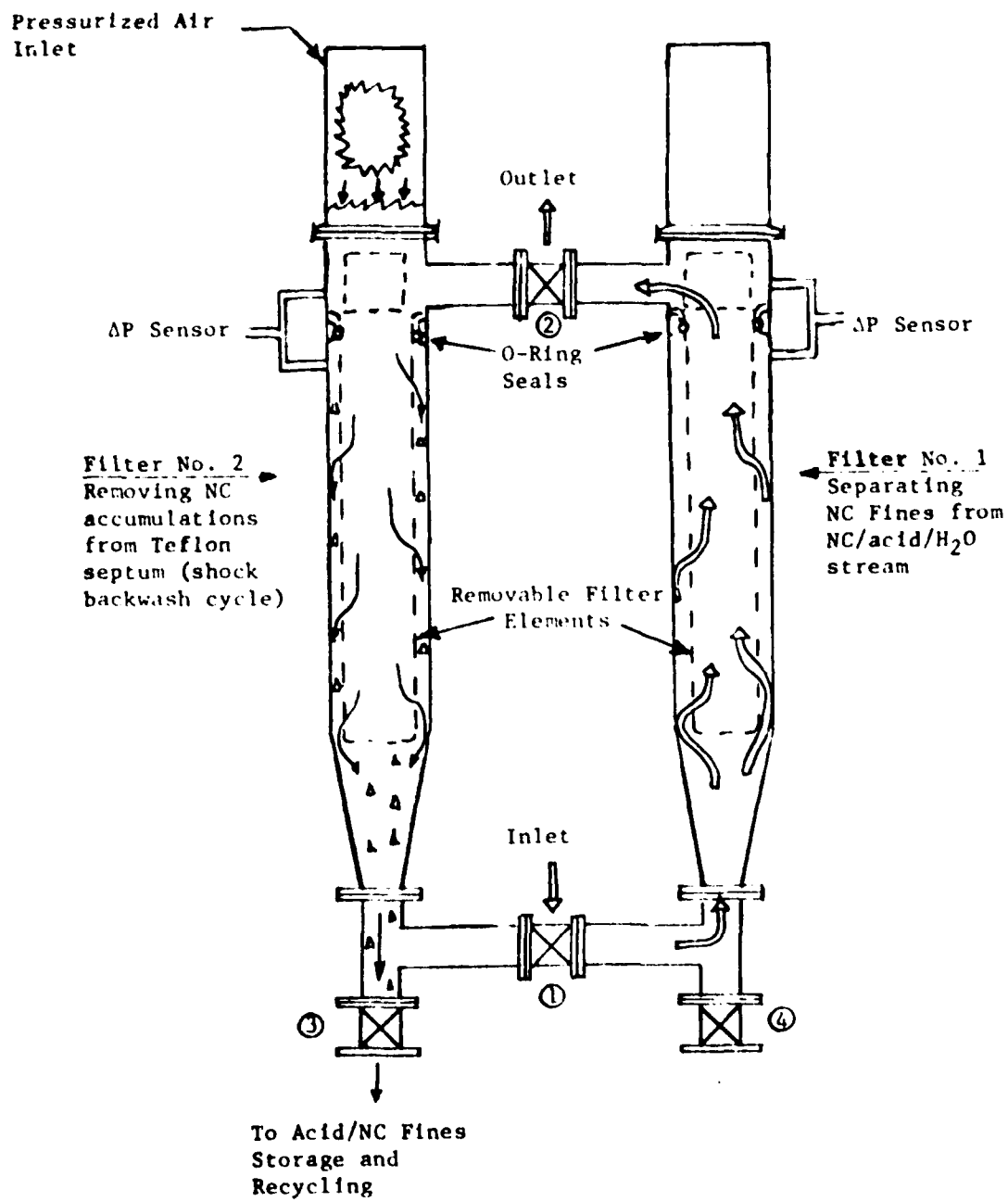


Figure 32. Durco dual tubular filter.

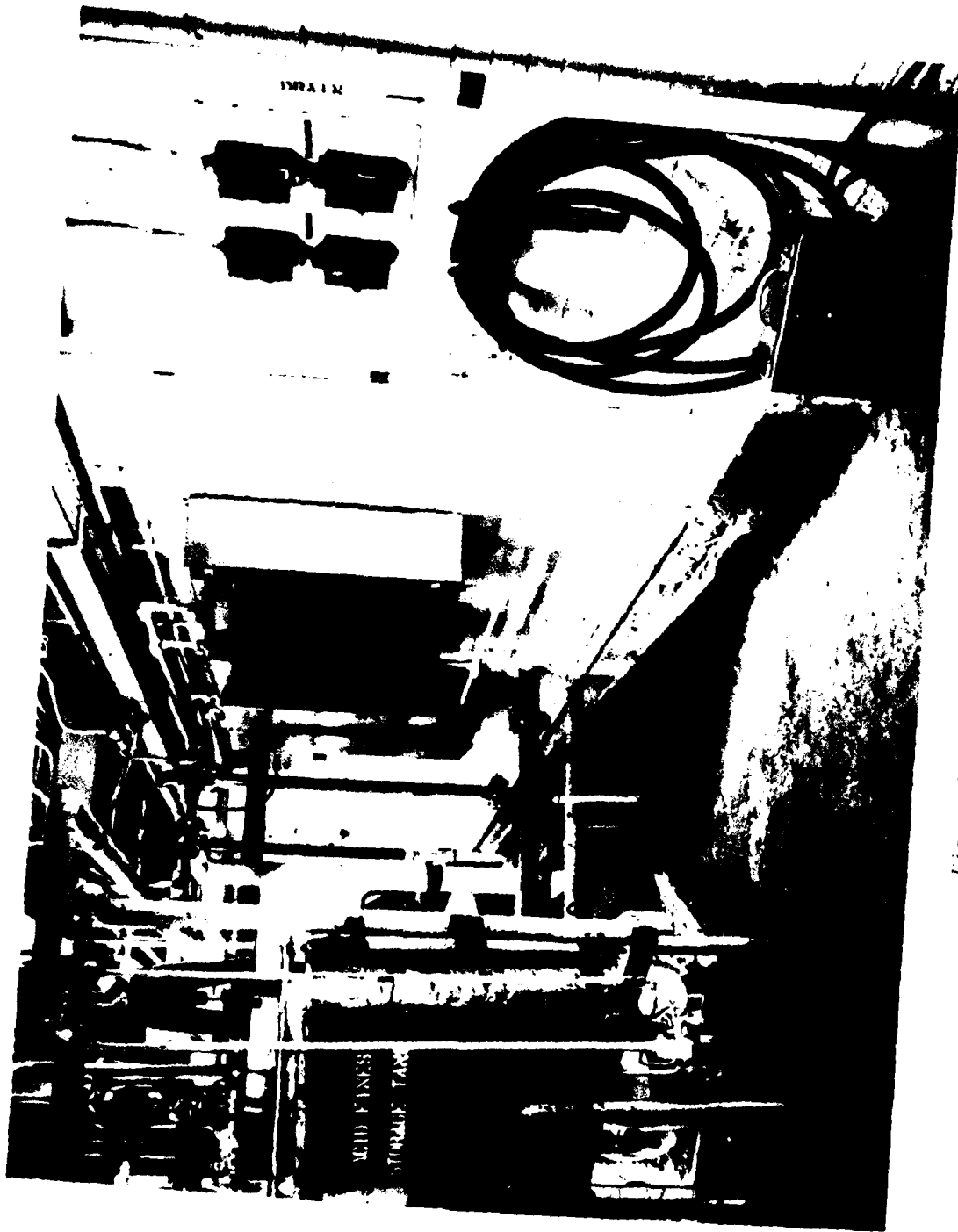


Figure 33. Acid fines storage tank.

mill is accomplished by a screw conveyor forced feed and the centrifugal force of the rotary grinding plates. Feed through the second (stage) attrition mill is by gravity and centrifugal force (fig. 21) NC is prevented from entering the bearings by Teflon-packed glands provided with lantern rings and flushed with a salt/acid solution (when salt-acid-cellulose is being processed) or water (when NC/water is being processed). Ground, partially nitrated salt-acid-NC flows by gravity into the nitrator and ground NC/water flows (by gravity) to NC hold tank No. 2.

### Ion Exchange System

Two two-bed ion exchange systems are used in the magnesium nitrate process. One small system is used to deionize filtered river water for use during processing, and a larger one is used to deionize process water so that it can be reused. The basic differences between the two systems are the tank (resin bed) sizes and type of ion exchange resins used.

The filtered water (makeup) deionizing unit (fig. 34) consists of two 406 mm (16 in.) diameter by 1,829 mm (72 in.) high polyvinyl chloride (PVC) lined reactor tanks with the necessary piping (PVC), valves, resin, and control elements needed to make up a working unit. The cation exchange tank contains 0.15 m<sup>3</sup> (5.3 ft<sup>3</sup>) of IWT C-211 resin and the anion exchange tank contains 0.11 m<sup>3</sup> (4.0 ft<sup>3</sup>) of IWT A-364 resin (IWT - Illinois Water Treatment Company). The resins are held in place by a quartz support bed. During normal operations, filtered river water containing dissolved mineral salts enters the cation exchanger first where sodium (Na<sup>+</sup>), calcium (Ca<sup>++</sup>) and magnesium (Mg<sup>++</sup>) ions are taken up on the cation resin and causing hydrogen (H<sup>+</sup>) ions to be displaced from the resin. This exchange, in effect, converts the mineral salts that were originally in the water to their corresponding acids. The acid water then passes into the anion exchanger where nitrate (NO<sub>3</sub><sup>-</sup>), sulfate (SO<sub>4</sub><sup>=</sup>) and chloride (Cl<sup>-</sup>) ions are displaced with hydroxide (OH<sup>-</sup>) ions. This exchange completes removal of the common minerals found in raw water supplies and the by-product is more deionized water (H<sup>+</sup> + OH<sup>-</sup> → H<sub>2</sub>O).

The process water deionizing unit (fig. 35) consists of two 760 mm (30 in.) diameter 2,438 mm (96 in.) high PVC lined reactor tanks and auxiliary equipment. The cation exchange tank contains 0.54 m<sup>3</sup> (19 ft<sup>3</sup>) of IWT C-381 (Amberlite 200 Rohm and Haas) resin and the anion exchange tank contains a like quantity of IRA-47 resin (Rohm and Haas). Ion exchange in this unit is accomplished in the same manner as in the filtered water deionizing unit (discussed above), except that Mg<sup>++</sup> and NO<sub>3</sub><sup>-</sup> ions are the principal ones removed from the process water (fig. 36).

A Dynatrol density sensing unit (fig. 37) monitors the density (i.e., acid concentration) of the incoming process water to assure that unusually high acid concentrations do not enter the exchanger. High acid concentrations may degrade the ion exchange resin(s). When the Dynatrol unit detects an unusually high acid content in the process water, it automatically adds deionized water to the process water stream to dilute it to an acceptable level using the mixing system shown in figure 37.



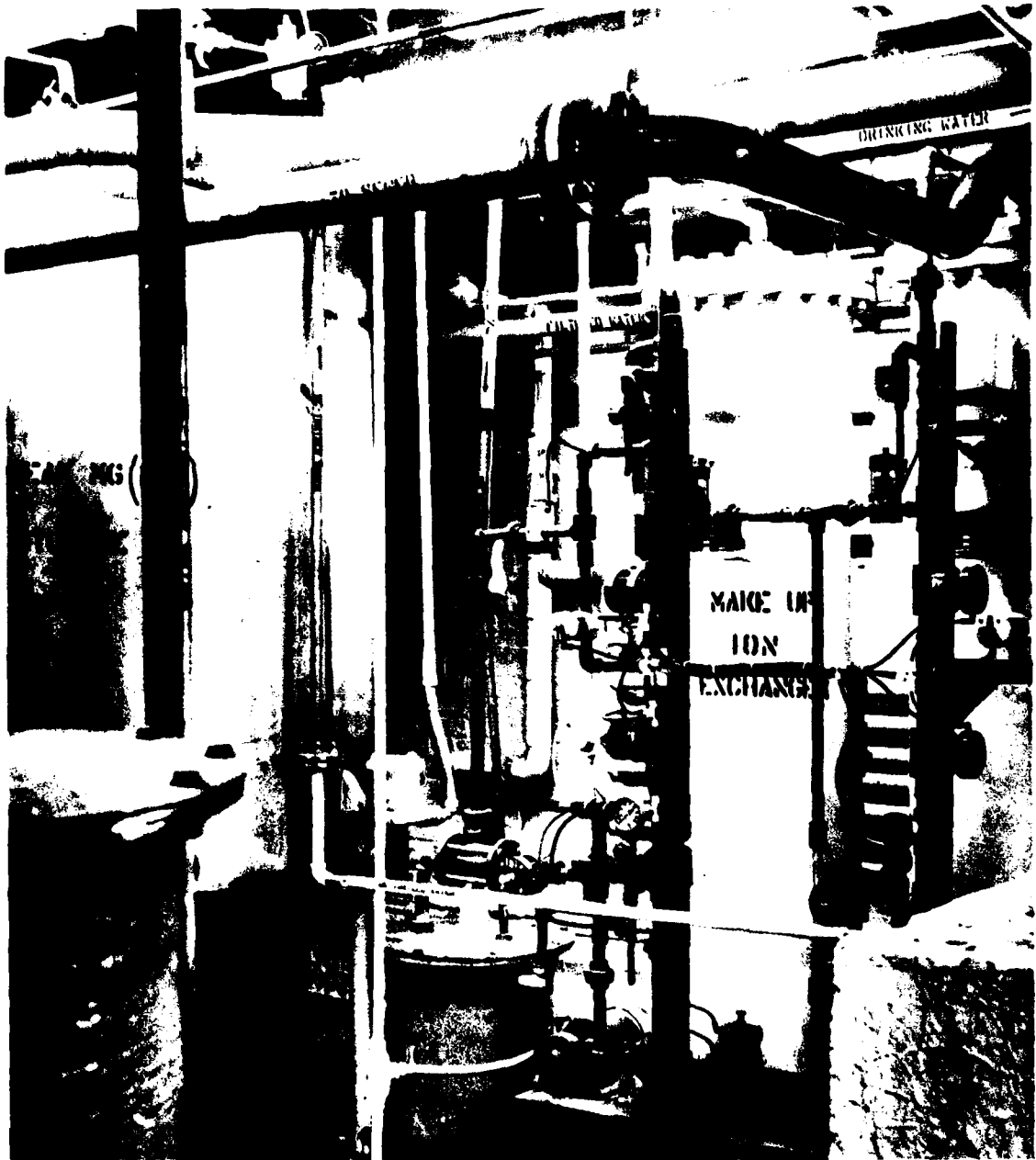


Figure 34. Make-up ion exchanger.

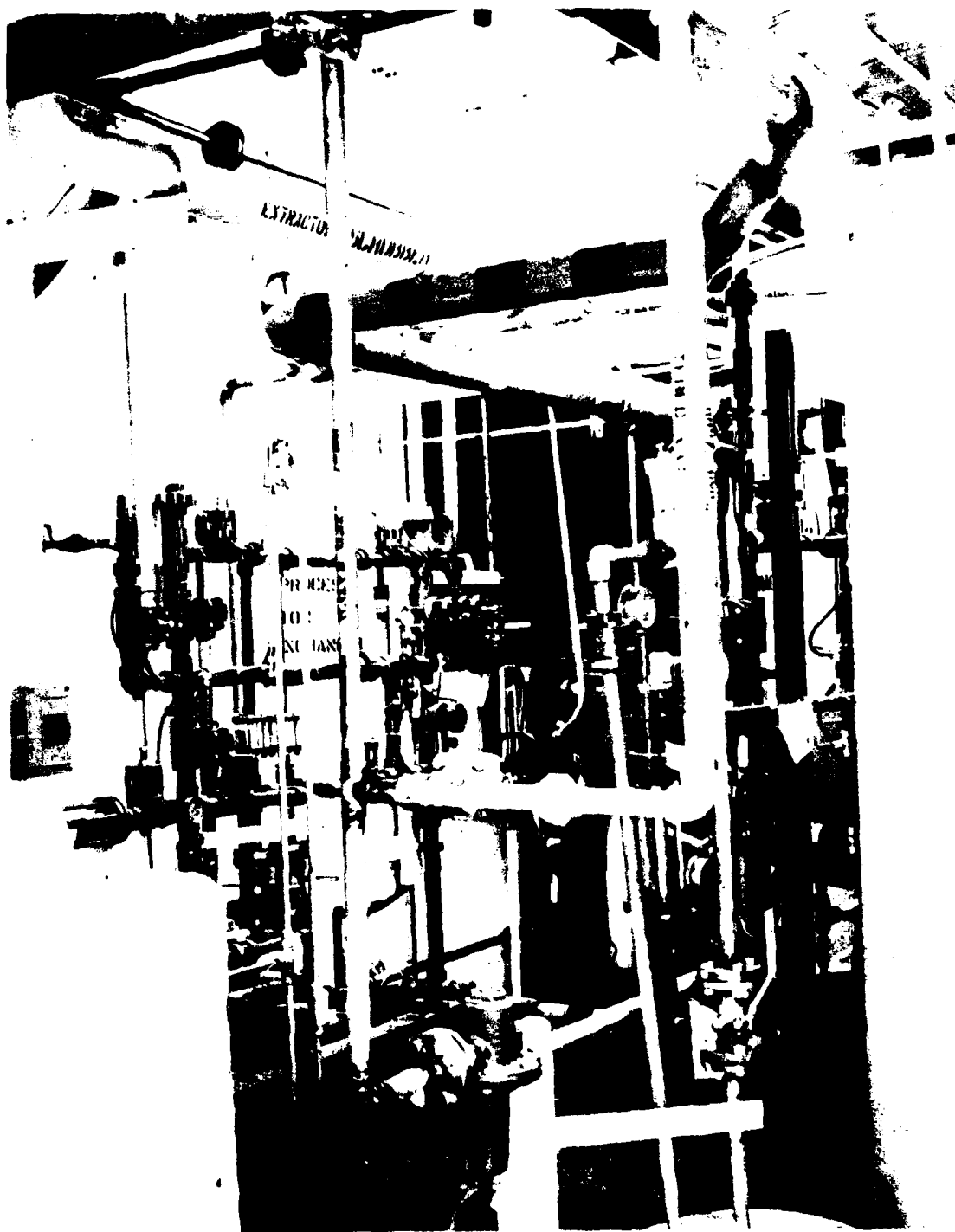


Figure 35. Process ion exchanger.

