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Final Report

One Step No Waste Composition C4 Production
ESTCP Project WP-201508

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14. ABSTRACT

(U) The Composition C4 (C4) hand-moldable M112 demolition blocks are used by all Department of Defense (DoD) services and are consumed at the rate of several hundred thousand blocks per year. Since 1950, C4 has been produced by a 10-step water slurry batch process that uses an organic solvent-based lacquer to apply a binder system to RDX nitramine crystals. The C4 batch method is labor and energy intensive with some inherent environmental risk. Then the C4 bulk powder is trucked to another facility to be post-processed into M112 demolition blocks via two additional manufacturing steps.

(U) The project demonstrated the manufacture of an alternate C4 composition (called PAX-52) which consolidated the 12-step manufacture of C4/M112 blocks into a single step by using a twin screw extrusion (TSE) technology. TSE is a waste-free and low-energy method for the continuous and safe production of M112 demolition blocks that are homogenous and ready-formed for packaging. The improved process was based on a “green” formulation which replaced the C4 binder system and binder coating media (solvent and water) with a silicone polymer and replaced the RDX nitramine with the far more eco-friendly HMX.

(U) Silicone polymers are environmentally-inert and exhibit low surface tension for highly efficient wetting and coating of high explosives such as RDX and HMX. The silicone polymer type and the particle size distribution of the energetic particles was tailored to generate an easily processed PAX-52 formulation that can be mixed, deaerated, and pressurized in any TSE and die-formed into the specified M112 shape.

(U) The techniques used to delineate the PAX-52 formulation features included characterization of the interfacial tension between the high explosive particles and the silicone binder, correlation between the rheological behavior of the silicone binder and the high-solids suspensions, mathematical modeling and 3-D Finite Element Method (FEM) numerical solution-based selection of the TSE screw and barrel configurations, operating conditions (temperature and pressure,) and model-based forming die design.

(U) A 500-pound batch of PAX-52 material was produced using a twin screw extruder then post-processed-packaged into M112 demolition blocks. The PAX-52 material was characterized against a select panel of energetic qualification tests and found to be within tolerance for all key quality and performance specifications. In particular PAX-52 was found to be cap sensitive (sensitivity to the shock from a standard blasting cap) and powerful enough to pass the energy output test where the demolition charge cut a 1.0 inch thick steel witness plate completely into two separate sections.

(U) Samples of the PAX-52/M112 demolition blocks were evaluated by the DoD C4-user community for a hands-on comparison to the C4/M112. The 595 Sapper Company, 5th Engineer Battalion at Fort Leonard Wood worked with PAX-52 by loading devices and demolition charges in a live training exercise. In addition, the Explosives Ordnance Disposal (EOD) Division, ARDEC, conducted subjective handling assessments of PAX-52 and its employment in various shaped charge containers and disposal procedures by representatives of three Armed Services (Army, Air Force and Marine Corps.) Mostly positive feedback was received from the evaluations but the clearest signal from the user groups was their enthusiasm for the cold-weather moldability of PAX-52.

(U) One 60-kilo batch of bulk PAX-52 was sent to Defence Research and Development Canada (DRDC) Valcartier, Canada and evaluated as a candidate to replace C4 as a low-no RDX hand-moldable demolition explosive. In addition to positive results in cold-weather moldability and initiation reliability, researchers detected only trace amounts of RDX in detonation residues with deposits of HMX that equate to toxicity levels nearly three orders of magnitude less than was detected in conventional C4.

(U) The continuous TSE technology and the novel PAX-52 formulation presented several key advantages over the existing production method. PAX-52 was demonstrated to be superior to C4 in hand-moldability through a range of temperatures (-40ºF and 140ºF) with no discernable difference in viscosity owing to the properties of silicone. PAX-52 was easily compounded to a high packing fraction and a desirable density using a mono-modal, large particle (200 µm) class of energetic powder. Post process analysis of PAX-52 showed a distinct shift to a bi-modal distribution (50 µm and 200 µm) due to a favorable *in-situ* shear reduction of larger particles into small. In addition, it was demonstrated that desirable formulations of PAX-52 could be produced using other methods, specifically: resonance acoustic mixing (RAM), planetary and sigma blade mixers; however, these methods were not modeled or optimized as part of this research.

(U) Historical data for the current C4/M112 production method were analyzed using time-value equations to set an average baseline cost of C4/M112 at $35 per block. Then an engineering model and cost assessment that used both empirical and industry standard data was developed for the demonstrated process technology, which estimated the baseline cost of PAX-52/M112 to be $67 per block. Though the single-step production demonstration validated manufacturing cost savings through reductions in labor, energy and process waste, and the several non-tangible cost benefits of PAX-52 related to hand-moldability, mission effectiveness, initiation reliability, manufacturing flexibility, etc; the unit cost of nitramine HMX was found to be the overwhelming cost driver in the manufacture of PAX-52. HMX costs nearly three times RDX which makes PAX-52 a pricey alternative for the C4 product managers; however; the project looked past the obvious HMX-to-RDX price differential to investigate several over-shadowed cost burdens associated with the current technology that would be greatly reduced by the alternative.

(U) Program Managers must consider sustainability during systems acquisition and select more sustainable systems that meet performance requirements; have fewer negative impacts on resource availability, human health, and ecosystem quality; and have a lower Total Ownership Cost (TOC). Here, a Sustainability Analysis assessed life cycle costs (internal to DoD) and impacts on resource availability, human health, and environmental quality (external costs) of both technologies and found PAX-52 to be significantly more sustainable.

(U) This report presents a comprehensive body of research that supports the proposed replacement of C4/M112 production method by a continuous processing method in conjunction with a green formulation, PAX-52. Some critical military information is redacted from this report but may be available by request to the project investigator.

