

HERCULES INCORPORATED  
ALLEGANY BALLISTICS LABORATORY  
CUMBERLAND, MARYLAND

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SHOCK GEL PROCESS  
FOR  
SMALL ARMS GUN PROPELLANT

FINAL REPORT

(Unclassified - Department of the Army)

E. H. Zeigler and N. F. Stanley

June 1976

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Prepared for  
Department of the Army  
Frankford Arsenal  
Philadelphia, Pennsylvania

Contract DAAA25-73-C-0172

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The findings in this report are not to be  
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ABSTRACT

A program was conducted to develop the shock gel process for the manufacture of small arms gun propellants. A 3-5 lb/hr facility to demonstrate the continuous manufacture of 13.4% N nitrocellulose, shock gel gun propellant was designed and operated. Overall operation of the demonstration plant was generally satisfactory. Additional studies to simplify and improve the operation of the continuous leaching process were recommended.

The manufacture of shock gel gun propellant utilizing syrups with high nitrocellulose concentration (15% NC) was demonstrated. The resulting propellant had high absolute and gravimetric loading densities. This propellant, deterrent coated with 5% DBP, exceeded the velocity of reference ammunition at chamber pressures below 50,000 psi.

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## FOREWORD

This report covers work performed during the period July 1974 through June 1975 by Hercules Incorporated at Allegany Ballistics Laboratory, Cumberland, Maryland, under Modification 3 to Contract DAAA25-73-C-0172 for the U. S. Army, Frankford Arsenal, Philadelphia, Pennsylvania. Mr. Robert Pizzola managed the program for the Army. The Hercules program team included Dr. J. C. Allabashi, Research Division Manager, Mr. T. C. Rissell, Department Superintendent; Mr. E. Hays Zeigler, the principal investigator; and Mr. Norval F. Stanley, co-investigator. Messrs. G. B. Lancaster and J. R. Huzinec assisted in the round-the-clock operations. Messrs. C. F. Hager, J. E. McMahon, and M. S. White, Research Technicians, assisted in daily shock gel operations. Messrs. W. J. Conway, C. D. Zembower, and L. E. Stouffer, Research Technicians, assisted in round-the-clock shock gel operations.

## TABLE OF CONTENTS

<u>Section</u>	<u>Title</u>	<u>Page</u>
I	INTRODUCTION	1
II	OBJECTIVES	3
III	TECHNICAL DISCUSSION	4
	A. CHARACTERIZATION OF SHOCK GEL SYRUPS AT HIGH CONCENTRATIONS	4
	1. Rheological Characterization	4
	2. Processing of 15% NC Shock Gel Syrup	7
	3. Characterization of Gun Propellant Made From High NC Concentration Syrup	12
	B. EQUILIBRIUM BETWEEN SHOCK GEL NC AND NITROGLYCERIN AT 60°C IN NITROGLYCERIN-WATER SOLUTIONS	16
	1. Experimental Procedure	16
	2. Experimental Results	17
	C. 3-5 POUND PER HOUR SHOCK GEL DEMONSTRATION PLANT	24
	1. Design, Construction and Procurement	24
	2. Single-Base Operations	42
	3. Double-Base Operations	55
	4. 3-5 Lb/Hr Demonstration Plant Performance	64
IV	CONCLUSIONS AND RECOMMENDATIONS	70
	A. CONCLUSIONS	70
	B. RECOMMENDATIONS	70
APPENDIX I	PICTORIAL VIEWS OF SHOCK GEL 3-5 LB/HR DEMONSTRATION PLANT	71

LIST OF FIGURES

<u>Figure No.</u>	<u>Title</u>	<u>Page</u>
1	Apparent Viscosity of X170-25 as a Function of Reciprocal Absolute Temperature	9
2	Particle Size Distribution Analysis for Leached Shock Gel Pellets X170-25 73.0% Water	11
3	Percent NG in 13.4% N NC Shock Gels as a Function of NG Concentration in NG/H <sub>2</sub> O Solutions @ 60°C	18
4	NG and Water Content as a Function of Contact Time	19
5	Preliminary Flow Sheet for Continuous Shock Gel Process	25
6	Shock Gel Solution Tanks	28
7	Detail of Tank No. 1	28
8	Shock Gel Syrup Metering Pump	30
9	Control Panel for Solution Tanks and Metering Pump	30
10	Shock Gel Syrup Filter and Housing	32
11	Shock Gel Streaming Head	32
12	Dewatering Screen No. 1	36
13	Screen No. 2 and Impregnation Column Feed Pump	41
14	Overhead View of Screen No. 2, Head Tank, Feed Pump and NG Impregnation Column	41
15	MeC Concentration as a Function of Column Height - Run X170-42	49
16	MeC Concentration as a Function of Leach Column Height - Run X170-44	52
17	Particle Size Distribution Analysis for Shock Gel X170-44	54
18	Hindered Settling Velocity of X170-44 Gels as a Function of Volume Fraction Gels	56

LIST OF FIGURES (CONT.)

<u>Figure No.</u>	<u>Title</u>	<u>Page</u>
I-1	Overall View of Shock Gel Plant	72
I-2	Lower Level Control Panel With 300 Gallon NG Tank Leach Column and NG Impregnation	73
I-3	Streaming Head in Operation With N <sub>2</sub> Sparge Shield in Place	74
I-4	Lower Portion of Leach Column, NG Impregnation Column	75
I-5	Dewatering Screen No. 1	76
I-6	Dewatering Screen No. 2 Feeding Gels to NG Impreg- nation Column Feed Pump	77

LIST OF TABLES

<u>Table No.</u>	<u>Title</u>	<u>Page</u>
I	LOT ANALYSIS OF SHOCK GEL NITROCELLULOSE LOTS	5
II	RHEOLOGICAL PROPERTIES OF SHOCK GEL SOLUTIONS	6
III	APPARENT VISCOSITY - BROOKFIELD MOD. LVT VISCOMETER, SPINDLE NO. 2	7
IV	VISCOSITY REDUCTION OF X170-25 SHOCK GEL SYRUP	8
V	ANALYSIS OF X170-25 SHOCK GELS	10
VI	COMPARISON OF ABL DUPONT DENSIMETER AND MOD. SLD	12
VII	FIRING RESULTS OF UNCOATED BASE GRAIN X170-25	13
VIII	DBP COATING VARIABLES (5% DBP)	13
IX	GUN FIRING TESTS OF DBP-COATED POWDER	15
X	GUN FIRING TESTS OF HERKOTE-COATED SHOCK GEL	16
XI	ANALYSIS OF 13.4% N NC SHOCK GELS FROM NG IMPREGNATION EXPERIMENTS	20
XII	DATA USED FOR CALCULATION OF MASS TRANSFER COEFFI- CIENTS	26
XIII	IMMERSION TESTS IN METHYL CARBITOL AT ROOM TEMPERA- TURE	35
XIV	CONDITIONS IN COUNTERCURRENT NG IMPREGNATOR DESIGNED FOR AVERAGE PELLET	38
XV	MATERIAL BALANCE-SHOCK GEL LEACH COLUMN AT STEADY STATE	51
XVI	SCREEN ANALYSIS OF 64% MeC-WET SHOCK GEL FROM RUN X170-44	53
XVII	DOUBLE-BASE SHOCK GEL PELLETS FROM RUN OF 11 JUNE 1975	59
XVIII	DOUBLE-BASE SHOCK GEL PELLETS FROM RUN OF 27 JUNE 1975	64
XIX	DEWATERING SCREEN OPERATING PARAMETERS	67



## SECTION I

### INTRODUCTION

Briefly, the shock gel process consists of dissolving fibrous nitrocellulose (NC) in a water-soluble solvent to form a syrup. The syrup is then forced through a multi-perforated plate to form streams which break up into spherical droplets and fall into a gelling bath. The gelling bath consists of a solvent/water solution outside the solubility range of the NC. In this bath, the NC precipitates out of solution and forms a spherical membrane with the syrup trapped inside. As the solvent concentration in the droplet is reduced by diffusion, a gel structure of precipitated NC is formed. Subsequent leaching exchanges solvent for water to produce a water-NC gel of spherical shape and uniform size. When the gels are dried, they shrink irreversibly into dense balls of hard, horny NC. To make a double-base propellant, the water-logged gels may be contacted with an NC/water solution. In this manner, NC contents of any practical level for gun propellants can be obtained.

The process was originally developed for the manufacture of double-base rocket propellants using nitrocellulose of 12.6% N and a diethylene glycol (DEG) solvent.

Contract DAAA25-70-C-0710 for Frankford Arsenal, performed in 1970-71, examined methods of adapting the basic process to the manufacture of small arms gun propellants using blended nitrocellulose of 13.2% and methyl "Carbitol" solvent. Results from this effort were encouraging, but the product manufactured had low gravimetric loading densities and poor particle shape. Ballistic results were unimpressive.

In Hercules-funded research on shock gel gun propellants performed under RI-65 in August and October 1972, further work was done utilizing blended NC of 13.2% N. Surface finish, shape, and gravimetric loading density were improved through the use of diethylene glycol - methyl "Carbitol" blended solvent combined with cushioning techniques and external energy input to the falling stream of shock gel solution.

Under Contract DAAA25-73-C-0172 for Frankford Arsenal during the period October 1972-January 1974, four NC-solvent, shock gel systems were compared: (1) 12.6% N - diethylene glycol (DEG); (2) 13.15% N (blended) - DEG/diethylene glycol monomethyl ether (MeC); (3) 13.15% N (directly nitrated) - DEG/MeC; and (4) 13.4% N - MeC. The 13.4% N - MeC system was found to be superior. Using this NC-solvent system, shock gel gun propellant base grain having a narrow particle size distribution, high absolute density, and a high gravimetric loading density was manufactured. Gun

firing tests were conducted which resulted in a deturred powder having a high gravimetric loading density and satisfactory 5.56mm ballistic performance at high, ambient, and low temperatures. A preliminary concept of a continuous shock gel process was developed, and a preliminary economic analysis was made for prototype and production scales which showed that shock gel gun propellants could be produced at a competitive cost.

The program described in this report, Modification 3 to Contract DAAA25-73-C-0172, sponsored by Frankford Arsenal, covered the period June 1974 through June 1975. Further investigations in the shock gel process were conducted with an emphasis on techniques of continuous gun propellant processing.

## SECTION II

### OBJECTIVES

The overall objective of this program was to demonstrate the feasibility of continuous operation of the critical stages of a 3-5 lb/hr facility for the preparation of shock gel gun propellants containing 13.4% N nitrocellulose and suitable for the 5.56mm cartridge.

Specific goals for this program were:

- (1) Demonstrate the feasibility of processing shock gel syrups of high nitrocellulose concentrations
- (2) Design and operate a 3-5 lb/hr, continuous ball formation and leaching facility
- (3) Design and operate a 3-5 lb/hr, continuous nitroglycerin impregnation facility

## SECTION III

### TECHNICAL DISCUSSION

#### A. CHARACTERIZATION OF SHOCK GEL SYRUPS AT HIGH CONCENTRATIONS

Shock gel syrups having high NC concentrations are of interest because of economic considerations. Numerous calculations of the economics of the shock process have shown that costs are reduced by processing a more concentrated syrup. Operating costs, per pound of powder, are reduced by 10-15% by changing from a 5% to a 10% NC syrup, depending on plant scale.<sup>(1)</sup> Similar reductions would be expected in changing from a 10% to a 15% syrup.

##### 1. Rheological Characterization

The rheological characteristics of shock gel syrups are of interest with regard to process design of critical shock gel operations such as syrup makeup, syrup pumping, filtration and streaming. The apparent viscosity of syrups measured at any arbitrary shear rate can be used for certain process control operations with enough experience in the particular means of measurement. However, the rigorous solution of certain types of fluid flow calculations requires knowledge of the shear stress as a function of shear rate.

For these reasons the rheological properties of shock gel syrups of 7%, 10% and 15% NC were examined using a Haake rotating viscometer at 50°C, 60°C, and 70°C. Two lots of nitrocellulose of 13.4% nitrogen were used for the measurements. One lot, X-495 (ABL lot No.), was procured from the Hercules Parlin, NJ, plant for use in the 3-5 lb/hr demonstration plant, and the other lot, X-492 (ABL lot No.), had been used for all previous studies of 13.4% N shock gel.

Table I shows the Hercules Parlin analysis of the two lots. Viscosity test solutions were made up utilizing the two nitrocellulose lots.

The Haake Rotoviscometer which was used to characterize the rheological properties of the solutions measures the torque necessary to rotate a cylindrical bob suspended in the sample. The sample is contained in a temperature-conditioned polyethylene cup. The readings for a particular

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(1) Zeigler, E. H. and Stanley, N. F., "Shock Gel Process for Small Arms Gun Propellants," Final Report, Vol. II, Hercules Incorporated/Allegany Ballistics Laboratory, February 1974.

shear rate can be converted to an apparent viscosity. By the use of regularly checked calibration factors for the unit, the shear stress can also be calculated. The data on shear stress, as a function of shear rate, can also be used for evaluation of the power law parameters  $m$  and  $n'$  which are defined by  $\eta_{app} = m\dot{\gamma}^{n'-1}$  where  $\eta_{app}$  is the apparent viscosity,  $m$  is the power law viscosity,  $\dot{\gamma}$  is the shear rate and  $n'$  is the power law exponent. For Newtonian fluids  $n'$  is unity and  $m$  reduces to the Newtonian viscosity. For the case of  $n'$  less than unity, the fluid is termed pseudoplastic while for  $n'$  greater than unity the fluid is dilatant. Both  $m$  and  $n'$  can be evaluated from a log-log plot of shear stress as a function of shear rate. The slope of the plot is  $n'$ , and  $m$  is the numerical value of shear stress at a shear rate of unity and is equal to  $\eta_{app}$  in magnitude only. Table II summarizes the rheological properties of the solutions as measured on the Haake Rotoviscometer.

TABLE I  
LOT ANALYSIS OF SHOCK GEL NITROCELLULOSE LOTS

	Lot No.	
	X-495	X-492
Nitrogen (%)	13.40	13.35
Acetone Insolubles (%)	Trace	Trace
Ash (%)	0.02	0.01
134.5°C Heat Test (min.)	30	30
Ether/Alcohol Solubility (%)	8.0	11.4
Viscosity (sec)	10	13
Fineness (ml)	91	88
Moisture (%)	31.0	32.4

Table III summarizes data on the two 7% solutions taken with a Brookfield viscometer.

The difference in apparent viscosity between solutions made with the 10-sec and those made with the 13-sec NC was not consistent when measured with either the Haake or the Brookfield viscometer. In general, when a

TABLE II  
RHEOLOGICAL PROPERTIES OF SHOCK GEL SOLUTIONS

% NC	Type NC	% MeC	% H <sub>2</sub> O	Power Law Parameters (l)												Remarks
				50°C				60°C				70°C				
				m	n'	m	n'	m	n'	m	n'	m	n'	m	n'	
7	13 sec	90	3	58.7	0.92	58.7	0.89	27.7	0.90	--	--	--	--	--	--	
7	10 sec	90	3	34.1	0.99	33.7	0.97	31.9	0.88	--	--	--	--	--	--	
10	13 sec	85.7	4.3	--	--	--	--	--	--	--	--	--	--	--	Gelled on solution	
10	10 sec	85.7	4.3	--	--	--	--	--	--	--	--	--	--	--	Gelled on solution	
10	13 sec	90	-	820	0.60	261	0.79	167	0.84	--	--	--	--	--	--	
10	10 sec	90	-	368	0.79	215	0.84	177	0.83	--	--	--	--	--	--	
15	13 sec	85	-	2197	0.55	1800	0.56	1178	0.59	--	--	--	--	--	--	
15	10 sec	85	-	3485	0.66	1038	0.85	300	1.00	--	--	--	--	--	--	

(11) Calculated from shear stress vs. shear rate plot.

large difference was observed, it was in the expected direction, i.e., a lower apparent viscosity for the solution made with the 10-sec NC.

TABLE III

APPARENT VISCOSITY - BROOKFIELD MOD. LVT VISCOMETER, SPINDLE NO. 2

% NC	Type NC	% MeC	% H <sub>2</sub> O	Spindle Speed (rpm)	Viscosity (cps)		
					50°C	60°C	70°C
7	13 sec	90	3	0.3	5320	3990	-
				0.6	5990	3660	2660
				1.5	5850	3860	2790
				3.0	5990	3920	2860
				6.0	6160	4000	2960
				12.0	Off	Off	2950
				Scale	Scale		
7	10 sec	90	3	0.3	-	-	-
				0.6	5990	4000	3330
				1.5	5850	3990	3190
				3.0	5920	3990	3130
				6.0	6060	4060	3000
				12.0	Off	Off	2960
				Scale	Scale		

## 2. Processing of 15% NC Shock Gel Syrup

As previously mentioned, high-concentration shock gel syrups are desirable for economic reasons. In previous programs the highest NC concentration used for shock gel gun propellant manufacture was 10% NC. As a feasibility demonstration, a 15% NC syrup was prepared and shock gelled in the batch system used in previous shock gel investigations. Nine pounds of X-495 nitrocellulose containing 3.2 lb of water (26.3% water-wet) was dissolved in 51 lb of methyl carbitol. An amount of 2-nitrodiphenylamine (2-NDPA) equivalent to 2% of the NC was included in the solution. The solution, as initially formed, was a gelatinous mass due to the high water concentration (~ 5.9% of the MeC plus H<sub>2</sub>O).

Prolonged heating at temperatures up to 85°C combined with vacuum and agitation was required to reduce the water content and solution viscosity to usable levels. A dry ice trap in the vacuum line was utilized to keep track of the water removed and the amount of MeC to be added back. Table IV shows the progress in viscosity reduction.

TABLE IV

## VISCOSITY REDUCTION OF X170-25 SHOCK GEL SYRUP

Time From Initial Makeup (hours)	% H <sub>2</sub> O In Solution	Brookfield Viscosity (cps) At Temperature (°C)
0	5.06	--
144	0.2	16,200 @ 79
168	0	11,200 @ 77
237	0	4,000 @ 81
261	0	3,100 @ 85

At 237 hours a viscosity-temperature profile was taken as shown in Figure 1. The viscosities measured at earlier times are shown for comparison. The recorded viscosities indicated that the solution should be usable at temperatures of 80°-85°C. This was confirmed by a short (10 minute) streaming run into a water gel bath using the vibrating streaming head mounted at a height of 8 feet.

For the actual streaming run the head was lowered to 7 feet above the gel bath. The run was conducted with the syrup at a temperature of 85°C. The syrup viscosity measured immediately before streaming was 3060 cps at this temperature (Brookfield viscometer).

The gel bath consisted of 55 gallons of 62% MeC at 20°C. This was less total gel fluid than normally used because the additional catch tank, recirculating pump, etc., had been dismantled to make room for the continuous demonstration plant.

Streaming of the solution took place in two runs because blinding of the streaming head filter screen by MeC (and acetone) insolubles severely reduced throughput as the run progressed. In the first run, streaming was initiated at 17 psig, but when the run was terminated after 100 min, 60 psig was necessary to obtain equivalent flow.

