LEVEL III

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DMSO RECRYSTALLIZATION OF HMX AND RDX

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This project was accomplished as part of the U.S. Army Manufacturing Methods and Technology Program. The primary objective of this program is to develop, on a timely basis, manufacturing processes, techniques, and equipment for use in production of Army materiel.

19. KEY WORDS *(Continue on reverse side if necessary and identify by block number)*

Recrystallization	Pilot plant
DMSO	Continuous cooling crystallization
RDX	Dilution crystallization
HMX	MMT-Process improvement

20. ABSTRACT *(Continue on reverse side if necessary and identify by block number)*

A pilot-scale process for recrystallization of RDX and HMX from DMSO was designed, procured, installed, and evaluated at Holston AAP. Process design was based on previous work which indicated cooling crystallization techniques using continuous nucleation and classified product removal to be most effective. Although all expected classes of material were successfully recrystallized, operating difficulties forced departure from the original equipment design and required implementation of alternate operating strategies. As a result, five distinct modes
(Continued)

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20. ABSTRACT (cont)

of operation were established, dependent upon the particular class of material being recrystallized. Two of these (for Class 5 RDX and Class 5 HMX) were batch.

Although the pilot plant failed to demonstrate the design concept of a single, multiclass recrystallization process using separation and recombination to give desired class, the direct recrystallization techniques developed were satisfactory and, in some cases, could be scaled-up based on the data generated. In other cases more piloting will be required in order to develop the data base necessary for sound scale-up.

Material recrystallized in the pilot plant was shown to meet all existing specifications. A variety of explosive formulations was produced for further testing which will include both interim qualification and end item tests.

A preliminary economic analysis indicates DMSO to result in significant cost reduction for recrystallization of HMX at production rates in excess of 150,000 pounds per month, while little or no cost reduction is anticipated for RDX.

CONTENTS

INTRODUCTION

Laboratory work performed during the Phase I study of "Recrystallization and Growth of HMX/RDX, a Study of Methods and Equipment"[1] indicated that significant increases in explosives recrystallization rates with corresponding decreases in recrystallization costs could be achieved in existing batch equipment using DMSO and cooling crystallization processes. The improvement was attributed primarily to the larger batch sizes possible due to the higher solvent power of DMSO for RDX and HMX when compared with the standard recrystallization solvents, acetone and cyclohexanone. Other benefits of recrystallization from DMSO included improved crystal characteristics such as clarity and shape. Cooling processes were also shown to provide safer, more controllable recrystallization systems which could provide more consistent product particle size distributions (PSD).

The Phase II pilot plant study[2] revealed that the predicted batch size increases were obtainable in batch, stirred-tank cooling crystallizers. Also, the products from these recrystallizations compared favorably with similar products recrystallized by conventional methods. However, it was also discovered that production of the larger particle size distributions in batch equipment was difficult. Therefore, the batch systems were judged practical only for producing the finer PSD's (Classes 1 and 5). Effective production of the coarser Class 3 and Class 4 distributions required the fines control capability of the draft-tube crystallizer which was also evaluated during this pilot plant. The draft-tube design had the disadvantages of low production rates and high equipment cost.

A need for the simplicity of the batch stirred tank equipment plus the fines control capability required for production of coarse explosives led to the investigation of a batch, controlled nucleation, classified product removal (CNCPR) crystallization system during the later part of the Phase II pilot plant. The high effective growth rates achieved in the batch CNCPR system as well as the high production rate potential of this system provided the incentive for the more detailed investigation presented in this report.

The accomplishments of the Project 4310 pilot plant phase included the following:

1. Process design of the CNCPR crystallization system and all auxiliary support systems.

2. Mechanical design of the pilot equipment and specification of instruments and control systems.

3. Procurement, modification, and installation of pilot plant equipment.

4. Development of a liquid chromatographic procedure for process control and pilot plant performance evaluation.

5. Completion of a hazards analysis based on the final pilot plant design.

6. Evaluation of the pilot plant including debugging, calibration, operator training, operation, and product manufacture.

7. Analysis of the data generated, the presentation of conclusions reached, and recommendations for future work based on the results achieved.

EXPERIMENTAL

EQUIPMENT

The equipment and facilities used to perform the experiments during the pilot plant phase are located at Building C-6, Holston Army Ammunition Plant. Most of the building equipment and the new process equipment were used to perform experiments as required.

Process and Instrumentation Diagram

The original process flow sheet (P&I) diagram for the Building C-6 pilot plant recrystallization process is presented as Figure 1. The equipment, instruments, and connected piping required to perform the basic experiments and produce recrystallized RDX and HMX are indicated on this figure. Changes in process configuration are not indicated on this figure but are discussed in detail in Section IV.

Equipment Layout

The original equipment layout plan for the pilot plant is detailed by Figures 2, 3 and 4. Table 1 presents a listing of each piece of equipment and its designated code number.

Process Equipment Description and Basic Functions

Slurry Mix Tank, CA-52 (Figure 5)

The slurry mix tank is a 50-gallon (0.189 m^3), agitated, 304L stainless steel tank used to blend the input materials for the recrystallization processes. The tank is positioned on three hydraulic load cells which are calibrated to determine the net weight of the tank contents. A dial indicator is provided for reading the weight. (Note: Due to failure of the hydraulic load cells the weigh system could not be used. The slurry mix tank was instead volumetrically calibrated for liquid measurement. Solids and make-up DMSO were weighed prior to addition).

Charging of explosives is accomplished by shoveling from a nutsche or charging from preweighed bags. Recycle solvent is pumped from storage tanks while any virgin solvent required is pumped directly from a drum as needed. A hydraulic, variable speed agitator is provided for mixing the ingredients. Slurry is transferred from the slurry mix tank to the evaporator feed tank by opening the air operated slurry mix tank drop valve and starting Pump CA-10.

Evaporator Feed Tank, CB-50 (Figure 6)

The evaporator feed tank is a 200-gallon (0.757 m^3), agitated, 304L stainless steel tank equipped with coils and jacket for heating or cooling. Material received from the slurry mix tank may be heated in this tank to prepare it for feeding to the evaporator. Automatic temperature regulating instrumentation is provided. The feed solution is continuously circulated through a low pressure steam-jacketed loop by Pump CB-10 with a feed take-off to the evaporator. The feed rate to the evaporator is controlled by the evaporator level signal and maintains proper evaporator level.

Evaporator, CB-51 (Figure 7)

The evaporator is a 100-gallon (0.379 m^3) 304L stainless steel, full vacuum rated tank equipped with coils and jacket for heating or cooling. Automatic temperature regulating instrumentation is provided to control the steam flow to the coils and jacket. Total system pressure is variable and automatically controlled by an air bleed to the two stage vacuum jets mounted on the roof. The solution in the evaporator is continuously circulated through a steam jacketed loop by Pump CB-11. Agitation of the tank contents is furnished by the returning fluid stream from this loop.

Feed to the dissolver is drawn from this loop via a control valve and magnetic flowmeter. The vapors from the boiling tank contents are fed directly into the rectifying column (CB-21) which is integrally mounted on top of the evaporator.

Rectifying Column, CB-21 (Figure 8)

The rectifying column is a packed column consisting of a 12-inch (0.30 m) diameter, 304L stainless steel shell packed with one-inch (25.4 mm) sodium-etched, Exlon rings. Active packed height is 18 feet (5.49 m) with liquid redistribution every 4.5 feet (1.37 m) and gas redistribution in the center of the column. Liquid feed distributors are located at six feet (1.83 m) and at 15 feet (4.57 m) from the bottom of the column. Reflux rate is controlled by action of a timed three-way valve connecting the distillate condenser to the distillate receiver and the reflux head. Vacuum is maintained by the two-stage vacuum jets located on the roof.

Dissolver, CB-52 (Figure 9)

The dissolver is a 100-gallon (0.379 m^3) 304L stainless steel, agitated atmospheric tank equipped with coils and jacket for

heating or cooling. Feed entering this tank is diverted from the evaporator circulating loop by a three-way metering valve. A magnetic flowmeter is provided to measure the flow rate of the feed stream and instruments are provided to allow this rate to be controlled and varied. Temperature measuring and regulating instrumentation is provided to control steam flow to the coils and jacket. The hot, DMSO/explosives solution in the dissolver is circulated to the crystallizers by Pump CB-12 through two independently controlled flow loops. The overflow from each crystallizer combines at the crystallizer overflow and returns by gravity to the dissolver. Level control in the dissolver is provided by limit switches which allow the level to fluctuate between two predetermined points. When the high level is reached, the air-actuated three-way valve in the crystallizer overflow line diverts the flow from the dissolver to the mother liquor receiver and the level in the dissolver drops. When the low level limit is reached, the three-way valve returns to its original position and the crystallizer overflow continues back to the dissolver. Loss of air supply diverts all flow to the dissolver.

Crystallizers, CC-50 and CC-51 (Figure 10)

Each crystallizer is a 9.5-gallon (0.036 m^3), 304L stainless steel, agitated, atmospheric tank equipped with coils and jackets for cooling. The solvent/explosive solution from the dissolver is independently circulated to both crystallizers by Pump CB-12. Feed rates to the crystallizers are individually controlled by separate control valves and measured by magnetic flowmeters. Instrumentation is provided to allow control and variation of these rates. The combined overflows from the crystallizers are returned to the dissolver or the mother liquor receiver as determined by the level in the dissolver.

Temperature is measured and regulated in each crystallizer by instrumentation which controls the automatic valve in the cooling water supply line. Product is removed through ram-type drain valves centered in the bottom of each crystallizer. The rate of product removal is controlled by manually setting the supply pressure to the valve operators to control the percentage of full open required. Signals to the valve operators are produced by electric, interval timers which control both the frequency of product removal and the volume of slurry removed. Separate timers are provided for each crystallizer with two set points to control the frequency and duration of the open/closed cycles. (Note: During experimentation, capacitance probes were added to each crystallizer timer circuit to allow more precise volumetric product removal).

Mother Liquor Receiver, CD-50 (Figure 11)

The mother liquor receiver is a 50-gallon (0.189 m^3), 304L stainless steel, agitated, atmospheric tank equipped with a tempered water jacket. The tank functions as a receiver for the recycle solvent stream from the decant system. The recycle solvent is held at ambient temperature or can be heated using the tempered water jacket. A high level alarm sounds when the level in the tank reaches approximately 85% of total tank volume. The tank contents are manually transferred periodically for recycle solvent processing or direct recycle.

Product/Mother Liquor Decant System (Figure 11)

The original product mother liquor screen equipped with a 270 mesh (53 μm) screen was replaced with the product/mother liquor decant system. The function of the screener was to receive the product stream from the crystallizers and to perform the initial solids/liquid separation. During initial trial runs constant plugging of the 270 mesh screen forced elimination of the screener altogether. The separation was done, instead, using the decant system. The crude decant system consists of several 2.5 gallon (0.0095 m^3) buckets for receiving the product slurry from the crystallizers. After settling, the liquid is manually decanted to the mother liquor receiver, the product solids are reslurried with water, and the slurry is manually transferred to the first wash screen.

First Wash Screen, CD-02 (Figure 11)

The first wash screen is a single-deck, 18-inch (0.46 m), electrically driven, Sweco vibrating screener of 304L stainless steel construction equipped with one 325 mesh (44 μm) screen and a self-cleaning kit. This screener receives the reslurried solids from the initial solids/liquid separation described above. A spray nozzle is used to provide a water wash to remove residual solvent from the product crystals. Washed solids overflow to the second wash screen (CD-03) and the wash liquor along with any precipitated fines underflows to Filter No. 2 (CD-52).

Second Wash Screen, CD-03 (Figure 11)

The second wash screen was originally a multi-deck, 18-inch (0.46 m), electrically driven, Sweco vibrating screener of 304L stainless steel construction equipped with 8, 50, 120 and 325 mesh (2380, 297, 125 and 44 μm, respectively) screens in a continuous stack. A self-cleaning kit was included on the bottom

screen and a metered filtered water wash was provided via a
spray nozzle. The function of this screener was to perform
a final water wash and physical separation of individual screen
fractions which could later be blended in various ratios to
produce different intermediate explosives products. During
trial runs constant plugging occurred on all the screens forcing
abandonment of the size separation idea. All the screens except
the 325 mesh were removed. The product from CD-02 was simply
washed a second time on CD-03 and then discharged into a 2.5
gallon (0.0095 m^3) polyethylene bucket. This material was then
manually charged into Vacuum Filter No. 3 (CD-53) for final
wash and dewatering.

Vacuum Filters; CD-51, CD-52 and CD-53 (Figure 11)

The three small vacuum filters are 20-inch (0.51 m) diameter,
304L stainless steel, tank-type filters with removable, poly-
ethylene cloth support grids. Each filter has a 38-gallon
(0.144 m^3) filtrate capacity and an 18-gallon (0.068 m^3) solids
capacity. CD-51 and CD-52 are equipped with tempered water
jackets. Removable cloth filter bags are used to collect the
solids. Each filter has an independent vacuum supply control
valve and level indicator.

Vacuum Filter No. 1 (CD-51) was designed to remove product
fines from slurry received from the mother liquor receiver.
Because of growth of crystals on and subsequent blinding of
the filter cloth, this filter was bypassed and this processing
step eliminated.

Vacuum Filter No. 2 (CD-52) receives the first wash water
underflow from the first wash screen which contains the fines
precipitated during the first wash. The cake required some
washing to remove the residual solvent present.

Vacuum Filter No. 3 (CD-53) receives the washed underflow and
overflow streams from the second wash screen. The material
accumulated in this filter provides the bulk of the product
and the filtrate provides the bulk of the first wash water.

Recycle DMSO Receiver, CF-51 (Figure 12)

The recycle DMSO receiver is a 150-gallon (0.568 m^3), 304L
stainless steel, agitated, tank equipped with both cooling
water and tempered water supplies to the jacket along with
temperature indicating instruments. The tank contents are
maintained at a predetermined temperature depending on the
explosive product present and the solvent content. Mother

liquor received from vacuum filter No. 1 (CD-51) or directly from the mother liquor receiver (CD-50) is cooled to precipitate excess dissolved explosives which are allowed to settle in the tank. The cooled solution overflows from the tank through the overflow nozzle provided to the recycle DMSO storage tank (CF-53). Accumulated impure solids are periodically dropped to vacuum filter No. 4 (CF-52) where the solids are collected for disposal or use depending upon the composition.

Product Line Wash Reservoir, CF-54 (Figure 12)

The product line wash reservoir is a 2.5- gallon (0.0095 m^3), inline holding tank used to store recycle DMSO overflowing the recycle DMSO receiver. A manually controlled, air-operated, drop valve is provided to allow the intermittent flushing of the product discharge line from the crystallizers (CC-50 and CC-51) to the decant system.

Vacuum Filter No. 4, CF-52 (Figure 12)

Vacuum Filter No. 4 is a 304L stainless steel filter with a 25-gallon (0.095 m^3) solids capacity and a 75-gallon (0.284 m^3) filtrate capacity. A removable cloth filter bag is used to collect solids from various recycle DMSO streams prior to further processing or disposal. Collected solids material is recycled or handled as waste explosives depending on its purity.

Recycle DMSO Storage Tanks; CF-53, CA-50 and CA-51 (Figures 12 and 13)

The recycle DMSO holding tank (CF-53) is a 150-gallon (0.568 m^3), polyethylene tank which is used to collect the recycle DMSO from either the recycle DMSO receiver or Vacuum Filter No. 4. The tank contains only the working inventory of recycled DMSO. Excess recycle DMSO may be pumped to either DMSO Storage Tank CA-50 or CA-51 or directly to the waste solvent storage tank. CA-50 and CA-51 are 350-gallon (1.32 m^3) agitated, 304L stainless steel tanks which are used for storing various solutions for start-up or shutdown operations. Dilution operations were also performed in these tanks to precipitate excess dissolved explosives prior to pumping to the waste solvent recovery equipment.

Condensate Holding Tank, CE-51 (Figure 14)

The condensate hold tank is a 100-gallon (0.379 m^3), polyethylene, atmospheric tank which is used to collect and store both the rectifying column overhead (distillate) stream and the second

wash water from vacuum filter CD-53. The combined material
is then pumped to the first wash screen and used as first
wash water.

Waste Solvent Recycle Tank, CB-53 (Figure 15)

The waste solvent recycle tank is a 75-gallon (0.284 m^3),
316 stainless steel, unagitated tank equipped with a heating
coil and temperature indicating and control instrumentation.
Filtered waste solvent is pumped as required to this tank and
preheated to the appropriate temperature. The waste solvent
is fed to the rectifying column during recovery operations.

Waste Solvent Storage Tank, CG-50 (Figure 16)

The waste solvent storage tank is a 5,000 gallon (18.9 m^3),
316 stainless steel, agitated tank which is used to store all
waste solvent containing less than 30% DMSO. Water is added
as required to correct the DMSO content to 10-20%. This material
is held for recovery of DMSO or for disposal.

Waste Solvent Filter, CG-51 (Figure 16)

The waste solvent filter is a 304L stainless steel, vacuum
filter with a 175-gallon (0.662 m^3) solids capacity and 300-
gallon (1.13 m^3) filtrate capacity. A removable cloth filter
bag is used to collect solids which will be discarded as waste
explosives. The filtrate contains 10-20% DMSO which is pumped
either to the waste solvent recycle tank for recovery or to a
solvent wagon for processing and/or disposal.

Vacuum Systems (Figures 17 and 18)

Vacuum is maintained on the five vacuum filters via two vacuum
receivers (CJ-50 ana CJ-51) using a set of two, single-stage,
steam ejectors (CJ-10 and CJ-11). Filters 1, 2 and 3 connect
to a common header while filters 4 and 5 share a second header.
Piping is arranged so that either or both receivers can be used
as the vacuum source. Vacuum is maintained on the rectifying
column (CB-21) and the evaporator (CB-51) using a separate two-
stage, intercondensed, steam ejector (CB-13). The pressure of
this vacuum supply system is controlled by an air bleed just ahead
of the main jet inlet. Steam flow to the first and second stage
jet and the water flow to the intercondenser are monitored.

Exhaust System (Figure 19)

Exhaust hoods are provided at all locations where the possibility
of operator contact with solvent fumes exists. The hoods are
located over the slurry mix tank (CA-52), the decant and
screener positions (CD-01, CD-02 and CD-03), Filter No. 1,
Filter No. 2, Filter No. 3 and Filter No. 4. The overflow
header from the crystallizers (CC-50 and CC-51) has a small
crossflow vent located behind the sampling port. The air
mover for the ventilation system is an exhaust fan (CH-10)
located on the building roof.

Pumps

All the pumps used in this process are 304 stainless steel,
centrifugal, slurry pumps equipped with water flushed, stuffing-
box type seals. High temperature shut-off as well as an inter-
lock for seal water failure protection is provided.

Waste Water Holding Tank, CG-52 (Figure 20)

The waste water holding tank is a $10' \times 15'$ ($3.0 m \times 4.6 m$)
stainless steel 8,000-gallon ($30.3 m^3$) storage tank which is
used to collect the excess water generated during the pilot
plant evaluations. This waste water overflows by gravity from
the condensate holding tank (CE-51) and accumulates during
the pilot operation. Disposal of this water depends upon the
concentration of DMSO as monitored by LC analysis. Most of
this stream was discharged during this evaluation.

Hydraulic System (Figure 21)

Two hydraulic oil drive units are used to furnish power to the
hydraulic drive motors for each agitated tank used in the
process. Each agitator has its own variable speed control.
One hydraulic power unit supplies the motors on the crystallizers
(CC-50 and CC-51). The other unit supplies motors on the
slurry mix tank (CA-52), the recycle DMSO receiver tank (CF-51),
the dissolver (CB-52), and the mother liquor receiver (CD-50).

Tempered Water System (Figure 22)

A Pick water heater is used to furnish the 40-50°C (313-323 K)
tempered water for the mother liquor receiver (CD-50), Filter
No. 1 (CD-51) and Filter No. 2 (CD-52). This water heater is
a thermostatically controlled steam/water mixer which supplies
constant temperature water over a variable range of demand con-
ditions dictated by the recrystallization process. Used

tempered water is not recirculated but is discarded after one pass.

Safety and Emergency Systems

Fire Protection

A sprinkler system of the wet-pipe design is provided for all operating areas of Building C-6. Activation of this system is by heat only. No manual activation is currently available. When the sprinkler system is activated, the fire bell is automatically engaged by the flow of water.

Emergency Alarm and Interlock

Paddle switches located at all the Building C-6 exits activate the building emergency alarm as well as the automatic building emergency shutdown sequence. The alarm switch is activated by personnel as they exit the building as soon as they realize that an emergency or uncontrolled dangerous situation exists. Activation of the emergency alarm automatically shuts down critical process equipment including all process pumps, the screeners, the vent fan, and the steam supply to all process tanks. The tank agitators continue to run.

Safety Showers and Eye Wash Stations

Safety showers are located as close to potential sites of personal contact with solvent and explosive solutions as physically possible. Eye wash units are located near the filtering area on the first floor, near the Sweco separators on the lower mezzanine, and near the slurry mix tank. Safety showers are located with the eye wash stations as well as at other strategic locations throughout the building.

Personnel Protective Equipment

Due to the potential for the existence of hazards associated with operator contact with DMSO/explosives solutions which might contain unknown contaminants from the crude explosives, care was taken to contain all DMSO/explosives solutions within the process equipment and to minimize operator exposure as much as possible. In those situations where potential operator exposure could not be avoided, the following additional personnel protective equipment was provided:

1. Face shields (clear plastic, full coverage)
2. Aprons (rubber, covering chest and upper legs)
3. Galoshes (rubber, worn over safety shoes)
4. Gloves (rubber, solvent resistent)

. MATERIALS

Process Materials

Dimethyl Sulfoxide (DMSO - See Appendix D for physical properties)

Crude RDX (Filtered, washed, nutsched)

Crude HMX (Filtered, washed, nutsched)

Analytical Materials

Hexane
Acetonitrile
Dimethyl Formamide (DMF)
Methanol
Chloroform

. PRODUCT REQUIREMENTS AND METHODS OF ANALYSIS

RDX

Attributes	Requirements	HDC Analytical Standard Method
Purity, % RDX	-	I-26+, I-153++
α HMX, %	< 0.01*	Microscope
Acidity, % as Acetic Acid	0.02 Max.	C-9
Melting Point, °C	190 Min.	P-12
Acetone Insolubles, %	0.05 Max.	C-6
Inorganic Insolubles, %	0.03 Max.	C-6
Insoluble Particles		
Retained on USSS No. 40	None	C-4
60	5 Max.	
Impact cm	33+	P-4
Color	White	Visual
Residual DMSO,	-	GC Procedure #33
Granulation	-	P-5

* Internal Control
+ Information Analyses
++ Process Control (C-6)

U.S. Standard Sieve No.	Weight % Passing							
	CLASS							
	1	2	3	4	5	6	7	8
8				100				
12			99-100					
20	96-100							
35		98-100		0-40				100
50	80-100	90-100	30-50				96-100	98-100
60						96-100		
80						91-100		
100	30-90	50-80	10-30				82-98	90-100
120						67-93		
170						43-80		
200	5-45	20-46	0-20				31-61	55-80
230						22-50		
325					97-100	8-36		40-60

HMX

Attributes	Requirements	HDC Analytical Standard Method
Purity, % HMX	Grade A - 93 Min. Grade B - 98 Min.	I-4, I-151+, I-153++
% α HMX	< 0.10*	I-4
% SEX	-	I-151+, I-153++
Polymorphic Form	Beta	I-4
Melting Point, °C	277 Min.	P-12
Acidity, % Acetic Acid	0.02 Max.	C-9
Color	White*	
Insolubles: Acetone, %	0.05 Max.	C-6
Inorganic, %	0.03 Max.	C-6
Insoluble Particles On USSS No. 40 60	0 5 Max.	C-4 C-4
Impact, cm	30 Min.*	P-4
Residual DMSO, %	-	GC Procedure #33

*Internal Control
+Information Analysis
++Process Control (C-6)

Particle Size,   P-5

USSS No.	% Passing for Class					
	1	2	3	4	5	6
8				100		
12			99-100	85-100		99-100
35				10-40		
50	84-96	100	25-55			90-100
100	40-60		10-30	0-15		50-80
120		98-100				
200	14-26		0-20			15-45
325	3-13	75-100			98-100	5-25

Composition A-3

Attributes	Requirements		HDC Analytical
	Minimum	Maximum	Standard Method
% RDX	90.3	91.7	C-36
% Wax	8.3	9.7	C-36
% Moisture	-	0.1	I-7
% Acidity	-	0.02	C-37
Insoluble Particles on			C-4
USSS 40	-	0	
60	-	5	
Granulation, % Passing			
USSS 6	100	-	
100	-	5	P-9
Bulk Density (gm/cc)			P-172
Army Procurement	0.77	-	
Navy Procurement	0.81	-	
Foreign Matter	-	None	P-60

. ## Composition A-5

Attributes	Requirements		HDC Analytical
	Type I	Type II	Standard Method
RDX %	98.5 - 99.0	98.5 - 99.0	C-29
Stearic Acid %	1.0 - 1.5	1.0 - 1.5	C-29
Moisture %	0.10 Max.	0.10 Max.	I-7
Bulk Density, g/ml	0.95 Min.	1.00 Min.	P-8
Insoluble Particles			
on USSS No. 40	None	None	C-4
60	5 Max.	5 Max.	

Comp A-5 (cont'd)

Attributes	Type I	Type II	HDC Analytical Standard Method
Granulation, % Passing			
USSS No. 12	99.0 Min.	99.0 Min.	P-9
200	2.4 Max.	2.4 Max.	

Composition C-4

Attributes	Requirements Minimum	Maximum	HDC Analytical Standard Method
RDX, % Class 1 and 2	99.0	92.0	C-48
Class 3	89.8	91.2	
Class 4	88.9	90.9	
Binder, % Class 1 and 2	8.0	10.0	C-48
Class 3	8.8	10.2	
Class 4	9.0	11.0	
Moisture, %	-	0.25	I-7
Plasticity, Class 1 and 4	-	0.030	P-29
Class 2	-	0.080	
Class 3	0.018	-	
Specific Gravity Class 2 only	1.50	-	P-30
Insoluble Particles on USSS No. 40	-	0	C-4
60	-	5	

Composition B

Attributes	Type I Min.	Max.	Type II Min.	Max.	HDC Analytical Standard Method
% RDX	57.5	61.5	57.5	61.5	C-7
% TNT	37.2	41.8	37.2	41.8	C-7
% Wax	0.7	1.3	0.7	1.3	C-8
% H_2O	-	0.20	-	0.20	C-3
Bulk Density, gm/cc	-	-	-	0.90	P-172
Insoluble Part. Ret'd. on USSS 60	-	5	-	5	C-4
Efflux Vis.,Sec.: Grade A	-	7.0	-	-	P-1
Grade B	-	17.0	-	-	
Granulation: % Pass. USSS No.10	-	100	-		P-9
200	-	-	1.0		

Cyclotol

Attributes	Requirements						HDC Analytical Standard Method
Type	I		II		II		
Class	-		A		B		
	Min.	Max.	Min.	Max.	Min.	Max.	
% RDX	73.0	77.0	68.0	72.0	67.6	71.6	C-2 C-7
% TNT	23.0	27.0	28.0	32.0	by Difference		
% Calcium Silicate	-		-		.35	.65	C-1
Viscosity Efflux Sec.		15.0	-	10.0	-	10.0	P-1
% Moisture		0.25		0.25		0.25	C-3
Insolubles Particles Retained on USSS No. 60		5		5		5	C-4

Octol

Attributes	Requirements						HDC Analytical Standard Method
Type	I		II		II		
Class	-		1		2		
	Min.	Max.	Min.	Max.	Min.	Max.	
% HMX	73.0	77.0	68.0	72.0	68.0	72.0	C-7
% TNT	23.0	27.0	28.0	32.0	28.0	32.0	C-7
Viscosity Efflux Sec.	-	15.0	-	12.0	-	8.0	P-1
Moisture	-	0.25	-	0.25	-	0.25	C-3
Polymorphic Form	-	Beta		Beta		Beta	I-4
Foreign Matter	-	None	-	None	-	None	Visual

	Min.	Max.	Min.	Max.	Min.	Max.	HDC Analytical Standard Method
Insolubles Particles Retained on USSS No. 60		5		5		5	C-4
% Acetone Insoluble	0.10		0.10		0.10		C-6

EXPERIMENTAL PROCEDURE

The DMSO-recrystallization process as originally designed underwent many physical changes throughout the evaluation phase of the project. This evaluation was primarily due to problems which arose and were solved using the most practical and available means possible. The resulting modified processes are described in general terms in Section IV. A. and in more detail in Section IV. B. A more detailed comparison of the modified processes with original processes may be accomplished using Standing Operating Procedure No. 1800-6702 at HSAAP.

General Process Description (Reference Figures 26 and 27)

Feed was prepared in the slurry mix tank by mixing crude explosives, recycle solutions of varying composition, and make-up DMSO. The mixture was pumped to the evaporator feed tank and then metered into the evaporator/rectifier system. DMSO was concentrated in the evaporator by removing water as an overhead stream from the top of the rectifying column. Concentrated DMSO/explosives solution in the evaporator became the feed for the CNCPR crystallization system. The ratio of explosives to DMSO as well as the evaporator conditions were controlled so that the feed solution was maintained at 85-90% DMSO by weight on a solids free basis and contained no undissolved explosives. This feed material could be introduced to the CNCPR system at several points depending on the particular product to be produced. Generally it was metered directly into the crystallizers or tne dissolver. Conditions within the CNCPR system were controlled to cause precipitation of explosive crystals. This was accomplished by water quenching to produce fine PSD's or by simply cooling the feed solution to produce the coarser PSD's. The conditions chosen determined both the particle size distribution and throughput rates obtainable.

Coarse explosives (Classes 1, 3, and 4) produced by cooling techniques were removed from the crystallizers at timed intervals in a thickened slurry containing approximately 40% solids and 60% spent solvent. The liquid was decanted into the mother liquor receiver and held at ambient temperature until transferred for recycle solvent processing or reused directly for making new feed solution. After decanting, the solids were diluted with water to reduce the solvent concentration and to precipitate the remaining dissolved material. Reslurried

product was then fed to the first wash screener and spray washed with a weak (0.5 - 2%) DMSO solution. The underflow fines obtained from the screening operation were collected, filtered, and washed with clean water in a vacuum filter. Filtrate was transferred to the waste solvent storage tank. The bulk of the product crystals overflowed onto the second wash screener and were washed again with clear water. The combined overflow and underflow were collected in another vacuum filter, dewatered and then placed in product containers. The filtrate was combined with the fractionating column distillate in the condensate receiver and used for first wash water. The excess was discarded.

Fine explosives (Class 5) produced by water quenching techniques were removed from the crystallizers in a slurry containing 10 to 20% solids by weight and liquid which contained approximately 20 - 50% DMSO by weight. This slurry was metered directly onto two vibrating screeners equipped with water sprays and 325 mesh (44 μm) screens for scalping oversized particles. The product solids in the underflow were collected in vacuum filters. ashed, dewatered, and placed in product containers. The combined filtrate and wash water were transferred to the waste solvent storage tank.

Recycle DMSO collected in the mother liquor receiver was used directly to make new feed solution or processed as recycle solvent. Material transferred to the recycle DMSO receiver was cooled to 20 - 25°C (293 - 298 K) to precipitate the remaining explosives. The solids were allowed to settle out in the receiver and the liquid overflowed to the recycle DMSO holding tank. The settled solids in the receiver eventually required filtration in filter #4. The filtrate was combined with the receiver overflow in the holding tank where it was stored until required for making new feed. The solids in filter #4 were water washed and stored as waste explosives. The wash water was transferred to the waste solvent storage tank.

Waste solvent generated from the various product washing operations plus the solvent purge generally contained 10 - 30% DMSO and 1 - 2% explosives by weight. Recovery of the DMSO involved filtering the slurry in the waste solvent filter to remove explosives and other solid impurities, fractionating the resulting solution to remove water and other volatile impurities and to concentrate the DMSO, and evaporating the concentrated DMSO as an overhead distillate to free the DMSO of dissolved impurities. The evaporator temperature, the column pressure, and the reflux ratio were controlled to obtain the desired concentration of DMSO in the evaporator and distillate. In general the DMSO was concentrated to 95 - 98% while the distillate contained less than 0.01% DMSO. The concentrated DMSO was collected in storage tanks and then later fed to the evaporator where it was evaporated as an overhead product and collected as 85 - 86% DMSO. (Note: The remaining 14% of this distillate is water which enters the system through the evaporator circulation pump packing gland.)

Detailed Procedure (Reference Figures 26 and 27)

Receiving Explosives

Crude RDX and HMX were individually received at the Building C-6 pilot plant in nutsches. The explosives had been filtered, washed, and dewatered prior to receipt. Upon receipt each nutsche was sampled and analyzed by liquid chromatography (L.C.) for composition and moisture content. This information was used for determining the amounts of explosives to be added to feed batches. The crude explosives were stored in the nutsches until used.

Receiving and Storing DMSO

DMSO was received at the pilot plant in 55-gallon (.208 m^3) drums as packaged at the supplier. Since the drums received from the solvent storage building (Quonset Hut 133) normally contained frozen DMSO, enough was kept at the pilot plant to allow time for thawing. Normally, 5 - 10 drums were received at a time.

Preparing Evaporator Feed

Feed batches were prepared in the slurry mix tank. Crude explosives, a recycle solvent stream, and make-up DMSO were all charged to this tank to produce a desired ratio of explosives to DMSO in the final mixture. The calculations required to determine the ratios of the various streams to each other involved the use of the temperature/solubility relationships presented in graphical form as Figure 23 and Figure 24. Equations were developed based on these relationships and programmed into a Texas Instruments (TI-59) calculator to facilitate the calculations performed at the pilot plant. The equations derived and a typical calculation are presented in Appendix C. In general the steps followed in making a typical feed batch are as follows:

1. Charge 30 - 45 gallons (0.11 - 0.17 m^3) of recycle solvent to the slurry mix tank. While agitating, sample the recycle solvent and analyze for percent explosives, percent DMSO, and % water.

2. Using the analysis of the recycle solvent and the crude explosives moisture content derived previously, perform the mixing calculation to determine the weights of wet explosives and make-up DMSO to be charged.

3. Using a shovel or scoop, transfer wet explosives from a nutsche into a plastic bag or bucket. Using the weigh scales provided, weigh out the proper amount and then charge the explosives to the slurry mix tank.

4. Weigh the make-up DMSO into plastic buckets and charge this to the slurry mix tank.

5. Agitate the mixture until homogeneous.

6. Transfer the mixture to the evaporator feed tank.

The evaporator feed tank was used only for hold-up of the feed mixture prior to feeding to the evaporator. (Although the evaporator feed tank has provisions for heating the feed material, this procedure was discontinued after initial plugging problems were experienced in the evaporator feed system caused by the circulation of the hot, 90 - 100°C (363 - 373 K) material.) The cold feed material was circulated in a continuous pump loop to the evaporator. A take-off for feeding the material to the evaporator was provided at the evaporator.

Concentrating the Solvent in the Feed Solution

Due to the decreasing solvent power of DMSO for both RDX and HMX with increasing water content, the extra water which enters the system with the explosives must be removed. Solvent is concentrated to 85 - 95% DMSO (solids free basis) which is high enough to dissolve all the explosives and to increase the efficiency of the process. Solvent concentration was accomplished using the evaporator/rectifying column system. The evaporator feed material was metered to the evaporator which was operated at 100 - 150 mm Hg (13.33 kPa) and 80 - 90°C (353 - 363 K). The water-rich vapor from the evaporator passed through the rectifying column and became richer in water as it approached the top of the column. The condensed liquid exited the top of the column with only a trace (less than 0.01%) of DMSO detectable. A reflux ratio (L/D) of 1/3 was required to achieve this result.

Crystallizing in the CNCPR System

Many experiments using the CNCPR system and several modifications of the CNCPR system were performed for the purpose of identifying the basic process conditions which would allow production of the required classes of explosives intermediates. After these basic conditions were discovered, system variables

were manipulated in order to optimize the production rate of the particular product within the physical constraints of the equipment. During the initial experimentation, the CNCPR system required several modifications to allow efficient production of the various explosives intermediates. These different systems are presented graphically in Figure 25 and are briefly described as follows:

Basic CNCPR System - The basic CNCPR system, as designed, uses two independent crystallizers and a common dissolver. Saturated or slightly unsaturated 80 - 90°C (353 - 363 K) feed material from the evaporator, which consists of a solution of explosives, DMSO, and water containing no undissolved material, is fed at a controlled rate to the 65 - 75°C (338 - 348 K) dissolver. The dissolver material is continuously circulated to each crystallizer at independently controlled rates. Nucleation and growth takes place in the crystallizers due to the increase in supersaturation which occurs as the warm dissolver solution cools in the crystallizers which are maintained at 35 - 45°C (308 - 318 K). Cooled, saturated solution, containing excess nuclei and fines, overflows from the crystallizers back to the dissolver where it is reheated to the dissolver temperature causing dissolution of the nuclei and fines. The feed rate to the dissolver is controlled to maintain an explosives concentration such that the solution is unsaturated by at least 5°C (5 K). Product crystals are periodically removed from the crystallizers at variable timed intervals. The length of the interval determines the average residence time of crystals in the crystallization environment and thus helps control particle size distribution of the product. The variables besides the particle residence time which most affect the product PSD and throughput rate are the dissolver/crystallizer circulation rate and the dissolver explosives composition.

CNCPR-MOD-1 - The first modification of the basic CNCPR system attempted was CNCPR-MOD-1. The difference from the basic configuration is that instead of feeding evaporator solution to the dissolver, feed material is introduced directly to the crystallizers with the feed point in the crystal bed. This produces a much higher localized supersaturation in the crystallizer and therefore a higher crystal growth rate is potentially possible.

CNCPR-MOD-2 - This modification is similar to CNCPR-MOD-1 in that feed is introduced directly to the crystallizers. However, the dissolver/crystallizer circulation loop is

not used. In this system the production of fines and nuclei is not controlled and therefore the PSD of the product is affected mostly by the feed rate and the crystal residence time.

<u>CNCPR-MOD-3</u> - The distinctive feature of this modification is the purposeful addition of water to the crystallizer. Otherwise, CNCPR-MOD-3 is the same as CNCPR-MOD-2. The addition of the water dilutes the DMSO and reduces its solvent power with a resultant precipitation of many very small particles. The addition of water is controlled to produce a desired final concentration of DMSO in the diluted solvent. The water addition exerts the single highest effect on the product PSD with the feed rate primarily affecting only production rate.

Crystallizing by Dilution

The dilution crystallization experiments performed in the pilot plant basically involved quenching hot DMSO/explosives solutions with cold water to precipitate fine PSD's of explosive crystals. The procedures attempted with RDX are as follows:

1. Hot feed material was added to an agitated crystallizer initially containing a heel of cold water. Feed was continued with agitation until the DMSO concentration in the crystallizer was 40 - 60%. When this point was reached, water addition was begun so that the ratio of water to feed would maintain the 40 - 60% DMSO concentration in the crystallizer. When the level in the crystallizer reached the operating level, continuous product removal was begun at a rate which would maintain the operating level. The temperature in the crystallizer was not controlled for these experiments.

2. Same as "a" except the water heel was initially 70°C (343 K).

3. Hot feed material and cold water were mixed in the crystallizer feed line in a ratio which produced a 45 - 65% DMSO concentration and then the mixture was piped directly to an agitated crystallizer. The batch was then heated to 70°C (343 K) and aged for 10 - 30 minutes (0.600 - 1.800 ks) prior to filtration.

4. Same as "c" except that the batch was not heated to 70°C (343 K) prior to the age.

5. Hot feed material and cold water were mixed in the crystallizer feed line in a ratio whicn produced an initial DMSO concentration of 65%. The batch was accumulated in an agitated crystallizer and aged for 10 - 30 minutes (0.600 - 1.800 ks). Additional water was then added to the crystallizer so that the final concentration of DMSO in the liquid was 45 - 50%. The batch was then dropped to the filtration equipment.

The procedures attempted with HMX are as follows:

1. Hot feed material and cold water were mixed in the crystallizer feed line in ratios which produced an initial liquid concentration of 65 - 80% DMSO. The mixture was accumulated in a crystallizer and agitated for ten minutes. A second addition of water reduced the DMSO concentration to 45 - 55% before filtration of the product fines.

2. Hot feed material was metered into an agitated heel of 95 - 100°C (368 - 373 K) water until the DMSO concentration was 20 - 25%. After a 20 minute (1.200 ks) age at 95°C (368 K), feed material and water were added separately to the crystallizer at a ratio which produced 20 - 25% DMSO in the mixture. The product slurry was continuously removed at a rate which held the crystallizer volume constant. Crystallizer temperature was maintained at 95°C (368 K) throughout the run.

3. Same as "b" except the heel contained beta-HMX seed material.

4. Hot feed material was metered into an agitated heel of 95 - 98°C (368 - 371 K) water containing beta-HMX seed material. The resulting mixture containing 15 - 20% DMSO was aged for thirty minutes at 95°C (368 K) and then cooled rapidly to 50°C (323 K). The batch was then dropped at 50°C (323 K) to filtration equipment.

5. Hot feed material and cold water were simultaneously metered into a agitated heel of cold water containing beta-HMX seed material. The final mixture containing approximately 25% DMSO was heated to 97°C (370 K), aged for 30 minutes (1.800 ks), cooled to 50°C (323 K), and then dropped to the filtration equipment.

6. Hot feed material and cold water were simultaneously metered into an agitated heel of cold water containing beta-HMX seed material. The feed was continued until the final mixture DMSO concentration was approximately 30%. At this point the feeds were stopped and the mixture was heated to 95°C (368 K) and aged for 30 minutes (1.800 ks). After the age period, the feed and water were restarted in a ratio which produced a DMSO concentration of approximately 45%. Product slurry was continuously removed at a rate which held the crystallizer volume constant.

7. Same as "e" except a heel of the material was retained in the crystallizer as seed for the next batch. Up to eight consecutive batches were run and the remaining product was filtered. New series were started by adding new seed material.

8. Same as "e" except no seed material was used.

Decanting Coarse Product Slurry

Coarse explosives were periodically removed from the crystallizer into 2.5 gallon (0.0095 m^3) plastic buckets. Approximately 1.5 - 2 gallons ($0.0057 - 0.0076 \text{ m}^3$) of product slurry were removed during each cycle. The slurry ideally contained 40 - 50% solids by volume and 50 - 60% spent mother liquor. After a brief settling period of 3 - 10 minutes (180 - 600 s) was allowed, the liquor was carefully decanted by pouring it into the mother liquor receiver. The heavier solids which remained in the bucket along with the remaining liquid were diluted and reslurried with enough water so that the slurry could be poured. The material was then manually poured onto the first wash screener.

Washing Coarse Explosives Products

Reslurried, coarse product explosives from the decant were poured onto the first wash screener. The water spray (0.5 - 2% DMSO) used with this screener removed most of the remaining DMSO from the product crystals. Precipitated fines, reslurry water, and wash water which passed through the screen were collected in filter #2. Fines were washed with additional clear water and stored as an impure fines fraction. The filtrate was transferred to the waste solvent storage tank. Coarse product overflow from the first wash screen passed to the second wash screen equipped with another 325 mesh

screen where it was exposed to a clean water spray wash
for removal of most of the remaining DMSO. The combined
overflow and underflow from this screener was collected
in filter #3, given a final clean water wash, dewatered,
sampled and stored as final product. The filtrate con-
taining the wash water (0.5 - 5% DMSO) was transferred to
the condensate holding tank, combined with fractionating
column distillate, and used for first wash water.

Screening, Washing, and Filtering Fine Explosives Products

Fine explosives from either batch or continuous processes
were fed at controlled rates directly to the first wash
screener. A water spray was used to promote efficient
wet screening of the fines through the 325 mesh screen and
to provide a product wash. The overflow from the first
wash screener (if any) passed to the second wash screener
also equipped with a 325 mesh screen and a water spray.
The underflow from each screener was collected in filter
#2 and filter #3, washed again with water, dewatered, and
combined as the final fines product. "Overs" (if any) from
the second wash screen were sampled and then placed in waste
explosive containers for burning. Filtrate from both filters
was transferred to the waste solvent storage tank.

Processing Recycle Solvent

Liquid from the decant process along with excess dissolver
material was collected as mother liquor in the mother liquor
receiver. This material was transferred to the recycle DMSO
receiver for processing or reused directly for making new
feed batches as will be explained later. Processing in the
recycle DMSO receiver involved cooling the liquid as much as
possible to precipitate some of the remaining dissolved
explosives. The precipitated solids were allowed to settle
out in this tank while the bulk of the cold, saturated liquid
overflowed to the recycle DMSO holding tank. This processed
recycle solvent was then reused from the holding tank as
required. The solids which precipitated in the recycle DMSO
receiver were accumulated until removal was required. The
solids were filtered and washed in filter #4.

Purging of solvent to remove impurities other than explosives
was accomplished by transferring part of the processed
recycle DMSO to the waste solvent storage tank where the
remaining explosives were precipitated by dilution and the
weak DMSO solution held for recovery. The purge was automa-
tic since all feed batches made included a percentage of new,
make-up DMSO.

Initially, all the spent solvent which was to be recycled
was processed before reuse. The idea for the processing
was to remove some of the remaining explosives by additional
cooling of the spent crystallizer solvent. The explosives
removed would be a relatively impure fines product since the
minor explosive component would concentrate in this stream.
Because the solubility of both RDX and HMX in DMSO is similar,
the resulting concentration of the minor explosive component
would be approximately 50%. For example, crude RDX contains
approximately 10% HMX as an impurity. During recrystalliza-
tion in DMSO, this HMX remains in solution until its satura-
tion is high enough to allow precipitation. Processing the
spent solvent stream by cooling and decanting (and/or filtra-
tion) removes the HMX component before recycle and thus does
not allow it to build up. Therefore, the RDX product can be
obtained 98 - 100% pure.

Experimental results indicated that the RDX impurity in
crude HMX was normally only present at levels below 3%.
Calculations led to the conclusion that the 15% solvent purge
rate was high enough to keep the level of RDX in the product
well below the desired limits and that processing the recycle
solvent would only lower the overall efficiency of the re-
crystallization process. In the case of RDX, HMX impurities
are not considered detrimental to the performance of the
final products. The only good reason for removing it is if
there is interference with the crystallization process. No
evidence of such interference was discovered; theretore,
most recycle solvent processing was eliminated and the spent
crystallization solvent containing dissolved and precipitated
explosives was reused directly. Processing of material removed
as purge was continued to reduce the loss of explosives as
waste.

Recovering Solvent

Due to cost considerations, separate solvent recovery equip-
ment was not purchased for the pilot plant. Instead, the
evaporator/rectifying column system was designed so that it
could be used on a limited basis to reconcentrate the DMSO
contained in the waste solvent so that it could be reused
for recrystallization.

Weak DMSO solution collected in the waste solvent storage
tank was generated as a result of the solvent purge and the
accumulation of the filtrate from the product wash. The
liquid portion of this material usually contained 10 - 20%
DMSO, 20 - 90% water, and traces of other accumulated

impurities such as acetic acid, soluble nitramines, fly ash and oil. The solids (1 - 2% of the total) were mostly explosives contaminated with fly ash and other insoluble impurities. The waste solvent was filtered to remove the solids and the clear liquid collected as feed for the re-concentration. The DMSO in this stream was concentrated by feeding the weak DMSO solution to the rectifying column using the evaporator as the reboiler. The evaporator was initially filled with feed solution and then the operating pressure, 75 mm Hg (10 kPa), was established in the system. Evaporator temperature was increased to the boiling point of the weak DMSO/water solution and the column was allowed to reach its equilibrium temperature. The reflux control was activated and water and other volatile impurities were removed from the top of the column as distillate. The set-point temperature for the reboiler, 90°C (363 K), corres-ponded to the boiling point of 98 - 99% DMSO. Until the set-point temperature was reached, the reboiler level was held constant by adjusting the feed rate to the column. When equilibrium was established, i.e., the reboiler set-point temperature was reached, the column feed rate was maximized by adjusting it so that the reboiler temperature remained constant. A draw-off of reboiler material was be-gun at a rate which maintained a constant reboiler level. The draw-off material was stored as reconcentrated DMSO. Feed to the column was continued until exhausted. The dis-tillate and bottom draw-off were periodically sampled and analyzed for percent DMSO by liquid chromatography. The 1/3 reflux ratio was the same as that used for feed pre-paration and resulted in a distillate concentration of 0.01% DMSO or less.

Since the reconcentrated DMSO from the fractionation also contained the non-volatile impurities which could not be removed by filtration, the decision was made to recover the DMSO from this stream as a distillate leaving these non-volatile impurities in the still as a sludge. The evaporator/rectifying column system was also used to accom-plish this final distillation. Distillation was begun using the heel of reconcentrated DMSO left from the fractionation. The reflux control was adjusted for "no reflux" so that all the condensate was removed at the top of the column. The conditions used allowed the system to operate at maximum output, i.e., the steam supply to the evaporator was maximum and the pressure was minimum. The feed material was intro-duced to the evaporator at a rate controlled to hold the level constant. Distillate was collected from the top of the column and placed in used DMSO drums. After completing the distillation the sludge left in the evaporator was trans-ferred to the waste solvent storage tank.

RESULTS

Recrystallization of RDX in the CNCPR Systems

The information presented in Table 2 describes the experimental conditions and procedures followed during the evaluation of various CNCPR processes for recrystallizing RDX. Table 3 presents the calculated results of each experiment.

Recrystallization of RDX in the Dilution Crystallizer System

Table 4 describes the experimental conditions and procedures attempted during RDX/dilution crystallization experiments. Table 5 presents the calculated results of these experiments.

