Closed Loop Energetics with VOC Emission Reduction (CLEVER)

Indian Head Division
Naval Surface Warfare Center

12 July 2002

Prepared for:	Prepared by:
Environmental Security Technology	Indian Head Division
Certification Program Office	Naval Surface Warfare Center
Arlington, VA 22203	Indian Head, MD 20640-5035

ESTCP Project Number PP-9704

Table of Contents

1. INTRODUCTION 5
1.1 Background Information 5
1.2 Official Department of Defense Requirement Statement 7
1.3 Objective of the Demonstration 8
1.4 Regulatory Issues 8
1.5 Previous Testing of the Technology 9
2. TECHNOLOGY DESCRIPTION 19
2.1 Background and Applications 19
2.2 Strengths, Advantages and Weaknesses 30
2.3 Factors Influencing Cost and Performance 30
3. SITE/FACILITY DESCRIPTION 32
3.1 Facility Background 32
3.3 Site Waste Characteristics 34
3.3 Site Maps & Photographs 34
4. DEMONSTRATION APPROACH 38
4.1 Performance Objectives 38
4.2 Physical Set-Up and Operation 39
4.3 Sampling Procedures 50
4.4 Analytical Procedures 54
5. PERFORMANCE ASSESSMENT 63
5.1 Performance Data and Assesment for Paste and Grains 63
5.2 Data Assessment 79
5.3 Technology Comparison 79
6. COST ASSESSMENT 83
6.1 Cost Performance 83
6.2 Cost Comparison to Conventional and Other Technologies 85
7. REGULATORY ISSUES 87
7.1 Approach to Regulatory Compliance and Acceptance 87
7.2 Regulatory Interactions 87
7.3 Review of Regulatory Drivers 88
7.4 Public Participation 89
8. TECHNOLOGY IMPLEMENTATION 89
8.1 DoD Need 89
8.1 Transition 91
9. LESSONS LEARNED 91
10. REFERENCES 92

Appendices

Appendix A: Points of Contact 99
Appendix B: Supporting Data and Documentation 101

(1) Detail Specification: High Energy Nitramine Gun Propellant (EX 99)
(2) Gallant, F.M.; *Discussion of Process Effects on EX 99 Propellant Characteristics and Continuous Extrusion Behavior*; IHTR 2310; January 5, 2001
(3) Lekander, Sven; *CLEVER Report, Additional Work*; Bofors Technical Report XU 5-99/420 October 8, 1999.
(4) Walsh, Christine; *Effects of Water Bath Collection on EX 99 Propellant Grains*; November 27, 2000.
(5) WGI Letter 79845.001.WGI:L-008 to Mr. Robert Thompson; Continuous Processing Scale-Up Facility VOC Emissions (Contract N00174-00-C-0039); January 2, 2001
(6) Raw Data From TSE Run #IH94000E-EX99-0081
(7) Raw Data From TSE Run #IH94000E-EX99-0083
(8) IHDIV, NSWC Analysis of Paste Batch 485/98
(9) IHDIV, NSWC Analysis of Paste Batch 486/98
(10) Microscopic Determination of Degree of Coating for Two Bofors Lova Powders; IHDIV, NSWC Memorandum 8000 Ser 3330H/162; October 20, 2000
(11) Small Scale Shock Sensitivity Test for EX 99 Lova Powder; IHDIV, NSWC Memorandum 2800 Ser 3310L/48/pw; August 13, 1998
(12) Sensitivity Testing for EX 99 Lova; IHDIV, NSWC Memorandum 5100 Ser 9410G/148/dr; August 28 2000
(13) Gap Test to Measure Shock Sensitivity (UN Test 7-3a(1)) Results of EX 99 TSE Grain Propellant Lot #IH-940-EX99-0088; IHDIV, NSWC Memorandum; October 31, 2000
(14) Cap Sensitivity Test (UN Test 5 (a)) Results of EX 99 TSE Grain Propellant Lot #IH-940-EX99-0088; IHDIV, NSWC Memorandum; October 31, 2000
(15) Ballistic Impact Chamber Test Results Comparing EX 99 Prepared by Twin Screw and Batch Processes; IHDIV, NSWC Memorandum; January 22, 2001
(16) Evaluation of EX 99 Lova Grains by Scanning Electron Microscopy; IHDIV, NSWC Memorandum 8010 Ser 3320I/062; March 16, 2000
(17) Variable Confinement Cookoff Test (VCCT) Results of Lova EX 99; IHDIV, NSWC Memorandum 8000 Ser 9510D/65/rh; December 5, 2000
(18) Greenberg, Dr. B and Kalyon, Dr. D; Structural Analysis of Twin Screw Extruded and Batch Processed EX 99 Lova Grains; December 2, 2000
(19) Closed Bomb Data for Twin Screw EX 99 Propellant Lot IH940-00E-EX-99-0088 and 0089; IHDIV, NSWC Memorandum 3965 Ser 3310E/54/kce; June 30, 2000
(20) Probe Testing of EX99 Twin Screw Processed Propellant; IHDIV, NSWC Memorandum 8030 Ser 621K/85; September 8, 2000
(21) Krawczyk, Jerry and Kowalczyk, Daniel; Closed Loop Energetics with VOC Emission Reduction Cost and Environmental Analysis; Booz-Allen & Hamilton, Inc. (8283 Greensboro Drive, McLean, VA 22102) Report for IHDIV, NSWC; December 2000

Table of Figures

Figure 1: EX 167 Propelling Charge 6
Figure 2: Bofors' Precipitation Process for Lova Nitramine Gun Propellant 10
Figure 3: 5"/54 Gun Maximum Pressure Data 13
Figure 4: 5"/54 Gun Velocity Data 14
Figure 5: M43 Batch Process Flow Diagram 15
Figure 6: Material Balance for Nitramine Fine Grind Facility (NFGF) 16
Figure 7: Material Balance for Nitramine Gun Propellant Facility (NGPF) -Virgin Mixing 17
Figure 8: Material Balance for NGPF - Rework Mixing 17
Figure 9: Material Balance for NGPF - Pressline (Blocking/Straining/Extrusion) 18
Figure 10: Material Balance for NGPF - Cutting 18
Figure 11: Material Balance for FA - Drying/Glazing/Blending 19
Figure 12: K-Tron Model T-37 Solid Feeder 24
Figure 13: Solid Refill Hoppers 25
Figure 14: W&P 40-mm Extruder Segmented Screws 26
Figure 15: W&P 40-mm Extruder Barrel Sections (Side View) 26
Figure 16: Sketch of Extruder Configuration for Lova TSE Processing 27
Figure 17: Twin-Screw Extruder Facility Control Room 28
Figure 18: Gun line at Main Range at DD, NSWC 29
Figure 19: IHDIV, NSWC 33
Figure 20: A Sketch of Bofors in Karlskoga, Sweden 35
Figure 21: TSE Facility Picture 36
Figure 22: TSE Building Floor Plan 37
Figure 23: Material Balance for CLEVER 38
Figure 24: Bofors Solid Feeder Refill System 40
Figure 25: IHDIV, NSWC Refill Cylinders 41
Figure 26: Lova Twin-Screw Extrusion and Pelletizing Equipment Set-Up 43
Figure 27: Barrel Configuration and Screw Configuration #48 44
Figure 28: Bofors Precipitation Process Sampling Locations 51
Figure 29: TSE Sampling Locations for EX 99 Propellant 52
Figure 30: Instrumented 5-inch Gun Barrel 61
Figure 31: Schematic EX 167 Propelling Charge Loaded for Proof Testing 62
Figure 32: Material Balance for Extruder Run IH94000E-EX99-0081 50
Figure 33: Material Balance for Extruder Run IH94000E-EX99-0083 50
Figure 34: DSC Trace for Paste Batch 485/98 65
Figure 35: Photomicrograph of Paste Batch 485/98 66
Figure 36: Cap Test Failure 68
Figure 37: Cap Test Configuration 69
Figure 38: Grain Mechanical Properties 70
Figure 39: Pressure vs. Time Comparison 77
Figure 40: Navy Need for Lova Nitramine Gun Propellant 90

Table of Tables

Table 1: Actual IH 1 Data 11
Table 2: Actual IH 1 Material Balance (Derived from Table 1 Data) 11
Table 3: 5"/54 Gun Firings at DD, NSWC, 12/16/96 13
Table 4: Precipitation Process Performance Factors 30
Table 5: Summary of Wastes from the Precipitation Process 34
Table 6: EX 99 Formulation 39
Table 7: Performance Objectives
Table 8: Precipitation Process Samples for EX 99 Propellant 51
Table 9: Description of TSE Samples 52
Table 10: Propellant Paste and Grain Test Matrix 53
Table 11: Sensitivity Ranges for Safety Testing 56
Table 12: Firing Lineup/Propelling Charge Configuration 63
Table 13: Bofors' Analytical Results of Paste Batches 64
Table 14: DSC Results 64
Table 15: RDX Particle Size 65
Table 16: Paste Safety Test Results 66
Table 17: Grain Safety Test Results 67
Table 18: Grain Dimensional Variation 71
Table 19: Paste and Grain Chemical Composition Comparision 72
Table 20: High Pressure Closed Bomb Data Summary for Lot IH94000E-EX99-0088 73
Table 21: High Pressure Closed Bomb Data Summary for Lot IH94000E-EX99-0089 74
Table 22: Gun Firing Results 75
Table 23: Data for the 26 lb Propellant Charges 76
Table 24: Project Assumptions for Cost Analysis 83
Table 25: EX 99 Propellant Material/Formulation 84
Table 26: CLEVER Cost Summary 84
Table 27: Net Present Value 86
Table 28: Internal Rate of Return 86
Table 29: Discounted Payback Period 86

List of Acronyms

AGS	Advanced Gun System
ABL	Allegheny Ballistics Laboratory
ANSR	Autonomous Naval Support Round
ARL	Army Research Laboratory
ATEC	Acetyl Triethyl Citrate
BDNPA/F	2,2 bis-dinitro propyl acetal/formal
BIC	Ballistic Impact Chamber
CAA	Clean Air Act
CAB	Cellulose Acetate Butyrate
CAS	Chemical Abstract Service Number
CLEVER	Closed Loop Energetics with VOC Emission Reduction
CWA	Clean Water Act
DDT	Deflagration-to-Detonation Transition
DoD	Department of Defense
DOE	Design of Experiments
DSC	Differential Scanning Calorimetry
EA	Environmental Assessment
EC	Ethyl Centralite
ERGM	Extended Range Guided Munition
ESD	Electrostatic Discharge
ESH	Environmental, Safety and Health
EtAc	Ethyl Acetate
FA	Finishing Area
FASM	Forward Area Support Munition
FY	Fiscal Year
HOE	Heat of Explosion
HPLC	High Pressure Liquid Chromatography
IHDIV	Indian Head Division
IHGP	Indian Head Gun Propellant
IHTR	Indian Head Technical Report
IRR	Internal Rate of Return
L/D	Length to Diameter Ratio
LICA	Liquid Coupling Agent
LOVA	Low Vulnerability Ammunition
MSDS	Material Safety Data Sheet
MEK	Methylethylketone
MJ	Megajoule
NEPA	National Environmental Policy Act
NFGF	Nitramine Fine Grind Facility
NGPF	Nitramine Gun Propellant Facility
NPV	Net Present Value
NSFS	Naval Surface Fire Support
NSWC	Naval Surface Warfare Center
NC	Nitrocellulose
NOS	Naval Ordnance Station (previous name of IHDIV)

nv	Non-volatiles
OMB	Office of Management and Budget
PBX	Plastic Bonded Explosive
RDX	Hexogen
RCRA	Resource Conservation and Recovery Act
SEM	Scanning Electron Microscopy
SOP	Standard Operating Procedure
TCIS	Thermal Catalytic Incineration System
TEGDN	Triethylene Glycol Dinitrate
TMETN	Trimethylol Ethane Trinitrate
TPE	Thermoplastic Elastomer
TPT	Temperature/Pressure Transducer
TSE	Twin-Screw Extruder
VCCT	Variable Confinement Cook-off Test
W&P	Werner & Pfleiderer
WA-XRD	Wide Angle X-ray Diffraction
WO	White Oak (previous division of NSWC)

Executive Summary

Background: The work reported herein was funded by the Environmental Security Technology Certification Program Office and Crane Division, NSWC (Code PM4).

Every year the Department of Defense (DoD) produces thousands of pounds of solvent mixed, nitramine based propellants in the production of gun propelling charges. The solvents tend to be Volatile Organic Compounds (VOCs). The conventional nitramine gun propellant production method (Batch process) does not recover or recycle the solvent VOC emissions. The VOCs are allowed to be released, untreated, into the atmosphere.

The Closed Loop Energetics with VOC Emission Reduction (CLEVER) process is a radical change from the conventional manufacturing method, which typically loses one pound of solvent directly to the atmosphere for every three pounds of propellant made. The CLEVER process is defined as the precipitation process followed by processing in a twin-screw extruder (TSE). The precipitation process, developed and patented by Nexplo-Bofors in Sweden, replaces conventional ingredient preparation and mixing. It is a closed loop process that fully dissolves all propellant ingredients into solvent and then processes the solution through a steam precipitator. The precipitator is used to evaporate the solvent thus precipitating a propellant powder. The evaporated solvent is recovered and recycled. The dried propellant powder is resolvated in a twin-screw extruder for final extrusion and cutting. Data on solvent usage, emissions and scrap generation were collected during both the precipitation process and twin-screw process to allow comparison with data developed from conventional production records.

Objective: The objective was to demonstrate the ability of the CLEVER process to cost effectively produce acceptable propellant while significantly reducing both the VOC emissions and the generation of hazardous solid wastes. The vehicle chosen for the demonstration was the production of two small lots of EX 99 propellant grains for testing in the new Extended Range Guided Munitions (ERGM) round for the Navy's 5-inch gun.

Results: The CLEVER process demonstration conducted between the Fall of 1997 and Spring of 2000 were highly successful. The CLEVER process demonstrated the ability to reduce VOC emissions by 47% and hazardous solid waste (scrap propellant) by 50% while reducing propellant cost by 42% based only on labor and materials costs. Using the net present value method, which includes facilities amortization costs, a net savings of 18% is realized when compared to the conventional batch process. The propellant quality was equal to or better than comparable batch produced propellant. Both demonstration lots were successfully gun fired and performance as measured by muzzle energy, chamber pressure, and velocity variation were outstanding, as good or better than anything that has been tested to date. On the critical parameter of muzzle energy, it exceeded the 18 MJ minimum requirement without exceeding 65,000 psi breech pressure.

Closed Loop Energetics with VOC Emission Reduction (CLEVER)

Indian Head Division
Naval Surface Warfare Center

1 August 2001

1. Introduction

1.1 Background Information

This demonstration was designed to address the need for an improved production process for nitramine based gun propellants. The Department of Defense (DoD) requires several million pounds per year of solvent processed propellants for gun propelling charges. Most of this propellant is solvent mixed in a process that uses Volatile Organic Compounds (VOCs) such as ethanol and ethyl acetate. The current process uses approximately one pound of solvent for every three pounds of propellant produced. This solvent is evaporated into the atmosphere during processing or thermally treated.

Presently, much of the current propellant processing capacity is idle because of reduced ammunition requirements. Production requirements are expected to increase with the introduction of new Navy gun ammunition and Army howitzer and tank ammunition. Development of a cost-effective propellant manufacturing process, with better pollution prevention capability, would significantly benefit current production facility restarts intended to meet increased production demands.

The demonstrated process, CLEVER, is a radical departure from the old process that emitted VOCs to the atmosphere via evaporation. The Bofors Explosives Company in Sweden has developed a closed loop process (US Patent Number 4,982,235, see Section 10 of this report) that fully dissolves all propellant ingredients into solvent. The solution is then processed through a steam precipitator that evaporates the solvent and precipitates a finely divided propellant powder. The evaporated solvent is recovered and recycled. This process is called the Bofors Precipitation Process. To transform the finely divided powder into the desired configuration for use in a gun, the precipitate is dried and then conveyed to a twin-screw extruder (TSE), for combination with only enough processing solvents to provide desired mechanical properties for extrusion and cutting (typically 10-13% by weight). The process was designed specifically for finely divided RDX and RDX propellants.

The Closed Loop Energetics with VOC Emission Reduction (CLEVER) process is defined as the precipitation process followed by processing in the TSE. The CLEVER process keeps nearly all solvents in a closed system for pollution prevention. The CLEVER process is also much less labor intensive than the existing conventional batch process and, therefore, it results in a lower cost propellant. The performance of the CLEVER propellant is equivalent to or better than that of the conventionally batch processed material. Therefore, the CLEVER process is a significant environmental improvement for the planned gun propellant production as well as a cost-effective approach.

The evaluation of the CLEVER process is very timely. Typically, it is difficult to insert new processing technology into ongoing programs. However, in this case, the Navy is currently developing two new propelling charges for the five-inch gun. The Navy has a new Extended Range Guided Munitions (ERGM) effort in RDT&E, which is the cornerstone of its Naval Surface Fire Support (NSFS) Program. A new propelling charge (EX 167, pictured below in Figure 1) is being designed for the ERGM.

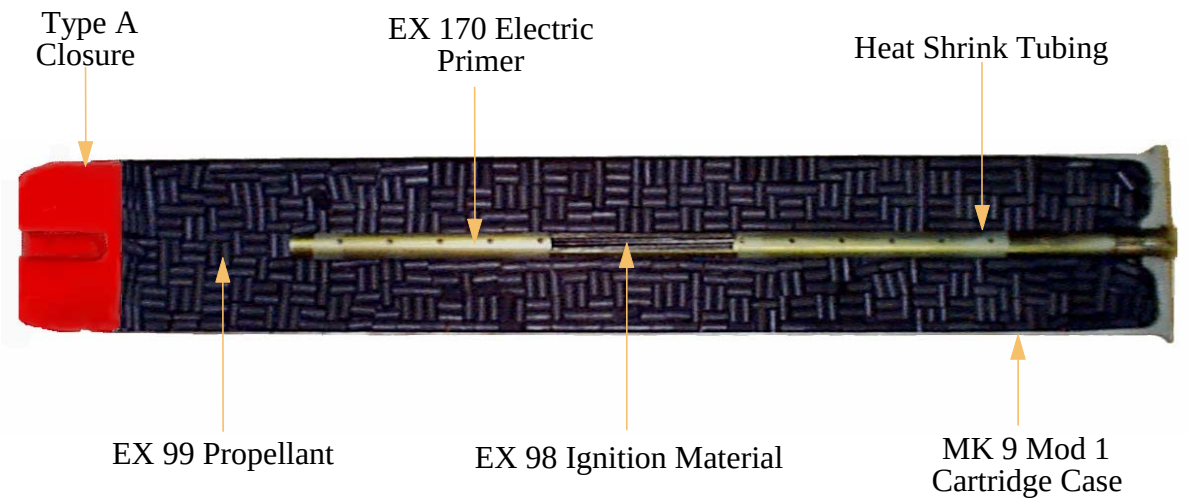


Figure 1: EX 167 Propelling Charge

The propellant planned for this propelling charge is EX 99 Lova nitramine gun propellant. EX 99 is essentially M43 Lova nitramine gun propellant without 0.5% Liquid Coupling Agent (LICA-12). EX 99 is Navy nomenclature and M43 is Army nomenclature. The EX 99 formulation planned for ERGM is the formulation that was demonstrated in the CLEVER project. Additionally, the Navy's new EX 167 Propelling Charge was the vehicle to both demonstrate the new technology and introduce the improved process into production. Production requirements for this new charge are currently projected at 2000 charges (60,000 pounds of propellant) per year for FY 05 through FY 14. This equates to a total of 600,000 pounds of propellant. The other propelling charge that the Navy is currently developing is a new EX 175 Propelling Charge that uses similar hardware and propellant. Once implemented, the EX 175 Propelling Charge will be used with the new Cargo projectile. Current production requirements for the Navy's Cargo Round are also projected at 2000 rounds (60,000 pounds of

propellant) per year for FY 03 through FY 14. This equates to a total of 720,000 pounds of propellant. Additionally, once the technology is proven, it will also be available to manufacture propellant for the Army, Air Force, and the private sector for use in automotive air bags.

1.2 Official Department of Defense Requirement Statement

1.2.1 Requirements. The development of this technology addresses two high priority requirements of the Compliance Pillar of the Department of Defense (DoD) Environmental Security program:

ID#: 3.I.6.c
Title: Energetics production pollution prevention
Tech Area: Pollution Prevention
Priority: High
Media: Air, Water, and Soil
Containment: Metals, Energetics
Drivers: Clean Air Act (CAA), Clean Water Act (CWA), and Resource Conservation and Recovery Act (RCRA)
Description: Manufacturing and processing of energetic materials for ordnance items results in large amounts of waste (e.g. extrusion scrap, contaminated salts, redwater) which are not recycled or recovered because of specification, safety, technological, or market constraints. ***New manufacturing processes are required to reduce hazardous waste and effluent generation, avoiding the necessity for open burning/open detonation, costly commercial disposal of hazardous waste, and expensive end-of-pipe treatment.***

ID#: 3.I.13.a
Title: Reuse/recycle of hazardous/polluting materials
Tech Area: Pollution Prevention
Priority: High
Media: Water, Solids
Containment: POL's, Paints, Solvents, other
Drivers: EO-12856; Resource Conservation and Recovery Act (RCRA)
Description: Successful effort will reduce hazardous waste generation, result in less environmental pollution, safer working conditions, and reduced disposal costs. Readiness and relations with the public will be improved. Disposing of hazardous waste is a Navy fleet/facility wide problem. Process improvements and advanced technologies in the reuse and recycle of hazardous waste must be developed. Disposal of hazardous waste is costly and will continue to be more so as disposal sites become scarcer.

1.2.2 How Requirements Were Addressed. The CLEVER process consists of the precipitation process to manufacture a readily feedable preblend that is then fed to a TSE for final extrusion. First, any reject or waste feedstock (from the precipitation process) or propellant (from the extrusion process) can be dissolved in the solvent and re-precipitated, thus, essentially eliminating or minimizing any propellant waste from any part of the process. Second, hazardous waste is also reduced at the extrusion process because all of the operations in the extrusion operation are conducted in a continuous and remote manner. Therefore, there is no need to clean out the machine until all of the production is complete and then the total amount of waste is less

than that from the conventional batch process. In the conventional batch process, there are wastes from each mix that is made, unusable heels of propellant from every block of propellant that is extruded into grains, and so on. The only explosive waste that is generated from the twin-screw process occurs during start up and shutdown.

The CLEVER process reduces VOC emissions in several ways. The Bofors Precipitation Process eliminates the need to dry and grind RDX to the proper particle size and, therefore, eliminates the VOC emissions from the drying step. The closed loop precipitation process uses solvents but the solvent is recycled. Both solvent waste and VOC emissions are minimized. Finally, products can be manufactured on a twin-screw extruder with less or no solvents thus reducing or eliminating VOC emissions. In the case of Lova nitramine gun propellants, solvents are still required but to a much lesser extent. A total of approximately 12% solvent is required to mix and extrude nitramine gun propellant versus 30% for mixing by the conventional batch process followed by 12% for extrusion.

1.3 Objective of the Demonstration

The project demonstrated the ability of the CLEVER process to cost effectively provide acceptable propellant with significantly fewer wastes than the conventional batch process. The U. S. Navy's 5-inch gun propelling charge provided a means for demonstration of this capability.

1.3.1 Scope and Location of the Demonstrations. The propellant paste was made by Bofors in Karlskoga, Sweden using their precipitation process. The paste was then transported to Indian Head Division, Naval Surface Warfare Center (IHDIV, NSWC) and extruded by the TSE process. Environmental impacts of CLEVER were then compared to existing data from the conventional process for the manufacture of nitramine gun propellant. Both CLEVER and conventionally processed propellant were loaded into propelling charges and ballistically evaluated at Dahlgren Division, Naval Surface Warfare Center (DD, NSWC) to determine whether the two processes produced similar results in actual end use.

1.4 Regulatory Issues

Code of Maryland Regulation 26.11.19.25 states: The owner or operator of existing explosives and propellant manufacturing equipment subject to this regulation shall install a VOC control device having a VOC destruction or removal efficiency of 85% or more overall on all active nitramine propellant manufacturing equipment that has a capacity of 150 gallons or more. The nitramine manufacturing facility is centered around a 150-gallon batch mixer. Currently, over $1,400,000 is being spent to install a Thermal Catalytic Incineration System (TCIS) at this facility. This system was installed because it was the only viable pollution control solution available at the time. Operating at its projected 99% efficiency, this system will reduce the VOC emissions from the current 27.0 lb/hr to 0.27 lb/hr during normal operations. This solution provides VOC emission reduction at the mixer only.

The Clean Air Act (CAA), Clean Water Act (CWA), and the Resource Conservation and Recovery Act (RCRA) are other underlying regulatory issues that emphasize the nationwide reduction or elimination of hazardous materials or waste. CLEVER will significantly reduce waste amounts by replacing both the grinding and mixing operations with the precipitation process. The precipitation process lowers waste significantly because of the recycling done on most of the resulting wastes. Waste is also reduced in the TSE process by replacing the mixing,

blocking, straining, and extruding processes with one continuous process.

1.5 Previous Testing of the Technology

The precipitation process used to make a multi-component single feedstock coupled with TSE processing into extruded grains has been used by Bofors in Sweden to manufacture nitramine based gun propellants commercially since the early 1990s. In the last several years, Bofors has also adapted this process for their commercial air bag propellant business.

IHDIV, NSWC and Bofors Explosives have been working together since the early 1990s to develop a process that would redefine the method of producing nitramine based gun propellants for the U.S. Navy. The contract (N00174-94-C-0041) began with small scale testing using various solvents and plasticizers. Once a baseline was established, the necessary testing performed, and data analyzed; the chosen formulation and process was manufactured on full-scale equipment. Since the joint venture first began, the nitramine gun propellant formulations have changed somewhat and the equipment has been modified to meet the needs of the Navy.

For the purposes of this demonstration, sampling shall be defined as efforts performed to collect data to conduct the material balance. Testing shall be defined as efforts performed to collect data through laboratory testing or gun firing. Various sampling and testing have been performed on previously processed Bofors material, TSE propellant and conventionally processed propellant. The lessons learned and appropriate data will be discussed below.

1.5.1 Sampling and Testing of the Bofors Precipitation Process during the Joint Effort Between Bofors and IHDIV on Lova Nitramine Gun Propellant Development. The information and data provided in this section is taken from the Bofors Technical Report XU 5-96/237 prepared by Bofors Explosives under IHDIV Contract N00174-94-C-0041; see reference 1.

1.5.1.1 Sampling. Figure 2 is a block diagram of the sample collection locations for the feasibility demonstration of the precipitation process. The dashed boxes represent closed systems for material balance purposes.

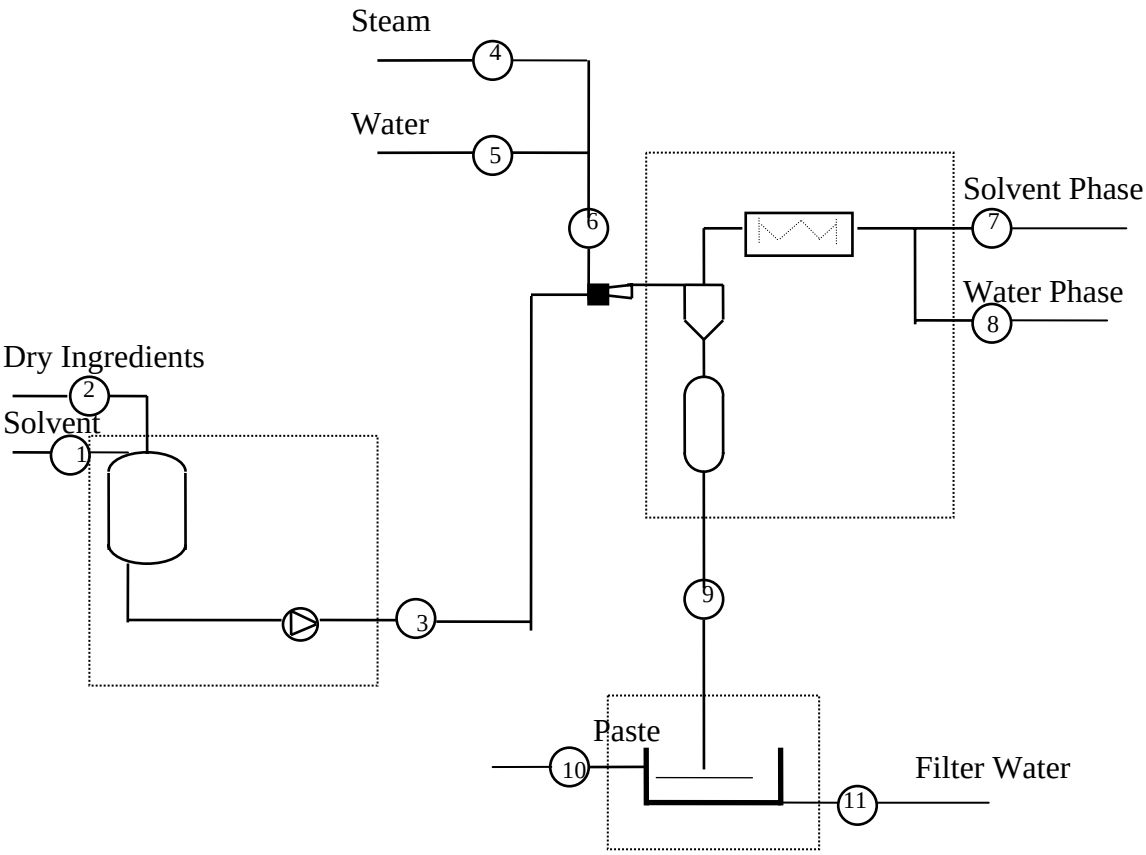


Figure 2: Bofors Precipitation Process for Lova Nitramine Gun Propellant

Material balance predictions were made for the precipitation process using the Aspen process simulation program and the results were compared with the actual figures as shown in Tables 1 and 2.

Table 1: Actual IH 1 Data

		1	2	3	4	5	6	7	8	9	10	11
		Solvent	Solids	Solution	Steam	Water	Wet Steam	Solvent Phase	Water Phase	Slurry	Wet Paste	Filter Water
Temp	°C	- - -	- - -	70	145	15	145	- - -	- - -	- - -	- - -	- - -
Press	bar	- - -	- - -	- - -	4.2	- - -	4.2	- - -	- - -	- - -	- - -	- - -
Vapor Frac	%	- - -	- - -	- - -	100	- - -	27	- - -		- - -	- - -	- - -
Total Flow	kg/hr	540	30	570	430	600	1030	507	274	819	60	759
Water	kg/hr	16	- - -	16	430	600	1030	18	253	775	30	745
EtAC	kg/hr	524	- - -	524	- - -	- - -	- - -	489	21	14	- - -	14
Solids	kg/hr	- - -	30	30	- - -	- - -	- - -	- - -	- - -	30	30	- - -
Water	%	3	0	2.8	100	100	100	3.6	92.4	94.6	50	98.2
EtAC	%	97	0	91.9	0	0	0	96.4	7.6	1.7	0	1.8
Solids	%	0	100	5.3	0	0	0	0	0	3.7	50	0

Table 2: Actual IH 1 Material Balance (Derived from Table 1 Data)

Material	One Batch (10 hour)	1000 kg RDX
RDX	300 kg	1000 kg
CAB, NC, EC	65 kg	217 kg
Ethyl Acetate (EtAc) (w/ 3% water)	5400 kg	18000 kg
Steam (4.2 bar)	4.3 metric tons	14.3 metric tons
Water	6 metric tons	20 metric tons
New/distilled EtAc for make- up	540 kg	1800 kg
Solvent phase (96.4% EtAc)	5070 kg (* 5100 kg)	16900 kg
Water phase (7.6% EtAc)	3740 kg (*2800 kg)	9133 kg
Filter water (1.8% EtAc)	7590 kg (*7.5 kg)	25300 kg

* - Aspen simulation predicted values

The material balances do not add up in all cases because the plant was not ideally equipped to recover the solvents or the paste; and the flow rates were not necessarily kept constant. For example, the cyclone receiver had to be flushed with extra water to remove the paste because the plant set-up and process was not optimized. However, this effort was only a feasibility demonstration. The plant set-up was improved for the CLEVER demonstration.

1.5.1.2 Testing. In the analysis of the chemical composition of the pastes, it was noticed that the nitrocellulose content was generally low. However, the analysis of the powder base(s), used to prepare the paste, showed that the nitrocellulose content was normal. As the

nitrocellulose obviously did not disappear, it was Bofors' conclusion that the determination must be effected by the presence of nitrate esters. For the CLEVER demonstration, no nitrate esters were included in the formulation so the effect is not applicable.

The paste was tested in large-scale card gap tests, however, the results indicated that this test was not suitable for obtaining useful data. The paste morphology limits test repeatability and introduces compaction density issues.

Scanning Electron Microscopic (SEM) analysis was conducted on sectioned, internal surfaces of the gun propellant grains. The micrographs revealed that the ingredients were well homogenized and plasticized, however, voids were also found. This initial effort was conducted to show feasibility and establish baselines for the precipitation process, not to optimize the precipitation or extrusion processes. The objective of this initial effort was met. SEM analysis of the propellant grains showed that there was a need for improvement in the twin-screw process. IHDIV, NSWC has made M43 Lova propellant, which is very similar to the CLEVER propellant (EX 99), via the twin-screw process without void problems.

1.5.2 Sampling and Testing of Previous TSE Efforts.

1.5.2.1 Sampling. Reducing waste is a key environmental and safety advantage of the TSE process. Validation of the projected waste reductions had not been performed by the energetics manufacturing community prior to the CLEVER project.