After the streaming head had been cleaned, a second run was initiated under similar conditions except that the head was lowered to



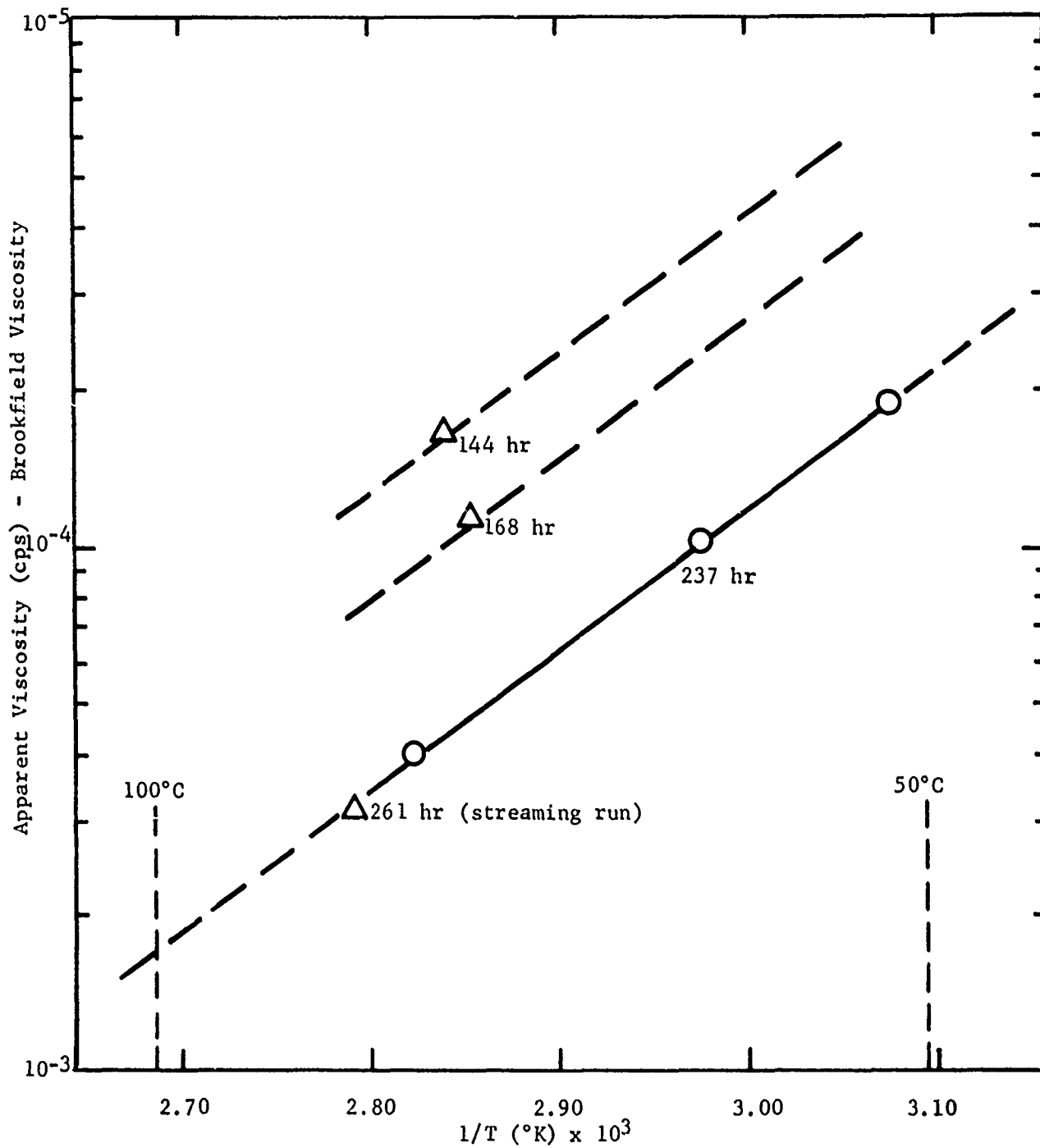


Figure 1. Apparent Viscosity of X170-25 as a Function of Reciprocal Absolute Temperature

6 feet and the gel bath concentration was reduced to 58% MeC by the addition of the supernatant leach liquid from the first portion of gels (which were being processed separately). During the second run pressure was varied from an initial 15 psig up to 50 psig at the termination after 67 min streaming.

Both batches were handled separately through leaching. Moisture analysis showed that both contained 73.0% internal moisture after leaching, and microscopic examination did not show any significant differences. The two batches were then combined and designated X170-25.

A screen analysis of the combined lot was made after all material had been passed through a No. 10 screen (0.0787 in. hole size) to remove gels that resulted from holes which were dripping rather than streaming as a result of screen plugging. The resulting particle size distribution analysis is shown in Figure 2. The weight median particle size indicated by this analysis was 0.042 in. for the leached pellets. A dry single-base pellet of the medium particle size would thus be ~0.024 in. in diameter.

Fifteen and one-half pounds of the leached pellets were NG impregnated to 5.42% NG in a single-step batch operation. An impregnation time of 2.75 hours at 60°C was allowed. After impregnation the gels contained 67.4% moisture.

The gels were dried to 0.72% moisture over a period of 225 min in the same "semi-fluidized" bed drier used in previous studies.

The dried gels were submitted for analysis with the results shown in Table V.

TABLE V  
ANALYSIS OF X170-25 SHOCK GELS

NG (%)	5.41, 5.42
NDPA (%)	0.78, 0.77, 0.77
Total Extractables (%)	6.12, 6.14
Absolute Density (gm/cc)	1.617, 1.627 (Hg Pycnometer)

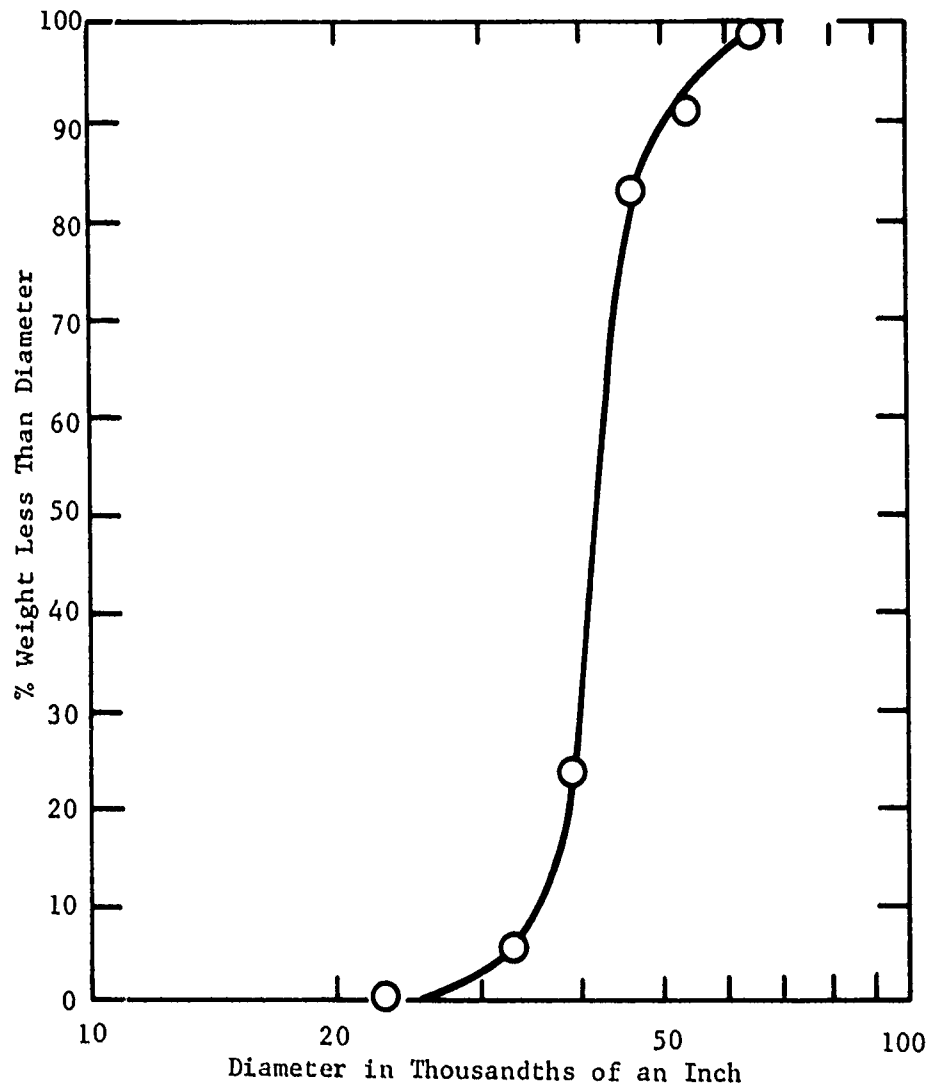


Figure 2. Particle Size Distribution Analysis for Leached Shock Gel Pellets X170-25, 73.0% Water

A 400-gm sample was glazed with 0.3% graphite, and the gravimetric loading density (GLD) was measured using a locally fabricated replica of a duPont Densimeter. The gravimetric loading density was determined to be 1.009 gm/ml which was equal to the highest previously measured GLD which was measured with a modified screen loading density (Mod. SLD). Both the ABL duPont Densimeter and Mod. SLD gave approximately equivalent results as shown in Table VI for two lots of WC-846 ball powder.

TABLE VI  
COMPARISON OF ABL DUPONT DENSIMETER AND MOD. SLD

WC-846 Lot No.	Gravimetric Loading Density		
	duPont Densimeter (gm/ml)	Mod. SLD (gm/ml)	Frankford duPont Densimeter (gm/ml)
AL 43136	0.987	0.987	-
AL 47101	1.020	1.017	1.021

Microscopic examination of X170-25 revealed well-formed spheres and spheroids with very smooth, almost glass-like surfaces.

The experiment indicated the basic feasibility of working with 15% NC shock gel syrups if the correct starting material was available. Starting with water-wet, 10 sec NC, a prolonged period of vacuum agitation was necessary to remove enough water to form a smooth solution, and additional cooking was then required to degrade the NC so that a usable viscosity was obtained. Some, if not all, of this time could be eliminated through the use of drier NC, partial replacement of water with MeC, more efficient water removal, use of a lower viscosity NC as a starting material, and the use of higher temperatures. All of these solutions would be considered in a large-scale production plant.

### 3. Characterization of Gun Propellant Made From High NC Concentration Syrup

The dry, double-base product from shock gel run X170-25 (15% NC syrup) was deterrent coated and test fired in a 5.56mm test fixture. Three 350 gm lots of the base grain were coated with dibutyl phthalate (DBP) and one lot was coated with Herkote. Before deterrent coating, some of the uncoated base grain was test fired with the results shown in Table VII.

TABLE VII  
FIRING RESULTS OF UNCOATED BASE GRAIN X170-25

Lot	Coating	Charge Weight (Grains)	Average Corrected Velocity @ 15 ft (ft/sec)	Average Corrected Peak Pressure-Copper Crusher (psi)	No. Shots
Reference	Lot LC-Y-5.56-905	-	3202	47,200	5
X170-25	None	15.0	2424	35,900	5

Based on these firing results, a coating level of 5% DBP was selected.

Each lot was coated by use of a slurry technique in a 3-liter, baffled container agitated by a high-speed propellant. The DBP coating runs differed from one another except in the coating amount as shown in Table VIII.

TABLE VIII  
DBP COATING VARIABLES (5% DBP)

Run	Powder Wt. (gm)	Water Wt. (gm)	Emulsifying Agent and Amount (gm)	Coating Temp. (°C)	Coating Time (Hr)	Loading Density (gm/ml)
X170-28	350	2500	Gelatin (3.5)	75	6	0.915
X170-29	350	1200	Mono-sulfonated (4.6) castor oil	70	12	0.896
X170-30	350	2500	Mono-sulfonated (5.0) castor oil Zonyl FSP (0.25)	75	3	0.916

A fluorochemical surface active agent, Zonyl FSP, was used in combination with the mono-sulfonated castor oil when it was found that

this resulted in a more stable DBP in water emulsion than when only the primary agent was used.

Agglomeration of the powder during or after coating was not a problem. The surface of the DBP-coated powder was characterized by severe dimpling as if localized swelling due to contact with the DBP emulsion had occurred. Only minor differences could be detected in the surface finish of the three lots. The gravimetric loading densities shown in Table VIII were measured in the ABL replica duPont Densimeter after the powder had been glazed with 0.3% graphite for 3-1/2 hours.

Gun firing tests of the DBP coated lots were conducted in a 5.56mm pressure barrel with the results shown in Table IX.

Lot Xi70-29, which had been subjected to a coating time of 1<sup>o</sup> hours, had the best velocity-pressure relationship. A velocity greater than that of the reference lot was achieved at a peak chamber pressure of < 50,000 psi.

The gravimetric loading density of the DBP-coated powder was disappointingly low because of surface degradation. A possible cause of this degradation was a difference in the susceptibility of the shock gel particle surface to attack by plasticizers as compared with ball powder. The "case hardening" undergone by ball powder during manufacture may make it more resistant to the type of deformation observed with DBP-coated shock gel powder.

A fourth coating run was conducted utilizing Herkote at the 4% level. Three hundred and fifty grams of base grain were coated in 2000 gm of water containing 3.5 gm of mono-sulfonated castor oil and 0.2 gm of Zonyl FSP. The Herkote was dissolved in 50 ml of 50/50 ethyl acetate/heptane and emulsified in 150 ml of the slurry liquid. The emulsion was added to the slurry all at once at a temperature of 50°C. The temperature of the slurry was then raised to 93°-94°C for 5 hours. After cooling, draining, and rinsing, the powder was dried at 60°C and glazed with 0.3% graphite for 3-1/2 hours. A gravimetric loading density of 0.969 gm/ml (duPont Densimeter) was measured for the finished powder.

The powder was subjected to gun firing tests but proved to be too "quick" ballistically. Gun firing results are shown in Table X.

Lot X170-31 with 4% Herkote had approximately the same performance as Lot X170-28 deterred with 5% DBP for 6 hours although the loading density was considerably higher.

TABLE IX

## GUN FIRING TESTS OF DBP-COATED POWDER

Lot	Coating	Charge Weight (Grains)	Average Corrected Velocity @ 15 ft (ft/sec)	Average Corrected Peak Pressure-Copper Crusher (psi)	No. Shots
Reference	LC-Y-5.56-905		3202	47,200	5
X170-30 - 3 hr coat- ing time	5% DBP	18.0	2508	29,100	2
"	5% DBP	20.0	2688	33,400	2
"	5% DBP	22.0	2880	39,100	2
"	5% DBP	24.0	3027	43,400	2
"	5% DBP	25.0	3148	46,600	2
"	5% DBP	25.5	3231	52,500	5
"	5% DBP	26.0	3211	52,200	2
X170-28 - 6 hr coat- ing time	5% DBP	18.0	2603	31,000	2
"	5% DBP	20.0	2748	34,600	2
"	5% DBP	22.0	2933	39,400	2
"	5% DBP	24.0	3082	44,900	2
"	5% DBP	25.0	3262	56,100	2
"	5% DBP	26.0	3326	60,300	1
X170-29 - 12 hr coat- ing time	5% DBP	18.0	2536	28,100	2
"	5% DBP	20.0	2703	33,100	2
"	5% DBP	22.0	2845	36,700	2
"	5% DBP	24.0	3054	42,200	2
"	5% DBP	25.0	3109	45,000	2
"	5% DBP	26.0	3156	47,100	2
"	5% DBP	26.5 (C)	3237	48,500	5
"	5% DBP	27.0 (C)	3263	52,000	2

(C) Compressed Charge.

TABLE X

## GUN FIRING TESTS OF HERKOTE COATED SHOCK GEL

Lot	Coating	Charge Weight (Grains)	Average Corrected Velocity @ 15 ft (ft/sec)	Average Corrected Peak Pressure-Copper Crusher (psi)	No. Shots
Reference	LC-Y-5.56-905	-	3202	47,200	5
X170-31	4% Herkote	22.0	2992	41,400	2
		24.0	3183	49,500	2
		24.2	3236	53,900	5
		25.0	3267	55,800	2

From the results of these gun firing test, it appeared that a coating level of ~5.5% DBP or 5% Herkote would offer superior ballistic performance. The 12-hour DBP coating period seemed to offer substantially improved pressure-velocity relationships as compared with a 3 or 6 hour coating period.

B. EQUILIBRIUM BETWEEN SHOCK GEL NG AND NITROGLYCERIN  
AT 60°C IN NITROGLYCERIN-WATER SOLUTIONS

Experiments were conducted to determine the equilibrium relationship between 13.4% N shock gels and NG/H<sub>2</sub>O solutions whose concentrations ranged from 0.04% to 0.24% NG at 60°C.

1. Experimental Procedure

The experimental procedure was to make 200 lb of NG/H<sub>2</sub>O solution by addition of the required amount of NG (as a 75% solution in acetone) to 200 lb of deionized water at 60°C. The NG was dissolved by rapid agitation for 10-15 min with an air-motor driven Cowles Dissolver. The acetone (b.p. 56.5°C) was driven off almost immediately upon addition.

One hundred grams of 80.0% water-wet shock gel pellets were externally dried and equally distributed between five small baskets made from 60-mesh stainless steel screen. The small baskets were hung inside a larger basket 5 in. in diameter by 15 in. deep which was suspended in the



center of the NG/H<sub>2</sub>O solution at the beginning of the experiment. The solution was gently agitated by a 4-in. propeller agitator mounted on an air motor-driven "Lightnin" mixer during the experiment. Samples were removed after 6 min, 12 min, 24 min, 2 hours and 4 hours immersion time. The samples were dried externally, weighed precisely, dried overnight at 60°C, then further dried at 105°C ± 5°C to remove the last traces of moisture. The samples were then reweighed and submitted for extraction and analysis.

The experimental procedure was slightly modified as the experiments progressed. After the first experiment, the 4-hour sample was placed in the large basket rather than in one of the smaller baskets. The experiment at 0.16% NG was repeated with all material placed in the large basket, and individual samples were scooped out at the designated time. The results of this experiment indicated that there was a substantial improvement in pellet contact with the solution, resulting in increased NG pickup for the samples. The succeeding experiments at 0.2% NG and 0.24% NG were also conducted in this manner. Table XI shows the results of pellet analyses for these experiments. The analysis for methyl "Carbitol" (MeC) was indirect in that MeC was assumed to account for the remainder of the ingredients not accounted for by direct analysis.

## 2. Experimental Results

Figure 3 shows the % NG in the pellets at equilibrium as a function of the solution concentration. An examination of the rate of NG pickup from experiments at 0.16, 0.20, and 0.24% NG (see Figure 4) indicated that the value at 4 hours was approximately 98-99% of the ultimate equilibrium concentration except for the experiment in 0.24% NG solution where the shape of the curve of NG pickup versus time suggested that this value was probably only about 95% of the ultimate NG pickup. The value plotted on Figure 3 for 0.16% NG was the average of two experiments. The value plotted for 0.24% NG (actually 0.234%) was the 4-hour value extrapolated to equilibrium (assuming the 25.1% NG represented 95% of equilibrium). The curve should be a good representation of the actual equilibrium between these pellets and NG/H<sub>2</sub>O solutions. The line passes through the origin and would be expected to be asymptotic with the saturation concentration (0.246% NG) since the NG is miscible with NC.

Figure 4 depicts the NG and water content of the shock gel pellets as a function of contact time with the impregnating solution at 60°C.