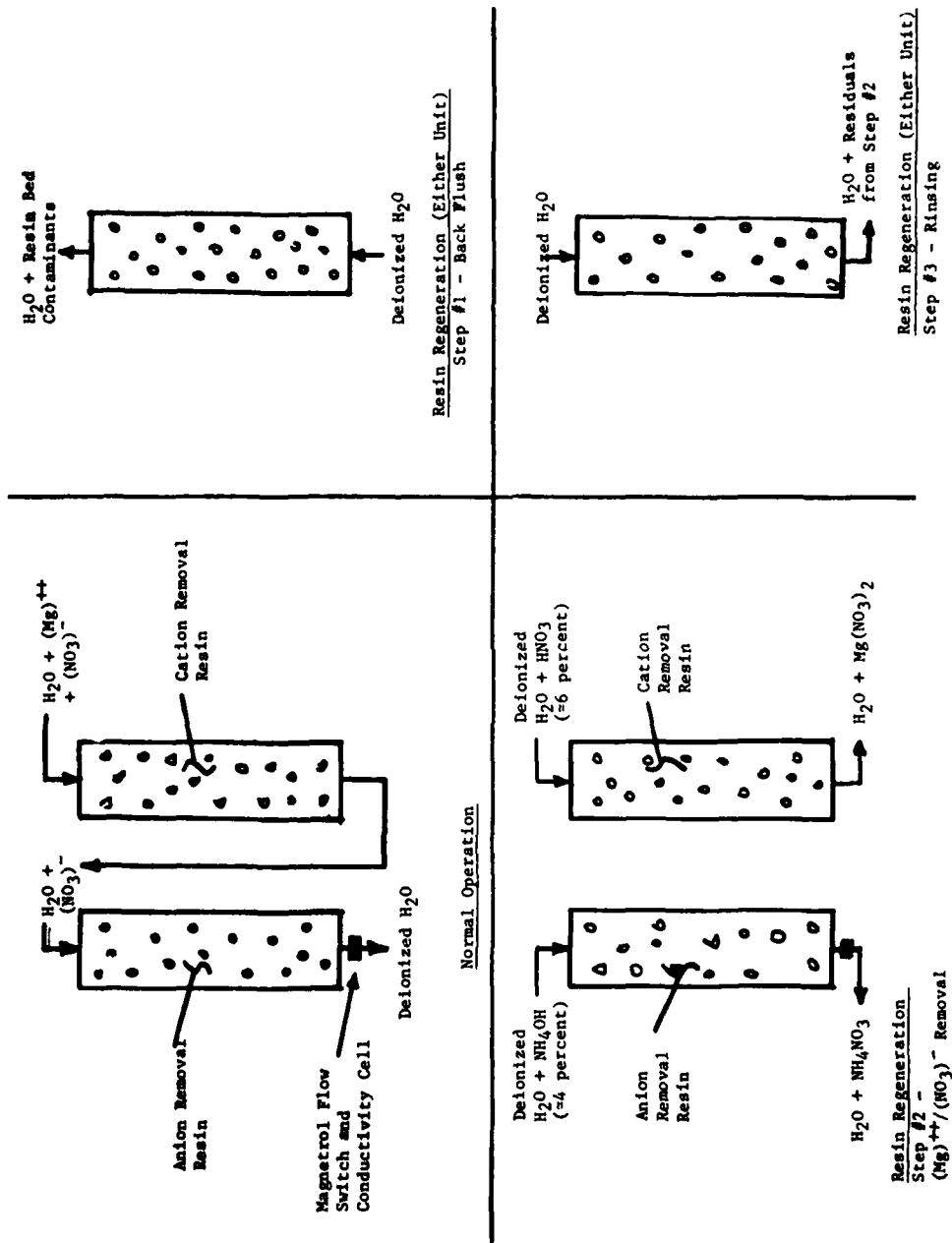


Figure 36. Ion exchange system.

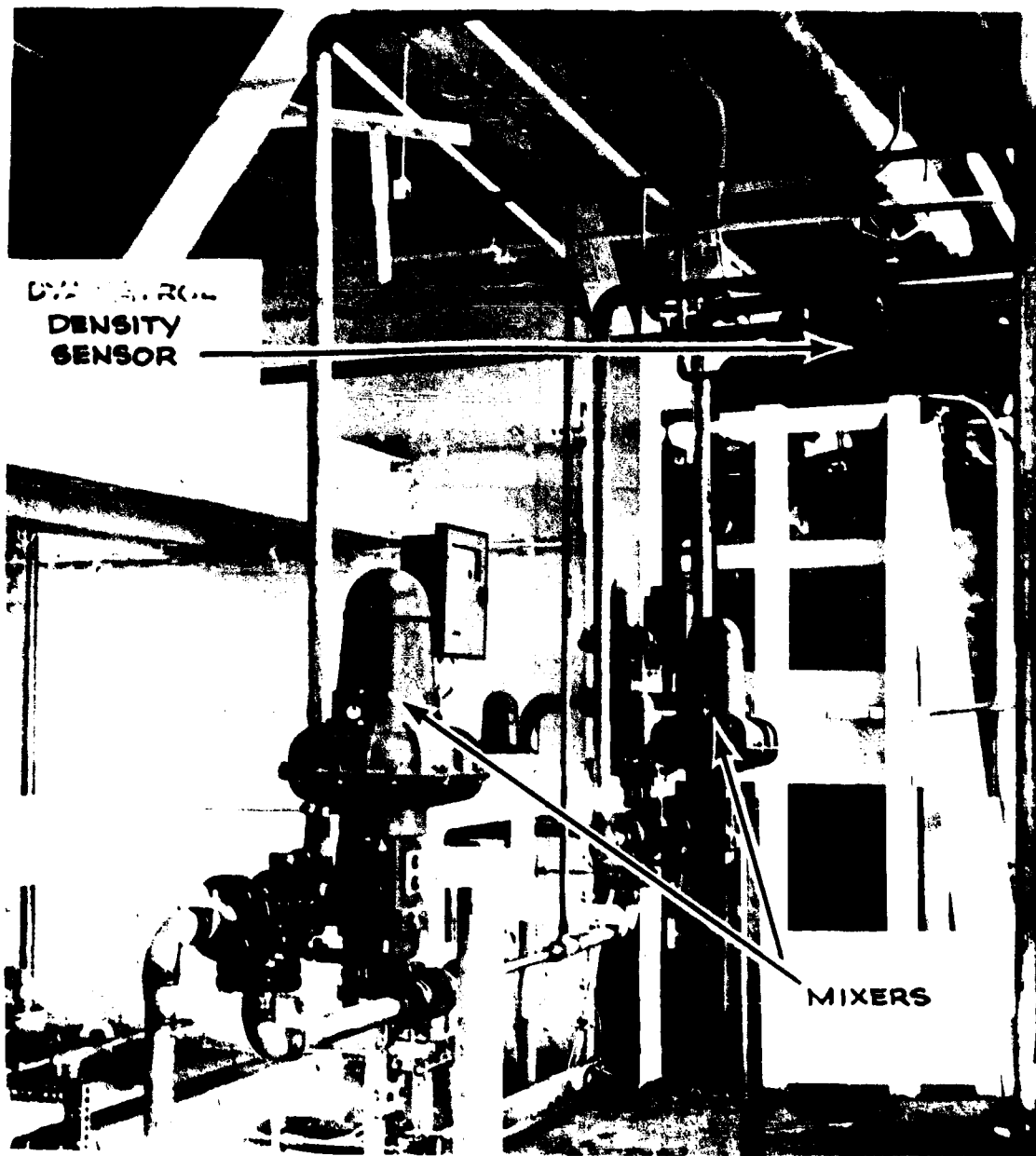


Figure 37. Process ion exchange feed mixing system.

Both ion exchange systems use nitric acid to regenerate the cation resin and ammonium hydroxide to regenerate the anion resin. When the resins need regeneration,  $\leq 30$  percent nitric acid or ammonium hydroxide solution are withdrawn by suction from storage tanks, diluted to approximately six percent solutions, and injected into the appropriate reactor circulation system by means of a Penberthy PVC ejector. Regeneration includes (1) a backwash cycle, (2) nitric acid (or ammonium hydroxide) entry for regeneration, (3) a slow rinse cycle, and (4) a fast rinse cycle (fig. 36).

System control for both units includes a pressure regulator and gages, water meter, conductivity cell, and magnetic flow switch. The conductivity cell continuously monitors water conductivity which is directly proportional to the ion concentration. When the conductivity (ion concentration) increases beyond a set point value, an alarm is sounded alerting the operator that the resin beds require regeneration (which is initiated manually or automatically). The magnetic flow switch detects a no-flow situation due to a plugged bed, broken line, etc. and (1) activates an alarm which alerts the operator that corrective action is required, (2) turns off the incoming water to the deionization unit, and (3) turns on the recirculation pump to maintain the resin in an operational condition until normal operations can resume.

#### Instrumentation and Controls

##### a. Control Room Layout

Consideration was given to the several processes that were visualized to be proceeding simultaneously when the control room instruments were laid out. Motor control relays and electrical breakers for the individual motors were placed in one room adjacent to the control room.

The manufacturing operations were divided into four categories and appropriate instrumentation was placed in consoles: (1) acid mix and storage; (2) nitration; (3) purification; and (4) concentration. Several other control panels for specific equipment were located on the structural walls around the room.

Figure 38 shows the general layout of the control panels in a horseshoe arrangement. The console on the left is devoted to acid mix and storage operations. The center panel is for control of nitrator feed, attrition mill operation, nitration, centrifuge operation, and water slurry of the centrifuged NC. The console on the right contains controls for the Eimco dewatering filter, boiling tubs, blend tank, NC storage, and ion exchange.

Figure 39 is a closeup of the left console for acid mix and storage. Across the top of this figure are readout gages for the four weigh tanks in the pilot plant. These are (left to right) the magnesium nitrate preparation tank, the salt/acid mix tank, the 60 percent nitric acid makeup tank, and the spent acid

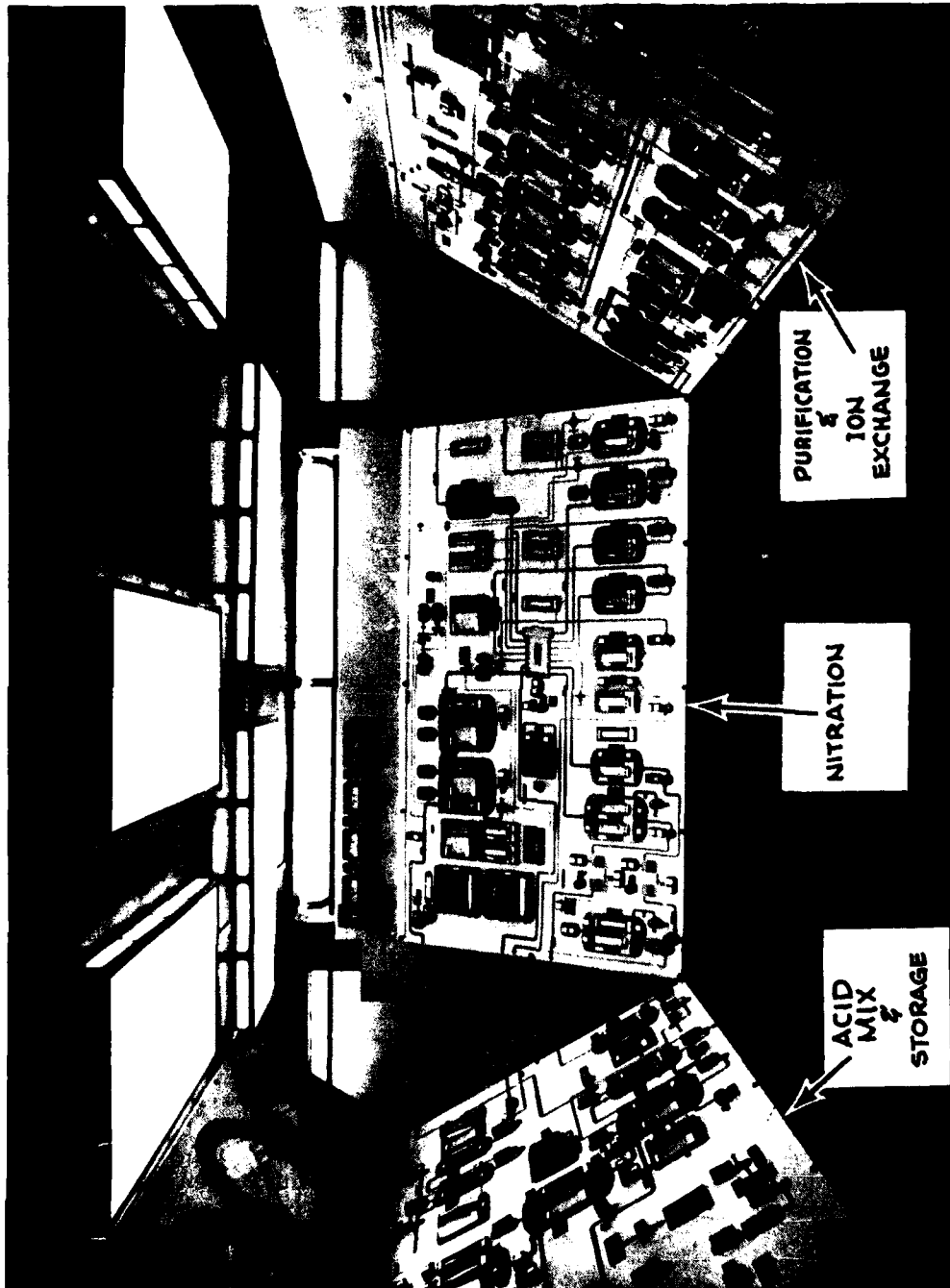


Figure 38. Control panel layout.

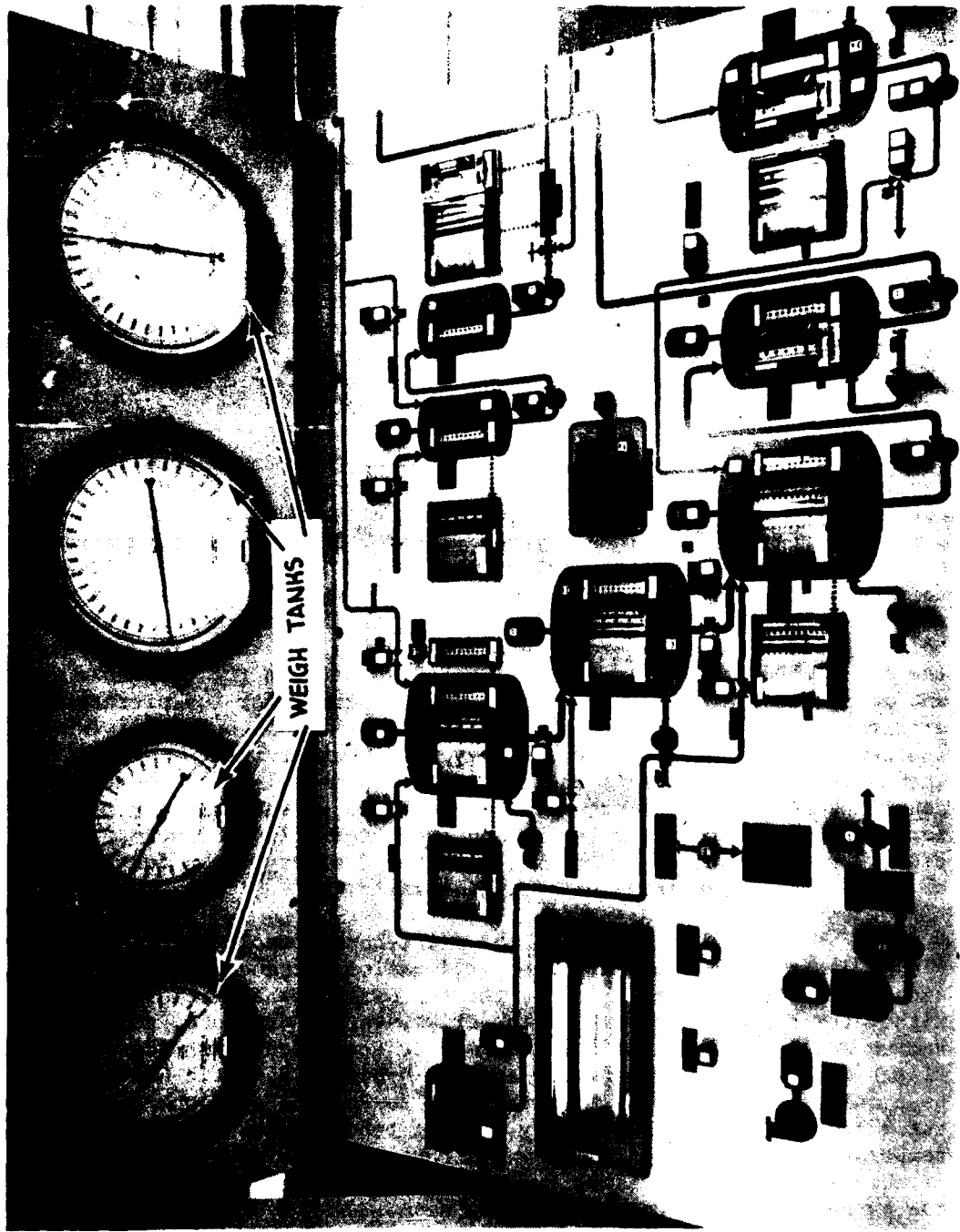


Figure 39. Acid mix and storage control panel.

tank. At the top left of the main panel is the 98 percent nitric acid storage tank with high and low level alarms, level indicator, and transfer pump. At the top center is the magnesium nitrate preparation tank with associated temperature and level controls, remote operated feed and dump valves, and agitator control. At the top right is the 60 percent nitric acid makeup tank and process feed tank with associated controls, remote operated valves, agitator, and pumps. Also, in this cluster is the control for the wash acid chiller for the fourth or spent acid wash in the centrifuge. Across the center of the panel a multipoint temperature recorder is at the left, with the magnesium nitrate storage tank and the alarm system for this panel. An alarm on any of the equipment controlled on this console will result in an audible alarm and flashing light from the alarm point. Acknowledgement will silence the alarm, but the light will remain lit until correction has been made. Across the bottom of the panel can be seen the system exhaust fan at the left, followed by the refrigeration brine makeup, refrigeration unit, salt/acid mix, salt/acid storage, and spent acid tanks with associated controls, remote valves, and pumps.