Analytical Results of Recrystallized RDX Samples

Tables 6, 7, 8, 9 and 10 present analytical results of tests performed on samples of the recrystallized RDX from the various recrystallization processes. Table 11 presents the results of additional analytical tests performed on selected representative samples from various experiments.

Recrystallization of HMX in the CNCPR Systems

Table 12 describes the experimental conditions and procedures used during the evaluation of various CNCPR processes used to recrystallize HMX. Table 13 presents the calculated results of each experiment.

Recrystallization of HMX in the Dilution Crystallizer System

Table 14 describes the experimental conditions or procedures attempted during HMX/dilution crystallization experiments. Table 15 presents the calculated results of these experiments.

Analytical Results of Recrystallized HMX Samples

Tables 16, 17, 18 and 19 present analytical results of tests performed on samples of the recrystallized HMX from the various recrystallization processes. Table 20 presents the results of additional analytical tests performed on selected representative samples from various experiments.

HMX Recrystallization Process Material Balances

Tables 21 and 22 present material balance information for 15 lb/hr (1.89 g/s) and 30 lb/hr (3.78 g/s) HMX/CNCPR systems,

respectively. Table 23 presents the material balance for a 7 lb/hr (0.88 g/s) HMX/dilution crystallization system. Flow stream numbers refer to Figure 26.

RDX Recrystallization Process Material Balances

Tables 24 and 25 present material balance information for 20 lb/hr (2.52 g/s) and 35 lb/hr (4.41 g/s) RDX/CNCPR systems, respectively. Table 26 presents the material balance for a 28 lb/hr (3.53 g/s) RDX/dilution crystallization system. Table 27 is a material balance for a 45 lb/hr (5.67 g/s) RDX/CNCPR-MOD-3 crystallization system. Flow stream numbers refer to Figure 26.

Solvent Recovery Material Balances

Figure 27 presents the material balance for the pilot plant solvent recovery system.

Process Capability Estimates

Feed Preparation System

Feed for explosives crystallization systems using DMSO can be produced efficiently in a feed preparation system similar to the pilot plant system. The pilot system consists of a mixing tank, an evaporator feed tank, a vacuum evaporator equipped with a rectifying column, condenser and reflux controls. The water entering the process in the crude explosives as well as the pump packing gland water can be effectively removed with very low solvent losses by evaporation and rectification of the vapor with reflux. Water removed from the top of the column can be expected to contain 0.01% (or less) DMSO.

CNCPR Crystallization System

Both RDX and HMX can be crystallized from DMSO using the CNCPR configuration. This system is most effective for producing Classes 3 and 4 explosives PSD's with through-put efficiencies of 65 - 80% and 60 - 80% of theoretical yield for RDX and HMX respectively. Production rate of 15 - 18 lb/hr (1.89 - 2.27 g/s) can be expected for both RDX and HMX produced in a CNCPR system using a single 9.5-gallon (0.036 m^3) crystallizer.

CNCPR-MOD-1 Crystallization System

RDX PSD's ranging from nominal Class 1 RDX (too large for actual Class 1 RDX) to a nominal Class 3 RDX (too large for

actual Class 3 RDX) can be crystallized from DMSO using the CNCPR-MOD-1 configuration. Production rates of 14 - 18 lb/hr (1.76 - 2.27 g/s) per 9.5-gallon (0.036 m^3) crystallizer and efficiencies of 46 - 56% of the theoretical yield can be expected with this system.

HMX PSD's ranging from Class 1 HMX to nominal Class 3 HMX (too large for actual Class 3 HMX) can be crystallized from DMSO using the CNCPR-MOD-1 configuration. Low production rates in the range of only 5 - 10 lb/hr (0.63 - 1.26 g/s) per 9.5-gallon (0.036 m^3) crystallizer and efficiencies averaging 60% of the theoretical yield can be expected.

CNCPR-MOD-2 Crystallization System

RDX PSD's ranging from nominal Class 1 RDX (too large for actual Class 1 RDX) to actual Class 3 RDX can be efficiently crystallized from DMSO using the CNCPR-MOD-2 configuration. Production rates averaging 21 lb/hr (2.65 g/s) per 9.5-gallon (0.036 m^3) crystallizer and efficiencies averaging 52% of the theoretical yield can be expected.

Although HMX was not recrystallized using this system, it is expected that results would show that the same general classes of HMX could be produced as with RDX. However, slightly lower production rates and efficiencies should be expected with HMX compared to RDX.

CNCPR-MOD-3 Crystallization System

RDX Class 1 can be efficiently crystallized from DMSO using the CNCPR-MOD-3 configuration. Production rates from 45 - 50 lb/hr (5.67 - 6.30 g/s) per 9.5-gallon (0.036 m^3) crystallizer and efficiencies of 75 - 80% can be expected.

Although HMX was not crystallized using this system, it is expected that the same general results would be obtained as with RDX.

RDX Dilution Procedure "a"

RDX Class 7 can be efficiently crystallized from DMSO using RDX Dilution Procedure "a". Production rates of 40 - 50 lb/hr (5.04 - 6.30 g/s) per 9.5-gallon (0.036 m^3) crystallizer and efficiencies of 90 - 95% can be expected.

RDX Dilution Crystallization Procedure

Class 5 RDX can be crystallized from DMSO by precipitation from an RDX/DMSO solution using water as the diluent. A batch procedure similar to "RDX Dilution Crystallization Procedure 'e'" can be expected to yield a 95% efficiency (95% of theoretical yield). Production rates of 14 lb/hr (1.76 g/s) of Class 5 RDX per 6-gallon (0.023 m^3) (working volume) crystallizer can be expected.

HMX Dilution Crystallization Procedure

Class 5 HMX can be crystallized from DMSO by precipitation from an HMX/DMSO solution using water as the diluent. A batch procedure similar to "HMX Dilution Crystallization Procedure 'g'" can be expected to yield 70 - 95% efficiency as a percent of theoretical yield). Production rates of 5.1 - 7.0 lb/hr (0.64 - 0.88 g/s) of Class 5 HMX per 7 gallon (0.026 m^3) (working volume) crystallizer can be expected.

Recrystallized Intermediate Product Quality

All RDX crystallized in "cooling - only" systems, i.e. CNCPR, CNCPR-MOD-1, and CNCPR-MOD-2, can be expected to exhibit an improvement in purity over the crude RDX charged. Purities for RDX crystallized this way generally ranged from 97 - 99% RDX compared to the 90 - 93% crude RDX input. The HMX impurity is concentrated in the fines and waste explosives streams. Classes 5 and 7 RDX produced by dilution procedures can be expected to exhibit purities which, in general, are approximately the same as the starting RDX purity. Class 1 RDX produced in the CNCPR-MOD-3 system shows a slight purity enhancement to the 95 - 96% RDX range.

Particle size distributions of all RDX crystallized in any of the CNCPR systems can be expected to be narrower and contain fewer fines than the usual distributions obtained from the evaporative crystallization processes used at HSAAP. Other physical properties of the RDX produced in these systems are comparable to RDX produced by current methods.

All HMX crystallized in "cooling-only" systems can be expected to exhibit purities of 99.5 - 100% HMX compared to the 98 + % crude HMX charged. HMX crystallized by dilution, like the RDX produced this way, can be expected to yield purities which

are approximately the same as the purity of the starting material.

Particle size distributions of HMX crystallized in any of the CNCPR systems are narrower than HMX crystallized using current methods. Other physical properties of HMX crystallized from DMSO are comparable to HMX produced by current methods.

Recrystallized Product Retrieval Techniques

Retrieval of product from the crystallizers can be accomplished by concentrating the crystals in the bottoms of the crystallizers using agitation control followed by the removal of a controlled volume of this thickened slurry on timed intervals. Coarse products can be removed with a high degree of efficiency with very little or no loss of product by way of the crystallizer overflow. The finer PSD's are much harder to concentrate in the crystallizer resulting in lower process efficiencies compared with the coarser product processes. It is expected that agitation improvements and a physically larger crystallizer will improve the effectiveness and efficiency of this product removal technique for all explosives PSD's.

Product Decant, Washing, and Filtration Systems

Decantation of the spent liquor from the solids removed from the crystallizers can be used to provide a very efficient and effective initial solids/liquid separation. Reduction of the solvent content of the solids to an acceptable level can be accomplished by reslurrying the solids with water, feeding the thick slurry to screening equipment, washing the material on the screen using water sprays, and collecting the product solids in filtration equipment for a final water wash. It is expected that use of continuous filtration equipment will improve the solids handling capability of this system and reduce the overall residual solvent content. However, reduction of the solvent content of the product below the levels obtained in the pilot plant using only the same or similar screening and filtration equipment is doubtful.

Solvent Recycle System

Spent solvent from the crystallization can be recovered and recycled directly to the feed preparation system without processing. The buildup of the minor explosive component in this recycle stream does not adversly affect the product purity partly because of the 15 - 20% purge of the recycle stream.

Accumulation of other impurities in the system detrimental to explosives crystallization was not observed during the pilot operation. It is expected that large scale systems will require a purge rate no greater than that used in the pilot plant to achieve purity comparable to the pilot plant levels. Control of the buildup of detrimental impurities will be accomplished as a side benefit of the solvent purge.

Solvent Recovery System

It is expected that a solvent recovery system can be developed based on the recovery procedure carried out in the pilot plant. The first step of the procedure includes, after an initial filtration, an evaporative, vacuum distillation which removes the water as a relatively solvent-free distillate and concentrates the DMSO to 95 - 99%. The distillate from this step also contains trace amounts of other volatile impurities. Non-condensible, volatile impurities are scrubbed out in the vacuum system. The concentrated bottoms stream contains, besides DMSO, all the solids, including explosives. The second step includes as a minimum, the evaporation of the concentrated DMSO from the bottoms stream as an overhead distillate to obtain a purity level as high as 90 - 95% DMSO. This recovered solvent stream can then be used directly as make-up solvent for the recrystallization processes. The evaporator bottoms contain all non-volatile impurities, including explosives. Some of these impurities can be filtered from the stream after precipitation by water dilution.

DISCUSSION

Equipment Performance

With only a few exceptions, most of the mechanical pilot plant equipment performed satisfactorily throughout the evaluation. Some equipment changes were actually made and evaluated during the pilot effort. Other potential improvements in mechanical equipment were simply noted as requiring changes to be evaluated later. Process control instruments also performed well. The process control liquid chromatograph performance was poor. Table 28 provides a listing of items which performed unsatisfactorily or required mechanical or process changes to make them usable or satisfactory. Further discussion of some of the mechanical problems is included in the Process Performance Section.

Process Performance

The basic pilot plant processes, with a few exceptions as noted,

performed very much as designed. Some changes were required,
however, because of mechanical problems which could not be
overcome or because of system interactions as changes made
in one sub-system affected another sub-system. For example,
early experiments with the particle size separation screener
revealed that with the equipment available, product particle
size separation was an impractical approach. Thus, process
modifications were required so that all the product particle
size distributions could be produced by direct crystallization
techniques. This change in the basic project philosophy will
not be discussed since it cannot be evaluated until the original
idea itself is evaluated and compared to the results of the
pilot work actually accomplished. However, the following
discussion will attempt to provide some insight as to the logic
behind the changes made as those processes actually evaluated
in the pilot plant evolved from the original process designs.

Feed Preparation Process

The feed preparation process did not suffer from any major
process design problems. Most of the changes required in
this system were mechanical in nature and are listed in
Table 28. The only process change actually made involved
the feeding of cold rather than hot feed to the evaporator
feed circulating line. Initially, the switch to cold feed
increased the heat load on the evaporator and severely
reduced its capacity. However, reduction of the pump
packing gland water flow was sufficient to allow the
evaporator to function with the capacity required to
support crystallization.

The point must be made that although the feed preparation
functioned well and was able to remove the unneeded extra
water from the process feed stream, there is one major flaw
in the process which will require refinement through further
investigation and experimentation. Because of the "on/off"
nature of the crystallization systems fed by the evaporator,
the evaporator and column were constantly under variable
load (usually either "no-load" or "full-load"). Besides
inefficiency in the column, this caused some fluctuations
in the feed composition which were not detected until the
crystallization system reacted to the particular change.
A possible solution would require the preparation of feed
"batches" while operating the evaporator system batchwise
at maximum efficiency. This would require intermediate
storage of the prepared feed for later use in the crystal-
lization system.

CNCPR Crystallization Process

The crystallization systems themselves as expected, provided the greatest challenge of all the areas of the pilot evaluation. The physical limitations of the crystallizers, i.e., relatively low cooling capacities and poor agitation, combined with the other mechanical problems discovered, produced situations which could only be partially overcome by manipulating the other process variables. The CNCPR system in principle performed very much as expected. However, there were several unanticipated factors which complicated the system and made it difficult to control.

One such factor which was recognized during the early experimentation was the dilution of dissolver material by water entering through the dissolver/crystallizer circulation pump packing gland. At first glance this does not appear to present a major obstacle to the success of the CNCPR crystallization system. It was, however, the root problem which interacted with other minor difficulties and situations to create a very complicated control problem that was never satisfactorily resolved. This interaction will be explained later. The obvious adjustment needed to counteract the constant dilution of the DMSO in the dissolver by the gland water is to increase the DMSO concentration in the feed solution by raising the evaporator temperature. This was done in the pilot plant but, as will be shown, did not provide a complete solution to the problem.

Another unanticipated factor was the inability to obtain reliable dissolver composition analyses capable of providing the necessary feedback for controlling the dissolver saturation. The lag-time between sampling and receiving the analyses was partly responsible. But, mechanical and procedural weaknesses in the liquid chromatograph and the LC procedure itself also contributed to the overall process limitations. Because of this "limited-capability" situation, alternate process control techniques had to be used which relied heavily on calculated material balance information plus visual observations of the dissolver and crystallizer material itself.

The successful production of coarse PSD's in the CNCPR crystallization process as well as the control of the process is dependent on the maintenance of an unsaturated condition in the dissolver. The unsaturated condition ensures that fines and nuclei returning from the crystallizer are dissolved and are not fed back to the crystallizers. Also, the unsaturated feed entering the crystallizer environment ensures that minimum nucleation and maximum growth rates will result. Control of the CNCPR was usually lost as the explosives concentration in the dissolver approached

the saturation point at the existing dissolver temperature and DMSO concentration. The rise in saturation point was not readily detectable by the operators and usually occurred due to a combination of product removal variations in the crystallizers and high feed rate to the dissolver. The usual response of the operators when this situation was discovered was to reduce or stop the feed to the dissolver or crystallizers in order to force the dissolver saturation back into an unsaturated condition. Because of the interaction of the pump packing gland water, this response proved to be too late to prevent upsetting the crystallizers. Because the inflow of packing gland water to the system was constant, the DMSO concentration began to fall when the feed was shut off. This caused precipitation of more fines in the dissolver and a general worsening of the condition. The response of the crystallizers included very fast depletion of the crystal bed and a very marked decrease in the average particle size of the decreasing amount of product removed. Eventually, though, enough material was removed from the crystallizers as product so that the dissolver saturation did drift back into an unsaturated condition. This became apparent to the operators when the crystallizer overflow began to show fewer fines and the dissolver material was free of solids. Also, the quantity and quality of the product removed from the crystallizers began to improve.

When improving conditions were observed, the feed to the dissolver was restarted to regain normal operating conditions. However, as soon as the feed was restarted, the DMSO concentration in the dissolver began to rise resulting in an increasing solubility for explosives and thus a continued decreasing dissolver saturation point. The resulting effect on the crystallizers was again a very fast depletion of the crystal bed although for a different reason. After feed was maintained to the dissolver for a sufficient period of time to allow the DMSO concentration to stabilize, the explosives saturation climbed to a level sufficient to allow the crystal bed to rebuild. Eventually control of the system was reestablished resulting in continued production of good quantities of high quality products.

Detection of the approaching critical dissolver saturation condition by the operators was almost impossible. The first indicator was the presence of a greater than usual number of fines and nuclei in the crystallizers which produced a milky appearance. A decreasing percentage of solids in the product

stream was observed shortly thereafter suggesting depletion of the crystal bed. As stated previously, if the CNCPR conditions deteriorated to this point, it was already too late to reverse the process and save the experiment. The order of the events described above could not then be altered and there was no choice but to follow the outlined procedure to reestablish control.

Reasoning applied to this problem led to the deduction that there existed only two possibilities for improving this situation. First, eliminating the circulating pump gland water altogether would allow quick recovery if control was lost since no dilution of the dissolver contents would occur. Second, improving the capability to detect saturation condition changes in the dissolver would help prevent over-saturation in the dissolver and thus eliminate loss of control itself. Because the existing HDC safety regulations require the use of packing gland flush water for all explosives processing pumps, the first possibility was eliminated. Thus, changes in the process were made to allow dissolver saturation condition changes to be detected before the critical saturation condition was reached. This was accomplished as follows:

1. Only one of the crystallizers at a time was used for crystallizing in the CNCPR system.

2. The second crystallizer was held in idle status with the cooling water off and dissolver material circulating through it.

3. The "saturated" or "unsaturated" condition of the dissolver was monitored visually by the operators by simply noting the presence or absence of cloudy solution or fine crystals in the idle crystallizer.

The processing change described above greatly reduced the incidence of complete loss of control due to oversaturation in the dissolver and allowed much longer runs without the same types of major process upsets. However, refinements of the overall process to provide an even greater degree of process control or to eliminate the control problems will be required before the continuous CNCPR system can be investigated in enough detail to fully characterize or exploit its potential as a coarse explosives crystallization system.

Modified CNCPR Crystallization Processes

Because of the basic control problems with the CNCPR system, various changes in the process configuration were evaluated

in order to simplify the system and thus improve the control over product PSD. CNCPR-MOD-1 provides introduction of feed solution directly to the crystallizers instead of the dissolver. This system experienced the same basic control problems during evaluation as the CNCPR system and the introduction of feed directly to the crystallizers actually produced a detrimental effect on particle growth rates. The quick cooling of the hot saturated feed in the crystallizer environment produced many fines and the resulting median crystal size was relatively small. The extra heat load on the crystallizers reduced the system potential since the resulting crystallizer temperatures were higher than the CNCPR system. The calculated efficiency of this system was high but this is misleading since the efficiency is simply a measure of the fraction of "system" potential obtained. The CNCPR-MOD-1 system offers no real or potential theoretical advantage over the CNCPR system.

CNCPR-MOD-2 is not actually a CNCPR system at all. Instead it is a mixed suspension crystallizer with intermittent product removal. CNCPR-MOD-2 was evaluated primarily for Class 1 RDX explosives production but was also successful in producing Class 3 RDX. The control problems of CNCPR and CNCPR-MOD-1 were entirely eliminated because the dissolver/crystallizer circulation loop was not used. However, the ability to produce the larger Class 4 particle size was also sacrificed. Crystallizer temperature could be maintained lower than either CNCPR or CNCPR-MOD-1 which increased the product rate potential over either system. Good system through-put efficiencies were attained of about the same magnitude as the CNCPR system. Like the CNCPR-MOD-1 system, hot, saturated feed solution introduced directly to the crystallizer environment favors the production of relatively fine crystals. But, because of the absence of fines control capability, more of the smaller crystals are retained in the CNCPR-MOD-2 crystallizer resulting in lower effective growth rates. The simplicity of this system, the high rate potential, and the good system efficiency makes it attractive as a nominal Class 1 or actual Class 3 explosives crystallization system.

CNCPR-MOD-3 is actually a variation of CNCPR-MOD-2. Water is metered into the crystallizer with the feed solution resulting in dilution of the solution and lowering of the DMSO concentration. The dilution effect precipitates additional fines in the crystallizer and causes an effective growth rate even lower than CNCPR-MOD-2. Product is removed intermittently. This system was evaluated for production of actual Class 1 explosives distributions since these could not be achieved by "cooling-only" methods as in CNCPR-MOD-2. This system had a very high production rate, high system potential, and high efficiency for producing Class 1 explosives. As in the CNCPR-MOD-2 system, the simplicity of the CNCPR-MOD-3 system along with the high

rate potential and high system efficiency make it attractive as an actual Class 1 explosives crystallization system.

Another variation of CNCPR-MOD-3 (RDX Dilution Procedure "a") was originally intended for Class 5 RDX production. Instead of reducing the DMSO concentration in the crystallizer by 5 - 10% using a small amount of dilution water as in CNCPR-MOD-3, enough water is added to the crystallizer to reduce the DMSO concentration by 30 - 40%. The product is removed continuously by maintaining a constant level in the crystallizer. Evaluation of this process indicated that it is incapable of producing Class 5 RDX but is effective for producing the Class 7 RDX distribution. A higher system potential than the CNCPR-MOD-3 system and an efficiency comparable to CNCPR-MOD-3 was achieved. The primary disadvantage of this system, as evaluated in the pilot plant, was the relatively high solvent usage rate compared to the other CNCPR systems. However, the solvent usage rate of this process compared to other dilution crystallization systems was significantly lower. Like the CNCPR-MOD-3 system, RDX Dilution Procedure "a" is an attractive process due to its simplicity, high system potential, and high efficiency.

RDX Dilution Processes

The original processes evaluated for production of Class 5 RDX (including RDX Dilution Procedure "a") failed to yield particle size distributions containing enough sub-325 mesh material to be practical. These systems included mixing hot, saturated feed solution and water in an agitated crystallizer vessel and removing the product either continuously or as a batch. Dilution of the feed material to DMSO levels of 30 - 50% produced product PSD's with a very low percentage of sub-325 mesh material. Additionally, dilution below the 50% DMSO level produced large quantities of alpha HMX and very impact sensitive product. A change in the process to allow instantaneous dilution of the feed solution to the desired final DMSO concentration solved the particle size problem and increased the percentage of sub-325 mesh particles to 90 + %. However, the alpha HMX content of the product remained high and the product was very impact sensitive (< 10 cm long impact).

Experimentation with the instantaneous dilution process indicated that dilution of the feed solution to the 65 + % DMSO level followed by a brief age of the precipitated material collected as a batch would convert the alpha HMX to the beta form. Further dilution of this material to any lower DMSO level did not yield any more alpha HMX. Because a significant quantity of RDX was still in solution at the 65% DMSO level, yield improvement was

realized by further dilution of the batch to the 45-50%
DMSO level. The final process (RDX Dilution Procedure "e")
proved to be a very simple process with both high system
potential and high system efficiency.

The primary process limitation with the pilot plant system
was caused by the lack of good agitation in the crystallization
vessels. Because of this problem, the entire volume of the
crystallizers could not be fully utilized. Refinement of
this process and equipment improvements could provide a
system which would have a production rate limited only by
the physical constraints of the equipment, i.e., crystallizer
size, degree of agitation possible, maximum feed rates avail-
able, and product slurry processing method and rate. The pilot
plant process used vibrating screeners to scalp over-sized
material from the product stream. The quantity of this over-
sized material averaged only 2 - 5 % of the batch. Therefore,
screening should not be considered as a necessary part of the
process. Separation of spent solvent and product and the
final product wash can be accomplished with equal or higher
efficiency using simple batch or continuous filtration equip-
ment.

HMX Dilution Processes

A two-step dilution procedure similar to the successful RDX
procedure was attempted using hot, concentrated DMSO/HMX
solutions. This procedure produced impact sensitive HMX
containing large quantities of both the alpha and gamma
polymorphs. Subsequent batch and continuous experiments
carried out in the laboratory[3] and the pilot plant revealed
that dilution of the HMX feed solution in a crystallization
vessel in the presence of fine beta HMX seed material (Class 5
pump-ground HMX, HGF) produced fine beta HMX with 90-100%
of the batch in the sub-325 mesh size range. However, the
impact values of this material were low (19-22 cm) compared
to standard values (30-32 cm). Further experimentation
revealed that the impact values of material produced this
way could be improved by heating the batch to 95°C (378 K)
and aging for 30 minutes (1.800 ks). Impact values of material
treated in this manner showed increases into the normal range
of 28-33 cm.

Other experiments performed without the HGF seed material
always produced mixtures of alpha and gamma HMX during the
initial dilution. Batches produced without seed at any
temperature followed by a heating cycle resulted in conversion
of the gamma HMX in the mixture to alpha HMX. All the batches
produced without beta HGF seed were impact sensitive. The
quantity and size of beta-HMX seed particles used determined

the rate of conversion of the gamma HMX formed during the
dilution to the beta polymorph. The heating cycle and
age period allowed stress relief of the "beta-strain" in
the converted beta crystals and thus reduced the sensitivity.

The successful batch process used to produce most of the
Class 5 HMX was HMX Dilution Procedure "g". The process
began by diluting hot, saturated HMX solution in a crystal-
lization vessel containing a 1.7% slurry heel of water and
beta HGF seed material. After the heating and age cycles,
most of the product material was removed from the crystallizer.
Part of the product slurry, however, was left as the heel
and to provide seed material for the next batch. Several
consecutive batches were produced using this procedure until
the sub-325 mesh fraction was reduced to a level less than
50% of the batch. The crystallizer was then emptied and a
new series started with new beta HGF seed.

As in the Class 5 RDX process, the Class 5 HMX production
rate was limited by the physical constaints of the crystal-
lizers themselves, i.e., poor agitation prevented utilization
of the entire volume of the crystallizer. During the consecutive
production method employed, the observation was made that
each batch of Class 5 HMX contained an increasing quantity of
material larger than 325 mesh. These "overs" decreased the
yield of each consecutive batch until eventually most of
the batch was too large. In general, the percentage of
material from each consecutive batch that was too large
(> 325 mesh) and had to be screened out as "overs" is as
follows:

Batch No.	% Overs
1	0-4
2	0-15
3	20-25
4	28-48
5	30-60
6	40-70
7	> 50
8	> 70

The Class 5 HMX process also used vibrating screeners to scalp
over-sized material from the product stream. Since initial
batches and the first consecutive batch yielded a high percentage
of sub-325 mesh material, screening should also not be considered
a necessary part of this process. Refinements of this process
using more suitable equipment could be made to provide a very
high yield, efficient Class 5 HMX process.

Product/Solvent Separation, Screening, Washing and Filtration Processes and Recycle Solvent Processing

The overall performance of the original process for separating the product from spent solvent followed by washing and separating the product into size fractions was poor. The poor process performance was primarily due to poor equipment performance. The initial separation of spent solvent from product was to be accomplished using a vibrating screener. Severe blinding of the original 270 mesh screen used and the 120 mesh screen used later forced early abondonment of this idea. A crude decanting process was substituted which proved to be very efficient.

Following the product/solvent separation, another screener was used to provide a primary wash. Blinding of the first screener caused overflow of spent solvent with the product to the 1st wash screener and eventual blinding of the screen in this unit, also. Use of the decant procedure eliminated this problem.

The final screener in the series was originally equipped with a stack of four screens for separating the product into four distinct size groups. The separation process was to have provided a broad capability with several advantages. The crystallization systems would not be required to provide exact particle size distributions of explosives meeting existing specifications for the various classes of explosives. The particular size fractions chosen could be recombined in ratios to produce these exact distributions if required. This would allow optimization of a system of multiple, independent crystallizers for the most efficient production of recrystallized explosives without regard to the production of specific distributions. Process variables would be manipulated to produce specific ratios of coarse to fines which could be separated and then recombined to meet the total plant demand for those individual products. Additionally, the system would have the ability to produce bimodel distributions directly instead of requiring the blending of other products. A much more consistent and repeatable PSD of the final products produced by blending specific particle size fractions would also be realized.

The screener intended to perform the separation and allow evaluation of this idea did not function. Each screen in the stack became clogged with explosives in such a way that retained product would not discharge. This was attributed to a poor screen-to-screen flow pattern and insufficient water flow to each screen. A better design of the screening equipment or a different arrangement of the same type of

equipment could possibly salvage the basic elements of this idea. However, it could not be evaluated during this pilot plant effort.

The performance of the screeners in providing product washing capability was satisfactory. Water spray nozzles distributed wash water over the product during its retention on the screen. Removal of a sufficient percentage of the fine particles (sub-325 mesh), which were precipitated during the reslurry of the product after decanting, was accomplished in the first wash screener. Since in most cases these precipitated fines contained a high percentage of the minor explosive component as an impurity, removal of this material improved the purity of the final product.

Filtration equipment used to filter explosives from relatively concentrated DMSO/explosives mixtures containing dissolved explosives produced severe filter cloth blinding and extremely long filtration times. The mother liquor filter, which was the first step in recycle solvent processing, was unable to maintain a filtration rate high enough to filter all the mother liquor produced. Therefore, this initial recycle solvent processing step was eliminated after only a few initial experiments. Mother liquor was transferred, instead, directly to the recycle DMSO receiver and held until filtered in the recycle DMSO filter. Although very slow filtration rates were experienced in this filter, also due to filter cloth blinding, the larger filter size provided enough capacity to process all the recycle solvent. Analyses of the filter cake, however, revealed that the purity level of the solids removed was as high in most instances as the input crude explosives. Therefore, recycle solvent processing was eventually discontinued altogether in favor of direct recycle of mother liquor. Direct recycle proved to greatly simplify the solvent recycle and purge and decreased the percentage of explosives lost as waste.

The modified processes used to handle the recrystallized product and recycle solvent, although different from the original processes, performed satisfactorily. Design of an effective decanting system for DMSO/explosives slurries will be required but should provide an efficient, dependable initial separation technique. Improvement of the screening process will be required if the original product PSD separation idea is to be incorporated into overall recrystallized explosive product scheme. If direct crystallization processes are to be used, continuous filters such as the Bird-Pannevis or Eimco designs would offer advantages over screening equipment to perform product washing functions.

Solvent Recovery Process

The solvent recovery process was not part of the original
pilot plant design. The original intention was to use
the evaporator/rectifier system to demonstrate that a simple
process could be used to reconcentrate the DMSO from the
waste solvent streams to a level which would allow its
reuse for crystallization. The reconcentration was also
to demonstrate that this procedure could be performed with
a minimum of solvent loss and that no problems would result
due to the presence of small quantities of explosives. Because
of the ease with which the reconcentration was accomplished and
the absence of any major problems, the decision was made to
carry the process one more step to recover the DMSO from the
concentrate by vacuum distillation to free the DMSO from the
explosives and any other non-volatile impurities. The resulting
distillate contained most of the DMSO and was free of any
detectable impurities other than water.

The apparent relative simplicity and efficiency of this
procedure carried out in equipment not designed for this purpose
implies that a system based on this procedure could be easily
designed to perform this function on a larger scale. Use of
the pilot plant data thus far obtained would facilitate this
design effort.

Process Material Balances

Crystallization Processes

Figure 26 is the flow diagram for the basic crystallization
processes evaluated in the pilot plant. Tables 21 through
27 present the material balance information corresponding
to Figure 26 for several of the recrystallization processes
previously discussed. The values used for flow rates and
compositions are average values based on analyses of samples
taken from the different process streams during the evaluation
phase. Internal streams in some cases bear calculated values
which may or may not represent actual situations observed
during evaluations. Instead, these values represent those
which would be expected using the results actually observed
in the product output streams with known input streams. Since
these values represent ideal situations, care should be
exercised if this information is to be used to design a
larger facility. More rigorous evaluation of the overall
system should be considered after the refinements are made
in the various sub-systems as discussed in the previous
section. Economic considerations expressed in this report,
however, are based on the material balances presented.

Solvent Recovery Process

Figure 27 is the flow diagram/material balance for the
pilot plant solvent recovery process. Rates and compositions
again reflect the average values observed through repeated
experimental runs. Internal stream rates and compositions
in some cases were calculated and represent those values
which would produce the output results actually observed
given the input rate and composition.

Experimental Results

RDX/CNCPR Experiments

The experiments conducted with the RDX/CNCPR systems are
described in Table 2, calculated results of these experiments
are presented in Table 4, and analytical results of samples
tested are presented in Tables 6, 7, 8, and 11. Brief analyses
of these experiments are as follows:

1. CNCPR-MOD-1 (Experiments 1 - 7) - Seven experiments were
   conducted using the two-crystallizer, CNCPR-MOD-1 flow
   configuration. Feed was introduced near the bottoms of
   both crystallizers in an attempt to provide high, localized
   supersaturation levels in the crystal bed where the largest
   crystals were. Most of these experiments were plagued by
   problems associated with poor dissolver saturation control
   described previously. Loss of control occurred frequently,
   as exhibited in Table 2, when the dissolver saturation
   temperature, T_s, exceeded the dissolver operating temperature,
   T_D. The particle size distribution of the product showed
   a great deal of variation corresponding to these upsets.

   The product removal cycle time provided very little control
   over product PSD except during the "in control" situations.
   The products from these experiments were for the most part
   nominal Class 3 RDX PSD. However, some actual Class 3 RDX
   was produced as well as actual Class 4 RDX. The Class 4
   RDX was all produced during experiment #4. Dissolver
   control had been lost and was being reestablished by
   allowing the system to drift without feed. Due to this
   "no-feed" situation, the system was actually a CNCPR
   system rather than a CNCPR-MOD-1 system. After control
   was regained and the feed was restarted, the product PSD
   drifted back into the Class 3 domain. This result demon-
   strates that the introduction of a highly saturated feed
   directly to the crystallizer produces a negative particle
   growth effect compared to normal CNCPR dynamics. There is
   also evidence that periods of increasing dissolver saturation

produce higher growth rates than periods of decreasing dissolver saturation.

The effect of dissolver saturation on product particle size is shown graphically in Figure 28. Figure 28 also shows the extremely erratic nature of the dissolver saturation and emphasizes the difficult control problem that existed. Note that the dissolver saturation seemed to have critical limits which if exceeded had detrimental effects on the product particle size. When the value of (T_D-T_S) approached zero or drifted into the negative region, the dissolver solution became saturated and the dissolver lost its ability to control the fines returning from the crystallizers. If (T_D-T_S) was <u>highly</u> negative, the dissolver itself even became a fines producer. Also, if (T_D-T_S) increased above $15^\circ C$ (288 K), the average product PSD decreased suggesting that growth rates were low and that nucleation effects dominated the production.

Also shown in Figure 28 is the effect of crystallizer feed supersaturation on the resulting product PSD. The crystallizer supersaturation is the difference between the dissolver saturation temperature, T_S, and the crystallizer operating temperature, T_C. (T_S-T_C) values as high as $40^\circ C$ (313 K) and as low as $10^\circ C$ (283 K) were experienced. Flunctuations in (T_S-T_C) contributed to the fluctuations noted in the product PSD but did not have as large a controlling effect as (T_D-T_S). However, when very low (T_S-T_C) values are experienced, as shown at the 32 - 36 hour (115.2 - 129.6 ks) and the 52 - 56 hour (187.2 - 201.6 ks) periods in Figure 28, (T_S-T_C) becomes the controlling factor and greatly depresses the apparent particle growth while nucleation and fines production remain fairly constant. Thus, a much smaller product PSD results.

The results of CNCPR-MOD-1 experiments, presented in Table 3, show that system production rates of 11.86 - 35.17 lb/hr (1.494 - 4.431 g/s) were achieved with corresponding system efficiencies of 45.8 - 67.7% of theoretical yield. The solvent usage rates ranged from 1.19 to 1.94 lb of DMSO/lb of product (1.19 to 1.94 kg of DMSO/kg of product). Note that the highest efficiency was achieved during the production of the Class 4 product during experiment Number 4. This can be attributed to lower loss of solids and dissolved material in the crystallizer overflow. Better settling of the larger, heavier particles in the crystallizers combined with a lower average dissolver saturation produced this effect.

2. CNCPR-MOD-2 (Experiments 8 - 13) - Six experiments were performed using the single-crystallizer, CNCPR-MOD-2 flow configuration. These experiments, which were designed primarily to produce Class 1 RDX PSD's, used several combinations of feed composition (28.4 to 32% RDX), feed rate (0.3 to 0.5 GPM (19 to 32 cm^3/s)), feed point (4 to 12 inches (0.1 to 0.3 m) from the crystallizer bottom) and product removal cycles (7, 10, and 20 minutes (0.420, 0.600, and 1.200 ks)). However, most of the product was nominal Class 1 too large for the actual Class 1 PSD. None of the controlled variables had any major effect on product PSD although there were several trends noted as follows:

   a. Raising the feed tube from the bottom of the crystal-lizer toward the crystallizer overflow reduced the product particle size.

   b. Increasing the feed rate reduced the product particle size.

   c. Changes in feed composition had no measurable effect.

   d. Increasing the product removal cycle time increased the product particle size. (The 20-minute (1.200 ks) cycles produced actual Class 3 RDX.)

   The results of the CNCPR-MOD-2 experiments, presented in Table 3, show that system production rates of 8.88 - 33.82 lb/hr (1.119 - 4.261 g/s) were achieved with corresponding system efficiencies from 32.1 - 79.8% of theoretical yield. The solvent usage rates ranged from 1.84 to 3.96 lb of DMSO/lb of product (1.84 to 3.96 kg of DMSO/kg of product). Due to the presence of the very many small particles, agitation was very critical. Too high agitation rates caused destruction of the crystal bed resulting in a homogeneous slurry in the crystallizer. During occasional upsets of this nature, a large amount of product was lost by way of the crystallizer overflow thus reducing both the production rate and the system efficiency. Constant readjustment of the agitator speed was required to properly maintain the crystal bed.

3. CNCPR-MOD-3 (Experiment 14) - One experiment using the CNCPR-MOD-3 flow configuration was performed. The conditions which resulted in the minimum PSD's in the CNCPR-MOD-2 experiments plus water dilution were used to produce the actual Class 1 PSD. The results of this experiment, presented in Table 3, show that a very high production rate of 48.72 lb/hr

(6.139 g/s), a system efficiency of 78.6%, and a solvent usage rate of only 1.18 lb of DMSO/lb of product (1.18 kg of DMSO/kg of product) were achieved. Careful control of agitation was maintained throughout the run and was the major factor responsible for the high production rate and efficiency.

4. CNCPR Experiment Number 15 - One experiment was performed using the basic CNCPR flow configuration using only one crystallizer. The idle crystallizer was used to visually monitor dissolver solution as previously discussed. The purpose of this experiment was to produce Class 4 RDX. Experience gained during the earlier CNCPR-MOD-1 experiments was used in attempts to provide greater control over dissolver saturation and thus prevent the negative values of (T_S-T_D) which contributed to the poor particle growth results obtained previously. The results of this experiment, presented in Figure 29, again show the effect of dissolver saturation on the product particle size. Although dissolver saturation control was successful and the (T_D-T_S) values were positive throughout most of the run, the product particle size was small indicating that the particle growth rates were again fairly low. However, Figure 29 indicates that the (T_S-T_C) values fell during the ititial part of the experiment and remained low during most of the run. The depressing effect of these low values on crystal growth rates is believed responsible for the small size of the product obtained.

    The results of the CNCPR experiment are presented in Table 3, These results show a production rate of 15.38 lb/hr (1.938 g/s) at 80% efficiency as well as a solvent usage rate of only 1.08 lb of DMSO/lb of product (1.08 kg of DMSO/kg of product).

5. CNCPR Products - No unusual qualities were discovered in any of the RDX intermediate products recrystallized from DMSO using the CNCPR process. Alpha HMX content was monitored by infra red and microscopic analysis. The analytical data presented in Tables 6, 7, and 8 show that occasional traces of alpha HMX were detected in some samples; but, these amounts were in all cases judged by microscopic analysis to be within acceptable limits less than 0.01% by weight. There were never, however, any impact sensitivity problems. Since this would not have been the case if alpha HMX contamination had been substantial, it is believed accurate to assume that no polymorph problems were experienced.

RDX/Dilution Experiments

The experimental conditions used to produce fine RDX by dilution are described in Table 4, calculated results of these experiments are presented in Table 5 and analytical results of samples tested are shown in Tables, 9, 10, and 11.

A total of two continuous and twelve batch experiments were performed in attempts to produce Class 5 RDX. The alpha HMX contamination problem, which occurred during the initial continuous experiments, as well as the discovery that growth rates were too high in the continuous system to allow production of the Class 5 PSD, led to the adoption of the successful batch process. The successful conditions used included an initial water dilution of the hot, DMSO/RDX solution by plug flow mixing in the crystallizer feed line, aging the mixture with agitation at ambient mixture temperatures, a second water dilution in the crystallizer, and filtration and washing to recover the product. The continuous product removal version of this process was successful in producing only Class 7 RDX.

The results of the RDX/Dilution experiments presented in Table 5 show that the final RDX Class 5 procedure using the pilot plant equipment was capable of producing 14 lb/hr (1.76 g/s) of product per crystallizer at efficiencies of 95%. Most of the product losses occurred during the filtration and washing steps. Impact sensitivity problems due to alpha HMX contamination were eliminated due to the aging step following the initial dilution. This is evident by observing the consistently high impact sensitivity values obtained on products from Experiments 9 through 14. Analytical results presented in Tables 9, 10, and 11 show that the Class 5 RDX produced meets all specifications for recrystallized RDX. No unusual properties were discovered as a result of any of the tests performed.

HMX/CNCPR Experiments

Experiments performed in the HMX/CNCPR systems are described in Table 12, calculated results of these experiments are presented in Table 13, and analytical results of samples tested are presented in Tables 16, 17, 18, and 20. Brief analyses of the experiments are as follows:

1. CNCPR-MOD-1 (Experiments 1 - 6) - Six experiments were performed using the two-crystallizer, CNCPR-MOD-1 flow configuration. Feed was introduced near the bottom of each crystallizer to provide high localized supersaturation

and high growth rates in the vicinity of the largest crystals. Better control of the dissolver supersaturation level was maintained during these experiments than during the corresponding RDX experiments. However, as shown in Figure 30, the saturation level was actually maintained too low to produce the desired high growth rates required to produce Class 4 HMX using the 20-minute (1,200 ks) product removal cycle. Also, the direct introduction of feed to the crystallizers, as was noted during the earlier RDX experiments, appears to increase the production of nuclei and fines resulting in depressed effective growth rates in the crystallizers.

Figure 30 also shows less erratic dissolver saturation variations than in the RDX experiments and therefore the corresponding changes in the product particle size distributions were not as drastic. However, the low dissolver saturation values resulted in low (T_S-T_C) values which depressed the growth rates and contributed to the failure to produce the desired larger particle size distributions.

The results of the CNCPR-MOD-1 experiments, presented in Table 13, show that system production rates of 6.75 - 20.85 lb/hr (0.851 - 2.627 g/s) were achieved with corresponding system efficiencies of 28.8 - 86.8% of theoretical yield. The solvent usage rates ranged from only 0.55 to 2.18 lb DMSO/lb of product (0.55 to 2.18 kg DMSO/kg of product). The most favorable conditions occurred at the beginning of the run when the values of dissolver saturation and (T_S-T_C) were high while (T_D-T_S) values were positive.

2. CNCPR (Experiments 7 - 11) - Five experiments were performed using the CNCPR flow configuration with only one operating crystallizer. The idle crystallizer was used as a visual reference of the dissolver saturation condition as in the HMX/CNCPR-MOD-1 experiments previously described. Figures 31, 32 and 33 show that the same general results were obtained during these experiments as with the other experiments, i.e., the use of the idle crystallizer was sufficient to keep the (T_D-T_S) values positive but the variations in both (T_D-T_S) and (T_S-T_C) caused corresponding variations in the product particle size distribution. However, the proper conditions were established and held long enough during part of the experiments to provide the growth and fines control necessary to produce the Class 4 PSD. These conditions included a combination of sustained high (T_S-T_C) values, sustained positive (T_D-T_S) values,

and 30-minute (1,800 ks) product withdrawal cycles.

Table 13 shows that system production rates of 14.69 - 17.13 lb/hr (1.851 - 2.158 g/s) were achieved with corresponding system efficiencies of 36.6 - 80.8% of theoretical yield. The solvent usage rates ranged rom 0.65 to 1.40 lb DMSO/lb of product (0.65 to 1.40 kg DMSO/kg of product).

3. CNCPR Products - Analytical data presented in Tables 16, 17, 18 and 20 show that all the DMSO-recrystallized HMX from the CNCPR process meet the specifications for recrystallized HMX. One of the samples tested for vacuum thermal stability (VTS) at 100°C (373.15 K) for 48 hours (172.8 ks) showed an atypical result of 0.10 cc/g. Moisture content of the test sample could be responsible for this result. All other test results were normal. No other unusual properties or qualities which would suggest chemical or physical interactions between DMSO and HMX were discovered during the performance of the tests.

HMX/Dilution Experiments

The experimental conditions used to produce Class 5 HMX by dilution are described in Table 14, calculated results of these experiments are presented in Table 15, and analytical results are shown in Table 19 and 20.

Sixteen batch experiments were performed in attempts to produce Class 5 HMX. Initial HMX experiments were based on the successful RDX procedures. Plug flow mixing to achieve the desired particle size and two-step dilution for polymorph control were attempted. These batches, however, were all impact sensitive and the material was predominantly gamma HMX. Seeding the dilution water with beta HMX seed from the regular Holston HGF production (pump-ground) process, even at very low concentrations, provided the needed polymorph control. The very small particle size required was easily obtained without plug flow mixing because of the very low growth rates of the HMX in the diluted solutions. This result could possibly be due to the relatively slow polymorph conversion observed. Leaving a heel of fine beta HMX seed for the next batch reduced the overall requirement for initial HGF seed. However, after a few consecutive batches, an obvious growth of material took place and reduction in process efficiency resulted.

The results of the HMX/Dilution experiments, presented in Table 15, show that the batch pilot Class 5 HMX procedure was capable of producing 5.1 - 7.0 lb/hr (0.64 - 0.88 g/s)

of product per crystallizer at total efficiencies of
70 - 95%. Reductions in production rate were experienced
as a result of the heel seeding method since a greater
percentage of product was lost as "overs" from the screening
process. 95 - 100% efficiency could be achieved if only
one or two consecutive batches were produced this way.
Other small product losses were attributed to the filtration
and washing steps. Alpha contamination and impact problems
were encountered primarily during the first 12 experiments.
However, definite improvement was shown during the final 4
experiments with most of products exhibiting impact values
above 30 cm. The 30-minute, $95^{\circ}C$ heating cycle used to
obtain the impact value improvement is believed to relieve
"beta strain" which occurs during solid phase conversion of
gamma HMX crystals to beta. Aging at $95^{\circ}C$ (368 K) allows
enough molecular mobility for realignment of the HMX molecules
into a more stable crystal structure.

Analytical results of Class 5 HMX samples tested at Holston
are presented in Tables 19 and 20. The samples representing
the pilot production process are H11191300.CD52/3S through
H11200500.CD52/3S. All specifications for Class 5 HMX were
met on each of these samples. Additional tests revealed that
one sample, when tested for vacuum thermal stability ($100^{\circ}C$ for 48
hr), showed an atypical value of 0.33 cc/g. High sample moisture
due to incomplete drying of the test sample is suspected to have
caused this result. No other unusual physical or chemical
properties were observed.

Residual Solvent in DMSO-Recrystallized Explosives

Previous concern was expressed that the quantities of residual
DMSO that remain with the intermediate explosives after recrystal-
lization and final washing could cause potential problems when
using these materials in final products. Subsequent limited
testing by other agencies has indicated that there might be some
compatibility problems, but, these must be dealt with individually.
Due to concerns of this nature, care was taken during the design
of the DMSO-recrystallization pilot equipment to provide sufficient
washing to reduce the total residual levels of DMSO in the products
as low as practical. The solvent levels obtained are, therefore,
representative of what would generally be expected for most DMSO
processes.

Residual DMSO test results presented in Tables 11 and 20 were
determined by gas chromatography. Two analyses were performed
on each sample. One analysis determined the total residual DMSO
content of the wet sample just as it left the washing step of
the recrystallization process. Another analysis determined the
occluded DMSO content after the sample was oven dried to remove

the surface moisture. Occluded DMSO averaged 0.03% and 0.08% by weight in tests of all the RDX and HMX samples, respectively. Total residual DMSO in the same samples averaged 0.35% and 0.41%. These values are not entirely representative since in all cases the finer intermediates (actual Classes 1, 5, and 7) were harder to wash and dewater than the coarser intermediates (nominal Class 1 and Classes 3 and 4). Thus, the finer material contained higher total residual moisture with higher levels of DMSO than the coarser material. The total residual solvent in the coarse RDX alone averaged only 0.16% while in the fines it averaged 0.64%. Comparable values for HMX were 0.25% DMSO in the coarse and 0.75% in the fines.

Final Incorporated Products from DMSO-Recrystallized Explosives

Tables 29 and 30 present analytical results of the final products made using DMSO-recrystallized intermediate explosives. These products, which include Compositions A-3, A-5, C-4, B, 70/30 Cyclotol, and 70/30 and 75/25 Octol, meet all applicable military specifications and will be used for qualification tests. No manufacturing problems were encountered which could be attributed to the chemical properties of the residual DMSO or the recrystallized explosives themselves. Viscosity specifications on the 70/30 Cyclotol and 75/25 Octol could not be achieved using standard incorporation techniques. The difficulties encountered were at least partly attributed to the narrow particle size distribution and relatively small average size of the coarse explosives (Class 4) compared to regular HSAAP Production material. However, a modified incorporation procedure, which was developed during the 1973 DMSO pilot effort, was successfully applied to the problem and the required amounts of "in-specification" test products were produced.

Solvent Recovery Capability

Since the development or evaluation of a solvent recovery process was not included in the project scope of work, no formal investigation was performed in this area. However, due to the accumulation of relatively large quantities of 10-30% DMSO/water solutions, reclaiming the DMSO in a form suitable for reuse was necessary. The available pilot plant equipment, i.e., the evaporator/rectifier system, was used for this purpose. During this crude recovery operation several observations were made which may prove helpful for designing a DMSO recovery process. These observations include the following:

1. No explosives migration by entrainment was detected by visual inspection of the internals of the rectifying column or condensor.

2. Concentration of the 10 - 30% DMSO to 95 - 99% DMSO is possible by single step fractionation.

3. The concentration of DMSO in the distillate from the concentration step can be maintained at less than 0.01% DMSO by weight using a reflux ratio (L/D) of 1/3.