1.5.2.2 Testing. A demonstration lot of M43 Lova nitramine gun propellant was manufactured via the TSE process in 1996 using a feedstock manufactured by a process different from the precipitation process. The propellant heat of explosion (965 cal/g) and density (1.669 g/cc) met the M43 Lova gun propellant specification requirements. The M43 propellant specification is MIL-P-70818A, see reference 7. The dimensions were all within specification except for the average web (see reference 7 or Appendix B of The CLEVER Technology Demonstration Plan, reference 10). The SEM overall ranking for the twin-screw produced material was 1 to 2 versus an average of 2 to 3 for batch produced M43 propellant. See paragraph 5.1.3.6 of this report for an explanation of this SEM ranking system. This propellant was ultimately gun tested and the results were compared to those obtained with the M43 Lova propellant made by the batch process.

A series of 5"/54 gun firings were made at DD, NSWC in December 1996 to evaluate several IHDIV, NSWC Lova nitramine gun propellants. This testing, funded by Crane Division, NSWC, was the first gun firing of twin-screw processed M43 Lova propellant in the U.S. and was conducted to compare the gun ballistics of TSE manufactured M43 Lova nitramine gun propellant with conventional, batch processed M43 Lova propellant (150 gallon horizontal batch mixer). In both instances, the propellant was extruded to the same size using a 0.454-inch diameter extrusion die. Closed bomb testing prior to the gun firings had shown the TSE M43 propellant burned slightly slower (0.92 times or approximately 8% slower) than the batch made M43 propellant. The burning rate of the batch propellant was 7.461 in/sec at 40,000 psi while that of the TSE propellant was 6.856 in/sec at 40,000 psi. Hence, it was expected that the TSE propellant charge weights required to achieve any given pressure would be correspondingly heavier than the batch propellant. The data obtained from the gun testing is tabulated in Table 3

and graphically presented in Figures 3 and 4.

Table 3: 5"/54 Gun Firings at DD, NSWC, 12/16/96

Process	Propellant Lot	Charge Weight (lb)	Pmax (kpsi)	Velocity (ft/sec)
Batch	0535	20.90	43.2	2703
Batch	0535	21.50	48.9	2856
Batch	0535	22.10	55.3	2959
Batch	0535	22.40	58.3	3033
Batch	0535	22.52	56.7	3031
TSE	0581	22.50	44.9	2778
TSE	0581	23.30	52.2	2952
TSE	0581	23.60	52.8	3008

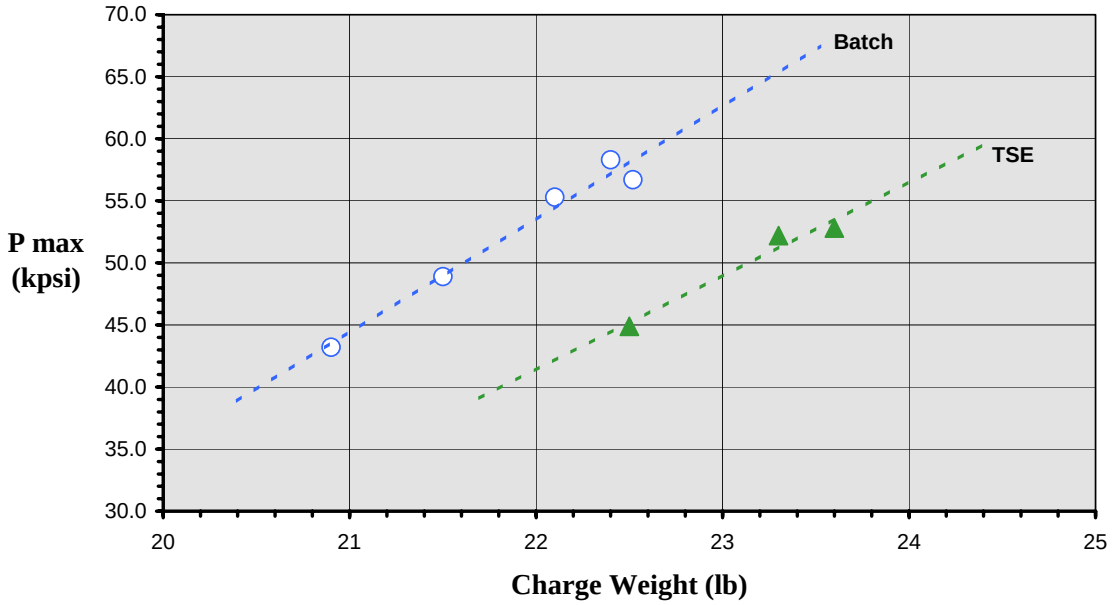


Figure 3: Maximum Pressure vs. Charge Weight for 70 lb Projectiles in a 5"/54 Gun

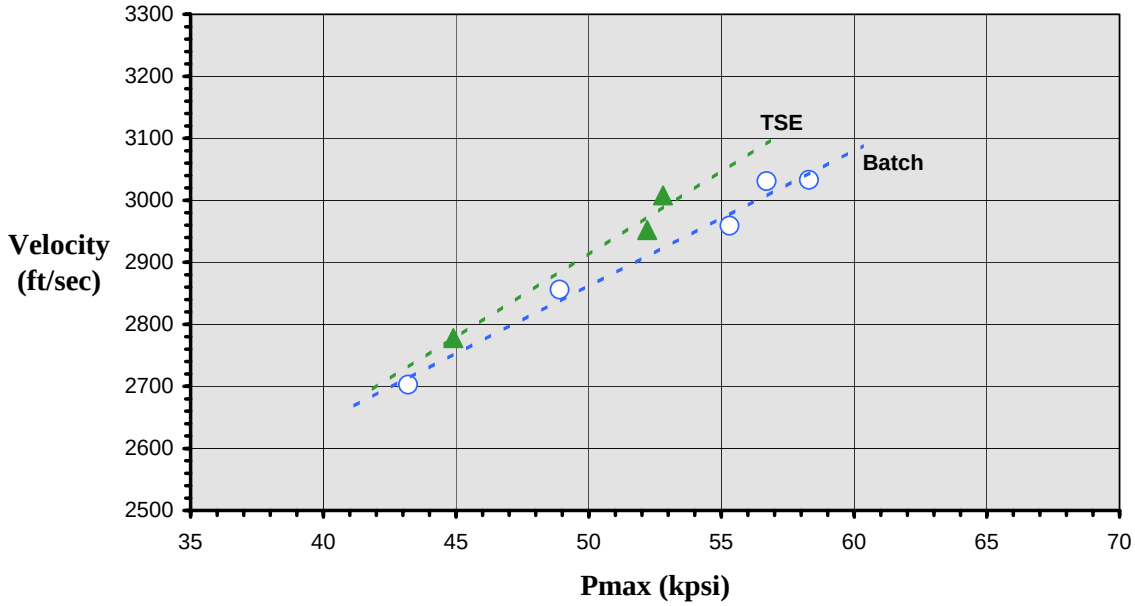


Figure 4: Velocity versus Maximum Pressure for 70 lb Projectiles in a 5"54 Gun

As shown by the data, five firings of the batch processed M43 propellant indicated that the proper charge weight to achieve 55 kpsi (normal operating pressure) was 21.8 pounds, and that at this pressure, the velocity would be 3000 ft/sec. Three firings of the TSE processed M43 propellant indicated that, as expected, a slightly heavier charge weight of 23.7 pounds would achieve 55 kpsi and a velocity of 3000 ft/sec. The ratio of the charge weights required (0.92) is the same value observed from the closed bomb testing data. While the TSE produced M43 propellant burned slightly slower than the batch produced M43, there was no difference in gun ballistics when the charge weights were adjusted to compensate for the slight difference in burning rate. Provided the propelling charge has the additional volume required to allow for more propellant, as is the case for the 5"/54, the burning rate difference is not an issue. Alternatively, the slower burning rate observed with the TSE M43 propellant can be easily compensated for by simply changing the propellant granulation (configuration), i.e. extrude through a smaller size die to give a proportionally smaller web.

1.5.3 Sampling and Testing of the Conventional (Batch) Processing Method.

1.5.3.1 Sampling. An IHDIV, NSWC Technical Report (IHTR 1814, see reference 2) was published in 1995 to summarize the life cycle aspects of conventional processing of 105-mm M900 APFSDS-T Projectile gun propellant (i.e. M43 Lova nitramine gun propellant). The material balance data used to prepare the report was used as a baseline to compare the batch process to CLEVER so that the waste reductions could be quantified. The flow in the conventional process is provided for informational purposes in Figure 5.

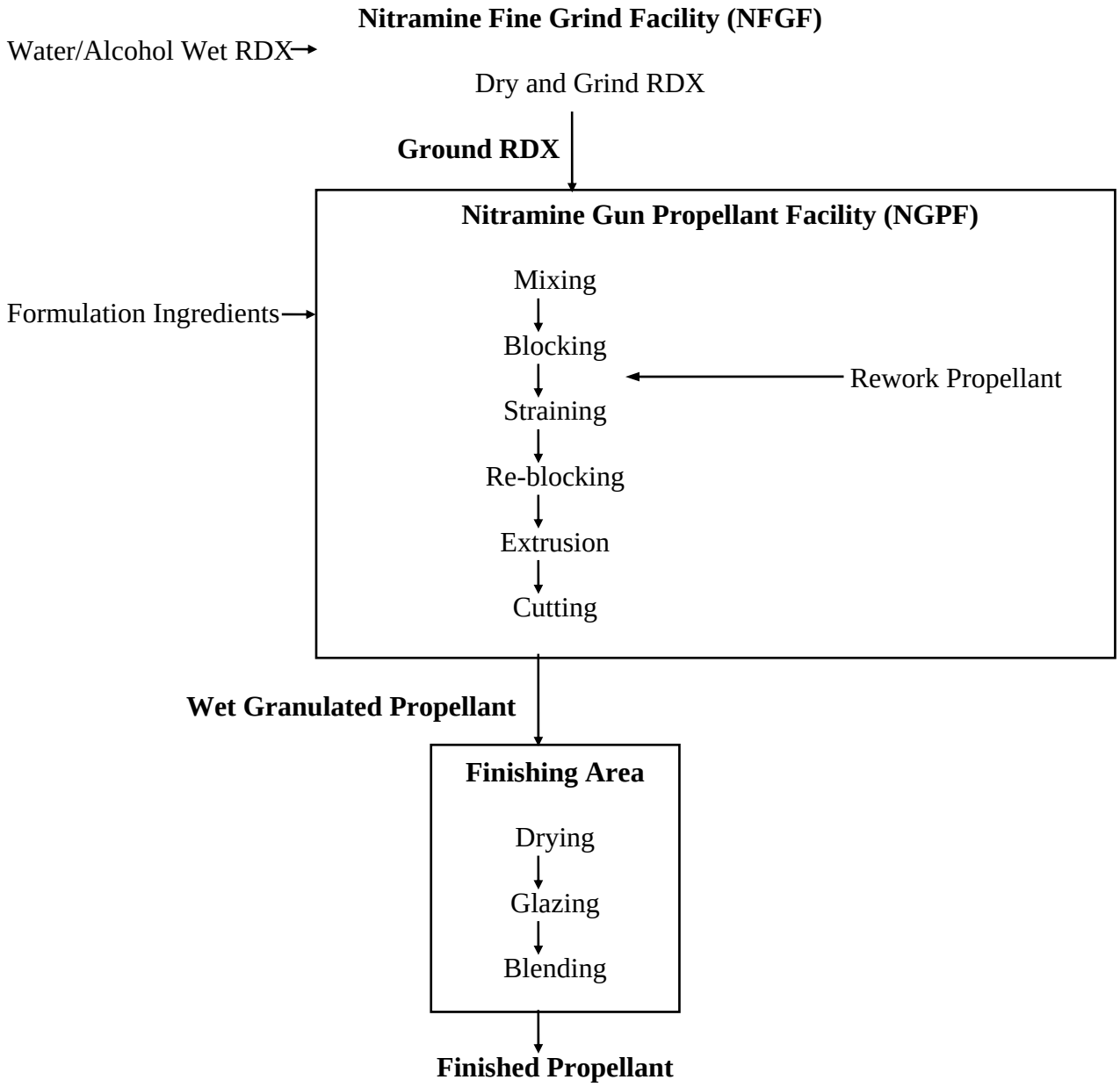


Figure 5: M43 Batch Process Flow Diagram

The actual data used to conduct the life-cycle analysis are provided in Figures 6-11. See Figure 23, Figure 32 and Figure 33 for similar data that were collected during the demonstration of the CLEVER process.

Note: Waste stream levels are presented in pounds (lb) per finished pound (fp) of product based upon a 525 lb dry yield per mix. Alcohol/water waste is segregated into two waste streams. A mixture with 60% alcohol and nitramine contamination is burned. A mixture of 20% alcohol undergoes nitramine extraction and is treated via biofiltration.

Process Information:
1. Batch Size-8000 lb non-volatile

10,400 lb total
2. Total Cycle Time - Drying, approx. 14 hours
Grinding, 500 lb/hr

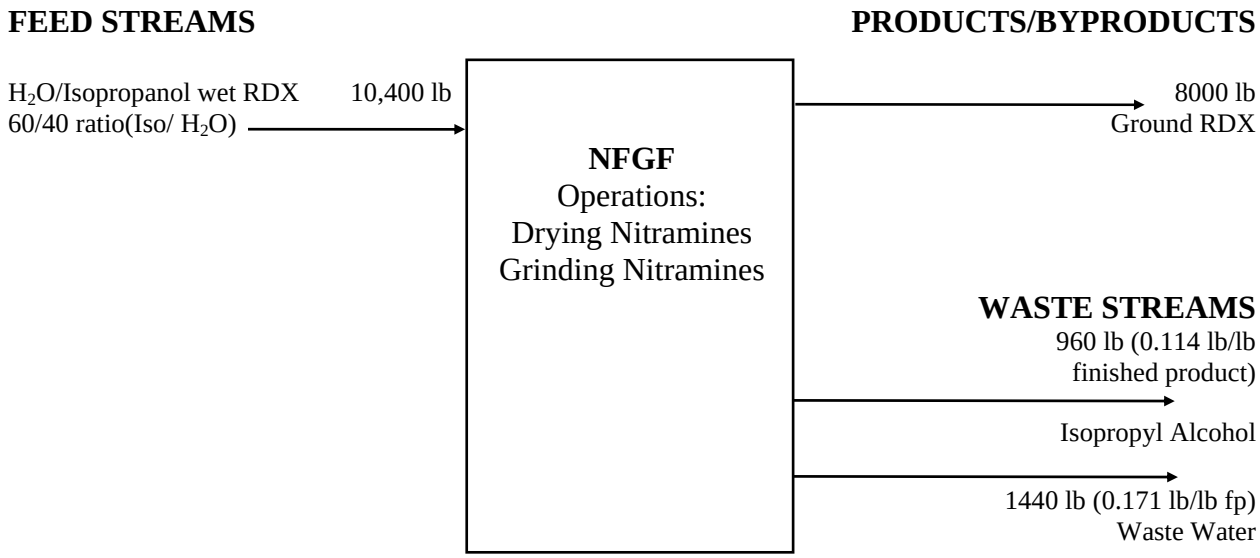
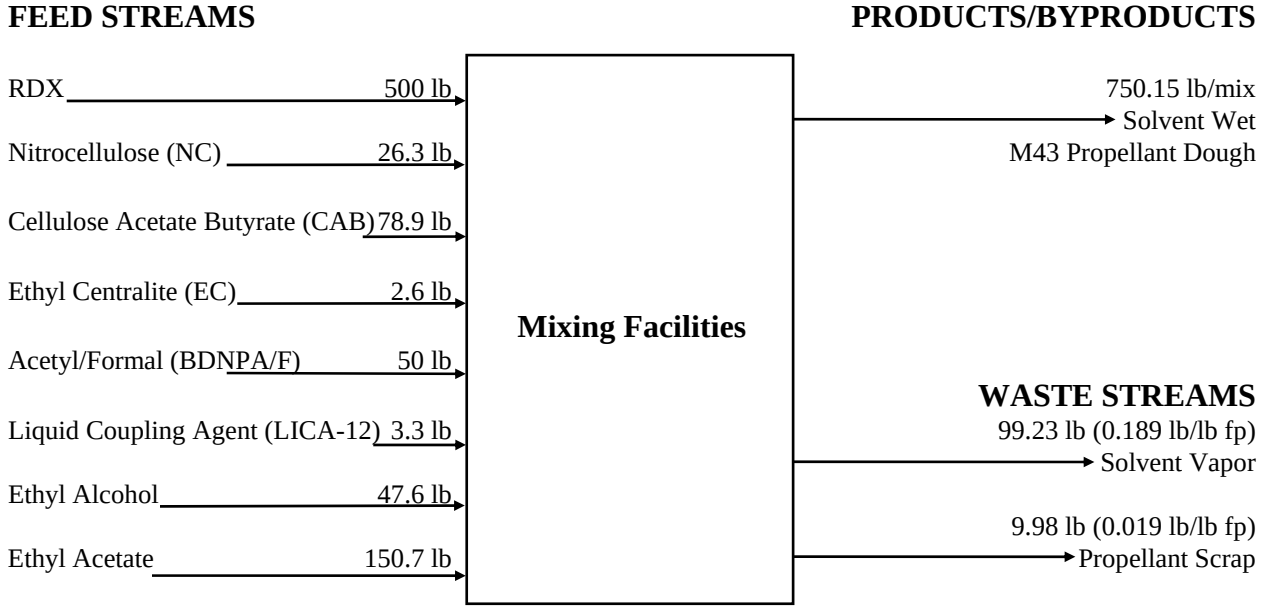


Figure 6: Material Balance for Nitramine Fine Grind Facility (NFGF)

Previous Operations:
1. Nitramine Fine Grinding

Process Information:
1. Batch Size-661 lb nonvolatiles (nv)
2. Total Cycle Time - 3.5 hours
3. Basis - 189 lb/nv/hr



Note: 50% of total solvent is lost at the mixer.

Figure 7: Material Balance for Nitramine Gun Propellant Facility (NGPF) - Virgin Mixing

Previous Operations:
1. Virgin Mix Processing

Process Information:
1. Batch Size-661 lb nv; 819 lb total
2. Total Cycle Time - 2.75 hours
3. Basis - 240 lb/nv/hr

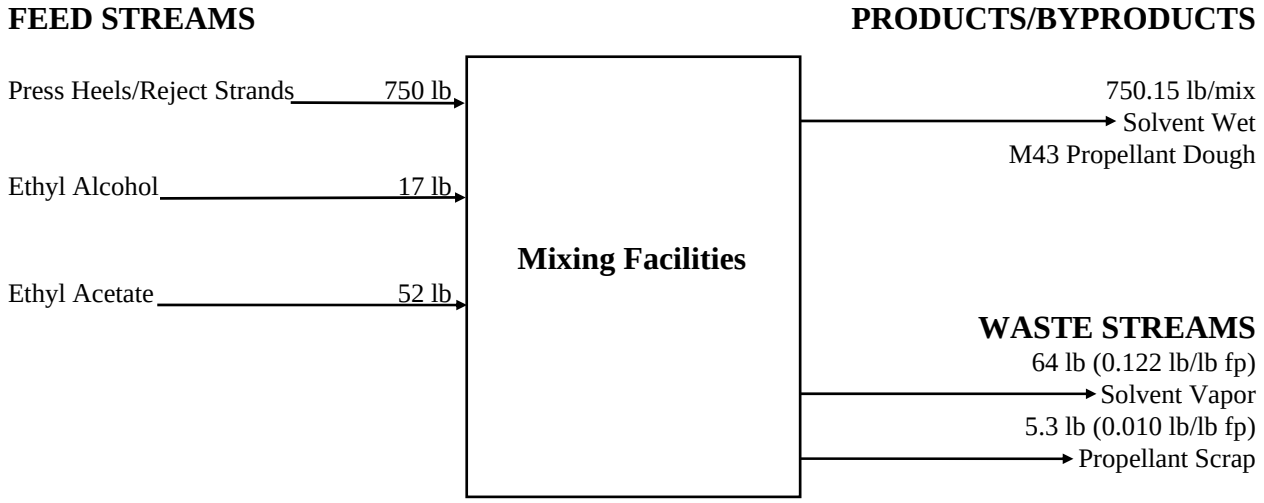


Figure 8: Material Balance for NGPF - Rework Mixing

Note: It is estimated 3% of the solvent is lost at the press line.

Previous Operations:
1. Propellant Mixing

Process Information
1. Batch Size: 1 mix
2. Total Cycle Time - 3.5 hours

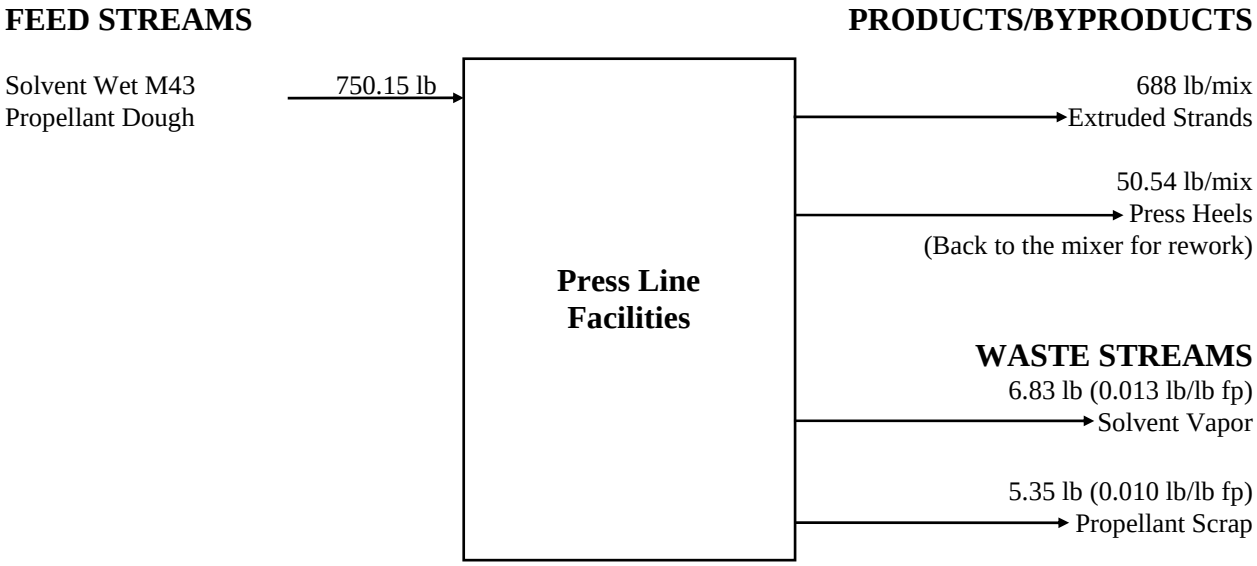
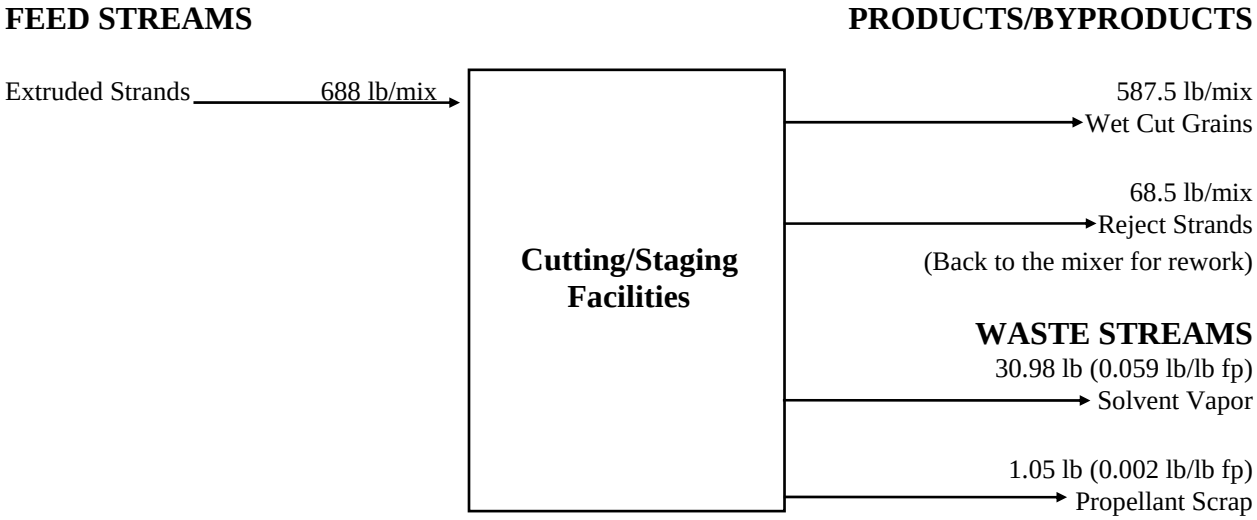


Figure 9: Material Balance for NGPF - Pressline (Blocking/Straining/Extrusion)

Previous Operations:
1. Blocking/Straining/Extrusion

Process Information:
1. Batch Size: 1 mix
2. Total Cycle Time - 3.5 hours



Note: 16% of the processing solvent is lost during cutting.

Figure 10: Material Balance for NGPF - Cutting

Note: 31% of the processing solvent is lost during the drying process.

Previous Operations:
1. Gun Propellant Manufacture

Process Information:
1. Batch Size- 5000 lb

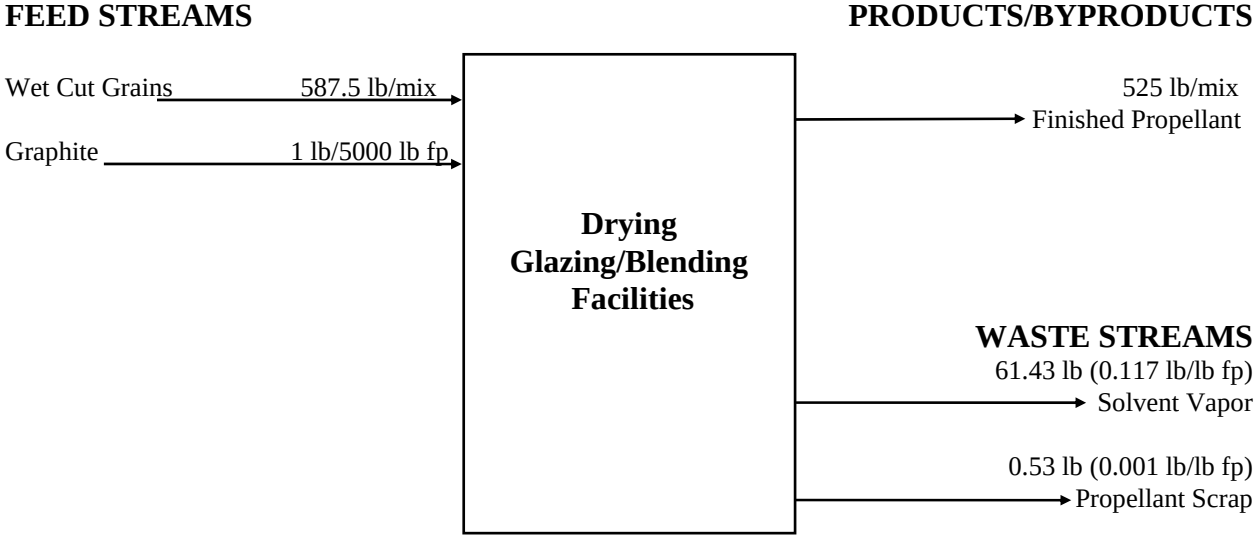


Figure 11: Material Balance for FA - Drying/Glazing/Blending

1.5.3.2 Testing. All testing of the M43 propellant was performed in conformance with the military specification for the M43 propellant for the 105 mm cartridge, MIL-P-70818A.

2. Technology Description

2.1 Background and Applications

2.1.1 Bofors Precipitation Process. The demand for fine particle size nitramines or explosive materials used in the production of propellant and plastic bonded explosives (PBXs) has increased in recent years. In order to be able to meet this demand, new avenues have been explored for producing fine particle size (less than 20 micron), crystalline high explosive materials such as RDX and HMX. The most widely used method for producing fine particle size high explosives is grinding the materials dry in a fluidized energy mill. The ground RDX is then batch mixed with the other components, typically in the presence of solvents, to produce a propellant or explosive composition for further processing. Numerous processing steps must be carried out at high cost and with significant VOC emissions, liquid wastes and solid wastes generated to accomplish the desired result. Bofors originally developed their precipitation process to avoid dry grinding of high explosive materials. Successful demonstration of precipitating fine powders led them to explore other applications such as powder paste feedstock for a TSE process. The Bofors Precipitation Process produces a fine material, which is acceptable for feeding to the TSE. Additionally, this process eliminates the grinding operation required for conventional manufacturing of nitramine gun propellants.

Nobel Chemicals AB, Sweden, a branch of Bofors, applied for a patent in April 1989 for the precipitation process for the manufacture of fine particle explosive substances. The patent, #4,983,235, was issued on January 8, 1991; see reference 3. The U. S. government has been interested in the process since it was first developed, and in 1994 initiated steps to test out various nitramine gun propellant formulations of interest.

Initially, Bofors used their precipitation process to manufacture a Lova nitramine gun propellant formulation very similar to the Army's XM-39 formulation. The difference was that the inert plasticizer used by Bofors was tributyl citrate versus ATEC (acetyl triethyl citrate) for the XM-39 formulation. The solvent used for this process was methylethylketone (MEK). From August 1995 to August 1996 Bofors and IHDIV, NSWC cooperated in a development study of an improved process for nitramine gun propellant manufacture. The task was to investigate the possibility of adapting the Bofors' process to manufacture nitramine propellants that met the specific requirements of IHDIV, NSWC at that time. The formulations of interest then were variations of the XM-39 formulation containing nitrate ester plasticizers (TEGDN and a combination of TEGDN/TMETN) instead of the inert plasticizer, ATEC, used in XM-39. During the development study, Bofors discovered that the nitrate ester plasticizers could not be added during the mixing phase of the precipitation process because the solubility of the nitrate ester plasticizers was too high in water. Too much of the plasticizers dissolved in the filter water and was lost. Therefore, Bofors developed a procedure to add the nitrate ester plasticizers to the product formed from the precipitation process. Additionally, as part of this development study, Bofors investigated the use of ethyl acetate instead of MEK because there are environmental concerns with the use of MEK. Bofors has shown that the substitution of ethyl acetate for MEK has no effects on the quality of the product manufactured. However, the use of ethyl acetate does affect the efficiency of the process. The product yield per unit time is lower.

The CLEVER demonstration utilized the EX 99 formulation that was ultimately selected for the ERGM propelling charge. The EX 99 formulation is a derivative of the XM-39 and the M43 Lova nitramine gun propellant formulations. EX 99 is Navy nomenclature and both XM-39 and M43 are Army nomenclature. Additionally, there are minor formulation differences. An energetic plasticizer (2,2 bis-dinitro propyl acetal/formal (BDNPA/F) or commonly known as acetal/formal) was substituted for the inert plasticizer in the XM-39 Lova formulation to increase the energy and the formulation was renamed M43 Lova. The M43 formulation was modified by removing the liquid coupling agent (LICA-12) because that ingredient provided no benefit to the composition and then it was renamed EX 99. The EX 99 formulation was manufactured by the batch process and the removal of LICA was demonstrated in 1991 to have no effect on the end product.

The precipitation process used for the CLEVER project was very similar to the original process for XM-39 formulation. There was one major difference; ethyl acetate was used in place of MEK. The work for the CLEVER project was divided into three different tasks: Tasks 1 and 2 included basic safety tests and test production at a pilot scale for the entire process (precipitation process as well as TSE). Task 3 consisted of full-scale production of 3300 kg of nitramine paste followed by the manufacture of 1000 kg of finished propellant grains from that paste feedstock.

2.1.1.1 Paste Manufacture Description. This description shows how the paste was

manufactured for this project. The EX 99 paste was made in two steps: powder precipitation and drying. After drying in a filter, these formulations required de-lumping.

2.1.1.1.1 Precipitation. The precipitation process begins with the charging of the raw ingredients. The solid ingredients are weighed up and charged into a bin. The bin is then transported to the plant and the ingredients are sucked, with the help of water, into the dissolver by vacuum transport. Once all of the solid ingredients are charged, the vacuum pump is shut off and the Acetal/Formal is manually charged through the manhole of the dissolver. The ethyl acetate is then pumped into the dissolver. The ethyl acetate slurry in the dissolver is heated to 70°C using indirect steam heating. The dissolver is kept agitated during the entire process. Once the desired temperature has been achieved, the solution is agitated for 20 minutes to ensure all of the ingredients have been dissolved. The precipitation begins after the dissolving is complete. The solution in the dissolver is pumped through a steam driven ejector to a cyclone. The separation of solvent from propellant paste slurry occurs in the cyclone. The slurry is collected in a receiving vessel that is initially filled with cold water. The downcomer from the cyclone ends below the water surface, thus creating a water seal. The precipitation continues until the dissolver is empty. The slurry is discharged in intervals into a filter neutsche where the powder is dewatered as much as possible by vacuum suction. The slurry passes through a strainer prior to the filter neutsche that removes lumps that may have formed during the process. The wet powder in the filter neutsche is transferred manually to large plastic boxes for transport to the drying facility. The cyclone overheads are condensed in a condenser. The condensate is separated into two phases, a lighter solvent phase and a heavier water phase. After the precipitation is finished, the water phase is pumped to a storage tank. The solvent phase is reused in future precipitations.

2.1.1.1.2 Paste Drying. The drying process is accomplished in a heated pressure vessel with a filter screen in the bottom. The dryer is heated indirectly by hot water circulating through the vessel jacket and a stirrer. The propellant paste can be introduced to the dryer by either re-slurrying in heated water, or by vacuum transport. Once the paste is introduced into the dryer, compressed air and a vacuum is applied to the filter, and water is siphoned off the paste and discharged to collection tanks for reuse in future process runs. The pressure in the vessel is released, and the energetic material is heated and agitated for about ten hours to drive off the remaining water, until the moisture content of the material is less than 0.5% by weight.