The rate data from the experiments conducted at 0.16% NG, 0.20% NG, and 0.24% NG was utilized for the calculation of overall mass transfer coefficients based on the rates of equilibration. The NG concentrations used for the calculation were based on NC and NG only, i.e., the mole fraction NG based on NC plus NG rather than NC plus all components. Interfacial mass transfer areas were assumed to be perfect sphere surface areas for the

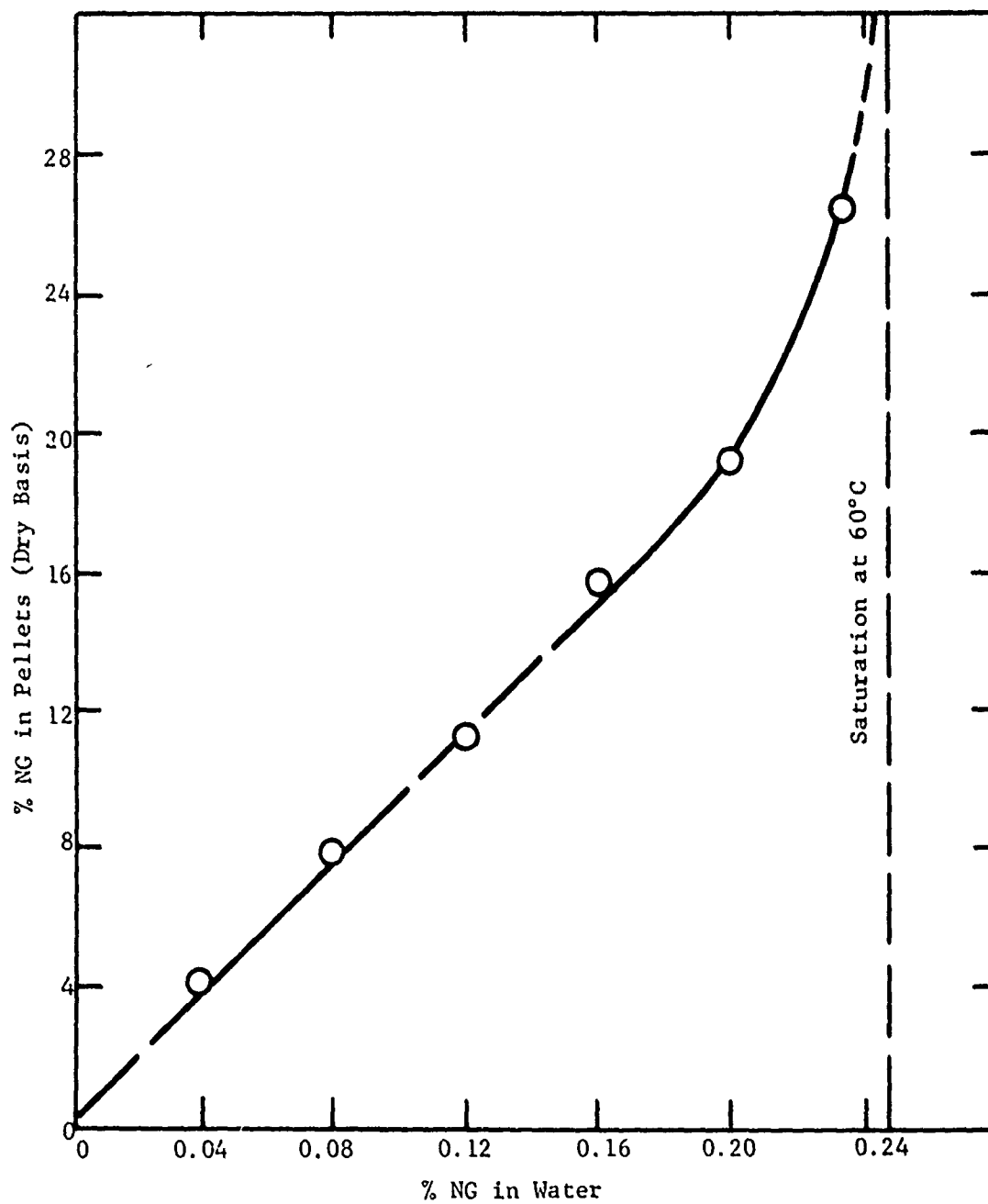


Figure 3. Percent NG in 13.4% N NC Shock Gels as a Function of NG Concentration in NG/H<sub>2</sub>O Solutions @ 60°C

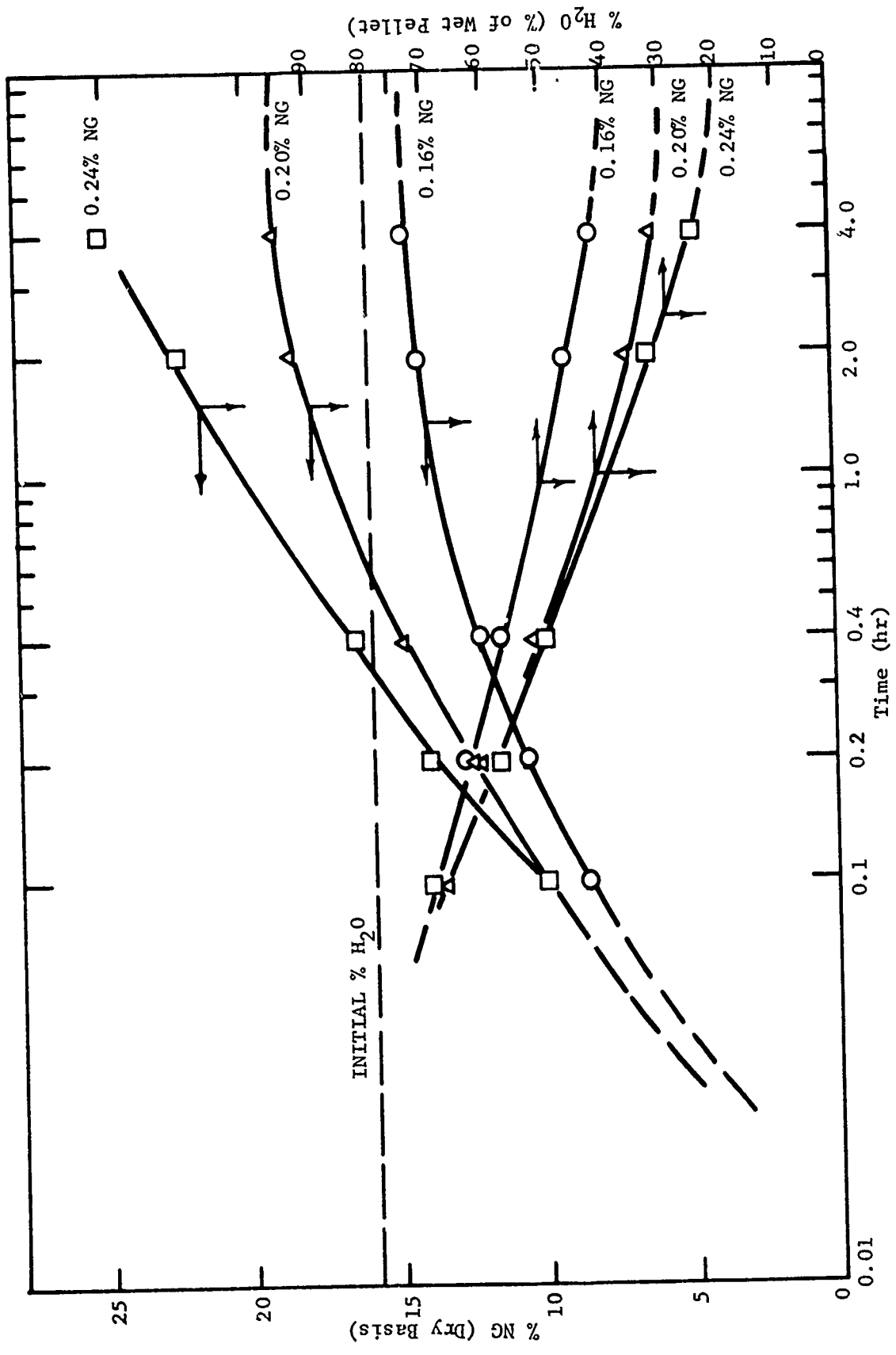


Figure 4. NG and Water Content as a Function of Contact Time

TABLE XI

ANALYSIS OF 13.4% N NC SHOCK GELS FROM NG IMPREGNATION EXPERIMENTS

% NG in Solution Beginning (End)	Impregnation Time				
	6 Min	12 Min	24 Min	2 Hr	4 Hr
% Water in Wet Pellets	78.90	77.9	76.8	72.9	70.3
% NC in Dry Pellets	98.50	97.75	97.57	95.43	94.42
% NG in Dry Pellets	0.58	1.27	1.42	3.52	4.09
% NDPA in Dry Pellets	0.50	0.52	0.56	0.59	0.83
% MeC in Dry Pellets <sup>(2)</sup>	0.42	0.46	0.45	0.46	0.66
<hr/>					
% Water in Wet Pellets	77.87	77.21	76.02	71.61	61.57
% NC in Dry Pellets	97.97	98.09	97.33	95.01	89.91
% NG in Dry Pellets	1.07	0.93	1.63	3.94	7.83
% NDPA in Dry Pellets	0.48	0.51	0.51	0.40	1.48
% MeC in Dry Pellets	0.48	0.47	0.53	0.65	0.78

$\frac{0.04}{(0.039)}$ <sup>(1)</sup>

$\frac{0.0806}{(0.0799)}$ <sup>(3)</sup>

NOTES: (1) All pellets in individual baskets.

(2) By difference.

(3) Four-hour sample in large basket in this and all succeeding experiments.

TABLE XI (CONT.)

		Impregnation Time				
		6 Min	12 Min	24 Min	2 Hr	4 Hr
<u>% NG in Solution</u> <u>Beginning (End)</u>	% Water in Wet Pellets	77.19	76.00	74.91	65.08	50.27
	% NC in Dry Pellets	97.48	97.36	96.60	90.48	86.02
	% NG in Dry Pellets	1.42	1.63	2.39	8.24	11.09
	% NDPA in Dry Pellets	0.53	0.53	0.51	0.66	1.75
	% MeC in Dry Pellets	0.57	0.48	0.50	0.62	1.14
<u>0.121</u> <u>(0.120)</u>	% Water in Wet Pellets	78.00	76.64	74.13	68.61	40.58
	% NC in Dry Pellets	97.55	97.03	95.19	91.64	80.28
	% NG in Dry Pellets	1.28	1.71	3.53	7.20	16.74
	% NDPA in Dry Pellets	0.52	0.52	0.54	0.52	2.06
	% MeC in Dry Pellets	0.65	0.74	0.74	0.64	0.92
<u>0.161</u> <u>(0.160)</u>	% Water in Wet Pellets	78.00	76.64	74.13	68.61	40.58
	% NC in Dry Pellets	97.55	97.03	95.19	91.64	80.28
	% NG in Dry Pellets	1.28	1.71	3.53	7.20	16.74
	% NDPA in Dry Pellets	0.52	0.52	0.54	0.52	2.06
	% MeC in Dry Pellets	0.65	0.74	0.74	0.64	0.92

TABLE XI (CONT.)

% NG in Solution Beginning (End)	Impregnation Time				
	6 Min	12 Min	24 Min	2 Hr	4 Hr
% Water in Wet Pellets	68.49	62.70	57.20	45.22	40.78
% NC in Dry Pellets	90.36	88.26	86.58	83.57	83.12
% NG in Dry Pellets	8.46	10.45	11.91	14.10	14.51
% NDPA in Dry Pellets	0.78	0.87	0.96	1.41	1.67
% MeC in Dry Pellets	0.40	0.42	0.55	0.92	0.70
<hr/>					
% Water in Wet Pellets	67.39	60.53	51.12	35.00	30.43
% NC in Dry Pellets	88.47	86.01	83.50	79.31	78.18
% NG in Dry Pellets	9.97	12.33	14.75	18.42	19.05
% NDPA in Dry Pellets	0.69	0.76	0.90	1.47	1.90
% MeC in Dry Pellets	0.87	0.90	0.85	0.80	0.87
<hr/>					
NOTE: (4) All samples in large basket in this and all succeeding experiments.					

$\frac{0.163(4)}{(0.160)}$

$\frac{0.202}{(0.198)}$

TABLE XI (CONT.)

% NC in Solution Beginning (End)	Impregnation Time				
	6 Min	12 Min	24 Min	2 Hr	4 Hr
% Water in Wet Pellets	68.04	57.46	49.18	31.33	23.04
% NC in Dry Pellets	88.98	84.96	82.19	75.20	71.39
% NG in Dry Pellets	9.95	13.85	16.33	22.37	25.10
% NDPA in Dry Pellets	0.53	0.65	0.75	1.51	2.56
% MeC in Dry Pellets	0.54	0.55	0.74	0.92	0.95
$\frac{0.239}{(0.234)}$					

median particle size whereas, in reality, the mass transfer area was composed of surface area plus internal pore area. The interfacial NG concentration was assumed to be the same as the average NG concentration at equilibrium (4 hours). The most important data required for the calculations are shown in Table XII along with the calculated mass transfer coefficients.

A constant mass transfer coefficient was not obtained because of the use of an artificial mass transfer area. A mass transfer coefficient based on the total mass transfer area should be essentially constant for constant conditions at the gel-liquid interface. It may be assumed that the measured mass transfer coefficients would be significantly higher under conditions of increased agitation which would prevent depletion of NG from the interfacial film.

#### C. 3-5 POUND PER HOUR SHOCK GEL DEMONSTRATION PLANT

To show the feasibility of the continuous operation of critical stages of a 3-5 lb/hr facility for the preparation of shock gel gun propellants containing 13.4% N nitrocellulose and suitable for the 5.56mm cartridge, such a facility was designed, constructed and operated.

##### 1. Design, Construction and Procurement

The demonstration plant was to include the following critical stages: syrup makeup, syrup transfer, syrup droplet formation, syrup gelling, gel leaching, and gel NG impregnation. Intermediate operations would also include transfer of the gels from stage to stage and removal of excess transporting fluid. A preliminary flow sheet prepared for use in economic analysis of the process was the starting point for designing the actual plant. The flow sheet is shown in Figure 5. Only the portions labeled "Gel and Leaching" and "NG Impregnating" were included in the demonstration plant.

##### a. Syrup-Makeup Equipment

The concept of operation for the demonstration plant envisioned syrup made in batches in two separate syrup makeup tanks as a 7% solution of 13.4% N nitrocellulose and pumped to the rest of the operation at a maximum rate of 5 lb/hr (dry NC basis).

At the maximum flow rate of 5 lb/hr, a volumetric flow of 0.152 gpm was necessary. Based on previous experience with 7% NC shock gel syrups, a total time period of 5 hours was thought necessary for complete solution makeup. Therefore a working volume of at least 45 gallons in each syrup tank was necessary.



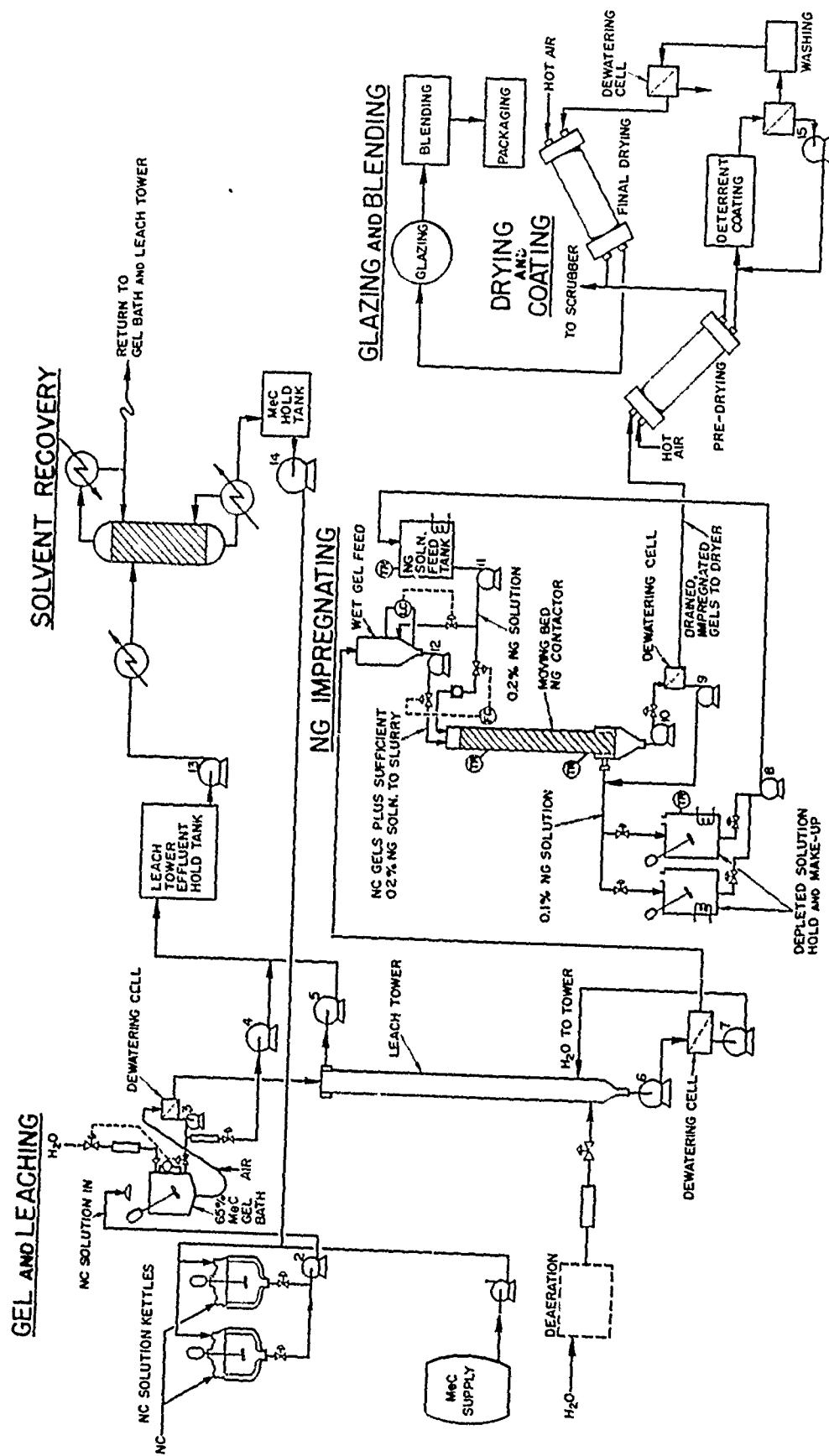


Figure 5. Preliminary Flow Sheet for Continuous Shock Gel Process

TABLE XII

DATA USED FOR CALCULATION OF MASS TRANSFER COEFFICIENTS

Contact Time (Hr)	0.16% NG					
	0	0.1	0.2	0.4	2.0	4.0
NG (mole fract.)	0	0.1050	0.1292	0.1470	0.1745	0.1795
Pellet Dia. (in.)	0.0430	0.0384	0.0354	0.0337	0.0308	0.0299
Pellet Density (gm/cc)	1.0858	1.1421	1.1715	1.2013	1.2708	1.2998
$Kx10^4$ $\frac{\text{No. Moles}}{\text{hr ft}^2 \text{ mole fraction}}$	-	3.15	1.96	1.28	0.83	0.82
				<u>0.20% NG</u>		
NG (mole fract.)	-	0.1238	0.1522	0.1812	0.2254	0.2339
Pellet Dia. (in.)	-	0.0372	0.0349	0.0323	0.0291	0.0284
Pellet Density (gm/cc)	-	1.1462	1.1822	1.2354	1.3378	1.3691
$Kx10^4$ $\frac{\text{No. Moles}}{\text{hr ft}^2 \text{ mole fraction}}$	-	2.77	1.61	1.45	0.99	1.06
				<u>0.24% NG</u>		
NG (mole fract.)	-	0.1229	0.1697	0.1994	0.2715	0.3059
Pellet Dia. (in.)	-	0.0375	0.0340	0.0320	0.0290	0.0280
Pellet Density (gm/cc)	-	1.1435	1.1988	1.2473	1.3621	1.4211
$Kx10^4$ $\frac{\text{No. Moles}}{\text{hr ft}^2 \text{ mole fraction}}$	-	1.88	1.65	0.88	0.68	1.27

Through competitive bids, two 60-gallon "Binks" pressure tanks manufactured by Binks Manufacturing Co. were purchased. These tanks were 110 psi rated ASME pressure vessels of galvanized steel construction with stainless steel fittings and a stainless steel liner. The tanks were locally modified to make them suitable for the intended use. The modifications to each vessel consisted of the following:

- (1) Installation of a hot water jacket.
- (2) Covering with 1 in. of "Ethafoam" insulation.
- (3) Installation of a bottom thermocouple.
- (4) Shortening the agitator shaft so that the agitator blade was 9 in. from bottom.
- (5) Attachment of a 6-in.-diameter "Cowles Dissolver" blade to the shaft and silver soldering in place.
- (6) Modification of the stuffing box to accept two Torrington needle thrust bearings and races; repacking with Chesterton Style 322 TFE-impregnated asbestos packing; and replacement of top support bushing with a precision ball bearing.
- (7) Attachment of a motor bracket to the stuffing box and a connection of a 2 horsepower air motor (Gast 6 AM) to the shaft via a cushioned drive coupling.

Previously, a 10-gallon vessel of similar height-to-diameter ratio fitted with a 1/2-horsepower drive unit and a 4-in. "Cowles Dissolver" blade had been used to make solutions of the type planned for the demonstration plant in a cycle time of ~ 4 hours at agitator speeds of ~ 1000 rpm. Based on geometric similarity of the two vessels and the plan to use agitator speeds of 2000 rpm for the larger vessel, the 6-in. agitator diameter and 2-horsepower motor were estimated to be correct for the 60-gallon vessels.