Figure 40 is a closeup of the center console for control of the nitration operations. Feed rate, level, and temperature controls are located on the schematic layout as are remote valves, pumps, and agitators. The nitrator agitator load meters may be seen above the top of the main panel at the left. These meters are designed to warn of an overload in the nitrator or loss of an impeller or shaft. In the event of these conditions, the agitators are shut down, resulting in feed stoppage of both cellulose and acid. Across the top of the panel from left to right can be seen the cellulose and acid feed controls, ratio controller controls for the nitrators, attrition mills, slug feeder, and chiller for the second acid wash in the centrifuge, and centrifuge slurry and countercurrent wash controls. The center of the panel is composed of the panel alarm and centrifuge controls. Across the bottom of the panel can be seen the controls for the NC fines receiver, spent and recovered acid receivers, centrifuge interstage wash tanks, and the NC slurry tanks for receipt of NC from the centrifuge or attrition mills.

Figure 41 is the right-hand console depicted in figure 38 and is essentially the purification, product, and ion exchange control center. Across the top of the panel from left to right, the Eimco dewatering filter drive, vacuum pressure, receiver, vacuum scrubber, and pump controls may be seen along with the dewatering filter wash water flow controls and the Dynatrol Specific Gravity Loop calibration controls. On the next row may be seen the temperature recorders for the five viscosity boiling tubs with associated agitator, temperature, level, and remote valve controls. At the right-hand side of the same row the NC fines tank and filters, along with the boiling tub water hold tank controls, may be seen. On the bottom row the ion exchange unit indicators plus feed and product tank controls may be seen next to the NC blend tank and NC storage tank controls. The alarm



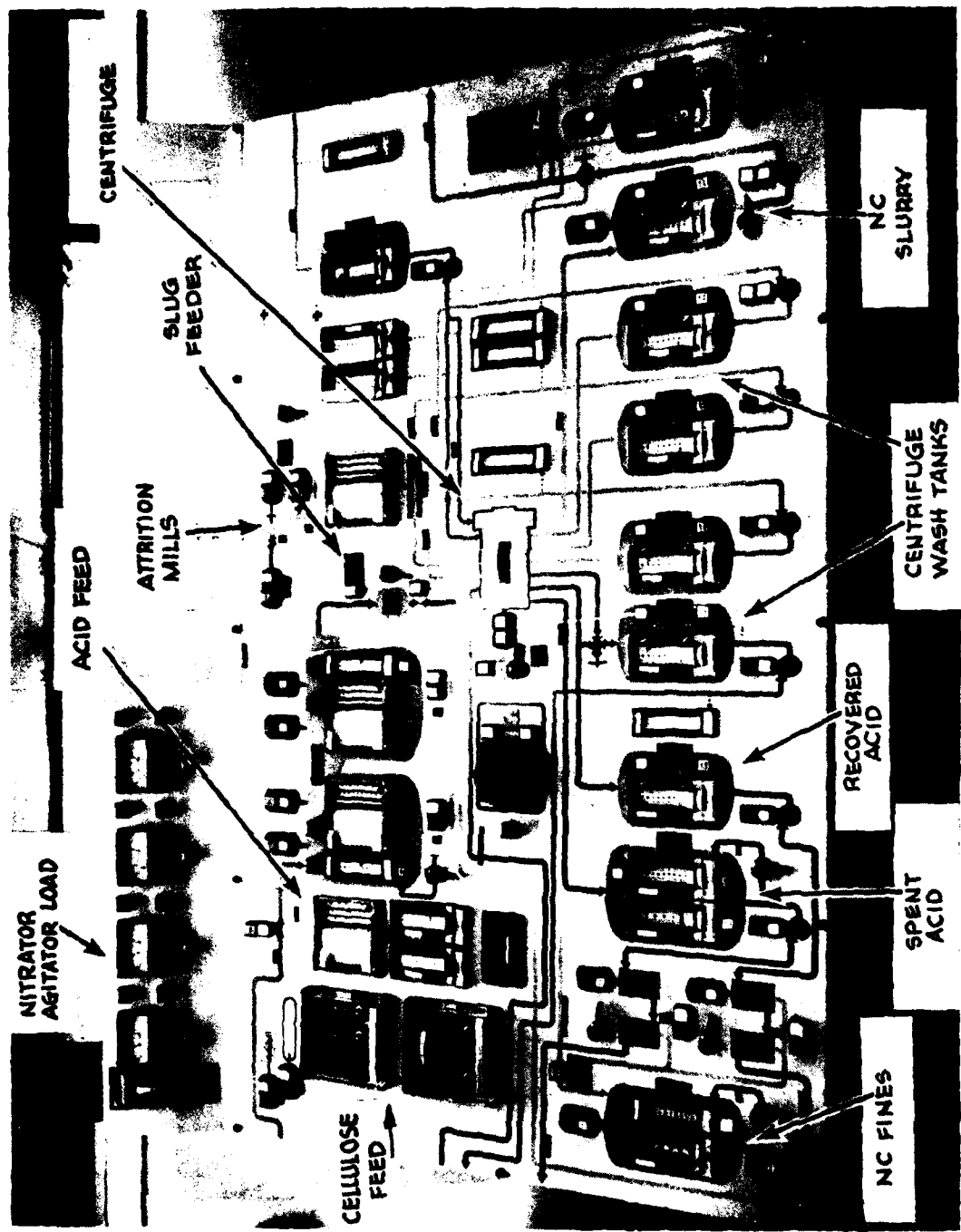


Figure 40. Nitration control panel.

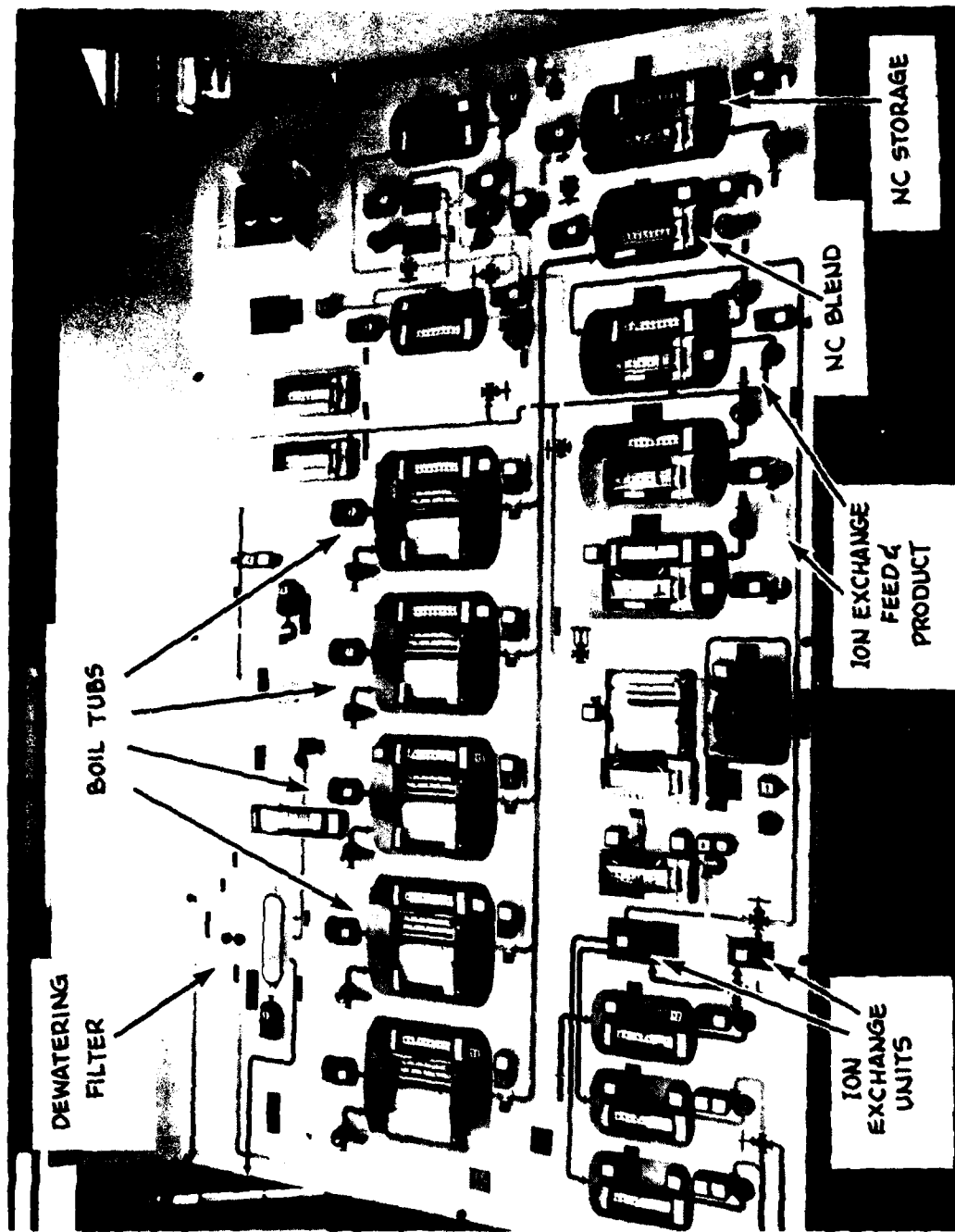


Figure 41. Purification and ion exchange control panel.

system for the purification section is located at the bottom center of this panel. Several of the ion exchange process timer controls may be seen in the background at the upper right-hand corner of figure 41. These controls are shown in detail in figure 42. Ion exchange equipment is discussed in another section of this report.

The concentrator instrument console was placed to the left of the acid mix and storage console. The size of this unit is the same as that of the other three consoles. All of the instrumentation was not placed in this unit because the program was terminated prior to completion of the concentrator section of the pilot plant.

#### b. Level Detection Systems

Several fundamental types of level detection systems were utilized on this project.

(1) Visual determinations were made of such things as height of cellulose in the cellulose feed hopper, coverage of the cellulose feed belt, slurry flow through the dewatering filter discharge hopper, and height of cake on the dewatering filter.

(2) Diaphragm Pressure Sensor - A pneumatically operated pressure transducer is located near the bottom of the applicable tank and is isolated from the tank contents by a stainless steel diaphragm. As the height of the tank contents varies, the contents pressure at the bottom of the tank varies directly. This pressure is transmitted via the diaphragm to the pressure transducer which, in turn, converts the measured pressure into a 21 to 103 kPa (3 to 15 lb /in.<sup>2</sup>) pneumatic signal. The signal pressure varies directly as the contents pressure (or contents level) and is used to operate a material height indicator and alarm.

(3) Differential pressure probes and suitable pneumatic control elements were also used in various tanks in the pilot plant with emphasis on the large tanks, such as the NC storage tank. The probe consists of a hollow pipe positioned vertically in the tank with its open end near the tank bottom. Air supplied at a constant rate is forced down and out of the pipe to then bubble to the surface of the tank material (liquid) and escape through venting provided. As the liquid height varies, the back pressure exerted by the liquid upon the air in the pipe varies. A pneumatically operated pressure transducer continuously measures the air pressure in the sensor pipe and converts the measured pressure into a 21 to 103 kPa (3 to 15 lb /in.<sup>2</sup>) pneumatic signal. The signal pressure varies directly with the liquid level in the tank and is used to operate a material height indicator/controller and alarm.

(4) "No Cote" Capacitance Probe - The "no cote" capacitance probe and suitable electronic package are manufactured by the Drexelbrook Engineering Company. The material level in the

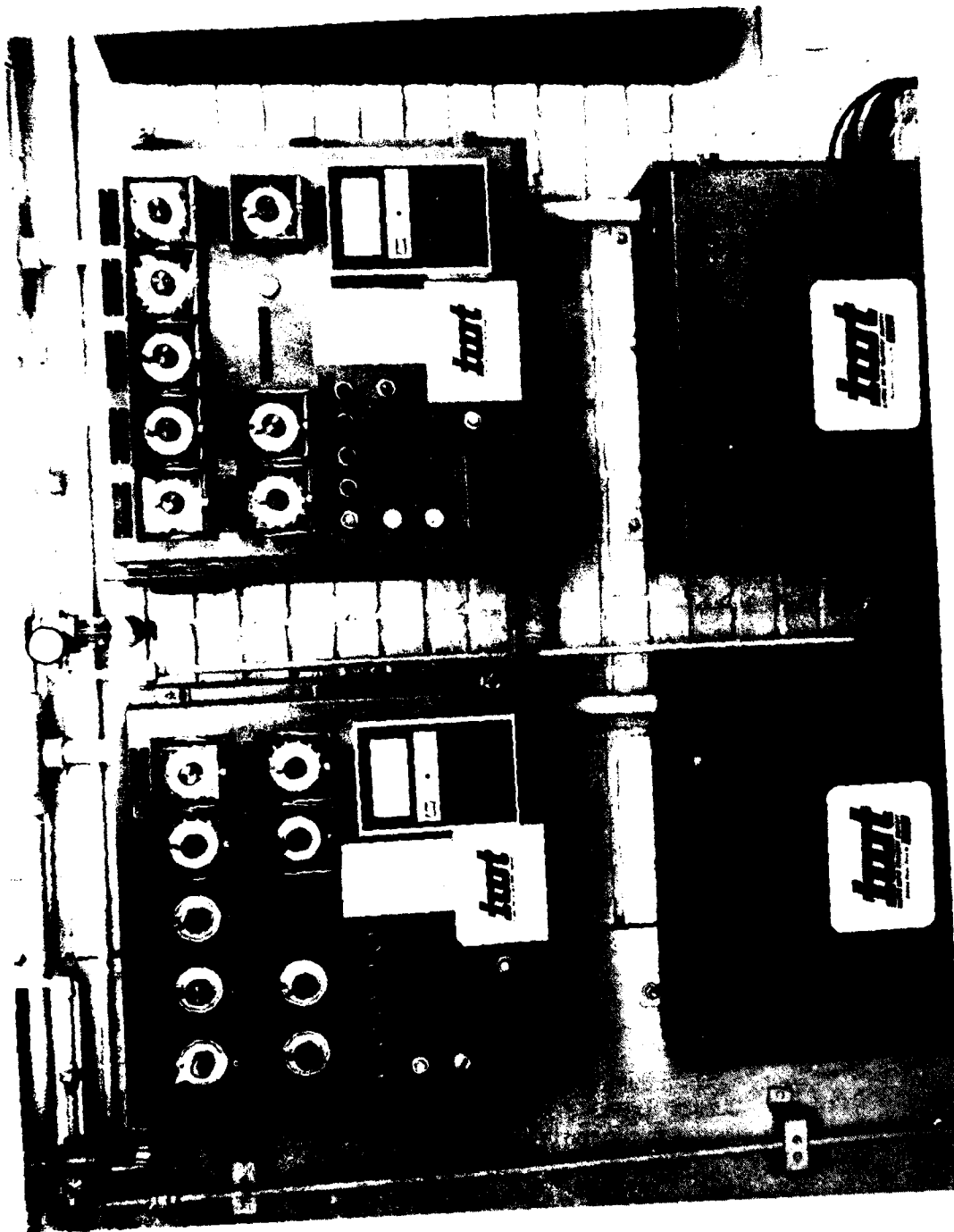


Figure 42. Ion exchange control panels.

nitrators and other process acid vessels was monitored through the use of these probes. The probe itself consists of an electrically charged, Teflon encased stainless steel rod positioned vertically within the tank. As the air-to-liquid interface (liquid level) moves up the probe, the capacitance between the charged probe and grounded metal tank increases proportionally and ranges from less than 5 pF to 3000 pF. The electronics package is basically a capacitance-to-current transducer which converts the probe capacitance into current. The current strength is proportional to the probe capacitance. A maximum of 0.1 ampere of current flows in any of the electronic package wires during operation. The output current is used to operate the material height indicator (or indicator/controller) and alarms.

c. Weigh Cell Systems

Four tanks in the pilot plant were installed as weigh tanks with readouts located in the control room. Three of these tanks used the two pilot and one load cell configuration. Emery Hydraulic Load Cells (Type AC-RB), Drawing C-17489, and Emery Pivots, Drawing C-16446, were used in these weighing systems. One of the tanks utilizes a three load cell weighing system.

d. Dynatrol Specific Gravity Meter

A Dynatrol Specific Gravity Meter was installed in the pilot plant to be used in controlling the feed composition to the Process Ion Exchange Unit and is discussed in another section of this report.

e. Utilities Consumption Measurements

Instrumentation was installed for the measurement of low pressure steam, high pressure steam, air, and electricity consumed on a continuous basis. Readouts are obtained in the control room.

f. Venturis

Liquid flows through a majority of the process lines in the pilot plant are controlled by measuring the pressure drop across a machined stainless steel venturi in the process stream and feeding the transmitted signal to an indicator, controller, or indicator/recorder/controller instrument at the appropriate control panel. Each venturi was sized for the rate of flow, viscosity, and temperature of the liquid flowing through the line.

Fume Removal

An existing acid fume exhaust stack and fan were used to remove fumes from the adjacent pilot plant. The 1.79 m<sup>3</sup>/s (4000 ft<sup>3</sup>/min) stainless steel fan was connected to venting nozzles on all process tanks containing acid in such a manner that the production facility fume recovery system would not be contaminated with magnesium nitrate from the pilot plant.

Boiling tubs were vented to the outside of the building. Provisions were made to maintain a positive fresh air pressure in the control room to protect operators and instruments in the event of excess acid fumes in the rest of the pilot plant.

Instrument cabinets which contain sensors and transmitters for the various controlled equipment throughout the pilot plant are purged with instrument air.