2.1.2 Twin-Screw Processing. Twin-screw processing has been used in the plastics and food industries for many years. The first twin-screw extruders for use in polymer processing were developed in the late 1930s in Italy. In the 1960s, thrust bearings were developed to increase their reliability. Several European countries have used twin-screw compounding and extrusion since the early 1970s to manufacture energetics; primarily single-base, double base, and triple-base gun propellants. The energetics community in the United States became interested in the application of twin-screw technology to the manufacture of energetic materials in the 1980s. About the same time, the Europeans began investigating the application of twin-screw technology to different types of energetic materials such as PBXs and composite propellants. IHDIV, NSWC began researching this technology in the early to mid-1980s. Within the last fifteen years or so, there have been remarkable improvements in technology areas such as machine design, controls, feeders, and computational models that have matured the development of continuous twin-screw process in it's application to the manufacture of energetic

materials and expanded the scientific understanding of the continuous twin-screw process.

2.1.2.1 Twin-Screw Processing Efforts at IHDIV. Extensive gun propellant experience as well as an excellent scientific knowledge base exists at the Indian Head Division, Naval Surface Warfare Center (see references 4,5 and 6). The history of the twin-screw processing efforts at IHDIV, NSWC follows:

1982 - 1984 *Technology Evaluation:* Research began on the technology. The initial application was the processing of Lova nitramine gun propellant. Visited facilities to determine what equipment current users were utilizing to process energetic materials. Funded universities and private companies to perform inert studies with continuous processing equipment.

1985 - 1987 *Pilot Plant Definition:* Although Lova nitramine gun propellant was the initial product, the facility was designed to be flexible so that all types of energetic materials could be processed in that facility. With this flexibility in mind, a 40 mm Werner & Pfleiderer (W&P) was procured and a facility chosen for the installation of the extruder and associated equipment.

1987 - 1988 *Process Development Facility Construction:* The initial facility construction and equipment installation was completed in 1988 at a cost of approximately one million dollars.

1988 - 1989 *Inert Studies (Lova):* The objective of the inert work with Lova was twofold. The first reason was to start up the facility and to learn the operation of the equipment with an inert material. The second reason was to develop operating parameters for the live processing of Lova. A total of 32 processing trials were performed to manufacture over 900 pounds of inert propellant. An extensive documentation package was prepared to obtain approval for live operations.

1990 - 1993 *Live Operations (Lova):* A similar facility at White Oak was built about the same time. The facility at White Oak was the first facility in the U.S. to process live material. Live processing at the Indian Head facility followed approximately six months later in May 1990. Both facilities were started up with live Lova nitramine gun propellant. The approach of the live processing work was purely an engineering approach with the goals of learning the operation of all of the equipment involved and learning how to process live material safely on a TSE. Lova was the vehicle chosen to accomplish this task. Since then, 40 processing trials at Indian Head have yielded over 500 pounds of live Lova. These trials used a Lova preblend manufactured by the old method (vertical mixer). Additionally, a rheological study of a high energy Lova propellant as processed on the twin-screw extruder was conducted with the use of an on-line, adjustable gap rheometer. This work was performed in collaboration with The Highly Filled Materials Institute at Steven's Institute of Technology and White Oak prior to the merger of White Oak and Indian Head Division.

1993 - 1994 *Facility Upgrade:* The initial facility design had a limited feed capacity because there were only two loss-in-weight solid feeders and there were no solid refill hoppers. Additionally, the facility lacked humidity control and other controls were minimal. The facility upgrade removed these limitations. The facility was upgraded specifically to provide the capabilities required to execute the Continuous Processing of Composite Propellants project. These capabilities included: additional loss-in-weight solid feeders, automatic solid refill, humidity control, improved extruder barrel temperature control and data acquisition/process control. This facility upgrade incorporated many of the lessons learned from the Lova processing work and from the community to provide a very flexible, modular and capable research and development facility.

1994 - 1997 *Continuous Processing of Composite Propellant:* IHDIV, NSWC entered into a cooperative agreement with the French to jointly develop continuous processing for the manufacture of composite propellants. This project combined the research and development resources of both countries to develop this technology much quicker than either country could achieve independently. IHDIV's secondary objective was to develop the science of TSE processing. During this time, a 2.75" extruded composite rocket motor grain was successfully extruded using the first iteration of a die designed with computational fluid dynamic modeling.

1995 - 1996 *Lova Demonstration Lot:* The objective was to test a demonstration lot of M43 nitramine gun propellant manufactured in the TSE and compare the test results to that obtained from the batch processed propellant. The TSE feed material was prepared in a manner similar to the Bofors Precipitation Process, however, ground RDX was the starting material. The propellant grains were successfully extruded and test fired.

1996 - 1997 *Inert TPE Processing:* Thermoplastic elastomers (TPEs) are ideal binders for "green propellants" because they do not require cross-linking and, therefore, can be more easily recycled. TPEs are very difficult if not impossible to process in larger sized batch mixers. However, TPEs can be processed readily in twin-screw extruders. The objective of this project was to develop the process for manufacturing a TPE based gun propellant. This work was funded under the Clean Agile Manufacturing of Energetics Project by SERDP.

Currently, there is another effort funded by the ESTCP program office related to the CLEVER effort. This project (ESTCP #PP-199804) is titled "Nitrocellulose Based Propellant Manufacturing Waste Minimization". This project is utilizing novel processing techniques to significantly reduce the amount of waste and emissions generated in the conventional manufacturing of MK 90 propellant grains for the 2.75-inch rocket motor. The team includes the current propellant grain manufacturer, Alliant Techsystems, Inc. at The Radford Army Ammunition Plant and the Army Research, Development and Engineering Center (ARDEC). The new process for the MK 90 grains uses a shear roll mill to manufacture solventless double

base propellant pellets that provides a feedstock to a twin-screw mixer/extruder. Indian Head Division is performing the twin-screw extrusion process development and demonstration portion of the project in close collaboration with Alliant Techsystems, Inc. who is performing the shear roll mill process development and demonstration. ARDEC is developing a process model for the shear roll mill and conducted an independent environmental and cost audit. The final manufacturing process will be cleaner, safer, more efficient, and have fewer operational steps.

Commonality exists between the two projects in the approach (the new advanced manufacturing process will be evaluated for three attributes relative to the conventional batch process as was the CLEVER process; i.e. acceptable product performance, minimum environmental impact, and low manufacturing cost) and in the demonstration of the twin-screw process. Twin-screw extrusion is again one of the two core technologies being demonstrated. However, although a twin-screw extruder is used for both processes, the equipment configuration and how it is operated very different and specific for each product.

2.1.2.2 Grain Manufacturing Process. The description below identifies how the final propellant grain is manufactured from the feedstock produced by the Bofors Precipitation Process. The TSE process includes material feeding, twin-screw compounding/extrusion and propellant cutting. These operations are done within one process, therefore, the manufacturing cost is lower when compared to the batch process. The entire TSE process is remotely controlled from a control room located approximately 300 feet from the processing building.

2.1.2.2.1 Material Feeding. The feed material characteristics are critical because the flow of material needs to be constant so the extruder will experience no interruptions. For some formulations, several loss-in-weight solid feeders are required to deliver the dry solid ingredients and several pump systems are required to meter the liquid ingredients to the extruder. However, for this demonstration, only one solid feed stream and one liquid feed stream were required. The Bofors Precipitation Process provides a feedstock that can easily be fed by the K-Tron twin-screw, vertically agitated, loss-in-weight solid feeder (Figure 12) with very little waste produced.

Figure 12: K-Tron Model T-37 Solid Feeder

The IHDIV, NSWC Twin-Screw Extruder Facility is a process development facility only and, therefore, the facility has a limited solid refill capability. The total amount of material that can be fed to the extruder determines the length of each processing run. The IHDIV, NSWC TSE Facility has one refill hopper for each of the four loss-in-weight solid feeders. Each refill hopper consists of four refill cylinders. Three of the four refill hoppers are shown in Figure 13.

Figure 13: Solid Refill Hoppers

These refill hoppers allow for longer TSE runs so that a more continuous manufacturing process can be demonstrated. When refill of the solid feeder is required, the contents of one refill cylinder is emptied into the feeder in a very short time period so the operation of the solid feeder is not disturbed. This procedure is repeated for the other three refill cylinders as required. Two of the four solid refill hoppers can be positioned over the K-Tron T-37 feeder for refilling.

The liquid feed stream consists of a 70/30 ethyl acetate/ethyl alcohol mixture. The ethyl acetate is the active solvent with the ethyl alcohol acting as a volume extender. This mixture is simply metered to the appropriate barrel section using a positive displacement pump. A closed loop control system based on mass flow measured by a flow meter is used to maintain an accurate delivery rate.

2.1.2.2.2 Twin-Screw Extrusion. The TSE Facility is centered around a Werner & Pfleiderer (W&P) ZSK-40 co-rotating twin screw extruder with cantilevered screw shafts. The extruder is powered by a 20 hp variable speed, explosion proof motor. A safety slip clutch is provided that disengages the motor from the extruder screws in the event of an over torque situation. This machine utilizes two 40-mm diameter screws (Figure 14) centered in the extruder barrels.

Figure 14: W&P 40-mm Extruder Segmented Screws

The screws are co-rotating, fully intermeshing and self-wiping. The screw profiles are designed so that the tip of one screw wipes the flank and root of the other screw resulting in a self-cleaning action. This type of twin-screw mechanism provides very good conveying, pressure build-up, and self-cleaning capabilities. Segmented screw sections are used to maintain flexibility in screw configurations. The screws can be made up of various different screw elements that slide onto a splined shaft. The various types of screw elements that are used are right-handed conveying elements, left-handed conveying elements, right-handed kneading blocks, left-handed kneading blocks and neutral kneading blocks. All of these elements can vary in length and pitch. Each type of screw element provides distinct conveying, shear, and pressure build-up action. Therefore, the various types of screw elements can be arranged on the screw shafts as needed to provide the required conveying or mixing actions at desired locations for making different energetic formulations. The screw configuration used for the manufacture of the M43 Lova Demonstration Lot was the starting point for the CLEVER demonstration. The final screw configuration used was selected based on the results of a designed experiment that was conducted during the course of the CLEVER project.

The TSE Facility was designed to have the capability of easily changing from one energetic formulation to another. Therefore, an extruder with a modular barrel design is used (Figure 15).

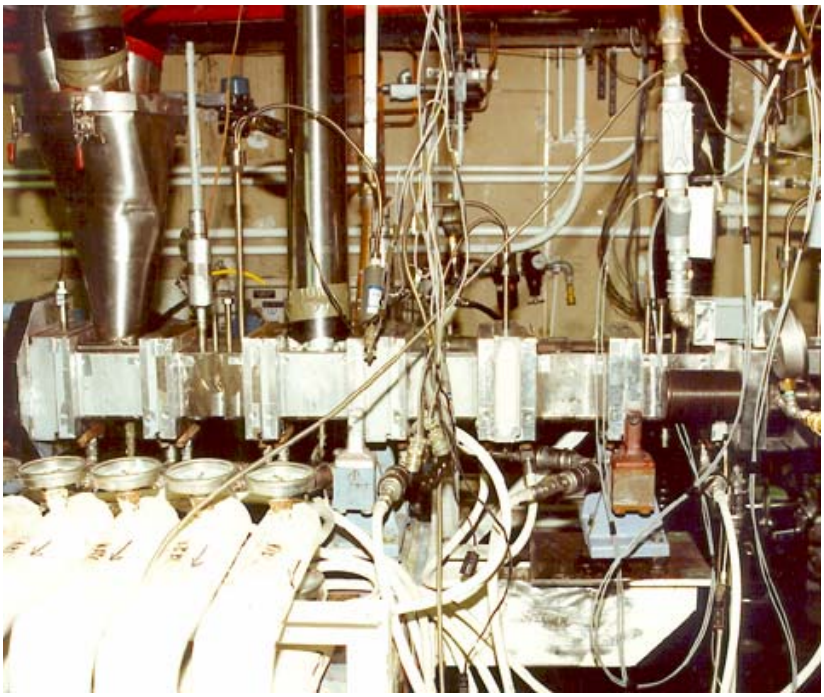
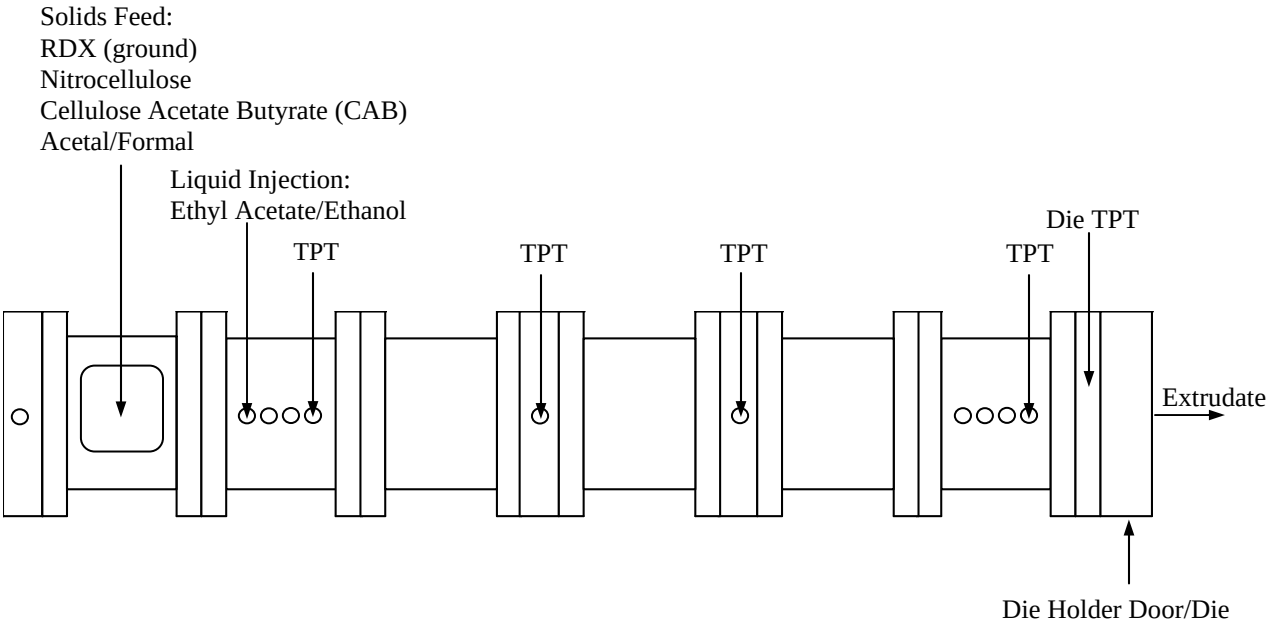


Figure 15: W&P 40-mm Extruder Barrel Sections (Side View)

This extruder has six segmented barrel sections and three liquid injection plates that can be arranged to serve different ingredient addition locations and operating requirements. Each individual barrel section is 165 mm in length and the total processing section of the extruder has a barrel length to screw diameter ratio (L/D) of 27. The extruder is equipped with five separate temperature control zones for maintaining process temperature. The extrusion pressure and temperature is measured by a pressure transducer/thermocouple located at the entrance region to the die. Pressures and temperatures along the length of the extruder are also measured at pertinent locations using combined Temperature/Pressure Transducers (TPTs).

The ingredients are mixed, consolidated, and pressurized for extrusion in the processing section of the extruder. The product is extruded through dies mounted in a die holder at the end of the extruder. The number of dies is dependent on the product dimensions and extruder size. A hydraulic clamping mechanism retains the die holder during operation. In the event of an over pressure situation, pressure is relieved by mechanical failure of a shear mechanism or relief of hydraulic clamping pressure.

A sketch of the barrel configuration used to manufacture the CLEVER demonstration lot of propellant is shown in Figure 16. The solid feed stream was fed into the first barrel section and the solvent mixture was fed immediately after the solid material. The temperature profile used is also shown. The pressure and temperature were measured at four places along the extruder barrels as well as at the extrusion die.



Note: The "Solids Feed" is the product of the precipitation process.

Figure 16: Sketch of Extruder Barrel Configuration for Lova TSE Processing

2.1.2.2.3 Propellant Cutting. Continuous twin-screw extrusion requires a unique approach to propellant cutting because it is a completely remote operation. The solvent wet extruded strand cannot be periodically removed from the extruder bay and manually cut into propellant grains as is done in the conventional batch process. The extruded strand must be cut

into real time and on-line with the extruder. Since it is almost impossible to match the cutter speed to the slightly varying strand extrusion speed, especially when you are cutting more than one strand, the extruder and cutter must be decoupled. There are two approaches to the decoupling problem. The first is to develop a slack loop between the extruder and cutter; and then turn the cutter on and off to maintain the loop while cutting the strand into propellant grains. The second approach is to cut the extruded strand into sticks that are then periodically fed into a continuously running cutter to cut the propellant grains. The second option was selected for the CLEVER project and demonstration. This option was chosen in order to minimize the amount of design work required for a remote strand handling system and, therefore, design cost. Equipment cost was also minimized because an existing cutter was used. The sequence of operation is as follows. An air-actuated guillotine cutter is mounted on top of the die holder and is used to separate the product from the start up material once steady state is achieved. Once the product reaches the desired consistency, a cut is made and strand-conveying tubes are lowered to catch the propellant strands and convey them to the pelletizer, or cutter, for final cutting. The alignment of the strand-conveying tubes in the down position with the die face is checked prior to the beginning of the extruder run. Water is used to help transport the strand as well as to cool the propellant strand and to extract some of the solvent so the propellant strand is stiff enough for final cutting. A second air-actuated guillotine cutter is used to cut the continuous propellant strand into approximately three foot sections called propellant sticks. The propellant sticks are then automatically fed to a pelletizer. The pelletizer is used to cut the propellant sticks to the appropriate grain length, approximately 0.6 inch. The entire extrusion and cutting operation is done remotely from a control room located approximately 300 feet from the processing building.

2.1.2.2.4 TSE Process Control. As discussed above, TSE process is controlled and monitored remotely from a control room, shown in Figure 17.

Figure 17: Twin-Screw Extruder Facility Control Room

An Allen Bradley programmable logic controller is used to continuously control and monitor the process conditions as well as to control the various alarms and interlocks of the system. The alarms and interlocks include features such as shutdown of the extruder in the event of an over torque or over pressure situation. A PC-based data acquisition system is used to log the process data at a rate of once per second. The system features trend screens, status screens, history recall, instrumentation displays, and subsystem overview screens. This system is ideal for monitoring feeder stability, process instrumentation, and extruder performance.

2.1.3 Gun Firing Operations. All firings were accomplished at the Dahlgren Division,

Naval Surface Warfare Center (DD, NSWC) Main Range. The Main Range (see Figure 18) forms a continuous range of 1200 feet of land and 25 miles of water (Potomac River).

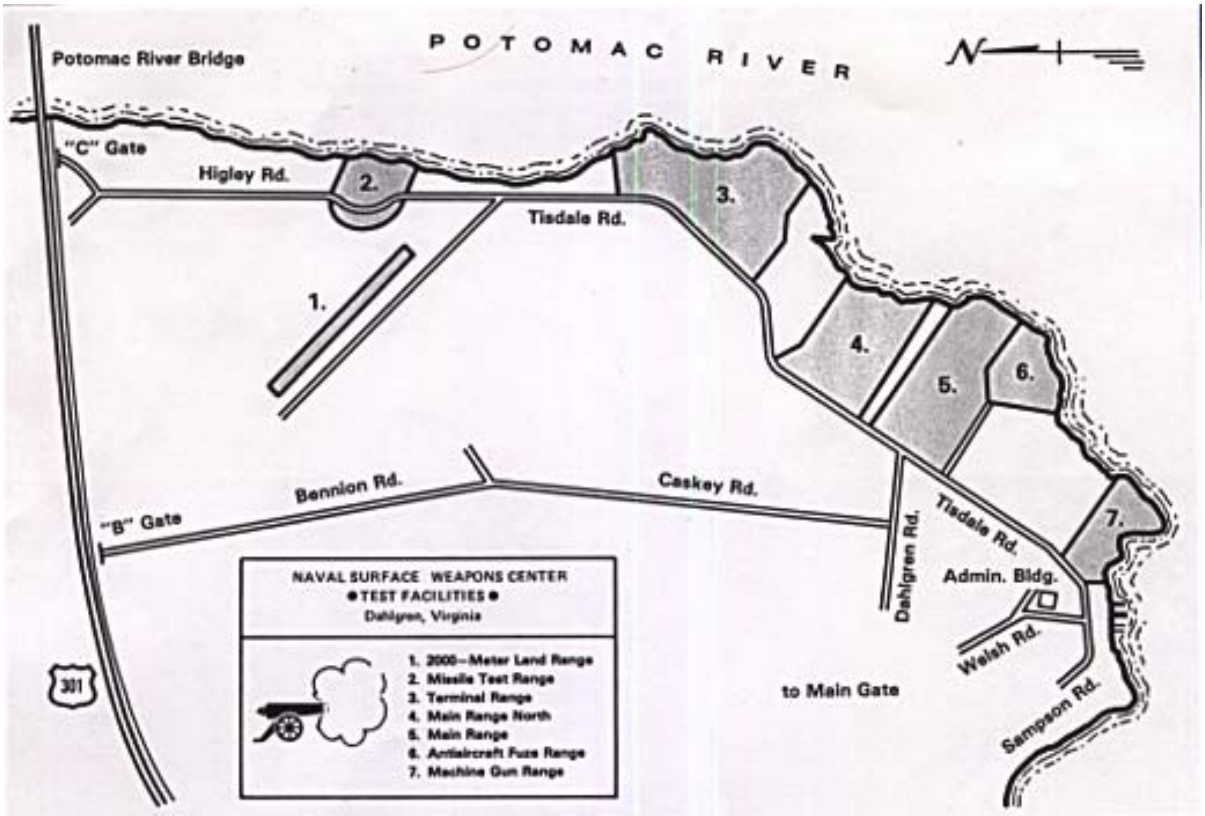


Figure 18: Gun line at Main Range at DD, NSWC

This complex is equipped with gun emplacements for firing all naval guns for:

(1) Acceptance testing of guns and ammunition components (propellants, cases, primers, projectiles, barrels, and oscillating assemblies); and

(2) Proof of gun propellants to determine the type and weight to be used for any gun and projectile combination.

Uniformity of propellant performance is determined by the measurement of projectile ejection time, projectile initial velocity, projectile water-impact range, and gun chamber pressure-time history. Support capabilities include: fabrication shops, temperature conditioning of explosive items, projectile and propellant charge assembly, gun component maintenance, and electronic and optical instrumentation.

Currently, qualification of the Extended Range Guided Munitions (ERGM) round with EX 99 propellant is being pursued by the Navy at DD, NSWC. The current 5-inch gun uses NACO, a single-base propellant that is less energetic than the EX 99 formulation. The ERGM round is designed to provide more energy and it uses a heavier projectile. DD, NSWC has the only modified 5"/54 gun which is capable of testing the ERGM round. The modified 5"/54 gun and

dummy rounds that simulate the weight characteristics of the ERGM round were used to test the CLEVER demonstration propellant. Since the CLEVER project used the same formulation as the ERGM propelling charge, as well as the same test configuration, the ERGM data was used as the batch baseline for comparison purposes.

2.2 Strengths, Advantages and Weaknesses

The CLEVER process could be used to produce all of the propellant for the new generation 5"/54 gun ammunition. The use of the CLEVER process would result in a 47% reduction of VOC emissions with a 41% cost savings. These reductions could be achieved at the two current active gun propellant manufacturing facilities - IHDIV and the Radford Army Ammunition Plant (RAAP). In addition to the gun propellant production base, the closed loop Bofors Precipitation Process has potential in explosive production processes. Although this program was not geared toward demonstrating the viability of explosive processing, it has provided an important "next step" in bringing the technology to the U.S. so that its full pollution prevention and efficiency benefits can be examined. CLEVER's current limitations are the lack of a large scale continuous processing facility dedicated to energetics and a Bofors precipitation plant in the U.S. Both limitations are being addressed by construction of dedicated facilities at Indian Head Division, Naval Surface Warfare Center.

2.3 Factors Influencing Cost and Performance

The factors that affect the performance of the Bofors Precipitation Process are either Major (M) or Critical (C). All of these factors are monitored during the process. A list of the parameters is presented in Table 4.

Table 4: Precipitation Process Performance Factors

Parameters	Criticality
Moisture content of RDX and NC	C
Ingredient weigh up	C
Dissolving temperature	C
Total amount of solvent	M (minimum amount)
Steam pressure	M
Flow, as observed, in sight glass	C
Flow of solution to cyclone	M (within a span)
Phase separation of condensate	C
Recovered condensate level	M
Level in receiving vessel	M (within a span)
Condensate temperature	M (maximum temperature)

These factors also can potentially impact cost since they control both batch size and batch cycle time. Larger batch sizes and shorter cycle times decrease per pound costs since labor costs decrease. Additionally, one could increase the maximum yearly plant capacity without increasing capital investment costs.

Manufacture of the final propellant by the TSE process has many variables as well. These include: paste delivery rate (lb/hr), solvent delivery rate (lb/hr), extruder screw configuration,

product throughput (lb/hr), screw speed (rpm), process temperature (can have multiple zones), extrusion die temperature, extrusion pressure (process measurement), and torque (process measurement).

The paste and solvent delivery rates are key areas that are simplified through the precipitation process. The paste is a free flowing and easily fed in a loss-in-weight solid feeder. There is only one solvent feed, which greatly simplifies the process design. Many calibration tests are conducted prior to a processing trial to verify feeder/pump flow rates and operational readiness. The screw configuration, product throughput, screw speed and process temperature are operating parameters that have been evaluated and established during past Lova process development efforts. These baseline parameters will be adjusted as required to suit the CLEVER process. Many similarities exist which enhances the success of CLEVER.

Several process measurements (the extrusion die temperature, extrusion pressure and torque) are used as an indication of the quality of the product. The approach is to reach a steady state operation as quickly as possible to minimize start up waste. The process measurements can be used to help minimize waste while still producing a quality product.

Minimizing waste and increasing the product throughput of the extruder are potential areas for process improvements to lower both material and labor costs per pound. As with the Bofors process, increasing the extruder throughput allows increasing the maximum yearly plant capacity without increasing capital investment costs.

2.3.1 Bofors Precipitation Process Scale-Up Issues. There are limited factors that affect the scale up of the manufacture of propellant paste by the precipitation process. The key to the precipitation process occurs at the nozzle of the precipitator. The only change would be the size of the supply tanks and number of precipitators. Material handling would be a greater issue; however, it should be handled in exactly the same manner so only flow rates would be affected and monitored.

2.3.2 TSE Process Scale-Up Issues. In order to scale a TSE process, it is necessary to create the same mass transfer conditions in both the model and target extruders. Practically, the key issues are matching shear rate and mechanical power input to the product. The following items should be followed when scaling between a lab machine and the larger scale unit:

- *Process length over Diameter (L/D)* - The L/D ratio of the barrel arrangement and the screw design of each process operation as well as the overall L/D should be matched as closely as possible between the different extruders.
- S_{mech} - This is a measure of the power input per unit mass of material. For a fixed screw design, this value varies with the screw speed and throughput.
- *Available Power* - The motor, gearbox, and shafts of the large-scale machine must be able to deliver the power and torque to the process section needed at the rates predicted by the scale-up calculations.
- *Degree of Fill* - The large-scale machine should generate the same average degree of fill as that of the small scale.
- *Shear rate* - This parameter is critical to mixing intensity. The average shear rate is proportional to the extruder screw speed. Each machine has its own shear rate constant.

The total shear input is, of course, dependent on the screw design.

3. Site/Facility Description

3.1 Facility Background

3.1.1 Bofors, Sweden Facility. At the beginning of the CLEVER project, Bofors AB consisted of the parent company with four divisions, several Swedish subsidiary companies, as well as sales companies and representatives in some 40 countries throughout the world. Since that time, Bofors AB has gone through a series of restructurings. The Bofors business concept to develop, manufacture, market, and maintain advanced material for the Swedish Defense Forces and the international market as well as to apply it's technologies to other projects within its sphere of competence remains unchanged. Bofors Explosives AB, now known as Nexplo is the specific company that performed part of the work for this demonstration. However, that specific company was called Bofors Explosives AB at the time that the demonstration was conducted so they will be referred to Bofors Explosives AB or Bofors for the purposes of this demonstration. They are located in Karlskoga, Sweden. A wide range of propellants and explosives are made there. Many products are made for both military and commercial use. Commercial products cover about 50% of sales. The company has made great advances in the development of insensitive munitions, such as Lova propellant and PBX. Lova is also used in vehicle safety applications, for example in airbags. The number of employees is approximately 100.

3.1.2 IHDIV, NSWC Facility. IHDIV, NSWC is the oldest, continuously operating Naval ordnance facility in the United States. IHDIV, NSWC was established in 1890 as the Naval Proving Ground. In 1932, it became the Naval Powder Factory; in 1958, the Naval Propellant Plant; in 1966, the Naval Ordnance Station; and in 1992, the Indian Head Division, Naval Surface Warfare Center. IHDIV, NSWC is located on a peninsula bordered by the Mattawoman Creek and Potomac River in Charles County, Maryland (Figure 19).

Figure 19: IHDIV, NSWC

The activity consists of 1600 buildings, 3500 acres, and approximately 2000 employees. The total plant asset value is $1.5 billion with over $50 million invested in the last five years in environmental efforts.

IHDIV, NSWC carries out a full spectrum of functions for energetics research, development, manufacturing, and in-service engineering. This activity possesses the unique capability to transition all energetics from laboratory to production and then to fielded product. Energetics is a term that applies to explosives, propellants, pyrotechnics, and specialty chemicals, including their immediately related component applications such as rocket and missile propulsion units, warheads, mines, gun projectiles and propelling charges, and cartridge-actuated and propellant actuated devices. The scope of capabilities at Indian Head Division, NSWC allows for efficient use of specialized expertise and expensive facilities required for research and development, scale-up, manufacture, and testing of energetics.

3.1.3 Dahlgren Division, NSWC. Since 1918, the Navy-maintained shore station to test ordnance materials has been located at Dahlgren, Virginia. DD, NSWC's mission is to conduct a comprehensive program of warfare analysis, research, development, test, evaluation, system integration, and fleet engineering support for surface warfare and related fields of technology. The facility consists of 4400 acres of land that includes a 2000-meter land range that has recently been added to accommodate Army and Marine Corps testing requirements and an instrumented water range 25 miles long and 5 miles wide. An extensive magazine complex adapted for segregated stowage and temperature conditioning of initiators, explosives, propellants, and pyrotechnics and complete facilities for explosives handling, assembling, disassembling, and fuzing are in close proximity to all firing ranges. A weight-handling system consisting of gantries, cranes, and rail networks is also available.

3.2 Site Waste Characteristics

3.2.1 Bofors Site. The yearly capacity of the Bofors plant is 225,000 kg of paste (consisting of 76% RDX by weight) based on a three-shift operation, 5 days per week, 40 weeks per year. In the precipitation process, 300 kg propellant paste is produced in each batch. Based on a three-shift operation, two batches are produced every 24 hours.

A summary of the wastes generated during the manufacture of one batch of material by the precipitation process and then normalized to a 1000 kg of RDX, is presented in Table 5. The costs are actual costs at Bofors, using ethyl acetate as solvent, calculated with a SEK/US dollar ratio of 8.00.

Table 5: Summary of Wastes from the Precipitation Process

Type of Waste	Waste Treatment	Amount (kg)	Cost ($US)
Waste water	Biological treatment	25,300	370
Condensate, water phase	Solvent (EtAc) recovery by distillation	9,133	870
Solid sludge from traps	Incineration	10	50

3.2.2 IHDIV, NSWC Site. All waste streams generated during the manufacture of energetics are handled in conformance with State and Federal regulations. Standard operating procedures (SOPs) are written, enforced, and monitored for all types of formulations and processes. These procedures detail the proper method of handling all waste generated during the process. The SOPs are reviewed by a special environmental group that is monitored by local authorities. Any waste energetic material is currently burned on site at a cost of $0.50/lb.

The TSE facility used for the CLEVER project is equipped with a ventilation and exhaust system that uses HEPA and carbon filters to capture all VOCs emitted from the process. Therefore, no VOCs are emitted to the atmosphere. Pure liquid solvent waste for the TSE process is negligible because all of it is incorporated in the propellant to aid in processing. However, water is used to cool the product as it exits the extruder to make the product stiff enough to cut to length. This water extracts a small amount of the processing solvent from the product during this cooling process. For the CLEVER project, we had initially planned to circulate the water through a distillation column to strip the solvents. This plan was abandoned when we found that the water volume of the recirculating system used was approximately 200 gallons. Given the short contact time (less than 5 minutes) and the water to propellant ratio of 3 to 1, we could not even approach the azeotropic concentration of solvent in the recirculated water used for strand conveying and cutting. The solvent contaminated water was drummed and disposed of as non-explosive hazardous waste at the end of a run.

3.3 Site Maps & Photographs

3.3.1 Bofors, Sweden Site. The Swedish Forces would not release a detailed map of the factory area. However, the precipitation process is located in the area marked with the number 22, Bjorkborn Industrial Area, in Figure 20 below.