Figures 6 and 7 show the shock gel solution tanks.

b. Syrup Transfer

Although the syrup vessels were capable of withstanding the pressures which might be necessary to pressurize the syrup to the streaming

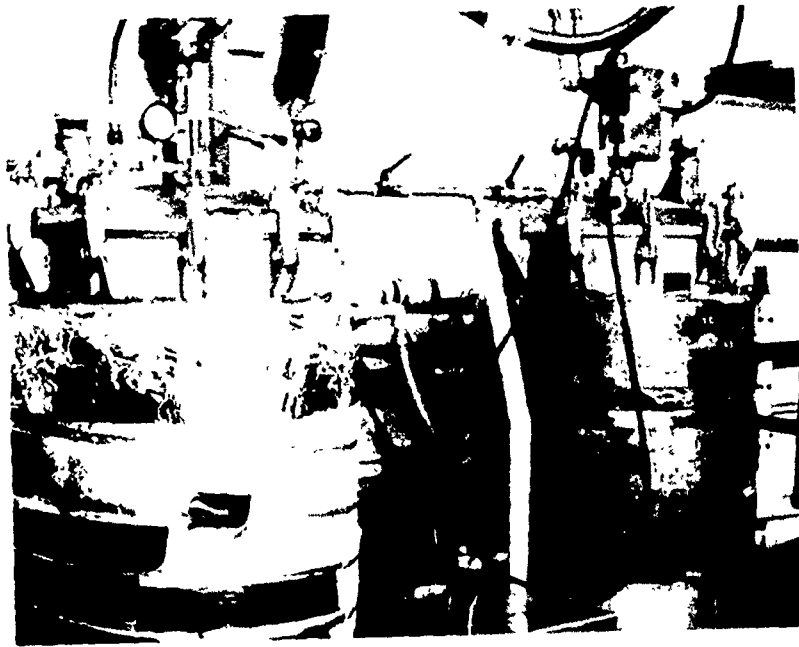


Figure 6. Shock Gel Solution Tanks

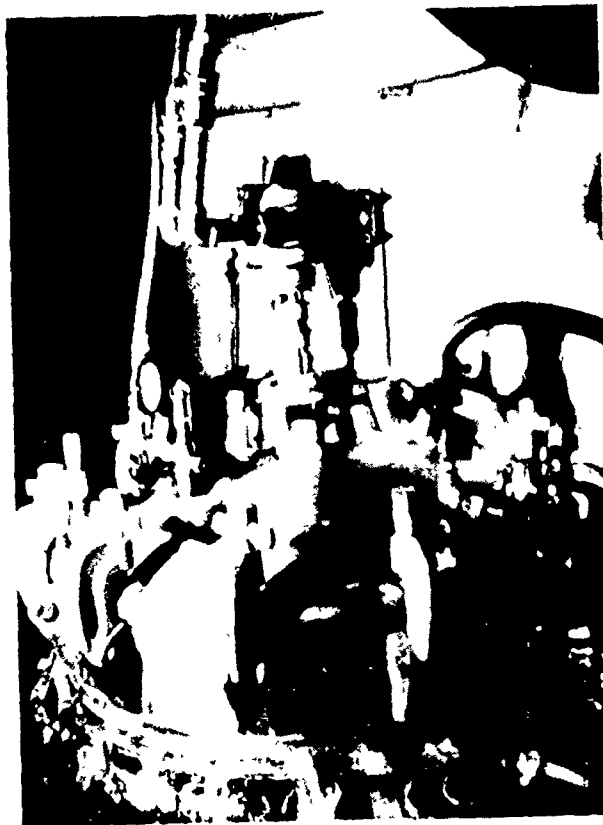


Figure 7. Detail of Tank No. 1

G-3584

head, this method did not seem to be capable of the desired metering accuracy without expensive flow controls.

Based on experience with precision metering of thick slurries of cellulose and nitric acid on Contract DAAA25-73-C-0711, in which a Zenith gear pump had proved eminently satisfactory, a similar type pump of larger capacity was chosen.

The pump used was a Zenith, three-gear, staple fiber pump, type BMC-5334-11, with cast iron body, stainless steel gears, and Teflon lip seal. The pump delivered a volume of 11.1 cc per revolution, which would keep operating speeds within the recommended 25-75 rpm range. The pump was trunnion-mounted and equipped for inlet and discharge lines of 1/2-in.-O.D. tubing.

The pump was driven by a Gast 4 AM air motor operating through a 30:1 gear reducer and a 72-tooth spur gear which meshed with the drive gear of the pump. Figure 8 shows the syrup metering pump. Figure 9 shows the control panel for solution tanks and metering pump.

The syrup was metered to an in-line filter for removal of undissolved material which might tend to plug the streaming orifices. The filter was chosen in collaboration with the manufacturer, Mott Metallurgical Corp., and consisted of a bundle of sintered stainless steel tubes with one closed end and with one open end welded to a manifold. The tubes were 18 in. long and had a 40-micron filtration rating. Total external area of the tube bundle was 2 ft<sup>2</sup>. The filter area was chosen to give a pressure drop, when clean, of < 10 psi at the maximum anticipated flow rate of 5 lb/hr of NC. The unknown factor in the filter choice was the running time which would be available before pressure drop became excessive.

The filter was housed in an ABL-fabricated housing made of stainless steel and sealed with an O-ring and flange to which the filter itself was silver-soldered. Figure 10 shows a filter and housing.

Connections from the syrup tanks to the pump and from the pump to the filter housing were made with 1/2-in.-O.D. stainless steel tubing and "Swagelok" connectors.

From the filter to the streaming head, a carbon steel 1-in. Schedule 40 pipe with welded fittings was used. The piping was hot-water traced and insulated, and the filter housing and tubing were wrapped with a resistance-type heating tape and insulated.

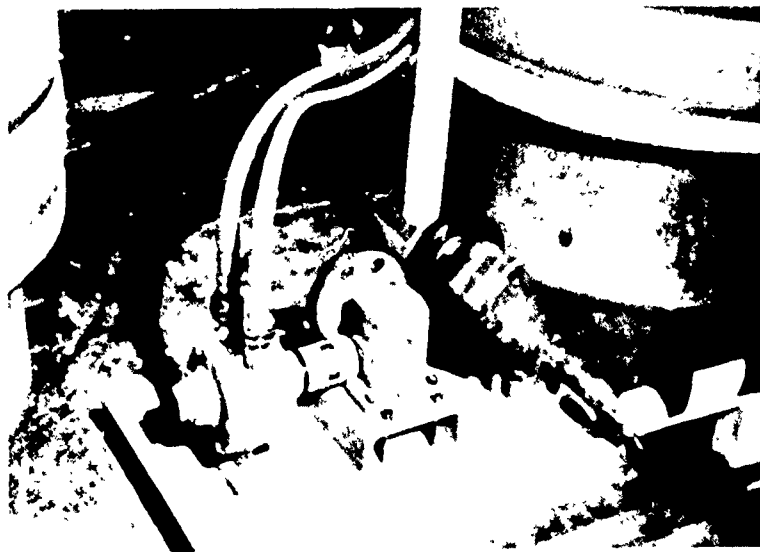


Figure 8. Shock Gel Syrup Metering Pump

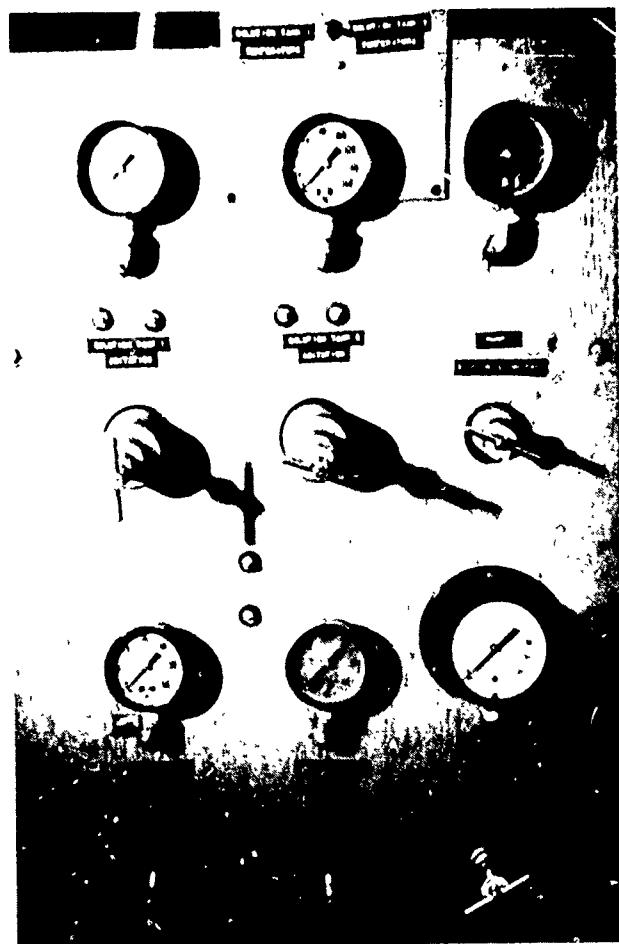


Figure 9. Control Panel for Solution Tanks and Metering Pump

G-3585

### c. Syrup Droplet Formation

The shock gel syrup was pumped to a streaming head for droplet formation. The streaming head was machined from a solid piece of 2024-T4 aluminum and consisted of head and orifice plate retaining cap with an integral lug for attachment of an air-operated vibrator.

Syrup entered the head through a 1-in. NPT aluminum pipe nipple screwed into the head and connected to the main syrup piping system by a flexible hose.

The streaming plate itself was made from 1/16-in.-aluminum sheet and was 5.4 in. in diameter with 161 holes 0.0135 in. in diameter. The holes were on 1/4-in. centers and were made by first dimpling the plate with a punch to give a depression 1/16-in. deep and then drilling through the center of the depression with a No. 80 drill. The plate was sealed by neoprene gaskets on either side.

The number of holes was chosen on the basis of the flow rate performance of the 85-hole streaming plate previously used for batch studies.

The streaming head was clamped to a NAVCO 1-in. matchplate-mounted, silent-type (air cushioned impact), air-operated vibrator with a typical vibrational frequency of ~ 200 cps. Figure 11 shows the streaming head mounted in normal operating position without N<sub>2</sub> sparge lines attached (heating tape not in place).

### d. Syrup Gelling

The 65% methyl Carbitol/water solution used for gelling the syrup droplets was contained in an open-top, jacketed, insulated, stainless steel, cone-bottomed tank. The tank had a bottom outlet of 1-in. pipe, a screened overflow to maintain a constant level with 70.3 gallons of liquid in the tank and was agitated with a NAR-33, air-operated, "Lightnin" mixer. The mixer was equipped with a 3.6-in.-diameter propeller agitator blade.

Gel and liquid take-off from the tank was via the bottom outlet through a 3/4-in.-I.D. hose leading to the No. 1 dewatering screen. The driving force for gel and liquid removal was air injected into the hose through a 6-in.-long, 16-gauge hypodermic needle.



Figure 10. Shock Gel Syrup Filter and Housing



Figure 11. Shock Gel Streaming Head

G-3586



Concentration of the gel liquid was maintained at 65% MeC by deionized water metered into the gel tank through a rotameter (Matheson Co., Model 604).

e. Gel Leaching

Gel leaching with water was performed in a one-step, counter-current operation. Although the gelling step was itself a partial leaching step (to 65% MeC), additional leaching was required to remove the MeC solvent completely and thus keep solvent losses to a minimum.

The leaching system was planned to keep solvent losses to less than 1% of powder weight which suggested that the typical leached gel (~ 90% liquid) should contain no more than 0.1% MeC.

The simplest design was thought to be appropriate for the early stages of continuous process work. The design chosen for evaluation was a simple vertical column without internal baffles or agitation. The gels were to be fed into the open top where they would settle under the influence of gravity and move downward, countercurrent to leach water introduced at the bottom of the column.

Based on previously measured hydraulic properties of shock gel pellets produced from a 7% syrup, which showed good agreement with the theoretical behavior of pellets with similar properties, the leach column was sized based on theoretical hydraulic properties for the following conditions:

- (1) Pellet diameter of 0.050 in.
- (2) 12% NC in leached particles
- (3) 4 lb/hr (dry NC basis) production rate
- (4) A 50% MeC effluent and a 25% MeC average concentration throughout the column
- (5) Ambient temperature (20°C) operation
- (6) Leaching height of 10 ft
- (7) Average residence time of two hours

The residence time of two hours was selected based on diffusion rates of methyl Carbitol during leaching which were measured during the previous shock gel investigation.

The free settling velocity of the specified particle was calculated to be 0.063 ft/sec. Since a residence time of 2 hours required a net downward velocity of 0.00139 ft/sec, it was apparent that the column would have to be operated under conditions of hindered settling wherein the hindered settling velocity minus the upward liquid velocity was equal to the desired net downward velocity.

For a 50% MeC effluent (assuming the gels enter the column with 15% free liquid), a liquid upflow of  $1.83 \times 10^{-4}$  ft<sup>3</sup>/sec was required. By use of equations relating the net downward velocity to the volume fraction liquid in the column, the volume fraction liquid was calculated to be 0.548, from which a column diameter of 6.4 in. was calculated.

A standard size (6.375 in. I.D. by 0.125 in. wall) of 6063-T6 aluminum tubing was used for the main tube. A 30-degree discharge cone (included angle) with 1-in. tubing outlet was welded to the bottom. Leach water was introduced from eight equally spaced hose fittings 3 in. above the bottom cone. At the top a 360-degree screened overflow weir (40 x 40 mesh SS) was provided. A 1-in. tube welded to the overflow carried the effluent to a cone bottom settling tank where NC fines could settle out. From there the overflow went to a rotameter (Brooks type size R-6-15-B with SS float) for measurement of effluent rate.

The leach water to the column was first passed through a Culligan Mod. D-25P deionizer and then metered to the column through a rotameter (Brooks tube size R-6-15-B with Carbaloy float). The water went to four low-flow rotameters (Brooks-Mite 0-10 gph) where it was distributed into four equal streams which were each divided into two streams going to the leach column at points 180 degrees apart.

The gels were fed to the column from a dewatering screen (Screen No. 1) which dewatered the gels as they were removed from the gel tank. The dewatering screen was fabricated from 1/16-in. SS sheet. The screened area was 2.5 ft long by 6 in. wide (40 x 40 mesh SS with 0.013 in. wire). The screen movement was stabilized by three 1 in. wide x 1/16 in. thick strips welded to the screen and box. The discharge aperture tapered to an opening 2 in. wide, and the sides of the box were 6 in. high. A vibrator (Vibrolator Mod. CD-35) was mounted on the rear wall of the box so that the vibration was in a plane perpendicular to the screen. The vibrator could be moved up and down ~ 1 in. above or below the center of gravity of the screen.

The screen was suspended in a frame with chains or cables adjustable for length so that the angle of the screen could be varied.

A full-length catch pan was situated under the screen to catch the gel bath liquid. A Jabsco, flexible impeller, 0-5 gpm pump driven by a Gast 4 AM air motor pumped gel bath fluid from the catch pan back to the gel tank. Figure 12 shows the No. 1 dewatering screen.

Leached gels, with accompanying water, were pumped from the bottom of the leach tower by a Moyno progressing cavity pump, Mod. 1L2CDB. The pump had a hard-chromed rotor rotating in a butyl rubber stator. The choice of butyl rubber for the stator material was based on immersion tests with samples of stator material in MeC at room temperature. The results of these tests are shown in Table XIII.

TABLE XIII  
IMMERSION TESTS IN METHYL CARBITOL AT ROOM TEMPERATURE

Length of Immersion (Days)	Buna-N Percent Change		Natural Rubber Percent Change		Butyl Rubber Percent Change		Viton A Percent Change	
	Volume	Weight	Volume	Weight	Volume	Weight	Volume	Weight
1	15.4	20.3	-2.0	0.3	-0.6	0.4	15.6	17.3
2	18.4	27.6	-9.3	0.2	-0.6	0.4	25.3	24.5
6	30.9	42.3	-5.4	0.04	-0.6	0.5	49.5	39.8
21	54.3	46.4	-5.9	-0.75	-0.6	0.9	59.5	49.9

The Moyno pump had an output of 0.0026 gallon per revolution and was driven by an Ingersoll-Rand variable-speed air motor through an integral 20:1 gear reducer.

The gels were discharged into a line transferring them to the upper level, to Screen No. 2, which fed the NG-impregnation operation. Several line arrangements were tried. The first consisted of a section of 2 in. I.D. rubber hose slipped over the pump stator housing with a 1½-in. x ½-in. reducer about 6 in. downstream. A hose adaptor for 5/8-in. hose led from the adapter to the second level to Screen No. 2. Water was injected into the discharge via three 20-gauge hypodermic needles to raise the liquid velocity in the hose sufficiently to carry the gels upward. Without liquid injection, the gels tended to bridge into a hard packed mass which blocked further flow.

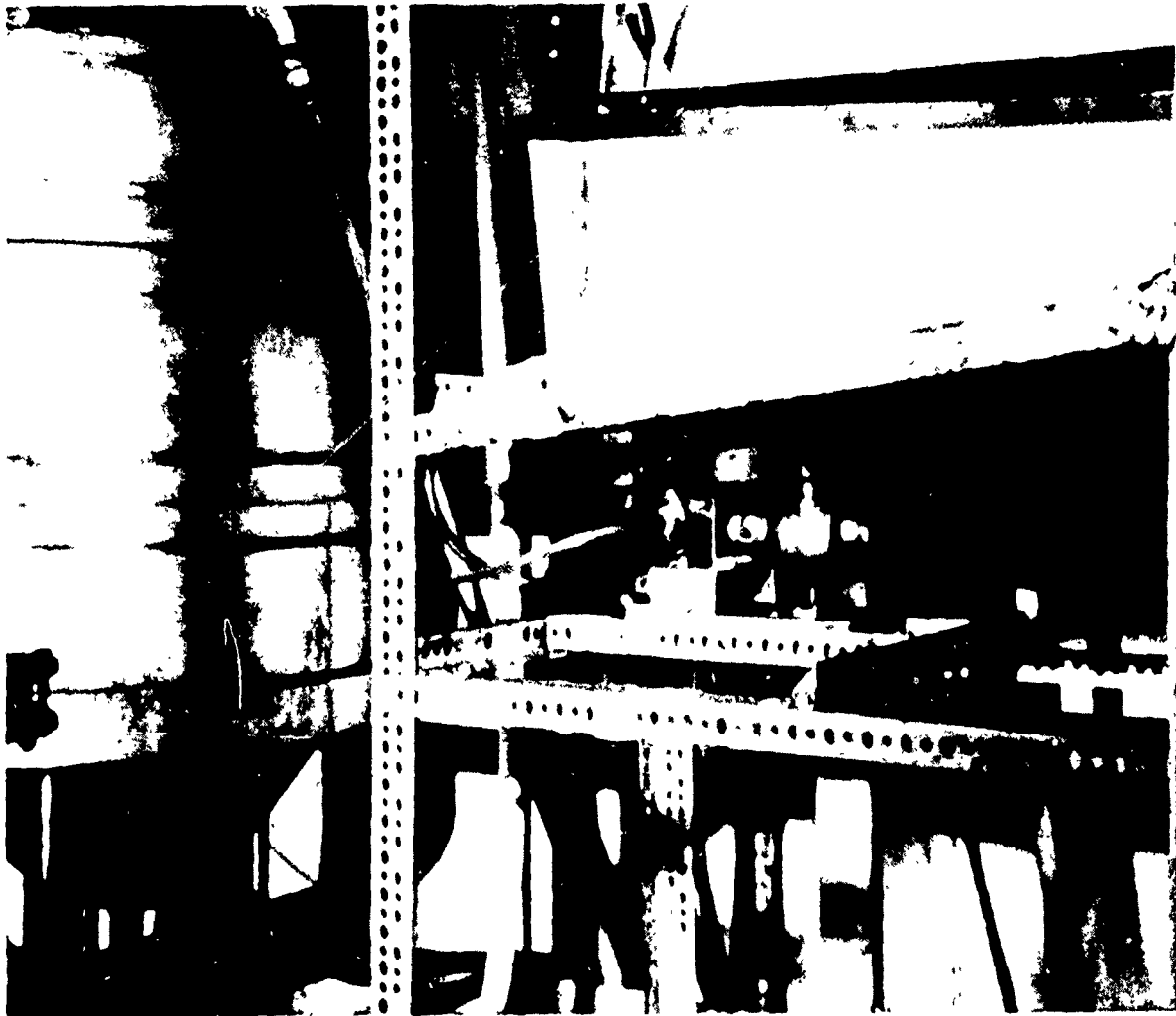


Figure 12. Dewatering Screen No. 1  
(with gel tank on left)

G-3587

f. Gel NG Impregnation

For the NG-impregnation step, both a countercurrent and a concurrent operating mode were considered. The countercurrent equipment was envisioned to be in much the same configuration as the leach column discussed in section e.

For an initial design of a countercurrent NG-impregnator, a number of assumptions were made. These assumptions were as follows:

Top

Shock Gels In: 4.65 lb NC per hour  
18.6 lb water per hour  
Average Diameter = 0.045 in.

NG/Water Solution Out: 230.4 lb per hour  
0.04% NG  
Temperature - 60°C.

Bottom

Shock Gels Out: 4.65 lb NC per hour  
0.35 lb. NG per hour  
0.29 lb water per hour  
Average Diameter = 0.0375 in.

NG/Water Solution In: 221.1 lb per hour  
0.2% NG  
Temperature - 60°C.

Height of Tower - 15 ft

Required Residence Time - 2 hours

Gels are perfect spheres of uniform size

Hydraulic properties of NG/water solution are the same as water at 60°C.

On this basis, the required tower diameter could be calculated to yield a hindered settling velocity for the pellets that would achieve the required residence time. The calculations, made for an "average" particle, showed that the correct diameter would be 3.9 in. A check of conditions at the top of the tower however indicated that at this diameter the entering pellets would not settle but would be swept out of the tower. This prediction was based on the difference between pellet and liquid density; this difference is approximately doubled from top to bottom because of shrinkage. At the same time the volume fraction of pellets decreases for the same reason, and thus further hinders the settling velocity. These conditions are illustrated in Table XIV.