#### Dump Tanks

Two dump tanks were installed for use at the pilot plant. Both tanks were made from batch system dipping pots filled with appropriate nozzles and valves. One of these tanks was placed near an acid sewer line and connected to all of the overflow lines from all acid tanks used in the pilot plant with the exception of the bulk acid storage tanks. This tank was vented to the fume removal fan and stack. This tank can be used as a hold tank for any acids which are to be dumped to the production waste acid treatment facilities.

A second dump tank was arranged adjacent to the loading dock and connected to a stainless steel box on the loading platform. This box contains a perforated false bottom, and over the top are arranged open ended pipes running from several tanks within the building. A filtered water deluge nozzle is also mounted above the open box. The bottom of the box drains through a controlled valve so arranged to allow the draining liquid to go into the acid sewer line directly or into the second hold tank made from a batch dipping pot. This hold tank drains into the acid sewer line when the valve on the bottom of the tank is opened. The top of the hold tank is vented to the fume fan and stack.

This box-tank arrangement will permit the pumping of NC slurry from the blend tank to the box where the water can drain out so that the NC can be put into cans for shipment or if the NC is contaminated or of the improper analysis it can be put into cans for disposal. Also, one of the lines terminating above the box runs from the bottom of the nitrators so they can be dumped, if necessary. The water nozzle will provide drowning water and the acid can drain either to the sewer or to the hold tank as the operator deems necessary.

Movement of NC from other locations in the pilot plant to the loading dock dump tank is indicated in Appendix B.

#### Valves

##### a. Process Valves

Stainless steel ball valves with Teflon seats are used in general throughout the pilot plant with the exceptions of utilities and fume removal systems. The fume removal system uses a

combination of ball valves and Posi-Seal vane-type valves with two-inch and larger valves being the vane-type. Flanged valves are used in all cases where NC is handled except in the ion exchange systems. All valves are arranged so that they may be removed for maintenance.

b. Control Valves

Control valves utilized throughout the pilot plant are stainless steel flanged ball valves with Teflon seats. With the exception of the previously discussed flush bottom tank valves control is by 21 to 103 kPa (3 to 15 lb /in.<sup>2</sup>) pneumatic actuation. Position of each valve is indicated by a pilot light on the applicable instrument console in the control room.

Interlocks

Several safety interlocks were designed into the pilot plant equipment for the operation of the cellulose feeder, the nitrator agitators and the attrition mills. These interlocks are detailed on the electrical drawings listed in Appendix A.

a. Nitrator Agitators

(1) Nitrator agitators are all started and stopped simultaneously under normal conditions. Before the agitators can be started, the following conditions must be satisfied:

(a) The liquid level must be at a point above the low level alarm point.

(b) The Merco screens must be running.

(2) The nitrator agitators also have a meter relay alarm to indicate the following:

(a) Agitator Shaft Overload - The high meter set point will sound an alarm and flash an indicator light and shut off the particular motor that is overloaded.

(b) Agitator Shaft Failure - Same as above except occurs when shaft breaks or fails.

b. Attrition Mills

(1) The attrition mills cannot be started unless the nitrator agitators are running. If agitators stop, the attrition mills will stop. The screw feeder is started and stopped with the mills and therefore has the same interlocks.

### c. Cellulose Feeder

(1) The cellulose feeder cannot be started until the following conditions are satisfied:

(a) The attrition mills must be running (unless the attrition mill selector switch is in bypass position).

(b) The salt-acid flow must be started and above set point flow.

(2) The cellulose feeder will shut down, should the following occur:

(a) High temperature on the nitrator tanks.

(b) High level on the nitrator tanks.

(c) Overflow on the nitrator tanks.

(d) Pressure loss on the CO<sub>2</sub> fire protection system for feeder.

(e) Attrition mill shutdown.

(f) Salt-acid flow below set point rate.

#### NOTES:

The "Run" pushbutton must be depressed and held in the depressed position. The salt-acid flow will start. The "Run" button can be released when flow is above set point. This will be indicated when light remains on after release.

### d. Salt-Acid Valves

(1) Valve can be opened or closed manually from control panel. The valve will not remain open, however, unless cellulose feeder is running. Salt-acid flow will start prior to feeder starting and will stop a short time after feeder stops.

(2) Should the salt-acid line become plugged or start leaking, the cellulose feeder will shut down and the salt-acid valve will close.

### Mixers

Conventional turbine (one used) and propeller-type mixers are used to achieve and/or maintain process slurry uniformity in the various pilot plant processing tanks. All mixer parts exposed to product contact, including the stuffing box/gland bearing lubrication retainer, are constructed from stainless steel. The stuffing boxes are packed with Teflon. The motors are fan cooled and totally enclosed



to prevent NC, water, or acid contamination from entering. A stabilizer ring is attached to many of the propellers (lower one only when there are two propellers to a shaft - none on the nitrator propellers) to minimize shaft vibration.

#### Centrifugal Pumps

Centrifugal pumps are used to transfer process slurries or fluids through pipes from one tank or operation to another. Transfer is accomplished by a rotating vaned impeller within a casing. The revolving blades of the impeller produce a partial vacuum at the entrance or eye of the impeller that draws the liquid or slurry into the casing from a suction (intake) pipe. The liquid or slurry is then forced outward along the impeller blades at an increasing velocity. The velocity head it has acquired when it leaves the blade tips is changed to a pressure head as it passes into the volute chamber and out of the discharge port.

All of the pumps used in the pilot plant, except for the Wilfley pumps, have a flushed and/or lubricated Teflon packing around the impeller drive shaft which prevents water, acid, magnesium nitrate, or NC from entering the shaft bearings or the pump surroundings. The Wilfley has a unique seal arrangement which eliminates the packing box, runs without packing friction during operation (except momentarily during starting and stopping), and prevents leakage through the use of vanes on the back side of the impeller. All of the pump parts contacting the process slurry or liquids are constructed of stainless steel to prevent corrosive deterioration and process contamination. All of the pump drive motors are totally enclosed and fan cooled (TEFC) to prevent entry of contaminants (especially NC) and motor overheating.

#### Countercurrent Wash Filters

##### a. Candle Type

Scaled down candle type filters were fabricated in accordance with Drawing Serial No. 15381 as listed in Appendix A. These filters were installed in the exit lines from each of the centrifuge wash acid spray pumps and in the exit line from the belt wash accumulator tank pump to catch any accumulated NC fines that were present in the liquids used for the countercurrent wash system.

##### b. Strainers

To further assure that no NC fines were present in the wash acid system, small stainless steel 50-mesh strainers were installed in the lines in front of the spray nozzles to the centrifuge washes and the spray nozzles at the Eimco filter.

### Temperature Sensors

Many of the process tanks have thermocouples located in them to measure the tank ingredients temperature. A thermocouple consists of two dissimilar metal wires (copper and constantan were used in this project) joined at one end. Each thermocouple is mounted inside a closed stainless steel tube for protection against mechanical or chemical damage and to secure it in the desired tank location. When the temperature of the welded junction of the wires changes and the free ends of the wires are kept at a known temperature, an electric potential is generated in the system and appears at the terminals. This potential varies proportionately to the temperature [approximately 10 mV at 204°C (400°F)], is measured continuously, and is converted to equivalent degrees of temperature via recorder-controllers on the instrument control panel. When the high or low temperature level set point on the recorder-controller is exceeded, a warning horn sounds and the appropriate (high or low temperature) indicator light is turned on which alerts the operator that corrective action is required.

### Hazards Analysis

Two of the inherent objectives of this project were the determination of hazards associated with the magnesium nitrate process and the minimization of these hazards by the proper designing of equipment and process flow. An extensive hazards analysis of all components of the pilot plant was conducted. Modifications to equipment necessary to comply with established hazard criteria for safe operations were made. Detailed hazard studies are on file at RAAP.

### MAGNESIUM NITRATE/NITRIC ACID CONCENTRATOR

The original scope of work for this project included the design, installation, and evaluation of a magnesium nitrate/nitric acid concentrator to recover the nitric acid and magnesium nitrate from the spent nitrating mix for reuse in the nitration process. The nominal capacity of this unit was intended to be 12 1/2 tons per day of 99 percent nitric acid.

At the direction of the Government, Chemical Construction Corporation (Chemico) was selected to design and install the pilot-scale concentrator. This was done in order to procure a pilot unit design based on the existing nitric acid/sulfuric acid concentrators which were being built by Chemico. It was felt that since these concentrators would be converted to the magnesium nitrate process if the evaluation proved successful, the pilot unit should be of a design compatible with the units to be converted.

After the contract was awarded and the design effort begun, Chemico submitted a quotation for the design and installation of the pilot-scale concentrator which exceeded the funds available for this effort. The contract was cancelled, and a decision was made

that Hercules Incorporated should design, procure, and install a subpilot-scale concentrator with a nominal capacity of one ton per day of 99 percent nitric acid.

The purpose of the subpilot-scale concentrator was to provide design criteria information for conversion of the nitric acid/sulfuric acid concentrators to the magnesium nitrate process, and to establish operating conditions for processing the spent salt-acid mix from the magnesium nitrate nitration process. The magnesium nitrate process for concentrating nitric acid has been in use in commercial installations and at Holston AAP for a number of years, but no information was available on the processing of a mixture of weak magnesium nitrate and nitric acid which might contain NC fines and NC nitration by-products.

The subpilot concentrator was designed at RAAP, with consulting services from the Hercules/Wilmington Engineering Staff. The columns, condensers, packing, and other glass components were procured from the Corning Glass Company. The reboiler and vacuum evaporator were designed and fabricated at RAAP. The pumps, tanks, piping, and instrumentation were procured from various vendors. Installation of all major equipment items and process piping was completed prior to suspension of work on this project. Installation of the instrumentation and electrical wiring was in progress but had not been completed.

The model of the subpilot concentrator, figure 23, illustrates how the concentrator appears when installed in the west end of the pilot plant building. Pumps and storage tanks are located on the first floor; the evaporator is supported on steel beams under the third floor; and the concentrator towers extend from the first floor area up into the cupola added to the building above Bay 7.

The process flow for the subpilot concentrator has magnesium nitrate solution from the evaporator (3) pumped to near the top of the stripping column (2). Spent acid from the storage of feed tank (8) is also pumped to near the top of the stripping column (2). Return acid from the reflex column (1) is pumped from the reflux drain acid pump tank (9) to the top of the stripping column (2) and the diluted magnesium nitrate solution flows down the stripping column to the reboiler (4).

Water vapor from the heated reboiler (4) flows into the stripping column (2) and provides additional heat to strip nitric acid from the countercurrent flow of magnesium nitrate solution. Nitric acid vapor flows under reduced pressure from near the top of the stripping column (2) to near the bottom of the reflux column (1) and is concentrated as it flows countercurrent to the reflux nitric acid. Nitric acid is condensed by water cooled glass condensers in the top of the reflux column (1) and the strong nitric acid product is drawn off, from the reflux acid, through a control valve to the strong nitric acid product tank (7).

Diluted magnesium nitrate solution from the reboiler (4) is pumped to the evaporator (3) for concentration. Excess magnesium nitrate solution, added from spent acid, is sent to the magnesium nitrate storage tank (5).

Diluted magnesium nitrate solution will be stored in a weak magnesium nitrate storage tank (6) during preparations for startup of the concentrator and during shutdown.

#### TEST PLAN FOR EVALUATION OF PILOT PLANT

##### Outline

A detailed test plan for evaluation of the magnesium nitrate pilot plant is given in Appendix C. Process flow sheets are given for nitration initiated in the attrition mills and for nitration initiated in the nitrator pots. The test plan includes both inert and live evaluation details.

##### Procedures

General Operating Procedures and Unit Operating Procedures for complete operation of the pilot plant are listed in Appendix D. Copies of all these procedures are on file at RAAP.

##### Control Room Data

Prior to startup of each test run in the pilot plant, Preoperation Check Lists (fig. 43) will be completed by the operating personnel so that continuity of operations can be maintained. Figure 44 provides examples of the Control Room Data Sheets that will be used during the course of each pilot plant run.

#### ENERGY CONSERVATION AND ECONOMIC ANALYSIS

The use of energy during the operation of the magnesium nitrate process was to be determined during the manufacture of NC in the pilot plant. Previously discussed recorders for the utilities consumed during operations were to furnish data for this evaluation.

Utilizing the above generated data, an economic analysis was to be made comparing the continuous mixed acid process to the magnesium nitrate process. However, these data and analysis were not made because of the termination of work.

Date \_\_\_\_\_ Time \_\_\_\_\_

Engineer \_\_\_\_\_

PRE-OPERATION CHECK LIST

	<u>Yes</u>	<u>No</u>
1. Refrigeration Unit Starter Breaker ON	---	---
2. Fume Removal System ON	---	---
3. Applicable Fume System Vent Valves Open	---	---
4. Water Line Valves to Nitrating Building Properly Positioned	---	---
5. Water Pressure Adequate	---	---
6. Process and Instrument Air Line Valves to Building and Through Air Dryer Properly Positioned	---	---
7. Air Pressure Adequate	---	---
8. Communication System Activated	---	---
9. Spent Acid Discharge Line from Spent Acid Storage Tank to the Durco Filters Properly Set and Valves Positioned	---	---
10. Line from Durco Filters to Spent Acid Storage Tank Properly Set and Valves Positioned	---	---
11. Acid Durco Fines Discharge Line to the Acid Fines Tank Properly Set and Valves Positioned	---	---
12. Recovered Acid Line from the Centrifuge to Recovered Acid Surge Tank Properly Set and Valves Positioned	---	---
13. Recovered Acid Line from Recovered Acid Surge Tank to Durco Recovered Acid Filters Properly Set and Valves Positioned	---	---
14. Acid Fines Discharge Lines from Durco Recovered Acid Filters to Acid Fines Tank Properly Set and Valves Positioned	---	---
15. Filter Recovered Acid Lines to the Acid Sewer and/or Centrifuge Properly Set and Valves Positioned	---	---
16. Acid Mix Lines Properly Set to Appropriate Nitrating Acid Feed Tank and Valves Positioned	---	---
17. All Applicable HAND/AUTO Starter Switches Set on AUTO Position	---	---
18. All Applicable STOP-RUN Switches Set on STOP Position	---	---

Figure 43 Preoperation check list

Figure 43 (cont'd)

	<u>Yes</u>	<u>No</u>
19. All Motor Breaker Switches Activated	---	---
20. Cooling Water Jacket Valves for Centrifuge Open	---	---
21. All Wash Acid Lines To and From Centrifuge Properly Set and Valves Positioned	---	---
22. Acid Conditioning Systems Properly Set and Valves Positioned	---	---
23. Salt Acid Feed Valve to Nitrator Closed	---	---
24. Nitrating Mix Circulating Line Properly Set and Valves Positioned	---	---
25. Valve on Nitrating Mix Take-Off Line from Circulating Line Open	---	---
26. All Nitrating Pot Drop Valves Closed	---	---
27. Nitrating Pot Overflow Weirs Properly Adjusted	---	---
28. Feed Valves to Eimco Filter Closed	---	---
29. Circulating Line from NC Hold Tank No. 1 to Eimco Filter Properly Set and Valves Positioned	---	---
30. Discharge Valve from NC Hold Tank No. 1 to Pump Closed	---	---
31. Gland Water Valve to NC Hold Tank No. 1 Pump Open	---	---
32. Line from Spent Acid Storage Tank Through Acid Chiller to Centrifuge Properly Set and Valves Positioned	---	---
33. Eimco Discharge Line Properly Set to Applicable Tank or Boiling Tub and Valves Positioned	---	---

Figure 43 (cont'd)

	<u>Yes</u>	<u>No</u>
<u>Prior to Utilization of Attrition Mills for Sizing</u>		
1. Lines from Applicable Boil Tub to Blending Tank Properly Set and Valves Positioned	—	—
2. Outlet Valve on Blending Tank Closed	—	—
3. Circulating Line from Blending Tank to Attrition Mills Properly Set and Valves Positioned	—	—
4. Valve on Take-Off Line Closed	—	—
5. Line from Attrition Mills to NC Hold Tank No. 2 Properly Set and Valves Positioned	—	—
6. Gland Water to Attrition Mills ON	—	—
7. Lines from NC Hold Tank No. 2 to Desired Boiling Tub Properly Set and Valves Positioned	—	—
<u>Prior to Blending</u>		
1. Lines from Desired Boiling Tub Containing High Grade Material to the Blending Tank Properly Set and Valves Positioned	—	—
2. Lines from Desired Boiling Tub Containing Low Grade Material to the Blending Tank Properly Set and Valves Positioned	—	—

CENTRIFUGE

NITRATION

Time	Nitrating Acid Mix Temperature Loop 1	Nitrating Acid Flow, percent Valve Open	Flow Rate	Acid Integrator	Cellulose Integrator	Temp. Pot 1	Temp. Pot II	Percent load on				Belt Wash Accumulator Flow, gpm	
								Agitators	Agitator Drive Settings	No. 1	No. 2		No. 3
								No. 1	No. 2	No. 3	No. 4	No. 4	Sturry Wash

DATE \_\_\_\_\_  
 RUN NUMBER \_\_\_\_\_  
 TYPE \_\_\_\_\_

Figure 44 Control room data sheet



Figure 44 (cont'd)

Time	Wash Acid Tanks Level		Temperature to Nozzles	Wash Acid Flow to Nozzle, gpm		Durco Filters Pressure		Slurry Feed Pressure	Filter Cake Thickness	Vacuum, Inch. Hg	Wash Water Flow Rate, RPM	Slurry Water Pressure	Durco Filter Pressure
	No. 1	No. 2		No. 1	No. 2	No. 1	No. 2						
No. 1	No. 2	No. 3	No. 4	No. 1	No. 2	No. 3	No. 4	No. 1	No. 2	No. 1	No. 2	No. 1	No. 2

REMARKS:



## PROTECTIVE STORAGE CONDITIONS

A decision was made by the U. S. Army Materiel Development and Readiness Command Project Manager to suspend development of the magnesium nitrate process until it was known if the mixed acid recycle process would be successful. After the mixed acid recycle process was proven to be technically feasible and more economical, it was accepted by the U. S. Army Armament Research and Development Command and the U. S. Army Armament Materiel Readiness Command. The Project Manager ordered the termination of the magnesium nitrate MM&TE project on 14 September 1976. These decisions were reported in a PMO letter, DRCPM-PBM-EP, dated 29 October 1976, Subject: DRC Project 5764013, MM&TE Magnesium Nitrate Nitrocellulose, transmitted to Hercules Incorporated by letter, SARRA-EN, dated 4 November 1976. The revised Contract Scope of Work (dated 18 July 1977) with instructions for placing the pilot plant equipment in a protective storage condition was transmitted to Hercules Incorporated by letter, SARRA-EN, dated 9 August 1977.