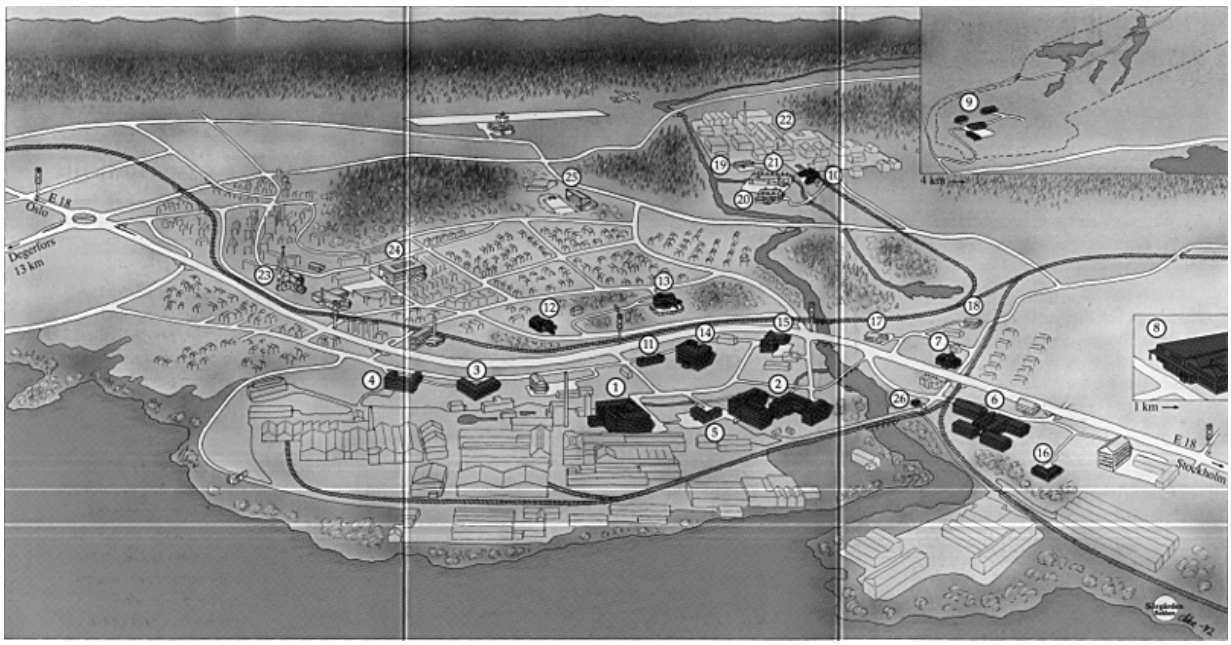


Figure 20: A Sketch of Bofors in Karlskoga, Sweden

3.3.2 IHDIV, NSWC. The TSE facility has two levels where all of the various components discussed in section 2.1.2.2 are located. An overall pictorial view of this facility is shown in Figure 21 while the building layout is presented in Figure 22. The TSE (twin screw extruder) is located on the first floor. The loss-in-weight solid feeders are located on the second floor mezzanine. The solid feeder refill hoppers are mounted above the feeders on hoists. The liquid feed systems are located on the main floor to the right of the extruder.

Refill Hoppers **Solid Feeders**

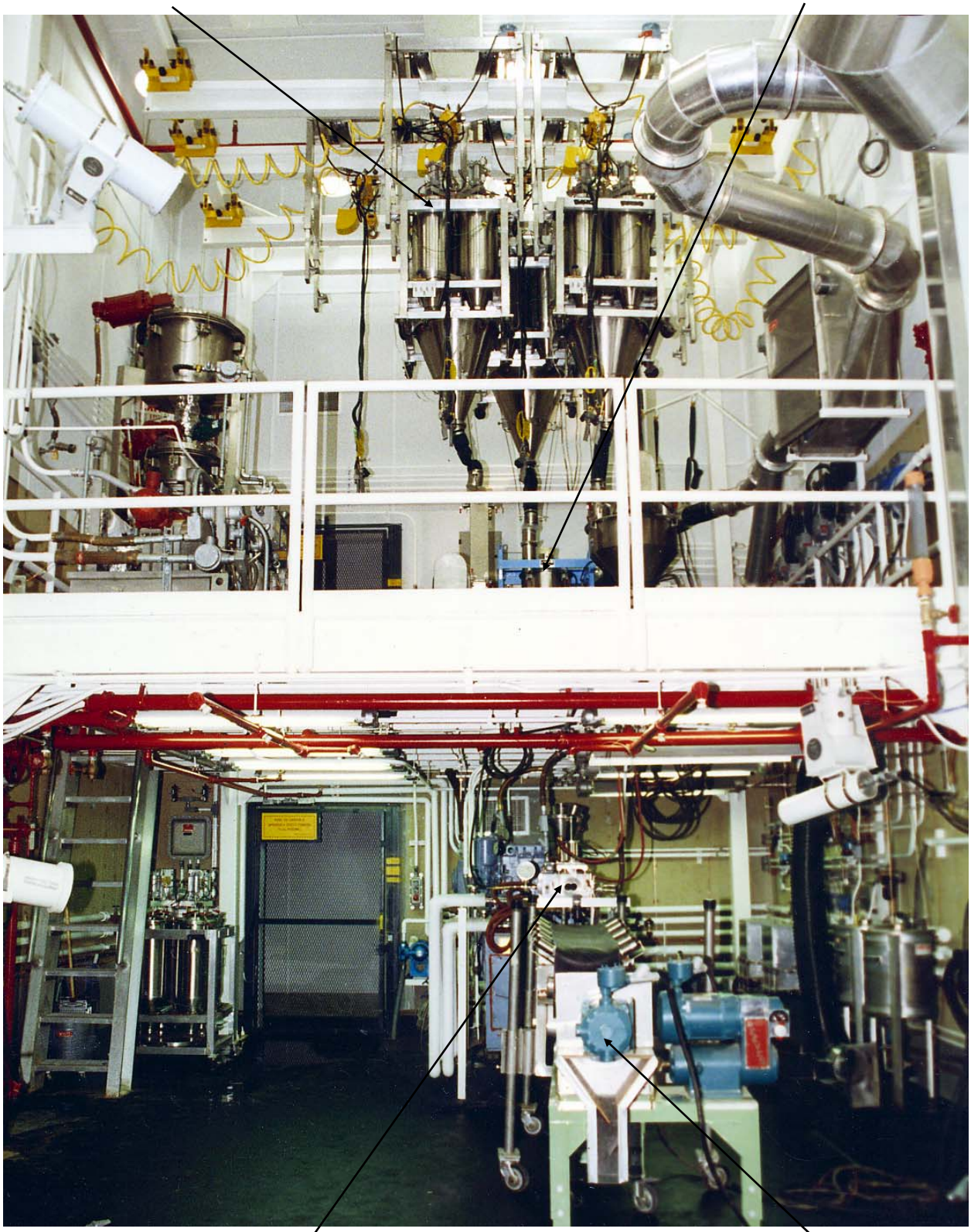


Figure 21: TSE Facility Picture

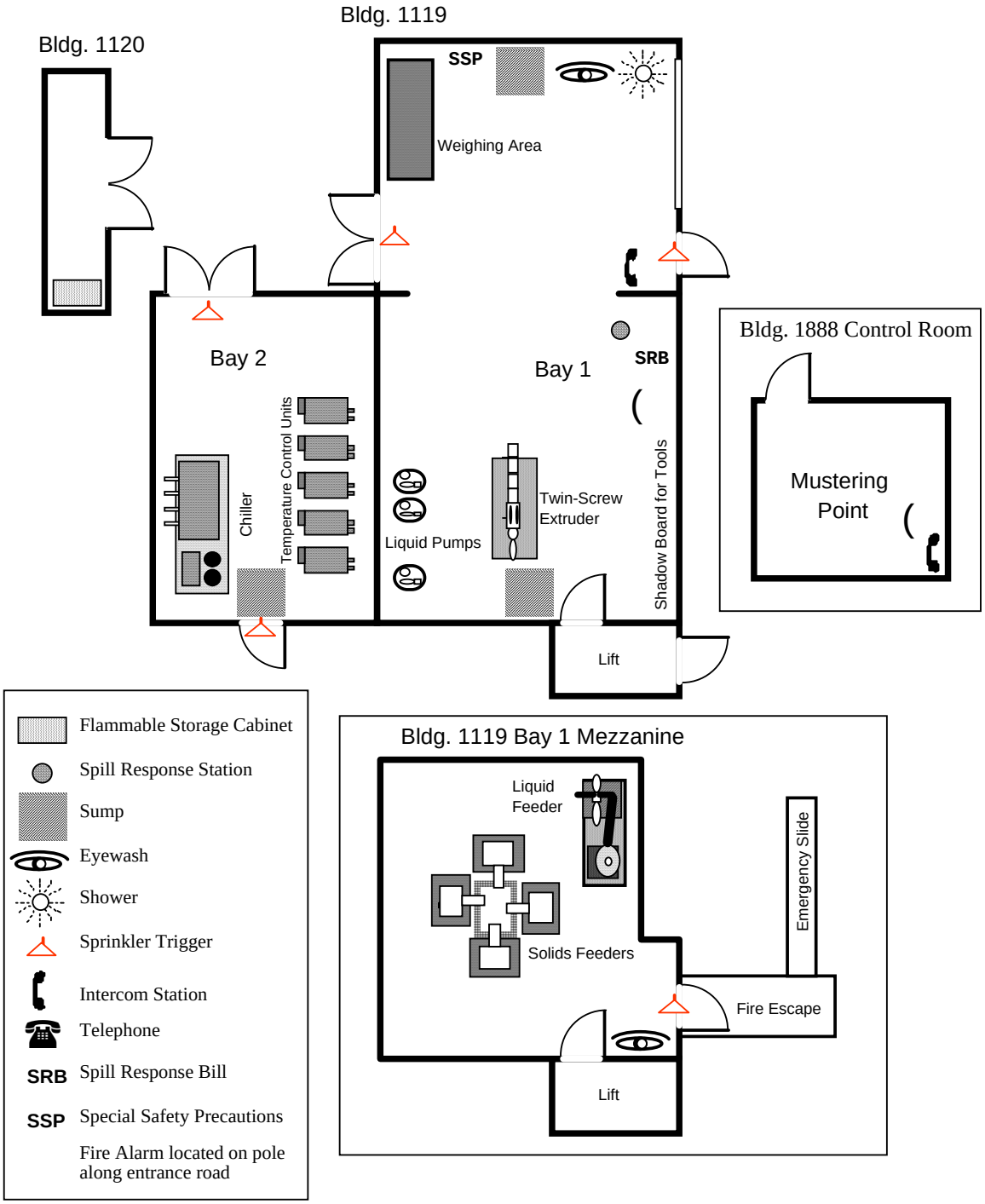


Figure 22: TSE Building Floor Plan

4. Demonstration Approach

4.1 Performance Objectives

The first performance objective was the reduction of emissions and waste realized as a result of replacing the conventional process with the CLEVER process. Emission reduction was verified through a validated material balance as was performed for standard batch processed Lova.

Input + Generation - Output - Consumption = Accumulation

Similar material balances for each operation in the CLEVER process were performed as shown in Figure 23.

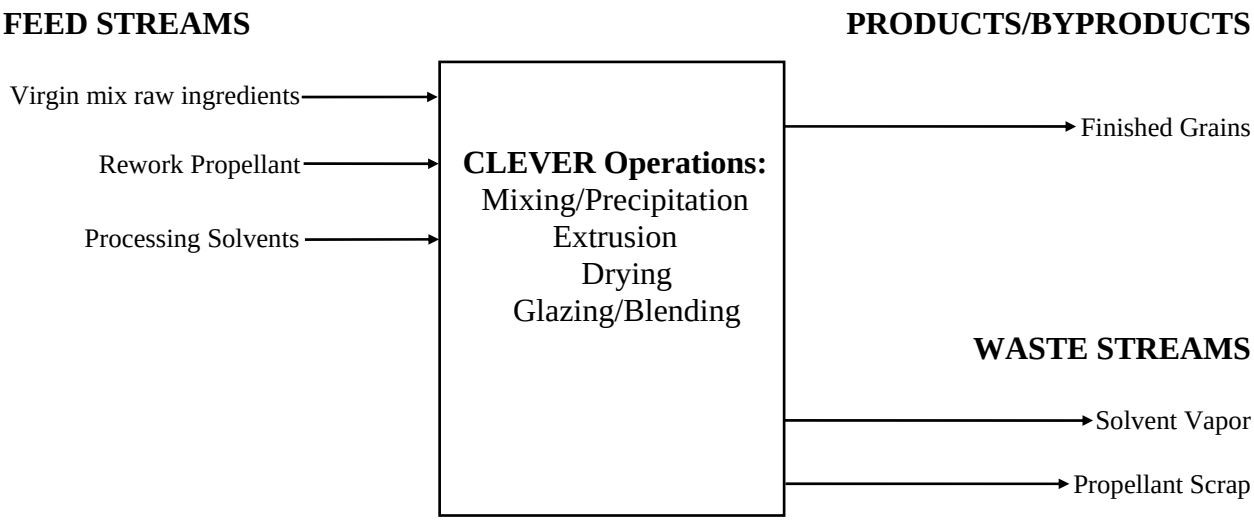


Figure 23: Material Balance for CLEVER

Each appropriate stream was chemically analyzed to verify if fugitive emissions exist. At both demonstration sites, waste streams were collected, analyzed, and measured to determine the waste quantities at the different locations.

A second objective of this project was to demonstrate that the new, cost efficient and environmentally friendly CLEVER process could produce an acceptable gun propellant. Acceptability for a gun propellant is determined by its ballistic and safety performance. The ERGM program is currently qualifying the EX 99 propellant formulation. A propellant specification (reference 8 and also provided in Appendix B) has been prepared for the ERGM program. This specification and the baseline data established by the ERGM program were used to determine acceptable propellant. Additionally, as discussed previously, the EX 99 propellant formulation is very similar to the M43 propellant formulation that was produced at IHDIV, NSWC for several years. All of this data was also available for comparison purposes.

A summary of the performance objectives is shown in Table 6.

Table 6: Performance Objectives

Performance Objective	Objective Met?
VOC Reduction	Yes
Waste Reduction	Yes
Ballistic Performance Comparable to or Better Than Conventional Propellant	Yes
Safety Performance Comparable to or Better Than Conventional Propellant	Yes

4.2 Physical Setup and Operation

4.2.1 Process Reconfiguration Design. The Process Reconfiguration Design phase addressed the issues associated directly with the redesign of the pre-demonstration process and equipment to enable it to produce the EX 99 formulation, shown in Table 7.

Table 7: EX 99 Formulation

Material	Weight Percent
Cellulose Acetate Butyrate (CAB)	12.0
Nitrocellulose (NC)	4.0
Ethyl Centralite (EC)	0.4
RDX	76.0
Acetal/Formal (BDNPA/F)	7.6
	TOTAL = 100%

The proof of concept work conducted prior to the CLEVER project addressed the initial concerns of process feasibility. The lessons learned from previous manufacturing experience were used to improve the pre-demonstration process design, equipment set up and operation. The result was a conceptual design for a technique for the manufacture of the demonstration propellant as well as for constructing a facility for the manufacture of Low Rate Initial Production (LRIP) and large-scale production quantities at a United States location.

Several United States representatives spent time in Sweden learning the process “first hand” which facilitated design change considerations utilizing the existing process equipment. Swedish representatives spent a similar amount of time in the United States to assess the process technology currently used and proposed locations for plant construction. Both exchanges were critical to the definition of the methods and equipment needed to establish the transfer of this Swedish technology to the United States.

4.2.2 Process Reconfiguration

4.2.2.1 Precipitation Process Reconfiguration. The Bofors Precipitation Process as operated at their Karlskoga facility normally uses methylethylketone (MEK) as the solvent to dissolve the propellant ingredients. We are required to use an alternative solvent as the

processing solvent because of environmental concerns. Ethyl acetate was chosen as the alternative solvent. The use of ethyl acetate in place of MEK required Bofors to operate their plant at a lower capacity than normal because the ingredients are less soluble in ethyl acetate. Also, our EX 99 Lova formulation required different ingredients than what Bofors normally uses to manufacture their Lova formulations. First, we required CAB 381-20 instead of CAB 171-15 that Bofors uses. Second, we required Acetal/Formal as an energetic plasticizer versus the typical non-energetic plasticizer that Bofors uses, Acetal Triethyl Citrate (ATEC). These formulation changes produced a somewhat stickier paste than Bofors was accustomed to handling. As a result, they had to modify how they operated their precipitator and cyclone separator to prevent clogging. However, no structural changes to their plant were required for the CLEVER demonstration.

4.2.2.2 TSE Process Reconfiguration. Prior to the CLEVER project, the twin-screw processing efforts on Lova formulations at IHDIV, NSWC were geared towards understanding the process and the process parameters required to safely obtain good mixing and a high quality product. The emphasis was not on waste minimization during this early work. Consequently, many of the extruder processing trials were fairly short in duration, several hours at most. During extended runs to produce a few hundred pounds of product for gun testing, the process had to be shut down frequently to manually refill the feeder and to remove the extruded strand. The extruded stand was then pelletized off-line into the final propellant grains. These frequent stops and restarts generated substantial quantities of scrap and waste.

Meeting the CLEVER project objectives of minimizing the generation of scrap and emissions while maximizing the yield of good material necessitated both remotely refilling the solid feeder while operating the extruder and pelletizing the extruded strand into the final propellant grains on-line. Accomplishing these two process changes required a substantial re-engineering effort of our refill hoppers and the development of new automatic and remote strand handling, pelletizing and product collection equipment. In an effort not to re-invent the wheel, we visited Bofors' production facility at Karlskoga to see how they addressed these same challenges. EX 99 propellant paste as currently manufactured is a fine cohesive powder similar in its flow characteristics to ground RDX. In order to refill their solid feeder, Bofors pneumatically conveys the paste from bins adjacent to the extruder bay to an intermediate hopper located above the feeder. This pneumatic conveying operation is an attended operation. The paste is gravity discharged from the intermediate hopper into the feeder hopper when refill is required. This set-up (depicted in Figure 24) while it worked quite well for Bofors, was not adaptable to the TSE facility at IHDIV, NSWC because of U.S. Navy safety regulations.

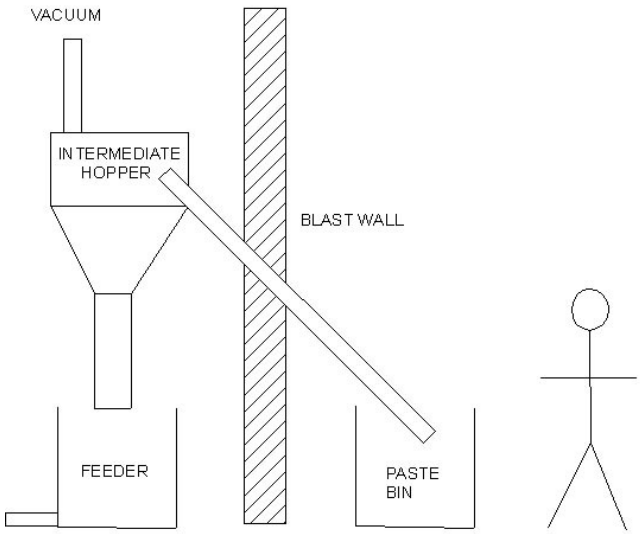


Figure 24: Bofors Solid Feeder Refill System

The existing system at the IHDIV, NSWC facility consisted of four refill racks suspended over each of the four solid feeders as shown previously in Figure 21. Each rack contains four individual refill cylinders, each of which has a nominal capacity of 1 cubic foot. As shown in Figure 25, a vertical plunger seals each refill cylinder.

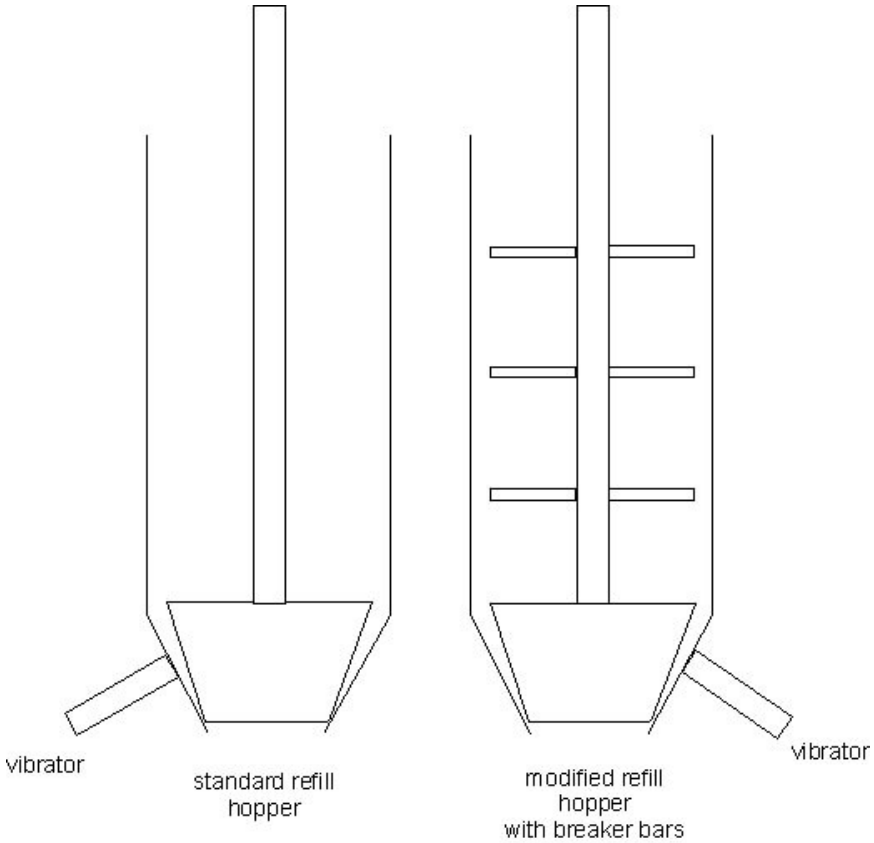


Figure 25: IHDIV, NSWC Refill Cylinders

When refill of the solid feeder hopper is required, a full refill cylinder is selected, the plunger is retracted and the cylinder vibrated to aid the gravity discharge of material into the feeder. The cohesive nature of the EX 99 paste caused considerable problems during refill when the standard refill hoppers were used. When the plunger was retracted to open the cylinder for discharge, the plunger compacted the paste bed above it causing the paste to bridge badly. Even vibration could not prevent the bridge from being formed and subsequently, stopping of the flow of material. The refill cylinders were modified to add radial breaker bars to the plunger shaft (as shown on the right sketch of Figure 25). This modification as well as cycling the plunger up and down during refill solved the bridging problem.

As stated earlier, pelletizing on-line was the second challenge we faced at the IHDIV, NSWC facility. Bofors accomplished on-line pelletizing by the following procedure. Once the extruder is operating at full formulation and is at steady state with good quality strand exiting the dies, the extruder and all feeders are simultaneously shutdown. Plastic tubes are clamped around the die bodies and placed through a blast wall to the cutter bay. The extruder is restarted at full throughput and formulation. A high flow of water is injected into the plastic tubes at the die face. The fast flowing water drags the strand through the tubes into the cutter bay. An operator in the cutter bay allows a slack loop to form and then hand feeds the strands into a bank of pelletizers, manufactured by Nobel Chematur in Sweden. Trip switches controlled off the slack loops turn the individual cutters off and on. The slack loop control is Bofors' method of decoupling the strand extrusion rate from the higher feed rate required by the pelletizer. Once the strands are pelletizing properly, the water flow is decreased to about one liter per minute per tube. This water flow is sufficient to both help transport the strands through the tubes and to firm up the strand by cooling so that it can be cut without smearing. The strand pelletizing is an attended operation with the operator being able to make adjustments and to correct any problems with the cutters while the extruder is operating.

While Lova propellant strands are routinely pelletized as an attended operation at IHDIV, NSWC, the cutting operation is done at a separate area away from the mixers and presses for safety reasons. According to safety regulations, the minimum distance between the IHDIV, NSWC extruder when processing a Class 1.1 material and an attended operation is 300 feet. This regulation clearly precluded us from using Bofors' method of pelletizing without a major design effort to develop a new pelletizing machine that would be self-threading and remotely adjusting.

Our approach to pelletizing (or cutting) on-line was as follows: Once the extruder was at steady state and the observed strand quality was good, a guillotine cutter was activated to cut the two propellant strands off flush at the die face and simultaneously align a set of four foot Teflon tubes with the die face. The strands were guided into the tubes via a funnel block, which also added water at the rate of a one-half liter per minute per tube. This water flow lowered the strand drag and cooled the propellant strands so they could be cut. On exiting the Teflon tubes, each propellant strand was fed through another guide block onto v-troughs. The v-troughs were flooded with water. The water again helped to reduce drag and cool the strands. When the parallel strands were extruded to approximately lengths of 30 inches onto the v-troughs, a guillotine cutter located at the end of the Teflon tubes cut the propellant strand, thus, producing

30-inch propellant sticks. The v-troughs were rotated to drop the 30-inch sticks on to the feed belts of a constantly running Nobel Chematur short cutting machine (i.e. pelletizer). The v-troughs then rotated back up to recapture the continuously extruded strands. The strand as it exits the die is very soft and pushing this strand down four-foot tubes and an additional three feet of v-trough is very close to trying to push a rope. Controlling material viscosity and minimizing drag was essential to preventing the strand diameter from ballooning at the die face causing the conveying tubes to clog. The entire strand conveying and pelletizing system was mounted in a tank so that the water used to convey and cool the strands could be recycled. The set-up used is depicted in Figure 26.

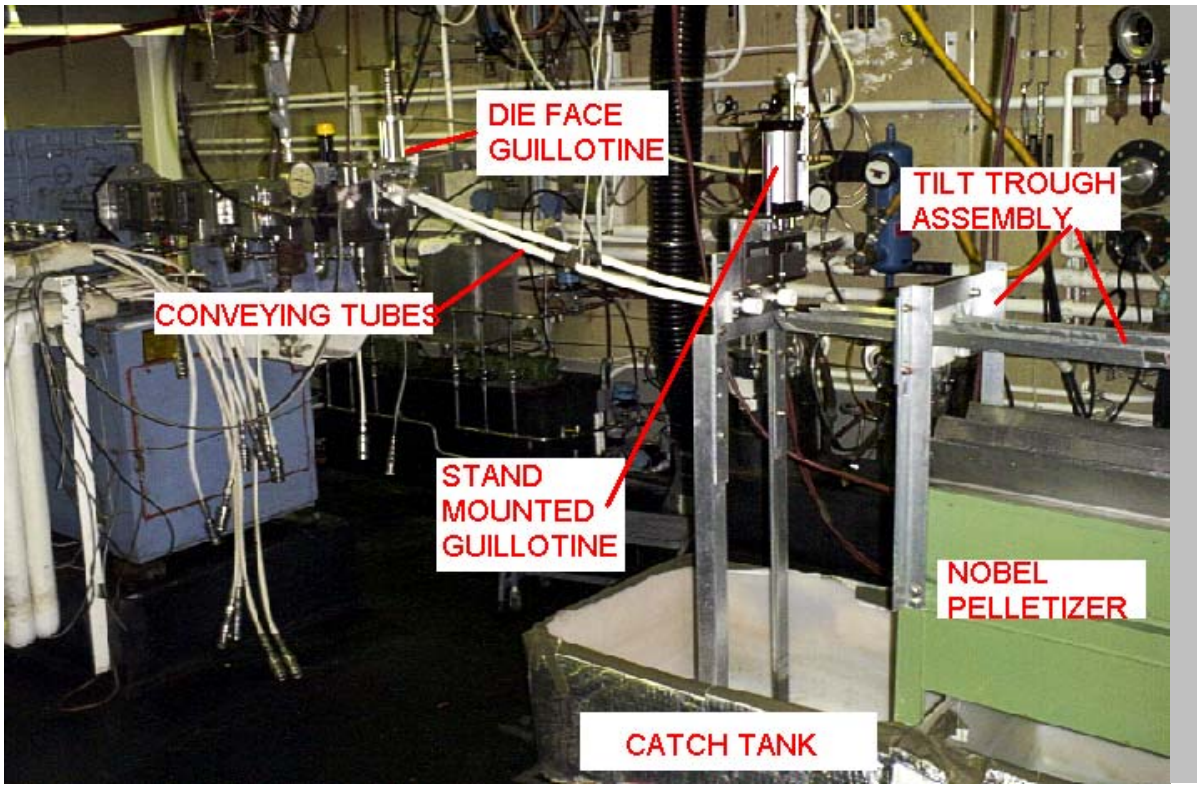


Figure 26: Lova Twin-Screw Extrusion and Pelletizing Equipment Set-Up

The cut grain collection was accomplished using two (2) gallon stockpots mounted on a 12-position rotary indexing table that passed under the pelletizer discharge chute. This set-up allowed us to process about 200 pounds of paste before it was necessary to temporarily shut down to both remove the propellant grains from the building and to refill the refill cylinders.

During routine maintenance of our extruder prior to the demonstration runs, we discovered abnormal wear of our barrel bores. An engineering investigation indicated that the wear was a direct match with the screw configuration we had previously used for Lova processing. This configuration was a very intense mixing screw profile and could have been a contributing factor to the observed wear. The decision was made to evaluate less intense screw designs prior to starting the demonstration runs. Given that we had made other substantial changes to our process and process configuration, we conducted a designed experiment, using design of experiment (DOE) techniques, to determine which combination of screw design and operating

conditions would produce a high quality strand with enough physical integrity to be reliably conveyed and pelletized. The major process changes included:

1. Changing from a single stand die to a two strand (or dual) die and increasing throughput to 30 lb/hr.
2. Using a new and unproven strand handling system
3. New feedstock (Bofors paste)
4. Cutting the propellant strands on-line

The results of the DOE testing, included in Appendix B, indicated the following operating conditions for the TSE process: screw configuration # 48 shown below in Figure 27, throughput of 30 lb/hr, solvent concentration of 12.25%, screw speed of 75 rpm and barrel temperature profile of 120˚F. At these conditions the strand exiting the die is quite soft and, during minor process upsets, is susceptible to mushrooming at the die face causing the conveying system to malfunction. In order to make the strand stiffer and the process more robust, we chose to operate the TSE process slightly off of the optimum point by using a solvent concentration of 12.0% instead of 12.25% and a barrel temperature profile of 110˚F instead of 120˚F.

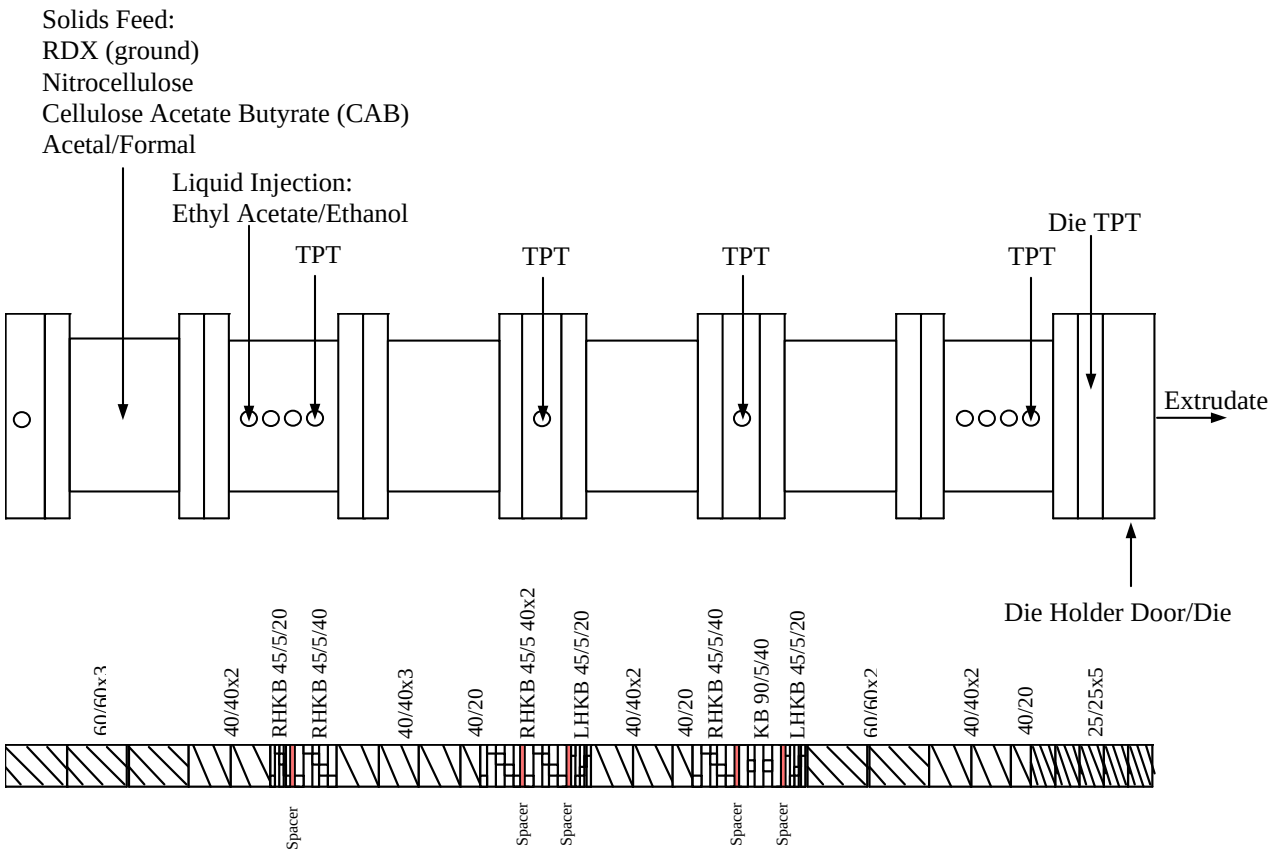


Figure 27: Barrel Configuration and Screw Configuration #48

4.3 Sampling Procedures

4.3.1 Sampling Locations

4.3.1.1 Precipitation Process Sampling Locations. The sampling in Sweden was identical to the previous effort, as described in section 1.5.1. The exact table and diagram are included below as Table 8 and Figure 28 for convenience.