15. SUBJECT TERMS Composition C4, PAX-52, M112 Demolition Block, Silicone, Energetic Nitramine RDX/HMX, Twin-screw Extrusion, Continuous Process, Environmental Impact, No Waste, Single Step.					
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Table of Contents

(U) LIST OF FIGURES

(U) LIST OF TABLES

(U) LIST OF ACRONYMS AND ABBREVIATIONS

(U) Acronyms and Abbreviations			
A&E	Ammunition & Explosives	**LCPP**	Life Cycle Pilot Process
AAP	Army Ammunition Plant	**LOT**	Specific identified amount produced
ABL	Allegany Ballistics Laboratory	**LOVA**	Low Vulnerability Ammunition
ACGIH	American Conference of Governmental Industrial Hygienists	**LTC**	Leutenant Colonel
AEC	U.S. Army Evaluation Center	**M112**	Military Demolition Block
AES	Accurate Energetic Systems	**METC**	Munitions, Engineering and Technology Center
AMC	Army Materiel Command	**MI**	Mixing Index
AMMO	Ammunition	**MIL-DTL**	Military Detail, Military Detail Specification
ANB	anhydride-free binder	**MO**	Missouri
AO	American Ordnance	**MSCoE**	Maneuver Support Center of Excellence
AOP	Allied Ordnance Publication	**MSDS**	Material Safety Data Sheet
ARDEC	Armaments Research,Development & Engineering Center	**NATO**	North Atlantic Treaty Organization
ARTI	Applied Research and Technology Institute	**NIOSH**	National Institute of Occupational Safety and Health
ASTM	American Society for Testing and Materials	**NJDEP**	New Jersey Department of Environmental Protection
ATL	Acquisitions Technology and Logistics	**NOL**	Naval Ordnance Laboratory
BAE	British Aerospace Systems	**NPDES**	National Pollution Discharge Elimination System
BAM	German Federal Institute for Testing and Materials	**NPL**	National Priorities List
BIP	Blow in Place	**NSWC**	Naval Surface Warfare Center
BP	Black Powder	**OEL**	Occupational Exposure Limit
BRAC	Base Realignment and Closure	**OSD**	Office of the Secretary of Defense
C-4, C4	Composition 4	**OSHA**	Occupational Safety and Health Administration
CAF	Canadian Armed Forces	**OUSD**	Office of the Under Secretary of Defense
CL-20	China Lake, nitroamine explosive	**PAX**	Picatinny Arsenal Explosive
DAG	Defense Acquisition Guidebook	**PAX-52**	Picatinny Arsenal Explosive #52
DA-PAM	Department of the Army Pamphlet	**PBX**	Polymer-bonded explosive
DE	Dentonation Efficiency	**PDMS**	Polydimethylsiloxane - Silicone oil
DETEF	Dentonation Efficiency	**PEO**	Program Executive Officer
DIO	Defense Input/Output (database)	**PETN**	pentaerythritol tetranitrate - High Explosive
DMDNB	2,3-dimethyl-2,3-dinitrobutane - Chemical Taggant	**PI**	Principal Investigator
DoD	Department of Defense	**PLC**	Programmable Logic Controller
DOTC	DOD Ordnance Technology Consortium	**PM-CCS**	Product Manager for Close Combat System
DR	Detonation Rate	**POM**	Program Objective Memorandum
DRDC	Defence Research and Development Canada	**PPE**	Personal Protection Equipment
DSC	Differential Scanning Calorimetry	**Q&A**	Questions & Answers
EFP	Explosively Formed Penetrator	**Q-D**	Quantity-Distance
EMQB	Energetic Materials Qualification Board	**RAM**	Resonance Acoustic Mixing
EOD	Explosive Ordnance Disposal	**RCRA**	Resource Conservation and Recovery Act
ERL	Energetics Research Laboratory	**RDX**	Research Department Explosive
ESD	Electrostatic Discharge	**RMSL**	Rocky Mountain Scientific Laboratory
ESMP	Explosives Safety Management Program	**RPM**	Revolutions per Minute
ESOH	Environment, Safety, and Occupational Health	**RTA**	Ranges and Training areas
ESTCP	Environmental Security Technology Certification Program	**SCADA**	Supervisory Control and Data Acquisition
FAT	First Article Testing	**SDS**	Safety Data Sheet
FLW	Fort Leonard Wood	**SEM**	Scanning Electron Microscope
FOD	Foreign Object Debris	**SERDP**	Strategic Environmental Research and Development Program
FREEDM	Future Requirements of Enhanced Energetics for Decisive Munitions	**SIT**	Stevens Institute of Technology
GOCO	Government Owned - Contractor Operated	**SOF**	Special Operations Forces
GOEX	Black Powder Manufacturer	**SOP**	Standing Operating Procedure
HASP	Health and Safety Plan	**SPPC**	Spill Prevention, Control, and Countermeasure
HMI	Human Machine Interface	**SSWG**	System Safety Working Group
HMX	High Melt Explosive, Her Majestys Explosive	**STANAG**	Standardization Agreement
HPLC	High Performance Liquid Chromatography	**TB**	Technical Bulletin
HSAAP	Holston Army Ammunition Plant	**TIL**	Threshhold of Initiation
I/O	Input/Output	**TLV**	Threshold Limit Value
IAW	In Accordance With	**TMDE**	Test, Measurement, and Diagnostic Equipment
IHE	Insensitive High Explosive	**TOC**	Total Ownership Cost
IM	Insensitive Munition	**TNT**	Trinitrotoluene
IPT	Integrated Product Team	**TSE**	Twin-screw Extrusion, Extruder
IRIS	Integrated Risk Information System	**TTA**	Technology Transfer Agreement
IWWTF	Industrial Waste Water Treatment Facility	**TWA**	Time Weighted Average
JIMTP	Joint Insensitive Munitions Technology Program	**U**	Unclassified
KOC	Soil Adsorption Coefficient	**UCL**	Upper Control Limit
LA	Louisiana	**USEPA**	United States Environmental Protection Agency
LANL	Los Alamos National Laboratory	**USSS**	US Sieve Size
LBR	Low-burning Rate	**UTSE**	Universal Twin Screw Extrusion Facility
LCA	Life Cycle Analysis	**UV-VIS**	Ultraviolet Visible Spectroscopy
LCC	Life Cycle Cost	**VCCT**	Variable Confinement Cook-off Test
LCCA	Life Cycle Cost Analysis	**VoD**	Velocity of Detonation
LCL	Lower Control Limit	**VTS**	Vacuum Thermal Stability