4. DMSO free of solid or liquid impurities other than water and possible trace quantities of acetic acid can be obtained by vacuum distillation of the concentrated DMSO. A sludge draw-off from the distillation contains all the non-volatile impurities present in the waste solvent feed.

Since the objective of the solvent recovery operation was not to perform a detailed solvent recovery study, but simply to recover solvent from the waste stream, no efforts were made to optimize the system through process variable manipulations or to determine what the system limitations were. Thus, additional work will be required to determine the most efficient conditions for solvent recovery with respect to minimum reflux ratio, optimum temperature and pressure conditions, the number of fractionators required (two are used in the Crown Zellerbach process), and the operating limits of the system. Another study should characterize the sludge from the evaporation step and determine the sludge draw-off rate required to control potential chemical interactions between the various impurity components and the requirements for disposition of this material.

Particle Size Separation/Recombination

The particle size separation/recombination idea could not be evaluated during this pilot plant evaluation because of the unsatisfactory performance of the size separation equipment. The direct recrystallization of the individual classes of explosives, which was not in the original scope of the project, became the new project objective after the separation system failed. This new project objective was met. Because the theoretical advantages of size separation/recombination have not yet been evaluated, the idea cannot be completely dismissed. However, the following facts should be considered before a final decision is made:

1. Individual processes are now available which can provide all the desired products without the additional processing required for separation and recombination.

2. Individual recrystallization processes are more efficient since the demand for particular products is relatively

independent of the demand for other products. (Size separation/recombination would produce variable mixes of all products).

3. Redesign and evaluation of separation equipment for performing the size separation would be required before the actual capability and benefits of size separation/recombination could be determined.

CNCPR Improvement

The lack of control over product particle size which was experienced during the evaluation of the continuous pilot CNCPR system was due to interactions between several controlled variables within the system. Due to the complexity of these interactions, it is felt that changes in the existing system controls will not result in sufficient improvement to provide a reliable process. Therefore, an alternate process configuration designed to eliminate these control problems should be considered for production of coarse explosives, particularly Class 4. The modified configuration will incorporate the best features of the previously demonstrated batch CNCPR technology with the successful features of the continuous CNCPR system to produce a semi-continuously fed, intermittent product removal CNCPR system.

ECONOMIC ANALYSIS

Recrystallized HMX

The approximate incurred costs for recrystallizing HMX by conventional methods to produce Classes 1 and 4 HMX and for recrystallizing, pump grinding, and classifying Class 5 HMX are shown in Attachment 1. Actual "out-of-pocket" unit costs of utilities used during the first quarter 1980, the 1980 HDC labor estimating rates, and the labor, material, and overhead values charged against various RDX and HMX products during June, 1980 are presented in Attachment 2. Assumptions made for estimating the costs of conventional HMX recrystallization include the following:

1. Utilities usages were calculated by performing energy balances on existing processes using current operating procedures and operating logs as bases. Costs were calculated by applying the "out-of-pocket" unit costs from Attachment 2.

2. Steam usage includes a 10% additional for miscellaneous but does not include building heat or ventilation.

3. Electrical usage does not include lighting or other miscellaneous usage.

4. The total of direct labor, materials, and overhead costs applied to recrystallization is the difference between the cost accumulated through the recrystallization step minus the cost accumulated through the filtration/washing step (represented by the value of crude explosives) at current production rates.

The estimated incurred costs for recrystallizing Classes 1, 4, and 5 HMX from DMSO in a hypothetical 864,000 lb/month (0.151 kg/s) recrystallization facility are presented in Attachments 3, 4, and 5, respectively. The assumptions used for these estimates include the following:

1. The hypothetical facility produces a maximum of 432,000 lb/month (0.076 kg/s) of nominal Class 1 HMX, 216,000 lb/month (0.038 kg/s) of Classes 3 or 4 HMX and, 216,000 lb/month (0.038 kg/s) of Class 5 HMX. These maximum rates are the bases for the calculated operating costs.

2. Solvent recovery facilities are included in the overall facility.

3. Utilities and materials usages are based on material and energy balances performed using the flowsheet presented in Appendix B, Figures 26 and 27.

4. Utilities costs were calculated by applying the "out-of-pocket" unit costs from Attachment 2.

5. Labor requirements are based on a total facility requirement of 5 men/shift, 3 shifts/day, and 7 days/week.

6. Labor rates are 1980 estimating rates for HMX recrystallization from Attachment 2.

7. Steam usage includes a 10% additional for miscellaneous but does not include building heat or ventilation.

8. Electrical usage does not include lighting or other miscellaneous usage.

Comparison of the calculated unit recrystallization costs of the selected products at the production rates chosen shows that DMSO recrystallization is less expensive than conventional processing. Because of physical and operational differences between the two systems being compared, ie., batch versus continuous systems, the unit operating cost differences depend to a great extent upon the production rates chosen for the comparison. In general, the unit costs of utilities and materials are independent of the production rates in either system and remain fairly constant. However, the unit labor cost is much more sensitive to production rate, especially

for the hypothetical DMSO facility. In order to provide a more meaningful comparison, the effect of production rate on the unit recrystallization costs must be analyzed and taken into account.

Because of the continuous nature of the DMSO facility, most of the system equipment must be operated at all times regardless of the production rate. For most operating rates a full operator crew is required and the total operator requirement at half rate is essentially the same as at full rate. Consequently, the unit operating labor cost will double at half the 864,000 lb/month maximum production rate. At very low rates, however, the labor requirement can be reduced by at least one operator since several of the operating phases such as solvent recovery and Class 5 recrystallization can be run intermittently instead of simply turning down to a lower rate. By contrast, the unit labor cost of conventionally recrystallized products is less sensitive to production rate changes since each recrystallization system is independent. At low production rates, only those systems actually needed are operated and thus operating labor is reduced accordingly. For purposes of this analysis, the production rate sensitivity of conventional processes is estimated to be approximately 15% (unit labor cost is 15% higher at 76,500 lb/month than at 612,000 lb/month).

The effects of production rate changes on the total operating costs of both the conventional and the hypothetical DMSO facilities described above are illustrated in Attachment 6. Note that the difference in the unit recrystallization costs of the two systems decreases rapidly as the production rate decreases. At approximately the 150,000 lb/month (0.026 kg/s) level, the DMSO facility loses its operating cost advantage. However, at very high rates a large advantage is apparent. Over the current normal operating range, the recrystallization cost advantage varies from $0.12 to $0.18 per pound. (Note: The costs compared to determine this cost advantage include only the direct charges incurred during recrystallization and are not total product costs.)

In considering other advantages of the DMSO facility over conventional facilities, it should be noted that three separate conventional facilities would be required to recrystallize the quantity possible in the hypothetical DMSO facility. Also, a 2 to 3 million pound per month (0.350 to 0.525 kg/s) DMSO facility is just as feasible as the 864,000 lb/month (0.151 kg/s) because of the relatively small size of the equipment required. Thus, a DMSO facility would provide a very high mobilization potential not presently possible with existing systems.

Recrystallized RDX

Actual calculations for comparing recrystallization costs for RDX by conventional versus DMSO methods were not undertaken for the following reasons:

1. Large scale RDX recrystallization from DMSO such as nominal Class 1 RDX for Composition B, is not considered practical due to the quantity, size, and cost of the equipment required and building load limit problems anticipated.

2. Large scale production of coarse RDX for coated compositions, such as nominal Class 3 RDX for Composition A-5, is not considered economical because of the extra processing steps required and thus the higher operating costs implied.

3. Previous estimates[2] comparing DMSO-recrystallization with conventional recrystallization projected only a "break-even" comparison for coarse RDX products. This projection is still considered valid.

4. Since the DMSO-recrystallized Class 5 RDX process is similar to the Class 5 HMX process, the same relative operating expense is anticipated for equivalent production rates. Thus, the cost of DMSO-recrystallized Class 5 RDX can be projected using the Class 5 HMX cost calculations as a basis.

In considering DMSO based facilities for production of nominal Classes 1 or 3 RDX for Compositions B, A-3, and A-5, it was noted that mobilization rate quantities of each of these products would have to be considered for comparison with facilities using conventional processes. A 7.5 million pound per month (1.313 kg/s) Composition B line would require a 4.5 million pound per month (0.787 kg/s) nominal Class 1 recrystallization facility. A 4.1 million pound per month (0.717 kg/s) A-Products line would require approximately a 3.8 million pound per month (0.665 kg/s) recrystallization facility. There are several problems associated with using DMSO technology for systems requiring rates of this magnitude. With cyclohexanone systems, approximately eight, 5,000-gallon (22.7 m^3) crystallizer-stills are required to recrystallize 4.5 million pounds per month (0.787 kg/s) of nominal Class 1 RDX. Since the recrystallization cycle ends with the recrystallized RDX in a hot water slurry, coating to produce any of the A-Products is easily accomplished. Also, pumps can be used to transfer the recrystallized RDX slurry to other buildings for any other end use. The load limits in such a facility could be maintained within acceptable limits by staging the batches.

DMSO facilities would require only three 5,000-gallon (22.7 m^3) crystallizers to recrystallize the 4.5 million pounds per month (0.787 kg/s) of RDX. However, the recrystallized product does not end up in a water slurry but must be reslurried if it is to be pumped or coated. This creates a minimum of one additional processing step which implies higher cost, at least for A-Products. In addition to the three crystallizers, two continuous filters

(one for dewatering incoming crude RDX and one for removing solvent from and washing the recrystallized product), an evaporator system, a product decant/recycle solvent handling system, a solvent recovery system (most likely a separate facility), and miscellaneous feed systems and controls would be required.

- Preliminary building layout of a DMSO-recrystallization process of the magnitude required within the existing recrystallization building floor plan results in an extremely overcrowded situation. The quantity of equipment needed demands a high personnel requirement. With only a "break-even" cost projection for DMSO-recrystallized RDX compared with conventional RDX, the additional capital expense would not be recoverable. As previously discussed, lowered production rates would not necessarily reduce the personnel requirements and would, therefore, additionally increase the unit cost of the product. In the case of the A-Products, where additional processing steps are required, it is doubtful that even a "break-even" cost situation would actually exist.

The load limits of the DMSO/RDX facility would also present some problems in that explosives involved in several of the processing steps, such as filtering and decanting, would have to be classified as Class 1.1. Also, due to the continuous nature of the feed preparation and product washing processes, even at reduced rates most of the equipment would be full most of the time and the explosives load could not be staged to reduce the explosives quantity for load limit purposes. In summary, for all production situations considered, DMSO processes for manufacturing the high volume RDX products mentioned appear neither practical nor economical compared with conventional processes.

Conventional methods for producing Class 5 RDX involve water quenching RDX/acetone solutions to precipitate the fine RDX crystals. DMSO procedures are similar but the batch sizes could be increased using the same crystallizer vessels by a factor of approximately four. The cost of recrystallizing Class 5 RDX using a DMSO process is expected to be approximately the same as Class 5 HMX (approximately $0.24/lb product). The cost of labor, materials, and overhead alone for the conventional Class 5 RDX is $0.4693/lb product (see Attachment 2). Thus, a favorable cost advantage with a factor of approximately two is expected for Class 5 RDX recrystallized using the DMSO process.

Attachment 1: Incurred Recrystallization and/or Grinding-Classification Costs of Conventionally Prepared HMX

Utilities	Class 1 HMX Batch Size = 850 lb Batch Time = 7 hr Usage	Cost	Class 4 HMX Batch Size = 1,200 lb Batch Time = 10 hr Usage	Cost	Class 5 HMX Batch Size = 3,000 lb Batch Time = 14 hr Usage	Cost
Steam						
1. Dissolving	2,247 lb	$ 4.9746	3,600	$ 7.9700	—	—
2. Seed Production	—	—	5,000	11.0695	—	—
3. Distillation	6,779 lb	15.0080	15,000	33.2085	—	—
4. Miscellaneous	903 lb	1.9992	2,400	5.3134	—	—
5. Total Steam	9,929 lb	$21.9818	26,000	$57.5614	0	0
Filtered Water	—	—	8,250 gal	$ 0.7004	1,800 gal	$0.1528
River Water	46,000 gal	$ 0.7498	72,000 gal	$ 1.1736	2,100 gal	$0.0342
Electricity	52.5 kw hr	$ 1.3353	74.6 kw hr	$ 1.9083	300 kw hr	$7.6740
Total Utilities Cost ($)	—	$24.0669	—	$61.3437	—	$7.8610
Unit Cost of Utilities ($/lb Product)	—	$ 0.0283	—	$ 0.0511	—	$0.0026
Labor and Materials Unit Cost ($/lb Product)	—	$ 0.2915	—	$ 0.3706	—	$0.7701
Total Recrystallization	—	$ 0.3198	—	$ 0.4217	—	$0.7727

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Utilities: 1st Quarter 1980 (Out-of-Pocket)

City Water	–	$0.4523/1000 gal
Filtered Water	–	$0.0849/1000 gal
River Water	–	$0.0163/1000 gal
Steam	–	$2.2139/1000 lb
Electricity	–	$25.5800/1000 kw hr
Air	–	$0.1056/1000 ft^3

Labor Rates for 1980:

Dept. 223 (HMX Recrystallization) - $20.04/hr; Labor and Benefits

Dept. 207 (RDX Manufacturing) - $22.12/hr; Labor and Benefits

Labor and Materials Costs:

DMSO - $0.62/lb (Crown Zellerbach)

* RDX ($/lb)	Labor	Materials	Overhead	Total
Crude	0.1197	0.1713	0.1272	0.4182
Class 1	0.1758	0.1764	0.1593	0.5115
**Class 3	0.3101	0.1458	0.1578	0.6137
Class 4	0.2448	0.1831	0.1995	0.6274
Class 5	0.3765	0.2277	0.2833	0.8875

*HMX ($/lb)				
Crude	0.4479	0.4331	0.6109	1.4919
Class 1	0.6701	0.5024	0.7077	1.8802
Class 4-2	0.7472	0.5094	0.7329	1.9895
Class 5	1.1715	0.4796	0.9291	2.5802

*Data from Contract Pricing Proposal No. 1585 for January – December, 1980.
**Actual for May 1980

Attachment 3: Cost of Recrystallizing Class 1 HMX Using
Hypothetical DMSO Facility; 432,000 lb/mo.

	Feed Prep.	Cryst.	Recy. Solv. Proc.	Prod. Filt. & Wash	Solvent Recovery	Miscl.	Unit Product Cost ($/lb Product)
Utilities							
Steam, lb/min	16.95	-	-	-	65.47	4.12	0.01916
Filtered Water, gpm	0.45	0.07	0.93	3.00	0.20	0.23	0.00004
River Water, gpm	73.80	47.32	-	-	391.46	25.63	0.00088
Electricity, Kwhr/mo.	30,461	4,300	4,801	24,597	26,927	4,550	0.00566
Labor, Man hr/day							
Crystallization	9.0	14.4	3.6	9.0	-	8.0	0.06123
Solvent Recovery	-	-	-	-	8.0	3.0	0.01531
Materials							
Solvent, lb/min	-	-	0.01	0.01	0.35	0.02	0.02418
Totals* ($/lb Product)	0.01821	0.02037	0.00592	0.01463	0.04956	0.01777	0.12646
Totals** ($/lb Product)	0.01859	0.02037	0.00592	0.01463	0.04497	0.01772	0.12220

*20% Solvent Purge Rate
**10% Solvent Purge Rate

Attachment 4: Cost of Recrystallizing Classes 3 or 4 HMX
Using Hypothetical DMSO FAcility; 216,000 lb/mo

Utilities	Feed Prep.	Cryst.	Recy. Solv. Proc.	Prod. Filt. & Wash	Solvent Recovery	Miscl.	Unit Product Cost ($/lb Product)
Steam, lb/min	8.50	7.13	-	-	33.43	2.45	0.02281
Filtered Water, gpm	0.23	0.20	0.45	1.50	0.10	0.12	0.00004
River Water, gpm	35.66	104.34	-	-	199.92	17.00	0.00116
Electricity, Kwhr/mo.	15,231	6,450	2,423	24,482	13,751	3,117	0.00775
Labor, Man hr/day							
Cystallization	4.5	7.2	1.8	4.5	-	4.0	0.06123
Solvent Recovery	-	-	-	-	4.0	1.0	0.01392
Materials							
Solvent, lb/min	-	-	0.01	-	0.18	0.01	0.02480
Totals* ($/lb Product)	0.01821	0.02430	0.00654	0.01545	0.05054	0.01667	0.13171
Totals** ($/lb Product)	0.01860	0.02430	0.00654	0.01545	0.04521	0.01661	0.12671

*20% Solvent Purge Rate
**10% Solvent Purge Rate

Attachment 5: Cost of Recrystallizing Class 5 HMX
Using Hypothetical DMSO Facility; 216,000 lb/mo

	Feed Prep.	Cryst.	Recy. Solv. Proc.	Prod. Filt. & Wash	Solvent Recovery	Miscl.	Unit Product Cost ($/lb Product)
Utilities							
Steam, lb/min	1.35	-	-	-	101.32	5.13	0.04773
Filtered Water, gpm	0.45	1.60	-	5.00	0.30	0.37	0.00013
River Water, gpm	-	-	-	-	605.75	30.29	0.00207
Electricity, Kwhr/mo.	10,213	2,150	-	21,930	41,667	3,798	0.00945
Labor, Man hr/day							
Crystallization	4.5	7.2	-	6.3	~	4.0	0.06123
Solvent Recovery	-	-	-	-	12.0	4.0	0.04453
Materials							
Solvent, lb/min	-	-	-	0.08	0.42	0.03	0.06572
Totals* ($/lb Product)	0.01434	0.02032	0	0.03014	0.13725	0.02881	0.23086
Totals** ($/lb Product)	0.01434	0.02032	0	0.03014	0.14621	0.02877	0.23978

*20% Solvent Purge Rate
**10% Solvent Purge Rate

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Attachment 6: Recrystallization Cost Comparison for Existing
Versus DMSO Recrystallization of HMX
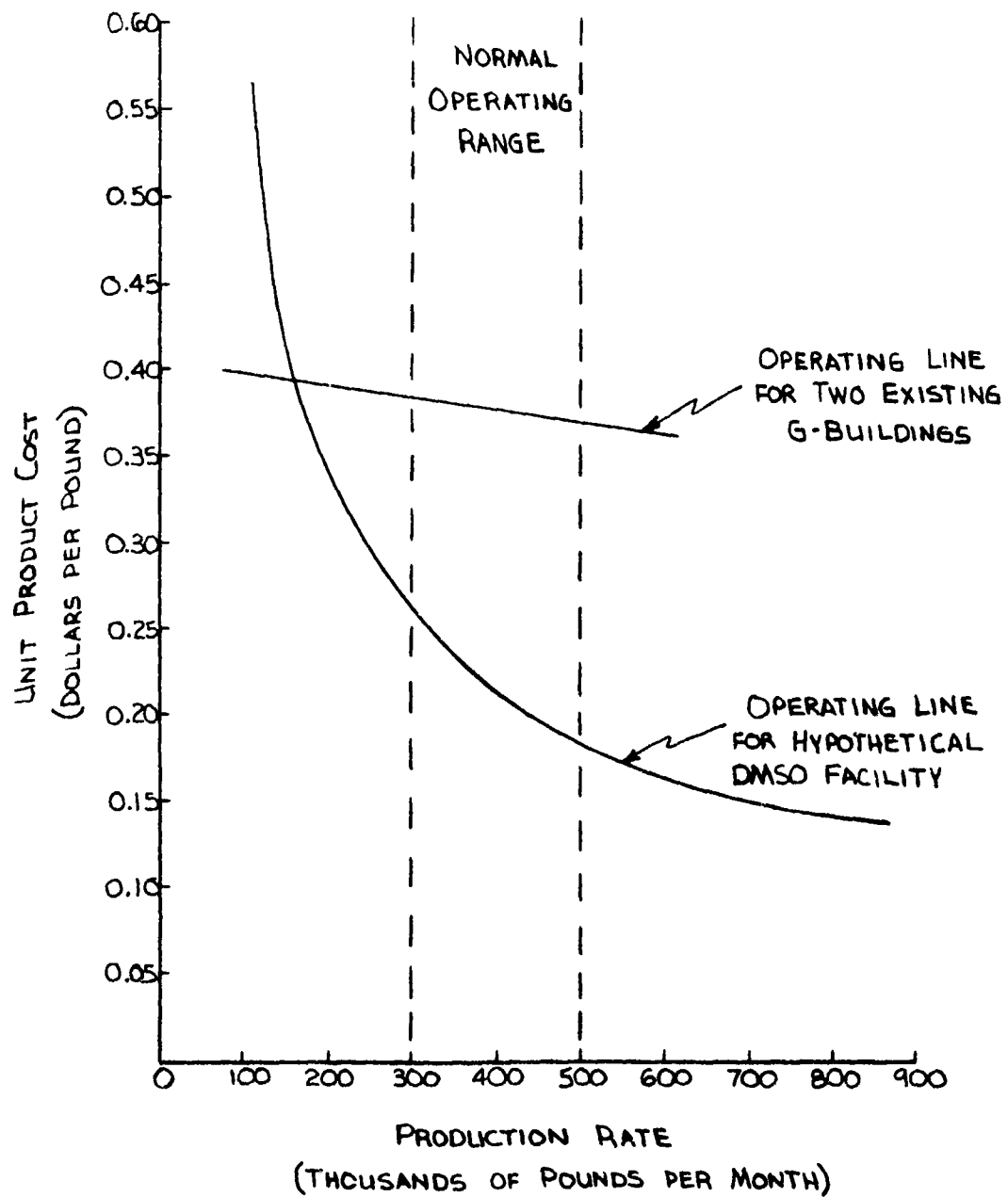
CONCLUSIONS

1. Feed solutions for RDX or HMX crystallization systems using DMSO as the crystallization solvent can be efficiently generated using a feed preparation system similar to the pilot plant system. The prepared feed solution, containing no undissolved solids, can be used directly as feed for crystallization systems.

2. The continuous CNCPR crystallization system can be used to crystallize both RDX and HMX from DMSO. Although the production of the coarse explosives distributions, Classes 3 and 4, was demonstrated, the capability of the continuous CNCPR system to consistently and repeatably produce the coarse distributions was limited by poor dissolver saturation control. The CNCPR-MOD-1 system, a variation of the CNCPR system, caused increased fines production and thus additionally reduced the effectiveness of this system to produce the coarse distributions.

3. The continuous CNCPR-MOD-2 crystallization system can be used to crystallize explosives particle size distributions ranging from nominal Class 1 to actual Class 3. Pilot plant data are considered sufficient for sizing a prototype crystallization unit for this purpose.

4. The continuous CNCPR-MOD-3 crystallization system can be used to crystallize actual Class 1 RDX and HMX particle size distributions from DMSO. Direct scale-up of this system to prototype scale is considered possible using the pilot plant data generated. A modification of the CNCPR-MOD-3 process which allows more dilution and continuous rather than intermittent product removal can be used to produce Class 7 RDX.

5. A batch, RDX dilution crystallization procedure is required for producing Class 5 RDX containing the stable beta polymorph of HMX. Sizing of equipment for a prototype Class 5 RDX production unit is considered possible using the existing pilot plant data. Good agitation during the aging period and a good product wash are considered of primary importance to a successful future design.

6. A batch, HMX dilution crystallization procedure is required for producing Class 5 HMX. The dilution must take place in the presence of fine beta HMX (HGF) and a thermal age period must follow to obtain the stable, beta HMX polymorph. Sizing of larger scale equipment for production of Class 5 HMX with this procedure is considered possible using existing pilot plant data. Good agitation and product washing are essential to a successful future design.

7. The RDX and HMX crystallized from DMSO meet all existing specifications for these products. Standard test results were comparable to RDX and HMX recrystallized by conventional methods.

8. A decant system is considered a desirable alternative to the original product/solvent separation technique which used a vibrating screener. The crude decant system used during the pilot evaluation performed the initial separation with good efficiency.

9. Screening equipment can be used to provide satisfactory initial and final product wash of the coarser products from the CNCPR systems.

10. Purity analyses of the various recrystallized RDX and HMX products indicate that processing the spent crystallization solvent by cooling and filtration to remove an impure explosives stream prior to recycle is not necessary. The build-up of the minor explosive component in the RDX or HMX processes can be effectively controlled by selection of an appropriate solvent purge rate. The present pilot plant data are considered adequate for sizing a recycle solvent handling system for a prototype production unit.

11. DMSO can be recovered from dilute water solutions which contain dissolved and undissolved solid and liquid impurities to obtain high purity solvent suitable for reuse. Although the present pilot plant data would be useful in designing a DMSO recovery system, it is not considered adequate for scale-up.

12. The direct recrystallization techniques developed in the pilot plant for recrystallization of individual classes of explosives are considered acceptable alternatives to the "multiclass explosives" idea using the size separation/recombination technique which failed due to unsatisfactory equipment performance.

13. The DMSO-recrystallized RDX from the pilot plant can be used as input materials for the manufacture of Composition A-3, Composition A-5, Composition C-4, Composition B and 70/30 Cyclotol. The resulting final products meet all specifications for these products.

14. The DMSO-recrystallized HMX from the pilot plant can be used as input materials for the manufacture of 70/30 and 75/25 Octol. The resulting incorporated products meet all specifications for these products.

15. Economic evaluation of hypothetical DMSO-recrystallization facilities indicate that DMSO processes can reduce the recrystallization cost of all HMX products at production rates higher than 150,000 pounds per month. The DMSO-recrystallization costs of the relatively large quantities of nominal Classes 1 and 3 RDX required for Composition B and A-Products are projected to be approximately the same as conventional processes. Class 5 RDX costs can also be reduced using the DMSO process. A lower recrystallization cost advantage results for both DMSO-recrystallized RDX and HMX compared with conventional methods if the DMSO facilities are operated at reduced capacity.

RECOMMENDATIONS

1. The following minimum efforts should be accomplished in support of any further DMSO recrystallization project work:

   a. All mechanical and general process improvements cited in this report relative to the existing pilot plant, including the design and installation of a product decant system, should be implemented. Individual and collective evaluation of the improvements should be completed before additional products are manufactured. Since most of these improvements will require redesign, modification, or purchase of additional capital items, additional project funding should be provided.

   b. Efforts should be made at HSAAP concurrent with Recommendation No. 1.a. to further evaluate the present analytical capabilities with respect to liquid chromatographic characterization of solutions containing RDX, HMX, DMSO, and water. If the present capabilities are determined to be unacceptable, improvements in the LC techniques or instruments should be pursued or alternate methods proposed which will provide the acceptable analytical capabilities required to more accurately evaluate the DMSO-recrystallization systems.

   c. Qualification tests of products from the current pilot plant evaluation should be completed as soon as possible. Results should be made available for review by HDC and HSAAP personnel as soon as the tests are completed in order to determine if additional improvements in the DMSO-recrystallization processes will be adequate to provide acceptable products.

   d. A study should be initiated to identify any accumulated impurities in the recycle and waste solvent streams generated during the pilot evaluations. Edgewood Arsenal should perform the appropriate toxicity studies of the impurities identified so that the potential hazards associated with the proposed recrystallization processes can be determined and evaluated.

2. After successful completion of Recommendation 1.a. and initiation of Recommendations 1.b. - 1.d., the pilot plant should be operated as a semi-works facility to provide additional test quantities of DMSO-recrystallized explosives as needed. These products could be provided as test material in the support of the development of new products or applications. Efforts should be made to solicit customer ideas and orders for these products.

3. Design and evaluation of a pilot DMSO recovery process should be accomplished to provide sufficient information for design and scale-up to prototype.

4. If future RDX and HMX production requirements justify prototype or
    production facilities, the following processes are recommended:

    (a) Batch cooling with dilution capability for producing actual and
        nominal Class 1 RDX and HMX, actual Class 5 RDX and HMX, and
        actual Class 7 RDX.

    (b) Batch CNCPR for producing actual and nominal Class 3 RDX and HMX
        and actual Class 4 RDX and HMX.

    (c) Continuous cooling with dilution capability for producing actual
        and nominal Classes 1 and 3 RDX and HMX and actual Class 7
        RDX.

5. Since demonstrated DMSO crystallization technology is available for
    efficient preparation of most individual classes of RDX and HMX, it
    is recommended that additional evaluation or use of the original
    continuous CNCPR concept (which includes particle size separation/
    recombination) for simultaneous production of all classes of explo-
    sives be abandoned unless justified by specific production need and/or
    economics.

6. Due to the complexity of parameter interactions within the continuous
    CNCPR system, it is recommended that no additional effort be expended
    to improve the existing continuous CNCPR process for production of
    coarse explosives.

7. If justified by economics and specific production needs for products
    requiring coarse explosives, is recommended that a semi-continuous
    CNCPR system, which is based on the combined successful features of
    the batch and continuous CNCPR systems, be evaluated in the existing
    pilot plant. The relatively minor modification recommended, which
    should eliminate the need for strict control of dissolver saturation,
    involves batch feeding of the dissolver to produce a semi-continuously
    fed, intermittent product removal system.

8. Based on the present economics and pending qualification of DMSO-
    recrystallized products from the pilot plant, it is recommended that
    decisions regarding future facilities projects for recrystallization
    of all classes of HMX or Class 5 RDX include consideration of DMSO
    processes as alternatives to conventional recrystallization process.

REFERENCES

1. HDC Technical Report, HDC-PE-15, Phase I, "Recrystallization and Growth of HMX-RDX, A Study of Methods and Equipment", D. M. Mahaffey, 1972.
2. HDC Engineering Report, HDC-19-74, "Recrystallization and Growth of HMX-RDX, A Study of Methods and Equipment, Phase II", M. D. Rothrock, 1974.
3. HDC Technical Report, 79-0088, "Precipitation of HMX Fines from DMSO", B. A. Hunter and F. D. Pilgrim, 1980.
4. HDC Progress Report, 20-P-26, "Physical and Chemical Properties of RDX and HMX", J. T. Rogers, 1962.
5. Treybal, R. E., "Mass Transfer Operations", 2nd Edition, McGraw-Hill Book Co., New York, 1968.
6. Crown Zellerback, Chemical Products Division Publications, "Dimethyl Sulfoxide, Recovery from Aqueous Solutions", Dimethyl Sulfoxide as a Reaction Solvent" and "Dimethyl Sulfoxide, Summary of Toxicological and Safety Information".
7. Military Specificatons, RDX, MIL-R-398C.
8. Military Specifications, HMX, MIL-H-45444A.
9. HDC Development Report, "Incorporations of DMSO-Recrystallized RDX and HMX to Make Cyclotols and Octols", R. E. Dawson, 1974.
10. McCabe, W. L. and J. C. Smith, "Unit Operations of Chemical Engineering", 2nd Edition, McGraw-Hill Book Co., New York, 1967.
11. Perry, R. H., C. H. Chilton, and S. D. Kirkpatrick, "Chemical Engineers' Handbook", 4th Edition, McGraw-Hill Book Co., New York, 1963.
12. HDC Technical Report, 77-0057, "Hazards Analysis of the DMSO Recrystallization Pilot Plant at Building C-6", S. A. Carmack, 1977.

APPENDIX A

TABLES

Table 1:   Equipment List

Equipment Name	Code Number
Slurry Mix Tank	CA-52
Evaporator Feed Tank	CB-50
Evaporator	CB-51
Rectifying Column	CB-21
Condenser	CB-22
Dissolver	CB-52
Crystallizers	CC-50, CC-51
Mother Liquor Receiver	CD-50
Product/Mother Liquor Screen	CD-01
First Wash Screen	CD-02
Second Wash Screen	CD-03
Vacuum Filter No. 1	CD-51
Vacuum Filter No. 2	CD-52
Vacuum Filter No. 3	CD-53
Recycle DMSO Receiver	CF-51
Product Line Wash Reservoir	CF-53
Vacuum Filter No. 4	CF-52
Recycle DMSO Storage Tanks	CA-50, CA-51
Recycle DMSO Holding Tank	CF-53
Condensate Receiver	CE-50
Condensate Holding Tank	CE-51
Waste Solvent Recycle Tank	CB-53
Waste Solvent Storage Tank	CG-50
Waste Solvent Filter	CG-51
Primary Steam Jets	CJ-10, CJ-11
Vacuum Receivers	CF-50, CJ-51
Evaporator Vacuum System	CB-13
Exhaust Fan	CH-10
Waste Water Holding Tank	CG-52
Feed Slurry Transfer Pump	CA-10
Evaporator Feed Pump	CB-10
Dissolver Feed Pump	CB-11
Crystallizer Circulating Pump	CB-12
Mother Liquor Transfer Pump	CD-10
Filter No. 2 Filtrate Pump	CD-11
Filter No. 3 Filtrate Pump	CD-12
Waste Solvent Pump	CG-10
Recycle Solvent Pump	CF-10
Condensate Pump	CE-10
Wash Water Pump	CE-11

Table 2: Experimental Conditions for Crystallizing RDX in the CNCPR Systems

Run No.	Time Factor (hr.)	Oper. Line Ratio	Evaporator Conditions		Composition Mass Fraction			Dissolver Cond		Apparent	Crystallizer Conditions				Feed Pt.		
			T (°C)	P (Torr)	Expl.	DMSO	Water	T_D (°C)	Feed Rate (GPM)	T_s (°C)	Oper. Temp. #1/#2 (°C)	Circ. Rate #1/#2 (GPM)	Feed Rate #1/#2 (GPM)	Drop Cycle #1/#2 (Min.)	Inches From Bottom	Samples Removed	t_b
1	t = 0 Init.																
CNCPR- MOD-1	Cond.=	0.300	80	100	0.275	0.634	0.091	75	0	75	30/30	1.0/1.0	0/0	15/15	4/4	-	-
	2.25	0.300	80	100	0.275	0.634	0.091	75	0	62	32/31	1.0/1.0	0/0	15/15	4/4	R10151915.CD53S	40
	3.25	0.300	80	100	0.275	0.634	0.091	75	0	53	32/32	1.0/1.0	0.25/0.25	15/15	4/4	R10152230.CD53S	37
	2.00	0.300	80	100	0.275	0.634	0.091	75	0	65	35/36	1.0/1.0	0.15/0.15	15/15	4/4	-	-
	3.50	0.300	80	100	0.275	0.634	0.091	75	0	70	35/36	1.0/1.0	0.15/0.15	15/15	4/4	R10150400 CD53S	43
	3.25	0.300	80	100	0.275	0.634	0.091	75	0	76	36/36	1.0/1.0	0/0	Off	4/4	R10160800 CD53S	49
2	t = 0 Init.																
CNCPR- MOD-1	Cond.=	0.343	80	100	0.275	0.634	0.091	70	0	76	42/45	1.0/1.0	0.25/0.25	15/15	4/4	-	-
	2.33	0.343	80	100	0.301	0.615	0.084	70	0	77	41/43	1.0/1.0	0.25/0.25	15/15	4/4	R10161300.CD53S	51
	1.00	0.343	80	100	0.312	0.612	0.076	70	0	72	41/43	1.0/1.0	0.25/0.25	15/15	4/4	R10161400 CD53S	43
	0.50	0.343	80	100	0.318	0.614	0.068	70	0	65	40/42	1.0/1.0	0.25/0.25	15/15	4/4	R10161430 CD53S	37
	1.50	0.343	80	100	0.320	0.612	0.068	70	0	69	40/41	1.0/1.0	0.25/0.25	15/15	4/4	R10161600.CD53S	57
	2.17	0.343	80	100	0.320	0.612	0.068	70	0	68	40/41	1.0/1.0	0/0	15/15	4/4	-	-
	0.83	0.343	30	ATM	0.320	0.612	0.068	70	0	60	35/36	1.0/1.0	0/0	Off	4/4	R10161900.CD53S	105
3	t = 0 Init.																
CNCPR- MOD-1	Cond.=	0.343	80	100	0.320	0.612	0.068	70	0	60	40/40	1.0/1.0	0.25/0.25	15/15	4/4	-	-
	1.83	0.343	80	100	0.320	0.612	0.068	70	0	74	40/41	1.0/1.0	0.25/0.25	15/15	4/4	R10160520.CD53S	45
	1.17	0.343	80	100	0.320	0.612	0.068	70	0	69	40/41	1.0/1.0	0/0	Off	4/4	R10160630.CD53S	60
4	t = 0 Init.																
CNCPR- MOD-1	Cond.=	0.343	80	100	0.320	0.612	0.068	75	0	69	40/40	1.0/1.0	0.25/0.25	20/20	4/4	-	-
	2.25	0.343	80	100	0.320	0.612	0.068	75	0	82	40/40	1.0/1.0	0.25/0.25	20/20	4/4	R10171230.CD53S	35
	1.00	0.343	80	100	0.320	0.612	0.068	75	0	75	40/38	1.0/1 0	0.25/0.25	20/20	4/4	R10171330.CD53S	46

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Table 2: (Continued)

Run No.	Time Factor (hr.)	Oper. Line Ratio	Evaporator Conditions					Dissolver Cond.			Oper. Temp #1/#2 (°C)	Crystallizer Conditions			Feed Pt. Inches From Bottom	Samples Removed	Lb
			T (°C)	P (Torr)	Expl.	DMSO	Water	T_D (°C)	Feed Rate (GPM)	Apparent T_s (°C)		Circ. Rate #1/#2 (GPM)	Feed Rate #1/#2 (GPM)	Drop Cycle #1/#2 (Min.)			
	0.83	0.343	80	100	0.320	0.612	0.068	75	0	73	40/40	1.0/1.0	0/0	20/20	4/4	-	-
	0.17	0.343	80	100	0.320	0.612	0.068	75	0	72	38/39	1.0/1.0	0/0	20/20	4/4	R10171500.CD53S	46
	1.25	0.343	80	100	0.320	0.612	0.068	75	0	60	41/43	1.5/1.5	0/0	20/20	4/4	-	-
	0.17	0.343	80	100	0.320	0.612	0.068	70	0	58	42/44	1.5/1.5	0/0	20/20	4/4	R10171625.CD53S	39
	1.42	0.343	80	100	0.320	0.612	0.068	70	0	52	45/46	1.5/1.5	0.25/0.25	20/20	4/4	-	-
	0.17	0.343	80	100	0.320	0.612	0.068	70	0	50	46/46	1.5/1.5	0/0	Off	4/4	R10171830.CD53S	37
5 CNCPR-MOD-1	t = 0 Init. Cond.=	0.343	80	100	0.320	0.612	0.063	70	0	50	43/43	1.5/1.5	0.25/0.25	20/20	4/4	-	-
	2.00	0.343	80	100	0.320	0.612	0.068	70	0	58	44/46	1.5/1.5	0.15/0.15	20/20	4/4	R10172315.CD53S	45
	2.08	0.343	80	100	0.420	0.612	0.068	70	0	60	42/43	1.5/1.5	0.15/0.15	20/20	4/4	R10170105.CD53S	42
	2.08	0.343	80	100	0.320	0.612	0.068	70	0	66	41/42	1.5/1.5	0.15/0.15	20/20	4/4	R10170310.CD53S	36
	2.58	0.343	80	100	0.320	0.612	0.068	70	0	75	42/42	1.5/1.5	0/0	Off	4/4	R10170550.CD53S	30
6 CNCPR-MOD-1	t = 0 Init. Cond.=	0.343	80	100	0.320	0.612	0.068	70	0	75	40/40	1.0/1.0	0.25/0.25	10/10	4/4	-	-
	2.25	0.343	80	100	0.320	0.612	0.068	70	0	83	41/42	1.0/1.0	0/0	Off	4/4	R10181130.CD53S	45
	Restart	0.343	80	100	0.320	0.612	0.068	70	0	83	40/42	1.0/1.0	0.25/0.25	10/10	4/4	-	-
	2.17	0.343	80	100	0.320	0.612	0.068	70	0	80	42/43	1.0/1.0	0.25/0.25	10/10	4/4	R10182345.CD53S	43
	1.17	0.343	80	100	0.320	0.612	0.058	70	0	70	43/44	1.0/1.0	0.25/0.25	10/10	4/4	R10180055.CD53S	56
	2.75	0.343	80	100	0.320	0.612	0.068	70	0	83	46/47	1.0/1.0	0.25/0.25	10/10	4/4	R10180240.CD53S	43
	1.83	0.343	80	100	0.320	0.612	0.068	70	0	67	48/48	1.0/1.0	0.25/0.25	10/10	4/4	R10180430.CD53S	61
	2.00	0.343	80	100	0.320	0.612	0.068	70	0	63	49/50	1.0/1.0	0.25/3.25	10/10	4/4	R10180630.CD53S	57
	0.50	0.343	80	100	0.320	0.612	0.068	70	0	62	50/50	1.0/1.0	0/0	10/10	4/4	-	-
	0.33	0.343	80	100	0.320	0.612	0.068	70	0	63	40/40	1.0/1.0	0.25/0.25	10/10	4/4	-	-
	0.83	0.343	80	100	0.320	0.612	0.068	70	0	65	48/48	1.0/1.0	0/0	Off	4/4	R10190900.CD53S	28

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Table 2: (Continued)

Run No.	Time Factor (hr.)	Oper. Line Ratio	Evaporator Conditions T (°C)	P (Torr)	Composition Mass Fraction Expl.	DMSO	Water	Dissolver Cond. Tb (°C)	Feed Rate (GPM)	Apparent Ts (°C)	Crystallizer Conditions Oper. Temp #1/#2 (°C)	Circ. Rate #1/#2 (GPM)	Feed Rate #1/#2 (GPM)	Drop Cycle #1/#2 (Min.)	Feed Pt. Inches From Bottom	Samples Removed	Lb
7 CNCPR-MOD-1	t = 0 Init. Cond.=																
	2.08	0.343	80	100	0.320	0.612	0.068	70	0	65	40/40	1.0/1.0	0.25/0.25	10/10	4/4	–	–
	2.08	0.343	80	100	0.320	0.612	0.068	70	0	77	42/43	1.0/1.0	0/0	10/10	4/4	–	–
	0.42	0.343	80	100	0.320	0.612	0.068	70	0	80	43/43	1.0/1.0	0/0	10/10	4/4	R10191200.CD53S	31
	0.42	0.343	80	100	0.320	0.612	0.068	70	0	62	42/42	1.0/1.0	0/0	Off	4/4	R10191230.CD53S	48

Run No.	Time Factor (hr.)	Oper. Line Ratio	Evaporator Conditions T (°C)	P (Torr)	Composition Mass Fraction Expl.	DMSO	Water	Cryst. Overflow Calc. Mass. Fract. % Expl. Solids(%)	Feed Rate (GPM)	Apparent Ts (°C)	Crystallizer Conditions Oper. Temp #1/#2 (°C)	Circ. Rate #1/#2 (GPM)	Feed Rate #1/#2 (GPM)	Drop Cycle #1/#2 (Min.)	Feed Pt. Inches From Bottom	Samples Removed	Lb
8 CNCPR-MOD-2	t = 0 Init. Cond.=							–	–	–	–/30	–			–/4	–	–
	4.08	0.343	80	100	0.320	0.612	0.068	0.260	10.8	60	–/26	–	0/0.3	–/10	–/4	R10222005.CD53S	62
	2.42	0.343	80	100	0.320	0.612	0.068	0.210	2.5	48	–/29	–	0/0.3	–/10	–/4	R10222230.CD53S	63
(one Cryst.)	1.00	0.343	80	100	0.320	0.612	0.068	0.228	4.7	53	–/32	–	0/0.3	–/10	–/4	–	–
	0.83	0.343	80	100	0.320	0.612	0.068	0.243	4.2	56	–/38	–	0/0.5	–/10	–/4	R10220020.CD53S	46
	0.67	0.343	80	100	0.320	0.612	0.068	0.245	4.4	57	–/38	–	0/0.5	–/10	–/4	–	–
	2.25	0.343	80	100	0.320	0.612	0.068	0.254	7.8	58	–/32	–	0/0.3	–/10	–/4	R10220235.CD53S	55
	1.33	0.343	80	100	0.320	0.612	0.068	0.195	1.0	45	–/32	–	0/0.3	–/10	–/4	R10220430.CD53S	39
	1.67	0.343	80	100	0.320	0.612	0.068	0.240	6.0	55	–/33	–	0/0.3	–/10	–/4	R10220615.CD53S	33
	2.50	0.343	80	100	0.320	0.612	0.068	0.275	9.4	67	–/35	–	0/0.4	–/10	–/4	–	–
Agitation Reduction →	1.58	0.343	80	100	0.320	0.612	0.068	0.307	12.2	75	–/38	–	0/0.4	–/10	–/4	R10231140.CD53S	50
	1.17	0.343	80	100	0.320	0.612	0.068	0.242	3.2	56	–/40	–	0/0.4	–/10	–/4	R10231420.CD53S	44
	2.92	0.343	80	100	0.320	0.612	0.068	0.276	5.7	67	–/43	–	0/0	Off	–/4	–	–
Cumulative Totals	22.42	–	80	100	0.320	0.612	0.068	0.261	6.8	60	–/34	–	0/0.35	–/10	–/4	–	392

Table 2. (Continued)

Run No.	Time Factor (hr.)	Oper. Line Ratio	Evaporator Conditions T (°C)	P (Torr)	Composition Mass Fraction Expl.	DMSO	Water	Cryst. Overflow Mass Fract. Expl.	Calc. % Solids	Apparent T_s (°C)	Oper. Temp #1/#2 (°C)	Crystallizer Conditions Circ. Rate #1/#2 (GPM)	Feed Rate #1/#2 (GPM)	Drop Cycle #1/#2 (Min.)	Feed Pt. Inches From Bottom	Samples Removed	Lb
9 CNCPR-MOD-2	t = 0 Init. Cond.=	0.343	80	100	0.320	0.612	0.068	-	-	-	-/25	-	0/0.5	-/10	-/8	-	-
	2.42	0.343	80	100	0.320	0.612	0.068	0.272	5.2	65	-/43	-	0/0.4	-/10	-/8	-	-
(One Cryst.)	0.25	0.343	80	100	0.320	0.612	0.068	0.266	5.9	62	-/41	-	0/0.4	-/10	-/8	R10232000.CD53S	53
	1.25	0.343	80	100	0.320	0.612	0.068	0.261	5.3	60	-/41	-	0/0	Off	-/8	R10232150.CD53S	26
Cumulative Totals	3.92	-	80	100	0.320	0.612	0.068	0.269	5.3	67	-/37	-	0/0.46	-/10	-/8	-	79
10 CNCPR-MOD-2	t = 0 Init Cond.=	0.343	80	100	0.320	0.612	0.068	-	-	-	-/36	-	0/0.5	-/10	-/12	-	-
	2.67	0.310	80	100	0.297	0.633	0.070	0.242	5.3	56	-/35	-	0/0.5	-/10	-/12	R10230400.CD53S	64
(One Cryst.)	2.00	0.310	80	100	0.286	0.643	0.071	0.224	3.0	52	-/35	-	0/0.5	-/10	-/12	R10230530.CD53S	48
	1.25	0.310	80	100	0.286	0.643	0.071	0.218	4.3	50	-/37	-	0/0	Off	-/12	R10240715.CD53S	48
Cumulative Totals	5.92	-	80	100	0.298	0.632	0.070	0.231	4.3	53	-/35	-	0/0.5	-/10	-/12	-	160
11 CNCPR-MOD-2	t = 0 Init. Cond.=	0.310	80	100	0.286	0.643	0.071	-	-	-	-/40	-	0/0.5	-/7	-/12	-	-
	1.58	0.310	80	100	0.286	0.643	0.071	0.198	1.0	45	-/32	-	0/0.5	-/7	-/12	R10241015.CD53S	58
(One Cryst.)	0.75	0.343	80	100	0.286	0.643	0.071	-	-	-	-/35	-	0/0.5	-/7	-/12	-	-
	1.50	0.343	80	100	0.299	0.631	0.070	0.203	0.0	47	-/37	-	0/0.5	-/7	-/12	R10241230.CD53S	44
	1.83	0.343	80	100	0.314	0.617	0.069	0.222	2.0	51	-/37	-	0/0.5	-/7	-/12	R10241420.CD53S	42
	1.17	0.343	80	100	0.320	0.612	0.068	0.232	3.3	53	-/37	-	0/0	Off	-/12	R10241625.CD53S	87
Cumulative Totals	6.83	-	80	100	0.298	0.632	0.070	0.212	1.3	47	-/36	-	0/0.5	-/7	-/12	-	231

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Table 2 (continued)

Run No.	Time Factor (hr.)	Oper. Line Ratio	Evaporator Conditions T (°C)	P (Torr)	Composition Mass Fraction Expl.	DMSO	Water	Cryst. Overflow Mass. Fract. Expl.	Calc. % Solids	Apparent T_s (°C)	Oper. Temp #1/#2 (GPM)	Crystallizer Conditions Circ. Rate #1/#2 (GPM)	Feed Rate #1/#2 (GPM)	Drop Cycle #1/#2 (Min.)	Feed Pt. Inches From Bottom	Samples Removed	Lb
12 CNCPR-MOD-2	t = 0 Init. Cond.=	0.307	80	100	0.284	0.644	0.072	-	-	-	-/40	-	0/0.4	-/20	-/4		-
	1.80	0.307	80	100	0.284	0.644	0.072	-	-	-	-/45	-	0/0.4	-/20	-/4		-
	3.83	0.307	80	100	0.284	0.644	0.072	0.258	3.4	59	-/45	-	0/0	Off	-/4	R1.051900.CD53S	50
Cumulative Totals	5.63	-	80	100	0.284	0.644	0.072	0.258	3.4	59	-/43	-	0/0.4	-/20	-/4	-	50
13 CNCPR-MOD-2	t = 0 Init. Cond.=	0.343	80	100	0.320	0.612	0.068	-	-	-	-/30	-	0/0.5	-/10	/12		-
	1.2	0.343	80	100	0.320	0.612	0.068	-	-	-	-/35	-	0/0.5	-/10	-/12		-
	2.0	0.343	80	100	0.320	0.612	0.068	0.257	4.9	58	-/37	-	0/0	Off	-/12	R11060100.CD53S	84
Cumulative Totals	3.2	-	80	100	0.320	0.612	0.068	0.257	4.9	58	-/35	-	0/0.5	-/10	-/12	-	84