Table 8: Precipitation Process Samples for EX 99 Propellant

		1	**2**	**3**	**4**	**5**	**6**	**7**	**8**	**9**	**10**	**11**
		Sol-	Sol-	Solu-			Wet	Solvent	Water		Wet	Filter
		vent	ids	tion	Steam	Water	Steam	Phase	Phase	Slurry	Paste	Water
Temp	°C	- - -	- - -	X	X	X	X	- - -	- - -	- - -	- - -	- - -
Press	bar	- - -	- - -	- - -	X	- - -	X	- - -	- - -	- - -	- - -	- - -
Vapor Frac	%	- - -	- - -	- - -	X	- - -	X	- - -		- - -	- - -	- - -
Total Flow	kg/hr	X	X	X	X	X	X	X	X	X	X	X
Water	kg/hr	X	- - -	X	X	X	X	X	X	X	X	X
EtAC	kg/hr	X	- - -	X	- - -	- - -	- - -	X	X	X	- - -	X
Solids	kg/hr	- - -	X	X	- - -	- - -	- - -	- - -	- - -	X	X	- - -
Water	%	X	0	X	X	X	X	X	X	X	X	X
EtAC	%	X	0	X	0	0	0	X	X	X	0	X
Solids	%	0	X	X	0	0	0	0	0	X	X	0

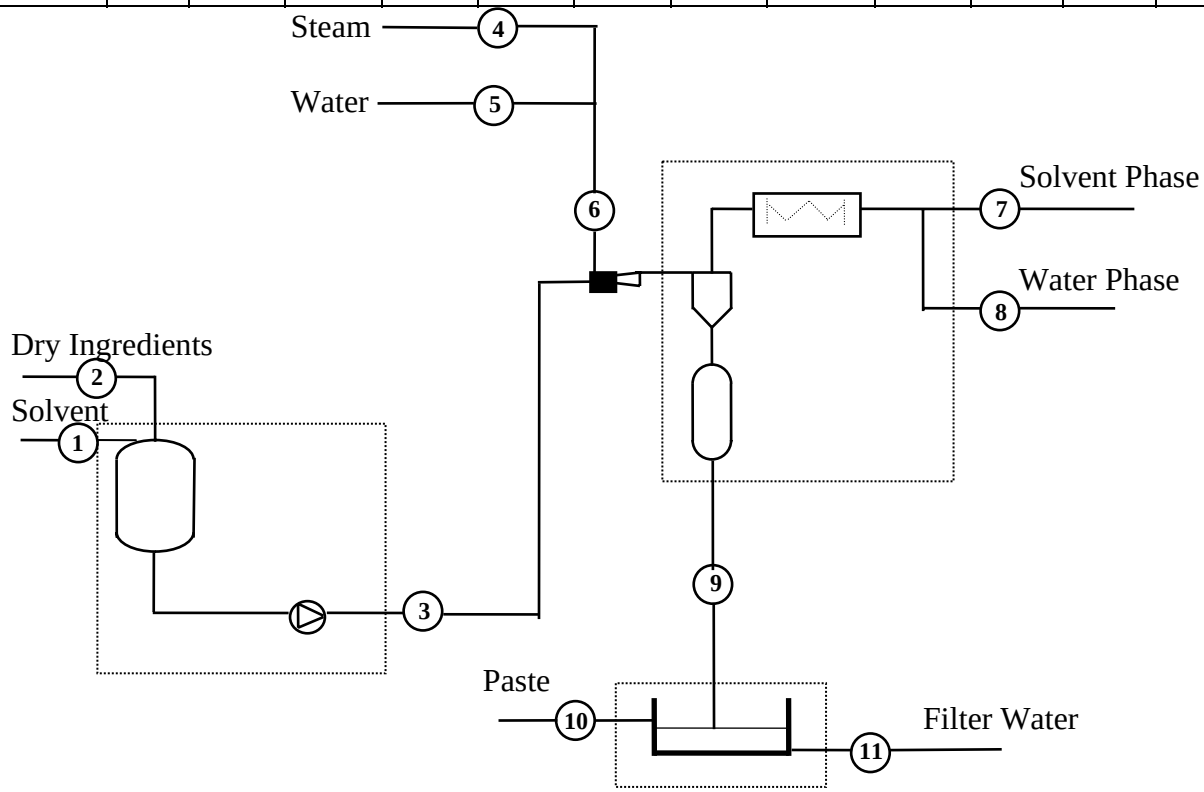


Figure 28: Bofors Precipitation Process Sampling Locations

4.3.1.2 TSE Sampling Locations. The sampling locations for the TSE process are described in Table 9 and depicted in Figure 29.

Table 9: Description of TSE Samples

Site	Type of Sample	Method of Measurement
1	Solid	Solid Feeder Scale
2	Liquid	Mass Flow Meter
3	Liquid	Volume
4	Solid	Scale
5	Solid/Liquid	Calculated from feed rates

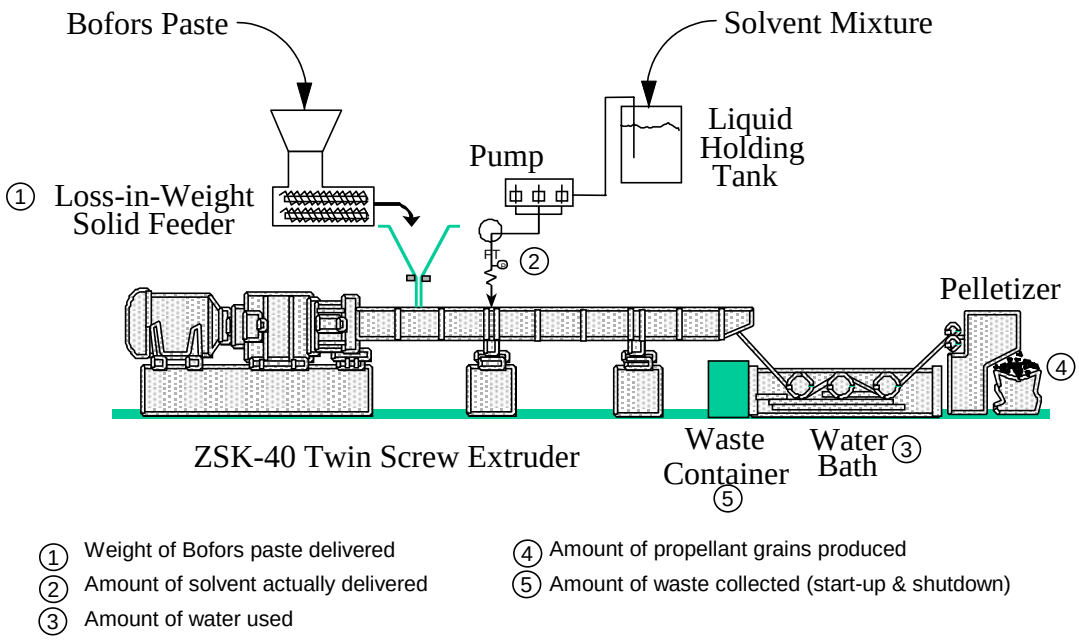


Figure 29: TSE Sampling Locations for EX 99 Propellant

4.3.1.3 Sample Collection. Several changes were made to the original ESTCP CLEVER Technology Demonstration Plan (reference 10). We had originally planned to measure the solvent loss into the processing bay air, however, this proved to be impractical. When we proposed to do this measurement, we overlooked the fact that the building has a once through ventilation system with a complete air change every 6 minutes resulting in extremely low solvent concentrations. Tracking the weight of powder paste refilled to the loss in weight feeder at each refill also proved to be difficult. The vibrators on the individual refill cylinders sometimes malfunctioned. The malfunctions resulted in incomplete emptying of the refill cylinders and the cylinders having to be cycled multiple times, making a running tally inaccurate. Calculating the weight fed from the loss in weight feeder set point and run times proved to be a more reliable and accurate method. We had also planned to individually collect and weigh the material from each ramp up and ramp down based on the assumption that we would have one ramp up and ramp down per run. Because of the mechanical difficulties we experienced, the material from

multiple starts and stops was commingled in the collection container along with some water leakage from the strand conveying tubes. Feeder data was also used to compute the ramp up and down quantities.

4.3.2 Test plan. The safety and small-scale sensitivity tests were conducted on paste samples from Batch 484/98 that was subsequently used during the processing DOE. Safety characterization of Batch 484 was considered to be representative of all three batches. All other paste tests were conducted on all three paste batches. Similarly grain testing was conducted on Lot IH94000E-EX99-0088 of which we had the most material and was considered representative of both lots. Final performance evaluation (i.e. gun firings) was conducted on both grain lots. The tests performed on the CLEVER propellant grains were also performed on propellant grains manufactured by the conventional batch process. The test matrix used to verify acceptable performance of the product produced by the CLEVER process is presented in Table 10 with the following exceptions: Paste Test #7, Deflagration-to-Detonation Transition (DDT) testing was not done. The rationale for not doing this testing was that the other safety and small-scale sensitivity tests characterized the paste material as Class 1.1 thus making this test mute. The test matrix was developed by the CLEVER IPT and documented both by the minutes of the IPT meeting as well as the CLEVER Technology Demonstration Plan (reference 10). There are no specific requirements for the paste other than chemical composition. Also, there are no specific requirements for the EX 99 propellant grains for many of the tests other than heat of explosion, propellant density, propellant chemical composition and gun firing results. In cases where there were no specific requirements, the conventional batch propellant data was used as the baseline requirement so a performance equivalent or better than the conventional batch propellant was the target. The requirements for each test are presented with the results of each test.

Table 10: Propellant Paste and Grain Test Matrix

* - CLEVER processed material only
** - Batch processed material only

PASTE TESTS	PROCEDURE	TEST SITE
1. Paste Chemical Composition*	Section 4.4.1.1	Bofors, Sweden
2. Thermal Stability – Differential Scanning Calorimetry (DSC) *	Section 4.4.1.2	IHDIV, NSWC
3. Microtrac Analysis for RDX Particle Size Distribution**; Malvern Analysis for Particle Size*	Section 4.4.1.3	IHDIV, NSWC; Bofors, Sweden
4. Microscopic Analysis (for free RDX) *	Section 4.4.1.4	IHDIV, NSWC
5. Safety Testing: Impact, Friction, and Electrostatic Discharge*	Section 4.4.1.5; SOPs P60028, P60029 and P60030	IHDIV, NSWC
6. Small Scale Shock Sensitivity: Cap Test and Card Gap Test*	Section 4.4.1.6; NAVSEA Inst 8020.8A	IHDIV, NSWC

7. Deflagration-to-Detonation Transition (DDT) Testing	Test not completed. See paragraph 4.3.2 above	IHDIV, NSWC
PROPELLANT GRAIN TESTS	**PROCEDURE**	**TEST SITE**
1. Safety Testing: Impact, Friction, and Electrostatic Discharge*	Section 4.4.2.1; SOPs P60028, P60029 and P60030	IHDIV, NSWC
2. Small Scale Shock Sensitivity: Cap Test, Card Gap Test and Unconfined Burning Test*	Section 4.4.2.2; Chapter 5 NAVSEA Inst 8020.8A	IHDIV, NSWC
3. Ballistic Impact Chamber (BIC) Test*	Section 4.4.2.3	IHDIV, NSWC
4. High Rate Mechanical Properties*	Section 4.4.2.4;	Army Research Laboratory (ARL)
5. Dimensional Analysis of Finished Grains	Section 4.4.2.5; MIL-STD-286C, Method 504.1.1 or Method 504.6.1	IHDIV, NSWC
6. Scanning Electron Microscopic (SEM) Analysis*	Section 4.4.2.6.	IHDIV, NSWC
7. Heat of Explosion	Section 4.4.2.7; MIL-STD-286C, Method 802.1	IHDIV, NSWC
8. Propellant Density	Section 4.4.2.8; MIL-STD 286C, Method 510.3.1	IHDIV, NSWC
9. Propellant Chemical Composition: CAB/NC, RDX/HMX, EC, A/F	Section 4.4.2.9; MIL-DTL-82965 (OS)A, Section 4.6.1	IHDIV, NSWC
10. Variable Confinement Cook-off Test (VCCT) *	Section 4.4.2.10	IHDIV, NSWC
11. Wide Angle X-ray Diffraction (WA-XRD) for Degree of Mixedness*	Section 4.4.2.11	Steven's Institute of Technology
12. Closed Bomb Burning Rate (High Pressure)	Section 4.4.2.12; MIL-STD-286C, Method 8-1.1.2	IHDIV, NSWC
13. Gun Firing	Section 4.4.2.13	Dahlgren Division, NSWC

4.4 Analytical Procedures

4.4.1 Paste Analysis and Testing. The general procedures used by Bofors and IHDIV, NSWC to characterize the Bofors EX 99 paste are presented in this section.

4.4.1.1 Paste Chemical Composition

4.4.1.1.1 RDX, HMX and Ethyl Centralite Content. The method used is based on High Pressure Liquid Chromatography (HPLC), reversed phase, with methanol/water as the mobile phase. Samples and references are dissolved in acetonitrile. RDX, HMX and Ethyl Centralite contents are determined in relation to the reference solutions, which have the same approximate contents as the samples. The samples are analyzed in a HPLC, supplied with a UV detector.

4.4.1.1.2 Nitrocellulose Content. The nitrocellulose (NC) content is determined by Gel Permeation Chromatography (GPC) and detected by a UV detector at 254 nm. Ethyl acetate is used as the mobile phase.

4.4.1.1.3 Cellulose Acetate Butyrate Content. The sum of the NC content and Cellulose Acetate Butyrate (CAB) content is determined by GPC and detected by a Refractive Index detector. Ethyl Acetate is used as the mobile phase. The CAB content is determined by subtracting the NC content from the sum discussed here.

4.4.1.1.4 Acetal/Formal Content. The acetal/formal content is determined by HPLC similar to the method described in paragraph 4.4.1.1.1.

4.4.1.1.5 Water Content. The water content is determined by using the Karl Fisher titration method.

4.4.1.2 Thermal Stability - Differential Scanning Calorimeter (DSC). DSC is used to determine the temperature at which an exothermic decomposition starts. The sample is heated at a constant rate, °C/minute, from room temperature until the decomposition has taken place. A Perkin-Elmer DSC-7 instrument is used with appropriate PC software that monitors the measuring conditions and calculates the onset and peak temperatures as well as the energy change. In general, the onset temperature is used as the decomposition temperature. A sample of approximately 1 mg is weighed in the crucible (aluminum made crucible with holes or a crucible with a suitable lid that can withstand the final pressure from the gases that are produced). The crucible is placed in the oven. A similar but empty crucible is placed in the reference oven. When the starting temperature is stable, the heating is started at 10 °C/min, through the PC software. When the temperature has increased beyond the total decomposition of the sample, heating is stopped and the calculation is started through the PC. The onset temperature is determined.

4.4.1.3 RDX Particle Size Analysis. Two methods, one for the batch process and one for the CLEVER process were used due to the characteristics of the material being analyzed for each process.

4.4.1.3.1 Microtrac Analysis. Microtrac analysis is a standard test that uses a laser light source to determine particle size distribution. This test is routinely used by IHDIV, NSWC in the conventional batch process to determine the RDX particle size.

4.4.1.3.2 Malvern Analysis. Malvern analysis is also a laser light scattering procedure

used by Bofors to measure the particle size distribution of RDX in their paste. The sample is suspended in a suitable liquid and introduced to a beam of light from a Laser. The light is scattered by the particles into a pattern that depends on the particle size distribution of the sample. The light is focused onto a detector in the form of concentric annular sectors. The detector provides an electronic signal that is recorded by a PC and processed. The RDX particles in the paste are coated with CAB and NC. During sample preparation, this coating needs to be dissolved off of the RDX particles without dissolving them. Therefore, the preparation of the saturated solution samples is critical.

4.4.1.4 Microscopic Analysis. Visual analysis with a microscope is performed to determine the amount of uncoated or free RDX. This quantity should be small for a quality product.

4.4.1.5 Safety Testing: Impact, Friction and Electrostatic Discharge. The purpose of the impact test is to determine the sensitivity of the propellant to initiation by impact forces. The NOS impact tester uses a 5 kg weight while the WO impact tester (ERL Bructeon machine) uses a 2.5 kg weight. The propellant sample (35 mg) is placed on an anvil. The weight is dropped from a specified height and the results of the impact are recorded as positive or negative. Any reaction (smoke, flame, sparks, odor, burn marks on the equipment or a noise above the ambient drop weight noise) is considered positive. Refer to Appendix E of reference 10 for the procedure, SOP P60029. The results are reported as a 50% height either in millimeters or centimeters. The sensitivity ranges are presented below in Table 11.

Table 11: Sensitivity Ranges for Safety Testing

Test	High Sensitivity	Medium Sensitivity	Low Sensitivity
IMPACT			
NOS Impact, 50% height (mm)	13 - 63	63 - 398	398 - 1000
WO Impact, 50% height (cm)	5 - 15	15 - 100	100 - 320
SLIDING FRICTION			
ABL Friction, 20 TIL (psig)	30 or less	40 - 420	560 - 1000
BAM Friction, 10 TIL (newtons)	6 - 54	60 - 144	160 - 360
ELECTROSTATIC DISCHARGE			
ABL ESD, 20 TIL (Joules)	0.0084 - 0.023	0.037 - 0.853	1.72 - 8.33

The purpose of the friction test is to determine the sensitivity of the propellant to initiation by frictional forces. There are two types of friction tests that can be used to determine propellant friction sensitivity: the ABL friction test and the BAM friction test. The ABL friction tester uses a steel wheel that slides one inch across the sample on a steel plate. The propellant sample size is 35-45 mg. The standard speed of the plate is 8 ft/sec. The pressure between the wheel and the plates is varied, using thirteen levels between 30 and 1000 psig to determine the threshold initiation level (TIL). The TIL is the level at which 20 negative results are observed with at least one positive result at the next higher level. Any reaction (smoke, flame, sparks, fumes, odor, or a noise, other than that caused by the equipment is considered a positive result. The BAM friction test is the NATO standard test. It uses a porcelain peg sliding back and forth one centimeter across the propellant sample that is on a porcelain plate. The propellant sample

size is 35-45 mg. The force between the peg and plate is varied between 6 and 360 newtons to determine the threshold initiation level with 10 negative results. Refer to Appendix E of Reference 10 for the procedure, SOP P60030. The sensitivity ranges for each test are presented in Table 11.

The purpose of the electrostatic discharge (ESD) test is to determine the sensitivity of the propellant to initiation by electrostatic discharge. The ABL ESD test uses an approaching gap electrode with a fixed voltage of 5.785 kv and a variable capacitance. The 20 TIL value is determined, ranging from 0.0084 to 8.33 Joules. The propellant sample size is 35-45 mg. Refer to Appendix E of reference 10 for the procedure, SOP P60028. The sensitivity ranges for each test are presented in Table 11.

4.4.1.6 Small Scale Shock Sensitivity: Cap Test, Card Gap Test and Unconfined Burning Test. These tests are conducted to determine the small-scale shock sensitivity of the propellant. The procedures for these tests are outlined in Chapter 5 of NAVSEAINST 8020.8A (refer to Appendix E of reference 10). The Cap or Detonation Test uses a No. 8 blasting cap in an attempt to detonate the propellant sample. A two-inch cube of propellant is placed on a lead cylinder. The blasting cap is placed perpendicular to the lead cylinder and in contact with the propellant sample. The cap is detonated and the result is recorded as a detonation, burned, or fragmented. Deformation (mushrooming) of the lead cylinder 1/8 inch or more is considered a detonation. This test is conducted a minimum of five times or until a detonation occurs, whichever is less.

The Ignition and Unconfined Burning Test consists three trials to burn propellant samples and record the results. The first two trials use a 2-inch cube of propellant each and the third trial uses four two inch cubes of propellant. In each case, the propellant samples are placed on a bed of kerosene soaked sawdust and ignited with an electric match head igniter. The results are recorded as to whether the propellant sample exploded and how long the propellant burned (seconds).

The Card Gap Test uses pentolite boosters to ignite propellant placed into a card gap tube. Cellulose acetate cards (2 inch x 1/2 inch x 1/2 inch) are placed between the booster and propellant. A witness plate is placed at the other end of the card gap tube. Detonation is indicated when a clean hole is cut in the witness plate. The number of cards used is varied until the 50 percent probability of detonation is obtained. The results are recorded and used to determine the hazard classification of the propellant.

4.4.2 Grain analysis and testing. The general procedures used by IHDIV, NSWC characterize the EX 99 Lova nitramine propellant grains are presented in this section.

4.4.2.1 Safety Testing: Impact, Friction and Electrostatic Discharge. The procedures for these sensitivity tests are described in section 4.4.1.5. A propellant grain is crushed and 35 milligrams of the fragments are used for each of the three tests.

4.4.2.2 Small-Scale Shock Sensitivity: Cap Test, Card Gap Test and Unconfined Burning Test. The procedures for these sensitivity tests are described in section 4.4.1.6. For the purposes of hazard classification, all testing may be done at 70 cards, the demarcation between Class 1.1 and Class 1.3 material for this test.

4.4.2.3 Ballistic Impact Chamber (BIC) Test. The BIC Test is a quasi-confined drop weight impact test used to predict the response of an energetic material to an arbitrary shock or impact. The growth of the reaction in the energetic material has a major role in determining its response to these stimuli. This test was designed to initiate a sample with a fixed impact that would be sufficient to ignite all energetic materials regardless of their sensitivity to impact. For propellants, a 2.5 kg drop weight is used to impact the sample contained in a confined volume in the WO Impact Tester. The sample size is 45 mg in the form of a 5 mm diameter disc approximately 1.2 mm thick. The hot gases from the impact induced ignition accelerate a pellet down a barrel and the resultant work and rate of work provide a measure of the extent and rate of growth of reaction. See reference 11 for more details.

4.4.2.4 High Rate Mechanical Properties. The purpose of this testing is to determine the fracture characteristics of the propellant grains under conditions similar to gun firing. This testing will be conducted at three temperatures representing the gun operating range (+20°F, +70°F, and 120°F). A servohydraulic tester is used to measure the mechanical response of the propellant under controlled compression measurements made at any rate up to 1000 s^{-1} for a specimen about 1 cm in length. The test produces a mechanical stress-strain curve from which the following parameters can be determined: modulus (E), maximum stress (σ_m), strain at maximum stress (ε_m), stress at failure (σ_f), strain at failure (ε_f) and failure modulus (E_f). While modulus, maximum stress, and strain at maximum stress have the usual engineering definitions, the point of failure and the failure modulus have special definitions. The failure modulus is the slope of the stress-strain curve in the near-linear region between strain at maximum stress and twice that value. If no maximum stress occurs in the region of failure because of work hardening and plastic failure, the failure modulus is measured between the strain at failure and three times that value. The failure point is determined by the intersection of the two lines that determine the modulus and the failure modulus. The strain at that intersection point is called the strain at failure, and the stress at failure is the corresponding stress on the response curve. See reference 12 for more details.

All of these parameters are required to characterize the material. The modulus describes how quickly the stress rises with strain, the stress and strain at failure show where the material begins to yield, and the maximum stress and strain at maximum stress provide ultimate strength information and the failure modulus describes the nature of the failure process (brittle fracture or plastic failure).

4.4.2.5 Dimensional Analysis of Finished Grains. The following dimensions (shown pictorially in Figure 2 of MIL-STD-286C, Appendix E of reference 10) are measured in accordance with either Method 504.1.1 or Method 504.6.1 of MIL-STD-286C: grain length (L) , grain diameter (D), perforation diameter (d), small and large outer web (W_{oS} and W_{oL}), middle webs (W_{m1}, W_{m2}, W_{m3} and W_{m4}), inner webs (W_{i1} and W_{i2}), and around-outer webs (W_{ao1} and W_{ao2}). All dimensions are measured with an image analyzer except the grain length and diameter that are measured with a micrometer. Measurements are taken on a minimum sample of 40 grains and averaged. The average for each specified dimension is reported as well as the standard deviation.

4.4.2.6 Scanning Electron Microscopic (SEM) Analysis. Grains will be prepared and

analyzed using the methodology developed for assessment of the quality of the M43 production grains. Five grains from each sample will be cleaved longitudinally. The fracture surface will be coated with approximately 30 nanometers of gold/palladium. Each surface will be divided into four quadrants, one region of each quadrant will be randomly chosen for SEM analysis, thus eliminating bias from the analysis. Micrographs of the selected regions will be obtained at 2000x magnification. A numerical ranking scheme was developed for assessing the quality of individual M43 batch production lots of material relative to one another. This same system was used to compare the CLEVER material and the conventional batch product. The numerical ranking scheme was used to evaluate the homogeneity (i.e. quality) of M43 Lova propellant based on the characteristics that were deemed to be undesirable. Six characteristics are evaluated and assigned a numerical value according to the quantity of undesirable characteristics that are present. The characteristics that are evaluated are: particle size distribution, binder rich regions, RDX rich regions, porosity/microporosity, void content and RDX large particle content (> 100 micron). The propellant sample is then assigned an overall ranking value from 1 to 5 based on the numerical values of the six characteristics (1 is the best and 5 is the worst). The numerical ranking scheme was developed in an attempt to quantitatively analyze the propellant quality and to help people, those not necessarily intimately involved with SEM analysis, understand the scanning electron micrographs. The assessment is subjective, however, the difference between an overall ranking of 1 vs. 5 is very evident.

4.4.2.7 Heat of Explosion. The heat of explosion provides an indication of the composition of the propellant. A weighed sample is burned in a bomb to measure the calorific value of the exothermic reaction. The procedure used is Method 802.1 of MIL-STD-286C.

4.4.2.8 Propellant Density. The propellant density provides an indication of the quality of the propellant sample. An air pychnometer is used to determine the propellant density as described in Method 510.3.1 of MIL-STD-286C. Refer to Appendix E of reference 10 for that specification.

4.4.2.9 Propellant Chemical Composition. The procedure for the chemical analysis for NC, RDX/HMX, CAB, EC, Acetal and Formal are described in Section 4.6.1 of MIL-DTL-82965(OS)A, reference 7. The requirements for propellant composition are outlined in Section 3.3 of MIL-DTL-82965 (OS)A, reference 7.

4.4.2.10 Variable Confinement Cook-Off Test (VCCT). The VCCT is a small-scale test that is used to evaluate the effects of confinement on the thermal behavior of propellants. The VCCT was designed to study the effects of a slow cook-off environment. The VCCT test assembly consists of a one-inch diameter by 2.5-inch long propellant billet contained within a 0.012 inch thick aluminum sleeve. The aluminum sleeve is centered within a steel confinement sleeve of variable thickness with steel washers. The thickness of the confinement sleeve may be varied from 0.015 to 0.120 inch in increments of 0.015 inch. This assembly is sandwiched between two mild steel witness plates. Heating of the test assembly is achieved using a laboratory oven or through the use of heating bands at a slow cook-off rate of 3.3°C per hour. Two thermocouples are used; one to control the temperature of the test assembly and the other continuously monitors the temperature as the test proceeds.

The VCCT test sequence starts by evaluating the behavior of the propellant sample at a

predetermined level of confinement. The temperature of the test assembly is set at 55°C below the expected cook-off temperature and maintained at this temperature for 2 hours. The temperature is then increased at a linear rate of 3.3 °C per hour until a reaction occurs. The reaction level is noted and the test repeated in step intervals with a steel sleeve of another thickness (0.015 inch thicker or thinner). The equivalent pressure of the confinement level, at which the threshold occurs from a pressure rupture to a more severe reaction, is used as a means of ranking propellants in terms of slow cook-off behavior. The reaction levels used are compatible with the definitions described in MIL-STD-2105B and are categorized as follows: burn, deflagration, explosion, partial detonation, and detonation. See reference 13 for more details.

4.4.2.11 Wide Angle X-Ray Diffraction (WA-XRD). The ultimate performance of heterogeneous materials such as propellants that constitute various ingredients critically depends on the "degree of mixedness" of the ingredients and the reproducibility of the mixing process. At Steven's, a novel x-ray technique is being explored to quantitatively characterize the "degree of mixedness" of highly filled materials. In the CLEVER project, this technology was applied to determine the "degree of mixedness" of the CLEVER propellant and to compare it with the conventional propellant.

In WA-XRD, a given substance produces a characteristic diffraction pattern upon irradiation with collimated x-rays. Qualitative analysis is accomplished by identification of the particular diffraction pattern of a substance from the control samples and the standard diffraction tables. When a mixture of ingredients is present in a given sample, the mixture gives rise to a convoluted diffraction pattern. With the computational techniques developed, the diffraction patterns are deconvoluted and each ingredient in a mixture is identified. Computerized analysis is carried out by utilizing the integrated intensity of the diffraction pattern of a particular ingredient that depends on the concentration of that ingredient in the given mixture. With this technique, typical mixtures with four or more ingredients can be distinguished and quantitatively analyzed. Furthermore, additional information such as polyforms present and degree of crystallinity of the particles is also obtained.

The quantitative "degree of mixedness" analysis is achieved by carrying out systematic "window" measurements at various locations of the sample and multiple samples of a given lot. Various "mixing indices" based on statistical variance of the individual concentration measurements are devised to characterize the degree of mixing. These include mixing index (MI) and coefficient of variation (v). The MI range from zero, for completely segregated system, to one for ideally mixed system. Thus, different processing conditions and different lots of products can be evaluated, indexed, and compared. Such measurements are carried out at desired scales of examination that are selected from the requirements of the mixing process.

4.4.2.12 Closed Bomb Burning Rate (High Pressure). This test method consists of igniting propellant, at a known loading density, in a closed bomb of fixed volume and measuring the rate of pressure increase (dp/dt) versus the pressure. From this information, the relative quickness and relative force of a propellant versus a standard can be determined as well as the linear burning rate (in/sec). The peak pressure developed by the test sample depends on the loading density that is defined as the weight of the sample divided by the volume of the closed bomb. This pressure should correspond to peak pressures expected in the actual gun system.

The closed bombs are temperature controlled to maintain specific test temperatures. In this case, the test temperature will be ambient temperature (70°F). The procedure used is Method 801.1.2 of MIL-STD-286C. Refer to Appendix E of reference 10 for this specification.

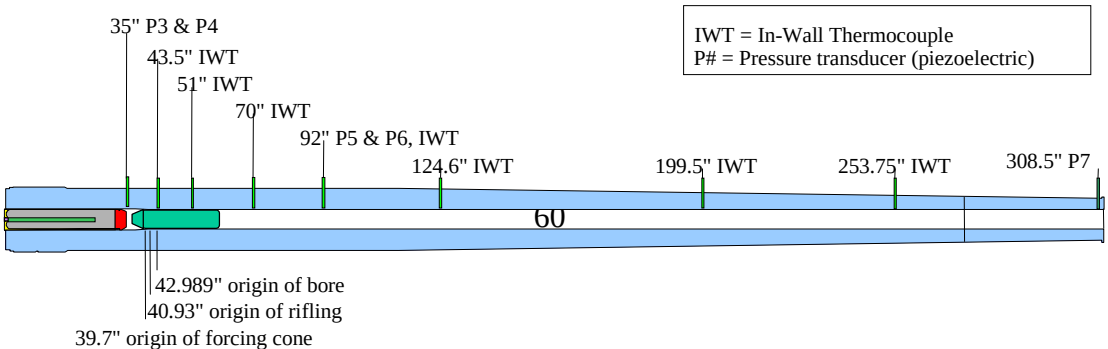
4.4.2.13 Gun Firing. In August 2000, both CLEVER processed grain lots (IH94000E-EX99-0088 and IH94000E-EX99-0089) were successfully fired in the 5"/54 gun. A batch processed gun propellant lot, IH23099DEX-99-FB01, was also tested as the control sample. In summary, the gun firing of the CLEVER propellant was conducted in the same configuration and the same manner used to test the EX 167 Propelling Charge of the ERGM round loaded with batch processed EX 99 propellant. A test slug, called HiJack and weighs 110 lb, was used for this testing to simulate the appropriate ERGM payload. Several rounds were initially fired to determine the proper charge weight (three for the CLEVER lot that was tested first, IH94000E-EX99-0088, and one for the second CLEVER lot tested, IH94000E-EX99-0089). The charge weight assessment consists of probing from 45 kpsi up to 75 kpsi to determine the required weight of propellant. The ERGM operating pressure is 65 kpsi and the proof pressure is 75 kpsi. Once the charge weight assessment was complete, the gun ballistics of each CLEVER lot was determined by firing five rounds, of the determined charge weight, at a temperature of 70˚F. Two tests were conducted with the batch standard, Lot IH23099DEX-99-FB01. From this testing the following data was generated: pressure time traces, ejection time, muzzle exit velocity, muzzle energy, temperature coefficient and uniformity. The data was compared to the performance requirements outline in ERGM performance specification, WS 33457 (Performance Specification for the 5-Inch Extended Range Guided Munition (ERGM) Propelling Charge EX 167), as well as to the data from gun testing batch processed material. Gun testing ultimately determined the success of the demonstration from a product performance aspect.

The testing was performed as an adjunct to other testing being performed for a 5"/54 gun product improvement program (PIP). Program and scheduling requirements dictated that all firings were conducted at +70˚F rather than the three temperatures (+20˚F, +70˚F, +120˚F) called out in the CLEVER Technology Demonstration Plan. The following is an excerpt of the testing details taken from the test report provided by the IHDIV, NSWC Gun System Engineering Branch, who has responsibility for the PIP program and conducted the testing.

4.4.2.13.1 Test Hardware. Testing was conducted in an eight-inch open gun mount that had been modified to accept a five-inch barrel. The barrel that was used was a 5"/54 instrumented autofrettaged Mk 19 Mod 2 barrel (S/N # 17451) with an eight caliber extension (S/N # 595). The barrel was instrumented with piezoelectric pressure gauges and in-wall thermocouples as shown in Figure 30.