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(U) ABSTRACT

(U) Introduction and Objectives: The Composition C4 (C4) and hand-moldable M112 demolition blocks are used by all Department of Defense (DoD) services and are consumed at the rate of several hundred thousand pounds per year. Since 1950, C4 has been produced by a water slurry process production process that is time, energy, and labor intensive. The project demonstrated the manufacture of an alternate C4 composition (called PAX-52) by using a twin screw extrusion (TSE) process. The improved process was based on a "green" formulation which replaced the C4 binder system with a silicone polymer (Polydimethylsiloxane) and replaced the RDX nitramine with the far more eco-friendly HMX.

(U) Technology Description: Twin screw extrusion is a continuous manufacturing platform relying on screw elements assembled on two parallel shafts, with the two screws enclosed in a figure-8 barrel with openings for feeding of the solid and liquid ingredients, devolatilization via the application of vacuum and a die for the shaping of the high-solids energetic suspension. The TSE technology presented difficult challenges for the processing of energetic materials so the development of multiple important scientific and technical capabilities prior to its demonstration were required to meet those challenges.

(U) Performance and Cost Assessment: A 500-pound batch of PAX-52 material was produced using the TSE at Picatinny Arsenal and then post-processed-packaged into 257 M112 demolition blocks. The production run met all expectations for steady-state continuous processing with no waste in the production of homogenous, well-formed bulk blocks. The PAX-52 material was characterized against a select panel of energetic qualification tests and found to be within tolerance for all key C4/M112 quality and performance specifications. In particular PAX-52 was found to be cap sensitive and powerful enough to pass the steel plate cutting test.

(U) The DoD may only have to bear a small price premium for the superior PAX-52/M112. The TSE method validated manufacturing cost savings through reductions in labor, energy and process aid waste (water and solvent.) A cost assessment which included labor, energy and materials, showed a production cost differential nearly twice that of C4 on a normalized basis, which at face value makes PAX-52 a pricey alternative to C4; however, the project looked past the obvious pound-for-pound HMX-to-RDX price differential to investigate several over-shadowed cost burdens associated with the current technology that would be eliminated by the alternative technology. These other costs (internal, external, and contingent) associated with the impacts C4 production has on ecotoxicity and climate change, human health and occupational, water, land and energy usage, and more, are studied by a Life Cycle Assessment (LCA) and compared to PAX-52. Here, a Sustainability Analysis assessed Life Cycle Costs (LCC) (internal to DoD) and impacts on resource availability, human health, and environmental quality (external costs) of both technologies and found PAX-52 to be significantly more sustainable with a lower Total Cost of Ownership (TCO).

(U) Implementation Issues: The PAX-52 TSE technology demonstration garnered positive feedback from the current C4 user community including; combat engineers, ordnance demolition units and future munitions developers. Each had an interest to see PAX-52 at a readiness level that lead to a technology transfer. The technology highlighted for the stakeholders the feasibility of a one-step M112 production process and the PAX-52 product met all their performance and

physical requirements; however, their full acceptance of the PAX-52/M112 was deferred until it matched the cost of the C4/M112.

(U) Publications: One preprint publication: J. He, S. Lee and D. Kalyon, “Shear viscosity and wall slip behavior of dense suspensions of polydisperse particles”, arXiv: 1806.01900 [cond-mat.soft] (2018) is currently prepared for submission to the journal Science.

1 (U) EXECUTIVE SUMMARY

1.1 (U) INTRODUCTION

(U) The Composition C4 (C4) and M112 hand-moldable demolition blocks are used by all Department of Defense (DoD) services and consumed at the rate of several hundred thousand pounds per year. C4 is produced in a multi-step water slurry process that requires an organic, solvent-based lacquer to mix a cohesive binder and plasticizer with energetic RDX nitramine crystals. This multi-step process, developed in 1948, has been largely successful owing to the availability of cheap labor, cheap energy, and lower environmental protection standards; however, six decades later it is still time, energy, and labor intensive and generates 1.5 million gallons of aqueous waste per year. The output of the C4 production process is a loose powder (or cake) that is boxed and transported to an entirely different packaging facility where it is further processed into M112 demolition blocks via two additional processing steps. **Figure 1**

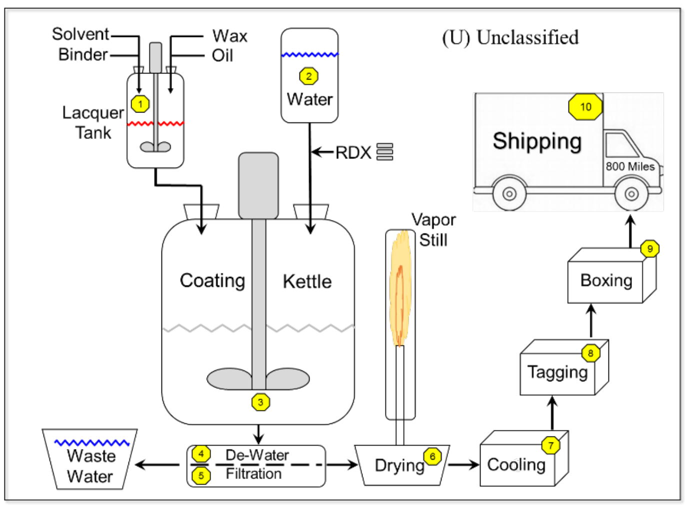

Figure 1 (U) Diagram of the Multi-step Process for Composition C4 Manufacturing. *The process requires large quantities of resources including energy for process heating and cooling, distilled water, waste water treatment, and solvent reclamation. Not depicted here, the process actually spans across several production plants and requires at least six consecutive labor shifts to complete a batch. The C4 is packaged in boxes and shipped 800 miles to be post-processed into the M112 demolition block configuration. It is there that the boxed C4 gets its moniker "cake" because it has a tendency to settle and partially consolidate during the long transport, becoming an agglomerate, cake-like formation.*

(U) This project's principle investigator proposed a manufacturing process that would replace the combined 12-steps of manufacturing C4 and M112 demolition blocks with a single step by using a continuous twin-screw mixing and extrusion process (TSE) **Figure 2**. In conjunction, a new "green" formulation would be developed to replace the current C4 binder system with a silicone polymer, eliminate the use of an organic solvent, and preclude any waste-water streams. The green formulation would provide another environmental benefit gained by replacing the RDX with the more environmentally-friendly HMX in the new C4, presently named PAX-52.