Run No.	Time Factor (hr.)	Oper. Line Ratio	Evaporator Conditions T (°C)	P (Torr)	Composition Mass Fraction Expl.	DMSO	Water	Cryst. Overflow Mass. Fract. Expl.	Calc. % Solids	Apparent T_s (°C)	Oper. Temp. (°C)	Water Rate (GPM)	Feed Rate (GPM)	Drop Cycle (Min.)	Feed Pt. In.From Bottom	Samples Removed	Lb
14 CNCPR-MOD-3	t = 0 Init. Cond.=	0.343	80	100	0.320	0.612	0.068	-	-	-	30		0.5	10	12		-
	1.17	0.343	80	100	0.320	0.612	0.068	-	-	-	43	0.017	0.5	10	12		-
	1.17	0.343	80	100	0.320	0.612	0.068	0.187	0.703	52	47	0.017 0	0	Off	12	R11060600.CD53S	114
Cumulative Totals	2.34	-	80	100	0.320	0.612	0.068	0.187	0.703	52	41	0.017	0.5	10	12	-	114

Table 2. (Continued)

Run No.	Time Factor (hr.)	Oper. Line Ratio	Evaporator Conditions T (°C)	P (Torr)	Composition Mass Fraction Expl.	DMSO	Water	Dissolver Cond. T_D (°C)	Feed Rate (GPM)	Apparent T_s (°C)	Crystallizer Conditions Oper. Temp #1/#2 (°C)	Circ. Rate #1/#2 (GPM)	Feed Rate #1/#2 (GPM)	Drop Cycle #1/#2 (Min.)	Feed Pt. Inches From Bottom	Samples Removed	Lb
15 CNCPR	t = 0 Init. Cond.=	0.343	80	100	0.320	0.612	0.068	70	0.30	75	-/37	-/1.0	0/0	-/20	-	-	-
	0.33	0.343	80	100	0.320	0.612	0.068	70	0.00	75	-/38	-/1.0	0/0	-/20	-	-	-
	1.00	0.343	80	100	0.320	0.612	0.068	70	0.00	72	-/40	-/1.0	0/0	-/20	-	-	-
	1.00	0.343	80	100	0.320	0.612	0.068	65	0.00	69	-/40	-/1.0	0/0	-/20	-	R11071612.CD53S	36
	0.83	0.343	80	100	0.320	0.612	0.068	65	0.00	64	-/45	-/1.0	0/0	-/20	-	-	-
	1.67	0.343	80	100	0.320	0.612	0.068	65	0.00	54	-/45	-/1.0	0/0	-/20	-	R11071825.CD53S	46
	0.33	0.343	80	100	0.320	0.612	0.068	65	0.30	54	-/45	-/1.0	0/0	-/20	-	-	-
	3.50	0.343	80	100	0.320	0.612	0.068	65	0.30	58	-/45	-/1.0	0/0	-/20	-	R11072230.CD53S	47
	1.33	0.343	80	100	0.320	0.612	0.068	65	0.60	58	-/45	-/1 0	0/0	-/20	-	-	-
	0.67	0.343	80	100	0.320	0.612	0 068	65	0.00	58	-/45	-/1 0	0/0	Off	-	R11070030.CD-3S	35

Operating Line Ratio - See Appendix C
T_D - Dissolver Temperature
T_s - Dissolver Saturation Temperature
#1/#2 - Refers to Crystallizers CC-50 and CC-51, Respectively

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Table 3: Results of RDX/CNCPR Experiments

Run No.	Run Duration (hr.)	Product Yield (lb. RDX)	Average Prod. Rate (lb/hr.)	Theoretical Yield (lb. RDX)	System Efficiency (% Theor. Yield)	Solvent Purge Rate (%)	Solvent Usage Rate (lb. DMSO/lb. RDX)
1	14.25	169	11.86	369	45.8	20	1.66
2	8.33	293	35.17	524	56.0	20	1.35
3	3.00	105	35.00	216	48.6	20	1.47
4	7.26	203	27.96	300	67.7	20	1.19
5	8.73	153	17.51	319	48.0	20	1.92
6	13.82	333	24.08	720	46.3	20	1.94
7	2.92	79	27.05	162	48.8	20	1.45
8	22.42	392	17.48	845	46.4	20	1.93
9	3.92	79	20.15	186	42.5	20	2.15
10	5.92	160	27.03	260	61.5	20	1.84
11	6.83	231	33.82	293	79.8	20	1.55
12	5.63	50	8.88	156	32.1	20	3.96
1.	3.20	84	26.25	168	50.0	20	1.85
14	2.34	114	48.72	145	78.6	20	1.18
15	10.66	164	15.38	205	80.0	20	1.08

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Table 4: Experimental Conditions for Crystallizing RDX by Dilution

Run No.	Dilution Procedure	Time Factor (Min.)	Heel Composition T (°C)	Heel Mass Fraction Expl.	Heel Mass Fraction DMSO	Heel Mass Fraction Water	Heel Amount Used (Gal.)	Feed Solution T (°C)	Feed Mass Fraction Expl.	Feed Mass Fraction DMSO	Feed Mass Fraction Water	Rate (GPM)	Dilution Water Rate (GPM)	Final Liquid Conc. Solids Free % DMSO Initial	Final Liquid Conc. Solids Free % DMSO Final	Dilution Point
1	a	t = 0	Ambient	0	0	1.0	2.5	80	0.325	0.605	0.070	-	-	-	-	-
		20	-	-	-	-	-	80	0.325	0.605	0.070	0.31	10	0	42.4	Cryst.
		30	-	-	-	-	-	80	0.325	0.605	0.070	0.16	5	42.4	47.4	Cryst.
		120	-	-	-	-	-	80	0.325	0.605	0.070	0.25	8	47.4	54.9	Cryst.
2	b	t = 0	70	-	-	1.0	5.0	80	0.240	0.685	0.075	-	-	-	-	-
		20	-	-	-	-	-	80	0.240	0.685	0.075	0.21	0	0	39.1	Cryst.
		17	-	-	-	-	-	80	0.240	0.685	0.075	0.21	14.5	39.1	39.5	Cryst.
3	c	8.9 (Heat batch to 70°C, hold for 30 min., screen hot through 325 mesh, filter)	-	-	-	-	-	80	0.240	0.685	0.075	0.67	30.0	-	3	Feed Tube
4	c	10.4 (Heat batch to 70°C, hold for 30 min., screen hot through 325 mesh, filter)	-	-	-	-	-	80	0.240	0.685	0.075	0.62	20	-	56.3	Feed Tube
5	d	10.4 (Age batch at ambient temp. for 30 min., screen through 325 mesh, filter)	-	-	-	-	-	80	0.240	0.685	0.075	0.62	20	-	56.3	Feed Tube
6	d	6.5 (Age batch at ambient temp. for 30 min., screen through 325 mesh, filter)	-	-	-	-	-	80	0.240	0.685	0.075	0.47	10	-	65.0	Feed Tube
7	d	6.5 (Age batch at ambient temp. for 30 min., screen through 325 mesh, filter)	-	-	-	-	-	80	0.240	0.685	0.075	0.46	15	-	56.4	Feed Tube

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Table 4 (Continued)

Run No.	Dilution Procedure	Time Factor (Min.)	Heel Composition T (°C)	Heel Mass Fraction Expl.	Heel Mass Fraction DMSO	Heel Mass Fraction Water	Amount Used (Gal.)	Feed Solution T (°C)	Feed Composition Mass Fraction Expl.	Feed Composition Mass Fraction DMSO	Feed Composition Mass Fraction Water	Rate (GPH)	Dilution Water Rate (GPM)	Final Liquid Conc. Solids Free % DMSO Initial	Final	Dilution Point
8	d	6.5	-	-	-	-	-	80	0.240	0.685	0.075	0.46	15	-	56.4	Feed Tube
		(Age batch at ambient temp. for 30 min., screen through 325 mesh, filter)														
9	e	6.5	-	-	-	-	-	80	0.240	0.685	0.075	0.47	10	-	65.0	Feed Tube
		10	-	-	-	-	-	-	-	-	-	0	10	-	45.4	Cryst.
		(Age batch at ambient temp. for 30 min.) (Screen through 325 mesh, filter).														
10	e	6.5	-	-	-	-	-	80	0.240	0.685	0.075	0.47	10	-	65.0	Feed Tube
		10	-	-	-	-	-	-	-	-	-	0	10	-	45.4	Cryst.
		(Age batch at ambient temp. for 20 min.) (Screen through 325 mesh, filter)														
11	e	6.5	-	-	-	-	-	80	0.240	0.685	0.075	0.47	10	-	65.0	Feed Tube
		10	-	-	-	-	-	-	-	-	-	0	10	-	45.4	Cryst.
		(Age batch at ambient temp. for 10 min.) (Screen through 325 mesh, filter)														
12	d	6.5	-	-	-	-	-	80	0.240	0.685	0.075	0.47	10	-	65.0	Feed Tube
		(Age batch at ambient temp. for 20 min., screen through 325 mesh, filter)														
13	d	6.5	-	-	-	-	-	80	0.240	0.685	0.075	0.47	10	-	65.0	Feed Tube
		(Age batch at ambient temp. for 10 min., screen through 325 mesh, filter)														
14	e	6.5	-	-	-	-	-	80	0.240	0.685	0.075	0.47	10	-	65.0	Feed Tube
		1.67	-	-	-	-	-	-	-	-	-	0	60	-	45.4	Cryst.
		(Age batch at ambient temp. for 10 min.) (Screen through 325 mesh, filter)														

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Table 5: Results of RDX/Dilution Crystallization Experiments

Run No.	Run Duration (min.)	Crude Yield (lb. RDX)	Crude Theoretical Yield (lb. RDX)	Actual Yield (lb. Class 5)	Product Impact (cm)	Alpha HMX Present *	System Efficienty (Amt. of Class 5 as % of Theor. Yield)	Solvent Usage Rate lb. DMSO/lb.Class 5
1	197	127	139	Cl. 7 – 98 Cl. 1 – 26 Cl. 5 – 3	43 52 –	no no no	(Crude Yield % of Crude Theor. Yield)= 91	2.03
2	77	18	19.2	4	<10	yes	20.8	13.34
3	74	12	13.3	12	<10	yes	90.2	3.16
4	75	14	15.5	14	<10	yes	90.3	3.16
5	65	13	15.5	13	<10	yes	83.9	4.97
6	46	5	6.9	5	47	small amt. (microscope)	72.5	4.19
7	46	5	7.4	5	45	small amt. (microscope)	67.6	4.19
8	46	6	7.4	6	31	small amt. (microscope)	81.0	3.42
9	61	7	7.4	7	42	no	94.6	3.00
10	51	7	7.4	7	43	no	94.6	3.00
11	41	7	7.4	7	43	no	94.6	3.00
12	36	5	7.4	5	46	no	67.5	4.19
13	26	7	7.4	7	48	no	94.6	3.00
14	28	7	7.4	7	52	no	94.6	3.00

*Microscopic Analysis

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Table 6: Analytical Results of Class 1 RDX Samples

Sample No.	Particle Size Distribution Percent Passing USS Sieve Nos.								Median Size (μ)	% RDX	% HMX	% SEX	%* α HMX	Poly- morph by IR	Long Impact (cm)	Crystal Acidity (%)
	8	12	20	35	50	100	200	325								
R10222005.CD53S	100	100	100	98	67	17	5	3	247	97.70	2.30	-	ND	Normal	42	0.020
R10222230.CD53S	100	100	100	97	74	26	6	4	223	-	-	-	-	-	50	0.000
R10220020.CE53S	100	96	90	85	62	17	4	2	258	-	-	-	-	-	51	0.000
R10220235.CD53S	100	100	100	94	64	22	6	4	248	97.92	2.08	-	-	-	51	0.000
R10220430.CD53S	100	100	100	99	75	30	15	12	215	94.64	5.36	-	-	-	50	0.000
R10220615.CD53S	100	100	100	99	77	29	11	8	214	95.62	4.38	-	-	-	48	0.000.
R10231000.CD53S	100	100	99	98	64	15	3	2	255	-	-	-	-	-	62	0.000
R10232000.CD53S	100	100	100	99	81	18	3	1	224	98.02	1.98	-	ND	Normal	-	-
R10230400.CD53S	100	100	99	98	62	18	7	6	257	97.36	2.64	-	ND	Normal	51	0.000
R10230530.CD53S	100	100	100	97	67	25	13	12	237	96.12	3.88	-	ND	Normal	54	0.000
R10240715.CD53S	100	100	99	90	66	18	4	3	248	97.85	2.15	-	ND	Normal	46	0.000
R10241015.CD53S	100	100	99	98	67	11	2	1	252	98.53	1.47	-	ND	Normal	44	0.000
R10241230.CD53S	100	100	100	100	79	24	5	3	219	98.36	1.64	-	< 0.01	Alpha Present	53	0.000
R10241420.CD53S	100	100	100	99	70	18	4	3	240	98.69	1.31	-	< 0.01	Alpha Present	48	0.000
R10241625.CD53S	100	100	100	99	73	24	13	12	228	98.77	1.23	-	ND	Normal	53	0.010
R11060100.CD53S	100	100	99	92	67	24	22	21	238	98.39	1.61	-	-	-	40	-
R10231420.CD53S	100	100	100	99	85	24	8	5	212	97.81	2.19	-	ND	Normal	-	-
R10232150.CD53S	100	100	100	99	87	28	7	3	204	97.40	2.60	-	ND	Normal	41	0.000
R10172315.CD53S	100	100	100	86	85	50	44	43	149	97.75	3.25	-	ND	Normal	44	0.000
R10231140.CD53S	100	100	100	99	96	46	31	29	161	96.31	3.69	-	-	-	58	0.000
R11060600.CD53S	100	100	100	99	98	64	18	5	126	95.19	4.81	-	-	-	-	-
R10252420.CD52S	100	100	100	99	86	37	11	3	188	88.07	11.93	-	ND	Normal	52	0.010

*Microscopic Analysis, Lower Detection Limit is 0.01%
ND - None Detected
RDX, HMX, SEX Composition analyses are by liquid chromatography, ASM 1-26.

Table 7: Analytical Results of Class 3 RDX Samples

Sample No.	Particle Size Distribution Percent Passing USS Sieve Nos.								Median Size (µ)	% RDX	% HMX	% SEX	%* α HMX	Polymorph by IR	Long Impact (cm)	Crystal Acidity (%)
	8	12	20	35	50	100	200	325								
R10161400.CD53S	100	100	99	79	40	3	0	0	349	98.77	1.23	-	<0.01	Alpha Present	56	0.000
R10161430.CD53S	100	100	99	78	34	7	5	5	371	98.40	1.60	-	ND	Normal	50	0.000
R10161600.CD53S	100	100	97	64	26	6	4	4	425	99.29	0.71	-	ND	Normal	47	0.000
R10161900.CD53S	100	100	96	62	27	6	3	2	430	99.34	0.66	-	ND	Normal	57	0.000
R10170105.CD53S	100	100	99	81	39	8	5	4	350	97.25	2.75	-	ND	Normal	59	0.000
R10170310.CD53S	100	100	100	71	24	6	4	3	409	98.80	1.20	-	ND	Normal	63	0.000
R10170550.CD53S	100	100	95	55	25	12	6	5	466	97.30	2.70	-	ND	Normal	50	0.000
R10182345.CD53S	100	100	99	78	32	7	3	2	376	98.35	1.65	-	ND	Normal	61	0.000
R10180055.CD53S	100	100	99	80	33	8	4	3	370	98.58	1.42	-	ND	Normal	55	0.000
R10190900.CD53S	100	100	95	57	24	10	7	6	457	97.83	2.17	-	ND	Normal	63	0.000
R10151915.CD53S	100	100	99	92	56	14	2	1	276	100.00	0.00	-	ND	Normal	47	0.000
R10191200.CD53S	100	100	99	88	54	15	5	3	282	97.38	2.62	-	ND	Normal	58	0.000
R10191230.CD53S	100	100	99	87	52	17	7	6	289	97.54	2.46	-	ND	Normal	52	0.010
R11071825.CD53S	100	100	99	90	58	12	2	1	271	99.21	0.79	-	ND	Normal	50	0.000
R11072230.CD53S	100	100	100	77	52	19	9	8	288	99.51	0.48	-	ND	Normal	49	0.000
R10161300.CD53S	100	100	99	84	46	9	1	0	318	97.93	2.07	-	<0.01	Alpha Present	52	0.000
R10180240.CD53S	100	100	96	66	36	9	3	2	392	98.05	1.95	-	ND	Normal	55	0.000
R10180430.CD53S	100	100	96	68	31	8	3	2	401	98.76	1.24	-	ND	Normal	54	0.000
R10152230.CD53S	100	100	99	87	47	14	8	7	312	98.24	1.76	-	<0.01	Alpha Present	43	0.000
R10150400.CD53S	100	100	97	77	46	23	14	9	323	97.16	2.77	0.07	<0.01	Alpha Present	41	0.000
R10160800.CD53S	100	100	97	71	40	21	15	10	362	98.06	1.94	-	<0.01	Alpha Present	50	0.000
R10150520.CD53S	100	100	99	88	50	10	3	3	297	98.29	1.61	-	ND	Normal	48	0.024
R10160630.CD53S	100	100	99	99	48	22	19	19	305	98.03	1.97	-	ND	Normal	48	0.000
R10171330.CD53S	100	100	84	51	37	17	5	4	486	98.43	1.57	-	ND	Normal	48	0.000
R10181130.CD53S	100	100	99	86	49	13	5	4	307	97.84	2.16	-	ND	Normal	57	0.010
R10180630.CD53S	100	100	96	71	36	11	6	5	378	98.39	1.61	-	ND	Normal	45	0.000
R11051900.CD53S	100	100	100	83	45	12	4	3	324	99.37	0.63	-	ND	Normal	57	0.300
R11070030.CD53S	100	100	100	75	50	15	7	5	297	97.51	2.49	-	ND	Normal	-	-

*Microscopic Analysis, Lower Detection Limit is 0.01%

ND – None Detected

RDX, HMX, SEX composition analyses are by liquid chromatography, ASM T-26.

Table 8: Analytical Results of Class 4 RDX Samples

Sample No.	Particle Size Distribution Percent Passing USS Sieve Nos.								Median Size (u)	% RDX	% HMX	% SEX	% α HMX*	Polymorph by IR	Long Impact (cm)	Crystal Acidity (%)
	8	12	20	35	50	100	200	325								
R10171230.CD53S	100	100	82	44	29	10	3	2	554	98.23	1.77	-	ND	Normal	50	0.000
R10171500.CD53S	99	98	78	33	20	8	4	4	628	98.57	1.43	-	ND	Normal	54	0.000
R10171625.CD53S	100	99	72	25	15	7	4	4	681	99.34	0.66	-	ND	Normal	56	0.010
R10171830.CD53S	100	98	68	23	15	8	5	3	704	99.19	0.81	-	ND	Normal	53	0.000

*Microscopic Analysis, Lower Detection Limit is 0.01%
ND - None Detected
RDX, HMX, SEX composition analyses are by liquid chromatography, ASM 1-26.

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Table 3. Analytical Results of Class 5 RDX Samples

Sample No.	8	12	20	35	50	100	200	325	Median Size (μ)	% RDX	% HMX	% SEX	% αHMX *	Polymorph by IR	Long Impact (cm)	Crystal Acidity (%)
R10311430.CD52/3S	-	-	-	-	-	-	-	99	-	95.08	4.92	-	ND	Normal	39	0.000
R10312215.CD52/3S	-	-	-	-	-	-	-	100	-	90.11	8.89	-	ND	Normal	49	0.000
R10310400.CD52/3S	-	-	-	-	-	-	-	100	-	90.75	9.25	-	ND	Normal	48	0.000
R10310645.CD52/3S	-	-	-	-	-	-	-	100	-	97.52	2.48	-	ND	Normal	51	0.000
R10011120.CD52/3S	-	-	-	-	-	-	-	100	-	98.07	1.93	-	-	-	48	-
R10011440.CD52/3S	-	-	-	-	-	-	-	100	-	98.31	1.69	-	-	-	52	-
R10012200.CD52/3S	-	-	-	-	-	-	-	100	-	100.00	0.00	-	-	-	36	-
R10010445.CD52/3S	-	-	-	-	-	-	-	100	-	93.65	6.35	-	ND	Normal	43	0.000
R10021130.CD52/3S	-	-	-	-	-	-	-	100	-	99.03	0.97	-	-	-	36	-
k10214 30.CD52/3S	-	-	-	-	-	-	-	99	-	96.58	3.42	-	-	-	35	-
R10021830.CD52/3S	-	-	-	-	-	-	-	98	-	97.02	2.98	-	-	-	52	-
R10022200.CD52/3S	-	-	-	-	-	-	-	97	-	97.14	2.86	-	-	-	46	-
R10020055.CD52/3S	-	-	-	-	-	-	-	100	-	92.02	7.98	-	-	-	47	0.000
R10160800.CD52S	100	100	100	81	76	70	68	99	-	80.08	19.92	-	<0.01	Alpha Present	45	0.000
R10161900.CD52S	-	-	-	-	-	-	-	65	-	74.79	24.89	-	ND	Normal	43	0.000
R10170830.CD52S	-	-	-	-	-	-	-	98	-	70.72	29.28	-	ND	Normal	47	0.000
R10171845.CD52S	100	100	100	96	91	86	85	84	-	66.80	33.20	-	ND	Normal	44	0.002
R10191230.CD52S	-	-	-	-	-	-	-	98	-	66.53	33.47	-	ND	Normal	56	0.000
R10230710.CD52S	-	-	-	-	-	-	-	97	-	-	-	-	-	-	55	0.000
R10230300.CD53S	-	-	-	-	-	-	-	99	-	62.50	37.50	-	ND	Normal	44	0.010
.10241640.CD52S	-	-	-	-	-	-	-	64	-	67.74	32.26	-	<0.01	Alpha Present	49	0.000
R10301045.CD52S	-	-	-	-	-	-	-	96	-	84.91	15.09	-	<0.01	Alpha Present	44	0.000
R11051900.CD52S	-	-	-	-	-	-	-	99	-	97.06	2.94	-	ND	Normal	55	0.000

*Microscopic Analyses, Lower Detection Limit is 0.01%
ND - None Detected
RDX, HMX, SEX composition analyses are by liquid chromatography, ASM I-26.

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Table 10: Analytical Results of Class 7 RDX Samples

Sample No.	Particle Size Distribution Percent Passing USS Sieve Nos.								Median Size (μ)	% RDX	% HMX	% SEX	%* α HMX	Polymorph by IR	Long Impact (cm)	Crysta Acidit (%)
	8	12	20	35	50	100	200	325								
R10252030.CD53S	100	100	100	100	100	96	44	11	83	90.08	9.92	-	ND	Normal	43	0.000
R10252115.CD53S	100	100	100	99	98	89	35	9	95	38.21	11.79	-	ND	Normal	46	0.000
R10252210.CD53S	100	100	100	100	99	82	33	11	100	88.15	11.85	-	ND	Normal	41	0.000

*Microscopic Analysis, Lower Detection Limit is 0.01%
ND - None Detected
RDX, HMX, SEX composition analyses are by liquid chromatography, ASM J-26.

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Table 11: Analytical Results of Representative Samples of DMSO-Recrystallized RDX

Sample No.	Residual Solvent (Weight % DMSO)		VTS at 100°C and 48 hr. (cc Gas/Gr. Sample)	% Acetone Insolubles	% Inorganic Insolubles	Number of Insoluble Particles on USSS No. 60 Screen	Melting Point (°C)
	Wet Sample	Dry Sample					
R10152230.CD53S	0.17	0.04	0.0346	0.00	< 0.03	None	203
R10161300.CD53S	0.08	0.04	0.0553	0.00	< 0.03	None	204
R10161900.CD53S	0.42	0.03	0.0373	0.00	0.00	None	202
R10171625.CD53S	0.07	0.04	0.0741	0.00	< 0.03	None	203
R10172315.CD53S	0.07	0.04	0.0431	0.00	< 0.03	None	203
R10182345.CD53S	0.14	0.04	0.0459	0.00	0.00	None	194
R10191200.CD53S	0.13	0.02	0.0360	0.00	0.00	None	202
R10222005.CD53S	0.53	0.03	0.0579	0.00	0.00	None	197
R10171330.CD53S	0.13	0.04	0.0236	0.00	0.00	None	199
R10171830.CD53S	0.04	0.03	0.0386	0.00	0.00	None	198
R10180630.CD53S	0.06	0.01	0.0458	0.00	0.00	None	201
R10220430.CD53S	0.12	0.03	0.0320	0.00	0.00	None	199
R10231140.CD53S	0.58	0.03	0.0360	0.00	< 0.03	None	198
R10241230.CD53S	0.22	< 0.01	0.0384	0.00	0.00	None	203
R10252115.CD53S	0.58	0.04	0.0394	0.00	0.00	None	196
R10252420.CD52S	0.03	0.03	0.0437	0.00	< 0.03	None	195
R10312215.CD52/3S	0.43	< 0.01	0.0359	0.00	< 0.03	None	196
R11011120.CD52/3S	1.30	< 0.01	0.0305	0.00	< 0.03	None	197
R11021130.CD52/3S	0.89	< 0.01	0.0268	0.00	0.00	None	197
R11022200.CD52/3S	1.72	0.19	0.0116	0.00	< 0.03	None	196
R10310400.CD52/3S	0.40	< 0.01	0.0416	0.00	< 0.03	None	200
R11020055.CD52/3S	0.34	< 0.01	0.0353	0.00	0.00	None	198
R11051900.CD53S	0.10	0.09	0.0456	0.00	0.00	None	204
R11060600.CD53S	0.12	0.03	0.0543	0.00	< 0.03	None	201
R11070030.CD53S	0.06	0.03	0.0418	0.00	< 0.03	None	204

Table 12: Experimental Conditions for Crystallizing HMX in The CNCPR Systems

Run No.	Time Factor (hr.)	Oper. Line Ratio	Evaporator Conditions T (°C)	P (Torr)	Composition Mass Fraction Expl.	DMSO	Water	Dissolver Cond. T_D (°C)	Feed Rate (GPM)	Apparent T_B (°C)	Crystallizer Conditions Oper. Temp #1/#2 (°C)	Circ. Rate #1/#2 (GPM)	Feed Rate #1/#2 (GPM)	Drop Cycle #1/#2 (Min.)	Feed Pt. Inches From Bottom	Samples Removed	Lb
1 CNCPR-MOD-1	t = 0 Init. Cond.*																
	2.00	0.300	80	100	0.270	0.631	0.099	75	0	80	42/43	1.5/1.5	0/0	20/20	–	–	–
	1.25	0.300	80	100	0.270	0.631	0.099	75	0	73	42/43	1.5/1.5	0/0	20/20	–	H10021600.CD53S	25
	1.00	0.300	80	100	0.270	0.631	0.099	70	0	61	42/43	1.5/1.5	0/0	20/20	:	H10021715.CD53S	40
	1.00	0.300	80	100	0.270	0.631	0.099	70	0	56	42/44	1.5/1.5	0.1/0.1	20/20	–	–	–
	1.08	0.300	80	100	0.270	0.631	0.099	70	0	51	42/44	1.5/1.5	0.1/0.1	20/20	4/4	H10021915.CD53S	32
		0.300	80	100	0.270	0.631	0.099	70	0	47	42/44	1.5/1.5	0/0	Off	4/4	H10020030.CD53S	35
2 CNCPR-MOD-1 3 gph Water Addition	t = 0 Init. Cond.* {0.83 0.33 0.83}																
	0.83	0.300	80	100	0.270	0.631	0.099	70	0	47	42/43	1.5/1.5	0.1/0.1	20/20	4/4	–	–
	0.33	0.300	80	100	0.270	0.631	0.099	70	0	45	42/44	1.5/1.5	0.1/0.1	70/20	4/4	–	–
	0.83	0.300	80	100	0.270	0.631	0.099	70	0	60	42/45	1.5/1.5	0.1/0.1	20/20	4/4	–	–
								70	0	50	42/45	1.5/1.5	0/0	Off	4/4	H10020400.CD53S	20
3 CNCPR-MOD-1	t = 0 Init. Cond.*																
	1.37	0.300	80	100	0.270	0.631	0.099	70	0	50	42/43	1.5/1.5	0.1/0.1	20/20	4/4	–	–
	1.30	0.300	80	100	0.270	0.631	0.099	70	0	49	42/44	1.5/1.5	0.07/0.07	20/20	4/4	–	–
		0.300	80	100	0.270	0.631	0.099	70	0	47	43/45	1.5/1.5	0/0	Off	4/4	H10031100.CD53S	48
4 CNCPR-MOD-1	t = 0 Init. Cond.*																
	1.75	0.300	80	100	0.270	0.631	0.099	70	0	47	40/42	1.5/1.5	0.07/0.07	20/20	4/4	–	–
	2.25	0.300	80	100	0.270	0.631	0.099	70	0	50	41/43	1.5/1.5	0.1/0.1	20/20	4/4	–	–
		0.300	80	100	0.270	0.631	0.099	70	0	56	44/46	1.5/1.5	0/0	Off	4/4	H10032030.CD53S	27
5 CNCPR-MOD-1	t = 0 Init. Cond.*																
	0.58	0.300	80	100	0.270	0.631	0.099	65	0	56	42/44	2.0/2.0	0.1/0.1	20/20	4/4	–	–
	0.67	0.300	80	100	0.270	0.631	0.099	65	0	57	42/44	1.5/1.5	0.1/0.1	20/20	4/4	–	–
	1.83	0.300	80	100	0.270	0.631	0.099	70	0	58	40/42	1.5/1.5	0.1/0.1	20/20	4/4	–	–
		0.300	80	100	0.270	0.631	0.099	70	0	59	42/44	1.5/1.5	0/0	Off	4/4	H10030400.CD53S	21

Table 12: (Continued)

Run No.	Time Factor (hr.)	Oper. Line Ratio	Evaporator Conditions T (°C)	P (Torr)	Composition Mass Fraction Expl	DMSO	Water	Dissolver Cond. T_D (°C)	Feed Rate (GPM)	Apparent T_B (°C)	Oper. Temp #1/#2 (°C)	Crystalliser Conditions Circ. Rate #1/#2 (GPM)	Feed Rate #1/#2 (GPM)	Drop Cycle #1/#2 (Min.)	Feed Pt. Inches From Bottom	Samples Removed	Lb
6 CNCPR-MOD-1	t = 0 Init.																
	Cond.=	0.300	80	100	0.270	0.631	0.099	70	0	59	35/35	1.0/1.0	0/0	20/20	4/4	-	-
	1.33	0.300	80	100	0.270	0.631	0.099	70	0	55	34/35	1.0/1.0	0.1/0.1	20/20	4/4	-	-
	1.33	0.300	80	100	0.270	0.631	0.099	70	0	53	39/38	1.0/1.0	0.1/0.1	20/20	4/4	H10041220.CD535	30
	2.00	0.300	80	100	0.270	0.631	0.099	70	0	56	40/39	1.0/1.0	0.1/0.1	20/20	4/4	H10041440.CD535	0
	0.83	0.300	80	100	0.270	0.631	0.099	70	0	55	40/40	1.0/1.0	0.15/0.15	20/20	4/4	-	-
	1.50	0.300	80	100	0.270	0.631	0.099	70	0	54	46/43	1.0/1.0	0/0	off	4/4	H10041650.CD535	59
7 CNCPR	t = 0 Init.																
	Cond.=	0.335	80	100	0.307	0.610	0.083	80	0	77	-/65	0/1.0	-	-/20	-	-	-
	1.08	0.335	80	100	0.307	0.610	0.083	80	0	74	-/35	0/1.0	-	-/20	-	-	-
	0.75	0.335	80	100	0.307	0.610	0.083	70	0	69	-/37	0/1.0	-	-/20	-	-	-
	0.50	0.335	80	100	0.307	0.610	0.083	70	0	67	-/37	0/1.0	-	-/20	-	H11261450.CD535	47
	2.58	0.335	80	100	0.307	0.610	0.083	70	0.20	55	-/38	0/1.0	-	-/20	-	H11261725.CD535	56
	0.33	0.335	80	100	0.307	0.610	0.083	70	0.40	50	-/38	0/1.0	-	-/20	-	-	-
	2.00	0.335	80	100	0.307	0.610	0.083	70	0.30	65	-/38	0/1.5	-	-/20	-	-	-
	2.00	0.335	80	100	0.307	0.610	0.083	70	0.15	71	-/38	0/1.5	-	-/20	-	-	-
	1.67	0.335	80	100	0.307	0.610	0.083	70	0.10	72	-/38	0/1.5	-	-/20	-	-	-
	1.00	0.335	80	100	0.307	0.610	0.083	70	0.10	73	-/39	0/1.0	-	-/20	-	H11260120.CD535	65
	0.83	0.335	80	100	0.307	0.610	0.083	70	0.10	73	-/40	0/1.0	-	-/20	-	-	-
	0.67	0.335	80	100	0.307	0.610	0.083	70	0	71	-/35	0/1.0	-	-/20	-	-	-
	0.25	0.335	80	100	0.307	0.610	0.083	70	0.20	70	-/37	0/1.0	-	-/20	-	-	-
	0.75	0.335	80	100	0.307	0.610	0.083	70	0.20	69	-/38	0/1.0	-	-/20	-	H11260620.CD535	34
	0.08	0.335	80	100	0.307	0.610	0.083	70	0	72	-/41	0/1.0	-	-/20	-	-	-
	3.08	0.335	80	100	0.307	0.610	0.083	70	0	71	-/42	0/1.0	-	-/20	-	-	-
	0.75	0.335	80	100	0.307	0.610	0.083	70	0.30	69	-/43	0/1.0	-	-/20	-	-	-
	0.92	0.335	80	100	0.307	0.610	0.083	70	0	68	-/43	0/1.0	-	-/20	-	H11270910.CD535	25
Cryst. Switch {	0.38	0.335	80	100	0.307	0.610	0.083	70	0	69	-/42	0/1.0	-	-/20	-	-	-
	0.33	0.335	80	100	0.307	0.610	0.083	70	0	69	40/-	1.0/0-	-	-/30	-	-	-

Table 12: (continued)

Run No.	Time Factor (hr.)	Oper. Line Ratio	Evaporator Conditions T (°C)	P (Torr)	Composition Mass Fraction Expl.	DMSO	Water	Dissolver Cond. T_D (°C)	Feed Rate (GPM)	Apparent T_s (°C)	Oper. Temp. #1/#2 (°C)	Crystallizer Conditions Circ. Rate #1/#2 (GPM)	Feed Rate #1/#2 (GPM)	Drop Cycle #1/#2 (Min.)	Feed Pt. Inches From Bottom	Samples Removed	Lb
7 (continued)	2.67	0.335	80	100	0.307	0.610	0.083	70	0.15	60	40/-	1.0/-	-	30/-	-	-
	1.67	0.335	80	100	0.307	0.610	0.083	70	0.15	60	41/-	1.0/-	-	30/-	H1127133O.CD53S	5?
	0.75	0.335	80	100	0.307	0.610	0.083	70	0.18	58	41/-	1.0/-	-	30/-	-	-
	0.75	0.335	80	100	0.307	0.610	0.083	70	0	59	41/-	1.0/-	-	30/-	H1127160O.CD53S	54
	1.00	0.335	80	100	0.307	0.610	0.083	70	0	54	41/-	1.0/-	-	30/-	-	-
	0.17	0.335	80	100	0.307	0.610	0.083	70	0.50	53	41/-	1.0/-	-	30/-	-	-
	0.25	0.406	80	100	0.307	0.610	0.083	70	0.22	55	41/-	1.0/-	-	30/-	-	-
	0.92	0.406	80	100	0.318	0.603	0.079	70	0.30	58	42/-	1.0/-	-	30/-	HJ127203O.CD53S	48
	3.17	0.406	80	100	0.357	0.575	0.068	70	0.30	70	42/-	1.0/-	-	30/-	-	-
	1.12	0.406	80	100	0.363	0.571	0.066	70	0.20	71	43/-	1.0/-	-	30/-	HJ127221S.CD53S	53
	1.08	0.406	80	100	0.370	0.569	0.061	70	0.20	70	43/-	1.0/-	-	30/-	-	-
	0.33	0.335	80	100	0.364	0.573	0.063	70	0	70	43/-	1.0/-	-	30/-	-	-
	0.42	0.335	80	100	0.364	0.573	0.063	70	0.20	69	43/-	1.0/-	-	30/-	H1127010O.CD53S	76
	2.00	0.335	80	100	0.347	0.584	0.069	70	0.20	65	43/-	1.0/-	-	30/-	-	-
	1.25	0.335	80	100	0.339	0.589	0.072	70	0	67	43/-	1.0/-	-	30/-	-	-
	0.25	0.335	80	100	0.339	0.589	0.072	70	0.30	66	43/-	1.0/-	-	30/-	H1127060O.CD53S	71
	3.50	0.335	80	100	0.316	0.604	0.080	70	0.30	73	44/-	1.0/-	-	30/-	-	-
	0.67	0.335	80	100	0.314	0.605	0.081	70	0	73	44/-	1.0/-	-	30/-	-	-
	0.83	0.335	80	100	0.314	0.605	0.081	70	0	66	44/-	1.0/	-	30/-	H1128083O.CD53S	54
	1.00	0.335	80	100	0.314	0.605	0.081	70	0	61	44/-	1.0/	-	30/-	H1128100O.CD53S	21
8 CNCPR-MOD-1	t = 0 Init. Cond.=															
	1.75	0.335	85	140	0.311	0.620	0.069	75	0	75	-/34	-/1.0	-	-/10	-	-
	1.12	0.335	85	140	0.311	0.620	0.069	70	0	62	-/34	-/1.0	-	-/10	H1128144S.CD53S	40
	1.60	0.335	85	140	0.311	0.620	0.069	70	0.20	54	-/35	-/1.0	-	-/10	-	-
	0.53	0.335	85	140	0.311	0.620	0.069	70	0.30	59	-/35	-/1.0	-	-/10	-	-
	0.50	0.335	85	140	0.311	0.620	0.069	70	0.30	60	-/35	-/1.0	-	-/10	H1128180O.CD53S	50
	1.50	0.335	85	140	0.311	0.620	0.069	70	0.20	64	-/36	-/1.0	-	-/15	-	-
	1.67	0.335	85	140	0.311	0.620	0.069	70	0.16	61	-/36	-/1.0	-	-/15	-	-
	1.83	0.335	85	140	0.311	0.620	0.069	70	0	59	-/36	-/1.0	-	-/15	H1128214O.CD53S	59
	1.00	0.335	85	140	0.311	0.620	0.069	70	0.30	48	-/37	-/1.0	-	-/15	-	-
	1.00	0.335	85	140	0.311	0.620	0.069	70	0.40	51	-/38	-/1.0	-	-/15	-	-
	0.33	0.335	85	140	0.311	0.620	0.069	70	0	54	-/39	-/1.0	-	-/15	-	-
	2.17	0.335	85	140	0.311	0.620	0.069	70	0.20	48	-/41	-/1.0	-	-/15	-	-
	2.00	0.335	85	140	0.311	0.620	0.069	70	0	53	-/45	-/1.0	-	-/15	H1128053O.CD53S	86

(continued)

Table 12: (Continued)

Run No.	Time Factor (hr.)	Oper. Line Ratio	Evaporator Conditions T (°C)	P (Torr)	Mass Fraction Expl.	DMSO	Water	Dissolver Cond. T_B (°C)	Feed Apparent Rate (GPM)	T_g (°C)	Oper. Temp #1/#2 (°C)	Circ. Rate #1/#2 (GPM)	Feed Rate #1/#2 (GPM)	Drop. Cycle #1/#2 (Min.)	Feed Pt. Inches From Bottom	Samples Removed	Lb
9 CNCPR	t = 0																
	Init. Cond. s	0.335	85	140	0.311	0.620	0.069	70	0	83	-/44	-/1.0	-	-/15	-	-	-
	1.67	0.335	85	140	0.311	0.620	0.069	70	0.25	68	-/42	-/1.0	-	-/15	-	H11291300.CD53S	59
	0.33	0.335	85	140	0.311	0.620	0.069	70	0	70	-/42	-/1.0	-	-/15	-	-	-
	0.67	0.335	85	140	0.311	0.620	0.056	70	0.25	65	-/44	-/1.0	-	-/15	-	-	-
	0.25	0.335	90	140	0.317	0.628	0.056	70	0.30	62	-/44	-/1.0	-	-/15	-	H11291445.CD53S	34
	0.50	0.335	90	140	0.317	0.628	0.056	70	0	61	-/44	-/1.0	-	-/15	-	-	-
	0.34	0.335	90	140	0.317	0.628	0.056	70	0.30	60	-/44	-/1.0	-	-/15	-	-	-
	0.88	0.335	90	140	0.317	0.628	0.056	70	0	55	-/45	-/1.0	-	-/15	-	-	-
	0.78	0.335	90	140	0.317	0.628	0.056	70	0.30	65	-/45	-/1.0	-	-/15	-	-	-
	1.50	0.335	90	140	0.317	0.628	0.056	70	0.30	55	-/45	-/1.0	-	-/15	-	-	-
	0.52	0.335	90	140	0.317	0.628	0.056	70	0.20	55	-/45	-/1.0	-	-/15	-	H11292300.CD53S	55
	0.60	0.335	90	140	0.317	0.628	0.056	70	0	60	-/46	-/1.0	-	-/15	-	-	-
	0.60	0.335	90	140	0.317	0.628	0.056	70	0	53	-/47	-/1.0	-	-/15	-	-	-
10 CNCPR	t = 0																
	Init. Cond. s	0.335	85	140	0.311	0.620	0.069	70	0	75	-/38	-/1.0	-	-/15	-	-	-
	1.00	0.335	85	140	0.311	0.620	0.069	70	0.20	60	-/36	-/1.0	-	-/15	-	-	-
	1.42	0.335	85	140	0.311	0.620	0.061	70	0.20	58	-/41	-/1.0	-	-/15	-	H11290130.CD53S	80
	1.67	0.335	85	140	0.311	0.620	0.069	70	0	55	-/40	-/1.0	-	-/15	-	-	-
	0.42	0.335	85	140	0.311	0.620	0.069	70	0	56	-/41	-/1.0	-	-/15	-	-	-
	1.33	0.335	85	140	0.311	0.620	0.069	70	0.20	52	-/41	-/1.0	-	-/15	-	H11290600.CD53S	75
	1.17	0.335	85	140	0.311	0.620	0.069	70	0	50	-/41	-/1.0	-	-/15	-	-	-
	2.00	0.335	85	140	0.311	0.620	0.069	70	0	51	-/41	-/1.0	-	-/15	-	-	-
	1.50	0.335	85	140	0.311	0.620	0.069	70	0.20	47	-/42	-/1.0	-	-/30	-	H11300800.CD53S	46
	0.30	0.335	85	140	0.311	0.620	0.069	70	0.20	42	-/62	-/1.0	-	-/30	-	-	-
	1.92	0.335	85	140	0.311	0.620	0.069	70	0.30	45	-/43	-/1.0	-	-/30	-	H11301130.CD53S	57
	1.58	0.335	85	140	0.311	0.620	0.069	70	0.30	56	-/43	-/1.0	-	-/30	-	-	-
	1.27	0.335	85	140	0.311	0.620	0.069	70	0	61	-/43	/1.0	-	-/30	-	-	-
	1.42	0.335	85	140	0.311	0.620	0.069	70	0.25	61	-/43	-/1.0	-	-/30	-	-	-
	0.17	0.335	85	140	0.311	0.620	0.069	70	0	64	-/43	-/1.0	-	-/30	-	-	-

Table 12:(continued)

Run No.	Time Factor (hr.)	Oper. Line Ratio	Evaporator Conditions T (°C)	P (Torr)	Composition Mass Fraction Expl.	DMSO	Water	Dissolver Cond. T_D (°C)	Feed Rate (GPH)	Apparent T (°C$\ddagger$)	Crystallizer Conditions Oper. Temp #1/#2 (°C)	Circ. Rate #1/#2 (GPM)	Feed Rate #1/#2 (GPH)	Drop Cycle #1/#2 (Min.)	Feed Pt. Inches From Bottom	Samples Removed	Lb
10 (cont'd)	2.00	0.335	85	140	0.311	0.620	0.069	70	0.20	66	-/43	-/1.0	-	-/30	-	-	-
	0.50	0.335	85	140	0.311	0.620	0.069	70	0.20	66	-/43	-/1.0	-	-/30	-	H11301830.CD53S	67
	1.25	0.335	85	140	0.311	0.620	0.069	70	0	68	-/44	-/1.0	-	-/30	-	-	-
11 CHCPR	t = 0 Init. Cond.																
	1.67	0.335	85	140	0.311	0.620	0.069	70	0	68	-/39	-/1.0		-/30	-	H11300030.CD53S	76
	1.33	0.335	85	140	0.311	0.620	0.069	70	0.20	68	-/37	-/1.0	-	-/30	-	-	-
	0.75	0.335	85	140	0.311	0.620	0.069	70	0.20	68	-/34	-/1.0	-	-/30	-	-	-
	2.25	0.335	85	140	0.311	0.620	0.069	70	0.20	65	-/35	-/1.0	-	-/30	-	-	-
	0.67	0.335	85	140	0.311	0.620	0.069	70	0	60	-/36	-/1.0	-	-/30	-	-	-
	1.08	0.335	85	140	0.311	0.620	0.069	70	0.20	61	-/37	-/1.0	-	-/30	-	-	-
	0.25	0.335	85	140	0.311	0.620	0.069	70	0.70	57	-/38	-/1.0	-	-/30	-	H11300430.CD53S	75
	2.75	0.335	85	140	0.311	0.620	0.069	70	0.25	56	-/38	-/1.0	-	-/30	-	-	-
	1.45	0.335	85	140	0.311	0.620	0.069	70	0.25	65	-/38	-/2.0	-	-/30	-	H12010730.CD53S	33
	0.55	0.335	85	140	0.311	0.620	0.069	70	0	68	-/39	-/1.0	-	-/30	-	-	-
	0.67	0.335	85	140	0.311	0.620	0.069	75	0	66	-/43	-/1.0	-	-/30	-	-	-
		0.335	85	140	0.311	0.620	0.069	73	0	65	-/43	-/1.0	-	-/30	-	H12011010.CD53S	42

Operating Line Ratio - See Appendix C
T_D - Dissolver Temperature
T_S - Dissolver Saturation Temperature
#1/#2 - Refers to Crystallizers CC-50 and CC-51, Respectively

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Table 13: Results of HMX/CNCPR Experiments

Run No.	Run Duration (hr)	Product Yield (lb HMX)	Average Prod. Rate (lb/hr)	Theoretical Yield (lb HMX)	System Efficiency (% Theor. Yield)	Solvent Purge Rate (%)	Solvent Usage Rate (lb DMSO/lb HMX)
1	6.33	132	20.85	152	86.8	15	0.55
2	1.99	20	10.05	56	35.7	15	1.62
3	2.67	48	17.98	60	80.0	15	0.96
4	4.00	27	6.75	64	42.2	15	2.05
5	3.08	21	6.81	73	28.8	15	2.18
6	6.99	89	12.73	158	56.3	15	1.25
7	42.95	654	15.23	1,094	59.8	20	1.21
8	16.00	235	14.69	506	46.6	20	0.91
9	8.64	148	17.13	265	55.8	20	1.25
10	20.92	325	15.54	402	80.8	20	0.65
11	13.42	226	16.84	618	36.6	20	1.40

Table 14: Experimental Conditions for Crystallizing HMX by Dilution

Run No.	Dilution Procedure	Time Factor (Min.)	Heel Seed Expl. (Wt.%)	Heel T (°C)	Heel Liquid Comp. Mass Fraction Expl.	DMSO	Water	Amount Used (Gal.)	Feed Solution T (°C)	Feed Composition Mass Fraction Expl.	DMSO	Water	Rate (GPM)	Dilution Water Rate (GPM)	Final Liquid Conc. Solids Free % DMSO Initial	Final	Dilution Point
1	a	7.0	-	-	-	-	-	-	80	0.220	3.675	0.105	0.64	10.0	-	67.7	Feed Tube
									-	-	-	-	-	10.0	67.7	52.9	Cryst.
		(Age batch at ambient temp. for 10 min.) 9.0 (Screen through 325 mesh and filter.)															
2	a	7.0	-	-	-	-	-	-	80	0.220	0.675	0.105	0.64	10.0	-	67.7	Feed Tube
									-	-	-	-	0	60.0	67.7	52.9	Cryst.
		(Age batch at ambient temp. for 10 min.) 1.5 (Screen through 325 mesh and filter.)															
3	a	5.0	-	-	-	-	-	-	80	0.220	0.675	0.105	0.66	7.5	-	72.0	Feed Tube
									-	-	-	-	0	60.0	72.0	47.6	Cryst.
		(Age batch at ambient temp. for 10 min.) 1.9 (Screen through 325 mesh and filter)															
4	a	5.0	-	-	-	-	-	-	80	0.220	0.675	0.105	0.82	6.0	-	76.6	Feed Tube
									-	-	-	-	0	60.0	76.6	50.8	Cryst.
		(Age batch at ambient temp. for 10 min.) 2.2 (Screen through 325 mesh and filter)															
5	b	t = 0	0	95	0	0	1.00	7.2	80	0.220	0.675	0.105	0.15	0	0	22.5	Cryst.
		18.0	-	-	-	-	-	-	80	0.220	0.675	0.105	0.15	38.4	22.5	15.7	Cryst.
		(Age batch at 95°C for 20 min.) 60.0 (Product continuously dropped to 325 mesh screen and filters)															

(continued)

Table 14 (continued)
(2)

Run No.	Dilution Procedure	Time Factor (Min.)	Seed T (°C)	Seed Expl. (Wt.%)	Heel Liquid Comp. Mass Fraction Expl.	DMSO	Water	Amount Used (Gal.)	Feed Solution T (°C)	Composition Mass Fraction Expl.	DMSO	Water	Rate (GPM)	Dilution Water Rate (GPM)	Final Liquid Conc. Solids Free % DMSO Initial	Final	Dilution Point
6	c	t = 0	95	6.25*	0	0	1.0	7.2	80	0.220	0.675	0.105	-	-	-	-	-
		30.0	-	-	-	-	-	-	80	0.220	0.675	0.105	0.15	38.4	0	14.1	cryst.
		(product continuously removed at 95°C to 325 mesh screen and filter)															
7	d	t = 0	98	1.67*	0	0	1.0	7.2	80	0.220	0.675	0.105	-	-	-	-	-
		5.0	-	-	-	-	-	-	80	0.220	0.675	0.105	0.50	0	0	21.2	cryst.
		(age batch at 95°C for 30 min., cool to 50°C, screen through 325 mesh, filter)															
8	e	t = 0	20	2.83*	0	0	1.0	3.5	80	0.220	0.675	0.105	-	-	-	-	-
		7.0	-	-	-	-	-	-	80	0.220	0.675	0.105	0.50	20	0	31.1	cryst.
		(heat to 97°C, age for 30 min., cool to 50°C. screen through 325 mesh, filter)															
9	e	t = 0	20	1.72*	0	0	1.0	3.5	80	0.220	0.675	0.105	-	-	-	-	-
		7.0	-	-	-	-	-	-	80	0.220	0.675	0.105	0.50	20	0	31.1	cryst.
		(heat to 95°C, age for 30 min., cool to 50°C, screen through 325 mesh, filter)															
10	f	t = 0	20	1.72*	0	0	1.0	3.5	80	0.220	0.675	0.105	-	-	-	-	-
		7.0	-	-	-	-	-	-	80	0.220	0.675	0.105	0.50	20	0	31.1	cryst.
		(heat to 95°C, age for 30 min.)															
		62.0	-	-	-	-	-	-	80	0.220	0.675	0.105	0.20	10	31.1	44.2	-

*HCP Seed Material

(continued)

Table 14 (continued)
(3)

Run No.	Dilution Procedure	Time Factor (Min.)	Heel Seed Expl. (Wt.%)	Heel T (°C)	Heel Liquid Comp. Mass Fraction Expl.	DMSO	Water	Amount Used (Gal.)	Feed Solution T (°C)	Feed Composition Mass Fraction Expl.	DMSO	Water	Rate (GPM)	Dilution Water Rate (GPM)	Final Liquid Conc. Solids Free % DMSO Initial	Final	Dilution Point
11	b	t = 0	0	20	0	0	1.0	3.5	80	0.220	0.675	0.105	-	-	-	-	-
		7.0	-	-	-	-	-	-	80	0.220	0.675	0.105	0.50	20	0	31.1	cryst.
		(heat to 95°C, age for 30 min., cool to 50°C, screen through 325 mesh, filter)															
12	e	t = 0	2.04+	20	0	0	1.0	3.5	80	0.220	0.675	0.105	-	-	-	-	-
		5.0	-	-	-	-	-	-	80	0.220	0.675	0.105	0.50	20	0	26.9	cryst.
		(heat to 95°C, age for 30 min., cool to 50°C, screen through 325 mesh, filter)															
13A	g	t = 0	2.83*	20	0	0	1.0	3.5	80	0.220	0.675	0.105	-	-	-	-	-
		7.0	-	-	-	-	-	-	80	0.220	0.675	0.105	0.50	20	0	31.3	cryst.
		(heat to 95°C, age for 30 min., cool to 50°C, screen through 325 mesh, filter, leave 3.5 gallons of product slurry in crystallizer)															
13B	g	t = 0	10.20++	40	0	0.313	0.687	3.5	80	0.220	0.675	0.105	-	-	31.3	-	-
		7.0	-	-	-	-	-	-	80	0.220	0.675	0.105	0.50	20	31.3	43.3	cryst.
		(heat to 95°C, age for 30 min., cool to 50°C, screen through 325 mesh, filter)															
14A	g	t = 0	1.70*	20	0	0	1.0	3.5	80	0.220	0.675	0.105	-	-	-	-	-
		7.0	-	-	-	-	-	-	80	0.220	0.675	0.105	0.50	20	0	31.2	cryst.
		(heat to 95°C, age for 30 min., cool to 50°C, screen through 325 mesh, filter, leave 3.5 gallons of product slurry in crystallizer)															

*HCF Seed Material
+Seed Material from a previous product batch.
++Seed Material retained in the heel of the previous batch.