Figure 30: Instrumented 5-inch Gun Barrel

4.4.2.13.2 Test Articles. The propelling charges, Figure 31, were loaded in accordance



with drawing 53711-7263283 with the following exceptions:

- Ethafoam (1/4" thick) was wrapped radially in the base of the cartridge case, as necessary to take up free volume.
- Wear liner and baseline closure plug (DWG. 53711-7263635) was replaced with the Type A closure plug/ablative paste (380 grams). No crimp or adhesive was used to secure the plug to the case.

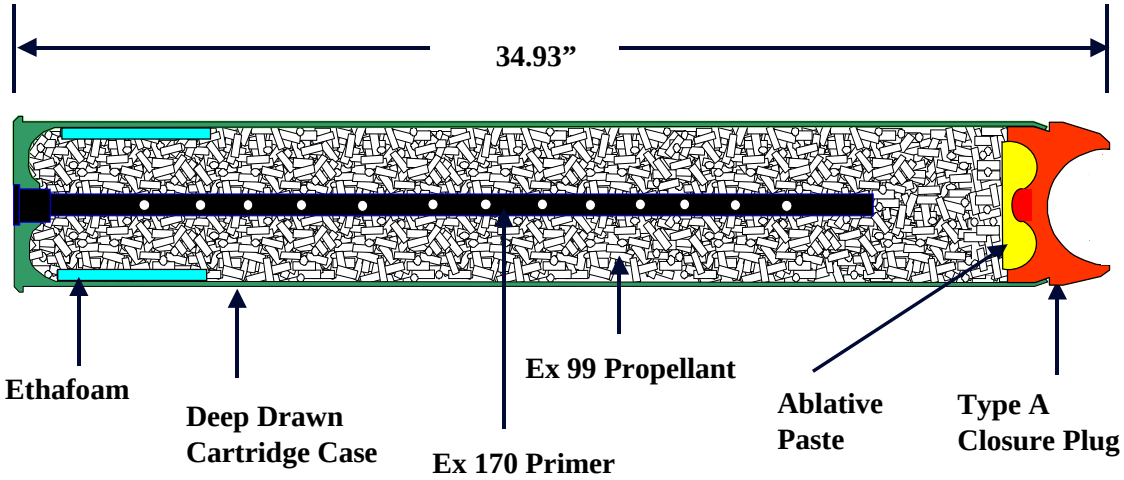


Figure 31: Schematic EX 167 Propelling Charge Loaded for Proof Testing

Test projectiles (aka HiJack) were manufactured in accordance with drawing 07609-SK76631. The firing lineup and propelling charge configuration is given in Table 12.

Table 12: Firing Lineup/Propelling Charge Configuration[a]

Date	Round #	Charge Weight (lbs)	PPD (in)	Projectile Seating Distance (in)	Projectile Type	Obturator Type	Propellant Conditioning Temperature (°F)	Projectile Weight (lbs)	Ablative Material	Primer S/N[b]	Ethafoam Size (in)
Practice Round (Lot SPCF-11247)											
8/22/00	1	20.26		35.10	MK 92 BL&P	COPPER	70	70	NONE	-	NONE
Lot 88 (CLEVER Process)											
8/22/00	2	23.90	2.0	38.00	Hijack	HiFrag	70	110.2	Paste	93	8x15.5
8/22/00	3	24.60	1.7	37.94	Hijack	HiFrag	70	110.0	Paste	96	8x15.5
8/22/00	4	25.70	1.8	38.25	Hijack	HiFrag	70	110.1	Paste	85	none
8/22/00	5	26.00	1.7	37.88	Hijack	HiFrag	70	110.2	Paste	104	none
8/22/00	6	26.00	1.7	37.81	Hijack	HiFrag	70	110.4	Paste	105	none
8/22/00	7	26.00	1.7	37.75	Hijack	HiFrag	70	110.1	Paste	46	none
8/22/00	8	26.00	1.7	37.81	Hijack	HiFrag	70	110.3	Paste	56	none
8/22/00	9	26.00	1.7	37.75	Hijack	HiFrag	70	110.0	Paste	60	none
Lot FB01 (Batch Process)											
8/22/00	10	24.80	2.0	37.81	Hijack	HiFrag	70	110.0	Paste	69	6x15.5
8/22/00	11	24.80	2.0	37.75	Hijack	HiFrag	70	110.3	Paste	70	7x15.5
Lot 89 (CLEVER Process)											
8/22/00	12	24.70	1.6	37.81	Hijack	HiFrag	70	110.2	Paste	71	8x15.5
8/22/00	13	26.00	1.6	37.88	Hijack	HiFrag	70	110.0	Paste	72	none
8/22/00	14	26.00	1.6	37.81	Hijack	HiFrag	70	110.2	Paste	83	none
8/22/00	15	26.00	1.6	37.88	Hijack	HiFrag	70	110.2	Paste	55	none
8/22/00	16	26.00	1.6	37.88	Hijack	HiFrag	70	110.1	Paste	78	none
8/22/00	17	26.00	1.6	37.88	Hijack	HiFrag	70	110.1	Paste	79	none

[a]All cases were MK9-1 from Lot # NIV87G001017
[b]The EX170 primers were from Lot #IH00BEX98P001

4.4.2.13.3 Test Procedure. The EX 170 primers and EX 99 propellant were temperature conditioned at +70°F for a minimum of 72 hours prior to testing. A test round was fired to check the instrumentation and set-up. The test round consisted of a Mk 67 propelling charge and a Mk 92 BL&P projectile. A probe test was performed where the charge weight was increased starting at the predicted 45,000-psi charge weight of 23.9 pounds. The propelling charges were loaded on the spot after analyzing the previous firings' results. The charge weight was increased after each firing until the desired energy of 18 MJ was achieved while staying at or below 65,000 psi. Additional firings were conducted at that same charge weight to evaluate its consistency. Two rounds were fired using the same configuration by replacing the EX 99 twin-screw processed propellant with the standard batch EX 99 propellant. This was performed to compare the continuously processed propellant to a standard. All charges, except one (round #15), were fired within 5 minutes after removal from temperature conditioning. All projectiles were hand-rammed.

5. Performance Assessment

5.1 Performance Data and Assessment for Paste and Grains

5.1.1 Project Demonstration: Propellant Manufacture

5.1.1.1 Paste Manufacture. The CLEVER project began in the fall of 1997 with the award of a contract to Bofors for the production of three propellant paste batches totaling 2030 pounds. This material was delivered approximately one year later in the fall of 1998. After some initial testing including some processing trials in the extruder, it was discovered that these three lots had been inadvertently manufactured using CAB 171-15 instead of CAB 381-20. CAB 171-15 is typically used by Bofors in their Lova formulations. The U.S. uses CAB 381-20 in its Lova formulations. Once Bofors was informed of the error, they obtained the proper ingredients and manufactured three replacement batches. These three batches (484/98,485/98 and 486/98) were delivered in January 1999.

5.1.1.1.1 Batch 484/98. This batch was the one produced with the proper CAB and significant problems were encountered. The paste that is precipitated and separated from the process water in the cyclone separator was much stickier than anticipated. The result was that the process was stopped four times due to clogging in the cyclone and it's associated piping. As a result of the frequent process interruptions, only 258 kilograms (kg) of useable paste was produced from this run. This amount is only 65% of theoretical and reflects the large amount of material lost in cleaning out the cyclone clogs as well as the losses in the transfer equipment and drier due to the paste's propensity to clump into agglomerates that then dried into large unusable lumps.

5.1.1.1.2 Batch 485/98. Based on the experience gained during Batch 484/98, Bofors increased both the steam and water flow rates to the cyclone by 50%. As a result, they were able to improve the flow through cyclone and only experienced two process interruptions due to clogging. This improvement increased the yield to 340 kg. This amount is 86% of theoretical.

5.1.1.1.3 Batch 486/98. No major process improvements were implemented for this batch since physical changes to the precipitation and cyclone equipment were required to prevent all clogging. Batch 486/98 was essentially a repeat of Batch 485/98. A total of 370 kg of paste was produced for a yield of 94%.

5.1.1.1.4 Mass Balance. Bofors' report (reference 9) that contains the mass balance information is included in Appendix B. The mass balance information is based on an average of Batches 485/98 and 486/98 only since there were too many problems during Batch 484/98 to obtain reliable data. It should be noted that the plant equipment was drained and cleaned out between batches to prevent cross contamination so we could look at batch-to-batch variation. This additional step resulted in additional material lost because material that is normally considered equipment holdup and carried over to the next batch was cleaned out and counted as material loss. A second source of confusion is in the area of solvent recovery. Bofors normally recovers solvent by distillation from their spent process water. However, since they did not want to contaminate their solvent recovery system (normally used for MEK) with ethyl acetate, their

solvent loss rates were partially estimated.

The mass balance shows a loss of 45 kg of solids per batch. A total of 22 kg of this loss was lumps of material that were collected on the strainer when the precipitation receiver was dumped to the filter. This material is normally rework, not waste, that is added into the next batch. This leaves 23 kg per batch unaccounted for. What is not included in this balance is the previously mentioned holdup, for example residual paste in transfer containers, dusting and paste lumps disposed of when the clogs were cleaned out. None of that material was weighed and recorded. It is estimated that the true solid waste for a plant run in a continuous manner will amount to less then 2% or less than 10 kilograms per batch.

Bofors reported an average 52 kg of solvent loss for Batches 485/98 and 486/98. This loss equates to 15 kg of solvent emissions per 100 kg of paste. However, they did not account for an additional 16 kg that could be recovered from the filter water and vacuum condensates. Bofors normally discharges the filter water and vacuum condensates to the sewer but we would send these streams to a solvent recovery still. When this change is considered, the solvent usage drops to 10.3 kg per 100 kg of paste manufactured during the demonstration. Bofors also calculated a non-recoverable solvent usage of 6 kg per 100 kg of product using the Aspen simulation code and an assumed batch size of 600 kg. This result seems low and the actual usage probably lies somewhere between 6 and 10 percent.

5.1.1.2 TSE Operation. The original intent was to manufacture sufficient EX 99 gun propellant material from each of the three paste batches (484/98,485/98 and 486/98) to conduct gun firings and access batch-to-batch variation. The initial estimate was a total of 1000 pounds of propellant grains for this testing. This proved to be optimistic. The need to conduct an extensive designed experiment to optimize our process and work some of the bugs out of the new strand handling and pelletizing equipment necessitated that we restructure this goal. The bulk of Batch 484/98 and a portion of Batch 486/98 were expended to do this initial work. After discussions with the IHDIV, NSWC gun systems engineer, interior ballistician and test personnel, we determined that 200 pounds of grains from Batch 486/98 and 400 pounds from Batch 485/98 would be adequate for gun firings. The plan was to produce the propellant in one continuous run by operating three shifts around the clock. The plan was to process 300 pounds of Batch 486/98 first followed by 600 pounds of Batch 485/98. Although the paste was delivered in early 1999, laboratory testing, tooling design and some initial processing trials needed to be completed before we were ready for extended extruder runs. This delayed the actual demonstration extruder runs to manufacture the gun test lots until Spring 2000.

5.1.1.2.1 TSE Run #IH94000E-EX99-0081. The run was started on May 4, 2000 using material from Batch 486/98. During the processing of this batch we experienced eight unplanned shutdowns. The cause of these shutdowns included mechanical failure of the conveying and pelletizing systems, power loss and computer failure. It must be emphasized that these shutdowns were not related to the quality of the paste batch. This run was the first time that these subsystems were integrated and subjected to the stresses of an extended run and subsequently numerous weaknesses of the process were exposed. Field repairs for most of these problems were implemented as we went along. We processed 300 pounds of Batch 486/98 and began processing material from Batch 485/98. Processing of Batch 485/98 was proceeding smoothly with only one shutdown for a minor mechanical fault. Approximately 30 hours into

the run and after we had processed first 300 pounds of Batch 486/98 followed by 256 pounds of Batch 485/98 (a total of 556 pounds of paste), we experienced a major process malfunction. The o-ring in the liquid injection nozzle that supplies the processing solvent to the extruder failed. The loss of the only liquid feed caused a jam within the extruder and an emergency shutdown. Jams of this type are definitely not experienced during normal operations. At this point the run was terminated and data collection for the demonstration ceased while the jam was safely cleared and the facility shutdown pending an engineering investigation of the problem. The failure was attributed to a long-term incompatibility between the o-ring compound and the process solvent. The problem was corrected and the demonstration run was resumed on May 24, 2000 as TSE Run IH94000E-EX99-0083.

5.1.1.2.2 TSE Run #IH94000E-EX99-0083. The purpose of this run was to complete the CLEVER demonstration by processing the remaining 20 pounds of paste from Batch 486/98 and 320 pounds from Batch 485/98. This goal was successfully accomplished during a 17-hour continuous run. There were two shutdowns during the course of this run. One shutdown was for a mechanical problem with the pelletizer, which was quickly corrected. The second was a planned shutdown during which the refill racks were reloaded and pelletized grains were removed from the building. This second extended run went much smoother than the first showing that we learned a lot from the first extended run.

5.1.1.2.3 Grain Drying and Sorting. During shutdowns, the pelletized grains were emptied from the collection stockpots onto Lova powder drying trays. Each tray held about 20 pounds of solvent wet grains. The grains were air dried at ambient temperature for two days followed by drying in a forced air oven at 140˚F for 4 days.

After drying it was necessary to sort through the grains to remove any which were too short, too long or damaged. These grains would normally be designated as rework material and added to the dissolver in the Bofors Precipitation Process. When pelletizing Lova in our batch production process, we expect less than 1% rejects from the large arms cutter. During the CLEVER demonstration runs we averaged a reject rate of 14% from the pelletizer. This reject rate was a direct result of having to adapt an attended cutter to a remote, on-line operation. In order to explain this large disparity, it is necessary to understand how the Nobel Chematur short cutting machine normally functions, the modifications and the impact of those modifications on the reject rate. The principle of the machine is as follows. Strand is conveyed to the cutter head by an in-feed conveyor. At the cutter head the strand is gripped between the belts of a tractor drive that is geared to the in-feed conveyor. The tractor drive feeds the strand through a nozzle that provides support to the strand while it is being cut. At the exit to the nozzle is a fixed speed motor driving a bar with a knife that chops the strand into grains. In the machine at IHDIV, NSWC there are actually two conveyor sections feeding a single rotating cutter bar. Since the cutter bar is driven at a fixed speed, grain length is determined by the tractor conveyor speed. Therefore, it is impossible to synchronize the cutter to the linear extrusion rate of two strands. We were faced with two choices. We could design and build a new prototype cutter at an estimated cost of $400,000 or we could use our existing cutter in a decoupled mode. Faced with funding constraints, we chose the second option. We decoupled the extrusion speed from the cutter speed by cutting the extruded strand into sticks approximately 30 inches long and then dropping those sticks onto the in-feed conveyors. As the leading edge of the stick arrives at the cutter knife the first grain is cut to a random length. Thereafter grain length is controlled by the

tractor feed speed. However, the last 2 to 3 inches of the stick is not well gripped by the tractor feed belts and is erratically cut. This flaw was responsible for approximately 50% of the reject grains. The remainder of the rejects was cut grains that rebounded off the walls of the collection chute and back into the path of the rotating cutter bar. We believe this was a result of a design fault in the cutter head made worse by the fact we were cutting strand of 0.430 in diameter when the design maximum for the cutter was 0.360. Initially almost 50% of the grains were being slashed, chipped or dented from impacting the cutter bar. After some modifications, the amount of damaged grains decreased to less than 10%. We could not alleviate the problem entirely.

5.1.1.2.4 Grain Lots IH94000E-EX99-0088 and IH94000E-EX99-0089. During the first extruder run, a total of 237 lb of grains were produced using paste Batch 486/98. During the second extruder run, an additional 20 lb of grains were manufactured using the remaining paste from Batch 486/98. Grains manufactured from Batch 486/98 from each of the two runs were blended, sorted, graphite glazed and re-designated as propellant Lot IH94000E-EX99-0089. The sorting yielded 212 lb out of the 257 lb produced for a reject rate of 17%.

Similarly for paste Batch 485/98, 229 lb of grains were produced from the first run and 267 lb were produced from the second run for a total of 496 lb. Again grains manufactured from Batch 485/98 from each of the two runs were blended, sorted, graphite glazed and re-designated as propellant lot IH94000E-EX99-0088. After sorting we yielded 433 pounds. The slightly lower reject rate of 13% is probably due to a significant improvement in pelletizer reliability during the processing of paste Batch 485/98.

5.1.1.2.5 Rework and Waste Generation Rates From TSE Operation. As part of the CLEVER process, the twin-screw extruder is incapable of making scrap propellant. This statement requires some explanation. The actual propellant is manufactured in the form of a powder paste during the Bofors Precipitation Process and it's chemical composition is fixed at this point and, thus, can not be altered by further processing steps. In the twin-screw extruder, a mixed solvent (70% ethyl acetate/ 30% ethanol) is added to the propellant powder paste. The extruder re-solvates the dry powder and mixes it into pliable dough that is extruded to shape through a die. The resultant propellant strand is then pelletized to the desired grain length and dried. There are four situations during which non-conforming material can be produced. They are:

1. The extruder is run solvent rich during start-up producing very soft dough. The feed rate of the propellant powder paste is increased until the strand exiting the die reaches the desired consistency to be handled and pelletized. During this start up process, called ramp up, the strand is too soft to pelletize and is therefore diverted. This material can be recycled back into the Bofors Precipitation Process and is actually rework material not scrap.

2. Either the loss-in-weight solids feeder used to feed the propellant powder paste or the pump used to feed the solvent mixture fail to hold set point. The resulting strand is the wrong consistency and must be diverted until the process is back under control. This material can also be recycled as rework material as in the preceding paragraph.

3. A malfunction with the cutter can cause the strands to be cut into dimensionally non-conforming grains. Once again this is rework material that can be recycled back to the

dissolver.

4. Prior to turning off the extruder, the powder paste feed rate is decreased and the process is run solvent rich to soften the material along the extruder screws and in the die to make cleanup easier. This process is called ramp down. The strand exiting the die during ramp down is too soft to pelletize and is once again diverted. The material produced during the ramp down procedure is also rework material that can be recycled back to the dissolver.

The primary waste stream of the part of the CLEVER process starting at the twin-screw extruder and ending with finished grains is the processing solvent added to the twin-screw extruder. During steady state operation the propellant strand exiting the extruder contains 12% processing solvent that is a 70/30 blend of ethyl acetate and ethanol. On a dry propellant powder weight fed basis, this is 13.63 lb of solvent per 100 lb. Using the lb/100 lb method of calculating normalizes the numbers with respect to throughput making it easier to scale between various extruders and throughput rates.

As the strand exits the die it is immediately transported in water to the pelletizer. Contact time with the ambient temperature water is approximately three minutes. During this time a very small percentage of the processing solvent is leached into the conveying water. Given that the concentration of solvent in the strand and in the water changes very little over the short contact period, the mass transfer problem is fairly simple. We conducted a solvent leaching test to determine the rate at which water leaches the solvent out of EX 99 Lova grains. A report is included in Appendix B. The solvent concentration in grains of the same size and initial solvent concentration of 12% dropped to 8% after being immersed for 14 hours. From the data generated by this test, it appears that the mass transfer to the water is controlled by the diffusion rate of solvent within the grain and is fairly constant. If we take this apparent average rate of 0.0047 lb solvent/100 lb propellant-minute and apply it to our situation, we calculate a loss of 0.014 pounds of solvent into the water per 100 lb of paste processed. This amount is negligible. In a more sophisticated computer analysis, Washington Group Inc. (WGI, the contractor hired by IHDIV, NSWC to design and build a scaled up twin-screw extruder facility) used the above data to determine how much solvent would leach into the conveying water. They calculated that for an 88-mm extruder processing 3000 lbs of propellant per shift, 1.08 lbs of solvent would be lost into the conveying water. When scaled to our size extruder, this equates to 0.036 lbs of solvent per 100 lbs of propellant processed. This estimate was a very conservative since WGI multiplied the expected mass transfer rate by a factor of 10. This result confirms our estimate. A summary of their analysis is included in Appendix B. Given these numbers, 99.7% of the process solvent mixture is lost to the atmosphere during the drying process.

Another source of waste is the solvent emissions from the material that is diverted during ramp up and ramp down. While the diverted propellant can be recycled as rework to the dissolver at the head end of the CLEVER process, it must be dried first. Solvent wet rework cannot be added to the dissolver. The dissolver uses 100% ethyl acetate as the active solvent and adding solvent wet rework from the extruder, which contains ethanol, would rapidly degrade the dissolver's efficiency. During the problem plagued run, IH94000E-EX99-0081, we ramped up ten times and ramped down seven times giving us a good basis from which to calculate the amount of paste and solvent used. The average ramp up used 5.98 lb of propellant paste and 0.95 lb of

solvent. The average ramp down used 3.15 lb of propellant paste and 0.6 lb of solvent. On a lb/100 lb basis, this is 15.88 lb and 19.05 lb, respectively, of solvent per 100 lb of paste used for ramp up and ramp down.

The last waste stream generated is during clean up of the twin-screw extruder. At the end of the run the powder paste feed rate is ramped down as previously explained and then shut off. The die holder door is then detached from the extruder barrel and the extruder is run until any remaining bulk powder is expelled. The extruder is then run for approximately 30 minutes while a mixture of process solvent and oatmeal is fed to it. This procedure purges any residual material from the extruder and helps clean the screws and barrel. The feed rates for the solvent and oatmeal naturally depend on the size of the extruder. For our 40-mm extruder, we use process solvent at a rate of 2 l/hr and oatmeal at a rate of 4 lb/hr. The purge procedure normally results in three to four pounds of Lova and solvent contaminated oatmeal that must be disposed of as explosive scrap. Additionally, another three pounds of solvent was used for both cleaning the die and general cleanup. The overall mass balances for the two runs are summarized in Figures 32 and 33. The complete balances for each run in spreadsheet format are included in Appendix B.

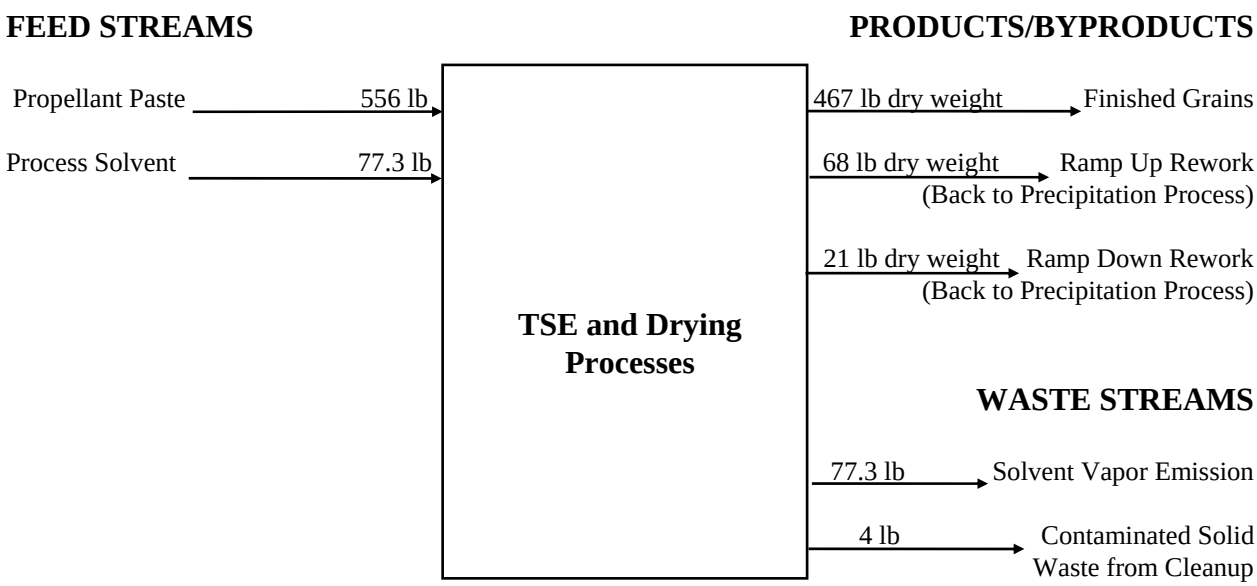
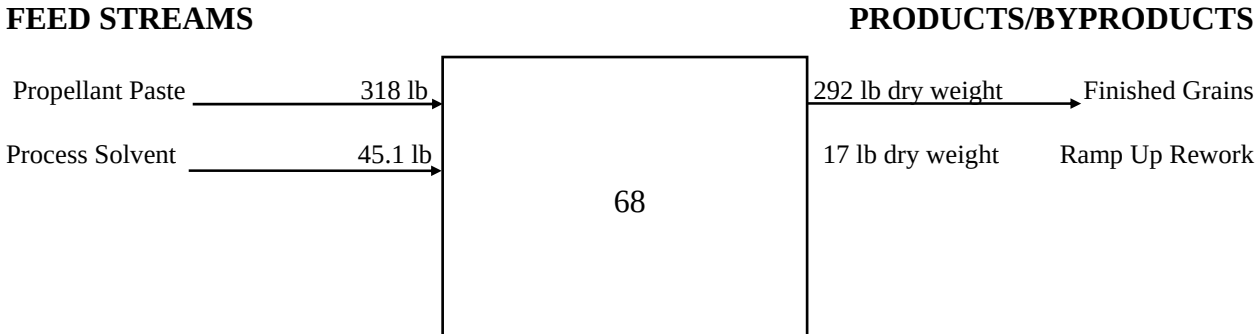


Figure 32: Material Balance for Extruder Run IH94000E-EX99-0081



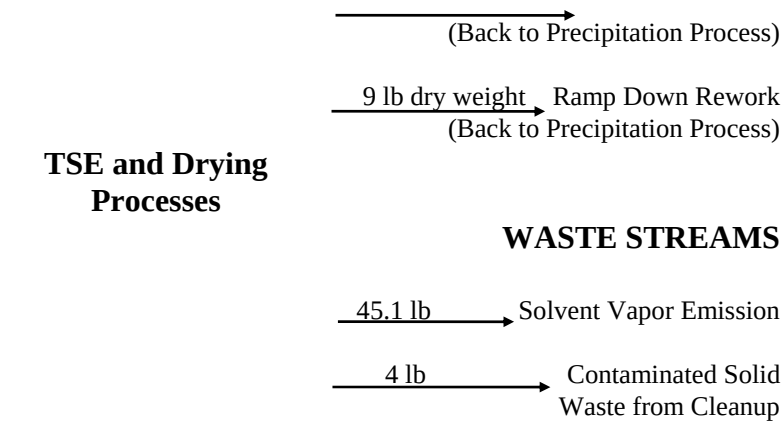


Figure 33: Material Balance for Extruder Run IH94000E-EX99-0083

5.1.2 Propellant Paste Data and Analysis

5.1.2.1 Paste Chemical Composition. The chemical analysis of paste Batches 484/98, 485/98 and 486/98 as reported by Bofors are presented in Table 13.

Table 13: Bofors' Analytical Results of Paste Batches

Analysis	Batch 484/98 Result	Batch 485/98 Result	Batch 486/98 Result	Requirements
Composition				
RDX/HMX, %	75.0	74.5	75.1	76.0+/-1.5
CAB, %	12.8	12.7	12.8	12.0+/-1.0
Ethyl Centralite, %	0.4	0.4	0.4	0.4+/-0.1
BDNPA/F, %	7.6	8.0	7.5	7.6+/-1.0
NC, %	4.2	4.4	4.2	4.0+/-0.5
HMX, %	2.2	2.5	2.5	<5
Volatile matter, %	0.10	0.10	0.15	<0.5
Through US sieve 10, %	100	100	100	100
Vacuum stability 100˚C, 40 h, ml/g	0.16	0.16	0.16	<3
Malvern, RDX, d_{50}, µ	3.70	3.40	3.15	<6.5
Malvern, RDX, 16µ, %	99.5	99.8	99.9	>90
Heat of explosion, cal/g	953	952	959	954+/-30

It should be noted that what Bofors lists as the compositional requirements were only guidelines, as the specification for EX 99 gun propellant was not finalized at the time of this demonstration. Bofors' paste was one percent lower in RDX and 0.8 percent higher in CAB than the nominal specification values. However, the batch-to-batch variation demonstrated is well within the specification tolerances for each component. The process is fully capable of meeting the compositional requirements of the specification. Chemical analysis and heat of explosion testing of Batches 485/98 and 486/98 conducted at IHDIV, NSWC were in substantial agreement with

Bofors' data. The IHDIV, NSWC analyses of the paste batches are included in Appendix B.

5.1.2.2 Thermal Stability. All three batches were analyzed for thermal stability. The lowest onset temperature observed was 213°C. This onset temperature is well above our maximum processing temperature of 45°C in the twin-screw extruder. Both the onset and peak temperatures for each batch are given in Table 14. Figure 34 is the DSC plot for batch 485/98 and is representative of all three batches.

Table 14: Paste DSC Results

Temperature	Batch 484/98	Batch 485/98	Batch 486/98	Requirement
Onset, ºC	214	215	213	>> 45
Peak, ºC	240	237	238	>> 45

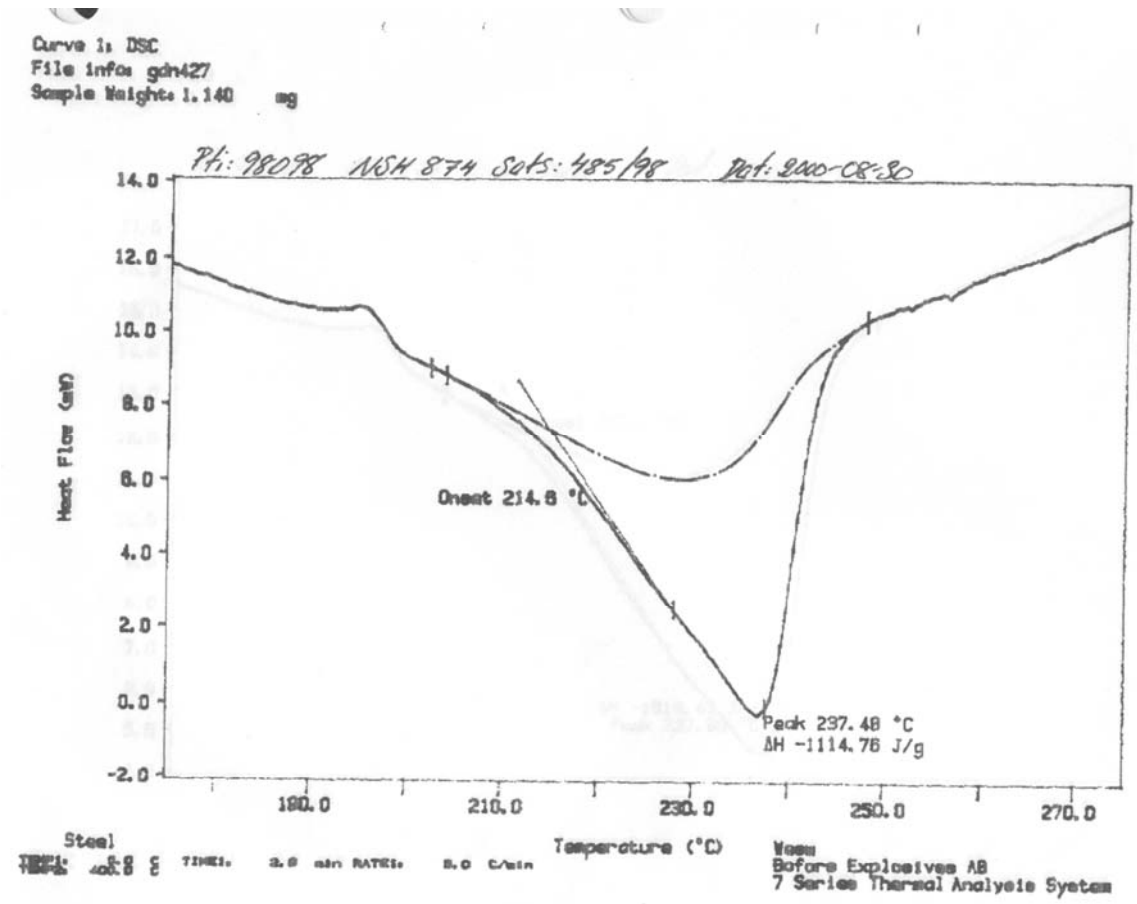


Figure 34: DSC Trace for Paste Batch 485/98

5.1.2.3 RDX Particle Size. The average RDX particle size of the batch processed material is typically 4-6 µ. This particle size is based on that obtained during the manufacture of over 2,000,000 pounds of Lova nitramine gun propellant via the batch process. The RDX particle size distribution for the three paste batches as reported by Bofors is presented in Table 15. The apparent decrease in mean particle size from the first to the last batch has no ready explanation

from their process records nor does it seem to correlate with other propellant properties. The reported difference maybe within the analytical error.

Table 15: RDX Particle Size

RDX particle size by Malvern	Batch 484/98	Batch 485/98	Batch 486/98	Requirement
d_{50}, μ	3.70	3.40	3.15	< 4
Less than 16μ, %	99.5	99.8	99.9	> 99

5.1.2.4 Microscopic Analysis for Free RDX. Paste samples from both Batches 485/98 and 486/98 were examined for free RDX, defined as uncoated RDX crystals. The samples were examined both by scanning electron microscopy and polarized light microscopy. While estimating the degree of coating is somewhat subjective, our senior microscopist estimates, based on his experience and observations, that less than 0.001% was free uncoated RDX. As can be seen from Figure 35, the paste is well agglomerated. The report and the photomicrographs are included in Appendix B.