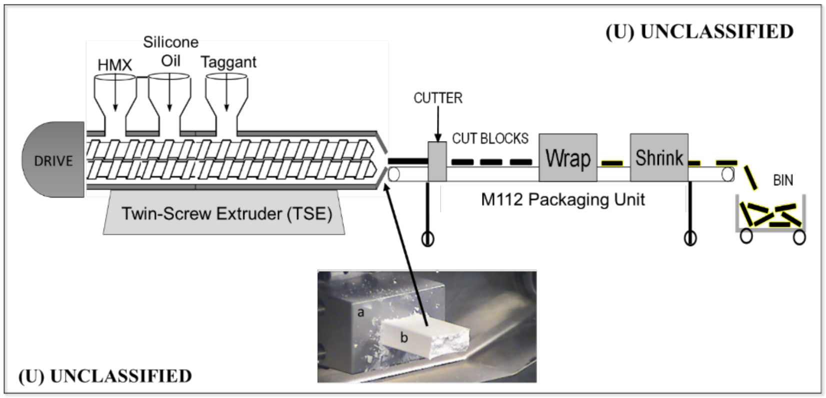

Figure 2 (U) Diagram of the Proposed Single-Step Manufacturing Process. *The single step production method would be demonstrated using a fully-intermeshing, co-rotating twin screw extruder at Picatinny Arsenal to validate a continuous process for the manufacture of both PAX-52 and the M112 demolition block on a single production platform. The two ingredients; HMX, and Silicone, (and a taggant); are metered into the TSE, homogeneously mixed by co-rotating screws, and finally extruded through a forming die (a) into the cross-section of the M112 demolition block (b.) The downstream packaging of the extrudate would not be validated by this project; however, it is depicted here to emphasize the efficiency gained by bundling all elements of M112 production into a single package.*

(U) Silicone polymers are environmentally-inert and exhibit low surface tension for highly efficient wetting and coating of high explosives such as RDX and HMX. The silicone polymer type and the particle size distribution of the energetic particles can be tailored to generate an easily processed PAX-52 formulation that can be mixed, deaerated, and pressurized in any twin screw extruder. With a specially designed forming die directly attached to the twin-screw discharge port, the mixed material can be shaped to any desired profile, here a simple rectangular cross-section specified for the M112. Though the continuous TSE process is fairly straightforward there was required much research that brought the project to the point of a demonstration.

1.2 (U) PROJECT OBJECTIVES

(U) The project demonstration would show that an analogue composition C4 explosive, called PAX-52, would be produced by compounding only two ingredients (and a taggant) in a twin-screw, continuous mixing process. Furthermore, that the compounded material would be extruded through an inverted forming die to the cross-section dimensions specified for the Army's M112 demolition block that the material, PAX-52, would meet C4/M112 quality specifications for plasticity and performance (1) (2).

(U) The demonstration would show that PAX-52 meets or exceeds sensitivity and performance characteristics of C4. The demonstration would show that the TSE PAX-52 product would not undergo a detonation response to heat ignition while confined.

(U) The demonstration would show that there are no by-products created by the TSE process. Only two ingredients (and a taggant) are required to produce PAX-52. All the energetic solids (HMX) and binder oil (PDMS) are metered into the TSE mixing process and compounded as the final product, PAX-52.

(U) The demonstration would show that there is no waste stream once the process achieves steady-state production. For the demonstration, small quantities of material associated with the start-and-stop of the process would not be considered a waste stream.

(U) The demonstration would show there are no process aids used in the manufacture of PAX-52. Use of a solvent in the mix to keep viscosity low through the mixing sections would not be required in the process.

(U) The demonstration would show that the PAX-52 process would not require external heating or cooling to maintain a steady-state level of production.

(U) The demonstration would show that the TSE PAX-52 product would be homogeneous with a density greater than 1.50 g/cc, which is the minimum density required for the M112 demolition block.

(U) The project would demonstrate that PAX-52 material can be packaged in the M112 configuration using an alternate method, with an allowance for off-site transportation.

(U) The project would demonstrate that PAX-52 in bulk form and in the M112 configuration is comparable to Composition C4, as evaluated by the user community. The demonstration would show that PAX-52 meets or exceeds the physical qualities and performance of C4 in a direct comparison.

(U) The demonstration would show that PAX-52 has an environmental impact three orders of magnitude less than C4 by virtue of the use of nitramine HMX energetic instead of nitramine RDX.
(U) The project would demonstrate cost savings as a result of the high rate of the one-step continuous processing method and the elimination of process aids and water waste.

(U) Environmental Benefits: The technology reduces overall energy consumption by eliminating multiple steps of the manufacturing process. Far fewer facilities, logistics, and process steps have to be energy managed because the technology brings together both elements of C4 and M112 production into one facility. The use of this technology eliminates at least 1.5 million gallons of process water used in the slurry-coating batch method and the need to treat the contaminated waste water stream. It is estimated that there are significant energy savings achieved by removing the need to heat **Table 1**, cool, filter, vacuum, distill, and treat the waste process water, and other constituents of C4.