(continued)

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Table 14 (continued)
(4)

Run No.	Dilution Procedure	Time Factor (Min.)	Heel Seed Expl. T (°C)	Heel Seed Expl. (Wt.%)	Heel Liquid Comp. Mass Fraction Expl.	Heel Liquid Comp. Mass Fraction DMSO	Heel Liquid Comp. Mass Fraction Water	Amount Used (Gal.)	Feed Solution T (°C)	Feed Solution Composition Mass Fraction Expl.	Feed Solution Composition Mass Fraction DMSO	Feed Solution Composition Mass Fraction Water	Rate (GPM)	Dilution Water Rate (GPM)	Final Liquid Conc. Solids Free % DMSO Initial	Final Liquid Conc. Solids Free % DMSO Final	Dilution Point
14B	8	t = 0	40	9.80++	0	0.312	0.688	3.5	80	0.220	0.675	0.105	-	-	-	-	-
		7.0	-	-	-	-	-	-	80	0.220	0.675	0.105	0.50	20	31.2	43.1	cryst.
		(heat to 95°C, age for 30 min., cool to 50°C, screen through 325 mesh, leave 3.5 gallons of product slurry in crystallizer)															
14C	8	t = 0	40	12.49++	0	0.431	0.569	3.5	80	0.220	0.675	0.105	-	-	-	-	-
		7.0	-	-	-	-	-	-	80	0.220	0.675	0.105	0.50	20	43.1	47.7	cryst.
		(heat to 95°C, age for 30 min., cool to 50°C, screen through 325 mesh, filter)															
15A	8	t = 0	20	1.70	0	0	1.0	3.5	80	0.220	0.675	0.105	-	-	-	-	-
		7.0	-	-	-	-	-	-	80	0.220	0.675	0.105	0.50	20	0	31.2	cryst.
		(heat to 95°C, age for 30 min., cool to 50°C, screen through 325 mesh, filter, leave 3.5 gallons of product slurry in crystallizer)															
15B	8	t = 0	40	9.80++	0	0.312	0.688	3.5	80	0.220	0.675	0.105	-	-	-	-	-
		7.0	-	-	-	-	-	-	80	0.220	0.675	0.105	0.50	20	31.2	43.1	cryst.
		(heat to 95°C, age for 30 min., cool to 50°C, screen through 325 mesh, filter, leave 3.5 gallons of product slurry in crystallizer)															
15C	8	t = 0	40	12.49++	0	0.431	0.569	3.5	80	0.220	0.675	0.105	-	-	-	-	-
		7.0	-	-	-	-	-	-	80	0.220	0.675	0.105	0.50	20	43.1	47.7	cryst.
		(heat to 95°C, age for 30 min., cool to 50°C, screen through 325 mesh, filter, leave 3.5 gallons of product slurry in crystallizer)															
15D	8	t = 0	40	13.49++	0	0.477	0.523	3.5	80	0.220	0.675	0.105	-	-	-	-	-
		7.0	-	-	-	-	-	-	80	0.220	0.675	0.105	0.50	20	47.7	49.5	cryst.
		(heat to 95°C, age for 30 min., cool to 50°C, screen through 325 mesh, filter, leave 3.5 gallons of product slurry in crystallizer)															
15E	8	t = 0	40	13.9++	0	0.495	0.505	3.5	80	0.220	0.675	0.105	-	-	-	-	-
		7.0	-	-	-	-	-	-	80	0.220	0.675	0.105	0.50	20	49.5	50.1	cryst.
		(heat to 95°C, age for 30 min., cool to 50°C, screen through 325 mesh, filter, leave 3.5 gallons of product slurry in crystallizer.)															

(continued)

Table 14 (continued)
(5)

Run No.	Dilution Procedure	Time Factor (Min.)	T (°C)	Seed Expl. (Wt.%)	Heel Liquid Comp. Mass Fraction Expl.	DMSO	Water	Amount Used (Gal.)	T (°C)	Feed Solution Composition Mass Fraction Expl.	DMSO	Water	Rate (GPM)	Dilution Water Rate (GPM)	Final Liquid Conc. Solids Free % DMSO Initial	Final	Dilution Point
15F	g	t = 0	40	14.0++	0	0.501	0.499	3.5	80	0.220	0.675	0.105	-	-	50.1	50.4	cryst.
		7.0	-	-	-	-	-	-	80	0.220	0.675	0.105	0.50	20			
		(heat to 95°C, age for 30 min., cool to 50°C, screen through 325 mesh, filter, leave 3.5 gallons of product slurry in crystallizer)															
15G	g	t = 0	40	14.1++	0	0.504	0.496	3.5	80	0.220	0.675	0.105	-	-	50.4	50.5	cryst.
		7.0	-	-	-	-	-	-	80	0.220	0.675	0.105	0.50	20			
		(heat to 95°C, age for 30 min., cool to 50°C, screen through 325 mesh, filter, leave 3.5 gallons of product slurry in crystallizer)															
15H	g	t = 0	40	14.1++	0	0.505	0.495	3.5	80	0.220	0.675	0.105	-	-	50.5	50.5	cryst.
		7.0	-	-	-	-	-	-	80	0.220	0.675	0.105	0.50	20			
		(heat to 95°C, age for 30 min., cool to 50°C, screen through 325 mesh, filter)															
16A	g	t = 0	20	1.70	0	0	1.0	3.5	80	0.220	0.675	0.105	-	-	0	31.2	cryst.
		7.0	-	-	-	-	-	-	80	0.220	0.675	0.105	0.50	20			
		(heat to 95°C, age for 30 min., cool to 50°C, screen through 325 mesh, filter, leave 3.5 gallons of product slurry in crystallizer)															
16B	g	t = 0	40	9.8++	0	0.312	0.688	3.5	80	0.220	0.675	0.105	-	-	31.2	43.1	cryst.
		7.0	-	-	-	-	-	-	80	0.220	0.675	0.105	0.50	20			
		(heat to 95°C, age for 30 min., cool to 50°C, screen through 325 mesh, filter, leave 3.5 gallons of product slurry in crystallizer)															
16C	g	t = 0	40	12.49++	0	0.431	0.569	3.5	80	0.220	0.675	0.105	-	-	43.1	47.7	cryst.
		7.0	-	-	-	-	-	-	80	0.220	0.675	0.105	0.50	20			
		(heat to 95°C, age for 30 min., cool to 50°C, screen through 325 mesh, filter, leave 3.5 gallons of product slurry in crystallizer)															
16D	g	t = 0	40	13.49++	0	0.477	0.523	3.5	80	0.220	0.675	0.105	-	-	47.7	49.5	cryst.
		7.0	-	-	-	-	-	-	80	0.220	0.675	0.105	0.50	20			
		(heat to 95°C, age for 30 min., cool to 50°C, screen through 325 mesh, filter)															

Table 15: Results of HMX/Dilution Crystallization Experiments

Run No.	Run Duration (min)	Crude Yield (lb HMX)	Crude Theoretical Yield (lb HMX)	Actual Yield (lb Class 5)	Product Impact (cm)	Alpha HMX Present*	Overall System Efficiency (Amt. of Class 5 as % of Theor. Yield)	Solvent Usage Rate (lb DMSO/lb Class 5)
1	42	9	9.8	8.4	22	yes	85.7	3.60
2	30	10	9.8	7.0	21	yes	71.4	4.32
3	34	7	7.3	0.3	14	yes	4.1	-
4	34	9	9.0	0.2	-	yes	2.2	-
5	150	25	25.7	15.1	<10	alpha + gamma	58.8	5.23
6	90	12	13.9	10.6	26	yes	76.3	2.86
7	90	6	6.5	5.7	24	small amt. alpha	87.7	2.95
8	98	3	8.5	3.0	23	no	35.3	7.87
9	80	8	8.2	8.0	31	no	97.6	2.95
10	140	33	35.5	23.1	36	small amt. alpha	65.1	4.64
11	100	5	7.7	5.0	20	yes	64.9	4.72
12	85	6	6.1	2.1	<10	alpha + gamma	34.4	8.03
13A	75	5	5.3	4.2	27	no	79.2	3.51
13B	85	11	10.9	10.5	28	no	96.3	3.09
14A	85	4	5.0	4.0	19	small amt. alpha	80.0	3.62
14B	70	6	6.8	5.0	32	no	73.5	4.09
14C	70	8	11.8	6.0	33	no	50.8	5.98

*Microscopic Analysis

Table 15: (continued)

Run No.	Run Duration (min)	Crude Yield (lb HMX)	Crude Theoretical Yield (lb HMX)	Actual Yield (lb Class 5)	Product Impact (cm)	Alpha * HMX Present	Overall System Efficiency (Amt. of Class 5 as % of Theor. Yield)	Solvent Usage Rate (lb DMSO/lb Class 5
15A	80	4	5.0	4.0	-	-	-	-
15B	63	6	6.8	5.5	-	-	-	-
15C	62	7	7.4	6.5	-	trace alpha	-	-
15D	64	7	7.6	5.1	-		-	-
15E	66	6	7.7	4.2	-	-	-	-
15F	71	6	7.7	3.5	29	trace alpha	-	-
15G	69	8	7.7	5.3	-	-	-	-
15H	78	12	12.3	4.0	-	-	-	-
15 Cumulative-553		56	62.2	38.1	30	trace alpha	61.3	4.95
16A	77	5	5.0	5.0	-	-	-	-
16B	58	6	6.8	5.6	-	-	-	-
16C	56	7	7.4	5.3	-	-	-	-
16D	69	13	12.1	6.2	-	-	-	-
16 Cumulative-260		31	31.3	22.1	33	no	70.6	4.27

*Microscopic Analysis

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Table 16: Analytical Results of Class 1 HMX Samples

| Sample No. | Particle Size Distribution Percent Passing USS Sieve Nos. | | | | | | | | Median Size (μ) | Purity* | | | | Poly-morph by IR | Long Impact (cm) | Crystal Acidity (%) |
	8	12	20	35	50	100	200	325		% RDX	% HMX	% SEX	%** α HMX			
H08202400.CD53S	100	100	99	98	90	37	10	5	185	0.00	99.84	0.16	0.00	Beta	29	0.014
H08212400.CD53S	100	100	99	98	92	39	12	6	180	0.00	99.77	0.23	0.00	Beta	30	0.012
H09262400.CD53S	100	100	98	86	68	35	15	8	216	0.20	99.80	0.00	0.07	Beta	31	0.000
H09270730.CD53S	100	100	98	87	72	36	21	9	207	0.57	99.43	0.00	0.00	Beta	30	0.000
H09270830.CD53S	100	100	99	88	69	32	19	7	221	1.17	98.83	0.00	0.00	Beta	27	0.000
H09271730.CD53S	100	100	98	87	72	36	21	9	207	0.64	99.33	0.03	0.00	Beta	30	0.000.
H10032030.CD53S	100	100	99	92	73	21	8	2	232	0.31	99.69	0.00	0.00	Beta	31	0.000
H10030400.CD53S	100	100	99	84	65	38	21	11	215	0.42	99.58	0.00	0.00	Beta	32	0.000
H11261450.CD53S	99	97	93	88	68	33	10	3	221	0.09	99.91	0.00	0.00	Beta	32	0.004
H11281800.CD53S	100	100	94	88	81	53	31	12	139	0.00	100.00	0.00	0.00	Beta	35	0.003
H11282140.CD53S	100	100	100	99	81	28	9	3	210	0.00	100.00	0.00	0.00	Beta	30	0.000
H112914,5.CD53S	100	100	100	97	90	38	12	4	183	0.15	99.85	0.00	0.00	Beta	28	0.000
H11270910.CD53S	100	100	100	99	98	63	21	10	126	0.25	99.75	0.00	0.00	Beta	30	0.000
H11281445.CD53S	100	100	99	94	90	72	41	18	96	0.15	99.85	0.00	0.00	Beta	33	0.003
H11300800.CD53S	100	100	100	100	92	86	26	2	104	0.00	100.00	0.00	0.00	Beta	29	0.012
H09172400.CD53S	100	99	97	94	82	48	24	13	158	0.00	99.81	0.19	0.00	Beta	27	0.000
H10021715.CD53S	100	100	100	98	82	40	21	8	184	0.34	99.66	0.00	0.00	Beta	30	0.000
H11290600.CD53S	100	100	100	99	97	47	16	6	158	0.00	100.00	0.00	0.00	Beta	30	0.006
H11280530.CD53S	100	100	100	99	92	42	15	6	173	0.00	100.00	0.00	0.05	Beta	28	0.003
H11291300.CD53S	100	100	100	99	94	40	17	5	176	0.00	100.00	0.00	0.00	Beta	30	0.000

*Liquid Chromatorgaphic Analyses, ASM I-151
**X-Ray Analyses, ASM I-4

Table 17: Analytical Results of Class 3 HMX Sample

Sample No.	Particle Size Distribution Percent Passing USS Sieve No.								Median Size (μ)	Purity*				Poly-morph by IR	Long Impact (cm)	Crystal Acidity (%)
	8	12	20	35	50	100	200	325		% RDX	% HMX	% SEX	% α HMX**			
H10041440.CD53S	100	100	98	59	26	7	4	3	445	0.00	100.00	0.00	0.00	Beta	32	0.000
H10041650.CD53S	100	91	61	56	35	10	5	2	442	0.88	98.85	0.27	0.00	Beta	32	0.000
H11272030.CD53S	100	95	76	59	34	8	2	1	426	0.00	99.85	0.15	0.00	Beta	33	0.000
H11270100.CD53S	100	100	95	52	19	2	1	0	488	0.00	99.84	0.16	0.00	Beta	31	0.000
H10031100.CD53S	100	100	99	91	60	16	3	1	263	0.00	100.00	0.00	0.00	Beta	30	0.000
H11261725.CD53S	100	100	100	90	67	18	4	2	246	0.41	99.43	0.16	0.00	Beta	29	0.000
H11260120.CD53S	100	100	100	97	68	24	5	1	236	0.00	100.00	0.00	0.02	Beta	31	0.000
H11260620.CD53S	100	100	100	98	63	23	11	5	249	0.00	100.00	0.00	0.00	Beta	30	0.000
H09252400.CD53S	100	100	99	71	27	7	4	3	403	0.00	99.83	0.17	0.00	Beta	29	0.000
H10021915.CD53S	100	100	99	87	24	12	4	2	381	0.27	99.73	0.00	0.00	Beta	31	0.004
H10041220.CD53S	100	100	99	88	57	19	8	5	270	0.24	99.76	0.00	0.00	Beta	34	0.003
H11270600.CD53S	100	98	67	55	24	9	4	2	467	0.00	100.00	0.00	0.00	Beta	31	0.000
H11292000.CD53S	100	100	100	66	58	16	4	1	269	0.22	99.78	0.00	0.00	Beta	30	0.000
H11301130.CD53S	100	100	99	77	37	7	3	2	363	0.13	99.87	0.00	0.00	Beta	33	0.012
H09272035.CD53S	100	100	99	84	28	10	4	3	377	0.54	99.33	0.13	0.00	Beta	31	0.000
H10021600.CD53S	100	100	99	94	53	15	3	2	285	0.00	99.78	0.22	0.00	Beta	32	0.002
H10020030.CD53S	100	99	95	71	43	15	7	5	348	0.42	99.58	0.00	0.00	Beta	31	0.000
H11271330.CD53S	100	100	99	91	52	17	8	6	289	0.21	99.59	0.20	0.00	Beta	31	0.009
H11272215.CD53S	100	100	90	71	43	18	13	8	348	0.00	99.76	0.24	0.00	Beta	29	0.000
H11290130.CD53S	100	100	100	93	55	10	3	2	281	0.28	99.72	0.00	0.00	Beta	31	0.000
H11301830.CD53S	100	100	73	46	35	20	8	4	550	0.48	99.52	0.00	0.00	Beta	27	0.000
H11300030.CD53S	100	100	99	81	53	16	7	5	285	0.00	100.00	0.00	0.00	Beta	29	0.004

*Liquid Chromatographic Analyses, ASM I-151
**X-Ray Analyses, ASM I-4

Table 18: Analytical Results of Class 4 HMX Samples

Sample No.	Particle Size Distribution Percent Passing USS Sieve Nos.								Median Size (μ)	Purity*			%** α HMX	Poly- morph by IR	Long Impact (cm)	Crystal Acidity (%)
	8	12	20	35	50	100	200	325		% RDX	% HMX	% SEX				
H11271600.CD53S	100	100	100	45	20	17	3	0	531	0.00	99.71	0.29	0.00	Beta	31	0.000
H12010730.CD53S	100	98	67	55	27	8	6	5	464	0.00	100.00	0.00	0.00	Beta	30	0.000
H11280830.CD53S	100	99	84	30	12	6	5	1	626	0.97	99.03	0.00	0.00	Beta	33	0.003
H11281000.CD53S	100	100	41	10	5	3	2	1	968	0.40	99.60	0.00	0.00	Beta	30	0.002
H11300430.CD53S	100	100	33	19	15	6	3	2	1053	0.00	100.00	0.00	0.00	Beta	28	0.003
H12011010.CD53S	100	100	85	32	12	7	6	4	615	0.00	100.00	0.00	0.00	Beta	34	0.000

*Liquid Chromatographic Analyses, ASM I-151
**X-Ray Analyses, ASM I-4

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Table 19: Analytical Results of Class 5 HMX Samples

Sample No.	Particle Size Distribution Percent Passing USS Sieve Nos.								Median Size (μ)	Purity+			% α HMX ++	Polymorph morph. by IR	Long Impact (cm)	Crystal Acidity (%)
	8	12	20	35	50	100	200	325		% RDX	% HMX	% SEX				
H11141810.CD52/3S	–	–	–	–	–	–	–	100	–	–	–	–	–	Beta*	27.5	–
H11140300.CD52/3S	–	–	–	–	–	–	–	100	–	–	–	–	–	Beta*	31.4	–
H11152025.CD52/3S	–	–	–	–	–	–	–	100	–	–	–	–	–	Beta*	27.2	–
H11152215.CD52/3S	–	–	–	–	–	–	–	100	–	–	–	–	–	Beta*	27.5	–
H11150140.CD52/3S	–	–	–	–	–	–	–	100	–	–	–	–	–	Trace* Alpha	3?.1	–
H11150240.CD52/3S	–	–	–	–	–	–	–	100	–	–	–	–	–	Beta*	33.3	–
H11162230.CD52/3S	–	–	–	–	–	–	–	100	–	–	–	–	–	Trace* Alpha	30.0	–
H11191300.CD52/3S	–	–	–	–	–	–	–	99	–	0.00	100.00	0.00	0.00	Beta	33.?	0.005
H11192030.CD52/3S	–	–	–	–	–	–	–	100	–	0.00	100.00	0.00	0.00	Beta	31.8	0.000
H11190510.CD52/3S	–	–	–	–	–	–	–	100	–	1.62	98.21	0.18	0.00	Beta	27.6	0.003
H11201305.CD52/3S	–	–	–	–	–	–	–	98	–	0.22	99.72	0.06	0.00	Beta	29.6	0.000
H11202130.CD52/3S	–	–	–	–	–	–	–	99	–	0.82	99.18	0.00	0.00	Beta	30.0	0.000
H11200500.CD52/3S	–	–	–	–	–	–	–	100	–	1.39	98.46	0.15	0.00	Beta	30.0	0.006
H10020400.CD52S	–	–	–	–	–	–	–	100	–	1.11	98.88	0.00	0.01	Trace Alpha	33.2	0.000
H10031100.CD52S	–	–	–	–	–	–	–	100	–	1.01	98.72	0.0?	0.00	Beta	30.?	0.000
H10030400.CD52S	–	–	–	–	–	–	–	100	–	0.00	100.00	0.00	0.00	Beta	29.8	0.000
H11280730.CD52S	–	–	–	–	–	–	–	100	–	1.31	98.51	0.18	0.00	Beta	29.4	0.002
H11281230.CD52S	–	–	–	–	–	–	–	98	–	1.19	98.77	0.00	0.04	Trace Alpha	28.7	0.004
H11290745.CD52S	–	–	–	–	–	–	–	98	–	0.00	100.00	0.00	0.00	Beta	26.9	0.003
H11300745.CD52S	–	–	–	–	–	–	–	72	–	1.90	98.04	0.00	0.06	Trace Alpha	30.8	0.021
H12011000.CD52S	–	–	–	–	–	–	–	69	–	0.00	100.00	0.00	0.00	Beta	31.7	0.012

*Microscope
+Liquid Chromatographic Analyses, ASM I-151
++X-Ray Analyses, ASM I-4

Table 20: Analytical Results of Representative Samples of DMSO-Recrystallized HMX

Sample No.	Residual Solvent (Weight % DMSO)		VTS at 100°C for 48 hr. (cc gas/gr. Sample)	% Acetone Insolubles	% Inorganic Insolubles	Number of Particles on USSS Nos.		Melting Point (°C)
	Wet Sample	Dry Sample				40	60	
H11281000.CD53S	0.12	0.08	0.0592	0.00	0.00	0	0	281
H11300430.CD53S	0.46	0.11	0.0427	0.00	0.00	0	0	279
H12011010.CD53S	0.12	0.08	0.0672	0.00	0.00	0	0	279
H11280830.CD53S	0.20	0.07	0.0505	0.00	0.00	0	0	280
H09172400.CD53S	0.26	0.08	0.0461	0.00	0.00	0	0	281
H11271600.CD53S	0.10	0.05	0.0253	< 0.03	0.00	0	0	280
H10030400.CD53S	0.44	0.06	0.0352	< 0.03	0.00	0	0	281
H11280530.CD53S	0.29	0.02	0.0429	< 0.03	0.00	0	0	283
H11291300.CD53S	1.43	0.08	0.0369	< 0.03	0.00	1 Fly Ash 1 Metal	1 Fly Ash	281
H11271330.CD53S	0.67	0.01	0.0223	0.00	0.00	-		278
H11290130.CD53S	0.87	0.04	0.0336	0.00	0.00	0	0	281
H08202400.CD53S	0.36	0.08	0.0368	0.00	0.00	0	0	278
H08212400.CD53S	0.10	0.07	0.0241	0.00	0.00	0	0	281
H09252400.CD53S	0.16	0.14	0.0429	0.00	0.00	0	0	280
H09272035.CD53S	0.09	0.04	0.0244	0.00	0.00	0	0	279
H10021915.CD53S	0.08	0.08	0.0413	0.00	0.00	0	0	279
H10041650.CD53S	0.08	0.04	0.0715	0.00	0.00	2 Fly Ash	0	280
H11261725.CD53S	0.11	0.07	0.1033	0.00	0.00	0	0	280
H09262400.CD53S	0.08	0.04	0.0220	0.00	0.00	0	0	280
H11140300.CD53/3S	0.19	0.16	0.0530	< 0.03	0.00	0	0	282
H11162230.CD52/3S	0.43	0.40	0.0284	0.00	0.00	0	0	278
H11192030.CD52/3S	0.20	0.01	0.0387	0.00	0.00	0	0	279
H11200500.CD52/3S	0.46	< 0.01	0.0210	0.00	0.00	0	0	280
H11201305.CD52/3S	0.43	0.10	0.3338	0.00	0.00	0	0	278
H11290745.CD52S	2.56	0.08	0.0361	0.00	0.00	0	0	281

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Table 21: Material Balance for 15 Lb/Hr HMX/CNCPR
Crystallization System
(Reference Figure 26)

System	Vessel Designation	Stream No.	Flow Rate (lb./hr.)	Percent Solids	Solids Composition Mass Fraction		Liquid Composition Mass Fraction			
					HMX	RDX	HMX	RDX	DMSO	Water
Feed Preparation	Nutsche	1	35.9	50.0	0.492	0.008	0	0	0	1.000
	Drum	2	11.1	0	-	-	0	0	1.000	0
	CD-50	3	64.9	7.6	1.000	0	0.064	0.016	0.736	0.184
	-	4	9.0	0	-	-	0	0	0	1.000
	-	5	120.9	22.8	0.218	0.010	0	0	0.596	0.404
	CB-10	GW-1	30.0	0	-	-	0	0	0	1.000
	CB-50	6	150.9	18.3	0.175	0.008	0	0	0.448	0.552
	CB-11	GW-2	30.0	0	-	-	0	0	0	1.000
	CB-22	7	91.9	0	-	-	0	0	0.0001	0.9999
	CE-51	8	150.0	0	-	-	0	0	0.005	0.995
	CE-51	8A	104.3	0	-	-	0	0	0.005	0.995
	CE-11	GW-3	30.0	0	-	-	0	0	0	1.000
Crystallization (CNCPR) One-Crystallizer	CB-51	9A	89.0	0	-	-	0.297	0.014	0.620	0.069
	-	9B & 9C	0	-	-	-	-	-	-	-
	-	DW-1	0	-	-	-	-	-	-	-
	CB-52	10	582.7	0	-	-	0.158	0.015	0.662	0.155
	-	11	0	-	-	-	-	-	-	-
	-	12	486.0	7.6	1.000	0	0.064	0.016	0.736	0.184
	CB-12	13	50.3	7.6	1.000	0	0.064	0.016	0.736	0.184
	CC-50 & CC-51	GW-4	7.7	0	-	-	0	0	0	1.000
	CC-50 & CC-51	14	46.4	38.5	1.000	0	0.064	0.016	0.736	0.184
		14A	0	-	-	-	-	-	-	-
Recycle Solvent Processing	CD-01	15	24.2	7.6	1.000	0	0.064	0.016	0.736	0.184
	CD-50	16	9.6	7.6	1.000	0	0.064	0.016	0.736	0.184
	-	DW-2	13.6	0	-	-	0	0	0	1.000
	CF-51	17	23.2	6.2	0.900	0.100	0	0	0.300	0.700
	-	WW-1	0.5	0	-	-	0	0	0	1.000
	CF-52	18	2.9	50.0	0.900	0.100	0	0	0.010	0.990
	CF-52	19	20.8	0.5	0.900	0.100	0	0	0.301	0.699
Product Filtration and Washing	-	SW-1	5.0	0	-	-	0	0	0	1.000
	CD-02	20	27.2	60.7	0.994	0.006	0	0	0.426	0.574
	-	21	20.0	75.0	0.998	0.002	0	0	0.272	0.728
	-	WW-2	125.0	0	-	-	0	0	0	1.000
	-	WW-3	5.0	0	-	-	0	0	0	1.000
	CD-53	22	17.6	85.0	0.998	0.002	0	0	0.014	0.986
	CD-53	23	132.4	0	-	-	0	0	0.010	0.990
	-	WW-4	1.0	0	-	-	0	0	0	1.000
	CD-52	24	2.0	50.0	0.950	0.050	0	0	0.010	0.990
	CD-52	25	156.2	0.3	0.950	0.050	0	0	0.025	0.975

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Table 22: Material Balance fo 30 Lb./Hr HMX/CNCPR
Crystallization System
(Reference Figure 26)

System	Vessel Designation	Stream No.	Flow Rate (lb./hr.)	Percent Solids	Solids Composition Mass Fraction		Liquid Composition Mass Fraction			
					HMX	RDX	HMX	RDX	DMSO	Water
Feed Preparation	Nutsche	1	77.0	50.0	0.984	0.016	0	0	0	1.000
	Drum	2	27.3	0	-	-	0	0	1.000	0
	CD-50	3	155.9	5.8	1.000	0	0.127	0.013	0.737	0.123
	-	4	22.0	0	-	-	0	0	0	1.000
	-	5	283.2	23.2	1.000	0	0.012	0.012	0.626	0.362
	CB-10	GW-1	30.0	0	-	-	0	0	0	1.000
	CB-50	6	313.2	21.8	0.963	0.037	0	0	0.556	0.444
	CB-11	GW-2	30.0	0	-	-	0	0	0	1.000
	CB-22	7	123.6	0	-	-	0	0	0.0001	0.9999
	CE-51	8	150.0	0	-	-	0	0	0.010	0.990
	CE-51	8A	164.5	0	-	-	0	0	0.010	0.990
	CE-11	GW-3	30.0	0	-	-	0	0	0	1.000
Crystallization (CNCPR)	CB-51	9A	219.6	-	-	-	0.300	0.011	0.620	0.069
		9B & 9C	0	-	-	-	-	-	-	-
Two-Crystalizer		DW-1	0	-	-	-	-	-	-	-
	CB-52	10	596.2	0	-	-	0.199	0.012	0.676	0.113
	CB-52	11	596.2	0	-	-	0.199	0.012	0.676	0.113
	-	12	965.1	5.8	1.000	-	0.127	0.013	0.737	0.123
	-	13	134.5	5.8	1.000	-	0.127	0.013	0.737	0.123
	CB-12	GW-4	7.7	0	-	-	0	0	0	1.000
	CC-50 & CC-51	14	92.8	37.3	1.000	0	0.127	0.013	0.737	0.123
		14A	0	-	-	-	-	-	-	-
Recycle Solvent Processing	CD-01	15	48.4	5.6	1.000	0	0.127	0.013	0.737	0.123
	CD-50	16	26.0	5.8	1.000	0	0.127	0.013	0.737	0.125
	-	DW-2	39.1	0	-	-	0	0	0	1.000
	CF-51	17	65.1	7.6	0.937	0.063	0	0	0.300	0.700
	-	WW-1	1.7	0	-	-	0	0	0	1.000
	CF-52	18	9.3	50.0	0.937	0.063	0	0	0.010	0.990
	CF-52	19	57.5	0.5	0.937	0.063	0	0	0.315	0.685
	-	SW-1	10.0	0	-	-	0	0	0	1.000
Product Filtration and Washing	-	20	54.4	61.5	0.997	0.003	-	-	0.445	0.555
	CD-02	21	40.0	75.0	0.998	0.002	0	0	0.272	0.728
	-	WW-2	125.0	0	-	-	0	0	0	1.000
	-	WW-3	10.0	0	-	-	0	0	0	1.000
	CD-53	22	35.2	85.0	0.998	0.002	0	0	0.014	0.986
	CD-53	23	139.8	0	-	-	0	0	0.019	0.981
	-	WW-4	2.5	0	-	-	0	0	0	1.000
	CD-52	24	5.0	50.0	0.971	0.029	0	0	0.010	0.990
	CD-52	25	161.9	0.6	0.971	0.029	0	0	0.050	0.950

Table 23: Material Balance for Nominal ? lb/hr
HMX/Dilution Crystallization System
(Reference Figure 26)

System	Vessel Designation	Stream No.	Flow Rate (lb./hr.)	Percent Solids	Solids Composition Mass Fraction HMX	RDX	Liquid Composition Mass Fraction HMX	RDX	DMSO	Water
Feed Preparation	Nutsche	1	14.2	50.0	0.984	0.016	0	0	0	1.000
	Drum	2	21.8	0	-	-	0	0	1.000	0
	-	3	0	-	-	-	-	-	-	-
	-	4	3.0	0	-	-	0	0	0	1.000
	CA-52	5	39.0	17.8	1.000	0	0.001	0.004	0.680	0.315
	CB-1C	GW-1	30.0	0	-	-	0	0	0	1.000
	CB-50	6	69.0	10.3	0.984	0.016	0	0	0.352	0.648
	CB-11	GW-2	30.0	0	-	-	0	0	0	1.000
	CE-11	GW-3	30.0	0	-	-	0	0	0	1.006
	CB-22	7	66.7	0	-	-	0	0	0.0001	0.9999
	CE-51	8	50.0	0	-	-	0	0	0.007	0.993
	CE-51	8A	126.6	0	-	-	0	0	0.007	0.993
	-	9A & 9B	0	-	-	-	-	-	-	-
Crystallization (Dilution)	CB-51	9C	32.3	0	-	-	0.216	0.004	0.675	0.105
	-	DW-1	11.7	0	-	-	0	0	0	1.000
One-Crystallizer	-	10	0	-	-	-	-	-	-	-
	-	11	0	-	-	-	-	-	-	-
	-	12	0	-	-	-	-	-	-	-
	-	13	0	-	-	-	-	-	-	-
	-	GW-4	0	-	-	-	-	-	-	-
	-	14	0	-	-	-	-	-	-	-
	CC-51	14A	44.0	16.1	0.984	0.016	0	0	0.495	0.505
Unused Equipment	-	15	0	-	-	-	-	-	-	-
	-	16	0	-	-	-	-	-	-	-
	-	DW-2	0	-	-	-	-	-	-	-
	-	17	0	-	-	-	-	-	-	-
	-	WW-1	0	-	-	-	-	-	-	-
	-	18	0	-	-	-	-	-	-	-
	-	19	0	-	-	-	-	-	-	-
	-	SW-1	0	-	-	-	-	-	-	-
	-	20	0	-	-	-	-	-	-	-
Product Filtration And Washing	CD-02	21	7.0	40.0	0.984	0.016	0	0	0.300	0.700
	CD-03	21A	5.3	40.0	0.984	0.016	0	0	0.005	0.995
	-	WW-2	50.0	0	-	-	0	0	0	1.000
	-	WW-3	30.0	0	-	-	0	0	0	1.000
	CD-53	22	1.8	38.9	0.984	0.016	0	0	0.016	0.984
	CD-53	23	79.9	0	-	-	0	0	0.016	0.984
	-	WW-4	60.0	0	-	-	0	0	0	1.000
	CD-52	24	10.0	40.0	0.984	0.016	0	0	0.016	0.984
	CD-52	25	137.0	0.2	0.984	0.016	0	0	0.127	0.873

Table 24. Material Balance for 20 Lb/Hr RDX/CNCPR Crystallization System (Reference Figure 26)

System	Vessel Designation	Stream No.	Flow Rate (lb./hr.)	Percent Solids	Solids Composition Mass Fraction		Liquid Composition Mass Fraction			
					RDX	HMX	RDX	HMX	DMSO	Water
Feed Preparation	Nutsche	1	31.9	75.0	0.937	0.063	0	0	0	1.000
	Drum	2	14.4	0	–	–	0	0	1.000	0
	CD-50	3	83.6	6.0	0.979	0.021	0.058	0.052	0.731	0.159
	–	4	10.0	0	–	–	0	0	0	1.000
	CA-52	5	139.9	25.4	0.833	0.167	0.020	0	0.688	0.292
	CB-10	GW-1	30.0	0	–	–	0	0	0	1.000
	CB-50	6	169.9	22.1	0.846	0.154	0	0	0.543	0.457
	CB-11	GW-2	30.0	0	–	–	0	0	0	1.000
	CB-22	7	82.6	0	–	–	0	0	0.0001	0.9999
	CE-51	8	150.0	0	–	–	0	0	0.005	0.995
	CE-51	8A	97.0	0	–	–	0	0	0.005	0.995
	CE-11	GW-3	30.0	0	–	–	0	0	0	1.000
Crystallization (CNCPR)	CB-51	9A	117.3	0	–	–	0.271	0.049	0.612	0.068
	–	9B & 9C	0	–	–	–	–	–	–	–
One-Crystallizer	–	DW-1	0	–	–	–	–	–	–	–
	CB-52	10	586.3	0	–	–	0.142	0.050	0.663	0.145
	–	11	0	–	–	–	–	–	–	–
	–	12	461.3	6.0	0.979	0.021	0.058	0.052	0.731	0.159
	–	13	63.0	6.0	0.979	0.021	0.058	0.052	0.731	0.159
	CB-12	GW-4	7.7	0	–	–	0	0	0	1.000
	CC-50 & CC-51	14	61.5	37.4	0.979	0.021	0.058	0.052	0.731	0.159
	CC-50 & CC-51	14A	0	–	–	–	–	–	–	–
Recycle Solvent Processing	CD-01	15	31.8	6.0	0.979	0.021	0.058	0.052	0.731	0.159
	CD-50	16	11.7	6.0	0.979	0.021	0.058	0.052	0.731	0.159
	–	DW-2	17.0	0	–	–	0	0	0	1.000
	CF-51	17	28.7	6.6	0.687	0.313	0	0	0.300	0.700
	–	WW-1	0.8	0	–	–	0	0	0	1.000
	CF-52	18	3.8	50.0	0.687	0.313	0	0	0.010	0.990
	CF-52	19	25.7	0.5	0.687	0.313	0	0	0.239	0.761
Product Filtration And Washing	–	SW-1	7.0	0	–	–	0	0	0	1.000
	–	20	36.7	59.9	0.959	0.041	0	0	0.430	0.570
	CD-02	21	26.7	74.9	0.980	0.020	0	0	0.206	0.794
	CD-03	21A	0	–	–	–	–	–	–	–
	–	WW-2	125.0	0	–	–	0	0	0	1.000
	–	WW-3	7.0	0	–	–	0	0	0	1.000
	CD-53	22	23.5	85.1	0.980	0.020	0	0	0.010	0.990
	CD-53	23	134.4	0	–	–	0	0	0.010	0.990
	–	WW-4	2.0	0	–	–	0	0	0	1.000
	CD-52	24	3.0	50.0	0.750	0.250	0	0	0.010	0.990
	CD-52	25	159.0	0.3	0.250	0.250	0	0	0.036	0.964

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Table 25. Material Balance for 35 Lb/Hr RDX/CNCPR Crystallization System
(Reference Figure 26)

System	Vessel Designation	Stream No.	Flow Rate (lb./hr.)	Percent Solids	Solids Composition Mass Fraction		Liquid Composition Mass Fraction			
					RDX	HMX	RDX	HMX	DMSO	Water
Feed Preparation	Nutsche Drum	1	59.2	75.0	0.927	0.073	0	0	0	1.000
		2	29.8	0	–	–	0	0	1.000	0
	CD-50	3	172.6	5.8	1.000	0	0.068	0.077	0.736	0.119
		4	10.0	0	–	–	0	0	0	1.000
	CA-52	5	271.6	25.0	0.824	0.176	0.031	0.019	0.733	0.217
	CB-10	GW-1	30.0	0	–	–	0	0	0	1.000
	CB-50	6	301.6	0	–	–	0.206	0.053	0.495	0.246
	CB-11	GW-2	30.0	0	–	–	0	0	0	1.000
	CB-22	7	87.7	0	–	–	0	0	0.0001	0.9999
	CE-51	8	150.0	0	–	–	0	0	0.008	0.992
	CE-51	8A	110.5	0	–	–	0	0	0.008	0.992
	CE-11	GW-3	30.0	0	–	–	0	0	0	1.000
	CB-51	9A	243.9	0	–	–	0.235	0.065	0.612	0.068
Crystallization (CNCPR) Two-Crystallizer		9B & 9C	0	–	–	–	–	–	–	–
		DW-1	0	–	–	–	–	–	–	–
	CB-52	10	596.6	0	–	–	0.148	0.071	0.671	0.110
		11	596.7	0	–	–	0.148	0.071	0.671	0.110
		12	941.7	5.8	1.000	0	0.068	0.077	0.736	0.119
		13	143.6	5.8	1.000	0	0.068	0.077	0.736	0.119
	CB-12	GW-4	7.7	0	–	–	0	0	0	1.000
	CC-50 & CC-51	14	108.0	37.2	1.000	0	0.068	0.077	0.736	0.119
	CC-50 & CC-51	14A	0	–	–	–	–	–	–	–
Recycle Solvent Processing	CD-01	15	56.6	5.8	1.000	0	0.068	0.077	0.736	0.119
	CD-50	16	27.6	5.8	1.000	0	0.068	0.077	0.736	0.119
		DW-2	41.4	0	–	–	0	0	0	1.000
	CF-51	17	69.0	7.8	0.626	0.374	0	0	0.300	0.700
		WW-1	2.0	0	–	–	0	0	0	1.000
	CF-52	18	10.2	50.0	0.626	0.374	0	0	0.010	0.990
	CF-52	19	60.9	0.5	0.626	0.374	0	0	0.313	0.687
Product Filtration And Washing		SW-1	13.6	0	–	–	0	0	0	1.000
	CD-02	20	65.0	60.0	0.967	0.033	0	0	0.430	0.570
		21	46.7	74.9	0.980	0.020	0	0	0.206	0.794
		WW-2	125.0	0	–	–	0	0	0	1.000
		WW-3	12.2	0	–	–	0	0	0	1.000
	CD-53	22	41.1	85.2	0.980	0.020	0	0	0.010	0.990
	CD-53	23	142.8	0	–	–	0	0	0.016	0.984
		WW-4	4.0	0	–	–	0	0	0	1.000
	CD-52	24	6.0	50.0	0.707	0.293	0	0	0.010	0.990
	CD-52	25	166.3	0.6	0.707	0.293	0	0	0.060	0.940

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Table 26: Material Balance for Nominal 28 Lb/Hr
RDX/Dilution Crystallization System
(Reference Figure 26)

System	Vessel Designation	Stream No.	Flow Rate (lb./hr.)	Percent Solids	Solids Composition Mass Fraction		Liquid Composition Mass Fraction			
					RDX	HMX	RDX	HMX	DMSO	Water
Feed Preparation	Nutsche	1	37.3	75.0	0.930	0.070	0	0	0	1.000
	Drum	2	79.9	0	-	-	0	0	1.000	0
	-	3	0	-	-	-	-	-	-	-
	-	4	12.0	0	-	-	0	0	0	1.000
	CA-52	5	129.2	17.2	1.000	0	0.035	0.020	0.746	0.199
	CB-10	GW-1	30.0	0	-	-	0	0	0	1.000
	CB-50	6	159.2	17.6	0.930	0.070	0	0	0.609	0.391
	CB-11	GW-2	30.0	0	-	-	0	0	0	1.000
	CB-22	7	72.6	0	-	-	0	0	0	1.000
	CE-51	8	100.0	0	-	-	0	0	0.0001	0.9999
	CE-51	8A	163.6	0	-	-	0	0	0.003	0.997
	CE-11	GW-3	30.0	0	-	-	0	0	0.003	0.997
Crystallization (Dilution)	-	9A	0	-	-	-	-	-	-	-
Two-Crystallizer	CB-51	9B	58.3	0	-	-	0.223	0.017	0.685	0.075
	CB-51	9C	58.3	0	-	-	0.223	0.017	0.685	0.075
	-	DW-1	43.6	0	-	-	0	0	0	1.000
	-	DW-1A	43.7	-	-	-	0	0	0	1.000
	-	10	0	-	-	-	-	-	-	-
	-	11	0	-	-	-	-	-	-	-
	-	12	0	-	-	-	-	-	-	-
	-	13	0	-	-	-	-	-	-	-
	-	GW-4	0	-	-	-	-	-	-	-
	-	14	0	-	-	-	-	-	-	-
Product Filtration And Washing	CC-50 & CC-51	14A	203.9	13.7	0.930	0.070	0	0	0.454	0.546
	CD-02	21	7.0	40.0	0.930	0.070	0	0	0.200	0.800
	CD-03	21A	1.8	38.9	0.930	0.070	0	0	0.002	0.998
	-	WW-2	100.0	0	-	-	0	0	0	1.000
	-	WW-3	60.0	0	-	-	0	0	0	1.000
	CD-53	22	4.2	50.0	0.930	0.070	0	0	0.020	0.980
	CD-53	23	161.0	0	-	-	0	0	0.005	0.995
	-	WW-4	120.0	0	-	-	0	0	0	1.000
	CD-52	24	49.0	50.0	0.930	0.070	0	0	0.020	0.980
	CD-52	25	367.9	0.2	0.930	0.070	0	0	0.215	0.785

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Table 27. Material Balance for Nominal 45 Lb/Hr RDX/CNCPR-MOD-3 Crystallization System (Reference Figure 26)

System	Vessel Designation	Stream No.	Flow Rate (lb./hr.)	Percent Solids	Solids Composition Mass Fraction RDX	Solids Composition Mass Fraction HMX	Liquid Composition Mass Fraction RDX	Liquid Composition Mass Fraction HMX	Liquid Composition Mass Fraction DMSO	Liquid Composition Mass Fraction Water
Feed Preparation	Nutsche	1	74.8	75.0	0.928	0.072	0	0	0	1.000
	Drum	2	36.2	0	-	-	0	0	1.000	0
	CD-50	3	205.9	4.8	0.996	0.004	0.067	0.078	0.739	0.116
	-	4	25.0	0	-	-	0	0	0	1.000
	CA-52	5	316.9	22.4	0.883	0.117	0.049	0.046	0.736	0.169
	CB-10	GW-1	30.0	0	-	-	0	0	0	1.000
	CB-50	6	346.9	24.5	0.771	0.229	0.036	0.002	0.691	0.271
	CB-11	GW-2	30.0	0	-	-	0	0	0	1.000
	CB-22	7	81.2	0	-	-	0	0	0.0001	0.9999
	CE-51	8	150.0	0	-	-	0	0	0.030	0.970
	CE-51	8A	124.9	0	-	-	0	0	0.030	0.970
	CE-11	GW-3	30.0	0	-	-	0	0	0	1.000
Crystallization (CNCPR-MOD-3) One-Crystallizer	-	9A	0	-	-	-	-	-	-	-
	-	9B	0	-	-	-	-	-	-	-
	CB-51	9C	295.7	0	-	-	0.253	0.067	0.612	0.068
	-	DW-1	8.2	0	-	-	0	0	0	1.000
	-	DW-1A	0	-	-	-	-	-	-	-
	CC-50 & CC-51	14	139.5	16.6	0.996	0.004	0.067	0.078	0.739	0.116
	-	14A	0	-	-	-	-	-	-	-
Recycle Solvent Processing	CD-01	15	62.0	4.8	0.996	0.004	0.067	0.078	0.739	0.116
	CD-50	16	20.5	4.8	0.996	0.004	0.067	0.078	0.739	0.116
	-	DW-2	31.4	0	-	-	0	0	0	1.000
	CF-51	17	51.9	7.4	0.594	0.406	0	0	0.300	0.700
	-	WW-1	2.0	0	-	-	0	0	0	1.000
	CF-52	18	7.0	50.0	0.594	0.406	0	0	0.010	0.990
	CF-52	19	64.0	0.5	0.594	0.406	0	0	0.226	0.774
	-	SW-1	20.0	0	-	-	0	0	0	1.000
Product Filtration And Washing	CD-02	20	97.5	53.6	0.951	0.049	0	0	0.482	0.518
	CD-02	21	75.0	60.0	0.960	0.040	0	0	0.277	0.723
	-	21A	0	-	-	-	-	-	-	-
	-	WW-2	125.0	0	-	-	0	0	0	1.000
	-	WW-3	20.0	0	-	-	0	0	0	1.000
	CD-53	22	56.3	80.0	0.960	0.040	0	0	0.012	0.988
	CD-53	23	163.7	0	-	-	0	0	0.050	0.950
	-	WW-4	6.0	0	-	-	0	0	0	1.000
	CD-52	24	12.0	50.0	0.900	0.100	0	0	0.010	0.990
	CD-52	25	166.5	0.8	0.900	0.100	0	0	0.108	0.892

Table 28. Equipment Performance Summary

Equipment Item	Equipment Performance Prior To Modification	Problem Areas	Solutions Attempted	Equipment Performance After Modification
Evaporator Feed and Circulation Pumps, CB-10 and CB-11	Poor	High water flow to packing gland caused low DMSO concentration in feed solution.	Reduced packing gland water flow.	Fair - fluctuations in water pressure at the low flow rates caused pumps to stop unexpectedly resulting in process upsets.
Slurry Mixing System	Weigh system did not function.	Deterioration of hydraulic load cells.	1. Maintenance-repair. 2. Procedure modification using stick measurements.	1. Did not function. 2. Fair - limited accuracy.
Evaporator Feed Circulation System	Poor	Evaporator feed line plugged.	Abandoned use of steam-jacket on the circulating line; discontinued heating evaporator feed.	Good - no more plugs experienced.
Evaporator Feed Valve	Fair	Evaporator feed valve plugged between valve and tank after long operating period.	1. Valve removed and cleaned. 2. Raised evaporator temperature.	Fair - valve continued to plug occasionally after long operating periods.
Dissolver Feed System	Poor	On/off feeding of the dissolver caused plugging of the feed line at the point where the feed enters the dissolver (below the liquid level). (Note: Feed rate fluctuated due partially to inadequate control by feed valve but also due to plugging by	1. Feed point to dissolver changed to a point above the liquid level. 2. Steam-jacketed, stainless steel feed line was replaced with a	1. Fair-plugging occurred occasionally at other points in the feed line (usually low point or in valves or tee's). 2. Good-plugging occurred with much lower frequency and usually only

(Continued)

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Table 28: Equipment Performance Summary (Continued)

Equipment Item	Equipment Performance Prior To Modification	Problem Areas	Solutions Attempted	Equipment Performance After Modification
		solids in the feed valve orifice.)	flexible polyethylene tubing system.	at the point where the polyethylene tubing joined the feed valve.
Dissolver/Crystallizer Circulating Pump. CB-12	Poor	1. High water flow to packing gland caused low DMSO concentration in CNCPR systems.	1. Reduced packing gland water flow.	1. Fair-fluctuation in water pressure at the low flow-rates caused pump to stop unexpectedly resulting in process upsets.
		2. Low water flow to packing gland also reduced DMSO concentration.	2. Increased DMSO concentration in feed from evaporator.	2. Fair-on/off feeding caused unsteady state conditions in dissolver.
Circulation Line Drain Valves (Evaporator Feed System, Evaporator Circulation Line, Dissolver/Crystallizer Circulation Line)	Poor	Drain valves plugged during operation of the systems in the nozzle between the circulation line and the drain valve.	Replaced the elbow from the drain nozzle to the valve with a straight nozzle.	Poor-plugging continued, problem never solved; will require redesign of a line drainage system.
Crystallizer Cooling System	Poor to Fair	Cooling capacity too low; resulted in low circulation rates at design temperature.	Water flow meters removed; increased cooling water supply pressure.	Fair-little noticeable effect.
Crystallizer Agitators	Poor	Poor agitation; product removal difficult; inefficient crystal classification.	Reversed agitator rotation.	Fair - product easier to remove, better crystal classification.