Tanks, centrifuge, nitrator, dewatering filter, NC fines filters and other process vessels\* and connecting lines were disassembled, where necessary, cleaned and thoroughly flushed with clean filtered water.

Electrical contacts of motors and associated starting equipment were cleaned and sprayed with a corrosion preventative. All electrical equipment was left in a de-energized position. The main 440 volt and 110 volt breakers were padlocked in the "OFF" position.

All instrumentation was disassembled, where necessary, cleaned, sprayed with a corrosion preventative, sealed in plastic bags, labeled and placed in a control console or, where appropriate, stored in place. The control console electrical contacts were cleaned and sprayed with a corrosion preventative. The concentrator instrumentation was also sealed and stored in a control console. Control consoles were then sealed to prevent moisture entry.

High pressure steam, low pressure steam, filtered water, and drinking water lines to the pilot plant were turned off and blanked or capped, as necessary. Water lines, tanks, and steam lines within the pilot plant were drained to prevent freezing.

The pilot plant communications system was loaned for use in the NC production facilities. The carbon dioxide fire suppression system, which was borrowed for use with the cellulose weigh feeder, was returned to production use.

\*See Appendix F, "A.S.M.E. Coded Pressure Vessels".

New drive pulleys and drainage belt for the dewatering filter, and materials for construction of the uncompleted nitric acid concentrator were stored in the pilot plant building. Process equipment that was used during the bench-scale studies was also cleaned and stored in the building.

A magnesium nitrate brine solution was left in the refrigeration unit and all associated equipment to prevent corrosion of the system.

Chemicals and ion exchange resins, which could not be stored economically or stored without damage from freezing, were placed on the excess materials list. These items were stored in a separate building.

### CONCLUSIONS AND RECOMMENDATIONS

#### Conclusions

Based on the work accomplished under this project, it is concluded that:

1. Manufacture of NC by the magnesium nitrate method, utilizing laboratory and bench-scale equipment, yielded a product which met the NC specification MIL-N-244A, except for the 134.5°C heat test (German rapid stability test).
2. A two bed, two resin, regenerative type ion exchange system can be used to recover magnesium and nitrate ions from the waste water effluents.
3. Materials of construction which provide satisfactory corrosion resistance for the magnesium nitrate process are available.
4. Based on laboratory and bench-scale studies, the magnesium nitrate method has advantages over the sulfuric/nitric mixed acid nitration process for NC. These are:
  - a. Reduction of stream pollutants from acid waste effluents.
  - b. Reduction of viscosity boil cycle time.
  - c. Elimination of soda ash poaching treatment.
  - d. Reduction of water consumption.

- e. Some possible negative aspects are the energy, capital cost, and operating cost impact which will occur if further evaluation shows that substantially more acid concentration capacity will be required.
5. The pilot plant can be used to evaluate NC processing conditions for either the magnesium nitrate or the mixed acid method.

#### Recommendation

It is recommended that the pilot equipment be maintained in a protective storage condition that will allow for reactivation to support future process development work on an as-needed basis.

APPENDIX A

DRAWING LIST

The following Drawing List tabulates the serial number and nomenclature of the drawings made for the construction and operation of the Pilot Plant. Drawings are stored at RAAP.

The full drawing numbers are not listed; just the serial number is listed. For example:

Dwg. No. 3001200 19518 F 35702 is listed by its serial number 19518.

DRAWING LIST

<u>Serial No.</u>	<u>Nomenclature</u>
13011	Weak Nitric Acid Day Tank
13012	Strong Nitric Acid Day Tank
15381	Wash Acid Filter Plan and Detail
15714	Acid Storage Tank
17496	Nitric Acid Storage Process Arrangement
18220	Instrument Arrangement - Second Floor
18300	Continuous Nitrator Assembly and Details
18301	Continuous Nitrator Details
18302	Continuous Nitrator Details
18303	Continuous Nitrator Details
18382	Continuous Nitrator Details
18400	Continuous Nitrator Arrangements and Details
18407	Continuous Nitrator Details
18799	Instrument Cabinet, Control Room
18800	Instrumentation for Acid Preparation
18801	Instrumentation for Nitration Process
18802	Instrumentation for Purification Process
18803	Instrumentation Control Loops 1-30
18804	Instrumentation Control Loops 31-93
18805	Instrumentation Control Loops 94 up
18818	Acid Preparation Process Flow Sheet
18819	Nitration Process Flow Sheet
18820	Purification Process Flow Sheet
18845	Instrument Panel, Acid Preparation
18846	Instrument Panel, Nitration

<u>Serial No.</u>	<u>Nomenclature</u>
18847	Instrument Panel, Purification
18853	Centrifuge Slug Feeder
18906	Control Room Architectural Plan and Sections
18936	Mag. Nitrate Make-Up Tank and Details
18939	Control Room Equipment Arrangement
18967	Spent Liquor Storage Tank Foundations
18988	Centrifuge Foundation Plan and Details
19179	Control Room Ventilation
19189	Plat Plan
19190	Equipment Arrangement, Sheet 1
19196	Equipment Arrangement, Sheet 2
19197	Equipment Arrangement, Sheet 3
19198	Equipment Arrangement, Sheet 4
19271	Decant Bulb
19272	Instrument Cabinet Arrangement, Acid Prep.
19273	Instrument Cabinet Arrangement, Nitration
19274	Instrument Cabinet Arrangement, Purification
19275	Instrument Cabinet Arrangement, Details
19276	Equipment Arrangement, Sheet 5
19288	Unloading Platform
19373	Instrument Arrangement, 1st Floor
19459	Instrument Arrangement, 3rd Floor
19461	Instrument Arrangement, Outside Tanks
19462	Eimco Frame Support
19464	Tank Foundations
19513	Piping Arrangement - NC Storage Tank
19514	Outside Tanks - Piping Plan



Serial No.Nomenclature

19515	Outside Tanks - Sections and Details
19516	Acid Unloading Station and Tank 11
19517	Piping Plan 1st Floor
19518	Piping Plan 2nd Floor
19519	Piping Plan 3rd Floor
19520	Piping Plan 4th Floor
19521	Process Piping and Details 2nd Floor
19522	Process Piping and Details 1st Floor
19526	Nitrator Mixer Support
19552	Refrigeration Unit Foundation
19555	Feeder Supports
19561	Attrition Mill Supports
19578	Instrumentation Control Loops 53-56
19630	Mg(NO <sub>3</sub> ) <sub>2</sub> Makeup Tank Support
19671	Lighting Plan 1st Floor
19672	Lighting Plan 2nd Floor
19673	Lighting Plan 3rd Floor
19674	Lighting Plan 4th Floor
19675	Outside Electric Lines
19676	Power Wiring Diagram
19677	Inst. Panel Wiring Connections - Acid Prep.
19678	Nitration Control Panel Wiring
19679	Purification Control Panel Wiring
19680	Relay Panel Arrangement - Electrical
19681	Power Plan 1st Floor
19682	Power Plan 2nd Floor

Serial No.Nomenclature

19683	Power Plan 3rd Floor
19684	Power Plan 4th Floor
19685	Power Plan Outside Tanks and Aux. Buildings
19686	Acid Preparation Process Control Wiring Diagram
19687	Acid Prep. Motor Control Wiring Diagram
19688	Nitration Process Control Wiring Diagram
19689	Nitration Process Control Wiring Diagram
19690	Motor Control Wiring Diagram - Nitration
19691	Motor Control Wiring Diagram - Purification
19692	Motor Control Wiring Diagram - Purification
19693	Durco Filter Wiring Diagram
19694	Merrick Scale A.C. and Signal Diagram
19695	Merrick Scale Connection Diagram
19696	Control Wiring Diagram - Cation-Anion (Two Bed) Units
19697	Cabinet Wiring Diagram - Cation-Anion (Two Bed) Units
19698	Typical Alarm Control Wiring Diagram
19699	Illuminated Push Button Wiring
19700	Centrifuge Strip Chassis and Connection Wiring Diagram
19701	Durco Filter Panel
19710	60% Nitric Make-Up Tank Support
19713	Pressure Indicators - Instrument Cabinet
19727	Inst. Air Supply 1st and 2nd Floor
19742	Inst. Air Supply 3rd Floor
19743	Inst. Air Supply 4th Floor and Outside Tanks
19760	Loop Arrangement 1st Floor
19761	Loop Arrangement 2nd Floor

Serial No.Nomenclature

19762	Loop Arrangement 3rd Floor
19763	Loop Arrangement 4th Floor
19764	Instrumentation - Outside Tanks
19769	Steel Arrangement 2nd and 3rd Floors
19839	Merco and Durco Support
19840	Acid Pump Supports
19841	Slug Feeder Supports
19848	Service Piping 1st Floor
19849	Service Piping 2nd Floor
19850	Service Piping 3rd Floor
19851	Service Piping 4th Floor
19582	Process Piping Details
19882	Service Entrance and Motor Control Layout
19883	Electrical Equipment Schedule.
19884	Elect. and Inst. Cover Sheet
19889	Steel Arrangement - 4th Floor
19893	Dump Hopper and Support
19894	Discharge Hopper and Support - Extractor
19898	Scale Feeder Hopper and Support
19899	Chute to Nitrator
19904	Centrifuge Discharge Adaptor and Att. Mill Chutes
19906	Fume Exhaust System 1st Floor
19907	Fume Exhaust System 2nd Floor
19908	Fume Exhaust System 3rd Floor
19909	Fume Exhaust System Outside Lines
19910	Process Piping - 1st Floor

Serial No.Nomenclature

19911	Process Piping - 1st Floor Details
19913	Percolator Tube Detail
19926	Pipe Line Description 3rd Floor
19930	Brine Piping
19933	Pipe Line Description 2nd Floor

Industrial Alloy Fabricators, Inc. Drawings (GBF 6198)

D-1071B	Drain Down Tank 61" (Boiling Tubs 1 and 2)
D-1070D	Drain Down Tank 75" (Boiling Tubs 3, 4, and 5)
D-1116A	Magnesium Nitrate Make-Up Tank

Process Equipment Corporation Tank Drawings (GBF 6169)

AY-16624	HNO <sub>3</sub> Makeup (100 Gallon "OVC")
AY-16623-21944	Acid Fines Storage (100 Gallon "JOVC")
AY-16623-21912	NC Blend (650 Gallon "OVC")
AY-16623-21913	NC Hold Tanks #1 and #2 (170 Gallon "OVC")
AY-16623-21908	H <sub>2</sub> O Fines Storage (100 Gallon "OVC")
AY-16729	Concentrator Mg(NO <sub>3</sub> ) <sub>2</sub> Storage (500 Gallon "JCVC")
AY-16626	Mg(NO <sub>3</sub> ) <sub>2</sub> Storage (665 Gallon "JOVC")
AY-16728	Spent Acid Feed To Concentrator (1000 Gallon "CVC")
AY-16627	Salt/Acid Storage (675 Gallon "JOVC")
AY-16625	HNO <sub>3</sub> Storage (160 Gallon "OVC")
AY-16629	Spent Liquor Storage (1075 Gallon "JOVC")
AY-16623-21910	Recovered Acid Surge (65 Gallon "OVC")
AY-16623-21945	Spent Acid Surge (65 Gallon "JOVC")
AY-16623-21911	Wash Acid Tanks #1, #2, #3, and #4 (10 Gallon "OVC")
AY-16623-21909	Belt Wash Accumulator (115 Gallon "OVC")
AY-16623-21914	Boil Tub Water Hold Tank (1075 Gallon "OVC")
AY-16623-21946	Weak Mg(NO <sub>3</sub> ) <sub>2</sub> Storage (600 Gallon "OVC")

Serial No.

Nomenclature

Process Equipment Corporation Tank Drawings (GBF 6169)

AY-16623	NH <sub>4</sub> NO <sub>3</sub> Storage (600 Gallon "OVC")
AY-16628	Salt/Acid Makeup (675 Gallon "JOVC")
D541-022	Attrition Mill Assy. 2430 FAB Swinghead V-Belt Drive
C1711-007	Screw Feeder

APPENDIX B

PILOT PLANT CAPABILITIES FOR MOVEMENT OF NITROCELLULOSE

Appendix B

Pilot Plant Capabilities For Movement Of Nitrocellulose

NITRATION AREA

1. Nitrator to
2. Slug Feeder to
3. Centrifuge to
4. NC Hold Tank No. 1

A. From H.T. #1 to Eimco Filter with 1-1/2" return to HT #1.

I. From Eimco to Boiling Tubs

a. From Boiling Tubs to Blend Tub

(1) From Blend Tub to Attrition Mill(s) with 1-1/2" return

(2) From Blend Tub to Boiling Tub #4 Bldg. 3019

(3) From Blend Tub to NC Storage Tank

II. From Eimco to Blend Tub

B. From HT #1 to Attrition Mill(s) with 1-1/2" return

I. From Attrition Mill(s) to Nitrator (Install hopper and lines)

II. From Attrition Mill(s) to NC HT #2 (Install hopper and lines)

a. From NC Hold Tank No. 2 to Eimco Filter

b. From NC Hold Tank No. 2 to Boiling Tubs with 1-1/2" return

c. From NC Hold Tank No. 2 to NC Storage Tank with 1-1/2" return

(Install blind)

(1) From NC Storage Tank to Drum Loading

(2) From NC Storage Tank to #4 Boiling Tub Bldg. 3019

(3) From NC Storage Tank to NC Blend Tub

d. From NC Hold Tank No. 2 to Attrition Mill(s)

C. From NC Hold Tank No. 1 to Boiling Tubs

D. From NC Hold Tank No. 1 to NC Storage Tank

**Notes:**

1. No facilities to pump direct from Boiling Tubs to Attrition Mill(s) and return to another Boiling Tub.
2. No facilities to pump direct from #1 Hold Tank to Drum Loading.
3. No facilities to pump from #4 Tub in Building 3019 to the Pilot Plant. (To get  $\text{Mg}(\text{NO}_3)_2$  NC out of #4 Tub.)



PURIFICATION AREA

1. From NC Hold Tank No. 1 to Eimco Filter With 1-1/2" Return

- A. Install blinds in the south flanges of both 2" Ts above No. 1 Hold Tank pump.
- B. Install blind in south flange of 1-1/2" return line T directly above tank TFA 18782 on second floor and north flange of L below Attrition Mill.
- C. Open Auto Valve in feed line above Eimco filter.

2. From Eimco Filter to Boiling Tubs

- A. NC comes off filter belt into hopper and into 2" line.
- B. Open three way 2" valve at north side of boiling tubs on third floor.
- C. Set other valves to receive into selected boiling tub.

3. From Boiling Tubs to Blend Tub

- A. Set three-way 3" valves from bottom of selected boiling tub.
- B. Open two three-way 3" valves above and on the north side of blend tub on first floor.
- C. Open outlet valve and plug from selected boiling tub.

4. From Blend Tub to Attrition Mills With 1-1/2" Return

- A. Open Auto Valve on bottom of blend tub.
- B. Set three-way valve between blend tub and boil water hold tank to pump to south line.
- C. Install blind in north side of T in 2" line above Tank No. RFA 18782 on second floor.
- D. Open Auto Valve under Attrition Mill on third floor.
- E. Change L in 1-1/2" return line south of nitrator on second floor.

5. From Blend Tub to Boiling Tub No. 4, Building 3019
  - A. Set three-way valve between blend tub and boil water hold tank first floor.
  - B. Set two three-way 3" valves west of NC storage tank.
  - C. Open Auto Valve on bottom of blend tub.
6. From Blend Tub to Drum Loading

No facilities.
7. From Blend Tub to NC Storage Tank
  - A. Set three-way 3" valve between blend tub and boil water hold tank to pump east.
  - B. Set first three-way 3" valve west of NC storage tank.
  - C. Open Auto valve on bottom of blend tub.
8. From Eimco Filter to Blend Tub
  - A. Set three-way 2" valve on top of third floor boiling tub, north side.
  - B. Set three-way 2" valve on bottom of third floor boiling tub, north side near ceiling of second floor, to pump south.
  - C. Blind in north flange above blend tank first floor.
9. From No. 1 Hold Tank to Attrition Mill(s) With 1-1/2" Return
  - A. Install a blind in the west flange of the first T in 2" line from No. 1 hold tank pump on first floor.
  - B. Install a blind in the west side flange of 2" T above tank RFA 18782 on second floor.
  - C. Install a blind in the 1-1/2" return line above the sight glass at bottom of nitrator, south side, second floor.
10. From Attrition Mill(s) to Nitrator to Centrifuge
  - A. Install proper hopper and lines at Attrition Mill(s) to deliver to the nitrator.

11. From Attrition Mill(s) to NC Hold Tank No. 2
  - A. Install proper hopper and lines at Attrition Mill(s) to deliver to the 3" line which goes straight to No. 2 Hold Tank.
12. From NC Hold Tank No. 2 to Eimco Filter With 1-1/2" Return
  - A. Set three-way 2" valve above No. 2 hold tank pump, first floor.
  - B. Install a blind in the east flange of 2" T above panel box No. 9, first floor.
  - C. Install a blind in the north flange of 1-1/2" T above hold tank pump No. 1, first floor.
  - D. Install a blind in west flange of 1-1/2" return line T above tank RFA 18782, second floor.
  - F. Open Auto. valve in 2" line above Eimco filter.
  - G. Open auto valve on bottom of No. 2 hold tank.
13. From NC Hold Tank No. 2 to Boiling Tubs With 1-1/2" Return
  - A. Set three-way 2" valve above No. 2 hold tank pump.
  - B. Install blind in west side flange of T above pump.
  - C. Set three-way valves to selected boiling tub.
  - D. Install a blind in the south flange of 1-1/2" return line above tank RFA 18782, second floor.
  - E. Open Auto valve east of boiling tubs on third floor.
14. From NC Hold Tank No. 2 to NC Storage Tank With 1-1/2" Return
  - A. Install blind in west side flange of 2" T above No. 2 hold tank pump.
  - B. Set three-way valve above T.
  - C. The 1-1/2" return line is open direct to No. 2 hold tank.
  - D. Open Auto Valve on top of storage tank.