TABLE XIV

CONDITIONS IN COUNTERCURRENT NG-IMPREGNATOR  
DESIGNED FOR AVERAGE PELLET\*

	Top	Bottom
Density of Gel (lb/ft <sup>3</sup> )	66.85	71.56
Density of NG/Water (lb/ft <sup>3</sup> )	61.38	61.38
Density Difference (lb/ft <sup>3</sup> )	5.47	10.18
Gel Diameter (ft)	$3.75 \times 10^{-3}$	$3.13 \times 10^{-3}$
Fractional Volume Occupied by Gels	0.572	0.328
Free Settling Velocity** (ft/sec)	0.436	0.564
Hindered Settling Velocity (ft/sec)	0.0074	0.064
Velocity of Liquid (ft/sec)	-0.029	-0.019
Net Downward Velocity (ft/sec)	-0.0216	0.045

\*Diameter of tower is 3.9 in.

\*\*Flow in the downward direction is considered positive.

A calculation of the required tower diameter at top and bottom using local conditions gave 4.34 in. and 3.38 in., respectively. Ideally, a countercurrent NG-impregnator would be tapered from top to bottom in such a manner as to lead to the correct average particle velocity throughout. As a practical matter such a tower would probably be built with an enlarged top section connected to a smaller diameter bottom section.

The problem described above was not encountered in the similarly designed leach column because large volume shrinkages did not occur.

The assumed residence time requirement of 2 hours was open to question but was probably a safe estimate.

In view of the high solubility of NG in NC and the low solubility of NG in water, it may be assumed that the primary resistance to NG diffusion resides in the liquid phase. Thus the flow conditions of the liquid phase should be quite important in determining the rate of mass transfer.

As far as the relative merits of a cocurrent versus a counter-current NG-impregnator are concerned, the following considerations were applicable.

Overall mass transfer coefficients for both are undefined, and the advantages of the higher average concentration difference in counter-current flow may be offset by the fact that the cocurrent mode operates at a higher Reynolds number which assists mass transfer.

Overall liquid flow rates are lower in countercurrent operation and thus reduce the cost of piping, pumping, storage, etc.

The cocurrent operating mode can be operated satisfactorily over a wider range of flow rates since the residence time is fixed by the rate at which gels are removed by the gel removal pump, and the solution flow can be varied independently of the gel flow rate without affecting residence time. In the countercurrent option, gel flow rate, liquid flow rate, and residence time are interrelated in such a manner that operation of a properly designed tower at other than the design rate would significantly affect residence time.

In terms of overall operating characteristics, the cocurrent mode of operation, although perhaps not as elegant in concept as a counter-current operation, seemed to offer the flexibility in operation which was necessary at the time.

The design of the equipment for the cocurrent operating mode was considerably simpler. Based on an average gel volume of  $0.274 \text{ ft}^3/\text{hr}$ , and thus a total gel volume of  $0.548 \text{ ft}^3$  for a 2-hour residence time, the required column volume could be calculated if a gel packing density was assumed. Assuming a gel volume fraction of 0.63, a total required volume of  $0.870 \text{ ft}^3$  was calculated.

Many combinations of height and diameter could be chosen. A 4 in. I.D. by 10 ft long column gave a volume of  $0.873 \text{ ft}^3$ . The high L/D ratio was attractive for reducing back-mixing of pellets, and the dimensions were a standard size for Pyrex pipe. The smaller diameter pipe also would give higher liquid phase turbulence which was thought to be beneficial for good mass transfer.

Based on the anticipated average liquid flow rate of 300 lb/hr of  $60^\circ\text{C}$ . NG solution, a Reynolds number of 500 was calculated for the flowing liquid. Further calculation showed that the bed pressure drop should be 1-2 psi which could be tolerated.

As finalized, the NG-impregnation column consisted of a single section of Pyrex pipe 10 ft x 4 in. I.D. At the top was a 1 in. x 4 in. Pyrex reducer through which the gels and liquid were introduced from the feed pump. At the bottom was an ABL-fabricated adapter which mated with the Pyrex pipe. The adapter was fabricated from aluminum sheet and had an enlarged annular area separated from the gels by a close-fitting circular screen (40 x 40 mesh) and connected by hose to a tank into which depleted NG solution could flow after passing through the gel bed.

Below the annular liquid-takeoff area, the adapter converged into a discharge cone with a 30-degree included angle and terminated in a 1-in.-diameter outlet tube. The tube was connected by rubber hose to the inlet of a Moyno pump identical to the one used on the leach column discharge except that it was driven by a Gast 4 AM, variable-speed air motor driving through a 10:1 gear reducer.

The screen (screen No. 2) on the upper level which received the gels discharged from the leach column was designed similar to screen No. 1 but was 6 in. shorter because of the lower expected flow rate. The vibrator on screen No. 2 was also a Vibrolator, Mod. C-35, and vibrated in a plane vertical to the screen. The vibrator was mounted above the screen box with its center  $2\frac{1}{2}$  in. above the top of the box and 1 in. forward of the rear wall of the box. The catch pan below the box drained by gravity into the building drain or a catch tank.

The screen discharged gels into a SS funnel 10 in. high x 6 in. I.D., flange-mounted to the inlet of a Moyno pump beneath the screen catch pan (see Figure 13).

Entering the inlet funnel with the gels was a 0.2% solution of nitroglycerin in water at 60°C. metered in by a rotameter (Fisher & Porter Co. Precision Bore Flowrator Tube No. 38-25) from a 2-gallon constant head tank mounted above screen No. 2. The Moyno pump pumped the slurry of gels and NG solution through a  $1\frac{1}{2}$ -in.-I.D. flexible PVC tube into the top of the impregnation column. Figure 14 shows an overhead view of screen, head tank, pump, and column.

The gels and liquid pumped from the bottom of the column were pumped through a  $1\frac{1}{2}$ -in.-I.D. flexible PVC hose to screen No. 3. This screen was identical to screen No. 2. The catch pan for this screen was connected to the inlet side of a 1 gpm peristaltic tube pump (Little Giant, Mod. LG-100) which pumped excess depleted NG solution from the catch pan to the depleted NG solution tanks.



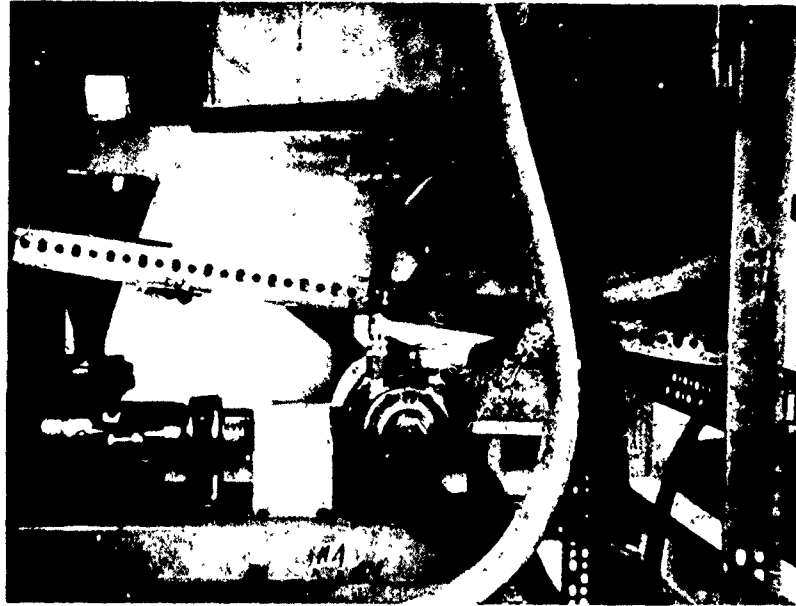


Figure 13. Screen No. 1 and Impregnation Column Feed Pump



Figure 14. Overhead View of Screen No. 2, Head Tank, Feed Pump and NG Impregnation Column

G-3588

The NG solution tanks consisted of one 300-gallon tank for containment of 0.2% NG solution and two 150-gallon tanks for containing depleted NG solution from the impregnation column and from the final screen. The tanks were sized on the basis of the projected column flow rates and the estimated time required to measure the depleted solution concentration, reconstitute the solution to 0.2% NG, and pump it back to the 300-gallon tank without interruption in NG solution feed. The analysis and reconstitution were estimated to require 4 hours which, at a 300 lb/hr liquid rate, was equivalent to 146 gallons.

The tanks were made by United Utensils Co. and were fabricated from 16-gauge stainless steel (SS). The tanks had bottoms which sloped toward the bottom outlet. The outlets were modified by insertion of a "goose neck" so that actual solution pickup was from a point 1 in. above the bottom.

Tank height to diameter ratio was 1:1. Each tank was equipped with a loose-fitting lid which had cutouts for hoses and agitator. The tanks were fitted with hot water jackets at ABL. The jackets on the 150-gallon tank were 18 in. high; that on the 300-gallon tank was 22 in. high. The circumference, lid, and bottom of each tank were insulated with 1 in. of "Ethafoam."

Each tank was equipped with an air-driven "Lightnin" mixer, Mod. NAR-33, having a 3.6-in.-diameter three-blade propeller. The propellers were silver soldered to the mixer shaft.

To pump reconstituted solution from the 150-gallon tanks to the 300-gallon tank, a 20-gpm Vanton "Flex-i-liner" pump, Mod. XB-T-120, was used. The pump utilized a Teflon body block and a butyl rubber Flex-i-liner.

Concentrated NG solution was pumped from the 300-gallon tank up to the constant head tank on the upper level by a 0-4 gpm peristaltic tubing pump using "Tygon" tubing.

## 2. Single-Base Operations

Five continuous single-base-only runs of various durations were made to determine the most effective running modes and to assess equipment operation.

a. Run X170-33

This was the first live run in the newly installed equipment for the demonstration plant. Of critical interest on this run was the ability of the NC syrup tanks to make up a 7% NC syrup in what was expected to be the minimum available time of ~ 5 hours. This was the pumping time for 50 gallons of syrup at the 5 lb/hr rate (4+ gallons remained below the dip tube when the tank was empty).

The first solution was made with a removable baffle attached to the dip tube, and it was found that the presence of the baffle prevented formation of a complete solution. This solution was scrapped and the baffle was removed.

Three hundred and forty pounds of solution (excluding weight of moisture), ~ 40 gallons, were made up on the next attempt. Then 316.2 lb of methyl Carbitol (MeC) was added to the tank and 221 gm of 2-NDPA were dissolved in the MeC while heating to 63°C. While the solution was agitated at ~ 1200 rpm, 32.51 lb of 26.8% water-wet NC were added to the MeC.

When all the nitrocellulose was in, the tank was sealed and high-speed agitation (~ 2000 rpm) under vacuum was begun. A vacuum of ~ 740 mm Hg was maintained. The vacuum line led to a trap cooled with dry ice so that collected liquid could be analyzed and MeC added back to the solution.

After three hours of vacuum agitation, the agitator was slowed down and stopped by overheating of the lower bearing assembly. Disassembly of the agitator assembly revealed that the additional downward force on the agitator shaft due to vacuum combined with the high speeds had caused the thrust collar on the shaft to severely gall and overheat the self-lubricated bronze bushing which served to provide radial alignment and vertical thrust capacity for the lower portion of the bearing assembly.

Inspection of the solution upon removal of the lid for agitator disassembly revealed essentially complete solution except three small, partially solvated lumps ~ 1 in. in diameter.

The apparent viscosity of the solution was measured at 57°C using a Brookfield viscometer. An apparent viscosity of 3450 centipoises was recorded. This indicated that such a solution would be streamable at a temperature of 65°-70°C after a makeup time that was not excessive for the mode of operation planned.

Repairs to the agitator of the NC solution tank were made, and the assembly was checked out by continued agitation at speeds of up to

2000 rpm. No overheating occurred. The solution was streamed after a total of 13 hours of vacuum agitation at temperatures up to 70°C. The viscosity of the solution shortly before streaming was 2350 cps at 62°C (apparent Brookfield viscosity) and 1730 cps at 69.5°C. The actual solution temperature during streaming was 66°C.

Due to moisture removal during vacuum agitation, the solution moisture was 1.76% and actual NC concentration was 6.92%.

For start-up the gel tank was filled with 547 lb of 60.7% MeC/H<sub>2</sub>O solution calculated to give 65.0% MeC after one hour of streaming.

The solution tank was pressurized to 40 psi to force solution to the gear pump inlet and the gear pump was set at 52 rpm (577 cc/min). Twelve minutes were required to fill the lines and initiate streaming. As soon as stream breakup and spherification were confirmed by strobe light, the streams were allowed to fall into the gel tank. The vibrating streaming head was located 72 in. above the gel bath.

After one hour of streaming, the constant level overflow on the gel tank began passing gel fluid whose concentration was found to be 65.0% MeC, as calculated. Gel bath take-off was then begun, by air lift, at a rate calculated to empty the gel tank every hour (70.3 gph). The gel bath discharge was directed to the dewatering screen above the leach tower and all of the liquid passing through the screen was pumped back to the gel bath. Water was metered into the gel bath at a rate calculated to maintain a 65.0% MeC concentration in the gel bath. Because of an error in the reading of a rotameter calibration chart, ~ 75 ml/min in excess of that actually required was added, resulting in a slowly dropping gel bath MeC concentration.

The water flow to the leach tower was set by four rotameters which distributed the entering water around the bottom and by a single rotameter which metered the overflow discharge. Since the overflow discharge was siphoning, it could not be accurately measured. Thirty minutes after gels began entering the leach tower, which had been previously filled with water, take-off of leached gels was begun at a pump speed calculated to maintain a constant bed level of leached gels in the bottom of the tower.

The leached gels were pumped out of the tower and up a 5/8-in. I.D. flexible hose to the dewatering screen on the upper level. Movement of the gels was assisted by water injected into the outlet throat of the Moyno pump. Gels were then recycled to an intermediate hold tank from the dewatering screen. The gels initially recovered from the leach tower contained 0.3% MeC after an undetermined leaching period.

Some of the streaming head orifices were observed to be plugged shortly after streaming was initiated, and more became plugged as the run progressed. Since the syrup flow rate was constant, the jet velocity from the streaming head orifices increased to the point where incompletely spherified droplets were gelled. In some cases these were actually strings several inches long. The leach tower eventually became plugged with an interlocking mat of these particles and operations had to be halted. At this point (after 3½ hours of streaming), approximately 75% of the streaming head orifices were plugged. Upon disassembly of the head, it was found that the syrup had carried weld scale and other contamination from inside the pipe to the streaming head and that this had caused the streaming plate orifices to be plugged.

At cessation of streaming, the system  $\Delta P$  measured at the filter inlet was 15 psig, created mostly by the plugged orifices.

The gel bath concentration had dropped from 65% MeC to 62.8% MeC because of the incorrect rotameter setting of the fresh water feed to the gel bath. The combined gel bath and leach tower effluents consisted of ~ 230 lb of liquid which was 40% MeC.

General operation of the system was encouraging but some modifications were needed. The leach tower was to be modified by the installation of Plexiglas windows top and bottom. This would allow direct observation of the gel bed in the column. By controlling the Moyno to keep this bed height constant, better control could be effected. The leach tower overflow was modified to eliminate siphoning and to permit accurate metering of the overflow. The water injection system was fitted with a rotameter for closer control. A precision rotameter was also installed to monitor the total flow to the four leach water distribution rotameters. The dewatering screen for the gel bath discharge was modified to relocate the vibrator from above the screen to the rear wall, for better action.

The actual NC feed rate to the streaming head during this run was 5.4 lb/hr (dry basis).

b. Run X170-37

Shock gel run X170-37 was designed for a feed rate of 3.8 lb/hr NC (dry basis). Solution tank No. 1 held 50 gallons of 7% NC syrup, and tank No. 2 held 45 gallons of 7% NC syrup. At a 3.8 lb/hr feed rate, each tank was good for 6+ hours of running; thus solutions could be made for continuous feed at this rate.

The solution was streamed at a syrup temperature of 74°C. After stream breakup and sphere formation were confirmed by stroboscopic observation, the droplets were allowed to enter the gel bath. After gelling had been in progress for 1 hr and 19 min, takeoff by air lift to the dewatering screen was begun at a rate calculated to empty the gel tank (70.3 gallons) every 1 hr and 19 min.

After the flow to the dewatering screen had been set, the dewatering screen gel discharge was allowed to enter the leach column. The rate of leach water upflow had been previously set at 310 ml/min based on desired steady-state conditions. Upon buildup of a visible static bed in the bottom of the column, the discharge pump was started and the static bed height was maintained constant. The leach column was operated in this mode for 2 hours without any apparent increase in gel residence time. Leach tower overflow MeC concentration was fairly constant at 32-34% MeC, but the leached gels contained 9-26% MeC, an excessive amount.

The leach liquid upflow was slowly increased to 670 ml/min over the next 1-1/2 hours in an attempt to increase the gel residence time. After a total of 5 hours streaming and 3-1/2 hours of leaching, a buildup of gels in the upper portion of the column caused operations to be halted. The cause was traced to the presence of occluded air in the later gels which so reduced their density that they could not sink in the column. The presence of air in the gels was the result of an inadequate period of solution evacuation (30 min) before streaming which left a layer of aerated syrup in the upper volume of solution. When the level in the solution tank decreased sufficiently to allow this syrup volume to be drawn from the dip tube, aerated gels resulted.

Operation and control of equipment during this run were satisfactory except for the operation of the leach tower. Operation of the leach tower seemed to be especially sensitive to start-up procedure and the means by which the required gel hold-up was established.

c. Run X170-40

Two new 7% NC syrup batches were made for X170-40 in the same manner as for X170-37. The first tank had an apparent viscosity of 1790 cps at 79°C, and the second tank at apparent viscosity of 2650 cps at 66°C.

For run X170-40 a syrup feed rate corresponding to 3.8 lb/hr of NC was used with a bulk syrup temperature of 74°C. The syrup was streamed into the 65% MeC gel bath for 2 hours before the gels were removed. The leach column was initially empty above the leach water feed point. After the dewatering screen had established a steady-state gel bed, a slurry of gels and 15% MeC at the desired steady-state gel-volume density (0.4) was quickly poured into

the leach column to the point where overflow began. The dewatering screen was then allowed to feed gels to the column. The gels would not sink and gels from the dewatering screen simply piled up. Even when the upflow of leach water was halted, the gels stayed in place as if bridged. The run was halted, and the gels were removed.

The run was restarted the following day by starting with the leach column filled with water and overflowing at the design rate while the discharge pump was pumping at a speed sufficient to remove gels at a rate somewhat greater than the feed rate (to prevent a buildup of gels from other than hindered settling). Leaching was performed for 2-1/2 hours at a steadily increasing rate of leach water upflow in an attempt to build up a loading of gels in the tower. The maximum leach water upflow rate was 1250 ml/min.

Streaming and leaching had to be terminated at this time because of excessive pressure drop across the syrup filter (150 psi). After leaching had been terminated, the leach tower had an accumulated load of gels equivalent to 13 minutes residence time. Just before the run was stopped, the lower sample tap on the column indicated a MeC concentration of 9.5% while the overflow contained 12.2% MeC.

The filter was cleaned and reinstalled in preparation for the next run.

d. Run X170-42

Two fresh solutions were made up for this run. Each solution tank contained ~ 50 gallons of solution which included a heel of 4+ gallons below the dip tube. The solutions had apparent viscosities (Brookfield viscometer) of 1850 cps and 2200 cps at 77°C and 74°C, respectively.

A streaming rate of 3.8 lb/hr of NC (dry basis) was used for the run. The temperature of the solution in the syrup tanks was 71°-72°C during the run. Gelling was commenced into the gel bath, which was maintained at 65% MeC and 19°C during the run, and continued for 2 hours before gel removal was initiated. After the gel removal rate was set and the operation of the dewatering screen checked, the gel flow was allowed to enter the leach column.

The leach column was filled with deionized water to the point of overflow, and the liquid displaced by the entering gels was continuously monitored. When the quantity of liquid displaced reached 67 lb, the gel flow was diverted to a catch container. The time required was 90 min (1-1/2 hours) and this quantity of gels was estimated to completely fill the leach column when expanded to the desired bed density (0.4 volume fraction) by the

projected liquid upflow. With this quantity of gels in the leach tower and no takeoff from the bottom, deionized water was fed to the leach column while the fluidization behavior of the pellets was observed. Liquid upflows of 500 ml/min to 2270 ml/min were used without obtaining the desired bed expansion. The 2270 ml/min upflow corresponded to a liquid velocity of  $1.5 \times 10^{-2}$  fps through a bed at 0.6 volume fraction. This velocity should be sufficient to expand the bed. It is believed that particle-to-particle friction was sufficient to prevent bed expansion.

When it was found that the bed could not be expanded once it had settled, it was decided to operate the column as a packed bed. Gels from the dewatering screen were allowed to enter the leach column for an additional 30 min to give a total volume of gels equivalent to 2 hours running time.