(Continued)

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Table 28: Equipment Performance Summary (Continued)

Equipment Item	Equipment Performance Prior To Modification	Problem Areas	Solutions Attempted	Equipment Performance After Modification
Crystallizer Product Removal Valves	Fair	Product drop valves plugged after long operating period; leaked during operation.	Removed and cleaned.	Fair - continued to plug after long operating periods; leaks became worse with time.
Crystallizer Product Removal Timing System	Poor	Product removal on timed cycle only caused large variations in the amount of material removed during one cycle.	Added capacitance probes to circuit which allowed removal of a constant, yet variable, volume during each cycle.	Good-much better system; gave no more problems.
Initial Solids/Liquid Separation System	Poor	Originally used Sweco screener; screen plugged resulting in poor separation and eventual plugging of other screeners.	1. Replaced 270 mesh screen with a 120 mesh. 2. Replaced screener with decant system.	1. Poor - same result. 2. Good - effective solids/liquid separation without further problems.
Second Wash, Size-Separation Screener	Poor	Product would not discharge from screens due to low water flow used.	Abandoned size separation idea; replaced screen stack with single 325 mesh screen.	Good - screen used only for product washing.
Mother Liquor Filter	Poor	Filter cloth blinded immediately on contact with saturated DMSO/ explosives solution.	By-passed filter; transferred mother liquor with solids to recycle DMSO receiver.	N/A

(Continued)

Table 28:   Equipment Performance Summary   (Continued)

Equipment Item	Equipment Performance Prior To Modification	Problem Areas	Solutions Attempted	Equipment Performance After Modification
Recycle DMSO Filter	Poor	Filter cloth blinded on contact with mother liquor; filtration extremely slow.	Used recycle solvent directly to make new feed batches; reduced demand on filter.	Good
Evaporator Pressure Control System	Poor	Large fluctuations in evaporator pressure; uncontrollable.	1. Changed pressure control point from evaporator to top of column. 2. Changed pressure control point from top of column to the vacuum supply line above the column condenser.	1. Fair – fluctuations less but very unsteady except at constant load conditions. 2. Good – fluctuations were reduced during variable load and were very low during constant load.
Steam Flow Totalizers for Evaporator and Dissolver	Poor – did not function.	Bad elements, improper installation, or bad design suspected.	Maintenance-repair and recalibration on several occasions.	Poor – did not function.
Water Flow Totalizers for Crystallizers	Poor	Flow element restricted the cooling water flow to the crystallizers so that supply pressure was too low.	Removed elements from cooling circuit.	Fair – some improvement in pressure.
Liquid Chromatograph (Process Control Unit at Pilot Plant)	Poor	1. Mechanical problems developed in almost all areas of the system including pumping, heating, and detection.	1. Maintenance-repair and recalibration on many occasions	1. Poor – problems continued to recur with even greater frequency as time progressed.

(Continued)

Table 28: Equipment Performance Summary (Continued)

Equipment Item	Equipment Performance Prior To Modification	Problem Areas	Solutions Attempted	Equipment Performance After Modification
			2. Replacement of bad detector; replacement of high-pressure pump and related controls; finally replacement of entire unit.	2. Fair – new unit better but eventually failed in same ways. Entire system was unreliable and results of sampling were sometimes erroneous and always in doubt.

Table 29: Analytical Results of DMSO-Recrystallized RDX Test Products

	Compositions		
	A-5	A-3	C-4
% RDX	98.84	91.11	89.94
% Stearic Acid	1.16	-	-
% H_2O	0.01	0.01	0.014
Bulk Density	1.03	0.82	-
% Binder	-	-	10.06
Plasticity	-	-	0.054
% Wax	-	8.89	-
Acidity	-	0	-
Foreign Matter	None	None	-
Insoluble Particles	None	None	None
Granulation:			
% Passing USSS No. 6	-	100	100
12	100	-	-
100	-	0.1	0.3
200	0.2	-	-

	Composition B	70/30 Cyclotol
% RDX	59.91	70.58
% TNT	38.91	29.42
% Wax	1.18	-
% Water	0.06	0.11
Viscosity, Sec.	4.4	9.5
Insolubles	None	None
Foreign Matter	None	None

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Table 30: Analytical Results of DMSO-Recrystallized
HMX Test Products

	70/30 Octol	75/25 Octol
% HMX	69.38	73.34
% TNT	30.62	26.66
% Water	0.10	0.13
Viscosity, sec.	11.12	14.1
Insoluble Particles, USSS No. 60	None	None
% Insolubles	0.00	0.00
Foreign Matter	None	None

APPENDIX B

FIGURES

121

122
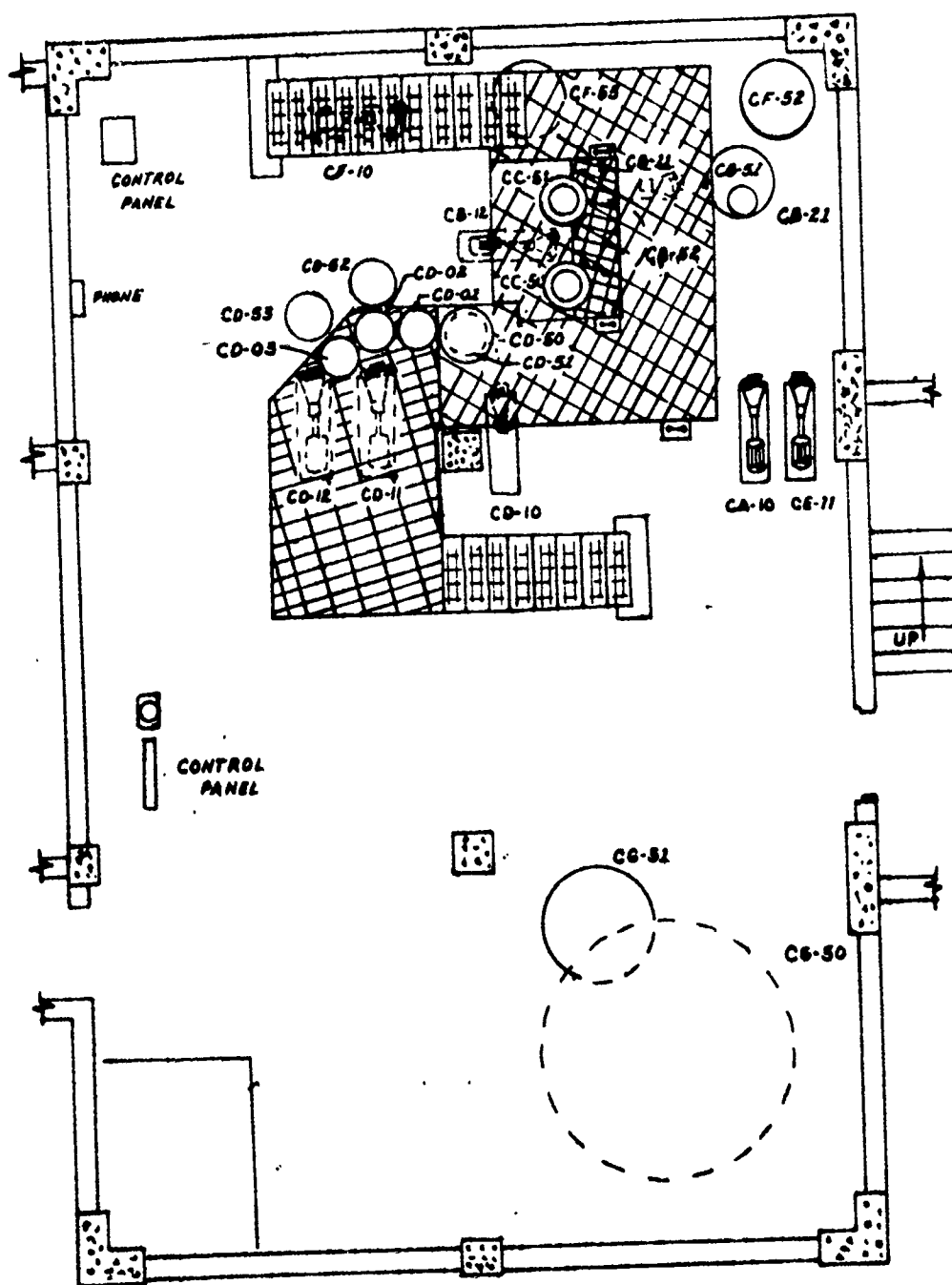
1st Floor Layout

FIGURE 2

JRC

Elevator

Control Rm.

CB-10　CB-50

CA-51

CA-50

CB-21　CB-22

CB-22

CF-51

CF-50

CA-52

UP

UP

DOWN

TABLE

SINK

CG-50
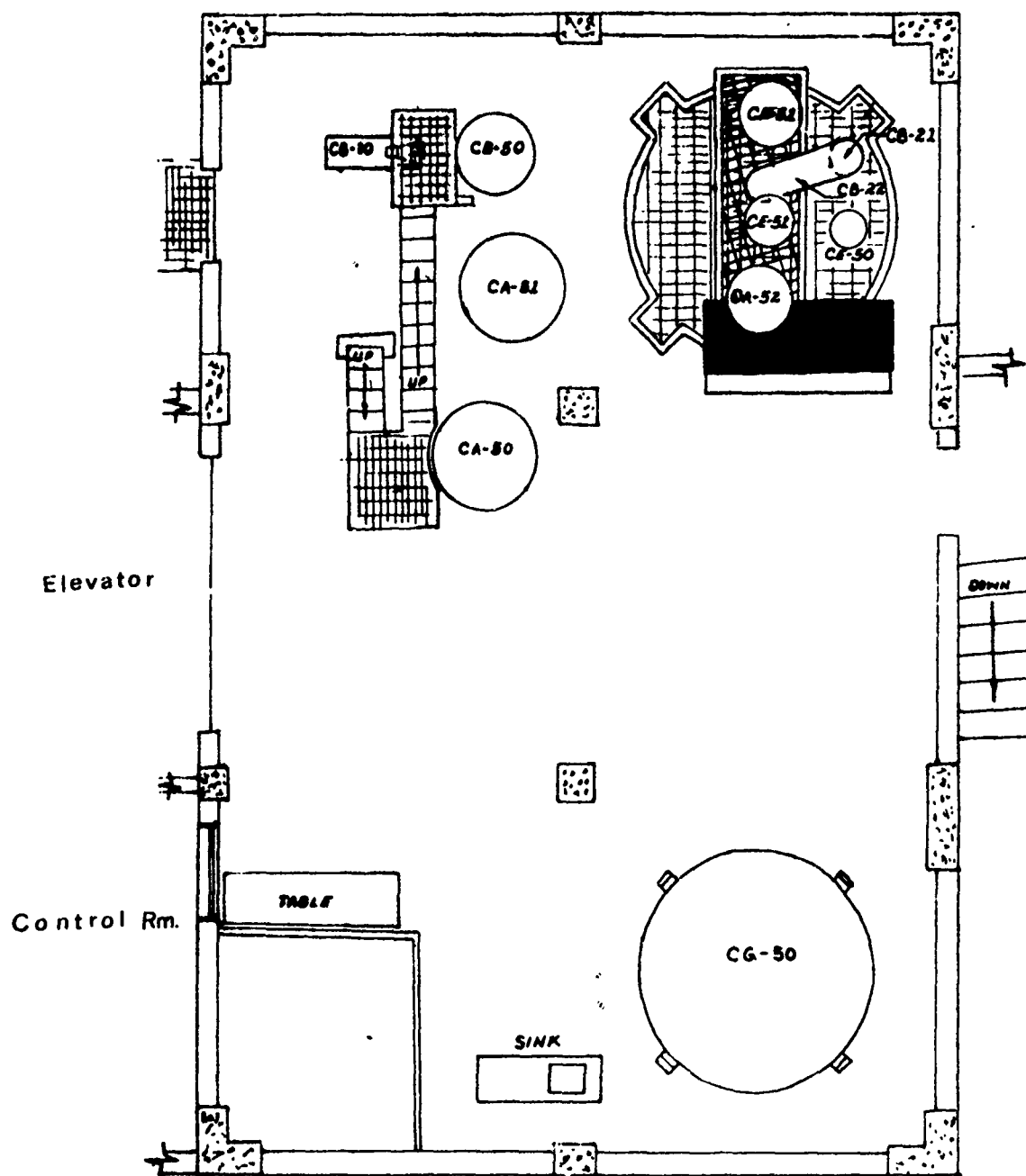
$\equiv$ 2nd Floor Layout $\equiv$

FIGURE 3

JRC

PROCESS AREA

DUAL PANE NON-OPENING WINDOW — 30" X 30" PLEXIGLAS

WINDOWS CAULKED AND BOLT. SHUT.

DESK

CONTROL & RECORDING ROOM

COMP

RECORD PANEL

ELEVATOR SHAFT

2'-6"

23'-6"

3'

9'-2"

WINDOWS CAULKED

SAFETY CHUTE

SAFETY CHUTE

≡ Control Room ≡

FIGURE 4

SCALE: 3/16" = 1'
SBQ 10/13/72

DMSO FROM DRUMS

RDX/HMX From NUTSCHES

CA-52

From
Recycle DMSO

HOLDING Tk. CF-53

WI
6

FCV
14

HC
14

To Evap. Feed Tk
CB-50

CA-10
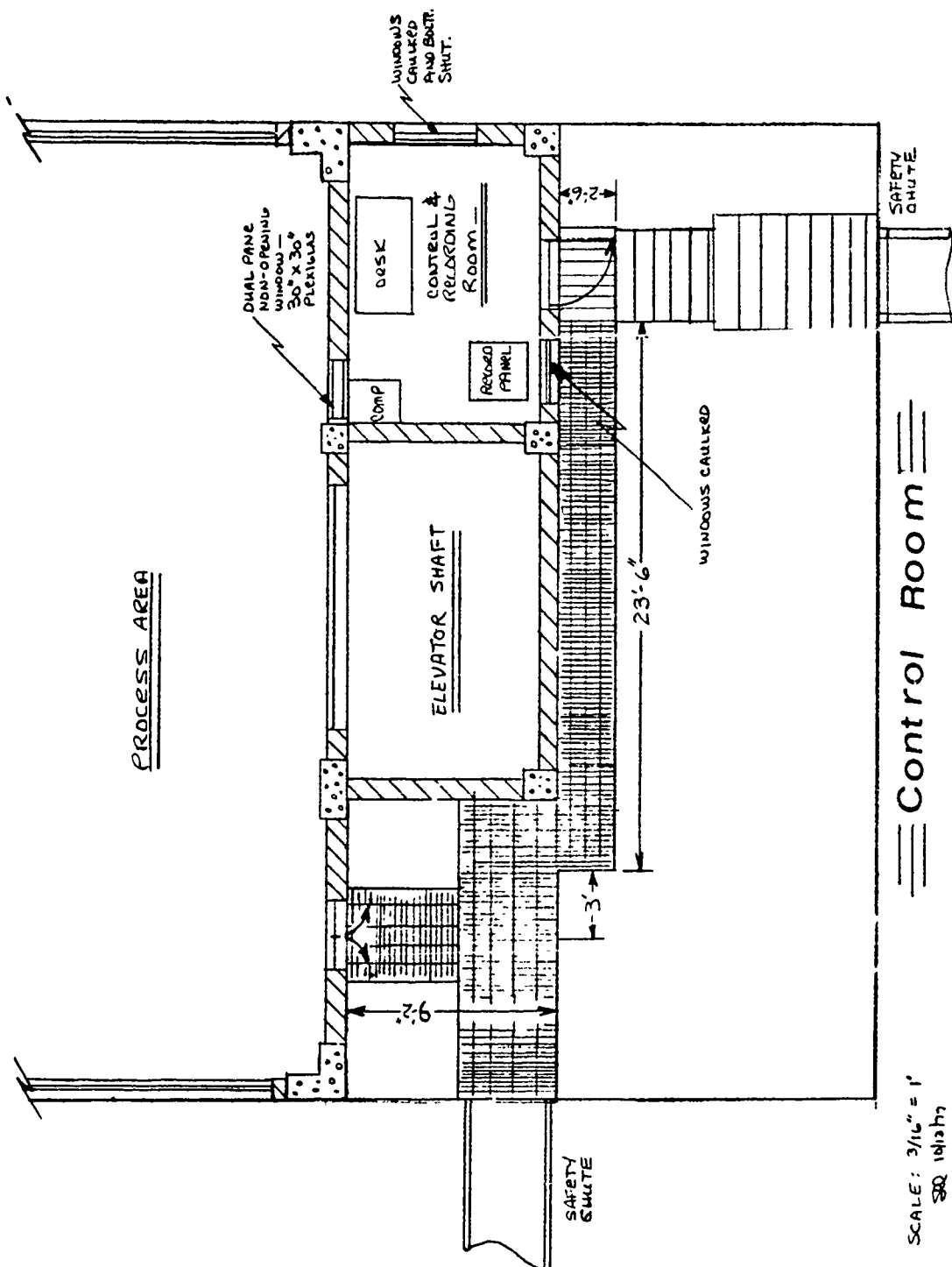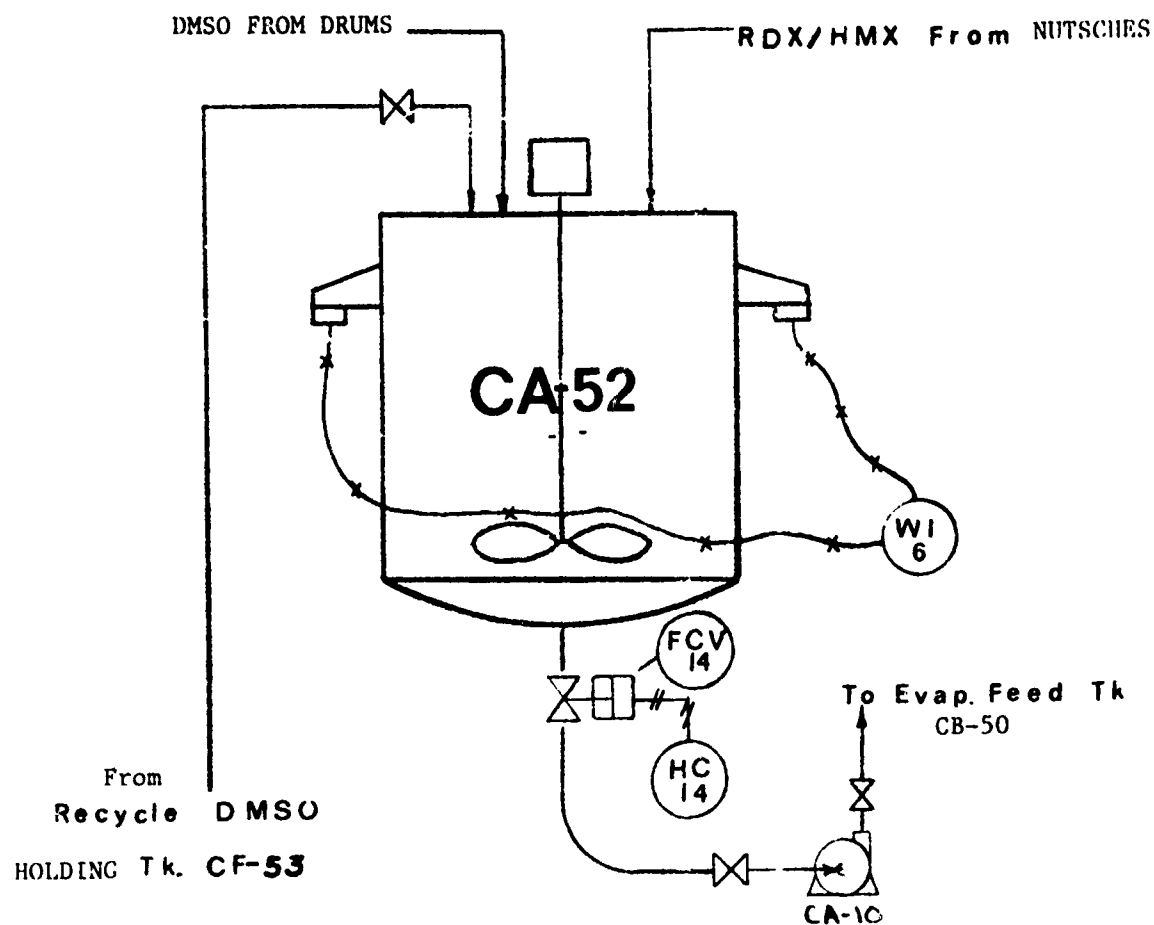
≡≡ Slurry Mix Tk. ≡≡

FIGURE 5

JRC

From CA-52

CB-50

LI 2C

TCV 1

15# Steam

TIC 1

TT 1

LIC 1

TO Evaporator CB-51

Filter No 4

CB-10

Washout
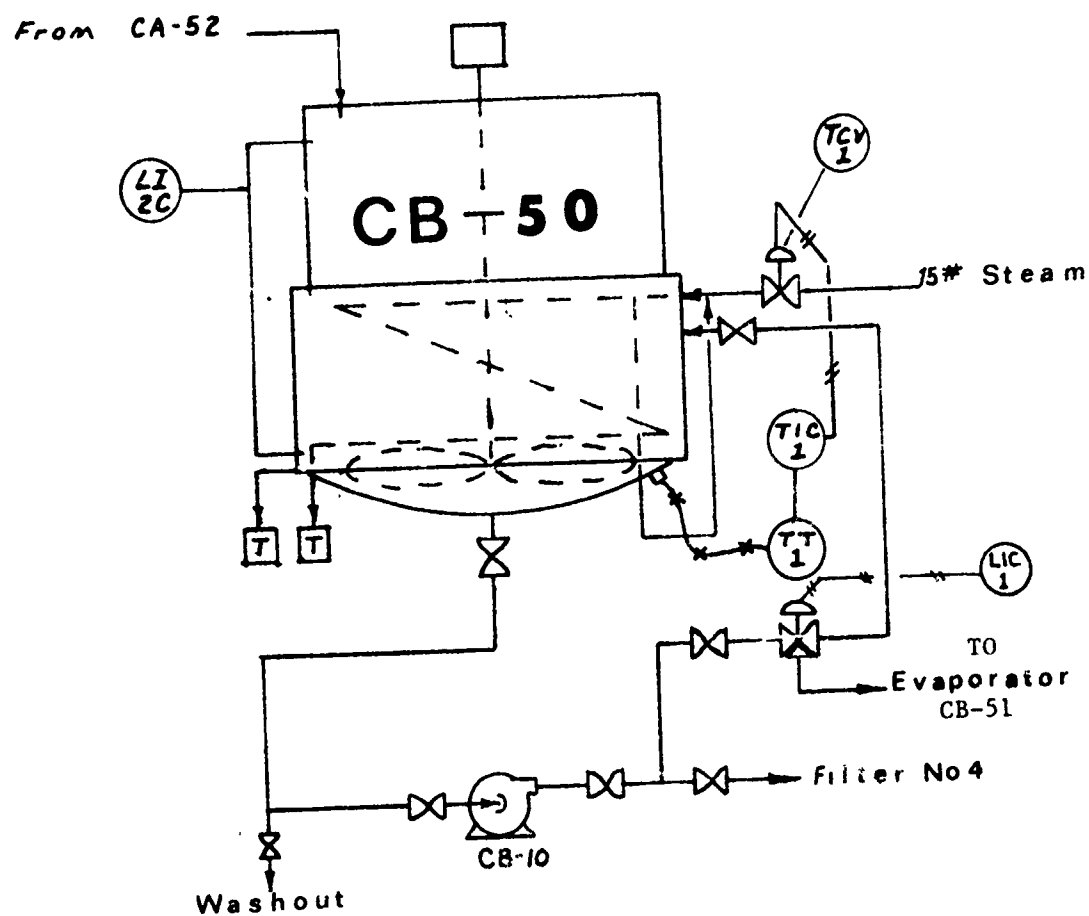
≡Evaporator Feed Tk≡

FIGURE 6

JRC

127

To
Rectifier

To
Evap.
Feed
Tank
(CB-50)

From
Evap.
Feed
Tank
(CB-50)

To Dissolver

LIC
1

LCV1

P1

PT
1

FIC
1

TIC
2

Steam

FE
4

CB-51

FW

Drain

Washout

CB-11

Sample

Evaporator
Feed Tank
(Shutdown)

Bypass

T
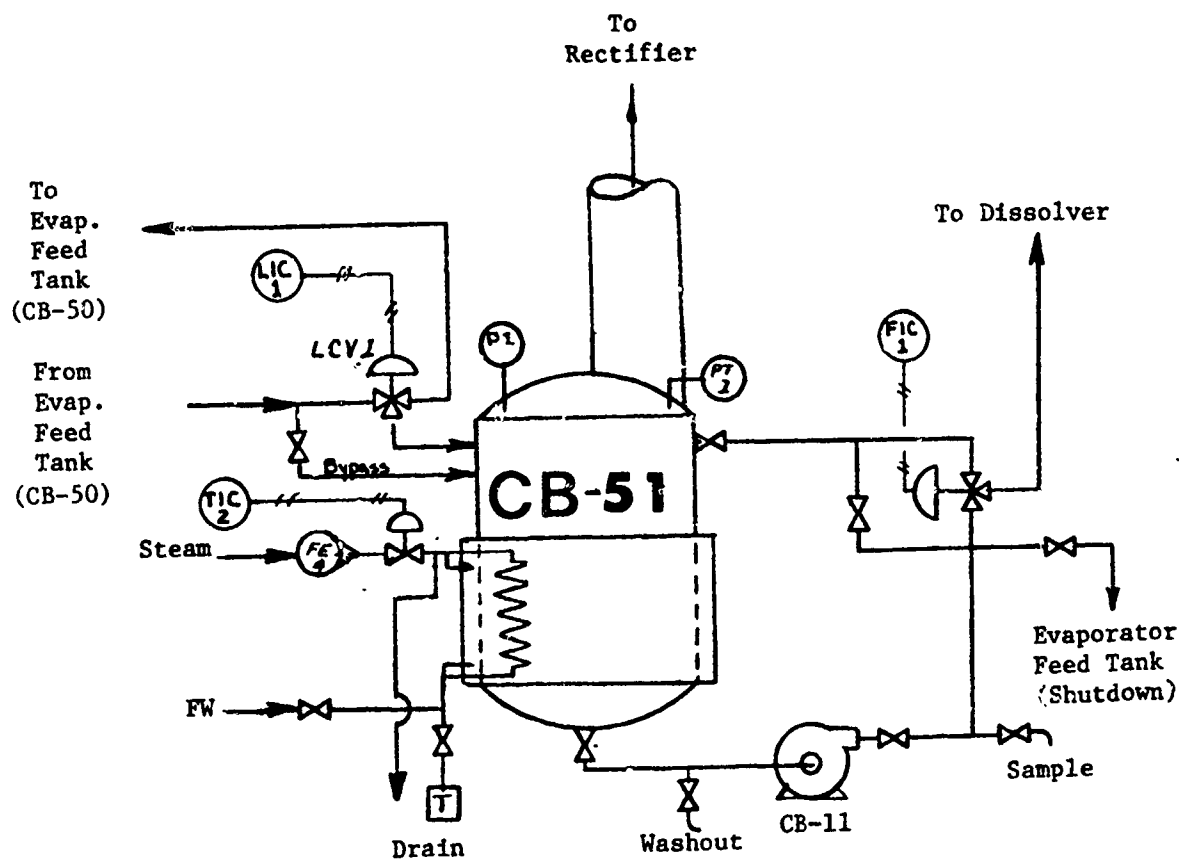
≡≡ **Evaporator** ≡≡
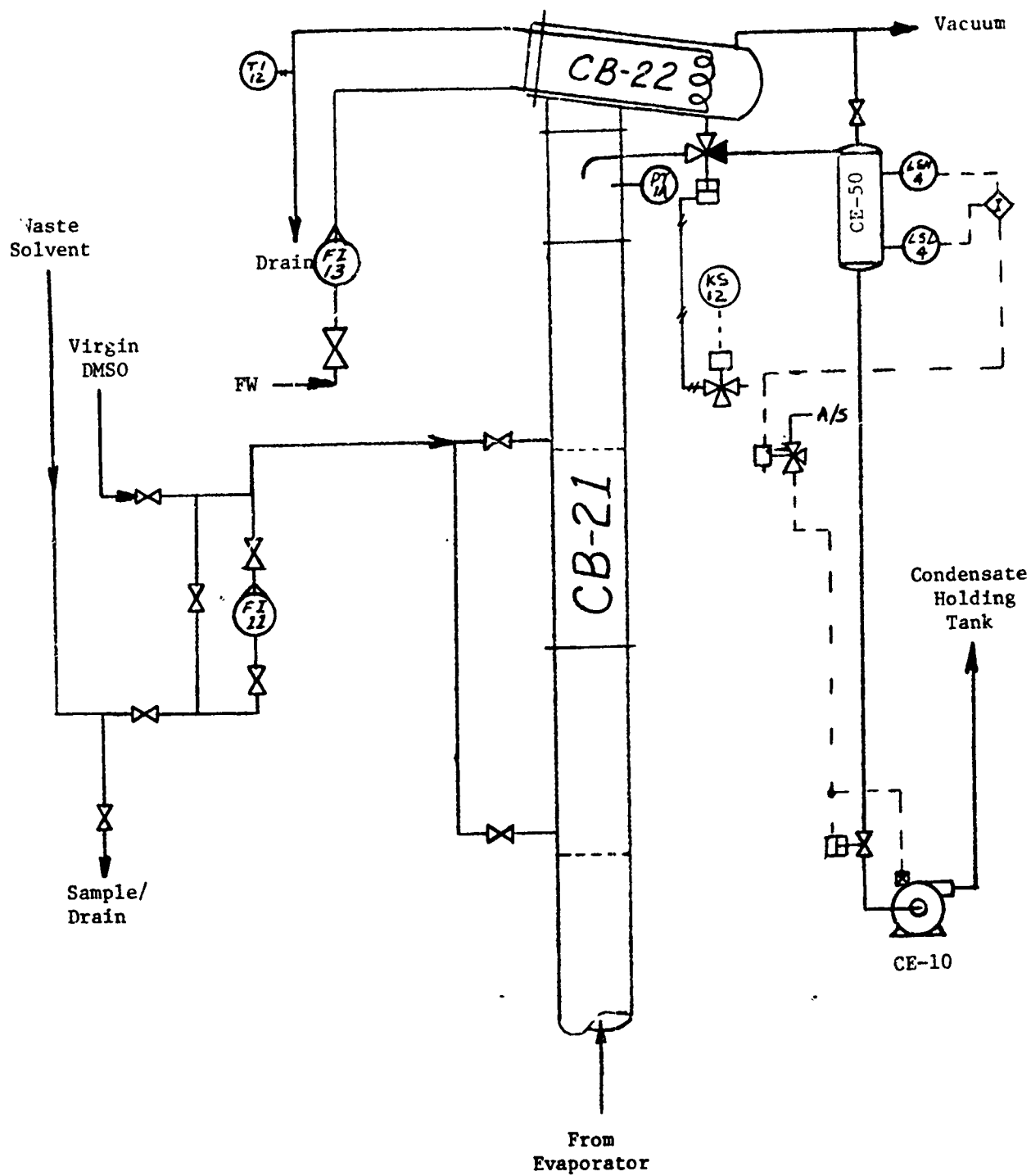
FIGURE 7

≡Rectifier≡

FIGURE 8

129
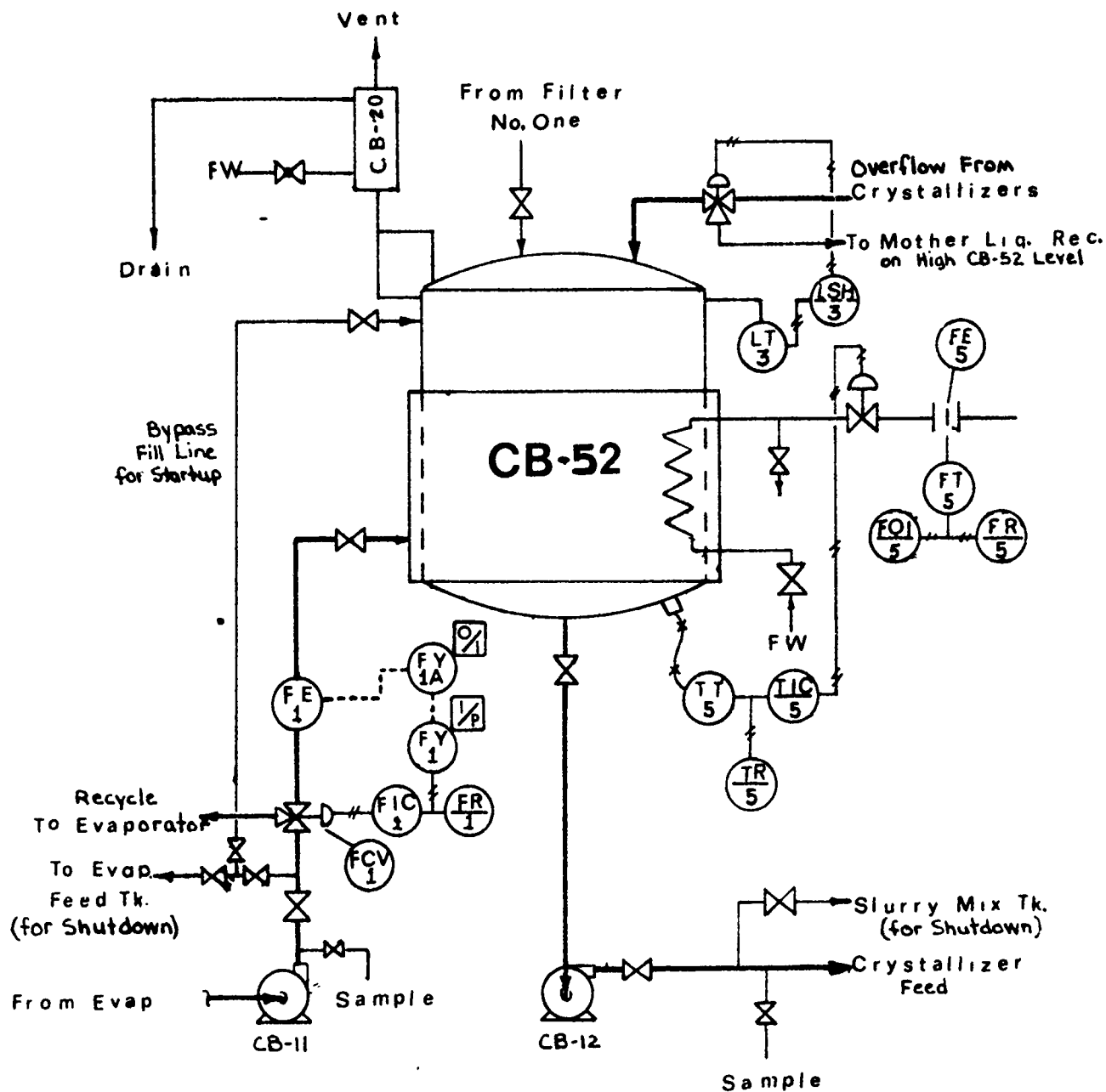
⟰Dissolver⟰

FIGURE 9

JRC
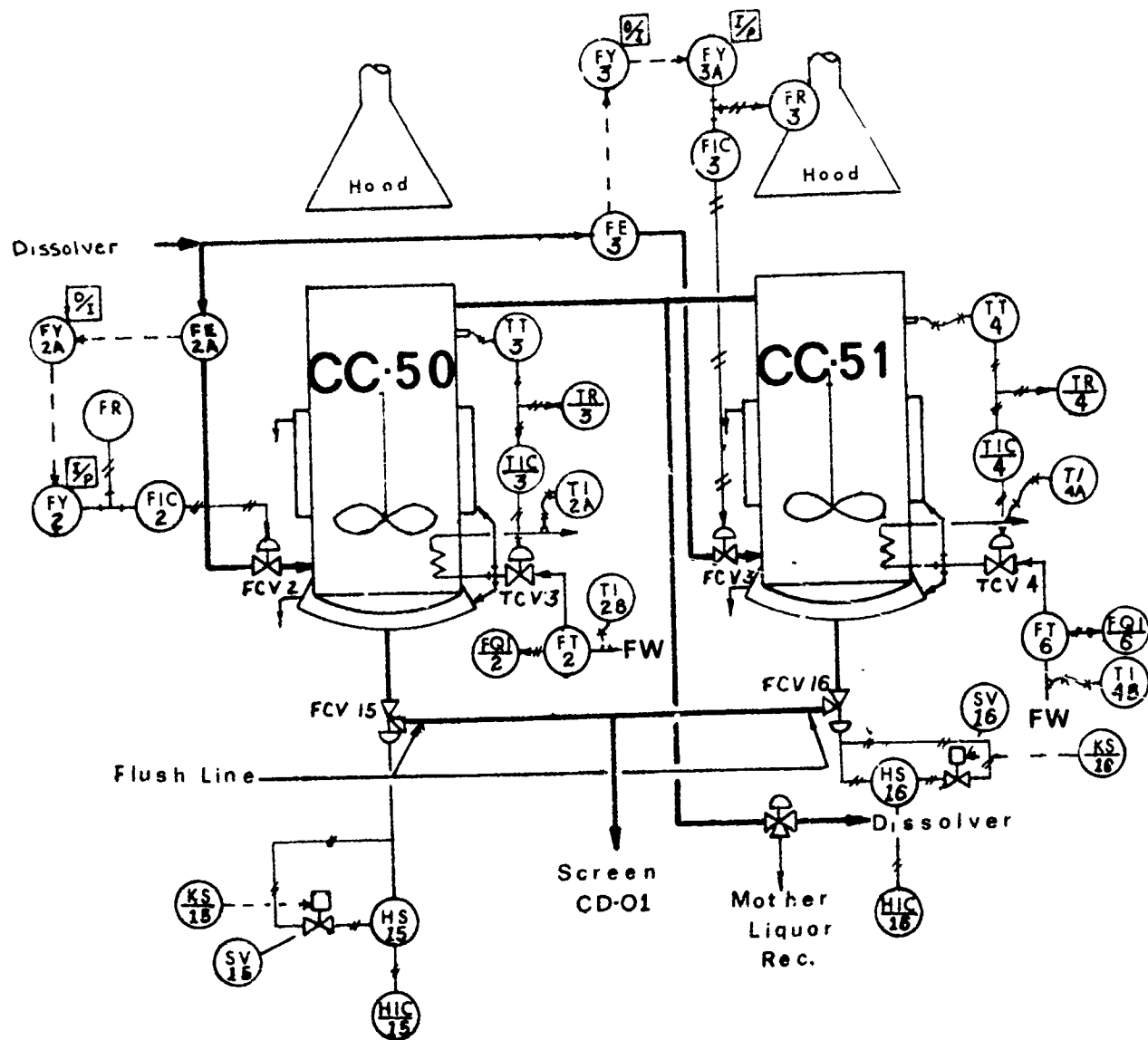
$\equiv$ **Crystallizers** $\equiv$

FIGURE 10

JRC

131
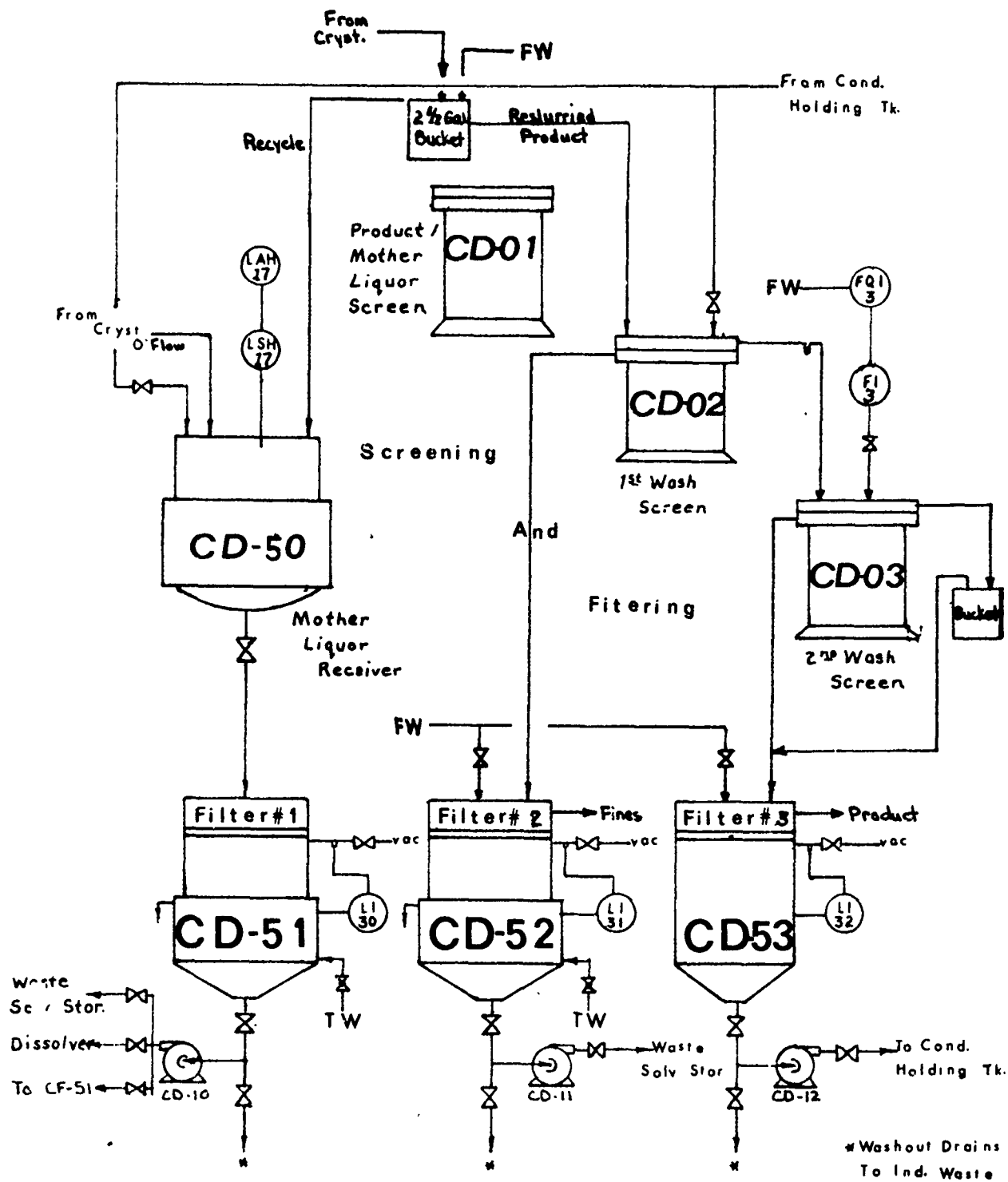
FIGURE 11: PRODUCT FILTRATION AND WASHING