Table 1 (U) Estimated Energy Required to Heat a Batch of C4. *Estimates were made based on information taken from the Composition C4 specification and some reasonable assumptions were made to calculate the estimated total energy required just to heat a batch of C4 in a slurry kettle (steps 1-3, Figure 1.) The total heat energy amounts to about a quarter-ton of coal or about 35 gallons of No-2 heating oil. This estimate for heating of the components in a single batch is only a fraction of the total energy requirements through the entire 12-step process.*

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(U) Batch Process Energy Consumption

Process	Component	Mass (g)	Specific Heat ($J \cdot g^{-1} C^{-1}$)	Temp 1 (C°)	Temp 2 (C°)	Energy E (J)	Combustion Efficiency η_C Q_{out}/Q_{in}	Process Steam Efficiency (η_p) (approximate)	Total Efficiency (η_T) ($\eta_C \eta_p$)	Total Energy (J) E/η_T	Btu	Boiler HP	Btu/H	Time to Temp (h)
Lacquer Process 25°C to 65°C	Component 1		2.15	25	65	172965516	0.85	0.9	0.8	2.79E+08	264837	300	7.63E+05	0.347
	Component 2		1.95	25	65	11109375.3								
	Component 3		1.75	25	65	23559568.5								
	Component 4		1.67	25	65	6120589.01								
		Sub-Total				213755049								
Lacquer Process 65°C to 100°C	Component	Mass (g)	Specific Heat ($J \cdot g^{-1} C^{-1}$)	Temp 1 (C°)	Temp 2 (C°)	Energy E (J)	Combustion Efficiency η_C Q_{out}/Q_{in}	Process Steam Efficiency (η_p) (approximate)	Total Efficiency (η_T) ($\eta_C \eta_p$)	Total Energy (J) E/η_T	Btu	Boiler HP	Btu/H	Time to Temp (h)
	Component 1		2.15	65	100	151344826	0.85	0.9	0.8	2.44E+08	231733	300	7.63E+05	0.304
	Component 2		1.95	65	100	9720703.36								
	Component 3		1.75	65	100	20614622.4								
	Component 4		1.67	65	100	5355515.38								
		Sub-Total				187035667								
Coating Process	Component	Mass (g)	Specific Heat ($J \cdot g^{-1} C^{-1}$)	Temp 1 (C°)	Temp 2 (C°)	Energy E (J)	Combustion Efficiency η_C Q_{out}/Q_{in}	Process Steam Efficiency (η_p) (approximate)	Total Efficiency (η_T) ($\eta_C \eta_p$)	Total Energy (J) E/η_T	Btu	Boiler HP	Btu/H	Time to Temp (h)
	Component 1		4.184	25	100	1927665740	0.85	0.9	0.8	2.78E+09	2633240	300	7.63E+05	3.450
	Component 2		0.966	25	100	74335518.8								
	Component 3		0.966	25	100	73941165.9								
	Component 4		0.966	25	100	49392698.8								
		Sub-Total				2125335124								
Distillation Process	Component	Mass (g)	Specific Heat ($J \cdot g^{-1} C^{-1}$)	Temp 1 (C°)	Temp 2 (C°)	Energy E (J)	Combustion Efficiency η_C Q_{out}/Q_{in}	Process Steam Efficiency (η_p) (approximate)	Total Efficiency (η_T) ($\eta_C \eta_p$)	Total Energy (J) E/η_T	Btu	Boiler HP	Btu/H	Time to Temp (h)
	Component 1		4.184	100	150	1285110494	0.85	0.9	0.8	1.85E+09	1755493	300	7.63E+05	2.300
	Component 2		0.966	100	150	49557012.5					$E = \int_{°C0}^{°C1} g \cdot \frac{J}{g \cdot °C} \cdot dT°C$			
	Component 3		0.966	100	150	49294110.6								
	Component 4		0.966	100	150	32928465.9								
		Sub-Total				1416890083	Total Estimated Heating Energy (J)			5.15E+09				

(U) UNCLASSIFIED

(U) The technology eliminates the need for an organic solvent required by the batch process to solvate a polymer binder into a lacquer to enhance coating of RDX crystals. The silicone polymer used by this technology is environmentally friendly and non-toxic to humans. The solvent used by this lacquer process is n-Octane, a normal, straight-chained molecule that is highly flammable (flash point of 13 °C) and very toxic to aquatic life (3). The use of TSE would enable the waterless and solvent-less production of PAX-52 and the forming of M112 blocks within a single process.

(U) The technology replaces RDX with the less toxic HMX nitramine. Environmental benefits gained by the green formulation have international ramifications (4) wherever the DoD operates RTA's near aqueous ecosystems. In addition, since HMX is more energy dense, the new greener PAX-52 formulation has the added benefit of requiring less energetic solids to achieve performance results equivalent to or better than the Composition C4.

(U) Based on evidence of waterway contamination, the DoD is not currently engaged in an effort to completely control the development of RDX-based munitions; however, our neighbors in Canada have a different stance on RDX contamination of their waterways. "Although not a regulated substance in Canada, published guidance values for RDX in drinking water and for the protection of surface water resources are low, in the parts per billion range. Recently, RDX concentrations approaching published guidance values were detected in water bodies of interest in Canada triggering a decision to restrict the use of RDX-based items in certain sensitive areas of a Canadian Army RTA. Thus, there is a pressing need for studying potential alternatives to replace RDX based explosives, as the continued use of such ordnance would exacerbate the contamination of water bodies both in and around CAF RTA's. As the plastic explosive C4 is widely used and as it was demonstrated to lead to the accumulation of RDX in the environment, alternative options were studied to replace it in the Canadian inventory with a non-RDX formulation. Many options were considered and two options are presently under study, one based on pentaerythritol tetranitrate (PETN) and one based on octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine (HMX)." (4) Here PAX-52 directly satisfies the Canadians need for replacing RDX with HMX and it is envisioned that the DoD would follow suit when presented with this viable technology for producing a C4 replacement.