Continuous gelling, dewatering and leaching were started. The leach column gel discharge was collected on the lower level in a screen-bottomed container rather than pumped to the dewatering screen on the upper level. This allowed easier sampling of discharged gels for the purpose of balancing the flow between discharged gel volume and entering gel volume. Leach column overflow was maintained at  $\sim 585$  gm/min while the speed of the discharge pump was adjusted to match the gel feed rate of  $\sim 37$  lb/hr (surface dry pellets with about 90% internal liquid).

Gel discharge rate was determined by collecting gels for 1 min, surface drying the gels and weighing them. At the initial pump speed of 50 rpm, gel flow was 14 lb/hr. The pump speed was increased to 132 rpm ( $50 \text{ rpm} \times 37/14$ ) and the gel flow was determined to be 57 lb/hr; the pump speed was then decreased to 100 rpm which gave a gel flow of 56 lb/hr. The pump speed was subsequently slowed to 65 rpm which, in two checks 1 hour apart, gave a gel discharge rate of 38 lb/hr and 41 lb/hr. The correct rate for fully leached gels would have been 35.5 lb/hr.

Fully leached gels were obtained shortly after continuous leaching was started, but they were the result of extra leaching which took place during attempts to expand the bed before continuous feed and discharge were started. Steady-state operation of the leach column was not obtained in the actual 5+ hours of continuous leaching, primarily because of the difficulty in obtaining a gel discharge rate which was constant and equal to the gel feed rate. For most of the leaching period, a slight differential rate existed which was higher at the discharge. This resulted in a falling bed height during most of the run, and at the end of the run the leach column contained 13.7 lb of pellets (surface dry) which corresponded to  $\sim 23$  min residence time at the intended throughput rate. Figure 15 shows the variation in MeC concentration in the column after 3+ hours of continuous leaching. The bed height was slowly decreasing at the time of sampling.



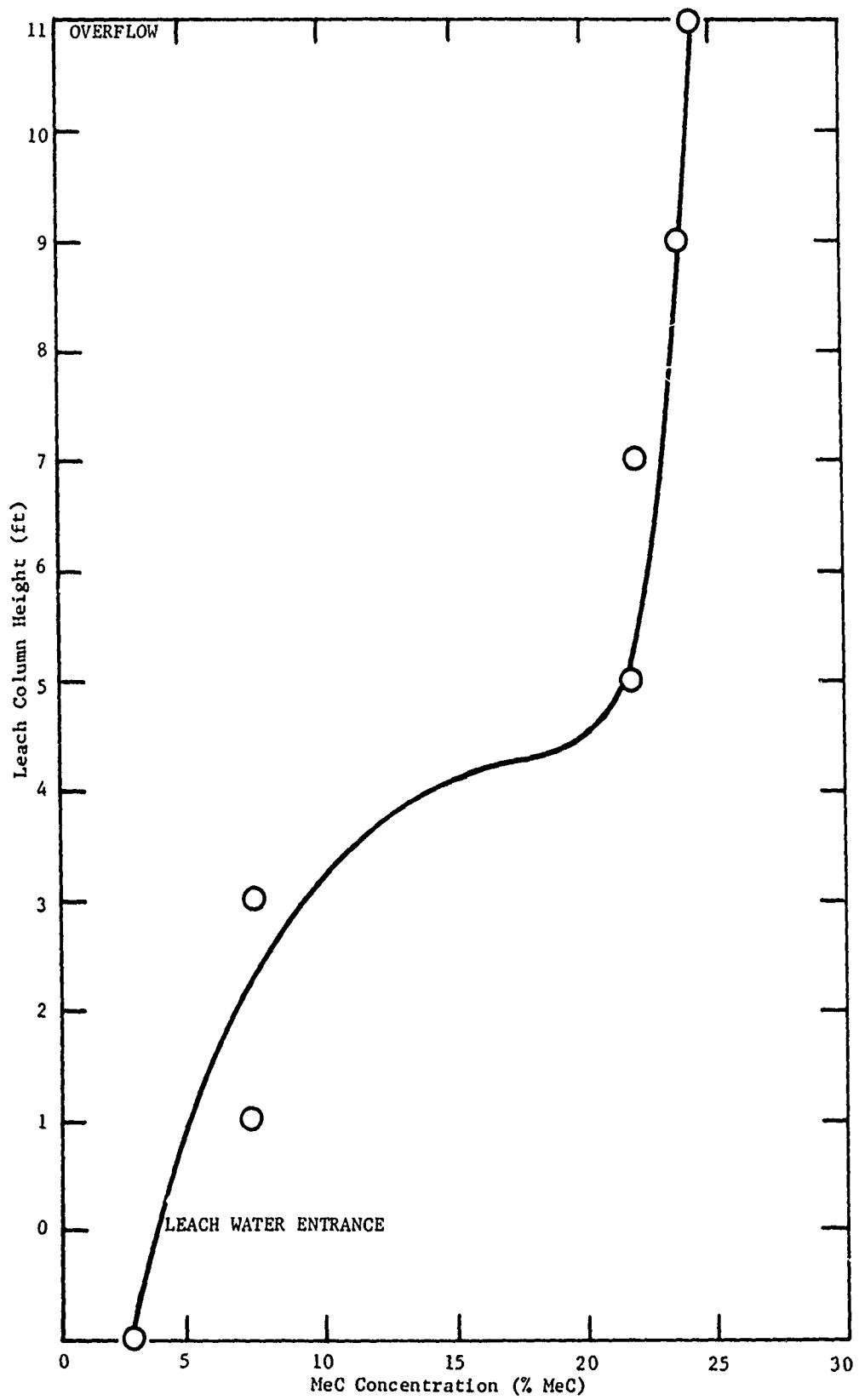


Figure 15. MeC Concentration as a Function of Column Height  
Run X170-42

Although completely leached gels were not obtained during continuous leaching on this run, the system worked essentially as designed; and it appeared that if a sufficiently long residence time could be established in the column, gels could be completely leached. Attempted operation with a 2-hour gel volume at this leach water rate made control of the bed level difficult because the top of the bed was below the upper sight window. Pumps speed adjustments were made after a sample had been collected, externally dried and weighed, which required at least 15 minutes. The initial excessive discharge rate was not reduced to match the gel input rate until near the end of the run. By this point, column residence time was less than 30 min.

During continuous leaching the gel bath concentration was maintained between 65.2% and 66.2% MeC and the leach column overflow varied between 23.0% MeC and 25.1% MeC. The MeC content in the discharged gels averaged about 3.5%.

For the next run it was decided to operate for three 8-hour shifts and to use a leach column loading which would maintain the bed height in the upper sight window for easier control. This was expected to give a gel residence time of 2+ hours and enhance the probability of obtaining fully leached gels.

e. Shock Gel Run X170-44

This run was initiated with two full solution tanks, each containing ~ 50 gallons of 7% NC syrup. Water-wet 13.4N NC of known TV was weighed out before the run to make up two additional solutions. The two initial solutions had been made using the standardized 5-hour solution cycle and exhibited apparent viscosities (Brookfield Viscometer) of 2400 cps and 2320 cps at 60°C.

Run X170-44 utilized a production rate of 3.8 lb/hr of NC (dry basis). Solution pumping was initiated 15 min. after the start of the first shift with the syrup at a temperature of 68°C. After two hours of gelling, takeoff from the gel tank was started. Initial performance of the dewatering screen at steady-state varied between 10.3% and 14.4% free liquid with the discharged gels.

Dewatered gels were diverted to the leach tower, and the leach column was fed for 2 hours and 33 min to bring the bed level up to the sight window. Leach water feed and gel takeoff were initiated to stabilize the bed at the desired level. Leach column flow was set to provide an effluent of 50% MeC. This flow proved insufficient to produce completely leached gels; after nine hours of operation, discharged gels contained 8% MeC while the column effluent contained 53.8% MeC. During this time the flow of water

to the gel bath to maintain a 65% MeC concentration had decreased unnoticed and gel bath MeC content had risen to 70% MeC.

The overflow rate was then increased to 660 ml/min and gel bath concentration control water increased to bring the gel bath back down to 65% MeC. The column stabilized at the new conditions after six hours of operation and was discharging completely leached gels. The gel bath MeC concentration had also been stabilized at 65-66%. Figure 16 shows the MeC concentration over the height of the column under steady-state conditions resulting in completely leached gels. The sample concentrations did not rise continuously over the height of the column.

A material balance around the leach column at the same time as the concentration distribution shown in Figure 16 is given in Table XV.

TABLE XV

MATERIAL BALANCE-SHOCK GEL LEACH COLUMN AT STEADY STATE

	In (lb/hr)			Out (lb/hr)		
	Gels	Free Liquid	Leach Water	Column Overflow	Gels	Free Liquid
NC	3.8	-	-	-	3.8	-
MeC	21.7	6.1	-	27.8	-	-
H <sub>2</sub> O	11.5	3.2	164.4	58.5	31.7	88.9

The actual upflow of water in the bottom of the column was 75.5 lb/hr or ~ 570 ml/min. The run was halted after a total of 23-3/4 hours of solution feeding at the 3.8 lb/hr rate.

As initially charged with gels to the upper sight window on run X170-44, the static bed (~ 2½ hours of gels) was packed to a volume fraction of gels equal to ~ 0.7 which indicated compression of the gels above what would be expected in a shallow bed (typically ~ 0.6). A check of the leach column at the end of run X170-44 showed that it contained a gel load of 66 lb, equivalent to a residence time of 1 hour 50 min and a volume fraction of about 0.44. Average upward liquid velocity through the bed (based on a residence time of 1 hour and 50 min) would be about  $2.8 \times 10^{-3}$  ft/sec and actual downward gel velocity would be about  $1.5 \times 10^{-3}$  ft/sec.

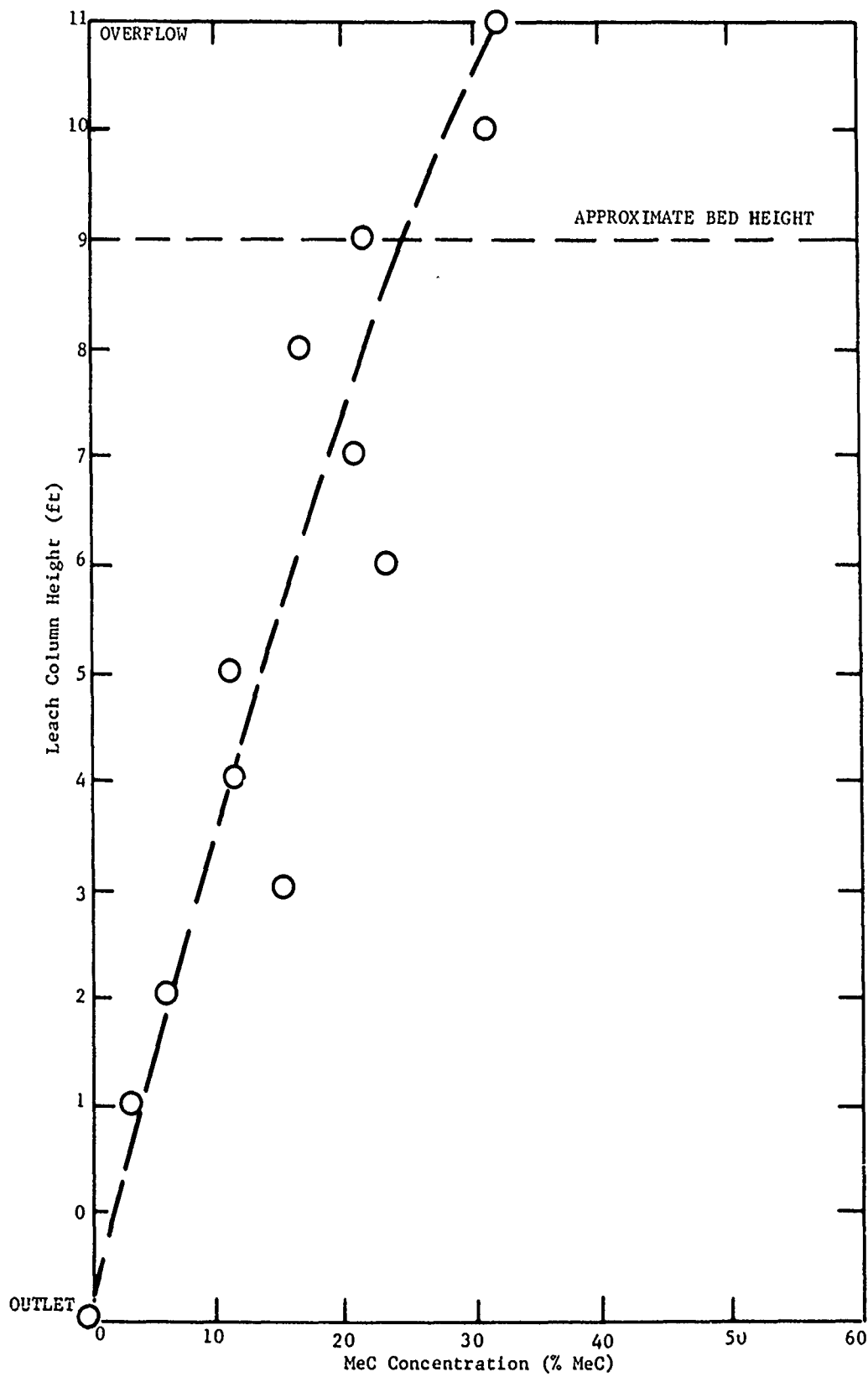


Figure 16. MeC Concentration as a Function of Leach Column Height - Run X170-44

This would suggest a relative velocity of the gels through the liquid of  $4.3 \times 10^{-3}$  ft/sec which agreed well with a measured hindered settling velocity of  $3.6 \times 10^{-3}$  ft/sec for these gels in 30% MeC. The actual average leach liquid concentration was closer to 15% MeC where a hindered settling velocity of about  $5.5 \times 10^{-3}$  ft/sec would be expected.

Thus, although startup was with a packed bed, the pellets went into a hindered settling mode during sustained operation.

The measured hindered settling velocity mentioned above was based on tests conducted with gels collected from the gel tank after the run. These gels, in 64% MeC, were wet screened to determine the average particle size and then used for settling tests at several different volume fractions. The screen analysis is shown in Table XVI and in Figure 17.

TABLE XVI  
SCREEN ANALYSIS OF 64% MeC WET SHOCK GEL FROM RUN X170-44

Screen No.	Hole Size (in.)	Wt. % On Screen	Cumulative % Less Than
45	0.0139	0.2	-
30	0.0234	0.1	0.2
25	0.0278	0.2	0.3
20	0.0331	0.4	0.5
18	0.0394	19.5	0.9
16	0.0469	49.6	20.4
14	0.0555	23.7	70.0
12	0.0661	3.5	93.7
10	0.0787	1.0	97.2
8	0.0937	1.8	98.0
7	0.110	0	100.0

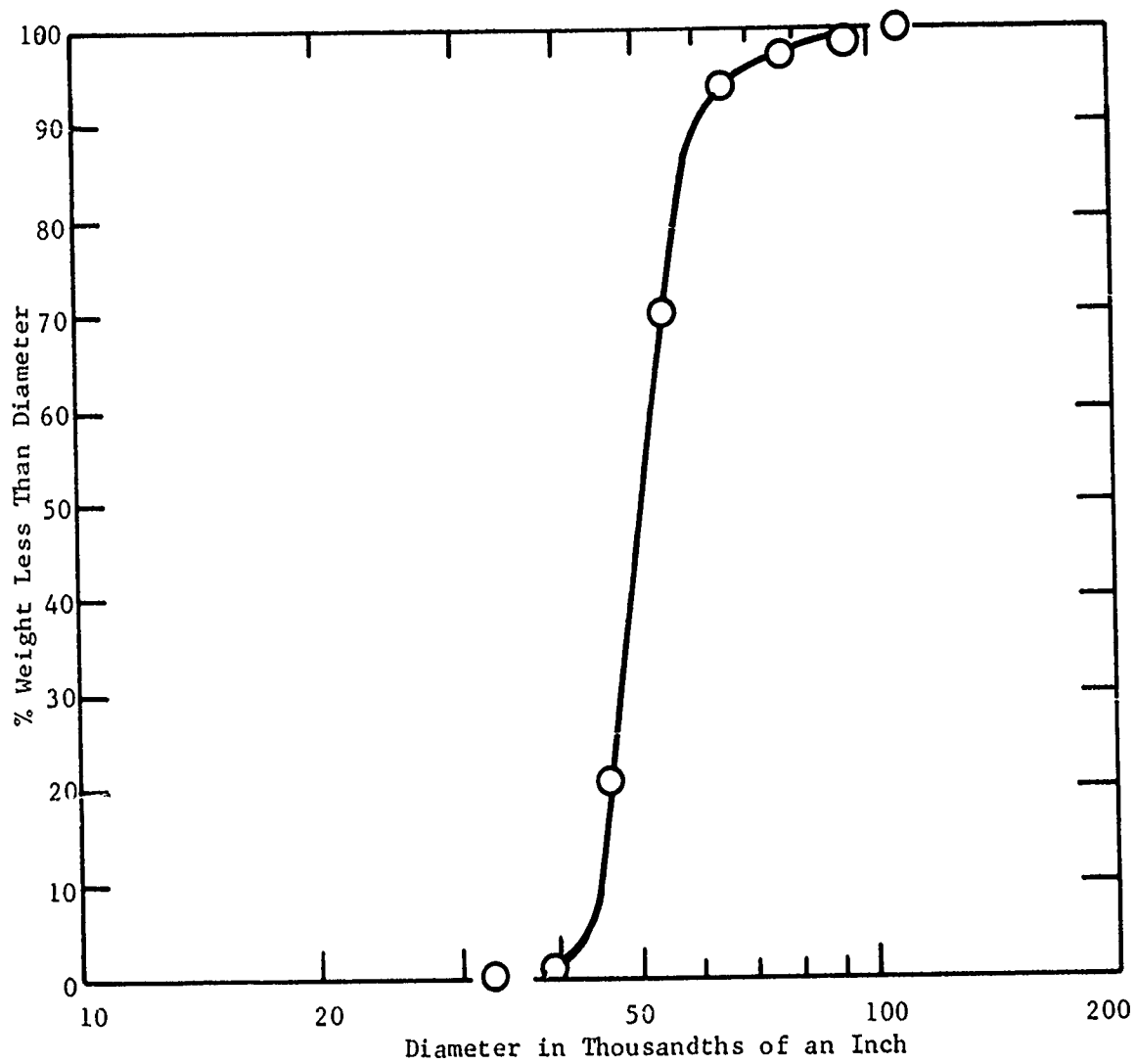


Figure 17. Particle Size Distribution Analysis for Shock Gel X170-44

Figure 18 shows the hindered settling velocity of these particles in 64% MeC, 30% MeC, and water as a function of volume fraction gels.

### 3. Double-Base Operations

Operations utilizing both the single- and double-base portions of the demonstration plant were conducted during June 1975. Single-base operations had indicated that, at a 3.8 lb/hr rate, completely leached gels could be obtained if leach water flow were such that a 30-32% MeC effluent resulted after a 2-hour residence time. Double-base operations were conducted at a 3 lb/hr rate, with a 2½ hour leach column residence time, in an effort to obtain a more concentrated effluent.

For double-base operations the leach column discharge system was modified to improve gel transport to the upper level by substitution of a 1½-in.-I.D. flexible PVC discharge line with air injection in place of the previously used 5/8-in.-I.D. line with water injection, which had encountered occasional complete plugging with gels at the point where the line was reduced in cross-section downstream of the 1½-in.-diameter Moyno pump discharge.

#### a. Twelve-Hour Double-Base Run of 11 June 1975

The first "live" double-base run, conducted on 11 June 1975, was made at a 3 lb/hr rate utilizing three shock gel supervisory personnel and the two regular shock gel technicians.

The gel tank was loaded with gels on 10 June 1975 by streaming for 2½ hours at the 3 lb/hr rate. The gels were maintained in suspension by agitating the gel tank overnight. The initial gel load was established the day before to allow an early state of continuous leaching operations the following day.

Two full solution tanks, each sufficient for eight hours of streaming at the 3 lb/hr rate, were available for the run. Three hours pumping at the 3 lb/hr rate had been used to load the gel tank, leaving 13 hours running time available for the following day.

Streaming into the gel tank and take-off from the gel tank was initiated at 9 a.m. on 11 June. One hour was spent adjusting and characterizing the flow into and out of dewatering screen No. 1. The flow of gels off the screen was diverted into a waste container during this time.