132

JRC

Recycled
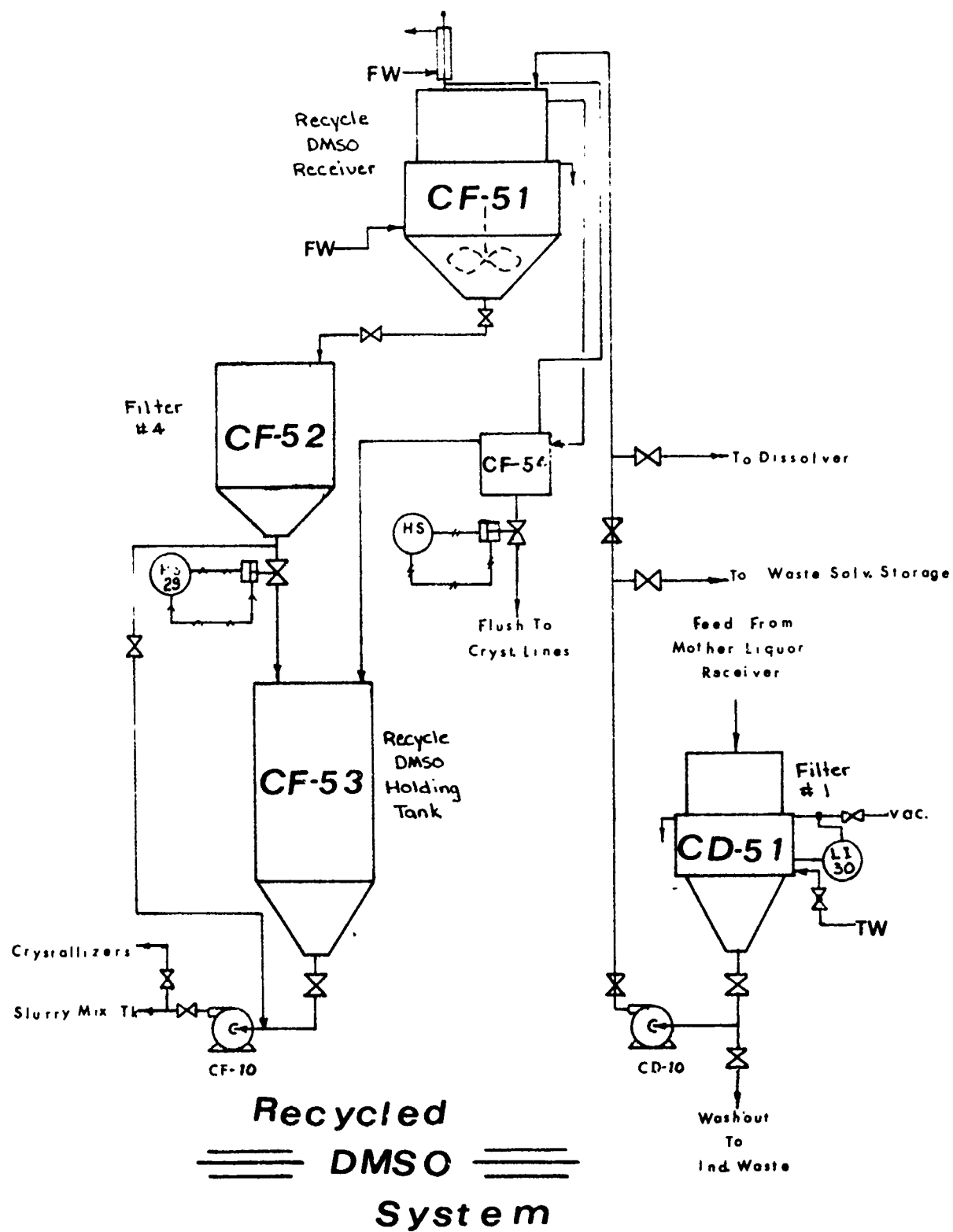
DMSO

System

FIGURE 12
133

JRC
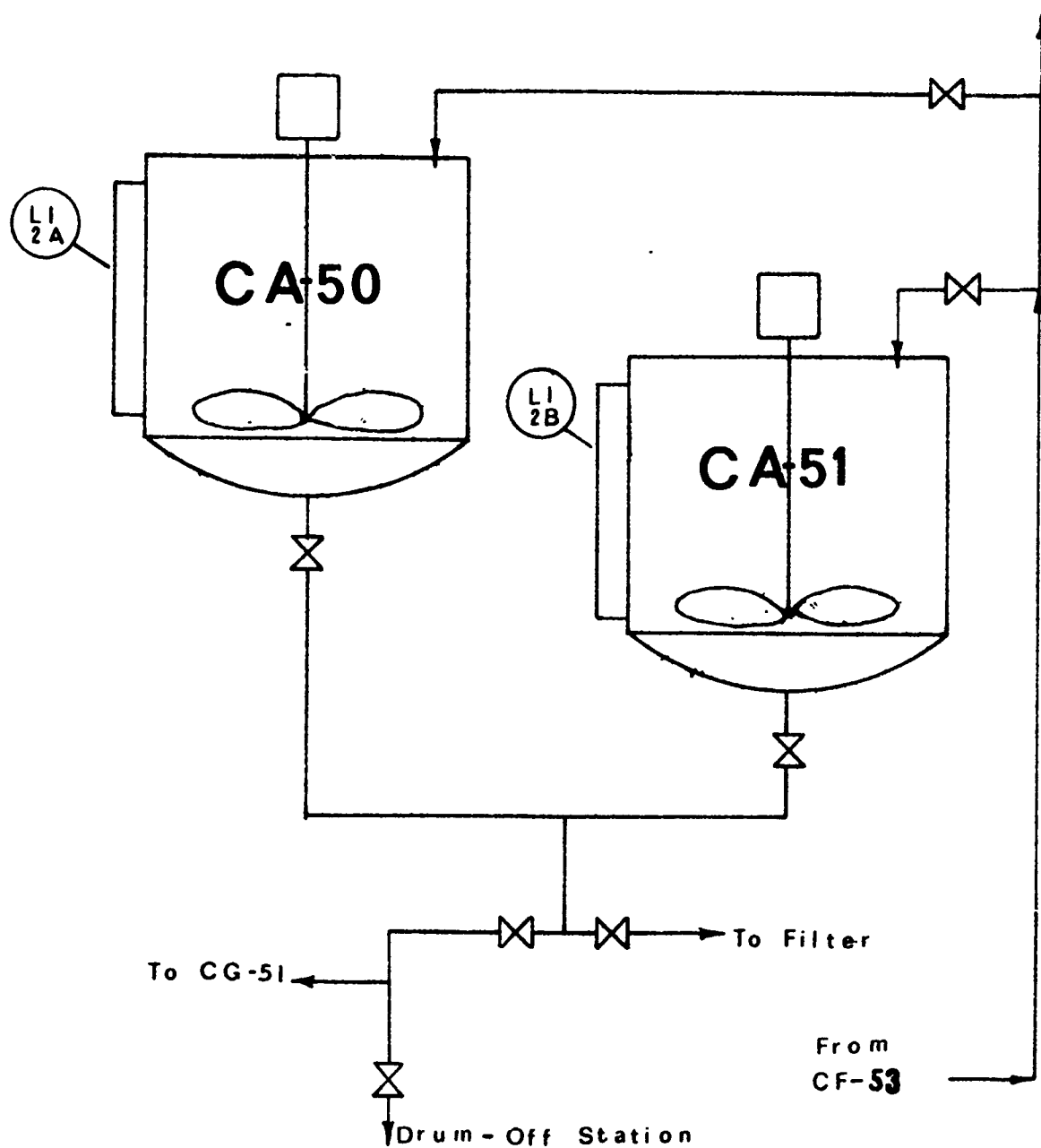
Recycle DMSO
Storage

JRC

FIGURE 13

FROM
FILTER No. 3

CONDENSATE
FROM CE-50

CE-51

WASTE
WATER
TANK

INDUSTRIAL
SEWER

FIRST
WASH
SCREEN

CE-11
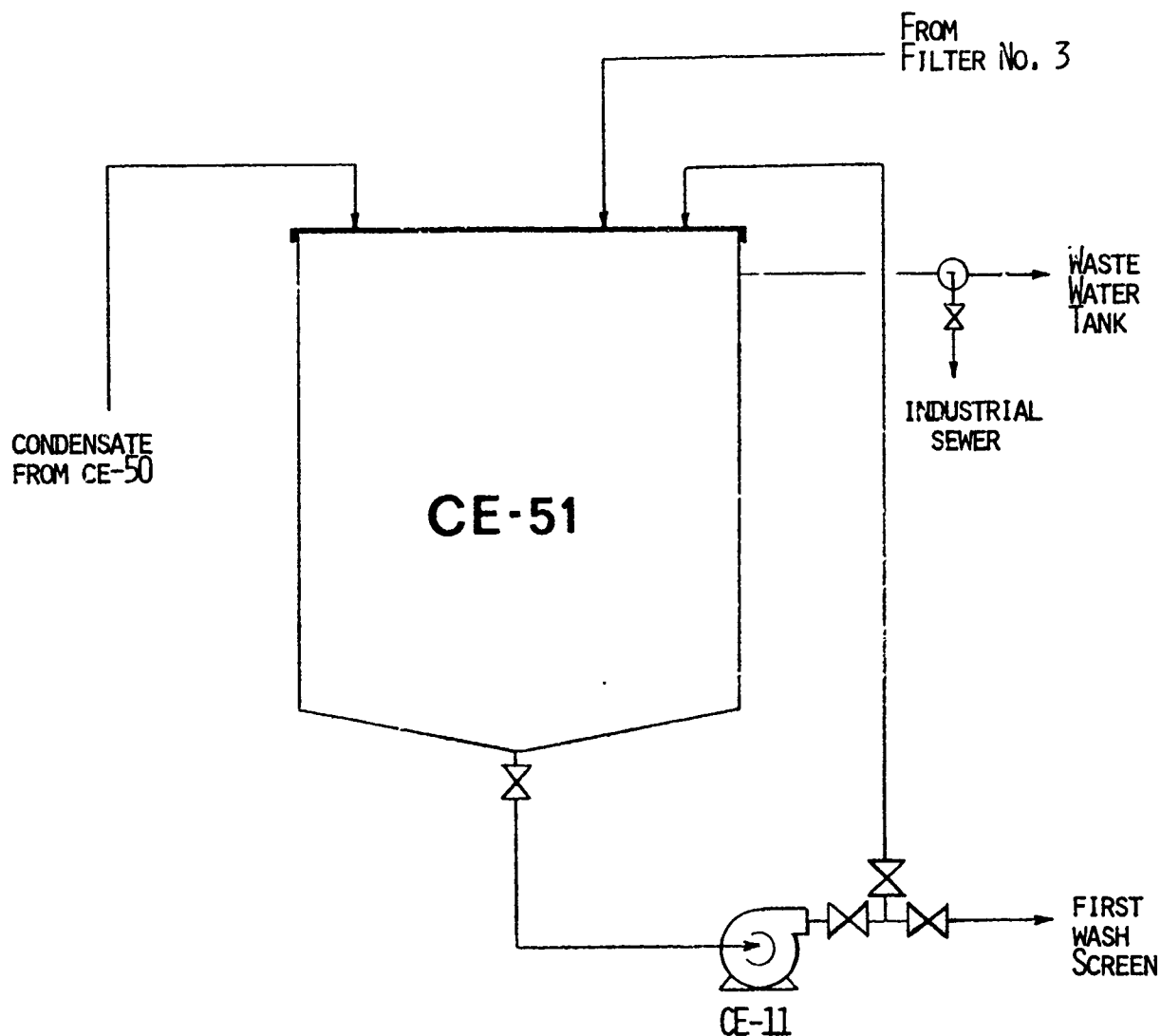
— CONDENSATE —
— HOLDING TANK —

FIGURE 14

FROM WASTE
SOLVENT FILTER

TIC
10

TT
10

TCV
10

15 PSIG
STEAM

CB-53

T

TO CB-21
OR CF-52
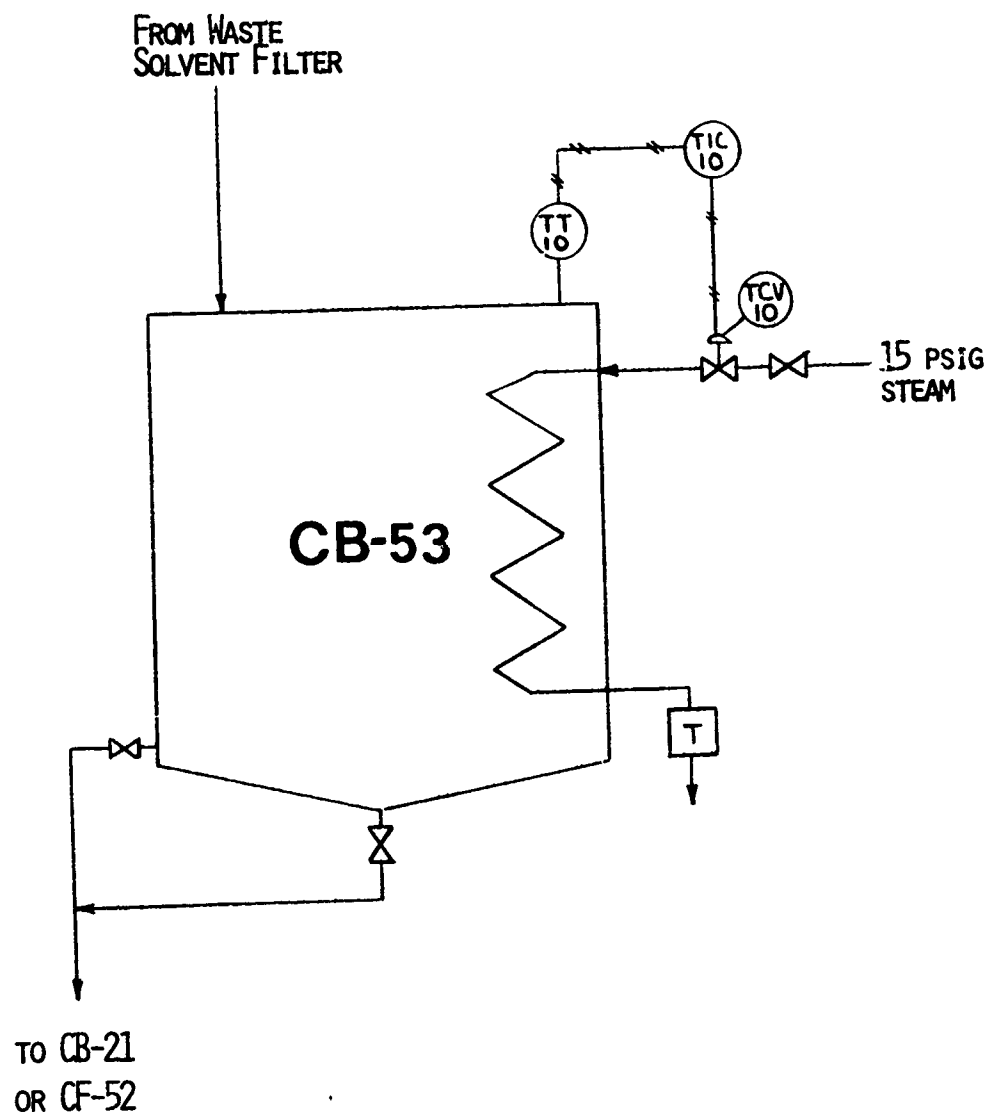
Waste Solvent
Recycle Tank

FIGURE 15

From Filter #1 (CD-51)

From Filter #2 (CD-52)

Recycled DMSO

Waste Solvent Storage Tank

CG-50

Recycled DMSO

Vacuum

Filter # 5

CG-51

Drum-Off Station

Waste Solvent Recycle Tank (CB-53)

Solvent Wagon

Washout to Industrial Waste

CG-10
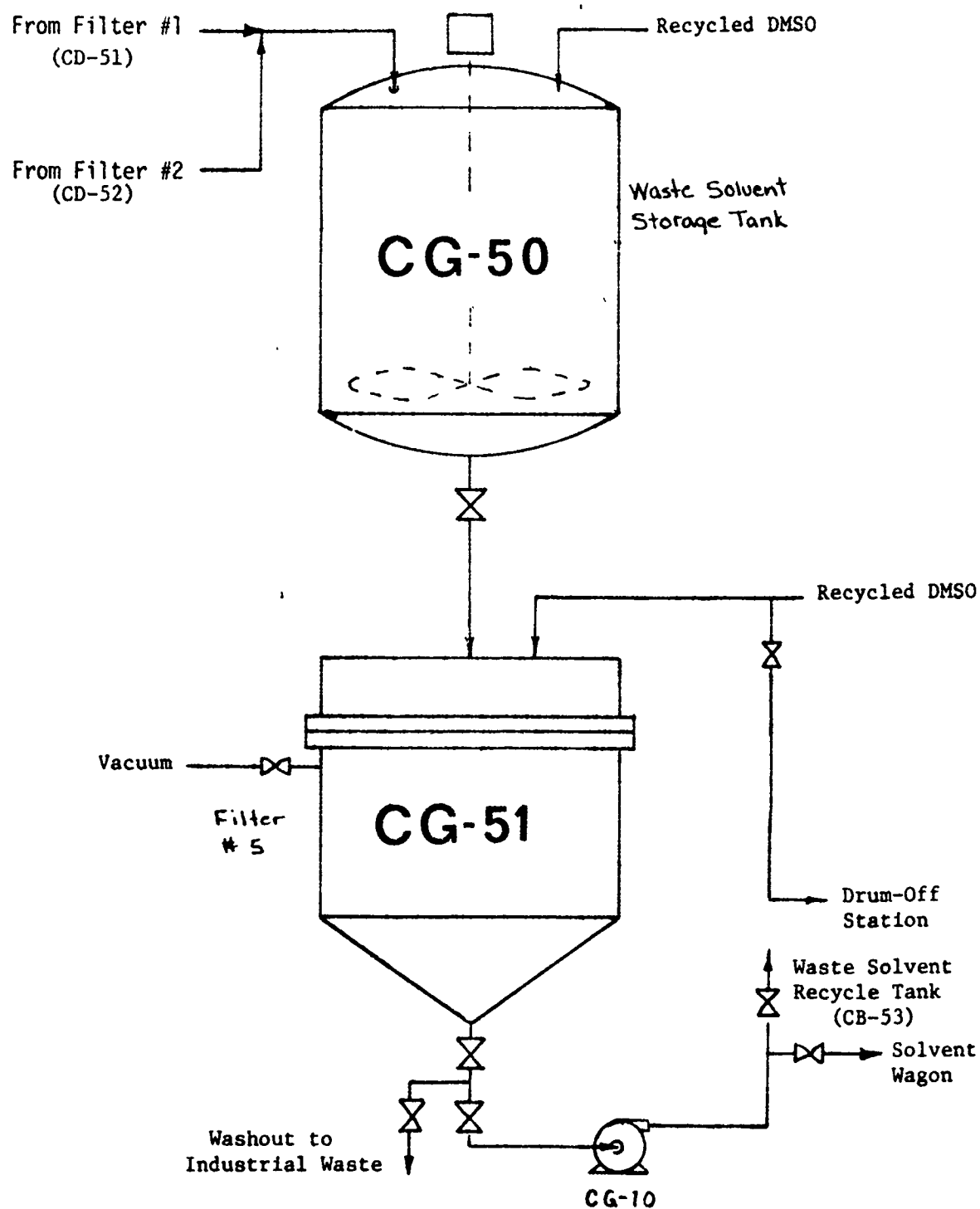
Waste Solvent
Storage & Filtering

FIGURE 16

JRC

100# Steam

100# Steam

FW CJ-10&11

PI 19

PI 18

CJ-50

vac. vac.

CD-51 CG-51
CD-52 CF-52
CD-53

CJ-51

Drum/Ditch
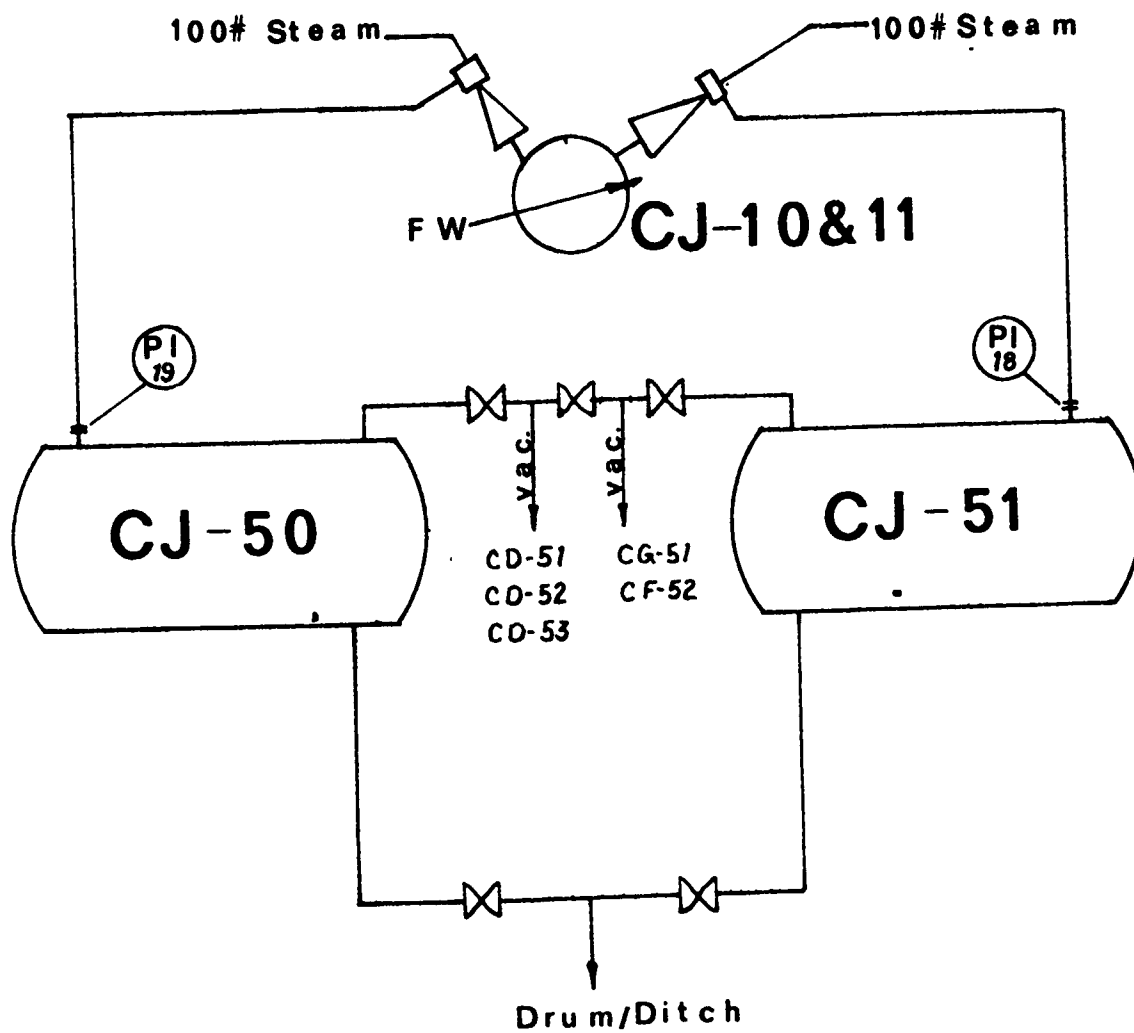
≡Vacuum System≡

FIGURE 17

JRC

138
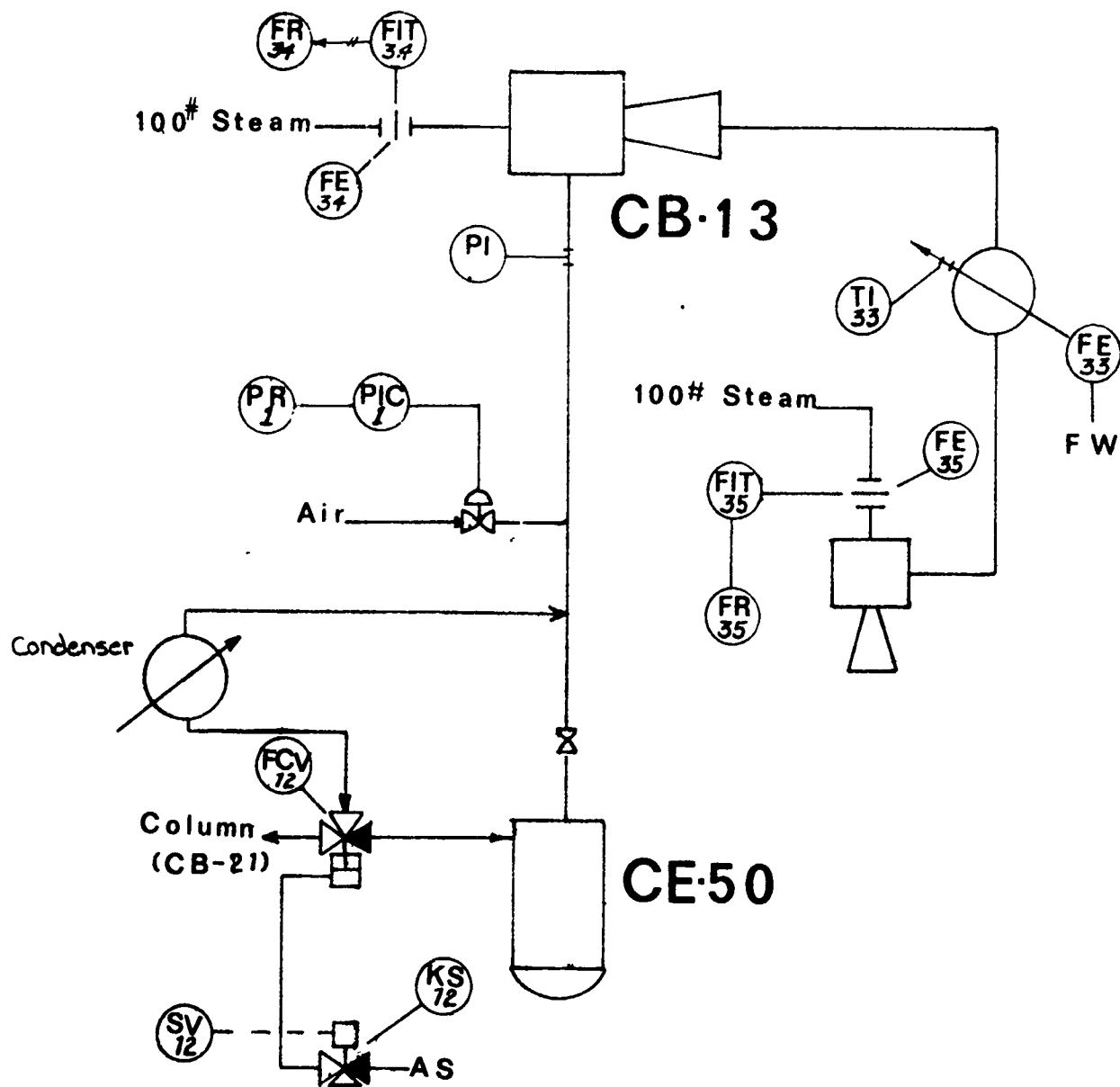
Steam Ejectors

For

Distillation Column

FIGURE 18   JRC

Atmo.

Exhaust Fan
(CH-10)

Roof

CC-50   CC-51

CA-52

2nd Floor

Mezzanine

CF-52   CD-01   CD-02   CD-03

1st Floor

CD-51   CD-50   CD-52   CD-53
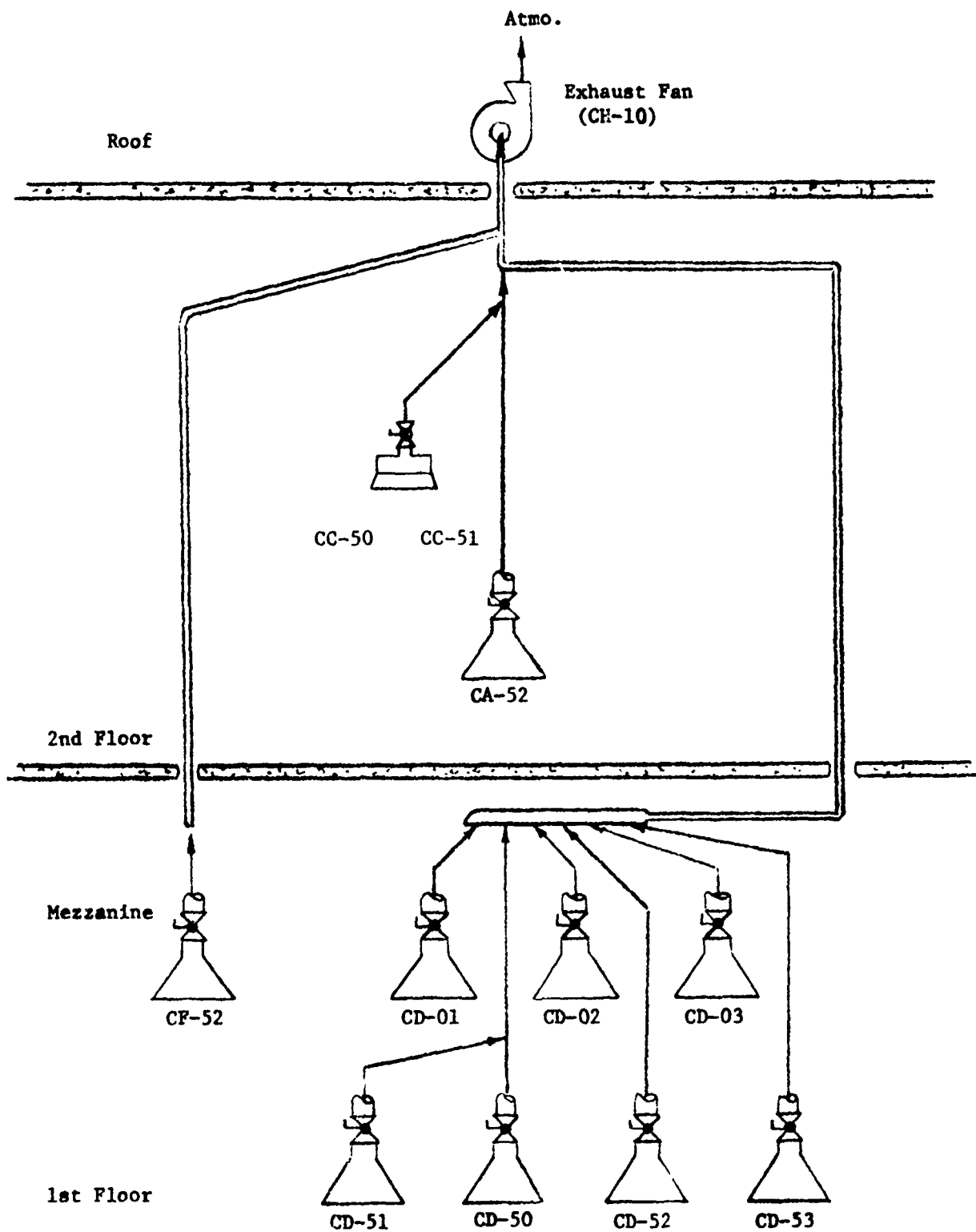
Exhaust   System

FIGURE 19

JRC

140

Cond. Holding Tk.
(CE-51)

CG-52

Ditch
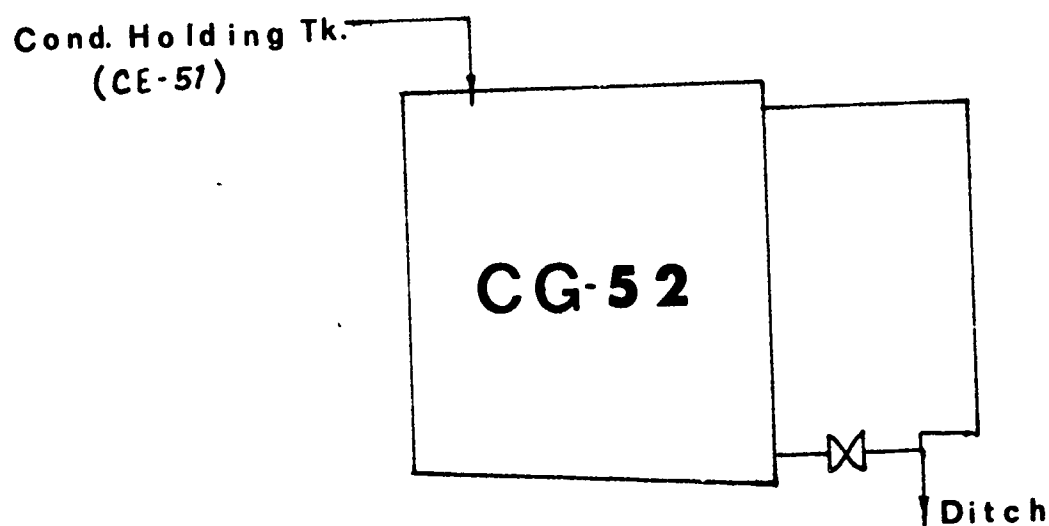
Waste Water
Holding Tk.

FIGURE 20

JRC

141
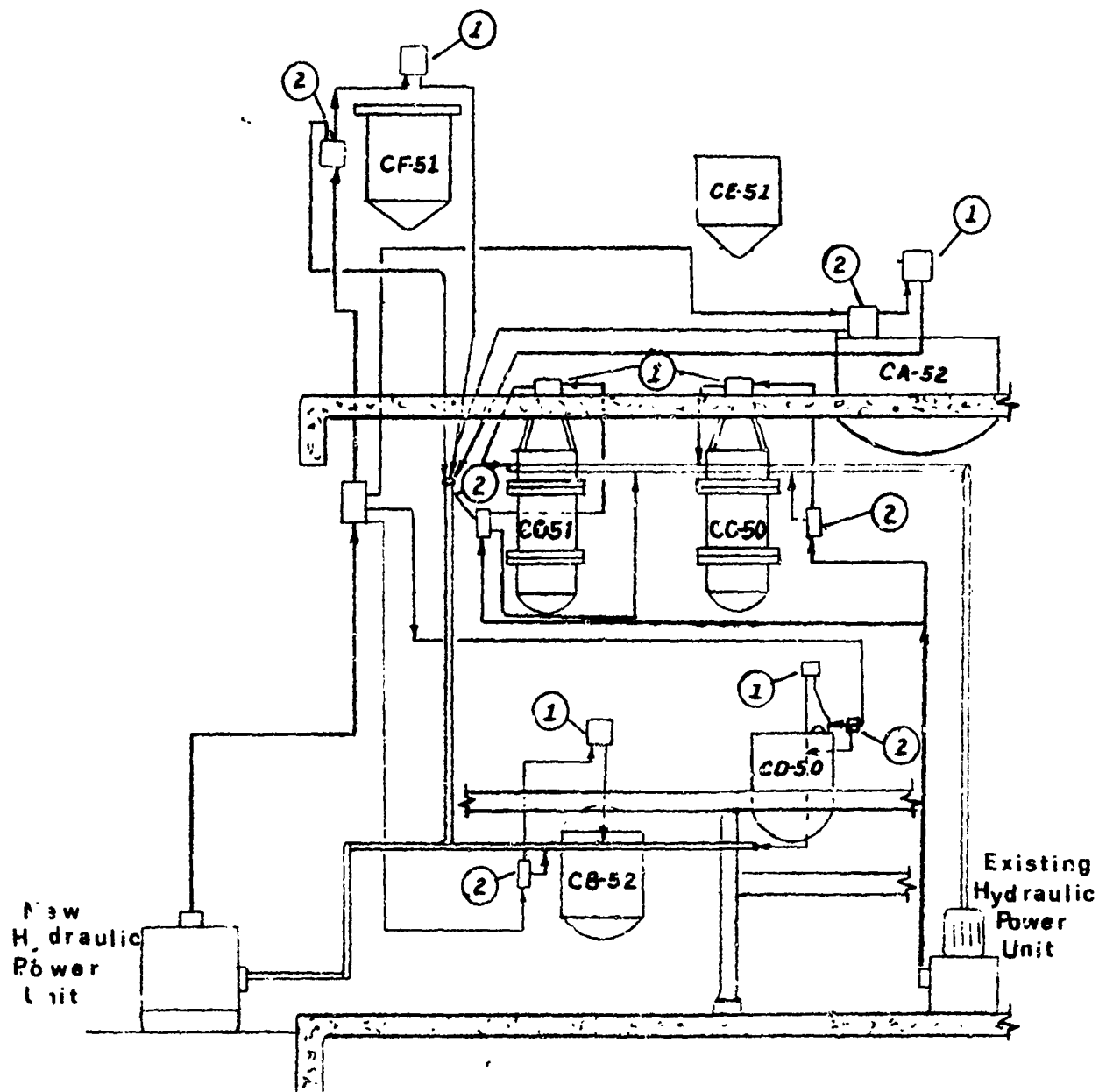
Hydraulic System

FIGURE 21

JRC

142
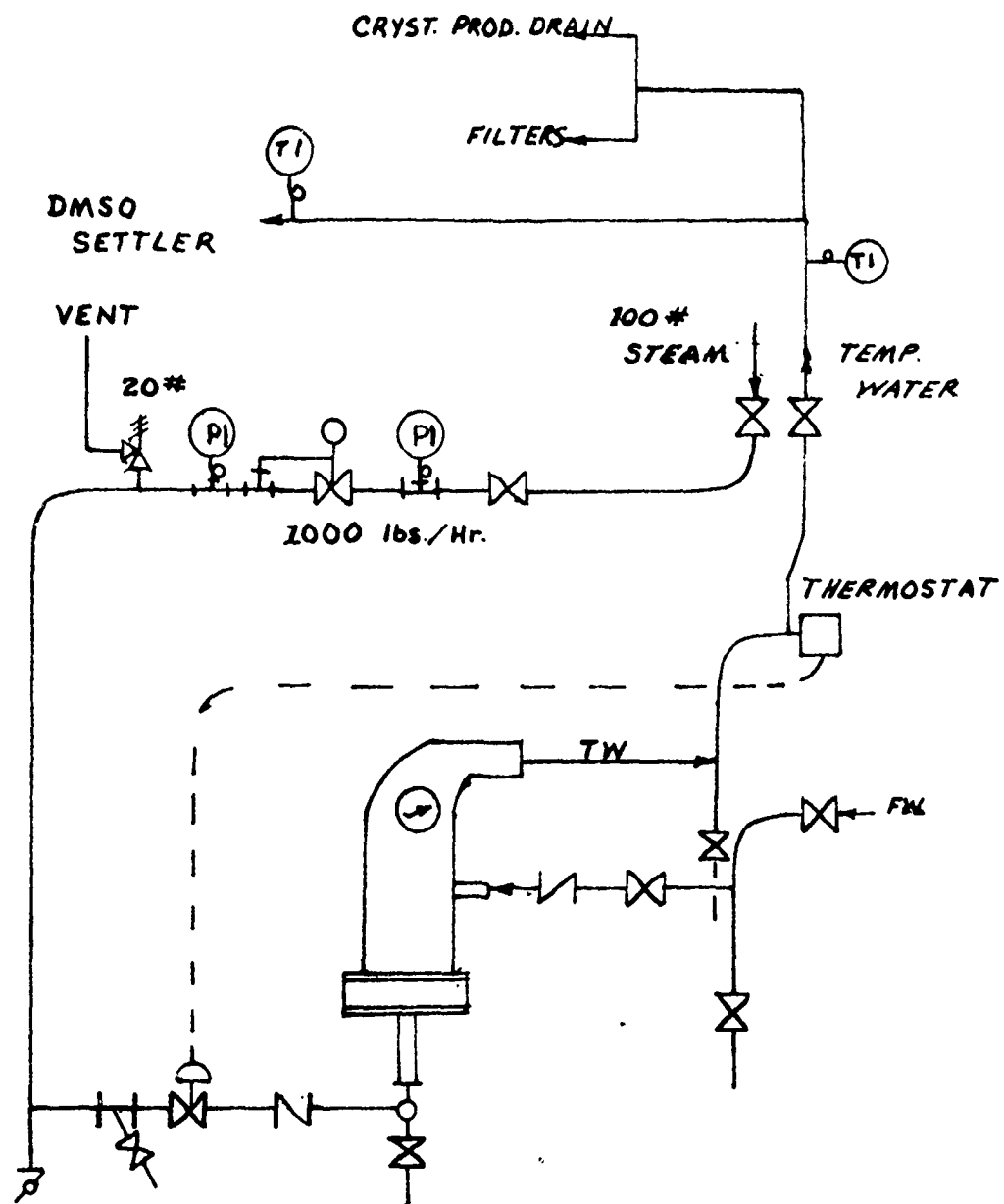
Pick Heater

FIGURE 22

JRC

Figure 23.Solution Equilibrium
For The System RDX-DMSO-H$_2$O

Saturation
Temperature, $^\circ$C

110
100
95
90
85
80
75
70
60
50
45
30
25

SOLID RDX

H$_2$O
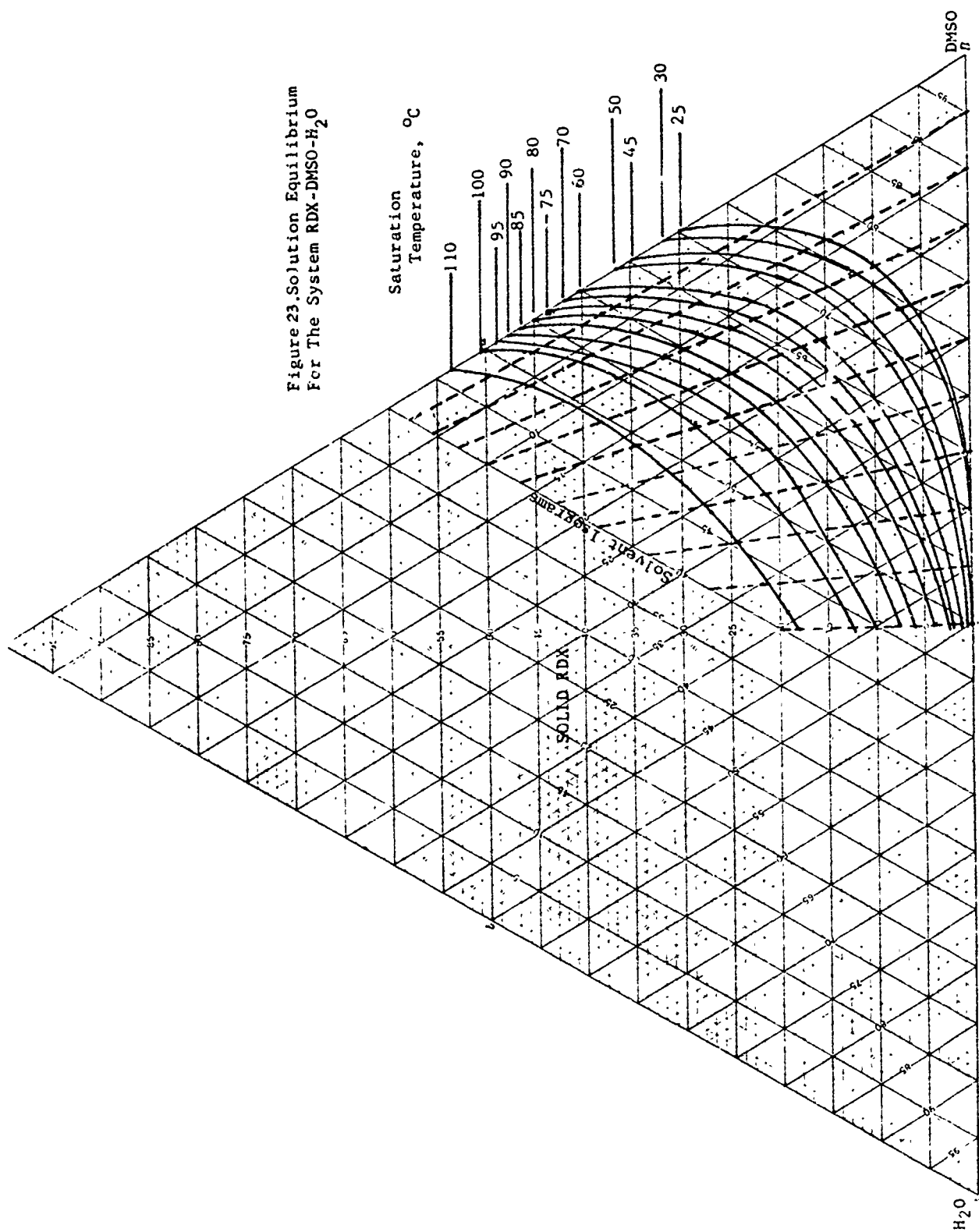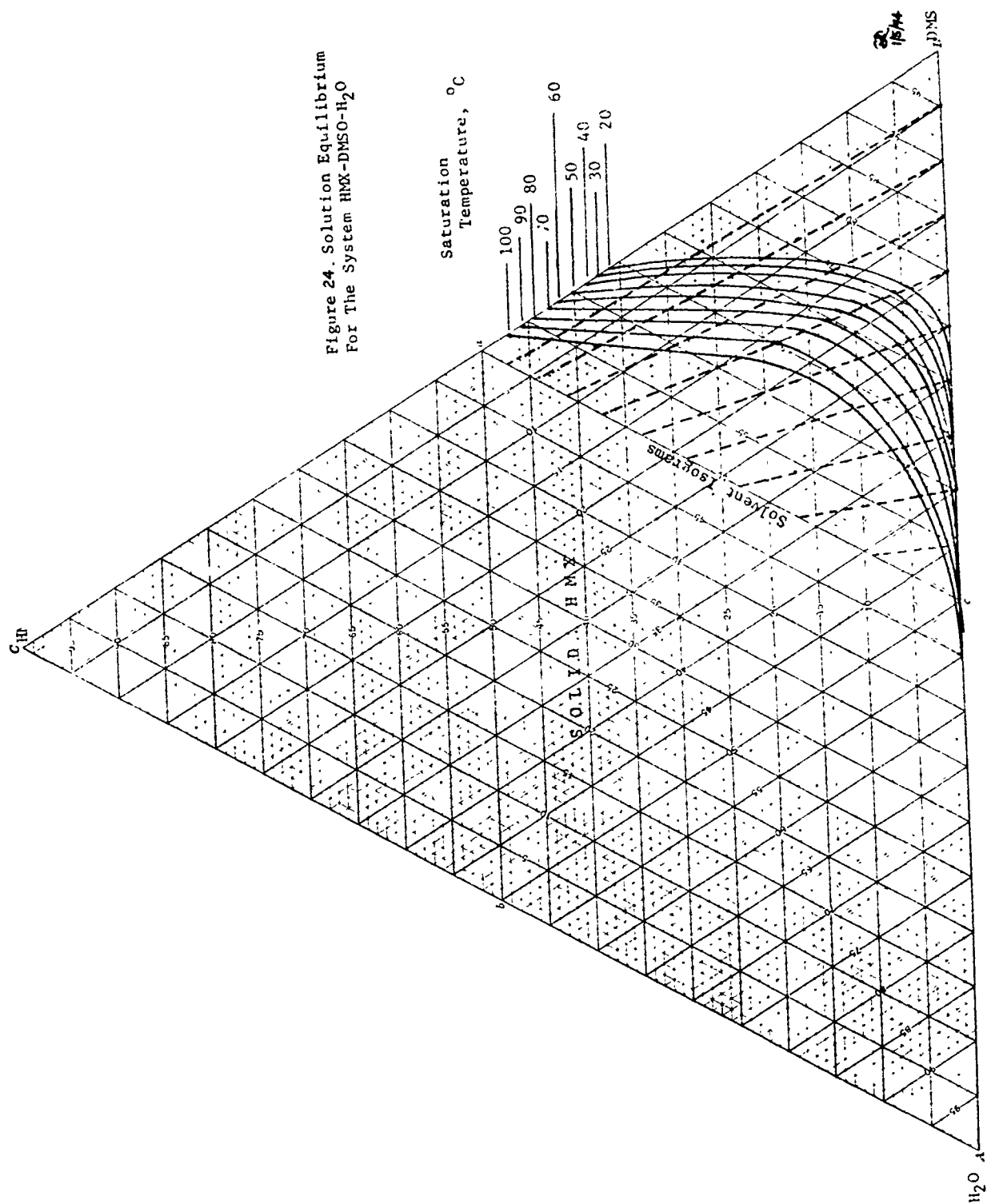
DMSO

Figure 24. Solution Equilibrium
For The System HMX-DMSO-H₂O

Saturation
Temperature, °C
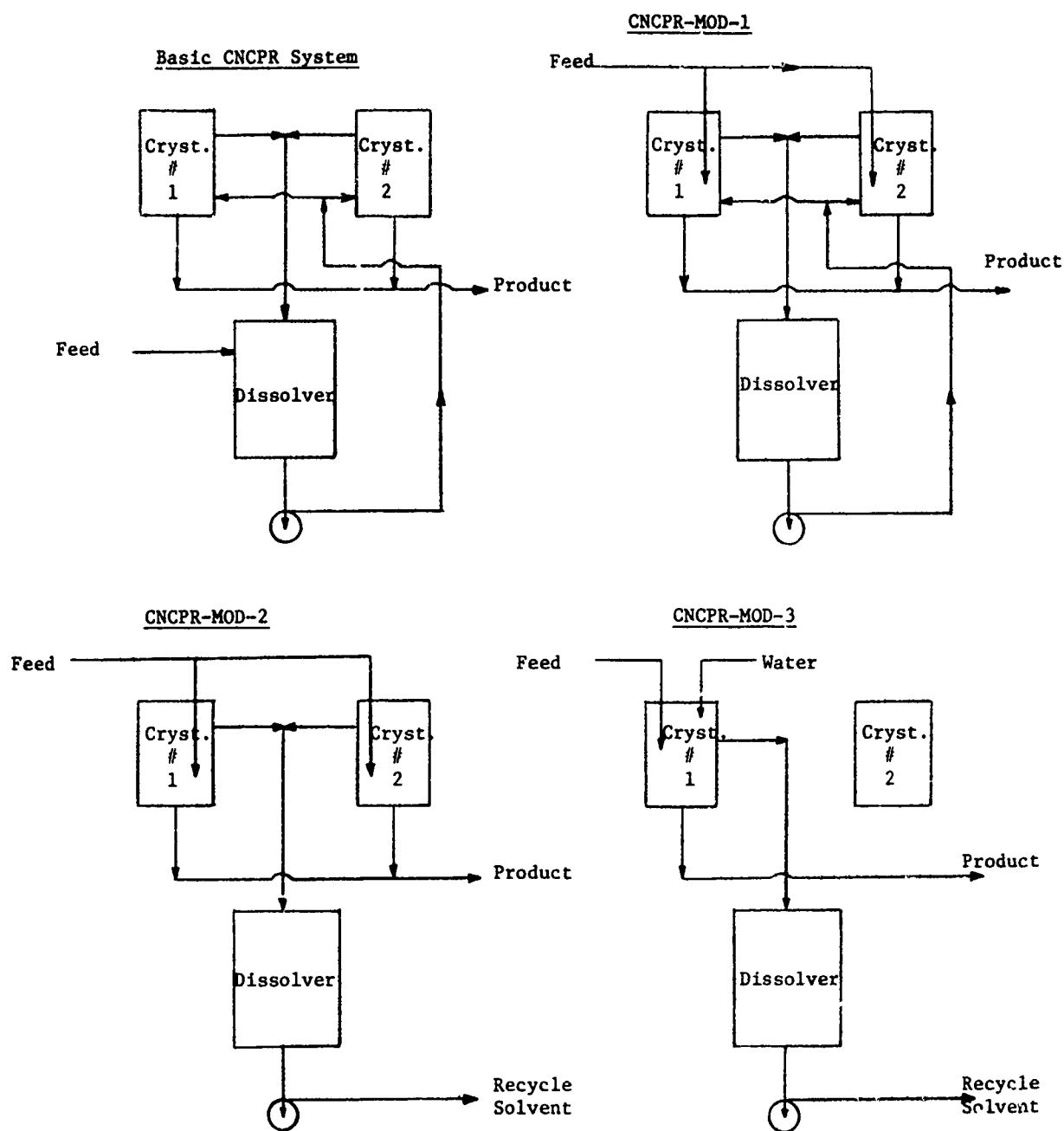
Figure 25: CNCPR Systems

Figure 26. Pilot Crystallization Flow Diagram

FIGURE 27: PILOT SOLVENT RECOVERY
MATERIAL BALANCE

Figure 27: Pilot Solvent Recovery Material Balance

Labels visible in diagram:

WASTE SOLVENT TANK CG-50

WASTE SOLVENT FILTER CG-51

WASTE EXPLOSIVES (423 LB)