1.3 (U) TECHNOLOGY DESCRIPTION

(U) The technology demonstrated for the solvent-less production of PAX-52 by continuous manufacturing via the twin screw extrusion (TSE) process. TSE is a continuous manufacturing platform relying on screw elements assembled on two parallel shafts, with the two screws enclosed in a figure-8 barrel with openings for feeding of the solid and liquid ingredients, devolatilization via the application of vacuum and a die for the shaping of the energetic mixture. Some of the possible screw elements, including fully-flighted screw sections and kneading disks staggered in reverse and forwarding modes, are shown in **Figure 3**. For the processing of energetic formulations, the commonly employed mode is the fully-intermeshing and co-rotating mode. In this mode the screws rotate in the same direction and the flank of one screw wipes off the root of the second screw. This eliminates the possibility of dead zones, i.e., stagnant energetic material in the flow channel. The following are the general advantages of the twin screw extrusion process.

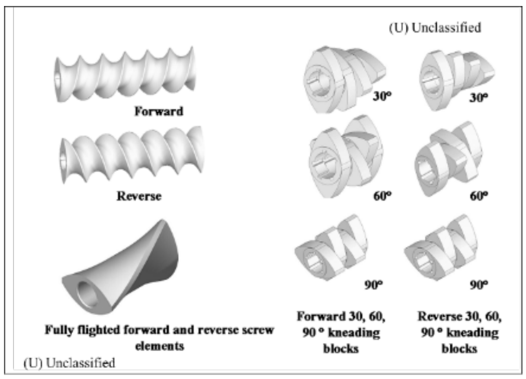


Figure 3 (U) Modular TSE Element

(U) GENERAL ADVANTAGES TSE OVER BATCH PROCESS

a. (U) Flexibility: The twin screw extrusion process is flexible because the screw elements can be selected and assembled on a shaft to generate a specific screw configuration targeting a given processing task. Mathematical modeling is used in the selection of the screw configuration and the associated die and operating conditions so that the processing of the energetic material occurs under safe conditions [71].

(U) One particular uniquely flexible extrusion technology that only the US Army has access to the Universal Extrusion Platform shown in **Figure 4**. This extrusion platform is unique because with a single machine it is possible to have single screw extrusion, twin screw extrusion with the screws co-rotating, twin screw extrusion with the screws counter-rotating, screws which are fully-intermeshed, screws which are not intermeshed, i.e., the "tangential configuration" or any degree of intermesh. The sensor and feed locations and the die type are interchangeable. During operation of the Universal Extrusion System, the die and the barrel can be detached fast to allow water impingement from an overhead deluge system.

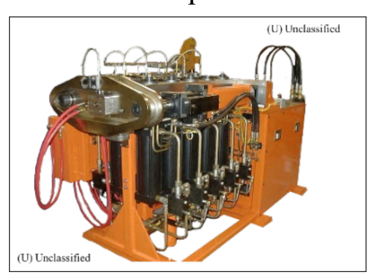


Figure 4 (U) UTSE Platform

b. (U) Inherent Safety: The residence time of the energetic time in a batch mixer is generally relatively long. For example, a typical mixing run in a vertical batch mixer can be 6 hours to mix a batch of 600 lb of propellant. This generates a production rate of less than 100 lb/hour. If there is an incident all of the 600 lb are involved. On the other hand, a similar production rate of 100 lb/hour can be achieved in a TSE with only about 15 lb of energetic in the extruder at any given time, with the possible damage of an incident limited to that of 15 lb.

c. (U) Surface to Volume Ratio: With the above example, the typical surface to volume ratio of the TSE would be conservatively at least one order of magnitude greater than that of the batch mixer. This provides much better temperature control in the TSE in comparison to a batch mixer.

d. (U) Mixing Capability: Superb distributive and dispersive mixing can be achieved with the twin screw extrusion process due to the much greater surface to volume ratio of the TSE. This superior mixing capability can be demonstrated experimentally [48, 51, 53, 55] and theoretically [45, 46, 59, 67, 77].

e. (U) Highly Viscous Suspensions: TSE can process highly viscous suspensions in comparison to batch mixers (the behavior of which approaches solid like behavior, i.e., viscoplasticity) whereas the batch mixing would likely result in stagnant regions and unmixed material when the shear viscosity of the suspension is relatively high.

f. (U) Process Control and Reproducibility: With a much greater surface to volume ratio and much better distributive and dispersive mixing capabilities [51, 53, 55, 77] twin screw extrusion provides better temperature control and a more homogeneous energetic product.

g. (U) Exposure Vulnerability: Exposure of production staff to process streams and the process, thus the vulnerability, is much less using TSE as compared to batch processes.

(U) TSE PROCESS APPLICATION:

(U) Continuous processing via the twin screw extrusion process is applicable to all processes that are currently applied on the batch mode. The current demonstration project is in the area of manufacturing of a hand-moldable high explosive called PAX-52. The application of the technology to other energetic formulations, especially those which involve significant concentrations of solvents, would introduce significant savings and environmental benefits at improved quality.

1.4 (U) PERFORMANCE AND COST ASSESSMENT

(U) The demonstrated technology essentially met all planned performance objectives with minor exceptions allowed for extenuating circumstances. The TSE mixing process achieved steady state without difficulty due to its well characterized silicone binder, particle size distribution and experienced process engineers. With only two ingredients fed into a TSE nearly 500 pounds of PAX-52 was compounded and extruded through a forming die to produce a well-formed, dense belt of material that met the specification for the M112 demolition block cross section. The bulk formed blocks were post processed into the M112 packaged configuration in specified packages, labels and double-sided adhesive tape then shipped to various test sites for characterization and performance testing **Figure 5**.