During steady-state flow of gels from dewatering screen No. 1, the gels entering the leach tower were determined to have an average 17.8%

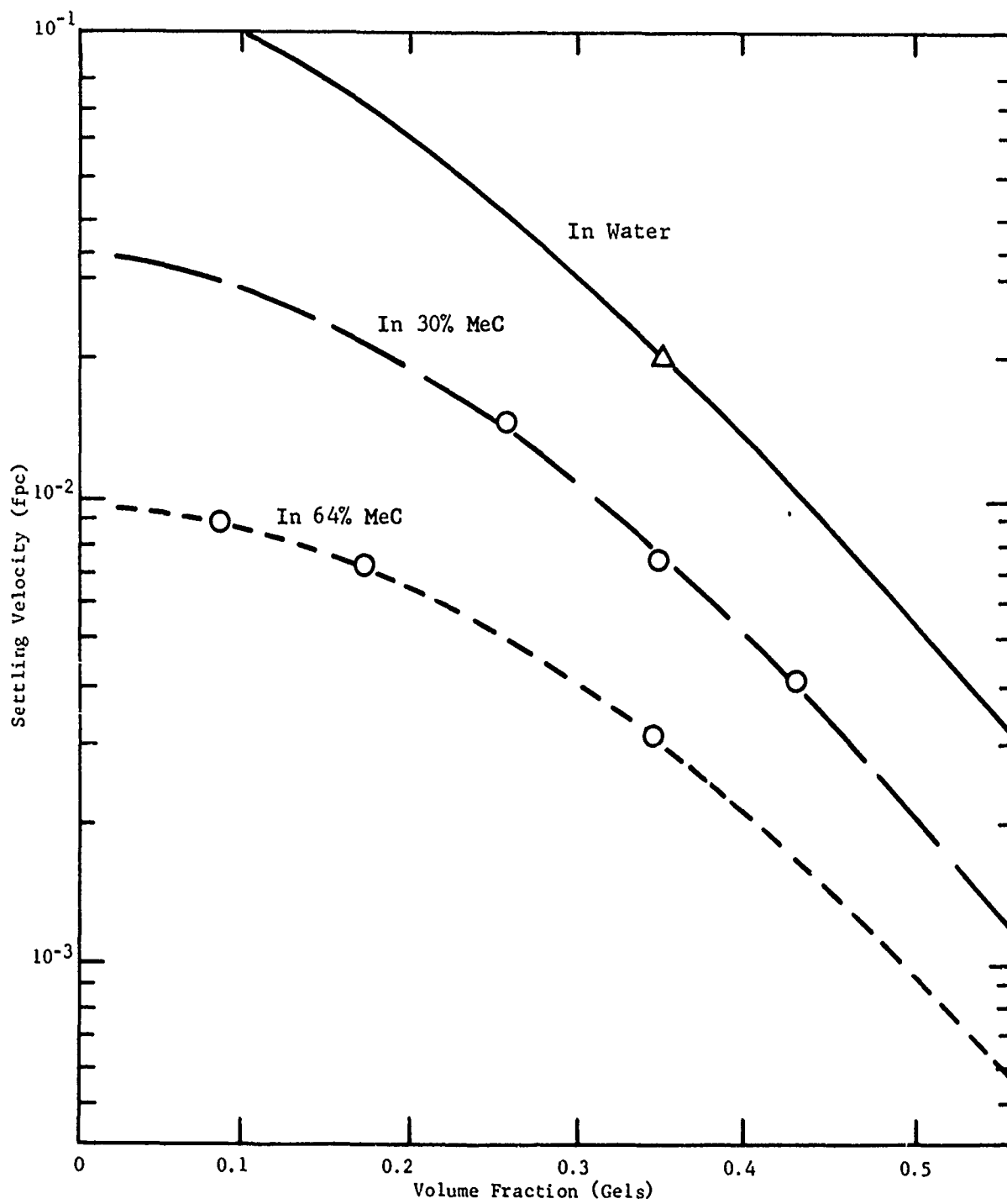


Figure 18. Hindered Settling Velocity of X170-44 Gels as a Function of Volume Fraction Gels



free liquid (65% MeC). A range of 11.7% to 22.3% free liquid was measured during the entire run.

The flow of deionized water to the leach column was set at 450 ml/min during the initial loading of the leach column which was expected to require  $\sim 2\frac{1}{2}$  hours. When the flow of gels was directed to the leach column, the overflow rate increased to 690-710 ml/min.

The gel bed reached the upper sight window (the normal operating level) after 3 hours and 40 min of gel feed. In contrast to previous start-ups (during single-base operations), leach water was fed to the column during the initial loading at the calculated rate of upflow which, if maintained after leach column discharge was initiated, would have eventually resulted in steady-state operation with an overflow of 31.5% MeC. In previous single base runs at 3.8 lb/hr, it was found that a 30-32% MeC overflow could be maintained while fully leached gels were discharged after a residence time of  $\sim 2$  hours. Also, it was thought that loading the leach column with an upflow of leach water present would prevent the previously observed initial high packing density in a static bed. Starting out with an initial upflow would also mean that the first gels discharged could be expected to be completely leached and thus suitable for immediate feeding to the NG impregnation column. This was desirable since the flow from dewatering screen No. 2 (feeding the impregnation column) was not easily diverted.

The 3 hours and 40 min required to reach the sight window in the leach column suggested that the gels were initially packed in the column to a volumetric packing density of 0.74-0.75.

Gel discharge from the leach column to dewatering screen No. 2 was begun when the bed became visible in the upper sight window. The NG/water solution was metered into the column with the gels as the NG impregnation column was filled. The concentration of the NG/water solution in the 300-gallon feed tank was measured shortly before startup and found to be 0.185% NG. The hot solution was metered from the constant head tank at 1865 ml/min. At this solution feed rate and with an NC rate of 3 lb/hr, an NG concentration of  $\sim 7\%$  was expected in the product.

The impregnation column was fed with gels for two hours with no gel take-off. The bed established in the column at this time was  $\sim 7$  feet high. The NG/water solution was able to percolate down through the bed by gravity. The gels acted as column packing to distribute the down-flowing solution. The solution passed out of the screened annulus in the bottom section to one of the 150-gallon NG solution make-up tanks.

The NG solution in the 300-gallon tank was maintained at a temperature of 62°C during the run. The slurry of gels and NG solution going into the impregnation column was at a temperature of 58.5°C. The temperature of the depleted solution discharged from the bottom of the column was 51°-52°C.

Free liquid carried into the NG impregnation column with the gels from dewatering screen No. 2 varied from 11.1% to 20%.

The free liquid carried with gels discharged from the final dewatering screen (No. 3) was measured to be 5.2%. Samples of the depleted NG solution were taken every 30 min after discharge of double-base pellets was begun from the impregnation column. Batches of discharged double-base pellets were collected hourly from the start of take-off. Small samples from each of these batches were dried for analysis.

The run was halted at 8:30 p.m. after having streamed for 12 hours. The leach column had operated from 10 a.m. to shut-down and had discharged pellets for 6½ hours. The NG impregnation column had been filled from 2:00 to 4:00 p.m. and had discharged double-base pellets from 4:00 to 8:30 p.m. The impregnation column had operated satisfactorily, and double-base propellant had indeed been made continuously.

At the end of the run, it was found that the gel tank contained gels equivalent to 2 hours 8 min running time; the leach column contained gels equivalent to 2 hours 32 min running time and the NG impregnation column contained gels equivalent to approximately 2 hours running time. These numbers suggested adequate control of the leach column and NG column content but a slightly excessive take-off rate from the gel tank.

Table XVII shows results of the analysis of the double-base gels from the process.

In the analyses shown in Table XVII, the "probable" % MeC was not directly analyzed but all extractable matter which was not NG or NDPA was assumed to be MeC. Typically, for past lots of batch-leached shock gel powder, this quantity would be approximately 0.5% for pellets which were thought to be completely leached.

For the dry double-base pellets to contain 8.5-12% MeC would suggest that the wet pellets must have contained, on the average, ~ 3-4% MeC. Refractive index analysis of liquid expressed from pellets on screen No. 2 indicated that the gels were usually completely leached; i.e., the refractive

TABLE XVII

DOUBLE-BASE SHOCK GEL PELLETS FROM RUN OF 11 JUNE 1975

	Gels Collected During Run				Gels in Column at End				
	4:00-5:00 P.m.	5:00-6:00 P.m.	6:00-7:00 P.m.	7:00-8:00 P.m.	8:00-8:30 P.m.	Column Top Quarter	Column 2nd Quarter	Column 3rd Quarter	Column Bottom Quarter
Moisture in Wet Pellets (%)	67.4	65.4	62.2	61.8	64.0	65.1	65.0	65.0	65.0
Extractables (%)	18.72	17.37	19.48	19.18	17.71	16.81	17.35	17.42	18.24
NC (%) (By Difference)	81.28	82.63	80.52	80.82	82.29	83.19	82.65	82.58	81.76
NDPA (%)	1.12	1.19	1.12	1.10	1.09	1.10	1.09	1.09	1.09
NG (%)	5.55	6.76	7.18	6.50	6.51	6.39	7.39	7.70	7.42
NG (as % of NC + NG + NDPA)	6.31	7.46	8.08	7.35	7.24	7.05	8.11	8.43	8.22
Probable % MeC	12.05	9.49	11.18	11.58	10.11	9.32	8.87	8.63	9.73
NC (Total Pounds in Lot)	1.63	2.23	2.74	3.88	4.11	1.54	1.42	1.88	1.23
Average NG in Depleted Sol Solution (%)	0.0631	0.0631	0.0645	0.0637	0.0690	-	-	-	-

index of the liquid in the gels was usually the same as that of deionized water. Occasionally a "wave" of partially leached pellets might be discharged from the leach column. For example, at 2:15 p.m. and at 6:20 p.m. during this run, pellets containing 2.1% MeC and 7.8% MeC, respectively, were found to be discharging from the leach column, but in both cases the pellets checked an hour later appeared to be completely leached.

A likely explanation for the apparent generally high level of MeC in the analyzed pellets is the distribution of MeC from these "waves" of unleached pellets throughout the impregnation column by the cocurrent flow of the liquid phase in the impregnation column. Thus, even if most of the pellets in the column had entered completely leached, by the time they were discharged, cocurrent flow of impregnating solution would have "smoothed" the MeC concentration throughout the column.

Based on the NG equilibrium studies described earlier in this report, gels in equilibrium with an NG solution of 0.064% NG (typical for this run) would be expected to have an NG content of ~ 6.1% NG if the equilibrium were at 60°C. Temperatures in the NG impregnation column were actually about 51°-52°C where a higher equilibrium NG concentration would be expected. That the discharged gels were at equilibrium was suggested by the analyses of gels in the column at the end of the run. Very little variation in NG content was seen except for the top quarter of the column. This indicated that the gels had reached their maximum NG content after approximately 30 min vs one hour of residence time.

b. 32-Hour Double-Base Run of 19 June 1975 and 20 June 1975

After the apparently satisfactory 12-hour run of 11 June 1975, it was decided to try to run for a 48-hour period. Some slight changes from the mode of operation used for the preceding run were instituted, including the use of a streaming plate with a Teflon-coated exterior, the addition of separate "Lightning" agitators for each NG solution tank, and the installation of a duplicate 2-ft<sup>2</sup> syrup filter in parallel with the original filter.

It was thought that the Teflon-coated plate would alleviate orifice plugging attributed to gelling of syrup on the surface of the plate by moist air, by eliminating spreading of the syrup across the plate.

While the two starting solutions were being made for the run, one of the agitator bearing boxes became noisy and overheated. When the bearing box was disassembled, it was found that the thrust collar on the shaft had been rubbing on the side of the housing. The condition was rectified by removing a small amount of material from the O.D. of the collar.

The bottom bushing was reimpregnated with oil, and the box was reassembled with new thrust bearings and packing and operated satisfactorily during the run. In addition to the two starting solutions, NC was preweighed for four additional solutions.

Gelling was initiated at 12:40 a.m. on 19 June 1975, and the gel tank was given a 1-hour load before gel take-off. This was a change from the previous run when a 2½ hour gel tank residence time had been used because the low take-off rate associated with the 2½ hour residence time was difficult to control accurately.

The leach column was filled in the same manner as on the previous run and required 3½ hours to reach the sight window. Leach column discharge was started at 6:35 a.m., and the NG impregnation column was filled to the 7-foot level by 8:15 a.m. Because of the initial high volumetric packing density in the leach column, the gels were initially removed at a rate higher than 3 lb/hr as the bed opened up. The 0.2% NG solution was fed into the NG impregnation column at a rate of 1765 ml/min.

Difficulty in maintaining a continuous discharge of gels from the leach column became a problem during the morning of June 19. The flow from the No. 1 screen had to be diverted several times to avoid overflowing the leach column. Usually gel discharge could be restored by squeezing the line between the column outlet and the pump inlet for several minutes. When the leach column was discharging gels poorly, the discharge pump had to be run at high speed to take out enough gels to maintain a constant bed level. When the pump was operated at high speed, pumping mostly water, column operation was upset due to insufficient leach water upflow. Thus the flow of leach water to the column had to be increased. An upset in the column discharge rate also caused an upset in the NG impregnation column and the column discharge could not be run continuously without drastic bed height reduction.

During the late afternoon of June 19, the Teflon-coated streaming plate was removed and the original plate substituted (all within ~ 5 min) because a number of holes had plugged and poorly formed gels were becoming evident.

Operations continued into the night and on into the morning of June 20 with the leaching operation requiring constant attention to avoid complete cessation of operations. New solutions were made up on schedule with the expectation that conditions would improve.

It finally became apparent on the morning of June 20 that conditions in the leach column were unlikely to improve. As the discharge line

was being manipulated to restore flow and the top of the gel bed was out of sight above the upper view port, the lower portion of the gel bed could be seen to separate from the upper portion through the lower view port. The separation was apparently caused by dissolved air coming out of the leach water during a prolonged period of zero gel flow (in or out). The run was finally terminated on the morning of June 20 after 32 hours of operation. Steady-state operation was not achieved during the run because of the problems with the leach column, and consequently very little operating data of any value were obtained.

During clean-up of the leach column after the run, the bottom cone of the leach column was found to have two metal washers lodged in the outlet. The washers had apparently fallen into the column during the previous run and had not been discovered. The washers were 1 in. in diameter with a 3/8-in. orifice and were acting in the manner of a random-closing butterfly valve.

c. 24-Hour Double-Base Run of 27 June 1975

For this run the washers were removed from the leach column and the streaming head was modified to allow the area immediately below the head to be sparged with dry nitrogen at 10-20 scfh. It was believed that the dry nitrogen sparge would reduce hole plugging in the orifice plate. Additional heating capacity was added to the tempered water system for the NG solutions so that higher temperatures (65°C) could be maintained.

Two NC solutions were made up before the run, and NC for a third solution was preweighed. Streaming at 3 lb/hr was initiated at 1 a.m. on June 27. The gel tank was given a 2-hour gel load before gel discharge into the leach column was begun. Without takeoff, the leach column required 3 hours for the gel level to reach the upper sight window. When leach column discharge was begun, the leach water was adjusted to maintain a column overflow of 670 ml/min.

Filling the NG column to the 7-foot level required 2 hours and 15 min. Feed of 0.2% NG/water solution was maintained at 1765 ml/min. Discharge of double-base gels was begun at 9:45 a.m. on June 27.

The gels initially discharged from the leach column were not fully leached and contained 3.9% MeC when leach column discharge was initiated. To gain additional experience with double-base operations, it was decided to go ahead with NG impregnation. Since the gel MeC content was dropping, it was also decided to maintain leach column parameters constant rather than increase leach water flow.

The leach column was operating smoothly and there was little difficulty in maintaining the bed level constant. The MeC content of the discharged gels continued to drop, and by noon (12:00) the discharged gels were completely leached and the MeC content of the leach column overflow was 28.8%.

During the period a new shock gel solution was made. At 12:30 p.m. the first 150-gallon tank of depleted NG/water solution was filled. The NG concentration was measured and found to be 0.0631%. The solution was reconstituted to 0.2% NG and pumped back to the 300-gallon tank. Because of the modifications to the tempered water system for the NG solution tanks, it was possible to keep them at 66°-69°C during the run. The gel/NG solution slurry in the impregnation column feed pump inlet funnel stayed at a temperature of 55°-58°C while the temperature at the bottom of the column was 52°-54°C.

Between 2:00 and 3:00 p.m. the leach column discharge pump drive motor began making noises and developed occasional seizures of its integral gear reduction unit. At about 3:25 p.m. the gear reduction unit seized and could not be restarted. Operations were halted while a replacement drive unit was sought. Between 4:00 and 5:00 p.m. a Gast 6 AM air motor was connected to the leach column discharge pump via a direct drive.

All systems were restarted at 5:30 p.m. and an attempt was made to continue operations. Unfortunately, the direct drive motor on the leach column discharge pump could be operated only intermittently because the minimum motor speed was too fast. An attempt was made to operate in this fashion until 10:30 p.m., but the results were unsatisfactory.

Analytical results for samples taken during this run are shown in Table XVIII. It can be seen from the analysis that the smooth operation of the leach column produced double-base pellets from the impregnation column with only a minor amount of MeC except for the last sample. During the period from 3:00 p.m. to 3:35 p.m., difficulties with the leach column discharge pump had ended the relatively smooth running experienced up to that time.

There was substantial plugging of streaming plate orifice holes during this run even though the N<sub>2</sub> sparge system was active during the entire run. Microscopic examination of the plate after the run revealed that only 77 holes out of 160 were completely open. Fifty-two holes were completely blocked and 31 were partially blocked. The material blocking the holes appeared to be very small pieces of scale from the inside of the syrup delivery pipe. This may be eliminated in future runs either by moving the syrup filters to just upstream of the streaming head or by incorporating a separate final filter.

TABLE XVIII

DOUBLE-BASE SHOCK GEL PELLETS FROM RUN OF 27 JUNE 1975

	9:45 a.m. to 11:00 a.m.	11:00 a.m. to 12:00 Noon	12:00 Noon to 2:00 p.m.	2:00 p.m. to 3:25 p.m.
Moisture in Wet Pellets (%)	62.6	62.8	64.5	60.3
Dry Weight of Sample (lb)	4.4	5.8	6.0	8.2
Extractables (%)	10.07	9.98	8.95	10.94
NC (%) (By difference)	89.93	90.02	91.05	89.06
NDPA (%)	0.98	1.00	1.03	1.04
NG (%)	8.32	8.59	7.50	8.75
NG (as % of NC + NG + NDPA)	8.38	8.62	7.53	8.85
Probable MeC (%)	0.77	0.39	0.42	1.15
Average NG in Depleted Solution (%)	-	-	0.0694	0.0618

4. 3-5 Lb-Hr Demonstration Plant Performancea. Leach Column

The apparent simplicity of the continuous shock gel leach column belied its actual operation. The leach operation itself was simple, but the problems of obtaining smooth, continuous functioning were not. As can be seen from Figure 16, the variation in MeC concentration over the height of the column did not increase smoothly from bottom to top, as would be expected, but varied sinously from point to point.

When first discerned, this effect was thought to be due to short-circuiting of gels and fluid within the column. On further reflection it seemed



more likely to be due to perturbations in gel feed rate, gel discharge rate, and leach water upflow rate. As an example, during the double-base run of 27 June 1975, the speed of the discharge pump on the leach column had to be changed about 5 times per hour to keep the top of the gel bed within view of the upper sight window. Each time the speed of the pump was changed, a compensating change in the total flow of leach water to the column was made to keep the water upflow constant. Variations in the discharge rate of gels from screen No. 1 were smaller but introduced an additional complication to the job of keeping the liquid upflow constant.

Overall mass transfer rates, as experienced in the leach column, were considerably lower than mass transfer rates which were previously measured under well-agitated conditions. Effective diffusivities for the diffusion of MeC from gel to leach water were much lower (perhaps as much as an order of magnitude). Presumably because of low turbulence a stagnant film existed on, and near, the gel surface, slowing diffusion.

The most effective method of improving mass transfer in the column without extensive redesign (e.g., complicated internal agitation mechanisms, etc.) is believed to be to increase the temperature. The leach water should be heated and deaerated in a single step. This should also eliminate the occasional leach stoppages believed to be caused by air released from the leach water and attaching itself to the gels.

To improve control of the rate of gel feed and thus eliminate the necessity of continually varying the discharge rate, direct gelling into the leach column should be used.

#### b. NG Impregnation Column

The NG impregnation column operated extremely well. No problems were experienced with the impregnation column which were not traceable to an upset in the leach column. Precise control of the rate of feed of NG solution should allow precise control over the NG content of the impregnated gel. If the rate of leached gel feed from the leach column can be made as precise as the feed of impregnating solution, then gel NG contents should be essentially constant.

The only area of improvement necessary for the impregnation column itself is to raise the equilibration temperature in the column. This can be done by simply raising the temperature of the incoming solution. A higher temperature will have the further benefit of reducing the water content of the impregnated gel.