15. From NC Storage Tank to Drum Loading
  - A. Set three-way 3" valve west of NC storage tank to pump to 3" line.
  - B. Set three-way 3" valve northwest of NC storage tank to drum loading line.
  - C. Open 1-1/2" return line valve above and south of storage tank.
  - D. Open valve at bottom of NC storage tank.
16. From NC Storage Tank to No. 4 Boiling Tub in Building 3019
  - A. Set three-way 3" valve west of storage tank to pump thru 3" line.
  - B. Set three-way 3" valve northwest of storage tank to pump thru 3" line to No. 4 boiling tub.
  - C. Install a blind in the 3" flange north of three-way valve.
  - D. Open valve at bottom of NC storage tank.
17. From NC Storage Tank to NC Blend Tub
  - A. Set three-way 3" valve west of storage tank to pump thru 2" line.
  - B. Open valve at bottom of NC storage tank.
  - C. Pump straight to NC blend tub.
18. From NC Hold Tank No. 1 to Boiling Tubs
  - A. Install blinds in the southeast flange 2" line first T above and south of elect. panel box No. 9.
  - B. Install blind in bottom of T above No. 2 hold tank pump.
  - C. Set three-way 2" valve in line to pump south.
  - D. Set three-way to selected boiling tub.
  - E. Install blinds (return line check).
  - F. Open Auto Valve on east side of boiling tubs on third floor.

19. From NC Hold Tank No. 1 to Storage Tank

- A. Install blind in 2" T to Attrition Mill(s) south.
- B. Install blind in 2" T west to extractor.
- C. Set three-way valve above No. 2 NC hold tank pump to storage tank.

APPENDIX C

Test Plan for the Continuous Manufacture  
of Nitrocellulose by the Magnesium Nitrate  
Process

APPENDIX C

TEST PLAN  
FOR  
THE CONTINUOUS MANUFACTURE OF NITROCELLULOSE  
BY THE MAGNESIUM NITRATE PROCESS

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TEST PLAN  
FOR THE CONTINUOUS MANUFACTURE OF NITROCELLULOSE  
BY THE MAGNESIUM NITRATE PROCESS

The test plan described herein is designed to (1) qualify the equipment installed in the Pilot Plant with regard to safety of operation, performance capability and reliability, and effectiveness of design to satisfy the intent of Pilot Plant processing and (2) define the processing approach to be employed in the nitration of cellulose by the magnesium nitrate method utilizing Pilot Plant facilities to establish final proof of process technical feasibility, determine the capital cost to convert the SCNC method to the magnesium nitrate method, indicate operating cost savings, and to provide data pertinent to the ion-exchange principle of reclamation of magnesium and nitrate ions for reuse in process for application to production facilities.

Phase I. Equipment - Mechanical Check

Check lists will be utilized to ensure that the equipment is checked for proper installation, including power source hookup, alignment of component parts, i.e., agitator drive unit, shaft, agitator, HNO<sub>3</sub> inlet line, water lines, air lines, MgCO<sub>3</sub> feed, steam lines, etc. Critical dimensions and clearances will be checked. All valves, gages, temperature sensors, orifices, and metering devices will be checked for proper installation and functionability. All level control devices, including air controls, high/low level alarms, and relays, will be checked for proper location and function and all systems will be checked for leakage. The weight indicating system, consisting of load cells, gages, transmitters, and air controls; detection systems and flow control systems; and motor speed controls will be checked for sensitivity to change and control response. Completed and signed copies of each check list will be placed on file with the official copy of the test plan. Equipment and installation drawings, manufacturers' data, and personal observations have been employed in preparation of module check lists as follows.



CHECK LIST

Merrick Scale Feeder

ITEM	YES	NO	REMARKS
<u>Installation</u>			
1. Power Source Hookup Correct			
2. Alignment of Component Parts Correct			
a. Drive Unit			
b. Feed			
c. Discharge			
d. Agitator			
e. Pump Impeller Rotation Proper			
3. Motors Properly Mounted			
a. Drive Guards Installed Properly			
4. Electrical			
a. Motor Properly Wired			
b. Grounding Megged and Functional			
c. Interlocks Functioning Properly			
d. Start-Stop Buttons Properly Wired			
(1) Automatic			
(2) Manual			
e. Alarms Function Properly			
f. Lighting Adequate			
5. Piping			
a. Adequately Supported			
b. Free of Leaks			
c. Installed for Proper Function			

Test Plan - Magnesium Nitrate Pilot Plant  
Page 3

Check List - Merrick Scale Feeder  
Page 2

ITEM	YES	NO	REMARKS
5. Piping (continued) d. Valves Properly Located  e. Valves Function Properly  f. High Level Liquid Sensors Function Properly.  g. Low Level Liquid Sensors Function Properly  h. Wash Out Capability Properly Installed  i. Gages Calibrated and Functional			
6. Bearings Properly Lubricated			
7. Bearings Properly Sealed			
8. Critical Dimensions Checked			
9. Critical Clearances Checked			
10. Air and/or Vacuum Systems			
a. Piping or Ducts Properly Installed			
b. Free of Leaks			
c. Pressure or Vacuum Valves Adequately Controlled			
11. Fail-Safe Valves Function Properly			
12. Other Checks Peculiar to This Equipment			

CHECK LIST

Attrition Mills

ITEM	YES	NO	REMARKS
<u>Installation</u>			
1. Power Source Hookup Correct			
2. Alignment of Component Parts Correct			
a. Drive Unit			
b. Feed			
c. Discharge			
d. Agitator			
e. Pump Impeller Rotation Proper			
3. Motors Properly Mounted			
a. Drive Guards Installed Properly			
4. Electrical			
a. Motor Properly Wired			
b. Grounding Megged and Functional			
c. Interlocks Functioning Properly			
d. Start-Stop Buttons Properly Wired			
(1) Automatic			
(2) Manual			
e. Alarms Function Properly			
f. Lighting Adequate			
5. Piping			
a. Adequately Supported			
b. Free of Leaks			
c. Installed for Proper Function			

Check List - Attrition Mills  
 Page 2

ITEM	YES	NO	REMARKS
5. Piping (continued) d. Valves Properly Located e. Valves Function Properly f. High Level Liquid Sensors Function Properly g. Low Level Liquid Sensors Function Properly h. Wash Out Capability Properly Installed i. Gages Calibrated and Functional			
6. Bearings Properly Lubricated			
7. Bearings Properly Sealed			
8. Critical Dimensions Checked			
9. Critical Clearances Checked			
10. Air and/or Vacuum Systems			
a. Piping or Ducts Properly Installed			
b. Free of Leaks			
c. Pressure or Vacuum Valves Adequately Controlled			
11. Fail-Safe Valves Function Properly			
12. Other Checks Peculiar to This Equipment			
		157	

CHECK LIST

Nitrators

ITEM	YES	NO	REMARKS
<u>Installation</u>			
1. Power Source Hookup Correct			
2. Alignment of Component Parts Correct			
a. Drive Unit			
b. Feed			
c. Discharge			
d. Agitator			
e. Pump Impeller Rotation Proper			
3. Motors Properly Mounted			
a. Drive Guards Installed Properly			
4. Electrical			
a. Motor Properly Wired			
b. Grounding Megged and Functional			
c. Interlocks Functioning Properly			
d. Start-Stop Buttons Properly Wired (1) Automatic			
(2) Manual			
e. Alarms Function Properly			
f. Lighting Adequate			
5. Piping			
a. Adequately Supported			
b. Free of Leaks			
c. Installed for Proper Function			

Test Plan - Magnesium Nitrate Pilot Plant  
 Page 7

Check List - Nitrators  
 Page 2

ITEM	YES	NO	REMARKS
5. Piping (continued) d. Valves Properly Located  e. Valves Function Properly  f. High Level Liquid Sensors Function Properly  g. Low Level Liquid Sensors Function Properly  h. Wash Out Capability Properly Installed  i. Gages Calibrated and Functional			
6. Bearings Properly Lubricated			
7. Bearings Properly Sealed			
8. Critical Dimensions Checked			
9. Critical Clearances Checked			
10. Air and/or Vacuum Systems			
a. Piping or Ducts Properly Installed			
b. Free of Leaks			
c. Pressure or Vacuum Valves Adequately Controlled			
11. Fail-Safe Valves Function Properly			
12. Other Checks Peculiar to This Equipment			

CHECK LIST

Centrifuge

ITEM	YES	NO	REMARKS
<u>Installation</u>			
1. Power Source Hookup Correct			
2. Alignment of Component Parts Correct			
a. Drive Unit			
b. Feed			
c. Discharge			
d. Basket			
e. Basket Rotation Proper			
3. Motors Properly Mounted			
a. Drive Guards Installed Properly			
4. Electrical			
a. Motor Properly Wired			
b. Grounding Megged and Functional			
c. Interlocks Functioning Properly			
d. Start-Stop Buttons Properly Wired (1) Automatic			
(2) Manual			
e. Alarms Function Properly			
f. Lighting Adequate			
5. Piping			
a. Adequately Supported			
b. Free of Leaks			
c. Installed for Proper Function			

Test Plan - Magnesium Nitrate Pilot Plant  
Page 9

Check List - Centrifuge  
Page 2

ITEM	YES	NO	REMARKS
5. Piping (continued) d. Valves Properly Located  e. Valves Function Properly  f. Wash Out Capability Properly Installed  g. Gages Calibrated and Functional			
6. Bearings Properly Lubricated			
7. Bearings Properly Sealed			
8. Critical Dimensions Checked			
9. Critical Clearances Checked			
10. Air and/or Vacuum Systems			
a. Piping or Ducts Properly Installed			
b. Free of Leaks			
c. Pressure or Vacuum Valves Adequately Controlled			
11. Fail-Safe Valves Function Properly			
12. Other Checks Peculiar to This Equipment			



CHECK LIST

Eimco Filter

ITEM	YES	NO	REMARKS
<u>Installation</u>			
1. Power Source Hookup Correct			
2. Alignment of Component Parts Correct			
a. Drive Unit			
b. Feed			
c. Discharge			
d. Pump Impeller Rotation Proper			
3. Motors Properly Mounted			
4. Electrical			
a. Motor Properly Wired			
b. Grounding Megged and Functional			
c. Interlocks Functioning Properly			
d. Start-Stop Buttons Properly Wired (1) Automatic			
(2) Manual			
e. Alarms Function Properly			
f. Lighting Adequate			
5. Piping			
a. Adequately Supported			
b. Free of Leaks			
c. Installed for Proper Function			
		16.	

Test Plan - Magnesium Nitrate Pilot Plant  
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Check List - Eimco Filter  
Page 2

ITEM	YES	NO	REMARKS
5. Piping (continued) d. Valves Properly Located  e. Valves Function Properly  f. High Level Liquid Sensors Function Properly on Receiving Tank  g. Low Level Liquid Sensors Function Properly on Receiving Tank  h. Wash Out Capability Properly Installed  i. Gages Calibrated and Functional			
6. Bearings Properly Lubricated			
7. Bearings Properly Sealed			
8. Critical Dimensions Checked			
9. Critical Clearances Checked			
10. Air and/or Vacuum Systems			
a. Piping or Ducts Properly Installed			
b. Free of Leaks			
c. Pressure of Vacuum Valves Adequately Controlled			
11. Fail-Safe Valves Function Properly			
12. Other Checks Peculiar to This Equipment			

CHECK LIST

Blending Tank

ITEM	YES	NO	REMARKS
<u>Installation</u>			
1. Power Source Hookup Correct			
2. Piping			
a. Adequately Supported			
b. Free of Leaks			
c. Installed for Proper Function			
d. Gages Calibrated and Functional			
3. Valves Installed and Functioning Properly			
a. Feed Lines			
b. Wash-out Water			
c. Pump Gland Water			
d. Steam Sparge			
4. Electrical			
a. Pump Properly Wired			
b. Grounding Megged and Functional			
c. Start-Stop Buttons Properly Wired			
5. Miscellaneous			
a. Platform Properly Spaced and Functional			
b. Alignment of Agitator Correct			

CHECK LIST

Slug Feeder

ITEM	YES	NO	REMARKS
1. Alignment of Component Parts Correct			
a. Main Unit Installed			
b. Acid Depth Indicator Installed			
c. Transition Pieces Installed			
2. Air Supply			
3. Controls			
a. Interlocks Functioning Properly			
b. Alarms Function Properly			
c. Seals Properly			

CHECK LIST

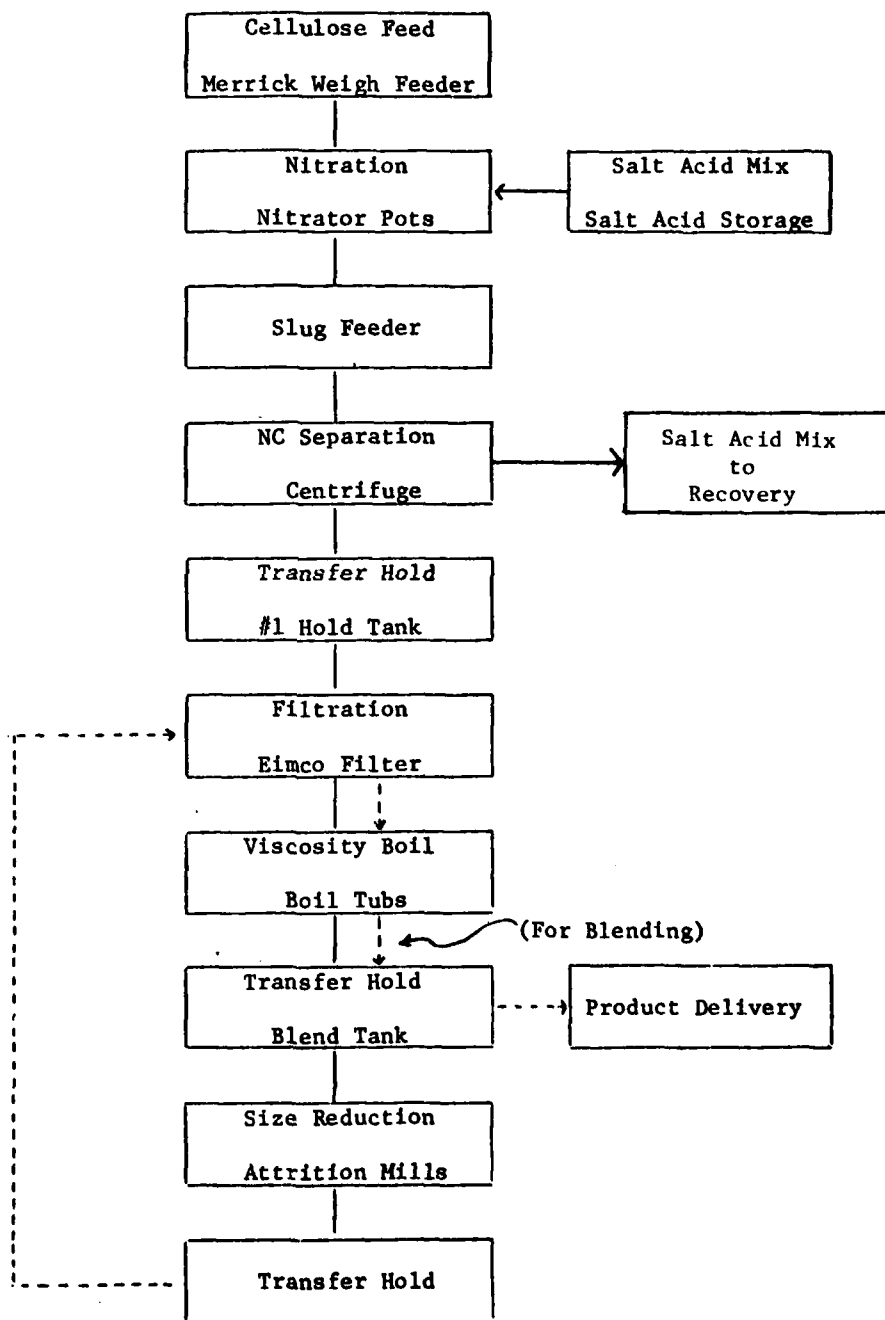
Boiling Tubs

ITEM	YES	NO	REMARKS
<u>Installation</u>			
1. Power Source Hookup Correct			
2. Valves Installed and Functioning Properly			
a. De-ionized and Filtered Water			
b. Decant Drain			
c. Slurry Exit			
d. Steam			
e. Air Line			
f. Slurry Entrance			
3. Piping			
a. Adequately Supported			
b. Free of Leaks			
c. Installed for Proper Function			
d. Vent to Atmosphere - Open			
e. Gages Calibrated and Functional			
4. Electrical			
a. Pump Properly Wired			
b. Grounding Megged and Functional			
c. Start-Stop Buttons Properly Wired			
5. Miscellaneous			
a. Platforms Properly Spaced and Functional			
b. Alignment of Agitator Correct		166	

FLOW

MAGNESIUM NITRATE PROCESS

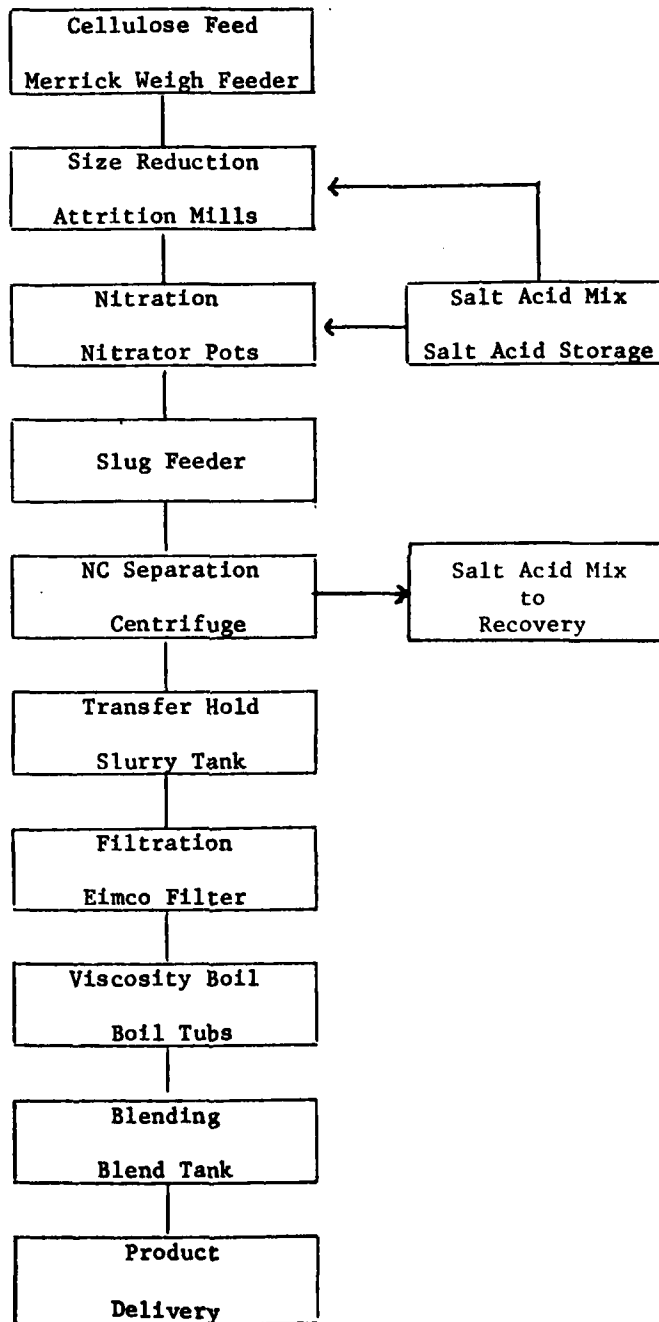
Nitration Initiated in Nitrator Pots



FLOW

MAGNESIUM NITRATE PROCESS

Nitration Initiated in Attrition Mills



## PHASE II.

Following the "dry run" in accordance with the check list, integration of the system will be evaluated by processing inert materials in conformance with the operational flow. Cellulose and water will be utilized as the inert materials and the ratio of water-to-cellulose will be varied as will the quantity in process to determine flow characteristics and capacities for handling slurries. This facility is designed and installed to provide flexibility in process technique with regard to utilization of the attrition mills. The design provides capability for initiation of nitration in the attrition mills or for initiation of nitration in the nitrating pots with subsequent utilization of the mills for sizing of product following the boiling operation.