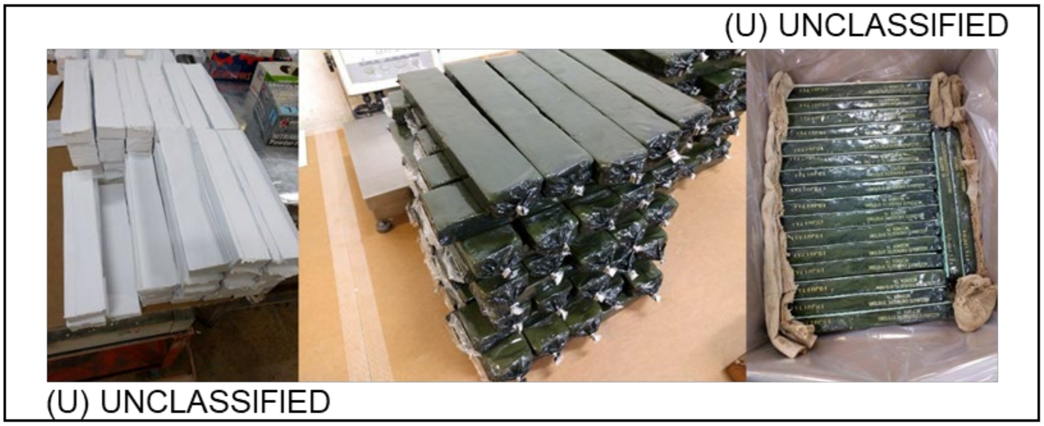


Figure 5 (U) M112 Production Steps. *Paper-wrapped blocks are stacked waiting to be unwrapped and inspected (right.) Regular copier paper proved to be more useful than thin waxed paper due to bond weight and no affinity for silicone to the paper surface. Blocks are slipped into Mylar bags which are heat-shrunk to form a secure container. The bag is closed with a metal c-clip and trimmed with a scissor (middle.) Lastly, 36 blocks are nested in a shipping box and packed with cellulose to prevent product movement during shipping (left.)*

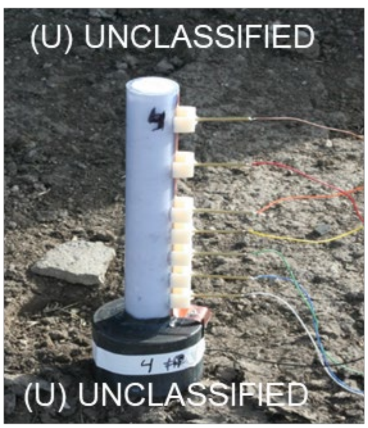


Figure 6 (U) Test Set-up.

(U) The PAX-52 material was characterized against a select panel of energetic qualification tests and found to be within tolerance for all key C4/M112 quality and performance specifications. In particular PAX-52 was found to be cap sensitive and powerful enough to pass the steel plate cutting test. In addition, the material was tested for detonation energy output with a detonation velocity and pressure comparable to C4. **Figure 6.**

(U) At the time of this writing no formal results were received from the deposition rate studies conducted by the DRDC, Valcartier, Canada. Informal email reports confirmed that PAX-52 met the planned performance objective by a measure far less than the success criterion of 1% combustion residue of RDX. Formal results will be published as an adjunct to this report at a later date and made available upon request.

(U) The continuous TSE technology and the novel PAX-52 formulation presented several key advantages over the existing production method. PAX-52 was demonstrated to be superior to C4 in hand-moldability through a range of temperatures (-40ºF and 140ºF) with no discernable difference in viscosity owing to the properties of silicone. PAX-52 was easily compounded to a high packing fraction and a desirable density using a mono-modal, large particle (200 µm) class of energetic powder. Post process analysis of PAX-52 showed a distinct shift to a bi-modal distribution (50 µm and 200 µm) due to a favorable *in-situ* shear reduction of larger particles into small. In addition, it was demonstrated that desirable formulations of PAX-52 could be produced using other methods, specifically: resonance acoustic mixing (RAM), planetary and sigma blade mixers; however, these methods were not modeled or optimized as part of this research.

(U) Historical data for the current C4/M112 production method were analyzed using time-value equations to set an average baseline cost of C4/M112 at $35 per block. Then an engineering model and cost assessment that used both empirical and industry standard data was developed for the demonstrated process technology, which estimated the baseline cost of PAX-52/M112 to be $67 per block. Though the single-step production demonstration validated manufacturing cost savings through reductions in labor, energy and process waste, and the several non-tangible cost benefits of PAX-52 related to hand-moldability, mission effectiveness, initiation reliability, manufacturing flexibility and other "….ilities"; the unit cost of nitramine HMX was found to be the overwhelming cost driver in the manufacture of PAX-52. HMX costs nearly three times RDX which makes PAX-52 a pricey alternative for the C4 product managers; however; the project looked past the obvious HMX-to-RDX price differential to investigate several over-shadowed cost burdens associated with the current technology that would be greatly reduced by the alternative.

(U) The Office of the Assistant Secretary of Defense OASD(A&S) partnered with the Army Armament Research, Development and Engineering Center (ARDEC) Explosives Development Branch (EDB) at Picatinny Arsenal on a pilot project to test DoD's Sustainability Analysis Methodology. This Sustainability Analysis evaluated PAX-52, an M112 demolition-block manufacturing process, as an alternative to the baseline C4 process. That baseline process consists of C4 production and M112 production processes, while the alternative system is a single-step process.[1]

(U) The Sustainability Analysis integrated Life Cycle Cost (LCC) estimating with Life Cycle Analysis (LCA) to compare the two technologies. Direct process information from ARDEC-EDB was combined with an integrated hybrid LCA database developed for DoD, which includes environmental flows related to economic activity for industrial sectors across the U.S. economy with integrated hybrid processes for transportation, energy, and other defense activities. Information from the baseline C4 and M112 processes was estimated using data provided by ARDEC-EDB. Use of the integrated hybrid LCA allowed for quantification of both direct impacts to the Army and impacts along the associated DoD supply chain. Additionally, the LCC estimates quantified internal costs to the Army over the life cycle on a per block basis (5).