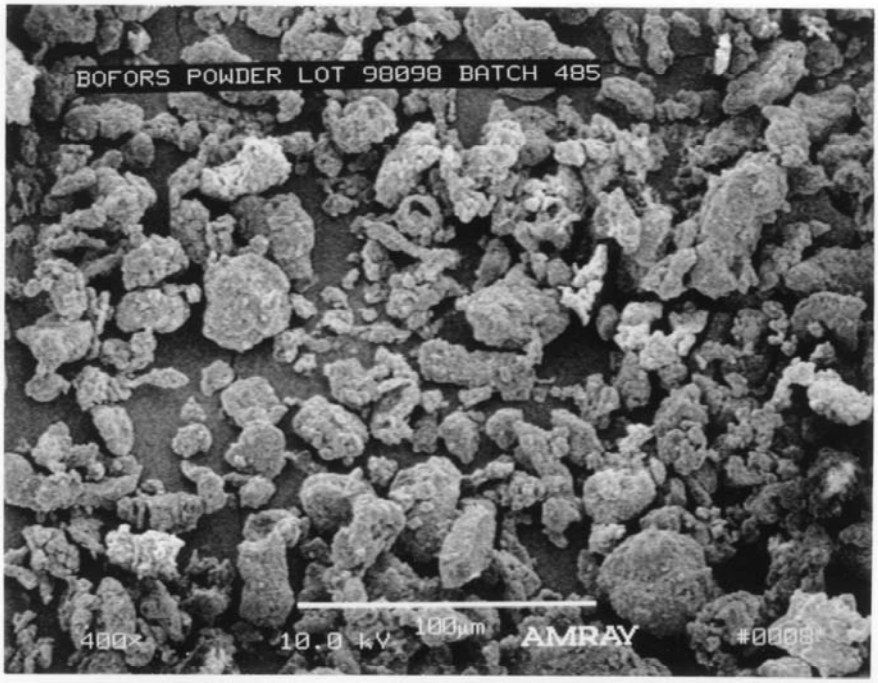


FIG. 1
BOFORS POWDER LOT 98098, BATCH 485, 400X

Figure 35: Photomicrograph of Paste Batch 485/98

5.1.2.5 Small-Scale Safety Testing. A sample from Batch 484/98 was subjected to NOS

impact, ABL friction and ABL electrostatic discharge (ESD) testing. The results along with those for both a RDX Class A standard and a M43 Lova paste produced at IHDIV in 1993 by a different process (i.e. water extraction process) are presented in Table 16.

Table 16: Paste Small-Scale Safety Test Results

Sample	NOS Impact 50% hgt. mm	ABL Friction 20 TIL psig	ABL ESD 20 TIL joules
Batch 484/98	267	420	0.037
RDX A standard	232	135	0.037
1993 Lova preblend	225	980	0.125
Requirement: Less sensitive than RDX A standard	Yes	Yes	Yes

The impact and ESD sensitivity of the paste made by the Bofors Precipitation Process is essentially that of RDX Class A. This fact makes sense since the paste is nominally 76% RDX. As would be expected, due to the smaller RDX particle size and the high degree of RDX coating (shown by the microscopy work), Batch 484/98 is less sensitive to friction than the RDX standard. Differences in the reported sensitivity between Batch 484/98 and 1993 M43 Lova paste are attributed to two factors. The first factor is the particle size difference between the M43 Lova paste made in 1993 and Batch 484/98. The IHDIV 1993 material was the consistency of sand while Batch 484/98 was closer to flour. It is estimated that there was probably an order of magnitude difference in the particle size for the two pastes. This would account for the difference in ESD and friction sensitivity. The second factor is the test equipment was refurbished since 1993, therefore, some of the difference may just be test bias. No material was retained from the IHDIV 1993 M43 Lova batch so we could not test it concurrent with Batch 484/98. Based on the results of the sensitivity testing, the Bofors precipitated paste was judged to be at least as safe to handle as the ground RDX we use in the batch process.

5.1.2.6 Paste Small-Scale Shock Sensitivity. Batch 484/98 was also subjected to card gap and detonation cap testing. The test report is included in Appendix B. Three card gap tests were conducted with 70 cards separating the paste sample and the pentolite booster. The witness plate was only dimpled in all three tests. This result is clearly a negative test or a Class 1.3 material. However, when we conducted the cap test during which the paste sample is subjected to the output of a blasting cap, we got two consecutive positive tests as evidenced by mushrooming of the lead witness cylinders. This result was a clear indication of the paste detonating and, therefore, the paste was classified as a Class 1.1 material. A positive test in either test results in a Class 1.1 classification. This result was not unexpected.

5.1.3 Propellant Grain Test Data and Analysis

5.1.3.1 Safety Testing. The safety tests are the same tests conducted on the paste, however, in this case a grain is crushed and 35 milligrams of the fragments are used for each test. Grains from Lot IH94000E-EX99-0088, made from paste Batch 485/98, were used for this and all subsequent propellant grain testing. The results are presented in Table 17. The test report is included in Appendix B.

Table 17: Grain Safety Test Results

Sample	NOS Impact 50% hgt. (mm)	ABL Friction 20 TIL (psig)	ABL ESD 20 TIL (joules)
CLEVER processed EX 99 Lova Lot IH94000E-EX99-0088	277	980	0.095
RDX Class A Standard	395	180	0.095
Typical conventional batch EX 99 Lova	230	980	0.095
Requirement: Equal to or less sensitive than conventional batch EX 99 Lova	Yes	Yes	Yes

Once again the results are similar to those obtained on the propellant paste and on the batch processed propellant. The impact numbers are somewhat lower than the RDX A standard and slightly higher than batch Lova. Given that the individual test results rely on operator interpretation and are subject to some variation especially when comparing testing done at different times, the reported differences are probably not significant.

5.1.3.2 Small-Scale Shock Sensitivity. Three propellant grain samples were tested at 70 cards. In all three cases, the witness plates were only dented or torn meeting the first criteria for a 1.3 hazard classification. Five additional samples, three for the test and two spares, were prepared for the cap test. The first three tests were negative meeting the requirement for a 1.3 hazard classification. At this point the test technician made the decision to fire the remaining two spare test samples. The fourth shot was also negative but the final shot resulted in a positive deformation of the lead witness cylinder as shown in Figure 36.

Figure 36: Cap Test Failure

Given the positive test indication, we were initially concerned that either the Bofors Precipitation Process or the TSE process had resulted in a more sensitive EX 99 Lova grain. However, after researching the matter, we found that other batch processed Lova lots had also failed the cap test. Based on an analysis by an IHDIV detonation physicist, this requirement was waived for Lova propellants in the larger grain sizes. This analysis concluded that Lova grains in our size range could have web thicknesses that were greater than the minimum critical diameter making it possible to drive the grain to detonation. However, given the random orientation of grains in a packed bed (as found in storage containers or shell casings) and the resulting small contact area between grains, sympathetic detonation from grain to grain was considered extremely unlikely. When researched how the test was performed, we discovered that the grains had been carefully stacked in the cardboard tube as shown in Figure 37 rather than poured in to randomly fill the tube.

Figure 37: Cap Test Configuration

This ordered orientation dramatically increased the grain-to-grain contact area, particularly from end to end. This test configuration makes the possibility of sympathetic detonation from grain-to-grain much more likely. Given these facts, we conclude that the one positive result out of five test shots was attributable to the test configuration used and not related to the CLEVER process. The test reports and the data analysis are provided in Appendix B.

5.1.3.3 Ballistic Impact Chamber Test. Samples from batch processed Lot IH2309DEX-99-FB01 and the CLEVER Lot IH94000E-EX99-0088 were both tested as described in Section 4.4.2.3 and reference 11. The reaction sensitivity for a material is proportional to the inverse of the initial reaction times. Test results of 10.2 (10^{-6}s) for Lot IH2309DEX-99-FB01 and 11.56 (10^{-6}s) for CLEVER Lot IH94000E-EX99-0088 indicate that these lots are essentially equal in sensitivity. The test report is provided in Appendix B.

5.1.3.4 High Rate Mechanical Properties. Grains from the batch processed EX 99 Lot IH2309DEX-99-FB01 and the CLEVER processed Lot IH94000E-EX99-0088 were both subjected to the testing. As expected both lots exhibited brittle fracture of the grains similar to that previously reported with M43 Lova. However, at both ambient and cold temperatures the CLEVER processed grains withstood higher stress levels prior to fracture as seen in Figure 38.

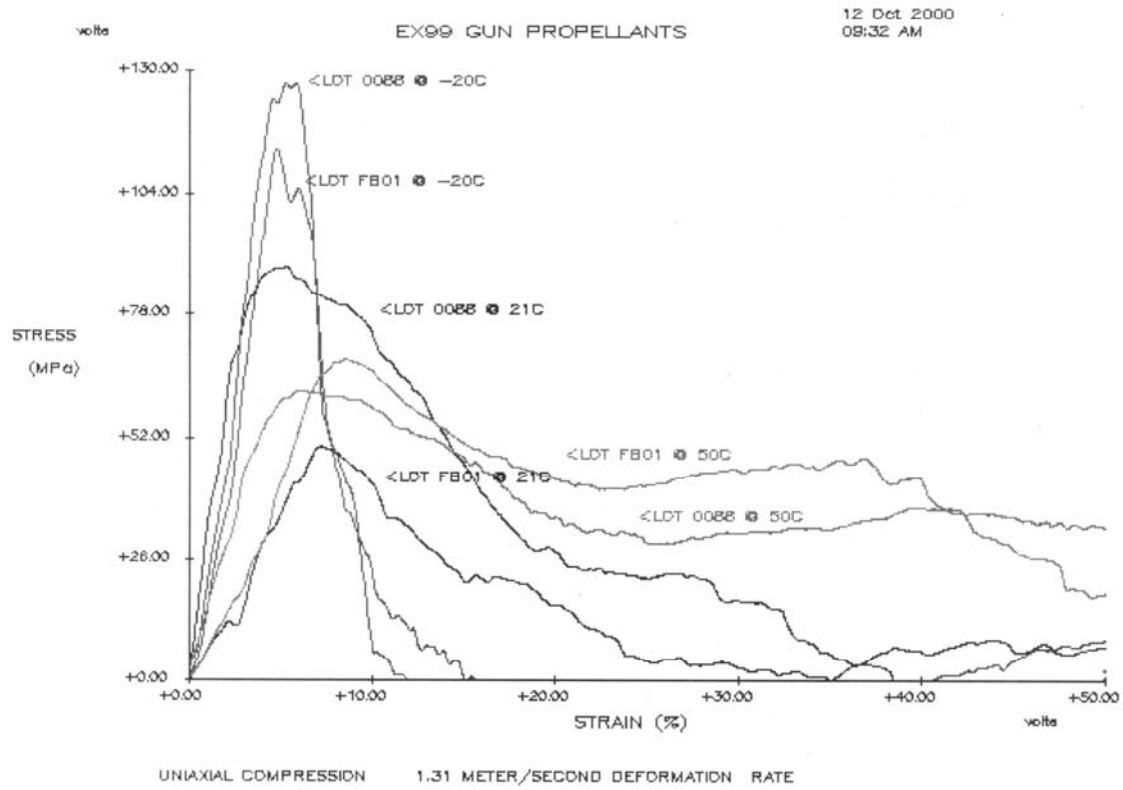


Figure 38: Grain Mechanical Properties

Photomicrograhic examination of the fracture zones showed a much better binder to filler interaction for the CLEVER processed grains as compared to the conventional batch processed grains. The higher fracture resistance was attributed to this observation. This data also agrees with the results of the scanning electron microscopy (SEM) and polarized light microscopy analysis that reported a very high degree of RDX particle coating.

5.1.3.5 Dimensional Analysis of Grains. The dimensional characteristics that most effect propellant ballistic performance are grain diameter, perforation diameter, inner web thickness and outer web thickness. The average dimensions for these characteristics are controlled by the tooling (die and pin plate). However, as one might expect, the variability is also affected by processing and can best be quantified by the standard deviations. Processing factors that can affect grain uniformity are solvent content, extrusion rate and even the orientation of the die (vertical as in the batch process vs. horizontal as in the TSE process). The twin-screw extruder allows for tighter control of the solvent content and a slower extrusion rate. These advantages over the batch process translate into less variable die swell and a more uniform strand going to the cutter. Additionally, the batch presses are vertical and the weight of the hanging strand can result in necking of the strand at the die. The twin-screw extruder extrudes the propellant strand in a horizontal plane and, therefore, is not susceptible to the problem. Standard deviations for a batch processed EX 99 Lot IH23000GEX99-0009 (a representative batch lot) and both CLEVER lots are presented in Table 18.

Table 18: Grain Dimensional Variation

Propellant Grain Lot	Diameter Standard Deviation	Perforation Standard Deviation	Inner Web Standard Deviation	Outer Web Standard Deviation
IH94000E-EX99-0088	0.003	0.001	0.002	0.004
IH94000E-EX99-0089	0.005	0.002	0.004	0.006
IH23000GEX99-0009	0.005	0.002	0.066	0.014
Requirement: Less than or equal to conventional batch standard deviation	Yes	Yes	Yes	Yes

All three lots were 0.4 inch nominal diameter, seven perforation grains. A larger data base on CLEVER processed lots than we currently have is required in order to make a definitive statement, however, the data suggests that CLEVER processing may have a distinct advantage over batch processing for grain uniformity. Smaller standard deviations of the web thickness result in a more uniform burn of the propelling charge.

5.1.3.6 Scanning Electron Microscope Analysis. Grains from each of the 12 sets of process conditions tested during the pre-demonstration DOE study were evaluated along with two representative batch processed EX 99 lots. The system developed in 1993 for scoring grain microstructure of M43 propellant grains gave a rating from 1 to 9 with 1 being the best. The rating was based on RDX rich regions, binder rich regions and porosity. Using this standard the CLEVER propellant samples score a 3 as compared to a 5 for the batch lots. Later the standard was expanded to include particle size distribution, voids, large particle content and particle coating. The ranking was change to a 1 to 5 scale. Under this expanded criteria, both the CLEVER and batch grains scored a 1. This score is satisfactory. The factor that evened the scoring was that the average RDX particle size for the CLEVER Lova is smaller resulting in more surface area for the binder to coat and subsequently thinner coating of the RDX particles. The microstructure of the CLEVER processed grains is at least as good if not better than batch produced grains. The SEM report is included in Appendix B.

5.1.3.7 Heat of Explosion. The heat of explosion (HOE) for Lot IH94000E-EX99-0088 was measured as 932 cal/g and 939 cal/g for Lot IH94000E-EX99-0089. These results correlate with the HOE values for paste batches 485/98 and 486/98, these values were 954 and 943 cal/g respectively. The fact that the HOE values for the paste are slightly higher than the finished grains is a phenomenon we have seen before. It appears to be related to a slight difference in how the grains combust in the calorimeter verses fine powders and not a physical effect of being processed through the twin-screw extruder. Both propellant grain lots were below the minimum specification value of 945 cal/g. This was expected since both paste batches were slightly low on RDX content. Additionally, the RDX that Bofors uses contains little or no HMX as a contaminant. US RDX contains 12% HMX on average. HMX has a higher heat of formation than a RDX and this would result in a higher heat of explosion for the mixture. Thus, since Bofors RDX has little or no HMX, it is not surprising that the HOE is lower. Ultimately, the

HOE can be easily adjusted for in future paste batches if necessary by adjusting the formulation within the specification range to aim for a slightly higher target HOE. The lot-to-lot variation was well within the specification requirement of +/-10 cal/g.

5.1.3.8 Propellant Density. The propellant density for the two propellant grain lots was 1.652 g/cc for Lot IH94000E-EX99-0088 and 1.657 g/cc for Lot IH94000E-EX99-0089. This is a tight distribution well within the specification limits of 1.63-1.68 g/cc.

5.1.3.9 Propellant Chemical Composition. The chemical composition of the paste is fixed during the Bofors Precipitation Process. The twin-screw extruder only resolvates the paste and extrudes the final propellant grain shape and cannot change the chemical composition. However, a chemical analysis of Lot IH94000E-EX99-0088 was conducted as a double check on the paste chemical composition analysis. As can be seen from the data in Table 19, the paste and propellant grain compositions are within the specification requirement except for the high Ethyl Centralite weight percent of the propellant grain which is believed to be an anomaly because the formulation is not altered in the twin-screw process.

Table 19: Paste and Grain Chemical Composition Comparison

Ingredient	Paste Batch 485/98	Grain Lot IH94000E-EX99-0088	Propellant Specification Requirement
RDX/HMX, %	74.5	74.9	76.0+/-1.5
CAB, %	12.7	12.8	12.0+/-1.0
Ethyl Centralite, %	0.4	0.73	0.4+/-0.1
BDNPA/F, %	8.0	7.6	7.6+/-1.0
NC, %	4.4	4.65	4.0+/-0.5

5.1.3.10 Variable Confinement Cookoff Test. Grains from Lot IH 94000E-EX99-0088 were used for the testing. Four tests were conducted as described in Section 4.4.2.10 and reference 13. All four were classified as burning reactions, the mildest of the six types of reaction. Grains from Lot IH23099DEX-99-FB01, a batch processed EX 99 Lova lot, were tested as a control. This testing resulted in three burning reactions and one pressure burst reaction. Both lots passed the test criteria, no reaction more severe than a pressure burst reaction. A full test report is included in Appendix B.

5.1.3.11 Wide Angle X-ray Diffraction Analysis. Grains from both the CLEVER Lot IH94000E-EX99-0088 and the batch Lot IH23099DEX-99-FB01 were shipped to Stevens Institute of Technology where they were examined using wide angle x-ray diffraction analysis and scanning electron microscopy (SEM). X-ray diffraction analysis showed both the CLEVER grains and the batch grains to be well mixed with mixing indexes of 0.997 and 0.994, respectively. These numbers are so close that the apparent difference may be due to RDX particle size distribution differences or minor differences in composition. With an index of 1 being perfect mixing, these numbers have to be considered the same given the unknowns in this relatively new technique that is still being refined. The SEM results showed no anomalies, porosity, voids or binder rich areas in the grains and support the IHDIV, NSWC SEM conclusion

that the RDX is well coated and that the particle size distribution is smaller in the CLEVER Lova which uses precipitated RDX instead of the ground RDX used in the batch formulation. The complete test report is included in Appendix B.

5.1.3.12 High Pressure Closed Bomb Testing. The primary reason for the high pressure closed bomb firings was to provide the ballisticians with data required to determine the propellant charge weights for the gun firings. Both CLEVER lots were found to be satisfactory for gun testing to proceed. Requirements based on the closed bomb firings have not been determined for EX 99 Lova as these values are normally set using production data. However, the summary data is presented in Tables 20 and 21 for both CLEVER lots. The within lot and lot-to-lot variability are very good especially for a process that has not been optimized in production. The full test report with graphs is included in Appendix B.

Table 20: High Pressure Closed Bomb Data Summary for Lot IH94000E-EX99-0088

Burning Rate, dP/dt, and Vivacity Summary Sheet
Type: Twin Screw EX99
Lot: IH940-00E-EX99-0088
Charge Wt: 75.0 grams
Igniter: 1 gram 700X
Bomb Volume: 198.88 cc

Pressure (psi)	10000	15000	20000	25000	30000	35000	40000	50000	60000	Pmax
Burning Rate (in/sec)										
28JUN02	1.34	1.75	2.34	2.84	3.45	4.03	4.61	5.75	6.94	96772
28JUN03	1.22	1.79	2.30	2.81	3.44	3.96	4.69	5.78	6.95	97397
28JUN04	1.23	1.76	2.32	2.90	3.48	4.01	4.62	5.78	6.95	97189
Average	1.26	1.76	2.32	2.85	3.46	4.00	4.64	5.77	6.95	97119
dP/dt (kpsi/sec)										
28JUN02	1801	2590	3656	4752	6065	7472	8960	12201	15883	
28JUN03	1704	2645	3624	4759	6060	7386	9067	12275	15950	
28JUN04	1698	2607	3666	4817	6117	7434	8985	12273	15920	
Average	1734	2614	3649	4776	6081	7431	9004	12250	15918	
Vivacity (100/(psi*sec))										
28JUN02	0.186	0.178	0.189	0.196	0.209	0.221	0.231	0.252	0.274	
28JUN03	0.175	0.181	0.186	0.195	0.207	0.217	0.233	0.252	0.273	
28JUN04	0.175	0.179	0.189	0.198	0.210	0.219	0.231	0.253	0.273	
Average	0.179	0.179	0.188	0.188	0.209	0.219	0.232	0.252	0.273	

Table 21: High Pressure Closed Bomb Data Summary for Lot IH94000E-EX99-0089

Burning Rate, dP/dt, and Vivacity Summary Sheet
Type: Twin Screw EX99
Lot: IH940-00E-EX99-0088
Charge Wt: 75.0 grams
Igniter: 1 gram 700X
Bomb Volume: 198.88 cc

Pressure (psi)	10000	15000	20000	25000	30000	35000	40000	50000	60000	Pmax
Burning Rate (in/sec)										
28JUN02	1.70	1.80	2.35	2.91	3.49	4.08	4.72	5.90	7.06	97439
28JUN03	1.70	1.75	2.29	2.93	3.50	4.13	4.76	5.95	7.10	96814
28JUN04	1.69	1.82	2.37	2.94	3.51	4.12	4.77	5.96	7.11	96855
Average	1.70	1.79	2.34	2.93	3.50	4.11	4.75	5.94	7.09	97036
dP/dt (kpsi/sec)										
28JUN02	2483	2667	3724	4850	6153	7551	9139	12500	16153	
28JUN03	2467	2588	3640	4848	6164	7612	9171	12552	16204	
28JUN04	2465	2686	3714	4886	6159	7612	9197	12557	16234	
Average	2472	2647	3693	4861	6159	7591	9169	12537	16197	
Vivacity (100/(psi*sec))										
28JUN02	0.182	0.182	0.191	0.199	0.210	0.221	0.234	0.257	0.276	
28JUN03	0.182	0.178	0.188	0.188	0.212	0.225	0.237	0.259	0.279	
28JUN04	0.182	0.185	0.192	0.192	0.212	0.225	0.237	0.259	0.279	
Average	0.182	0.182	0.190	0.190	0.212	0.224	0.236	0.258	0.278	

5.1.3.13 Gun Firings. In August 2000, both CLEVER processed grain lots (IH94000E-EX99-0088 and IH94000E-EX99-0089) were successfully fired in the 5"/54 gun and equaled or exceeded the performance of the batch processed grain lot IH23099DEX-99-FB01, the control sample. The full test report is provided in Appendix B. Maximum piezoelectric pressure (ksi) reading for each gauge location, muzzle velocity (ft/sec), ejection time (ms), and energy (MJ) are shown in Table 22.

Table 22: Gun Firing Results

Round #	Muzzle Velocity (fps)	M11 Average (ksi)	P3=35" (ksi)	P4=35" (ksi)	P5=92" (ksi)	P6=92" (ksi)	P18=308" (ksi)	Eject Time (ms)	Comments	Energy (MJ)
Practice Round (SPCF-11247)										
1	2712		46.30	45.85	35.79	35.78	6.99	19		10.85
Lot 88 (CLEVER Process)										
2	2453	43.1	42.06	41.07	36.47	39.10*	12.88	24	*spiked	13.97
3	2578	49.7	48.46	46.90	46.71*	44.74*	12.58	834	*spiked	15.40
4	2729	58.1	57.58	55.48	48.66	48.67	12.41	21		17.28
5	2793	62.5	62.74	60.17	51.40	52.09	12.45	20		18.11
6	2773	60.5	60.99	57.94	50.78	51.12	12.34	404		17.89
7	2788	62.4	62.10	58.26	53.07	55.06	12.31	21		18.03
8	2785	61.7	62.11	58.02	54.94	56.30	12.30	355		18.03
9	2785	61.6	60.00	57.45	50.53	54.48	11.87	21	no trigger, #s from oscilloscope	17.98
Lot FB01 (Batch Process)										
10	2743	62.5	62.40	60.47	59.87	52.12	11.85	240		17.44
11	2748	62.7	63.80	61.52	50.67	51.64	12.44	409		17.55
Lot 89 (CLEVER Process)										
12	2619	51.1	50.26	48.47	41.90	42.99	12.15	23		15.93
13	2805	63.4	63.42	60.89	53.47	52.34	12.26	20		18.24
14	2811	64.3	64.45	61.67	51.82	58.06	12.44	20		18.35
15	2816	65	65.29	62.73	58.03	60.68	11.84	21	5+ min. outside cond. temp	18.41
16	2815	64.3	64.27	61.91	61.23	57.69	12.25	262		18.38
17	2782	61.4	61.73	58.87	53.94	57.56	12.55	21		17.95

The propellant charge was loaded to 26 pounds in order to produce 18 MJ of energy and keep the breech pressure at or below 65,000 psi. Table 23 shows the results of only the 26-pound loaded charges. This was the maximum propellant weight available before configuration modifications would have to be made to the propellant charge. These data also show that while using the proper propellant weight of 24.8 pounds for the standard batch propellant (FB01), it produced a lower amount of energy than the continuously processed (26 pounds) even though it produced the same breech pressure.

Table 23: Data for the 26 lb Propellant Charges

Round #	Muzzle Velocity (fps)	Breech Pressure (kpsi)	P3=35" (kpsi)	P4=35" (kpsi)	P5=92" (kpsi)	P6=92" (kpsi)	P18=308" (kpsi)	Ejection Time (ms)	Energy (MJ)	Seating Distance (in)
Lot 88 (CLEVER Process)										
5	2793	62.5	62.74	60.17	51.40	52.09	12.45	20	18.11	37.88
6	2773	60.5	60.99	57.94	50.78	51.12	12.34	404	17.89	37.81
7	2788	62.4	62.10	58.26	53.07	55.06	12.31	21	18.03	37.75
8	2785	61.7	62.11	58.02	54.94	56.30	12.30	355	18.03	37.81
9	2785	61.6	60.00	57.45	50.53	54.48	11.87	21	17.98	37.75
Average	**2784.80**	**61.74**	**61.59**	**58.37**	**52.14**	**53.81**	**12.25**	**164.20**	**18.01**	**37.80**
St. Dev.	**7.36**	**0.80**	**1.09**	**1.05**	**1.85**	**2.15**	**0.22**	**197.30**	**0.08**	**0.05**
Lot 89 (CLEVER Process)										
13	2805	63.4	63.42	60.89	53.47	52.34	12.26	20	18.24	37.88
14	2811	64.3	64.45	61.67	51.82	58.06	12.44	20	18.35	37.81
15*	2816	65.0	65.29	62.73	58.03	60.68	11.84	21	18.41	37.88
16	2815	64.3	64.27	61.91	61.23	57.69	12.25	262	18.38	37.88
17	2782	61.4	61.73	58.87	53.94	57.56	12.55	21	17.95	37.88
Average	**2805.80**	**63.68**	**63.83**	**61.21**	**55.70**	**57.27**	**12.27**	**68.80**	**18.27**	**37.86**
St. Dev.	**13.99**	**1.40**	**1.35**	**1.47**	**3.84**	**3.03**	**0.27**	**108.00**	**0.19**	**0.03**
Total										
Average	**2795.30**	**62.71**	**62.71**	**59.79**	**53.92**	**55.54**	**12.26**	**116.50**	**18.14**	**37.83**
St. Dev.	**15.28**	**1.48**	**1.65**	**1.92**	**3.41**	**3.07**	**0.23**	**158.16**	**0.19**	**0.05**

As shown in Table 21, the largest deviations are noticed in the muzzle velocity and ejection time. The data shows that a charge weight of 26.00 pounds produced an average breech pressure of 62.7 kpsi with a standard deviation of 1.48 and an average muzzle velocity of 2795 ft/sec with a standard deviation of 15.3. The energy ranged from 17.9 to 18.4 MJ.

Figure 39 shows the slight differences in pressure-time performance between the batch and twin screw processed propellant, as well as the differences between the two different lots of CLEVER processed propellant. The pressure versus time plots for all the firings are available in the full test report.

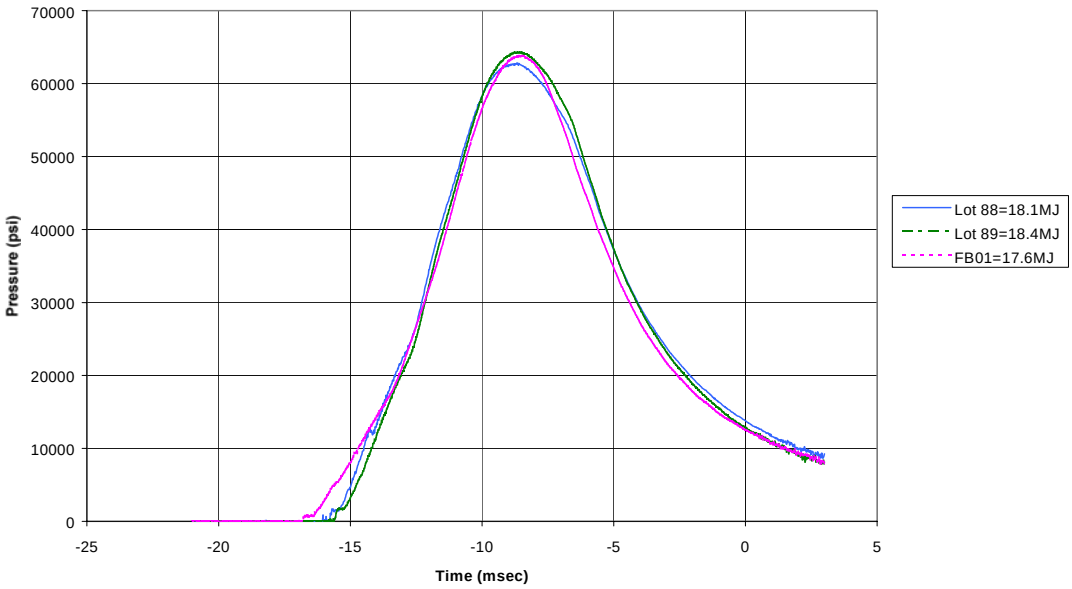


Figure 39: Pressure vs. Time Comparison

5.1.3.13.1 Conclusions. Overall, the twin screw processed EX99 propellant met expectations by consistently producing 18 MJ of energy while remaining at or below 65,000 psi with a charge weight of 26 pounds. This charge weight just filled the available cartridge case volume. For this particular test application (110 lb Hijack projectile seated at a nominal 37.8" seating distance), the granulation was nearly optimum. According to the pressure-time plots, Lot 89 does seem to reach a higher pressure and burn slightly faster, which is more efficient than Lot 88. The twin-screw processing team noted the following differences:

- Two different Bofors paste batches were used to make grain Lots IH94000E-EX99-0088 & IH94000E-EX99-0089.
- One production run had mechanical issues, which caused it to shut down several times. Even though the muzzle velocity seems to have a large overall standard deviation compared to the other parameters, it is within previous EX 99 batch processed results. The standard deviation for the individual lots was very good, and if these lots were blended, which is normal practice, the standard deviation should have been reduced.

The ejection time varied considerably. This is due most likely to the inconsistency currently seen with the EX170 primers. IHDIV, NSWC is researching this issue and is trying to solve the problem with various modifications to the existing configuration.

The gas blowback along the cartridge cases for these firings is consistent with previous results. The gun barrel thermal data for the CLEVER processed propellant is consistent with the batch processed propellant.

5.2 Data Assessment

The bulk of the specific comments were presented along with the actual data in section 5.1 as this seemed the most coherent way to address them. However, there are several general observations that while broad in scope are fairly important.

CLEVER processed grains were equal or superior to batch processed grains in all respects. This said, we have to qualify the statement to some extent. The CLEVER grains were made using brand new die tooling that was subjected to minimal wear and tear during the demonstration as compared to what production tooling experiences. The tighter dimensional tolerances exhibited by the CLEVER grains may reflect this fact. Another possible factor was that the process both at Bofors and at IHDIV, NSWC had a very high degree of hands on participation by senior engineers in lieu of normal production labor. Whether this helped or hurt grain quality is open to discussion.

Bofors did an excellent job of manufacturing the test batches but they did encounter significant processing problems that they solved on the fly. When we look at their yield and mass balance data we need to keep in mind that it is an average of only the last two batches and does not represent a process that was fine tuned over an extended production run.

IHDIV, NSWC experienced similar problems during the twin-screw extruder processing. The heart of the twin-screw technology, the extruder, worked flawlessly but we were plagued with mechanical problems with the strand conveying and cutting system. This equipment had been developed and tested during relatively short runs and was found lacking when it came to running for 10 hours without maintenance. As a result we had a lot of unplanned down time with multiple starts and stops during the first run. It was not until the second run that we obtained mass balance data representative of normal operations.

5.3 Technology Comparison

The only competing technology to the CLEVER process is the current batch Lova production process. These two processes can be compared in five main areas that are propellant grain quality, process yield, solvent emissions, scrap generation and complexity. These areas are discussed in the following sections.

5.3.1 Quality. The quality of Lova grains manufactured by the CLEVER process was equal to or better than batch processed grains in all areas. The uniformity of the three paste batches produced by the Bofors Precipitation Process was particularly encouraging considering that these were the first three batches made with this particular Lova formulation and solvent. The fact that the paste varied little as Bofors changed process parameters to solve the adhesion problem shows the process is robust and tolerant of upsets.

5.3.2 Process Yield. The best way to compare the two process yields is to look at their respective rework rates. The batch process has two main sources of rework material, which must be cycled back into a dedicated rework mix where it is resolvated. The first source is the batch pressing operation during which the propellant is extruded into strands of the proper geometry. On the average 7.6% of a mix can not be extruded and is retained as press heels which are reprocessed in a rework mix. The strands are then conveyed to a pelletizing operation where they are cut into grains. Typically an additional 10.4% of the mix is rejected at this point because the perforation are off center or the strands are damaged or have dried out to the point

they can not be cut. These reject strands are also recycled into a rework mix. This gives us a combined rework rate of 18% for the batch process.