EVAPORATOR FEED TANK CB-50

EVAPORATOR CB-51 (75 mm Hg) 90°C

EVAPORATOR FEED TANK CB-50

EVAPORATOR CB-51 (50 mm Hg) 105°C

RECTIFYING COLUMN CB-21

RECTIFYING COLUMN CB-21

CONDENSER

CONDENSER

55-GAL. DRUM

VACUUM SUPPLY

VACUUM SUPPLY

292 LB { EXPL - 0.1431, DMSO - 0.8484, H2O - 0.0085 }

42,300 LB @ 102 LB/HR { SOLIDS (1%) { RDX - 0.5000, HMX - 0.5000 }, LIQUID (99%) { EXPL - 0.0010, DMSO - 0.0999, H2O - 0.8991 } }

49,930 LB @ 102.4 LB/HR { H2O - 0.9999, DMSO - 0.0001 }

4,394 LB @ 266.5 LB/HR

4,394 LB @ 10.6 LB/HR { EXPL - 0.0095, DMSO - 0.9509, H2O - 0.0396 }

4,597 LB @ 211.2 LB/HR { DMSO - 0.8550, H2O - 0.1450 }

495 LB H2O @ 20 LB/HR

101 LB/HR { EXPL - 0.0010, DMSO - 0.0999, H2O - 0.8991 }

12,447 LB H2O @ 30 LB/HR

CG-10

CB-10

CB-11

CB-11

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Figure 28: Effect of Dissolver Saturation and Crystallizer Supersaturation on Product Particle Size in the CNCPR-MOD-1/RDX System

149
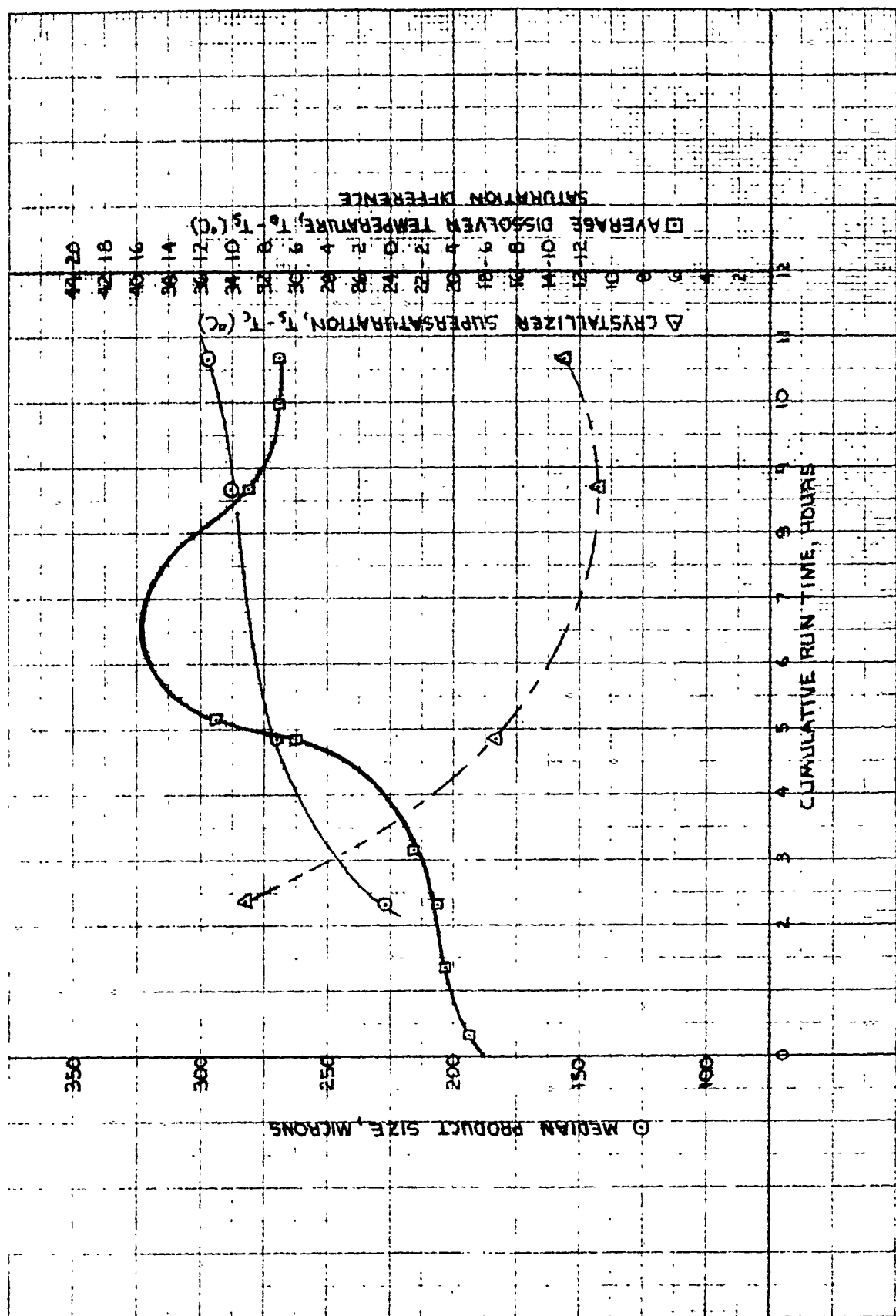
Figure 29: Effect of Dissolver Saturation and Crystallizer Supersaturation on Product Particle Size in the CNCPR/RDX System

150

Figure 30: Effect of Dissolver Saturation and Crystallizer Supersaturation on Product Particle Size in the CNCPR-MOD-1/HMX System

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Figure 31: Effect of Dissolver Saturation and Crystallizer Supersaturation on Product Particle Size in the CNCDR/HMX System (Expt. No. 7)

Figure 32 : Effect of Dissolver Saturation and Crystallizer Supersaturation on Product Particle Size in the CNCPR/HMX System (Expts 8 and 9)

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Figure 33: Effect of Dissolver Saturation and Crystallizer Supersaturation on Product Particle Size in the CNCPR/HMX System (Expts 10 and 11)

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APPENDIX C
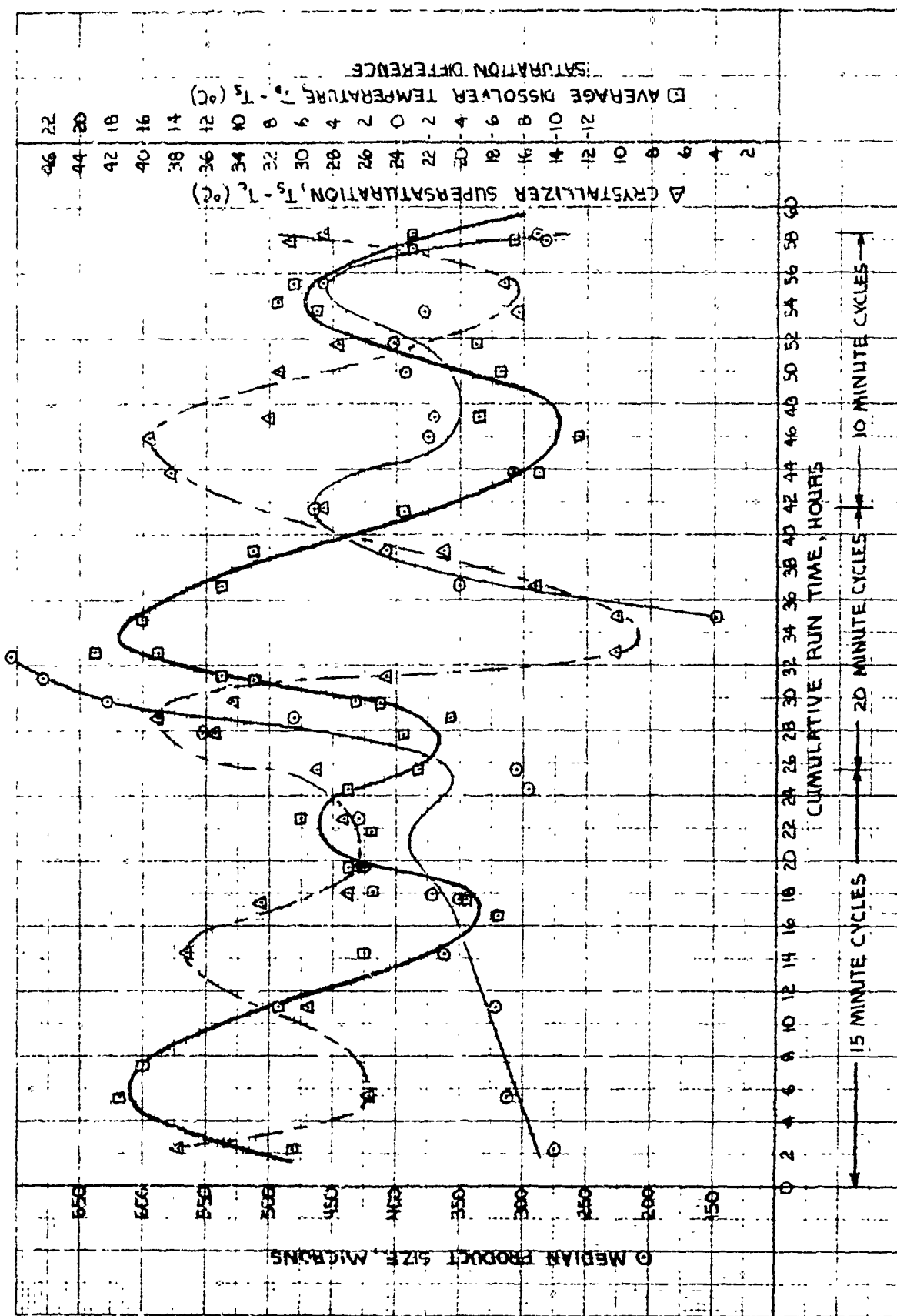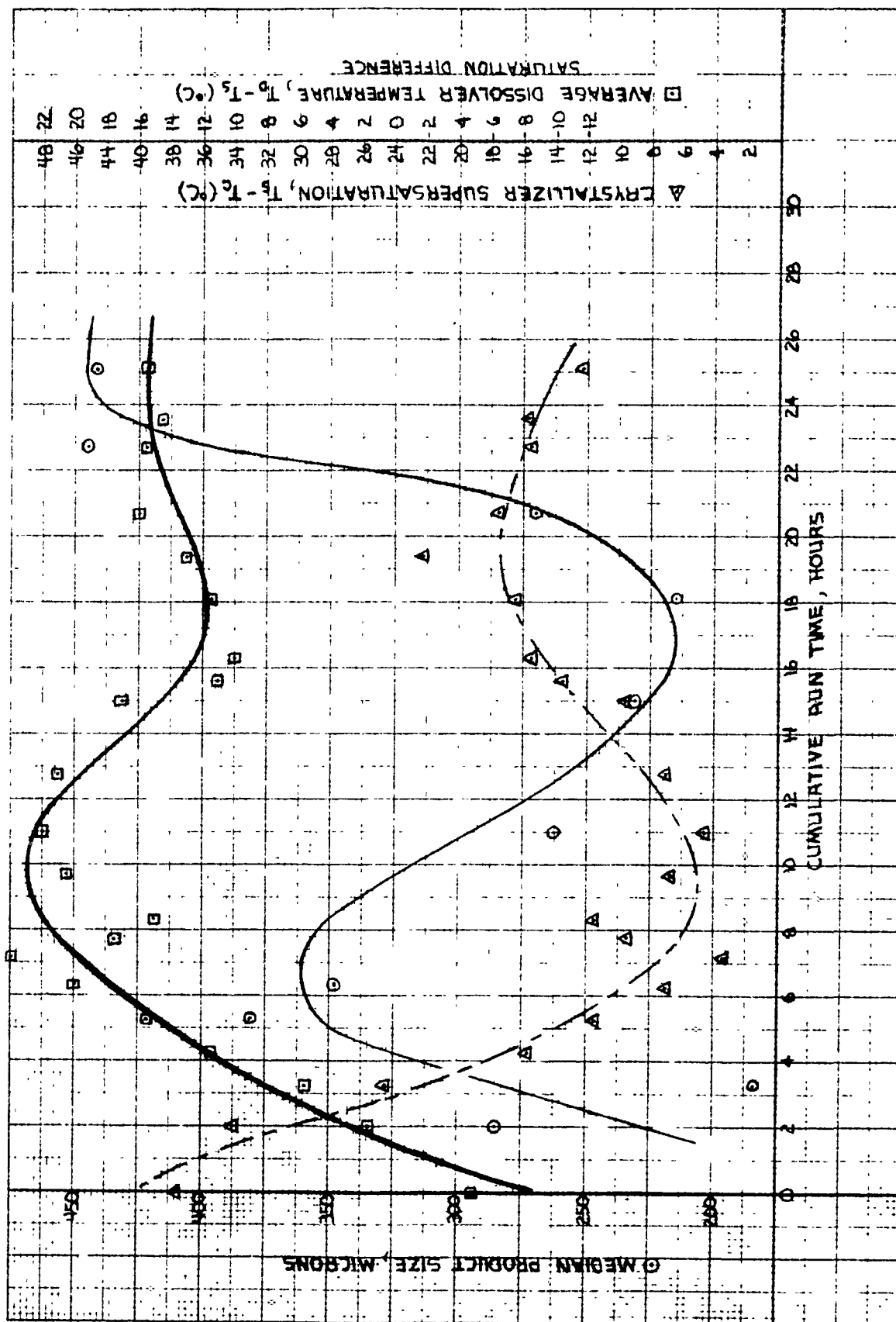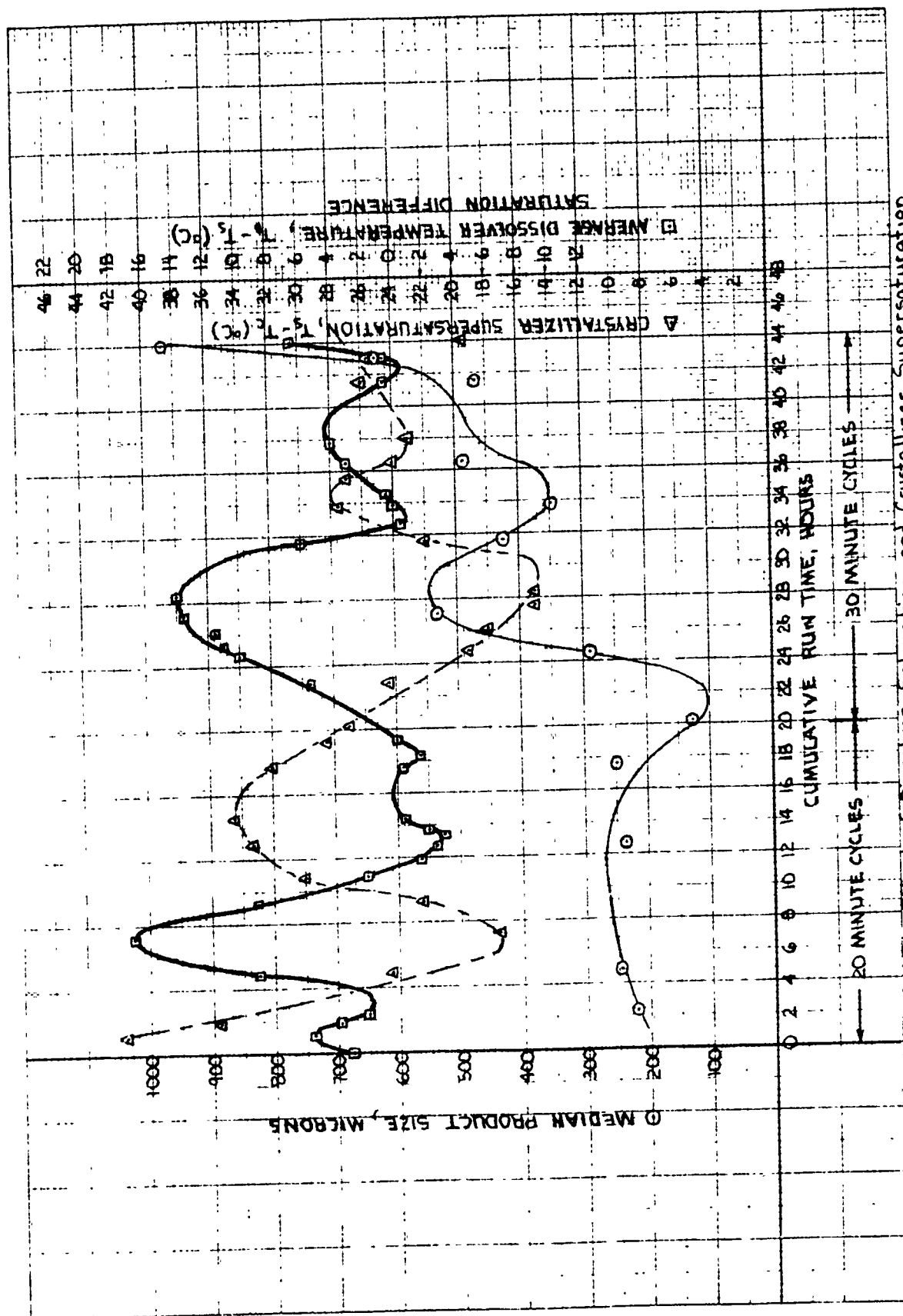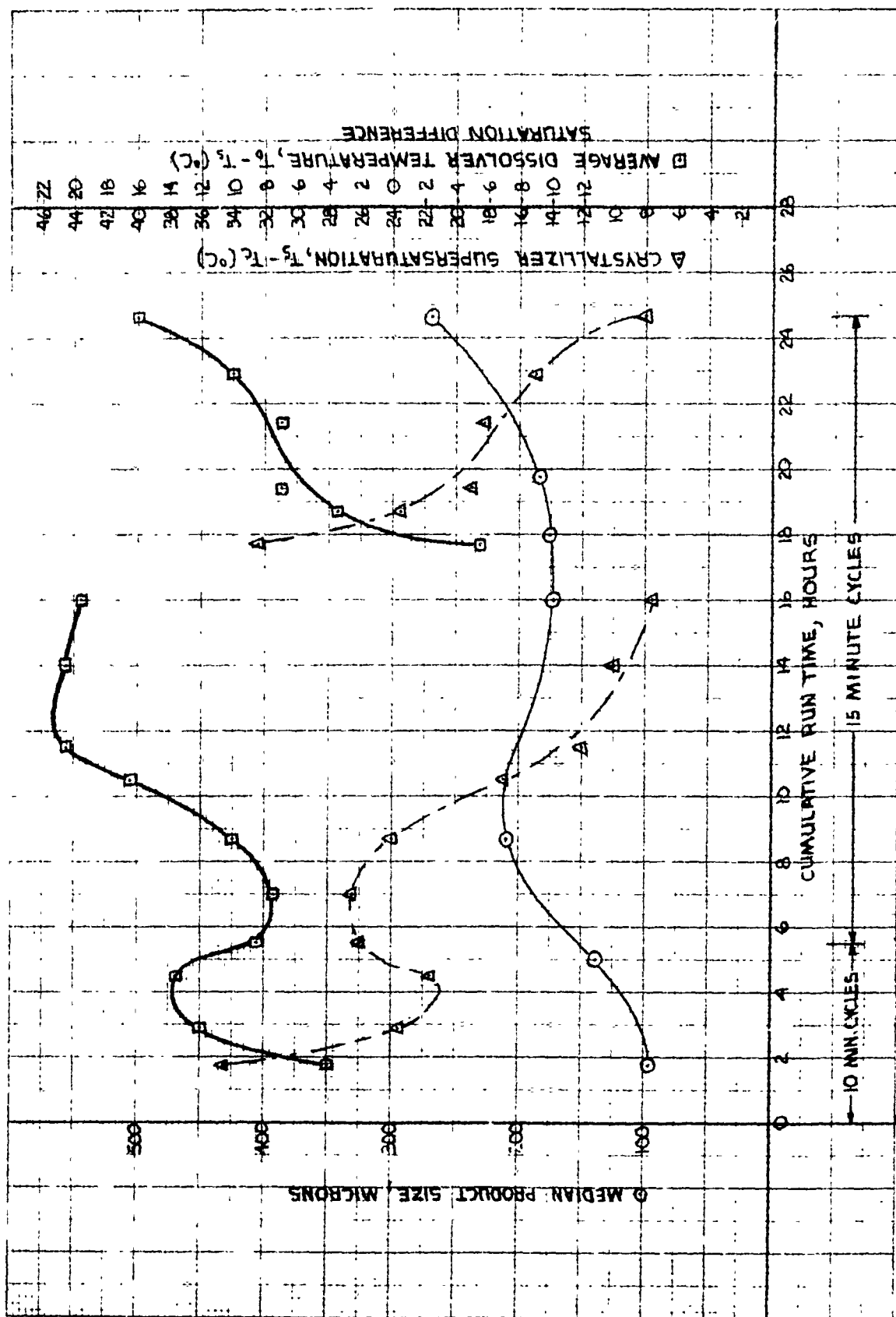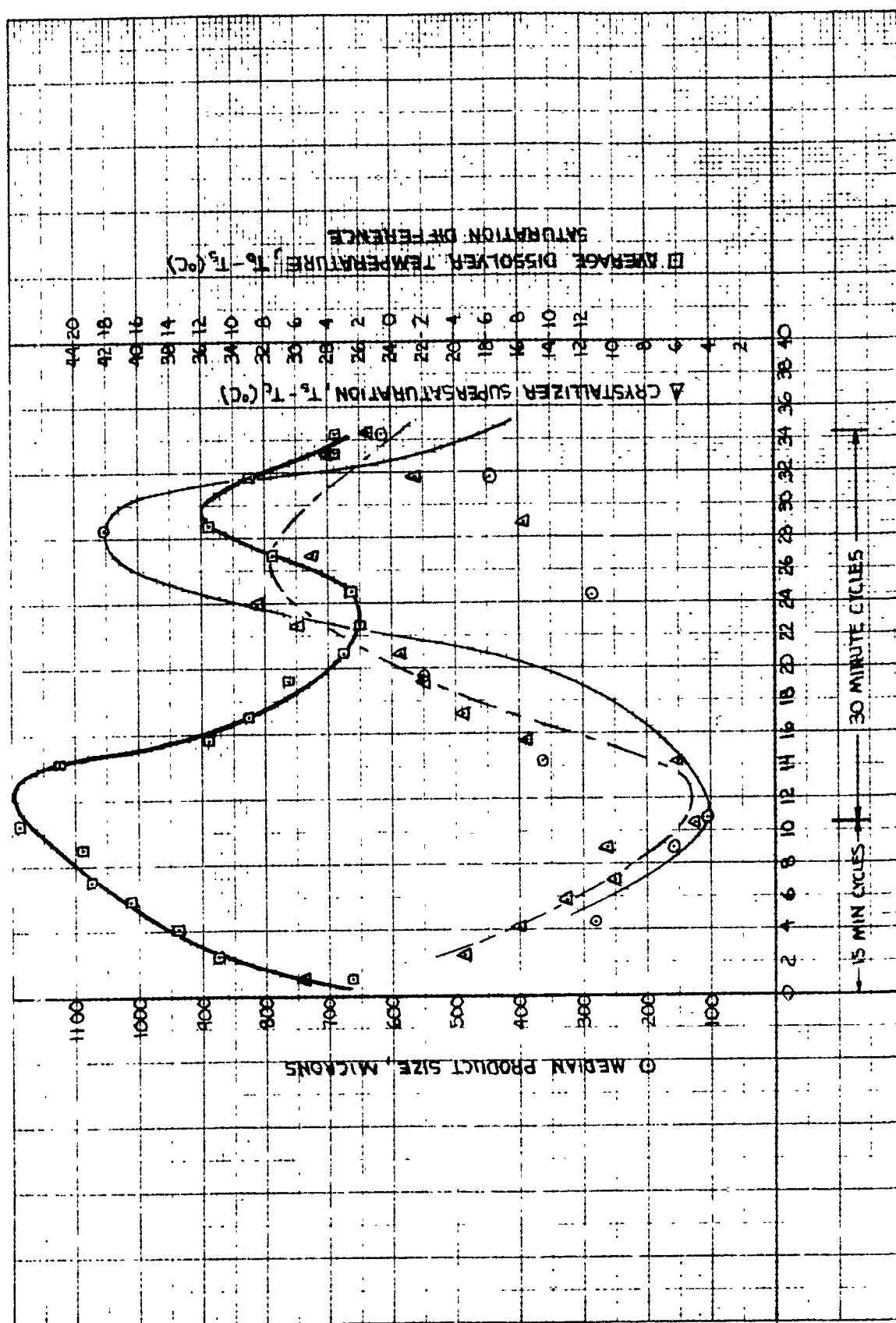
MIXING EQUATIONS FOR RDX/HMX,

RECYCLE SOLVENT AND DMSO

Mixing Equations for RDX/HMX, Recycle Solvent, and DMSO:
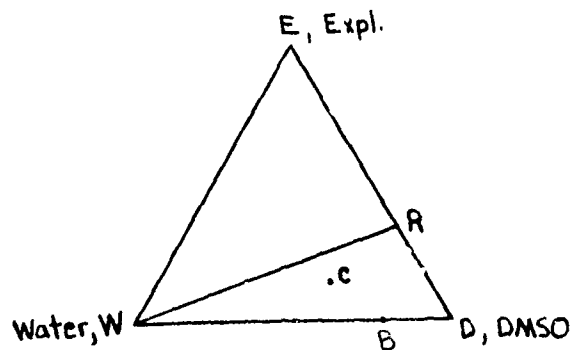
R = Operating Line Ratio = $\dfrac{\text{1b dry explosives}}{\text{1b dry explosives + 1b DMSO (100\% Basis)}}$

WR = Operating Line

M = Make-up Solvent Rate = $\dfrac{\text{1b DMSO (100\% Basis) in Make-up DMSO}}{\text{1b DMSO (100\% Basis) in Recycle Solvent}}$

A = Lb of Wet Explosives = Component A

B = Lb of Make-up Solvent = Component B

C = Lb of Recycle Solvent = Component C

W_i = Mass Fraction of Water in Component i.

D_i = Mass Fraction of DMSO in Component i.

E_i = Mass Fraction of Explosive in Component i.

ρ_i = Density of Component i (1b/gal)

Equations: (1) $\rho_c = \dfrac{1}{\dfrac{W_c}{8.34} + \dfrac{D_c}{9.17} + \dfrac{E_c}{\text{*Sp}^G{}_E(8.34)}}$ $\text{*Sp}^G{}_E$ — RDX = 1.8
                                                                                                HMX = 1.9

(2) $C = \rho_c \times$ (gals. of C) Measured in Mix Tank

(3) $B = \dfrac{MCD_c}{D_B}$

(4) $A = \dfrac{R(CD_c + BD_B) - CE_c\,(1-R)}{(1-W_A)\times(1-R)}$

Calculation Procedure:

1. Measure gallons of recycle solvent (Component C) in the slurry mix tank.

2. Sample and analyze Component C using LC for explosives (E_c), water (W_c), and DMSO (D_c).

3. Determine moisture content of wet, crude explosives (Component A) as W_A.

4. Calculate the density of Component C (ρ_c) using equation (1).

5. Calculate C using equation (2).

6. Given M and D_B, calculate B using equation (3).

7. Given R, calculate A using equation (4).

Example Calculation for HMX System:

Given – R = 0.335, D_B = 0.86, M = 0.20

Determined – gallons of C in mix tank = 42
W_A by LC analysis = 0.400
D_C by LC analysis - 0.729
E_C by LC analysis - 0.142
W_C by LC analysis - 0.129

Calculated – $\rho_c = \dfrac{1}{\dfrac{0.129}{8.34} + \dfrac{0.729}{9.17} + \dfrac{0.142}{(1.9 \times 8.34)}}$ = 9.622 lb/gal

$C = 9.622 \times 42 = 404.1$ lb recycle solvent

$B = \dfrac{(0.2)(404.1)(0.729)}{0.86} = 68.51$ lb. make-up solvent

$A = \dfrac{0.335\left[(404.1)(0.729)+(68.51)(0.86)\right] - \left[(404.1)(0.142)(1-0.335)\right]}{(1-0.4)(1-0.335)}$

= 201.2 lb wet HMX

APPENDIX D

PHYSICAL PROPERTIES OF DMSO

PHYSICAL PROPERTIES

TABLE 1 PROPERTIES OF DMSO

Molecular Weight.	78.13
Boiling Point at 760 mm Hg	189°C (372°F)
Melting Point	18.55°C (65.4°F)
Specific Gravity at 25°C (77°F)	1.096 (9.15 lb per gal)*
Refractive Index at 25°C.	1.477
Vapor Pressure at 25°C	0.62 mm Hg*
Surface Tension at 25°C	42.85 dynes per cm
Viscosity at 25°C.	2.00 Centipoise*
Specific Heat at 29.4°C.	0.47 ± 0.015 cal/(gm)(°C)*
Heat of Vaporization at 25°C.	12.64 Kcal/mole (291 Btu/lb)
at 189°C	10.31 Kcal/mole (237 Btu/lb)
Heat of Solution in Water at 20°C.	60 cal/gm
Heat of Fusion	44 cal/gm
Heat of Combustion at 25°C	6050 cal/gm
Flash Point (TOC).	95°C (203°F)
Ignition Temperature in Air.	300-302°C (570-575°F)
Flammability Limits in Air Lower	3-3.5 vol %
Upper	42-63 vol %
Thermal Coefficient of Expansion	0.00088 cc per degree C
Electrical Conductivity 20°C	3×10^{-8} ohm^{-1}cm^{-1}
80°C	7×10^{-8} ohm^{-1}cm^{-1}
Water Solubility.	Completely Miscible

*Additional data are found in the following tables and figures

FIGURE 1

VAPOR PRESSURES OF DMSO

TEMPERATURE °C

FREEZING POINT CURVES FOR DMSO—WATER SOLUTIONS

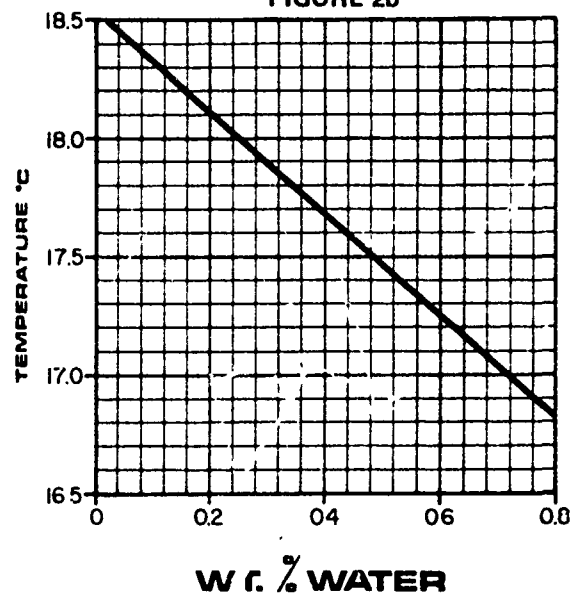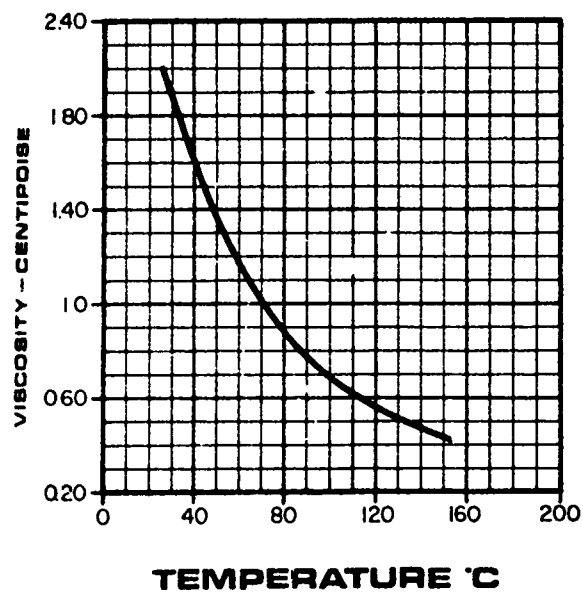## FIGURE 2a
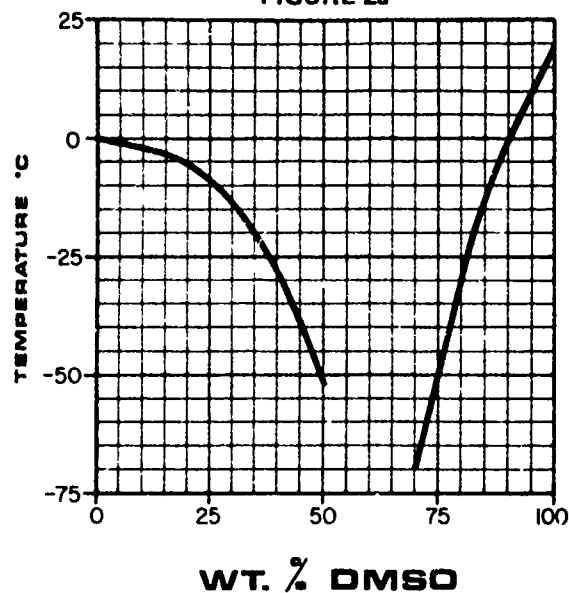
FIGURE 2b

FIGURE 3
VISCOSITY OF DMSO

FIGURE 4
VISCOSITY OF DMSO —WATER SOLUTIONS
25°C

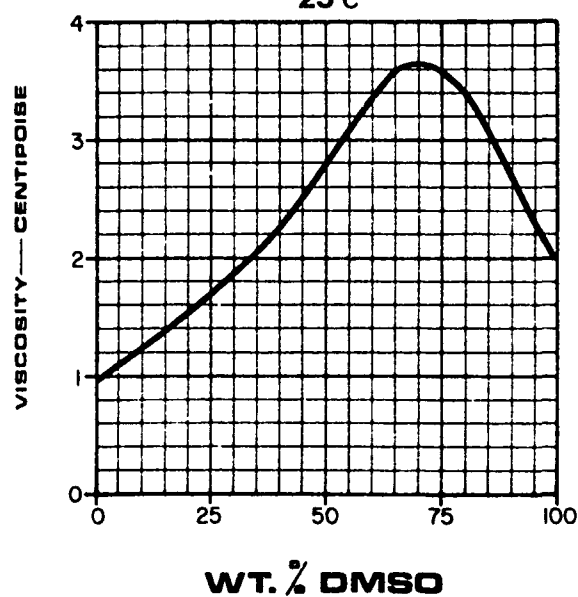## TABLE 2   HEAT CAPACITY OF DMSO

Temp, °C	Cp (liquid), cai/(g)(°C)
30	0.47
60	0.47
100	0.48
150	0.52

TABLE 3   DENSITY OF DMSO

Temp, °C	Density, grams/cc
25	1.096
60	1.062
100	1.023
150	0.974

TABLE 4 VAPOR–LIQUID EQUILIBRIUM DMSO–WATER

(ONE ATMOSPHERE PRESSURE)

TEMPERATURE °C	MOL FRACTION WATER IN LIQUID	MOL FRACTION WATER IN VAPER
100.0	1.000	1.000
100.6	0.988	0.9998
101.0	0.975	0.9997
102.0	0.945	0.9994
103.3	0.909	0.9989
105.0	0.865	0.9983
108.0	0.810	0.997
113.0	0.740	0.994
118.0	0.675	0.990
120.0	0.645	0.986
130.0	0.513	0.964
143.0	0.378	0.921
149.0	0.313	0.890
165.0	0.176	0.773
174.5	0.100	0.628
177.0	0.081	0.573
183.0	0.046	0.353
184.6	0.034	0.282
187.7	0.011	0.100
189.0	0.000	0.000

FIGURE 6
BOILING TEMPERATURE CURVE
FOR DMSO — WATER SOLUTIONS
(ONE ATMOSPHERE PRESSURE)

WEIGHT FRACTION—WATER
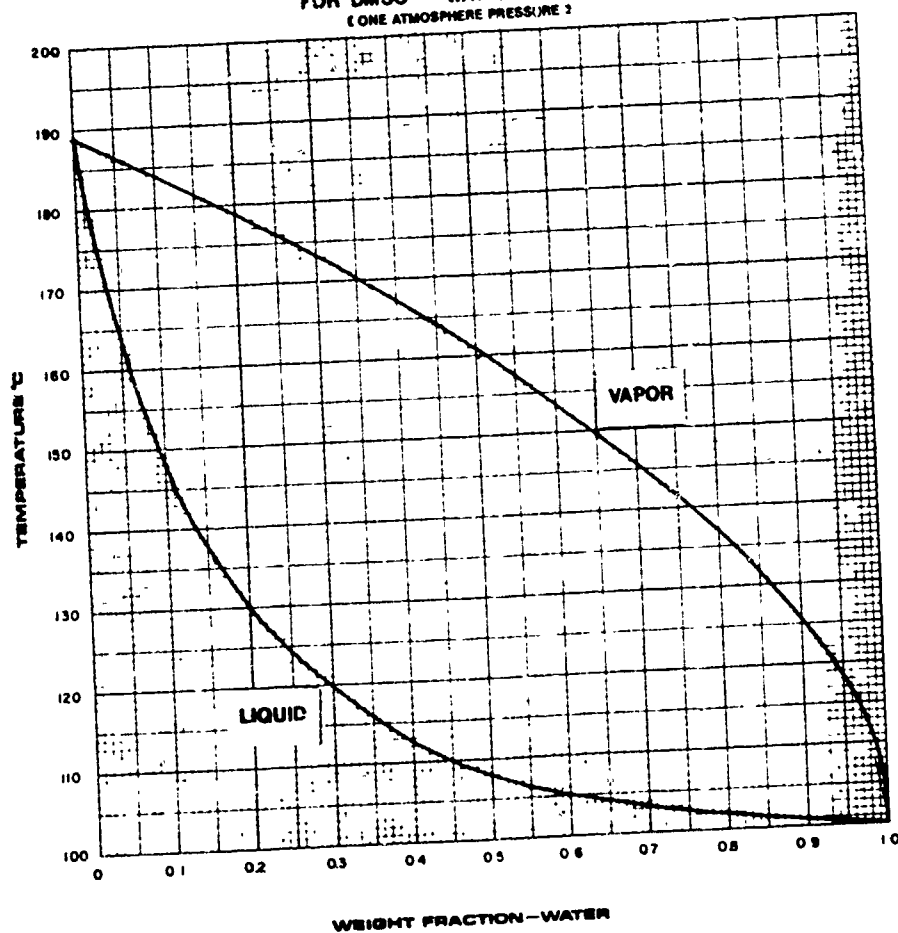
FIGURE 7
VAPOR LIQUID EQUILIBRIUM CURVE
DMSO WATER AT ONE ATMOSPHERE

163
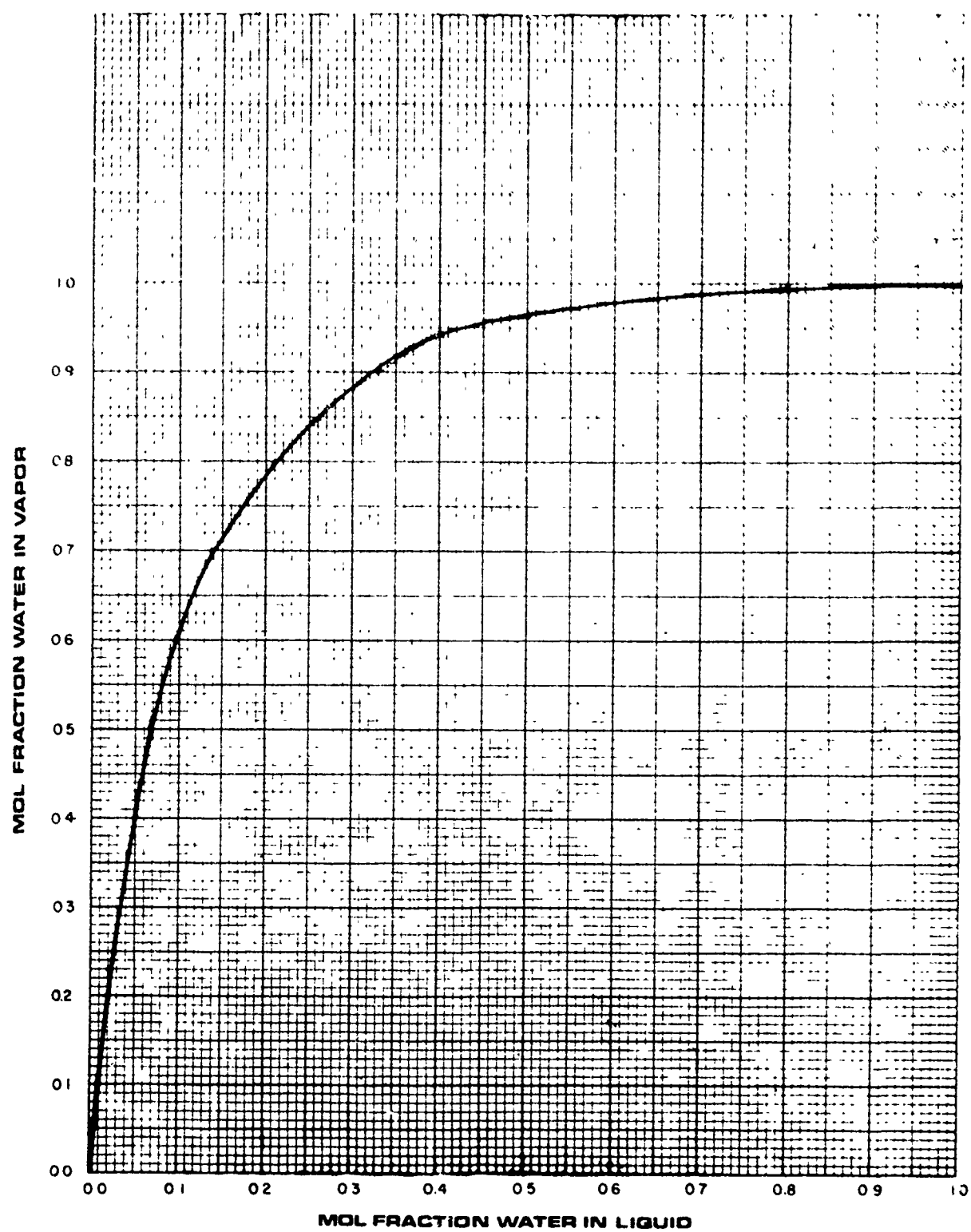
FIGURE 8

THEORETICAL PLATE REQUIREMENT
DMSO/WATER SYSTEM

CONDITIONS:

REFLUX RATIO	1:1
OVERHEAD	500 PPM DMSO
BOTTOMS	500 PPM H_2O
PRESSURE	1 ATMOSPHERE
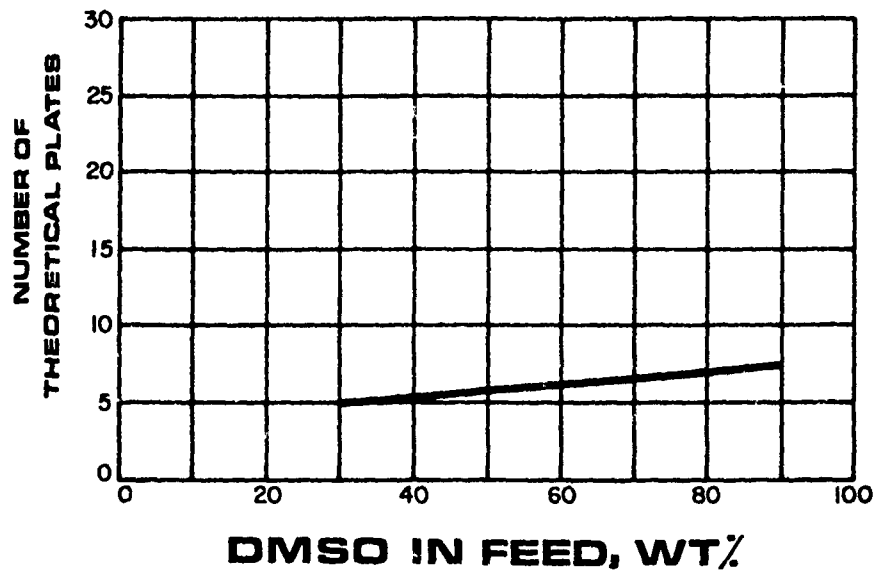
FIGURE 9

REFLUX REQUIREMENTS — DMSO/WATER SYSTEM

CONDITIONS:

THEORETICAL PLATES	12
OVERHEAD	500 PPM DMSO
BOTTOMS	500 PPM H_2O
PRESSURE	1 ATMOSPHERE

164
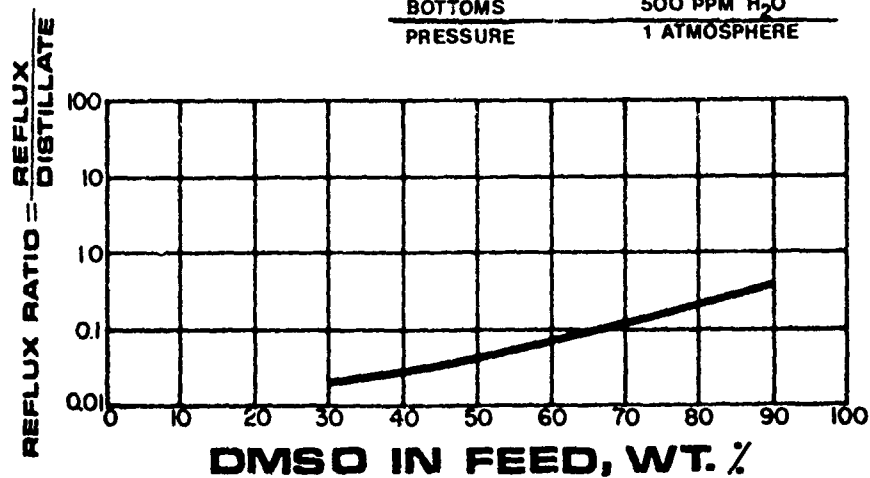
FIGURE 10

THERMAL DECOMPOSITION OF DMSO

165
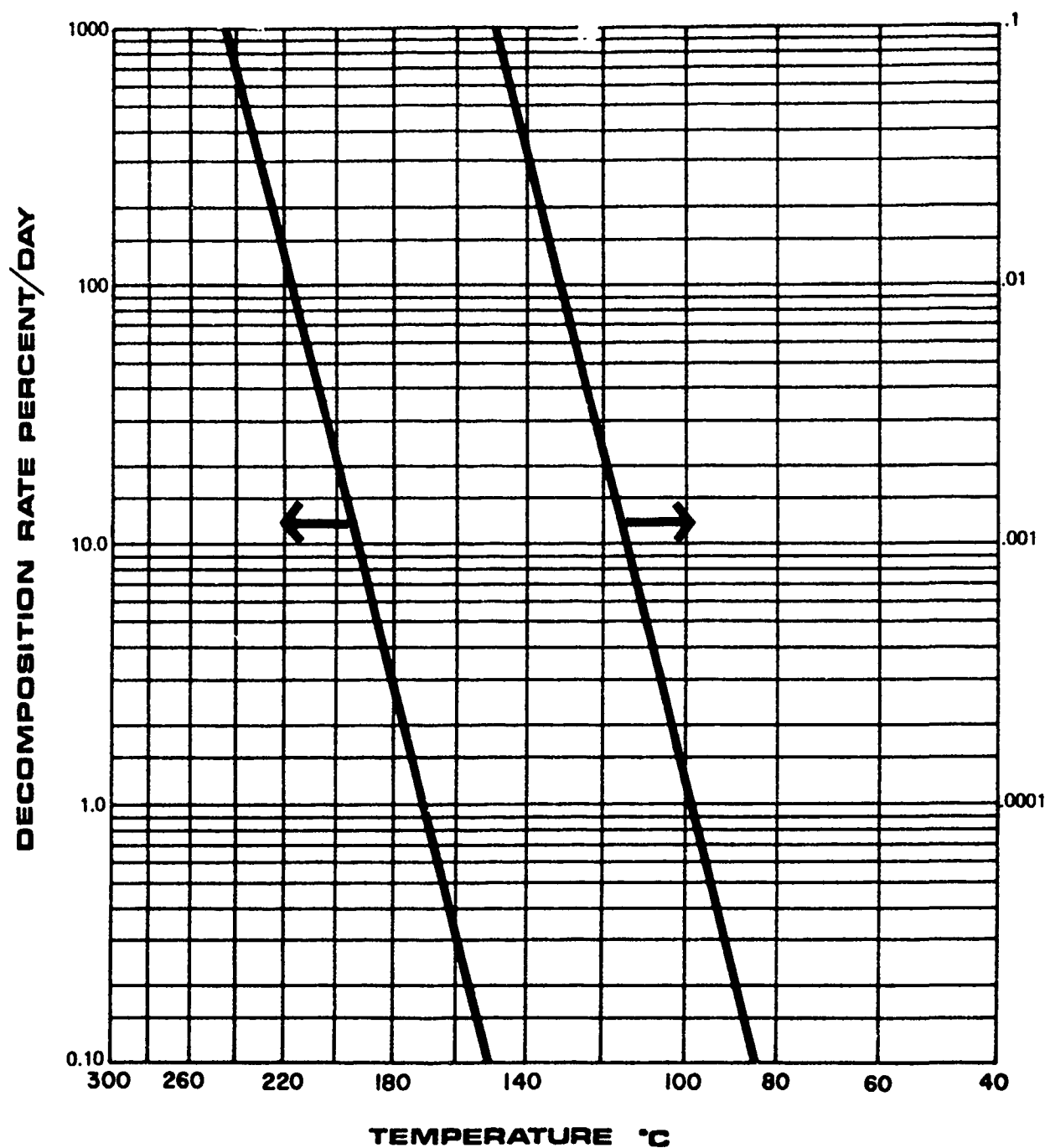
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