Mass transfer within the column is fast enough that the column probably has a capacity three or four times the present 3 lb/hr rate.

c. Solution Formation

Typically, 7% solutions could be made up in 4-5 hours from the time feed from a tank was halted. With the lid slid back, 27.25 lb of NC (usually about 38 lb water-wet) was added to the tank in increments of 2-3 lb each along with MeC in increments of about 30 lb each. At the same time, 243 gm of NDPA was added. The NC was broken up during addition with a nylon-bladed paddle with assistance from the agitator once the liquid level was high enough. When all the NC and 254 lb of the MeC had been added, the lid was moved into place and clamped tight. The mixture was agitated under vacuum at 2000 rpm with maximum heating for one hour. Agitation was then halted, the vacuum released, and 94.75 lb of MeC added through the addition port. Agitation at 2000 rpm under vacuum was then continued for a total of 5 hours from the start of the cycle. Maximum heat (90°-95°C jacket temperature) was maintained until the desired temperature 74°C was reached.

Seven-percent solutions made by this technique typically had apparent viscosities of ~2000 cps at 74°C. The exact viscosity depended upon the amount of heel in the tank at the time of solution makeup.

d. Syrup Metering

No problems were experienced with the syrup-metering system. A 10-psig head was maintained on the tanks while pumping to ensure a full feed. The syrup flow rate was measured occasionally by catching the falling syrup from the streaming head for a measured time period and weighing it. In all cases the measured flow rate corresponded exactly to the calculated flow.

e. Syrup Filtration

The two 2-ft<sup>2</sup> sintered metal syrup filters did a good job of filtration. However, cleaning the filters after a long run was time-consuming.

The filters were cleaned by backflushing with clean MeC and brushing the exterior of the tubes. The filters were then cooked in boiling caustic solution (3N KOH) for two hours. This was followed by washing with warm soapy water and drying. When dry, the filters were soaked in two changes of either 95% H<sub>2</sub>SO<sub>4</sub> or 85% HNO<sub>3</sub> for an hour each, followed by washing with warm soapy water and drying.

Although the filters worked well, the labor involved in cleaning them suggests that disposable filters would actually be cheaper.

f. Streaming

No difficulty was experienced in obtaining good streaming and stream breakup so long as the syrup was maintained at 74°C. Other than orifice plugging, which was at first incorrectly attributed to moisture gelling of syrup on the plate, no problems arose with the streaming system. Plugging was found to be caused by pieces of pipe scale.

g. Gelling

The 70.3-gallon, jacketed gel tank was agitated by an air-driven "Lightnin" mixer. Although it served well in the gelling function, the difficulty in maintaining a precise discharge rate suggested that the separate gelling step should be eliminated. Typically, a sufficient gel concentration gradient existed in the tank from top to bottom such that the tank was slowly depleted of gels over a long period of running. Direct gelling will eliminate this difficulty.

h. Dewatering

Typical flows to the three dewatering screens were 35 gph for screen No. 1, 6 gph for screen No. 2 and 7 gph for screen No. 3. Typical pellets from screen No. 1 had about 20% free liquid, those from screen No. 2 about 4%, and those from screen No. 3 about 5% free liquid. Typical screen operating parameters, shown in Table XIX, suggest that screen No. 1 was undersized.

TABLE XIX

DEWATERING SCREEN OPERATING PARAMETERS

Screen No.	Free Area (ft <sup>2</sup> )	Liquid at Temp. °C	Typical <sup>(1)</sup> Flow (gph/ft <sup>2</sup> )	% Free Liquid on Gels
1	0.29	65% MeC at 20°C	108	15-20
2	0.23	H <sub>2</sub> O at 20°C	8.9	3-5
3	0.23	H <sub>2</sub> O at 50°C	28.7	4-7

(1) Based on DB run of 27 June 1975.

i. NG Solution Makeup

Reconstitution of depleted NG solutions was done in the two 150-gallon depleted solution tanks. These tanks were equipped with air-driven, "Lightnin" mixers and 3.8-in. propeller agitator blades.

Solutions were made up by adding the required amount of NG (as a 50% solution in acetone) in four equal increments and agitating at maximum speed for 15 min after each increment addition. This procedure resulted in complete solutions with no difficulty.

j. Fast Analytical Technique for Dilute NG/Water Solutions

Operation of the 3-5 lb/hr demonstration plant required analysis of the depleted NG/water solutions for NG concentration followed by reconstitution of the solutions to 0.2% NG concentration and transfer of the reconstituted solutions to the 300-gallon feed tank. At the 3 lb/hr (dry NC basis) rate, each 150-gallon tank must be reconstituted approximately every 5 hours. The analytical technique used in the past (method 208.2.2 of MIL-STD 286A) required a considerable time period, was tedious, and lacked accuracy at low NG concentrations. A new technique utilizing polarography and referenced to a calibration curve was developed for use by shock gel operating personnel; this technique gave good accuracy and yielded results within 1 hour, or less, from the time the sample was taken.

The technique made use of the reducibility of nitroglycerin by a dropping mercury cathode. The reduction results in a current which is proportional to the concentration of the nitroglycerin at voltages where complete concentration polarization occurs.

A standard NG/water solution of known NG concentration was first made up, and a series of polarograms were made at various dilutions using a Sargent Recording Polarograph. Potassium chloride at 0.08N was the supporting electrolyte with a quiet pool mercury anode and a dropping mercury cathode. Twenty-five milliliters of liquid consisting of 5 ml of the sample and 20 ml of 0.1N KCl was contained in the electrolysis vessel which was thermostatically controlled at 29°C.

A plot of diffusion current as a function of concentration was prepared which was linear and passed through zero at zero concentration. A fully developed wave was obtained at a cathode potential of -0.9 volt. Some interference from the simultaneous reduction of 2-NDPA (the NG solvent used for the standards was 99/1 NG/2-NDPA) was apparent but appeared to account for < 1.5% of the diffusion current when the NG was at the 0.1% level.

For shock gel process control purposes only, the total wave height at -0.9 volt was measured, and by reference to the calibration curve, the NG concentration could be determined.

Accuracy on unknown samples in the 0.05-0.1% NG concentration range was about  $\pm$  1-2%. Procedures were written for the analytical technique, and three persons were trained in its use to obviate the need for analytical support during around-the-clock shock gel operations.

## SECTION IV

### CONCLUSIONS AND RECOMMENDATIONS

#### A. CONCLUSIONS

Completion of this contract has resulted in the following conclusions:

Processing of high NC-concentration shock gel syrups (as high as 15% NC) into gun propellants suitable for 5.56mm was shown to be feasible but will require either the use of low-viscosity NC and the development of techniques for handling dry (or nearly dry) NC, or else quick moisture removal from very high-viscosity syrups wherein the NC is not in complete solution.

The feasibility of continuous processing of shock gel syrups of moderate concentration (7% NC) through nitroglycerin impregnation was demonstrated at a rate of 3 lb of NC per hour with shock gel pellets of a size suitable for further processing into a gun propellant for 5.56mm.

The shock gel continuous leach column used for the 3-5 lb/hr demonstration plant did not perform as well as expected because of low rates of mass transfer under the conditions of operation and control difficulties which prevented smooth operation.

The shock gel, continuous NG-impregnation system performed well in the intended mode of operation and was capable of operation at throughput rates greater than 3-5 lb/hr.

#### B. RECOMMENDATIONS

The following recommendations are made for further investigations of the shock gel process for the manufacture of small arms gun propellants:

Demonstrate a continuous leach column that incorporates the gelling function and which can operate at high leach temperatures to improve mass transfer.

Use the shock gel process to demonstrate the capability of making shock gel pellets suitable for further processing into a gun propellant for 20-40mm ammunition.

Define the starting NC viscosity requirements for an NC suitable for processing into an immediately usable 10-15% NC shock gel syrup.

Investigate, as an aid to eventual scale-up, the completely continuous formation of shock gel syrups.

APPENDIX I

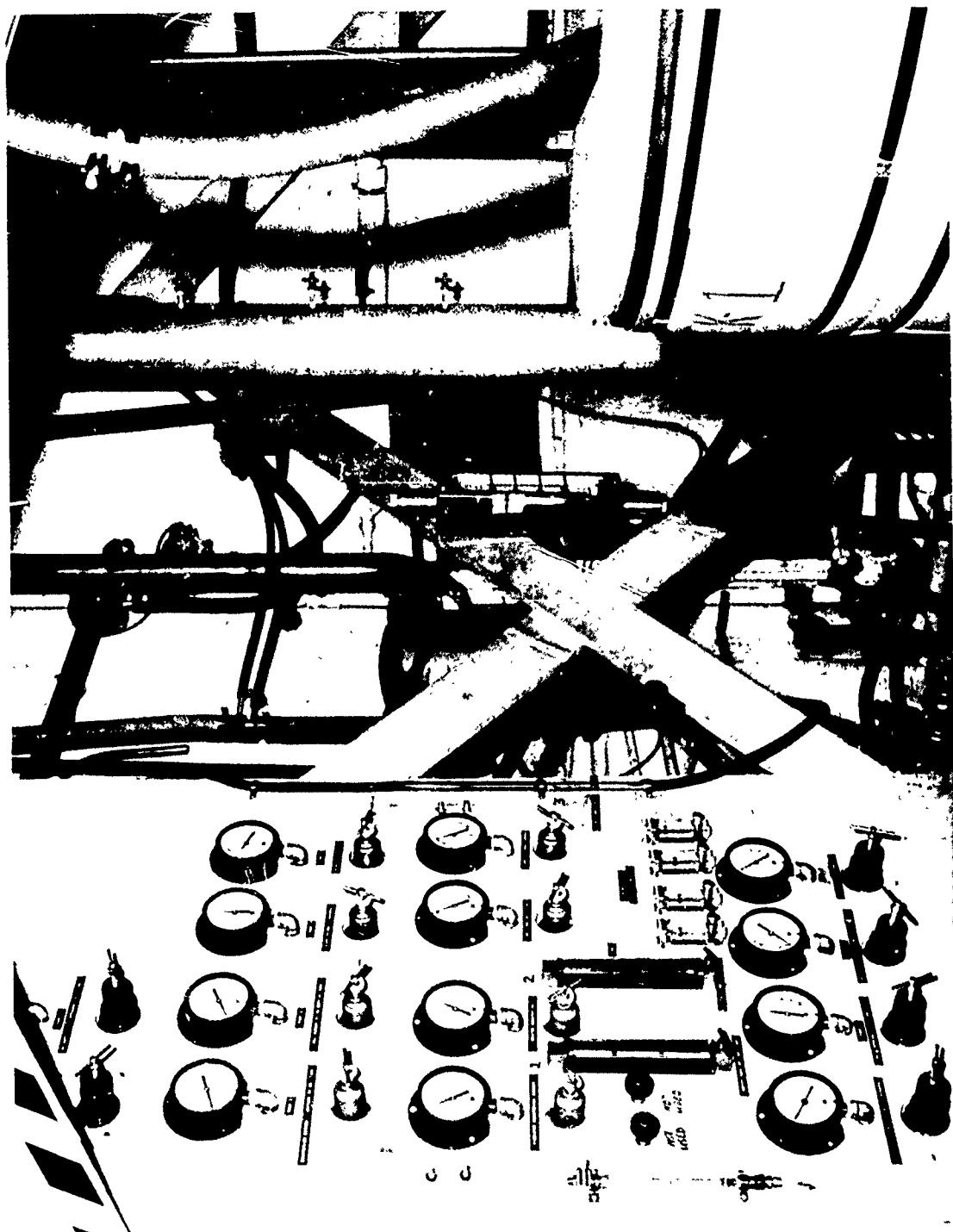
PICTORIAL VIEWS OF SHOCK GEL 3-5 LB/HR DEMONSTRATION PLANT

Some views of the shock gel 3-5 lb/hr demonstration plant are shown in the following pages. The photos were taken during the 24-hour run of 27 June 1975.



Figure I-1. Overall View of Shock Gel Plant (Solution Making and Transfer Equipment are out of sight to left)





G-3592

Figure I-2. Lower Level Control Panel with 300-Gallon NG Tank (in foreground),  
Leach Column and NG Impregnation Column (in background)

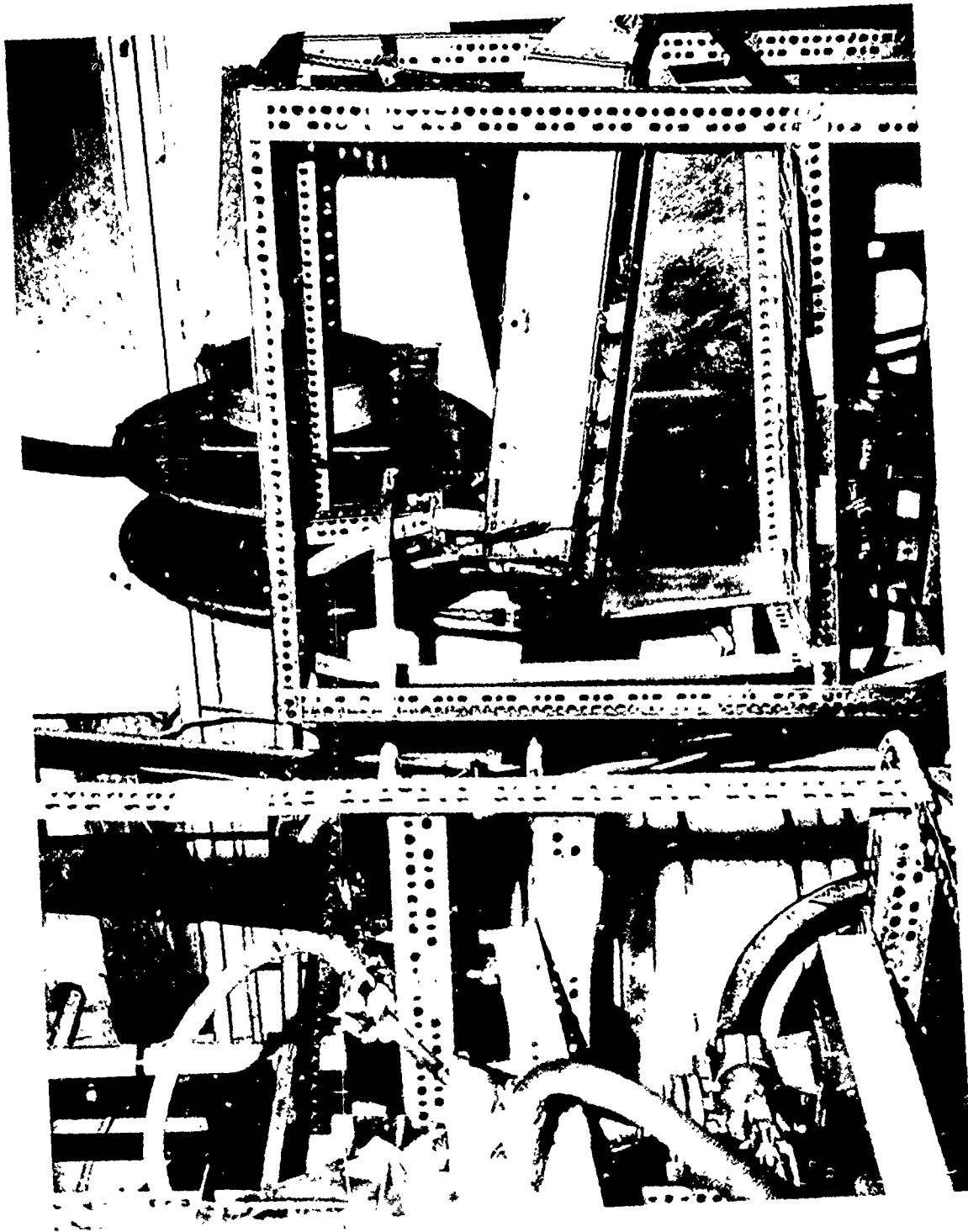


Figure I-3. Streaming Head in Operation with N<sub>2</sub> Sparge Shield in Place

G-3593

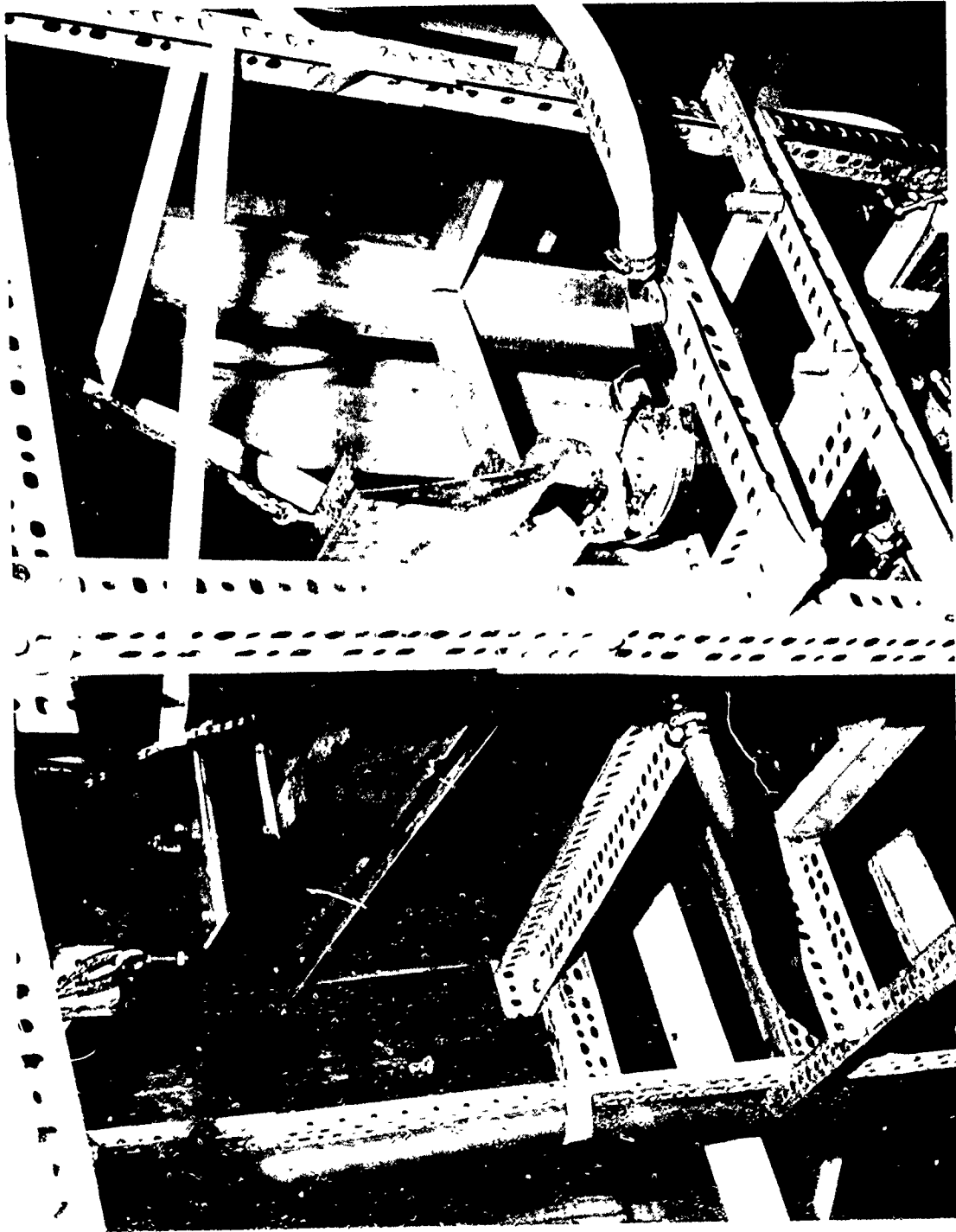


Figure I-4. Lower Portion of Leach Column (on left) and NG Impregnation Column (on right) G-3592



G-3589

Figure I-5. Dewatering Screen No. 1



G-3591

Figure I-6. Dewatering Screen No. 2 Feeding Gels to NG Impregnation Column Feed Pump

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A program was conducted to develop the shock gel process for the manufacture of small arms gun propellants. A 3-5 lb/hr facility to demonstrate the continuous manufacture of 13.4% N nitrocellulose, shock gel gun propellant was designed and operated. Overall operation of the demonstration plant was generally satisfactory. Additional studies to simplify and improve the operation of the continuous leaching process were recommended.		

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ABSTRACT (cont.)

The manufacture of shock gel gun propellant utilizing syrups with high nitrocellulose concentration (15% NC) was demonstrated. The resulting propellant had high absolute and gravimetric loading densities. This propellant, deterrent coated with 5% DBP, exceeded the velocity of reference ammunition at chamber pressures below 50,000 psi.

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