The CLEVER process needs to be looked at as two sequential unit operations. The first operation is the Bofors Precipitation Process that manufactures the paste feedstock for the twin-screw extruder operation. Based on the average of the last two Bofors paste batches we had 22 kilograms of material per batch which needed to be recycled back into the dissolver. This equates to a rework rate for the demonstration lots of 5.5%, the first batch 484 is not included in this calculation since it was considered atypical because of the extensive problems during it's manufacture. As previously stated the process is still undergoing some changes to increase the particle size of the paste, it is estimated that the final rework rate will be under 2% when we have gained some production experience with the precipitation process. The second contributor is the twin-screw extruder process. For these runs the rework was defined as the sum of the startup and ramp down material that would normally be added to the dissolver for the next paste batch. Grains that were damaged by the pelletizer were not included in this calculation since we would not use this type of pelletizer in actual production. The first extruder run had a rework rate of 19%, however, this was the run where we had a lot of mechanical problems. Even with some problems the second extruder run had a rework rate of 8%. It is our estimate that rework generated by a twin-screw extruder production facility will be less than 4%.

The CLEVER process rework rate during the demonstration was 13.5% as compared to a batch rate of 18%. Given that there is a lot of room for improvement as we gain experience with the CLEVER process, we expect the rework rate to decrease to less than 6% in production.

5.3.3 Solvent Emissions. To compare the CLEVER and the batch processes we again need to look at them as two sequential unit operations. Since the process rates are also different we need to normalize the emissions to a pound per pound of product basis. In addition, for simplicity, the comparison was based on virgin material and did not include rework.

The batch process starts with the preparation of the RDX that involves drying and grinding it to the required particle size. During this operation 0.114 pounds of isopropanol per pound of RDX is lost as solvent emissions. Since Lova is 76% RDX this equates to 0.087 pounds per pound of Lova. The dried and ground RDX along with other ingredients is then charged to a mixer along with 0.3 pounds of mixed solvent (70/30 ethyl acetate/ ethanol blend). All this solvent is lost as emissions during the mixing and subsequent extrusion pelletizing and grain drying operations. This gives us a total of 0.387 pounds of solvent emitted per pound of propellant made.

For the CLEVER process we start with the Bofors Precipitation Process that emits 0.103 pounds of ethyl acetate per pound of paste. At steady state, the twin-screw process uses 0.136 pounds of mixed solvent, 70/30 ethyl acetate/ethanol blend, per pound of Lova. When this ratio is adjusted for solvent used during the ramp up and ramp down it increases to 0.154 pounds solvent emissions per pound Lova. The total for the CLEVER process as demonstrated is 0.257 pounds of VOCs per pound of Lova. This represents a 35% decrease over the conventional batch process. If Bofors' projection of 6% emissions proves true in production and the rework rate at the twin screw extruder can be reduced to 4%, which seems feasible, the projected VOC emissions drop to 0.204 pounds per pound Lova. This would increase the reduction in VOC emissions to 47%.

5.3.4 Scrap Generation. For the purpose of comparison, scrap was defined as any material that would be disposed of as hazardous explosively contaminated waste. Since everything in the plant is presumed to be explosively contaminated, this covers not only scrap propellant but also includes rags, solvent and other materials used to clean the process equipment. Once again to make the comparison quantities are normalized to a per pound of Lova basis.

For the batch process there are three main points of scrap generation. The primary source is cleanup of the mixer bowl that generates 0.019 lb per lb Lova of scrap propellant, rags and solvent. Another important source is the blocking and extrusion presses that generate an additional 0.01 lb per lb. Finally the pelletizing operations generate an additional 0.003 lb per lb of scrap for a total of 0.032 lb scrap per lb Lova. Not included in this total were scrape generated from die cleaning which is minimal. Another source that was not included was scrap from the RDX grinding operation since we did not have data on this.

For the CLEVER process the scrap generation is as follows. If we allocate the 22 kilograms per batch which Bofors cannot account for as scrap the rate per pound from the precipitation process is .058 lb per lb. If the process yield increases to 98% as expected this would drop to 0.02 lb per lb. During the twin screw extruder run we only had cleanup data available for the second run since the first was terminated by the extruder jam. Based on the second run we generated 7 pounds of scrap which was a combination of oatmeal and solvent, used to purge the extruder, Lova propellant and rags. On a per pound basis this amounted to 0.024 lb/lb Lova for a total of 0.082 for the CLEVER process.

While for the demonstration the scrap generation rate was significantly higher than the batch rate there is lots of room for improvement. If the 98% yield assumption for the precipitation process is correct the CLEVER rate drops to .044. Further more the twin-screw contribution is entirely dependent on how long yow run since you only clean up at the end of a run. While we do not have a lot of data with the CLEVER process to estimate our actual scrap rates in production we expect that they will be similar to the batch process.

5.3.5 Process Complexity. The batch process is fairly complex involving seven operations in separate facilities, they are:

1. RDX drying and grinding
2. Propellant mixing
3. Blocking and straining
4. Strand extrusion
5. Pelletizing (cutting)
6. Grain drying
7. Grain Blending and glazing

Operations two through five are very dependent on the solvent content of the propellant. The entire process requires rigorous coordination and scheduling. Delays at any operation caused by equipment failure, transportation delays or other problems can result in the propellant losing too much solvent by evaporation and not being processable at the next operation. Since the operations are sequential and time related labor requirements are high.

The CLEVER process is considerably simpler than the batch process since it has four basic operations:

1. Bofors Precipitation Process
2. Twin-screw extruder processing
3. Grain drying
4. Grain blending and glazing

The Bofors Precipitation Process makes the feedstock for the twin-screw extruder. The feedstock can be made ahead of time and stockpiled until the extruder is ready to run. Also since the RDX no longer needs to be ground another variable that can affect quality is eliminated and safety is enhanced. The fact that the processes are decoupled with respect to time gives us the flexibility of running them concurrently if maximum yearly output is required or for smaller quantities sequentially with the same crew to cut labor requirements.

Overall the CLEVER process has less unit operations and is a simpler and more robust process than the batch process. Additional advantages are less capital investment in facilities, a smaller facilities footprint and enhance flexibility.

5.3.6 Charge Design and Performance. One of the main factors controlling burning rate in Lova nitramine gun propellants is the RDX particle size. Generally a smaller RDX particle size results in a lower burning rate. This allows the ballistician designing the propelling charge to use a smaller grain diameter, thereby, increasing the charge weight to volume ratio and ultimately increasing the available energy. Although the relationship is complex, the shock vulnerability of a propelling charge also tends to decrease both as the grain web decreases and as the RDX particle size decreases.

The conventional batch process for Lova relies on mechanical grinding of the RDX. The fine grind process for RDX has a lower particle size limit of approximately 4μ. The Bofors Precipitation Process relies on in situ recrystallization to form the RDX particles and is capable of producing an RDX particle size of less than 4 μ, thus increasing the design options available to the ballistician and ultimately improving the gun performance. Essentially, the propellant produced has a slower burning rate which result in smaller grains to give the desired performance. Smaller grains tend to pack better in the propelling charge so the charge weight can be increased. A higher charge weight results in higher total chemical energy and ultimately a greater velocity. Therefore, the performance can be improved by using the CLEVER process versus the conventional batch process to make propellant of the same formulation because more propellant can be packed in the propelling charge.

6. Cost Assessment

An essential criterion for evaluating ESTCP programs is the project affordability. This section

presents the detailed cost assumptions and estimates for both Batch and CLEVER processes. The cost analysis captures the (1) the investment cost for EX 99 CLEVER propellant production, (2) the direct EX 99 propellant production cost and (3) direct production support activities. The results from the cost analysis were used in a cost effectiveness analysis to determine the NPV, IRR, and Payback Period. The complete environmental and cost analysis prepared by Booz-Allen & Hamilton, Inc. using the ECAM methodology (see references 14 and 15) is provided in Appendix B.

It should be noted that the CLEVER cost estimate should not be interpreted as the total ownership cost (TOC) of production. The cost estimate is based on input from production experts, process experts and extrapolation of production data.

6.1 Cost Performance

6.1.1 General Cost Assumption. TOC represents the Total Ownership Cost of an asset. This cost includes, at a high level, design and development, production, operations and maintenance and final disposal. The CLEVER cost estimate is not a TOC estimate because it only addresses part of the design and development effort and the production costs. The CLEVER estimate does not include the cost of monitoring and testing after deployment, nor does it estimate the disposal cost.

The following assumptions (Table 24) are the result of interviews with the technical people involved in the CLEVER project. These project assumptions establish a general framework to make an equitable cost and environmental comparison between the Batch process and CLEVER process.

Table 24: Project Assumptions for Cost Analysis

Description	Value	Data Description/Source
Fiscal Year (FY)	2000	All cost estimate values are based in constant year FY 2000 dollars.
Period of Performance	10 Years	The Batch/CLEVER process analyses are over a 10-year period of performance.
Discount Rate (Real)	4.0%	OMB Circular A-94, Revised January 2000
Annual Production Quantity (pounds)	250,000	Comparison quantity of EX 99 propellant.

The formulation (raw materials) to produce EX 99 propellant is presented in Table 25.

Table 25: EX 99 Propellant Material/Formulation

Ingredients	Percentage

RDX	76.0%
Cellulose Acetate Butyrate (CAB)	12.0%
Acetal/Formal	7.6%
Nitrocellulose	4.0%
Ethyl Centralite	0.4%

6.1.2 Cost Summary. Table 26 presents the Cost Summary for the CLEVER project. Utility resource consumption and costs may be a significant cost driver in evaluating alternative manufacturing technologies. Utility analysis consisted of reviewing data for resource consumption from Batch and CLEVER technical reports. The Batch process relies primarily on electricity, while the CLEVER process requires electricity, steam, and water. Preliminary analysis of the utility consumption for each process revealed that this cost is negligible to the overall cost analysis.

Table 26: CLEVER Cost Summary

CLEVER Cost Summary			
Start-Up		Operations & Maintenance (10 year estimate)	
Actvity	Dollars	Actvity	Dollars
Capital Equiment	$ 14,247,472	**Operations Labor**	$ 23,821,080
Twin Screw Extruder	$ 2,585,000	**Supervision/Technical Supp**	$ 4,550,750
Bofor Precipitation Process	$ 11,652,600	**Maintenance**	$ 1,626,330
Solvent (11,894 pounds)	$ 9,872	**Raw Material**	$ 29,574,420
Site Preparation & Construc	$ 5,502,000	**Processing Material**	$ 395,870
Planning/Engineering	$ 563,000	**Waste Disposal**	$ 379,470
Planning	$ 370,448	**Environmental Management & Monitoring**	$ 2,159,966
Permitting	$ 12,000	**Other Direct Cost**	$ 1,250,000
TOTAL INVESTMENT	**$ 20,694,920**	TOTAL INVESTMENT	**$ 63,757,886**
All Costs are Presented in Constant Year 2000 Dollars			

The CLEVER production process will require a capital investment for:

- The Bofors Precipitation Process equipment.
- Construction of a new production facility. (The total cost estimate for construction of the new processing facility is $11.0 million dollars, split evenly between the EX 99 propellant production and other energetic material production programs.)

- The 92mm Twin Screw Extruder (TSE). (The total cost estimate for the TSE $ 5.2 million dollars, split evenly between the EX 99 propellant production and other energetic material production programs.)

In addition to the equipment capital investment, additional investment requirements include:

- The CLEVER demonstration and validation effort,
- The review and development of standard operating procedures (SOPs),
- The initial environmental assessment, permitting and testing.

The production cost estimate is based on an analysis of current production processes and an impact analysis of how the CLEVER production process will reduce cost and the generation of waste streams.

6.2 Cost Comparison to Conventional and Other Technologies

Please refer to Appendix B for the detailed cost comparison contained in the Closed Loop Energetics With VOC Emission Reduction (CLEVER) Cost and Environmental Analysis. The results presented in this analysis are summarized below.

The cost comparison compares the baseline Batch process to the CLEVER process in terms of Net Present Value (NPV), Internal Rate of Return (IRR) and Payback Period. Each one of these measures helps the decision-maker with the investment decision. The financial analysis is based on production of 250,000 pounds of EX 99 propellant per year, over 10 years.

6.2.1 Net Present Value (NPV). The NPV is the present value of the future net revenues or cost savings of an investment less the investment's current and future cost. An investment is profitable if the NPV of the net revenues it generates in the future exceeds its cost (e.g., a positive NPV). The NPV method is based upon the concept that a dollar today is worth more than a dollar tomorrow. The methodology, using a Discount Rate[1], progressively reduces the value of costs and revenues over time. Federal facilities typically use the discount rate published by the Office of Management and Budget (OMB).

The NPV of the CLEVER cost analysis is $16.1 million (Table 27). This is calculated by estimating 10 years of cost for producing 250,000 pounds of EX 99 propellant per year and the investment cost for the CLEVER process. The 10-year cash flow is then discounted at the real OMB rate of 4.0% per year and summarized.

Table 27: Net Present Value

	Investment Cost	10 Year Cost Estimate Constant Year	Total Cost Estimate	Present Value Total Cost Estimate

[1] Discount Rate: The interest rate used to discount future cash flows to their present value. This represents the rate of return that could be earned by investing in a project with risks comparable to the project being considered. Federal facilities generally use a discount rate determined by the office of Management and Budget (OMB).

				(Discount Rate 4%)
Batch Process	$ 0	$ 109,011,742	$ 109,011,742	$ 88,418,288
CLEVER Process	$ 20,694,920	$ 63,757,886	$ 84,452,806	$ 72,408,277
NPV				$ 16,010,011

6.2.2 Internal Rate of Return (IRR). While the IRR is not commonly applied in Federal investment decision-making, it has been estimated for one more point of reference. The purpose of the IRR calculation is to determine the interest rate at which the NPV is equal to zero. If the IRR exceeds the "hurdle rate" (defined as the minimum acceptable rate of return on a project), the investment may be deemed worthy of funding.

The estimated ten-year IRR for the CLEVER process (Table 28) is 17.5%.

Table 28: Internal Rate of Return

	Internal Rate of Return (10 year)
CLEVER Process	17.5%

Because the IRR (17.5%) is greater than the discount rate (4.0%), this financial metric supports the decision to invest and implement the CLEVER process.

6.2.3 Discounted Payback Period. The discounted payback period analysis is the investment performance indicator most commonly used by many Federal Agencies (see reference 16). The purpose of a discounted payback analysis is to determine the length of time the discounted future savings of a project will repay the investment costs. Those investments that recoup their costs before a set "threshold" period are usually determined to be worth funding. The estimated discounted payback period is approximately 5.15 years (Table 29). The decision-maker needs to consider the acceptability of this time frame.

Table 29: Discounted Payback Period

	Discounted Payback Period
CLEVER Process	5.15 Years

7. Regulatory Issues

The Indian Head Division, Naval Surface Warfare Center (Indian Head) has been in the business of developing, testing and manufacturing ordnance and energetics to support the defense

requirements of the nation for over a century. Indian Head began operation in 1890 as a Naval gun test facility and has evolved from a "powder factory" into a critical source serving the tri-services with thousands of specialized ordnance devices and components.

Because of the significant environmental, safety and health (ESH) considerations associated with the production of defense energetic materials, federal, state, and local authorities regulate activities at IHDIV, NSWC. IHDIV has evaluated and inserted pollution prevention technologies throughout the energetic materials lifecycle in order to become more effective and efficient at addressing new and emerging environmental issues. In an era of increasingly stringent regulatory requirements, the innovative nature of the CLEVER Program proactively addresses pollution prevention and compliance aspects of many regulatory requirements. The CLEVER process will help Indian Head ensure compliance with these requirements because it has been designed and engineered to incorporate pollution prevention into the process itself. The success of this approach is evident in that the amount of regulated media (such as VOC solvent emissions, and hazardous and solid waste) generated as a result of the production process is significantly reduced compared to conventional batch processing techniques. In addition, CLEVER technology provides positive safety and health benefits to decrease operator exposure to toxic and/or hazardous materials and contribute to a safer work environment for operators.

7.1 Approach to Regulatory Compliance And Acceptance

The CLEVER Project Has Designed And Engineered Pollution Prevention Into The Process, And Has Utilized A Comprehensive, Multi-Pronged Approach To Ensure Both Regulatory Compliance And Regulator And Stakeholder Acceptance Of The CLEVER Process. The CLEVER Strategy Includes:

- Openly engaging Environmental Division personnel throughout the construction and operation of equipment an facilities;
- Identifying major regulatory drivers applicable to energetic materials production, assessing the ESH issues and impacts associated with energetic material chemical constituents, and quantifying waste streams; and
- Ensuring stakeholder participation throughout the process.

This approach has served to enhance the standing of Indian Head Division among federal, state, and local regulators; and the surrounding community, by demonstrating the installation's commitment to environmental protection and the minimization of safety and health hazards to workers and the surrounding community.

7.2 Regulatory Interactions

Production operations at Indian Head Division cannot proceed unless the proper environmental permits have first been secured from the appropriate regulatory authorities. The introduction of a new process; or changes/modifications to existing, permitted processes, necessarily requires IHDIV to determine the need for potential environmental permit modifications. Because the environmental permitting process can be a lengthy experience, the facility’s personnel have established a close working relationship with federal, state, and local authorities. Production engineers and environmental personnel with expertise in specific media work closely to ensure that they have a clear understanding of the propellant production processes and associated ESH considerations. Prior to actual construction and operation of CLEVER process equipment at the

larger scale, production engineers and personnel from the Environmental Division will review the project and determine if any new permits or permit modifications will be required. Changes to the process may also be identified to facilitate permitting. This cooperative effort ensures the project will meet all environmental regulations and permit requirements. Permit modifications and applications are made through appropriate agencies of the Maryland Department of the Environment, Department of Natural Resources and Department of Housing and Community Development. These agencies are contacted and consulted as part of the process of modifying of, or applying for a new permit. These agencies include the Air and Radiation Management Administration (ARMA), the Waste Management Administration (WSA), the Water Management Administration (WMA) and the Office of Preservation Services.

The CLEVER project has benefited from a history of close cooperation with federal, state and local regulators. Consultation and cooperation with environmental personnel and regulatory authorities will continue as CLEVER matures, and will continue throughout the effective life cycle of the new production equipment. This will serve to minimize the potential down time attributable to regulatory issues, and serves to ensure that the ability of Indian Head Division to carry out its national defense missions is not unduly impacted.

7.3 Review of Regulatory Drivers

The CLEVER project conducted a comprehensive review of federal, state, and DoD-specific regulatory drivers impacting the implementation of the new production process. The review considered major environmental statutes and regulatory requirements addressing different environmental media (e.g., solid, liquid, and gaseous emissions), as well as safety and health implications. In addition, the CLEVER project assessed the gun propellant's chemical composition to determine the ESH effects of each individual chemical constituent and their regulation under major environmental, safety and health regulations. In evaluating the individual chemical constituents, literature reviews were conducted, Material Safety Data sheets (MSDS) obtained and analyzed, and Chemical Abstract Service numbers (CAS#s) identified. The CAS#s were then used to search regulatory databases to determine the regulations applicable to the energetic material constituents. The thorough review of potential issues associated with regulatory drivers further demonstrates the commitment of both Indian Head Division and the CLEVER project to an environmental management approach that is comprehensive, open and transparent to stakeholders.

7.4 Public Participation

As an active DoD installation, IHDIV has numerous mechanisms at its disposal to encourage public participation and stakeholder involvement in different aspects of its operations. Several are briefly discussed below.

Under Section 112r of the Clean Air Act, Indian Head Division is required to have developed a detailed risk management plan (RMP) to minimize the potential consequences that may result from an accidental release of hazardous materials. Under the Emergency Planning and Community Right-to-Know Act (EPCRA), facilities that store or manage certain hazardous or extremely hazardous substances in quantities greater than established threshold planning quantities are required to:

- Participate in the local emergency planning process. IHDIV is a member of the Local Emergency Planning Committee (LEPC);
- Notify state and local emergency planning officials of the presence of hazardous substances above established thresholds;
- Provide appropriate ESH data to state and local emergency planning officials; and
- Report releases of such substances that exceed reporting thresholds.

IHDIV aggressively implements the requirements of the National Environmental Policy Act (NEPA). NEPA requires federal agencies to evaluate the environmental consequences of proposed federal actions, identify and assess reasonable alternatives to proposed actions in order to minimize or avoid adverse environmental effects, and to have procedures in place to ensure that relevant environmental information is made readily available to decision makers and the public before decisions are made and federal actions taken. An Environmental Assessment (EA) is being conducted for both the construction of the large-scale Bofors Precipitation Process and TSE facilities. Notice of intent to conduct the EA, and the findings of the EA itself, are required to be published in the local paper for a 30 day comment period.

Public notices are also published when wastewater and hazardous waste permit modifications or applications are being finalized. If warranted by public response to the permit notifications, public meetings may be conducted. These meetings are generally held off plant at locations convenient to the public. The meetings are announced in the “Legal Notices” sections of local newspapers and announced on local radio stations depending on the regulators guidelines. As discussed above, the permitting process results in close cooperation and interaction with federal, state, and local government officials. IHDIV also has a Public Affairs Office with well established procedures for distributing information related to plant operations and handling other related inquiries; both military and contractor personnel are always ready to meet with the public and local authorities on an as needed basis to address community concerns as they may arise.

8. Technology Implementation

8.1 DoD Need

EX 99 Lova nitramine gun propellant is used in the propelling charges being developed and qualified for the Navy's NSFS Extended Range Guided Munition (ERGM, EX 167 Propelling Charge) and for Navy's 5-inch Cargo Round (EX 73 Propelling Charge). Current production requirements for Navy's NSFS ERGM propelling charge are projected at 2000 rounds (60,000 pounds of propellant) per year for FY 05 through FY 14. This equates to a total of 600,000 pounds of propellant. Current production requirements for the Navy's Cargo Round are also projected at 2000 rounds (60,000 pounds of propellant) per year for FY 03 through FY 14. This equates to a total of 720,000 pounds of propellant.

The other propelling charge that the Navy is currently developing is a new EX 175 Propelling Charge that uses similar hardware and propellant. Once implemented, the EX 175 Propelling Charge will be used with the new Cargo projectile. Production requirements have been

projected at 60,000 pounds per year. Additionally, once the technology is proven, it will also be available to Current procurements for these systems are planned at 2000 rounds (60,000 pounds) per year for each of the two propelling charges.

Additionally, the Navy has several other development programs that are underway. The Navy's gun efforts are shown in Figure 40.

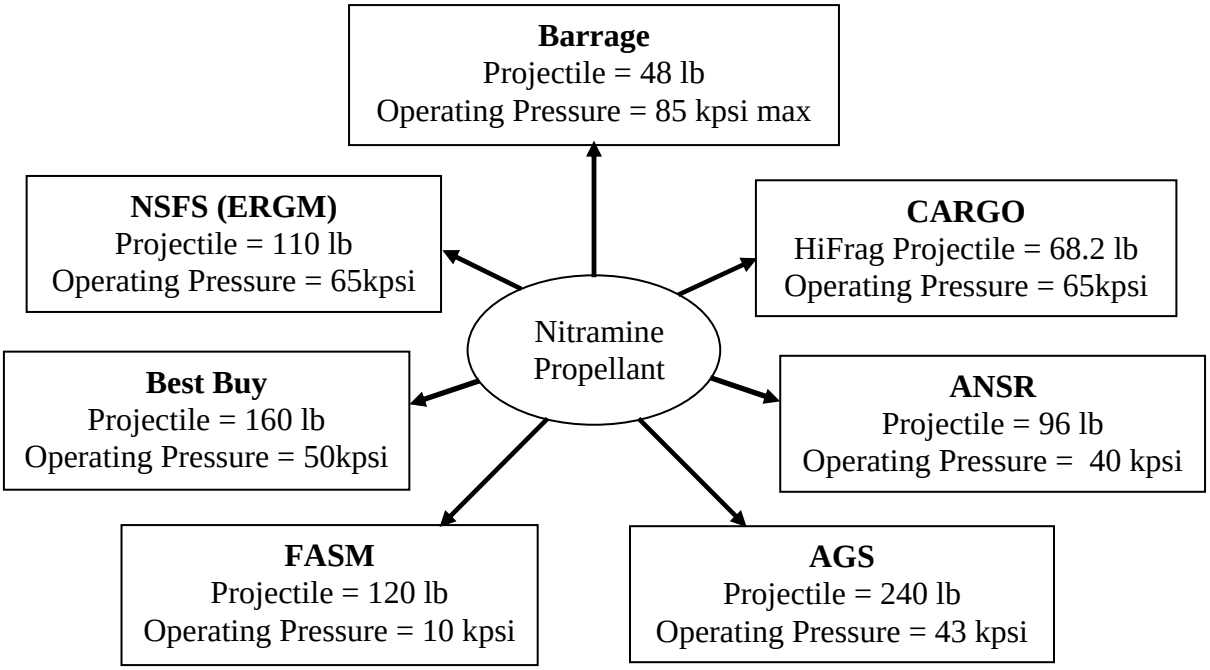


Figure 40: Navy Need for Lova Nitramine Gun Propellant

Besides ERGM and Cargo programs, Best Buy, Forward Area Support Munition (FASM), Advanced Gun System (AGS) and Barrage are all to meet various missions for the Navy's 5-inch gun. Autonomous Naval Support Round (ANSR) is a new 6-inch Naval gun. Some of those programs are farther along in the development process than others but none of those are far enough along to have procurements established. This interest in nitramine gun propellants clearly shows that there is currently a Navy need for this propellant and will be a Navy need for this propellant.

Additionally, the technology is also available to manufacture propellant for the Army to meet their near term Future Combat System needs, the Air Force, and the private sector for use in automotive air bags.

8.2 Transition

Indian Head Division is bringing the CLEVER technology to the Navy. Contracts to design, build, and start-up a Nitramine Intermediates Facility (for the Bofors Precipitation Process) and Continuous Processing Facility (centered around and 88-mm twin-screw extruder) were awarded in FY00. The contract for the Nitramine Intermediates Facility was awarded to Weatherly, Inc; a supplemental contract was awarded to Nexplo Explosives (formerly Bofors Explosives) for a license to use their patented technology. Washington Group International, formerly Raytheon,

was awarded the contract for the Continuous Processing Facility. The start-up and turn over to the government is scheduled for FY04 for the nitramine precipitation plant and FY02 for the continuous processor. The paste feedstock required for any propellant manufactured in the Continuous Processing Facility between FY02 and the completion of the start-up of the NIF in FY04 will be procured from Nexplo Explosives. The two facilities will transform IHDIV propellant and explosive processing technology, reduce environmental impact, improve operational safety, and lead the transition of the technology to industry.

9. Lessons Learned

The most succinct lesson learned during the demonstration was to keep it simple. Overloading the test agenda by adding items, which although they may be important are peripheral to the core technology demonstration, is inviting problems. We committed this error when we decided to pelletize all the Lova on line. Working the bugs out of a prototype cutter caused numerous unnecessary stops during the first extruder run.

When feasible structure the testing so that it can be done in segments by the same crew during normal working hours. For reasons of economy and facility workload we chose to operate around the clock with multiple shifts. This required that personnel accustomed to working the day shift be assigned to the midnight shift. Expecting people to work at maximum efficiency on a highly technical task without giving them time to adjust to new working hours is asking a lot.

Rely on your normal methods for recording process data and automate as much of the data collection as possible. Our most reliable data came from these sources. Introducing new forms that require a lot of data entry by personnel at 3 AM while they are solving technical problems that normally accompany the startup of a new process can overload the operators no matter how dedicated they are.

If your resources allow it conduct a full-scale practice run prior to the actual demonstration. We did not have enough propellant paste to do this; if we had we could have worked out a lot of the minor problems prior to the actual demonstration run. Estimate your material requirements and then double them.

10. References

1. Lekander, Sven; *Nitramine Gun Propellant Development: Joint Effort Between Bofors Explosives and NSWC*; Bofors Technical Report XU 5-96/237 prepared under Contract N00174-94-C-0041; August 31, 1996.

2. Hardy, Richard A., Newman, Kirk E., and Peranich, Robert G.; *Data Collection for Life Cycle Assessment Models, Part2, 105-mm M900 APFSDS-T Projectile*; IHTR 1814; Indian Head Division, Naval Surface Warfare Center; July 31, 1995.

3. US Patent Number 4,982,235, *Method for the Production of Fine-Grained Explosive Substances*; Nygrist, Jan-Olof, Hafstrand, Anders, and Kaml, Nobel, Nobel Kaml, AB, Karlskoga, Sweden, April 28, 1989.

4. Murphy, Constance; *Continuous Processing of Composite Propellants*; Joint Army, Navy, NASA, Air Force (JANNAF) Conference Proceedings; Monterey, CA; November 1993.

5. Muscato, Richard and Murphy, Constance; *Continuous Processing Study of LOVA Gun Propellant on a Twin Screw Extruder*; Joint Army, Navy, NASA, Air Force (JANNAF) Conference Proceedings; Pasadena, CA; March 1995.

6. Muscato, Richard; *Lova Twin Screw Processing & Propelling Charge Gun Firing*; Continuous Mixer and Extruder Users Group Meeting Proceedings, Hoboken, NJ; June 1997.

7. *Propellant M43 For Use In Cartridge 105mm*, APFSDS-T, M900; MIL-P-70818A(AR); August 23, 1994.

8. *High Energy Nitramine Gun Propellant Detail Specification*; MIL-DTL-82965(OS)A; June 27, 2001

9. Lekander, Sven; *CLEVER Report, Additional Work*; Bofors Technical Report XU 5-99/420 October 8, 1999.

10. Murphy, Constance; *ESTCP Closed Loop Energetics with VOC Emission Reduction (CLEVER) Project Technology Demonstration Plan*; Indian Head Division, Naval Surface Warfare Center; February 2, 1998.

11. Coffey, C.S. and DeVost, V.F.; *The Ballistic Impact Chamber (BIC) Test*; Propellants, Explosives, Pyrotechnics 20, 1995, pages 110 - 113.

12. Lieb, Robert; *Mechanical Response and Morphological Characterization of Gun Propellant*; Army Research Laboratory Technical Report (ARL TR-1205); September 1996.

13. Alexander, Kim, Gibson, Kevin and Baudler, Brian; *Development of the Variable Confinement Cookoff Test*; IHTR 1840; September 1996.

14. *Environmental Cost Analysis Methodology Implementation Report*; National Defense Center for Environmental Excellence operated by Concurrent Technologies Corporation; prepared under Contract DAAA21-93-C-0046, Task N.098, CDRL A012; January 7, 1998.

15. *Environmental Cost Analysis Methodology* may be downloaded from the ESTCP web site

at http://www.estcp.org/documents/guidance/index.cfm and selecting Environmental Cost Analysis Methodology (ECAM SM) Handbook and Software or by going directly to http://www.ndcee.ctc.com/ecam/.

16. *Federal Facility Pollution Prevention Project Analysis: A Primer for Applying Life Cycle and Total Cost Assessment Concepts*; Office of Enforcement and Compliance Assurance, U.S. Environmental Protection Agency, Washington DC, June 1995.

Appendix A
Points of Contact

Program Managers:

Mr. Ashley Johnson
Surface Weapons and Ammunition Program Manager, Code PM2
Indian Head Division, Naval Surface Warfare Center
101 Strauss Avenue
Indian Head, Maryland 20640-5035
Phone: 301-744-6704
Fax: 301-744-4187
E-mail: johnsonag@ih.navy.mil

Mr. Charles R. Painter
Environmental and Chemicals Program Manager, Code PM4
Indian Head Division, Naval Surface Warfare Center
101 Strauss Avenue
Indian Head, Maryland 20640-5035
Phone: 301-744-6772
Fax: 301-744-4187
E-mail: paintercr@ih.navy.mil

Project Managers:

Bofors Precipitation Process Technology:
Mr. Ulf Larsson
Nexplo Bofors AB
SE-691 86 Karlskoga, Sweden
Phone: 4658683148
E-mail: ulf.g.larsson@nexplo.se

Twin-Screw Extrusion Technology:
Mrs. Constance M. Murphy
Continuous Processing Project Manager, Code 2330A
Indian Head Division, Naval Surface Warfare Center
101 Strauss Avenue
Indian Head, Maryland 20640-5035
Phone: 301-744-6497
Fax: 301-744-4843
E-mail: murphycm@ih.navy.mil

Gun Systems Engineering:
Mr. Michael R. Thornton
Gun Systems Technology Branch Manager, Code 4210
Indian Head Division, Naval Surface Warfare Center
101 Strauss Avenue

Indian Head, Maryland 20640-5035
Phone: 301-744-1438
Fax: 301-744-6223
e-mail: thorntonmr@ih.navy.mil

Technical:

Bofors Precipitation Process Technology:
Mr. Sven Lekander
Chiragene Inc. (formerly Bofors Explosives)
7 Powder Horn Drive
Warren, New Jersey 07059
Phone: 732-805-3664
E-mail: sven.lekander@cambrex.com

Ms. Valerie L. Plummer *(ESTCP Principal Investigator)*
Chemical Engineer, Code 2120P
Indian Head Division, Naval Surface Warfare Center
101 Strauss Avenue
Indian Head, Maryland 20640-5035
Phone: 301-744-1926
Fax: 301-744-4522
E-mail: plummervl@ih.navy.mil

Twin-Screw Extrusion Technology:
Mr. William F. Newton
Chemical Engineer, Code 2330B
Indian Head Division, Naval Surface Warfare Center
101 Strauss Avenue
Indian Head, Maryland 20640-5035
Phone: 301-744-2458
Fax: 301-744-4843
E-mail: newtonwf@ih.navy.mil

Gun Systems Engineering:
Mrs. Jennifer McKee
Gun Systems Engineer, Code 4210H
Indian Head Division, Naval Surface Warfare Center
101 Strauss Avenue
Indian Head, Maryland 20640-5035
Phone: 301-744-1188
Fax: 301-744-4697
E-mail: mckeejj@ih.navy.mil

Appendix B
Supporting Data and Documentation