### Process Evaluation When Utilizing Attrition Mills for Initiation of Nitration

Utilization of the mills for initiation of nitration during the systems evaluation with inert materials will proceed as follows. The inert material will be charged into the scale feeder which, in turn, will charge preweighed quantities into the attrition mills. Simulation of salt-acid flow to the attrition mills will be initiated. Interlocks which preclude the feeding of material into the mill unless the acid feed is operating will be checked for effectiveness under load conditions. From the attrition mills, the material will be charged by gravity into the nitrators. Checks will be made to determine the effectiveness under load of the interlocks which preclude the charging of material unless the agitators are operating and salt-acid is flowing. From the nitration pots the material will be charged by gravity into the centrifuge through the slug feeder which controls the quantity charged to ensure minimum and uniform cake thickness. Material is continuously discharged from the centrifuge by a reciprocating internal pusher. Wash systems will be checked under load conditions. Slurry make-up water, which is pumped into the discharge end of the centrifuge, will be varied to determine the desired consistency for most efficient processing.

Following centrifuging, the slurried material will flow into NC Hold Tank #1 and from there pumped to the Eimco dewatering filter. The functionability of the extractor under a load condition will be checked and optimum cake thickness, wash water feed, and belt speed will be established. Material will flow by gravity to the boil tubs. From the boil tubs the material will be fed into the blending tank and from there pumped into NC storage tank.

### Process Evaluation When Utilizing Attrition Mills for Sizing

Utilization of the mills for sizing following the boiling operation will proceed as follows. The inert material will be charged into the scale feeder which in turn will charge preweighed quantities into the nitration pots. Checks will be made to determine the effectiveness under load of the interlocks which preclude the charging of material into nitrators unless the agitators are operating and



salt-acid is flowing. From the nitration pots, the material will be charged by gravity into the centrifuge through the slug feeder which controls the quantity charged to ensure minimum and uniform cake thickness. Material is continuously discharged from the centrifuge by a reciprocating internal pusher. Wash systems will be checked under load conditions. Slurry make-up water which is pumped into the discharge end of the centrifuge will be varied to determine desired consistency for most efficient processing. Following centrifuging, the material will be fed into NC Hold Tank #1 and from there pumped to the Eimco dewatering filter. The functionability of the extractor under a load condition will be checked and optimum cake thickness, wash water feed, and belt speed will be established. Material will flow by gravity to the boil tubs.

Following boil, the material will be pumped into the Transfer Hold (blend) Tank and from there to the Attrition Mills for sizing. The material will flow out of the Attrition Mills into a specified boil tub and then be pumped into NC Hold Tank No. 2.

The  $Mg(NO_3)_2$  make-up system will be checked with water as the inert material. The performance of the agitator, metering devices, level control devices, high and low level alarms, and orifices will be observed. Load cells, gages, transmitters, and air controls for the weight indicating system will be checked with varying quantities of water. Detection and control systems for flow and motor speed will be checked under load conditions. Any necessary adjustments to settings will be made.

Upon completion of all inert runs, the desired ratios and load capacities for the system will be determined. Power requirements for motors under no-load and load conditions will be determined and recorded. Any changes to equipment and operating procedures which may have become apparent during the inert runs will be effected.

### PHASE III.

Confirmation of the process and the establishing of processing parameters utilizing live materials will begin following evaluation of the inert run results and the incorporation of any changes deemed necessary in equipment or operating procedures. This phase of work will consist of the manufacture of small batches of nitrocellulose by the magnesium nitrate process while varying ratios, temperatures, times, etc., to determine control ranges and to establish optimum operating conditions. Nitrocellulose will then be processed for utilization in propellant manufacture for comparison. The following studies will be included in this phase for nitrocellulose produced by the magnesium nitrate process:

1. Initiation of nitration in the attrition mills for correlation with conventional continuous nitrations.
2. Separation of nitrocellulose from nitrating acid, and determination of spent acid composition.

3. Centrifuge and countercurrent wash efficiency studies.
4. Purification boiling, blending variable analysis.
5. Assessment of corrosion of the pilot size to the full size manufacturing line.
6. Correlation of the pilot size nitric acid concentrator to the full size concentrator.

Evaluation of Scale Feeder Utilizing Inert Materials

1. Operation of unit interlocks will be checked so unit should not run when (a) acid mix, (b) attrition mills, or (c) nitrator agitators are not functioning.
2. Calibrate:  
Set controller in manual mode. Adjust output to 5 milliamps (maximum speed). Operate for warm-up at least one-half hour. Follow procedure in Merrick handbook for tare calibration and span calibration. Latter is accomplished with a test chain.
3. Operate feeder and mixed acid feed for proportional control checkout. Use cellulose collected and weighed in boxes and liquid feed to water.
4. Check out angle drive coupling alignment.
5. Establish best hopper capacity to maintain adequate flow and minimum variation in moisture in cellulose.

Evaluation of Scale Feeder Utilizing Live Materials

Equipment will be operated in accordance with applicable Unit Operating Procedure and General Operating Procedure. No samples will be taken for testing at this point.

Nitrating Acid Preparation Test Plan Utilizing Inert Materials

- A. Check out equipment.
  1. Check nitric acid feed line for proper valve opening.
  2. Check operation of nitric acid pump.
  3. Check scale tank that vents are open.
  4. Check transducer electrical continuity to panel board indicator.
  5. Check operation of scale tank pump.
  6. Check operation of agitator.

B. Preoperational checkout.

1. Water check scale tank and pump.
2. Water check high and low level alarms.
3. Check temperature control.
4. Add water above low level alarm.
5. Add 50-pound calibrated weights to tank and record. Add 50-pound increments up to 200 pounds.
6. Add water close to high level alarm position. Repeat Step 2.

Nitrating Acid Preparation Test Plan Utilizing Live Materials

1. Close discharge valve on salt/acid make-up tank.
2. Add 4,000 pounds of nitric acid (98%) to scale tank.
3. Record scale weight reading.
4. Start agitator and circulate.
5. Add 200 pounds of magnesium nitrate. Stop agitator and record weight. Circulate for 20 minutes.
6. Sample mix for laboratory analysis.
7. Add increments of 200 pounds of magnesium nitrate as in Steps 5 and 6. The total number of increments will be directed by Production Engineering engineer.

Evaluation of Nitrators Utilizing Inert Materials

1. Water check nitrator for leakage.
2. Water check high and low level alarms.
3. Calibrate weir setting for desired flow rate and nitrator cycle time.
4. Operate agitators to establish optimum speed for each pot to adequately drown cellulose and stir.
5. Check acid feed ring with water to ensure good wetting of cellulose.
6. Operate cellulose feeder and acid flow meter to check for safety interlock. System should stop when acid flow is interrupted.
7. Measure inlet and exit temperatures of water flowing through nitrator jacket.
8. Check efficiency of air purge line.
9. Dump cellulose and water to dump tank to check efficiency of dump system.

Evaluation of Nitrators Utilizing Live Materials

Equipment will be operated in accordance with applicable Unit Operating Procedures and General Operating Procedures. Samples of NC will be taken with a thief from the fourth nitrating pot. Samples from nitrator will be drowned in water.

Evaluation of Slug Feeder, Centrifuge, and Countercurrent Wash System Utilizing Inert Materials

1. Water check slug feeder for leakage.
2. Water check high level alarm in slug feeder.
3. Operate centrifuge and set timer on slug feeder.
4. Calibrate quantity of cellulose and water fed from the slug feeder per stroke.
5. Check efficiency of wash lines.
6. Check rate of cake buildup and uniformity of cake in the centrifuge.
7. Check efficiency of countercurrent washes.
8. Calibrate countercurrent wash sprays.
9. Check slurry tub overflow alarm.
10. Check centrifuge vibration switch operation.
11. Check use of Teflon rod to unplug feed line from slug feeder to centrifuge.
12. Check efficiency of slurry wash lines at front of centrifuge.
13. Check consistency of slurry in slurry tank (NC Hold Tank #1)
14. Establish method of sampling slurry at entrance to slurry tank.
15. Adjust countercurrent wash lines and calibrate flow meters.
16. Measure pressure of countercurrent washes.
17. Establish method of sampling from countercurrent wash lines.
18. Check emergency shutdown procedures for the slug feeder, centrifuge, and countercurrent wash system.

Evaluation of Slug Feeder, Centrifuge, and Countercurrent Wash System Utilizing Live Materials

After mechanical and inert check-outs have been performed, approved PPCB field review and ARMCOM safety pre-operational inspections are completed, a live evaluation will be conducted.

Follow applicable section of Test Plan.

Eimco Extractor Test Plan Utilizing Inert Materials

Equipment will be operated in accordance with applicable Unit Operating Procedure and General Operating Procedure. Cellulose and water slurry will be fed onto the belt at various, controlled feed rates to establish the rate commensurate with the optimum cake thickness on the belt. In addition, filter speed will be varied and the various speeds evaluated for effect on equipment efficiency until the optimum speed is established. Wash water flow rates will be varied and an optimum wash water rate determined. All values will be recorded as determined and incorporated into the operating instructions.

Eimco Extractor Test Plan Utilizing Live Materials

When material from No. 1 NC Hold Tank is being processed over the filter the wash water will be sampled twice per shift and analyzed for total acid.

Evaluation of Boiling Tubs and Blending and Storage Tanks Utilizing Inert Materials

A. Boiling Tubs

1. Close exit feed valve on boiling tub(s).
2. Close bottom drain valve.
3. Open entrance valve from Eimco filter to boiling tub(s).
4. Open vent line to atmosphere.
5. Check feed line to see that material is coming to tub. When material level is above agitator blades, start agitator motor.
6. Fill tub to approximately 12 inches below the overflow line.
7. Open valve to percolator line and adjust air feed.
8. Turn on steam to tub.
9. Check tub temperature periodically until temperature reaches 96°C (consider on boil).
10. During the time of bringing to boil, check system for leaks, etc.
11. Shut off steam.
12. Shut off air to percolator.
13. Tub can be drained to the sewer or transferred to the blend tub.
14. Repeat Steps 1 through 13 for each of five tubs.

B. Blending and Storage Tubs

1. Blending Tub

- a. Close outlet feed valve.
- b. Open inlet valve from boiling tubs.
- c. Check inlet line to assure material is being received from boiling tub.
- d. Start agitator.
- e. Check for leaks, etc.
- f. Material can be pumped to storage tubs, attrition mills, or drum loading.

2. Storage Tub

- a. Close outlet feed valve.
- b. Open inlet valves from blender tub tank.
- c. Turn on gland water to blender pump.
- d. Start blender pump.
- e. Open blender tank outlet feed valve.
- f. Check storage tank inlet line to assure that material is being received.
- g. Start agitator.
- h. Material can be pumped to blend tank, drum loading, or other storage tank.

Evaluation of Boiling Tubs and Blending and Storage  
Tanks Utilizing Live Material

After mechanical and inert check-out, approved PPCB field review and ARMCOM safety pre-operational inspection are completed, a live evaluation will be conducted.

A. Boiling Tubs

This evaluation will be conducted to determine the optimum purification treatment and treatment times needed to adjust viscosity and stabilize magnesium nitrate nitrocellulose from the prototype operation. Data accumulated from the bench-scale studies will be used to initiate this work.

The operation of all units will be performed in accordance with the applicable UOPs.

Analysis of data within the frame of testing may reveal the necessity to modify this Test Plan to obtain information at differently prescribed conditions of treatment and testing.

1. Summary of Tests to Be Accomplished

- a. After boiling tub is loaded, adjust acidity of material to less than 0.25 percent by either draining and washing or adding nitric acid. Sample for viscosity and  $N_2$ .
- b. Acid boil NC to adjust the viscosity of the NC and wash (boil time selected by Production Engineering engineer).
- c. Neutral boil and wash the NC twice to remove the residual acid. Sample the NC for a complete nitrocellulose analysis.
- d. Repeat Steps a through c for each boiling tub loaded.

B. Blending

1. When treatment of NC in the boiling tubs is completed and samples have been analyzed by the laboratory, a mathematical blend ratio of high grade and low grade NC will be calculated.
2. The calculated weights of each type NC will be pumped to the blend tub, agitated one hour, and sampled for a complete blend analysis (blending time may be increased or decreased, based on operating experience).
3. If the laboratory analysis meets the blend acceptance requirements, the material can be transferred to a storage tub, to containers for further processing, or sent to waste disposal.
4. If the laboratory analyses do not meet the blend acceptance requirements, adds will be made, the blend agitated, and analyzed until the blend acceptance requirements are met. The blend then can be transferred as outlined in Step 3.

C. Storage Tub

This tub can receive NC from the blender or from Slurry Tub #2, and can transfer NC to the blender, attrition mills, drum loading, or #4 Boiling Tub in C-Line NC Boiling Tub House.



Tests Required for Each NC Lot and Blend

A. Boiling Tubs (Directly After Nitration)

Tests: nitrogen, viscosity

B. Boiling Tubs (After NC Treatment)

Tests: nitrogen, solubility, viscosity, German, KI

C. Blender

Tests: nitrogen, solubility, viscosity, KI, German, AI, ash, fineness

The Magnesium Nitrate Pilot Plant facility is designed to provide flexibility in process technique. Variation of process flow and of equipment processing parameters will be pursued in plant evaluation to establish optimum parameters. The program plan for controlled variation of processing parameters is delineated in the test plan which will govern evaluation activities at the facility.

The need to vary parameters and process flow to properly evaluate the potential of the Magnesium Nitrate system precludes the initiation of a conventional training program, since some actions will be contingent on the results obtained from previous actions. The approach to personnel training, therefore, will be accomplished as follows. Personnel assigned to this project will thoroughly familiarize themselves with equipment layout, piping arrangements, and control consoles. Each engineer having a primary responsibility toward procurement and installation of the individual equipment items will explain his equipment to the group on site with regard to its process function, capabilities, alarms, and relationship to other equipment. Operating procedures will be discussed and hypothetical failures or malfunctions with appropriate corrective action(s) will be reviewed. Upon completion of this phase of training, inert runs will be initiated for system check-out and to provide on-the-job training for personnel.

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Sampling will be accomplished as specified on the chart below. All data will be recorded and identified in a manner congruous with efficient comparison of the effects realized from parameter variations. Data will be thoroughly analyzed prior to the establishment of optimum operating parameters.

<u>Sample Location</u>	<u>Frequency</u>	<u>Analysis</u>
Acid Mix	1/mix	TA
Acid Mix Storage		
Spent Acid Surge	1/shift	% HNO <sub>3</sub> , % Mg(NO <sub>3</sub> ) <sub>2</sub>
Spent Acid Storage	1/shift	% HNO <sub>3</sub> , % Mg(NO <sub>3</sub> ) <sub>2</sub>
Recovered Acid Surge	1/shift	% HNO <sub>3</sub> , % Mg(NO <sub>3</sub> ) <sub>2</sub>
Centrifuge Wash (Acid)	1/shift, each of 4 tanks	% HNO <sub>3</sub> , % Mg(NO <sub>3</sub> ) <sub>2</sub>
Belt Wash Accumulator	1/shift	TA solids
Eimco Wash	2/shift	TA
Spent Acid Fines		
Recovered Acid Fines		
Boiling Tub	2/tub 1/tub	TA N <sub>2</sub> , Sol., Germ., KI, Visc., AI, Fine.
NC Blend	1/blend, if acceptable	N <sub>2</sub> , Sol, Germ., KI, Visc., AI, Fine., Ash
NC Storage	As needed	
NC Hold Tank #1	As needed	
NC Hold Tank #2	As needed	
Poacher	No poaching on MCNC	
Boil Water Hold	As needed	
Water Fines		
Eimco Wash Line	2/shift	
60% Nitric Make-up	1/mix or tank	% HNO <sub>3</sub>
Ammonium Hydroxide Make-up	6 to calibrate	Then water temperature will be used to control
99% Nitric Storage	1/tank delivered	% HNO <sub>3</sub>
Magnesium Nitrate Make-up	1/mix	EDTA
Magnesium Nitrate Storage	1/mix	EDTA
Refrigeration Unit - Brine	1/month	% Mg(NO <sub>3</sub> ) <sub>2</sub> ; % Zn, Cu, Sn, Fe; SpG
Water Overflow Sump		Total solids; TA; ppm nitrates; ppm Mg <sup>++</sup>
Acid Overflow Sump		Total solids; TA; ppm nitrates; ppm Mg <sup>++</sup>
Air Samples		ppm NO; ppm NO <sub>x</sub>
Noise Level		Decibels
Nitrogens at Nitrator	3/shift	N <sub>2</sub>
Nitrogens at Centrifuge	3/shift	N <sub>2</sub>

SCALE FEEDER

<u>Run</u>	<u>Hopper Capacity</u>	<u>Chain Setting for Cellulose Feed, lb/hr</u>	<u>Water-Acid Feed, gal/min</u>	<u>Water Meter</u>
1		30	2.5	40:1
2				
3				
4		30	3.0	50:1
5				
6				
7		30	2.0	30:1
8				
9				
10		60	4.0	30:1
11				
12				
13		60	5.0	40:1
14				
15				
16		60	6.0	50:1
17				
18				
19		100	7.0	30:1
20				
21				
22		100	8.0	40:1
23				
24				
25		100	9.0	50:1
26				
27				

NITRATOR

Run	Weir Setting	NC Flow Rate, lb/hr	Cycle Time, min	Range Agitator Speed	Cellulose Feed Rate, lb/hr	Acid Feed Rate, gal/min	Temperature, °C		Ratio, A:C
							Inlet	Outlet	
100	70	90	35	A	60	2	50		30:1
101	70	68	35	A	45	2	55		40:1
102	70	54	35	A	36	2	60		60:1
103	105	150	25	A	100	4	50		30:1
104	105	126	25	A	84	4	55		40:1
105	105	90	25	A	60	4	60		60:1
106	90	117	30	A	78	3	50		30:1
107	90	90	30	A	60	3	55		40:1
108	90	72	30	A	48	3	60		60:1
109	90	150	20	A	100	4	50		30:1
110	90	126	20	A	89	4	55		40:1
111	90	90	20	A	60	4	60		60:1

ATTRITION MILLS (Nitration Initiation)

<u>Run</u>	<u>Ratio Controller Setting</u>	<u>Acid Feed Rate, gpm</u>	<u>Cellulose Feed Rate, lb/hr</u>	<u>Ammeter Reading</u>		<u>Weir Setting</u>	<u>Cycle Time, min</u>	<u>Nitrogen, percent</u>
				<u>Start</u>	<u>Operating</u>			
1	40:1					Top		
2	40:1					Top		
3	40:1					Top		
4	30:1					Top		
5	30:1					Top		
6	30:1					Top		
7	40:1					Bottom		
8	40:1					Bottom		
9	40:1					Bottom		
10	30:1					Bottom		
11	30:1					Bottom		
12	30:1					Bottom		
13	40:1					Halfway		
14	40:1					Halfway		
15	40:1					Halfway		
16	30:1					Halfway		
17	30:1					Halfway		
18	30:1					Halfway		

ATTRITION MILLS (Particle Size Uniformity)

<u>Run</u>	<u>Ammeter Reading</u>	<u>Quantity of Material</u>	<u>Total Sizing Time</u>
Each Boiling Tub	To be established to meet a fineness of 90-110 ml	600 or 1,000 gallons of slurry relative to two tub sizes	To be established

BLENDING

<u>Run</u>	<u>Blend - 13.15 N<sub>2</sub></u>		<u>Adds</u>	<u>Agitation Time Following Add</u>	<u>N<sub>2</sub> percent</u>	<u>Test Results</u>				
	<u>High Grade percent</u>	<u>Low Grade percent</u>				<u>Solubility percent</u>	<u>Viscosity sec</u>	<u>KI, min</u>	<u>Ash, percent</u>	<u>Fineness, ml</u>
Each Blend Tub	Approx. 70	Approx. 30	To be determined by analyses of blend	1 hour	13.10 to 13.20	37 to 47	8 to 20	35, 15, min.	0.4	90 to 110

POACHING

<u>Run</u>	<u>Weight of NC</u>	<u>Soda Ash Add</u>	<u>Tub Temperature</u>	<u>Boil Times</u>			
				<u>#1</u>	<u>#2</u>	<u>#3</u>	<u>#4</u>

There is no poaching treatment on the Magnesium Nitrate system.



MANUFACTURE OF 60% OR 30% NITRIC ACID

<u>Run</u>	<u>Make-up Tank Weight</u>	<u>Water Weight</u>	<u>99% HNO<sub>3</sub> Weight</u>	<u>Agitation Time</u>	<u>Tank Temperature</u>	<u>Approx. % HNO<sub>3</sub></u>	<u>% HNO<sub>3</sub> By Analysis</u>
1				5		30	
2				10		30	
3				15		30	
4				20		30	
5				25		30	
6				5		60	
7				10		60	
8				15		60	
9				20		60	
10				25		60	

BOILING TUB - INERT

<u>Run</u>	<u>Slurry Consistency</u>	<u>Settle Time</u>	<u>Agitation</u>	<u>Temperature Build Time</u>	<u>Tub Temperature</u>	<u>Air Feed Rate</u>	<u>Boil Time</u>	<u>Cool Time</u>	<u>Product Acidity</u>	<u>Product Viscosity</u>	<u>N2</u>
1	3%	1 hr	Full								
2	5%	1 hr	Full								
3	7%	1 hr	Full								
4	10%	1 hr	Full								

INERT CELLULOSE

LIQUID AND CELLULOSE SCALE FEEDER

Pulp - 100-108; Lint - 200-208; Same conditions.

<u>Run</u>	<u>Ratio</u>	<u>Cellulose Feed, pph</u>	<u>H<sub>2</sub>O, pph</u>
100	50:1	50	2,500
101	50:1	100	5,000
102	50:1	150	7,500
103	35:1	50	1,650
104	35:1	100	3,500
105	35:1	150	5,250
106	20:1	50	1,000
107	20:1	100	2,000
108	20:1	150	3,000

EIMCO EXTRACTOR - INERT

<u>Run</u>	<u>Feed Rate, percent</u>	<u>Cake Thickness</u>	<u>Filter Speed, rpm</u>	<u>Wash Water Flow Rate</u>	<u>Total Acid of Wash Water</u>	<u>Vacuum Reading, inch/Hg</u>
1	10		2, 5, 10, 15			5, max
2	20					(below atmospheric)
3	30					
4	40					
5	50					
6	60					
7	70					
8	80					
9	90					
10	100					

TEST PLAN

RECOVERY OF MAGNESIUM NITRATE AND NITRIC ACID FROM THE PROCESS STREAM

Run	Timer Settings, minutes										Record ppm Nitrates
	Cation					Anion					
Counter Setting	Back Wash	Acid In	Slow Rinse	Fast Rinse	Record ppm Mg <sup>++</sup>	Back Wash	Ammonium Hydroxide In	Slow Rinse	Final Rinse	Record Time	
1a	3	15	11	15	15	15	24	15	Until water quality is as good as set point on the conductivity monitors -	Record Time	
1b	3	20	11	15	15	20	24	15	"	"	
1c	3	10	11	15	15	10	24	15	"	"	
1d	3	15	15	15	15	15	24	15	"	"	
1e	3	15	8	15	15	15	24	15	"	"	
1f	3	15	11	15	15	15	28	15	"	"	
1g	3	15	11	15	15	15	20	15	"	"	
1h	1	15	11	15	15	15	24	15	"	"	
1i	2	15	11	15	15	15	24	15	"	"	
1j	4	15	11	15	15	15	24	15	"	"	

TEST PLAN

RECOVERY OF MAGNESIUM NITRATE AND NITRIC ACID FROM THE PROCESS STREAM

Run	Recirculation Auto/Man	Water Meter Readings, gal		Regeneration Auto/Man	Influent flow, gal/min	Conduc- tivity Setting	Regenerate Used, gal HNO <sub>3</sub> NH <sub>4</sub> OH	Pressure Reading, psig						
		Start	End					Tank 1	Tank 2	Tank 3	Tank 4			
1	Auto			Auto	28	0								
2	Auto			Auto	28	2								
3	Auto			Auto	28	4								
4	Auto			Auto	28	6								
5	Auto			Auto	28	8								
6	Auto			Auto	28	10								
7	Auto			Auto	28	12								
8	Auto			Auto	28	14								
9	Auto			Auto	28	16								
10	Auto			Auto	28	18								
11	Auto			Auto	28	20								
12	Man			Man	28	10								
13	Man			Man	28	12								
14	Man			Man	28	14								
15	Man			Man	28	16								
16	Auto			Auto	28	To Be Determined								

TEST PLAN

Recovery of Magnesium Nitrate and Nitric Acid from the Process Stream

<u>Run</u>	<u>Record Analysis of Influent</u>		<u>Water Required to Rinse, gal</u>		<u>Volume Influent Treated, gal</u>	<u>Total Cycle, minutes</u>	<u>Rinse Flow, gal/min</u>	<u>Remarks</u>
	<u>ppm Mg<sup>++</sup></u>	<u>ppm NO<sub>3</sub></u>	<u>Cation Column</u>	<u>Anion Column</u>				
1							28	
2							28	
3							28	
4							28	
5							28	
6							28	
7							28	
8							28	
9							28	
10							28	
11							28	
12							28	
13							28	
14							28	
15							28	
16							28	

TEST PLAN

RECOVERY OF MAGNESIUM NITRATE AND NITRIC ACID FROM THE PROCESS STREAM

<u>Run</u>	<u>Remarks</u>
2	Conditions as determined under 1 above.
16	
17	Both influent and rinse flow set for 24 gal/min.
18	Both influent and rinse flow set for 26 gal/min.
19	Both influent and rinse flow set for 30 gal/min.
20	Both influent and rinse flow set for 32 gal/min.



TEST PLAN

MANUFACTURE OF DEMINERALIZED WATER

Run	Recirculation <u>Auto/Man</u>	Record Water Meter Reading, gal		Regeneration <u>Auto/Man</u>	Water Flow, gpm	Conduc- tivity Setting	Record Pressure Readings, psig		Regenerate Used, gal		Remarks
		Start	End				At Start Tank 1	At End Tank 2	HNO <sub>3</sub>	NH <sub>4</sub> OH	
1	Auto			Auto	10.8	0					
2	Auto			Auto	10.8	2					
3	Auto			Auto	10.8	4					
4	Auto			Auto	10.8	6					
5	Auto			Auto	10.8	8					
6	Auto			Auto	10.8	10					
7	Auto			Auto	10.8	12					
8	Auto			Auto	10.8	14					
9	Auto			Auto	10.8	16					
10	Auto			Auto	10.8	18					
11	Auto			Auto	10.8	20					
12a	Man			Auto	10.8	8					
b	Man			Man	10.8	8					
13a	Man			Auto	10.8	10					
b	Man			Man	10.8	10					
14a	Man			Auto	10.8	12					
b	Man			Man	10.8	12					
15a	Man			Auto	10.8	14					
b	Man			Man	10.8	14					
16a	Man			Auto	10.8	16					
b	Man			Man	10.8	16					

TEST PLAN

MANUFACTURE OF DEMINERALIZED WATER

Run	Timer Settings, minutes										Total Cycle, minutes	
	Cation					Anion						
	Backwash	Acid In	Slow Rinse	Fast Rinse	Backwash	Ammonium Hydroxide In	Slow Rinse	Final Rinse	Final Rinse	Final Rinse		
1a	15	20	15	Until methyl orange indicator changes color - Record Time	15	20	15	Until water quality is as good as set point on the conductivity monitor - Record Time	15	20	15	15
1b	20	20	15	"	15	20	15	"	15	20	15	15
1c	10	20	15	"	10	20	15	"	15	20	15	15
1d	15	15	15	"	15	15	15	"	15	20	15	15
1e	15	25	15	"	15	25	15	"	15	20	15	15
1f	15	20	20	"	15	20	20	"	15	20	20	15
1g	15	20	10	"	15	20	10	"	15	20	10	10

2 As determined under Run 1 above.

As determined under Run 1 above.

16

Record  
Cake  
Moisture

Record  
Cake  
Thickness

Discharge  
Slurry

Wash #5

Wash #4

Wash #3

Wash #2

Wash #1

Centrifuge  
Speed

Slug  
Feeder,  
Cycles/min

Pusher  
Cycles/min

Ratio,  
Acid to  
Cellulose

Feed  
Rate,  
lb/hr

Maximum  
Mid Range  
Minimum

Continuous  
30  
25  
20  
15  
10  
5

Water  
Acid Wash  
Chilled Acid  
Recycle  
Belt Wash  
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Water  
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Chilled Acid  
Recycle  
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Water

(8 x 7 x 7 x 3 x 3) = 3528 Runs to Obtain All Variables)

Maximum  
Mid Range  
Minimum

Continuous  
30  
25  
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Water  
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(11 x 8 x 7 x 3) = 1848 Variables)

APPENDIX D

Pilot Plant Operating Procedures On File At  
Radford Army Ammunition Plant

## APPENDIX D

PILOT PLANT OPERATING PROCEDURES ON FILE AT RADFORD ARMY  
AMMUNITION PLANT

<u>Item</u>	<u>Procedure Number</u>	<u>Title</u>
1	4-26-40	General Operating Procedure
2	4-26-40(A)	General Sampling Procedure
3	4-26-40(B)	Carrier Refrigeration Unit Operation
4	4-26-40(C)	Operating Procedure for the Manufacture of Demineralized Water
5	4-26-40(D)	Manufacture of 30 Percent Ammonium Hydroxide
6	4-26-40(E)	Manufacture of 60 Percent or 30 Percent Nitric Acid
7	4-26-40(F)	Nitration Scale Feeder
8	4-26-40(G)	Nitrating Acid Feed and Nitrator
9	4-26-40(H)	Operation of S-8 Baker Perkins Continuous Centrifuge
10	4-26-40(I)	Dewatering Nitrocellulose Prior to Viscosity Boil
11	4-26-40(J)	Boiling Tub Operations
12	4-26-40(K)	Poaching Operations
13	4-26-40(L)	Attrition Mills - Sizing of Cellulose Nitrated by the Mixed Acid Method
14	4-26-40(M)	Blending Operations
15	4-26-40(N)	Operating Procedure for the Recovery of Magnesium Nitrate and Nitric Acid from the Process Stream
16	4-26-40(O)	Merco Rotary Strainer
17	4-26-40(P)	Durco Tubular Filters
18	4-26-40(Q)	Operating Procedure for Operation of the C. W. Thomson Communication System
19	4-26-40(R)	Use of the M.S.A. Demand Air Mask
20	4-26-40(S)	Operation of Dumbwaiter

APPENDIX E

A.S.M.E. Coded Pressure Vessels  
(Documentation on file at RAAP)

APPENDIX E

ASME CODED PRESSURE VESSELS IN PILOT PLANT

<u>Number</u>	<u>Item Name</u>	<u>Location</u>	<u>Design or Max. Operating Pressure</u>	<u>Manufacturer</u>	<u>RFA No.</u>	<u>National Board No.</u>	<u>Date of Manufacture</u>	<u>Date of Installation</u>	<u>Remarks or Vendor S/N</u>
1	Acid Fining Tank	Basement	Jacket 100 psi @ 360°F	Will-Flow Corp.	18783	2568	1974	August 1975	S/N 21944
2	Spent Liquor Surge	Basement	Jacket 110 psi @ 360°F	Will-Flow Corp.	18736	2569	1974	August 1975	S/N 21945
3	Brine Compression Tank	Basement	125 psi @ 375°F	Bell & Gossett Products	Catalog No. 60	396008	1973	Not used for pressure atmospheric tank	P.O. No. BR 27616
4	Make-Up Ion Exchange	Basement	100 psig	Watubo Ind.	18762	10986	1973-1974	Not in service	S/N 117310
5	Make-Up Ion Exchange	Basement	100 psig	Watubo Ind.	18762	10987	1973-1974	"	S/N 117311
6	Process Ion Exchange	Basement	100 psig	Watubo Ind.	18760	11031	1974	"	S/N 117312
7	Process Ion Exchange	Basement	100 psig	Watubo Ind.	18760	11032	1974	"	S/N 117313
8	Magnesium Nitrate Storage	2nd Floor	110 psi @ 360°F	Will-Flow Corp.	18731	2564	1974	"	S/N 21940
9	Spent Liquor Storage	Outside SE End	110 psi @ 360°F	Will-Flow Corp.	18734	2567	1974	August 1975	S/N 21943
10	Salt/Acid Storage (Nitrating Acid Feed)	Outside SE End	110 psi @ 360°F	Will-Flow Corp.	18733	2566	1974	August 1975	S/N 21942
11	Salt/Acid Mix (Recovered Acid Storage)	Outside SE End	110 psi @ 360°F	Will-Flow Corp.	18732	2565	1974	August 1975	S/N 21941
12	Magnesium Nitrate Make-Up	3rd Floor	250 psig @ 400°F	Industrial Alloy Fabricators, Inc	18742	83	1974	Not in service	S/N 25793
13	Instrument Air Dryer	3rd Floor West	200 psig @ 350°F	Henderson Engr. Co., Inc.	N/A	2205	1974	April 1975	S/N 3022
14	Instrument Air Dryer	3rd Floor West	200 psig @ 350°F	Henderson Engr. Co., Inc.	N/A	2173	1974	April 1975	S/N 2990
15	Concentrator Jacketed Tank	Basement West	110 psi @ 360°F	Will-Flow Corp.	19763	2833	1975	Not in service	500 gallon

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Commander  
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Readiness Command  
ATTN: DRSAR-LEP-L  
Rock Island, IL 61299

US Army Materiel Systems Analysis Activity  
ATTN: DRXSY-MP  
Aberdeen Proving Ground, MD 21005