(U) Program Managers must consider sustainability during systems acquisition and select more sustainable systems that meet performance requirements; have fewer negative impacts on resource availability, human health, and ecosystem quality; and have a lower Total Ownership Cost (TOC). Here, a Sustainability Analysis assessed life cycle costs (internal to DoD) and impacts on resource availability, human health, and environmental quality (external costs) of both technologies and found PAX-52 to be significantly more sustainable (5) **Figure 7**.

[1] Analysis presented here is from a preliminary report submitted for inclusion in this ESTCP final report. A comprehensive analysis will augment this report as an addendum.

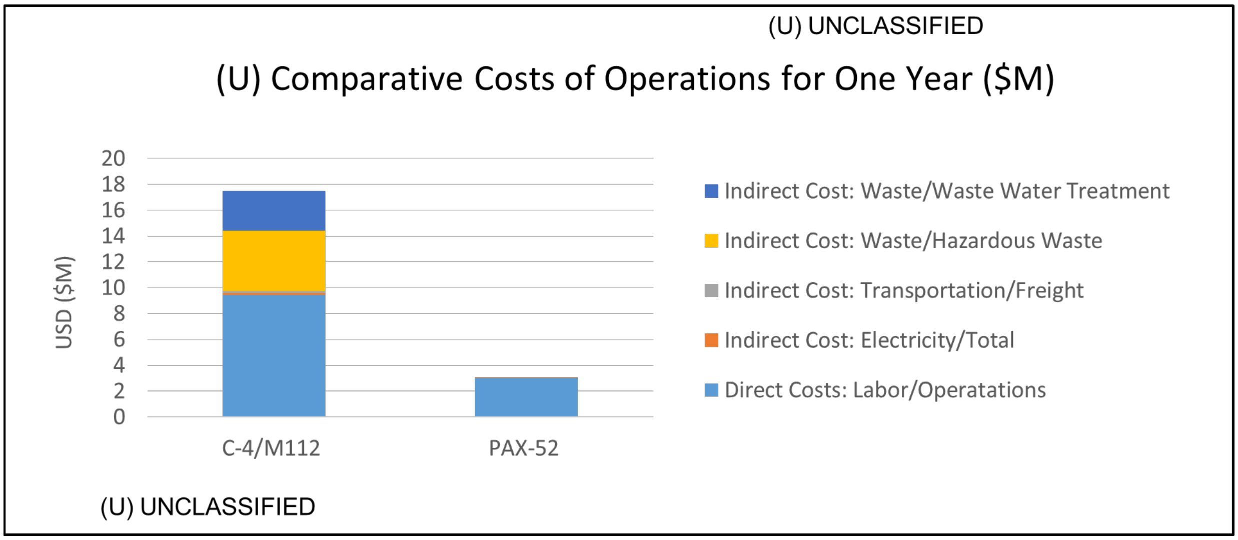


Figure 7 (U) Life Cycle Costs. *Estimate of selected annual operating costs for M112 production (both systems produce M112 blocks, but the baseline system is identified as C-4/M112 to reflect that it includes two steps). A preliminary estimate of annual operating costs was conducted. Costs are split into direct (labor) and indirect (electricity, transport, waste-treatment) costs. The largest driver of operating costs is labor for both alternatives (>50%; Figure 1). For the baseline (C-4) system, the remaining costs are driven by utilities and waste management. Labor costs for the baseline are based on engineering estimates and are higher than the alternative, given the difference in the number of processing steps. (This difference is also illustrated by the difference in electricity consumption: C-4 electricity is approximately five times higher than PAX-52 labor, though electricity is ~100 times lower than labor). Given the magnitude of labor costs, the overall analysis is sensitive to the engineering estimates used to quantify labor cost, and these should be explored further. The alternative process (PAX-52) reduces operating costs per pound of M112 to 20-30 percent of the baseline.* (5)

1.5 (U) IMPLEMENTATION ISSUES

(U) The demonstrated technology has had wide exposure in various venues including the 2017 ESTCP Symposium where this PI presented the technology to a host of military and defense industry stakeholders (**Figure 8**). The PAX-52 TSE technology demonstration garnered positive feedback from the current C4 user community including; combat engineers, ordnance demolition units and future munitions developers. Each had an interest to see PAX-52 at a readiness level that leads to a technology transfer. The technology highlighted for the stakeholders the feasibility of a one-step M112 production process and the PAX-52 product met all their performance and physical requirements; however, their full acceptance of the PAX-52/M112 was deferred until it matched the cost of the C4/M112.

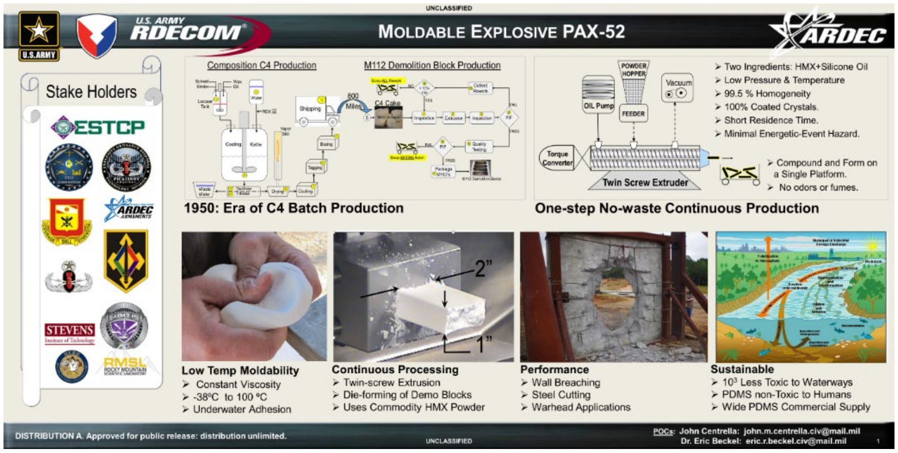


Figure 8 (U) Technology Presentation *The technology was presented in both the technical and poster sessions at the 2017 ESTCP Symposium in Washington, DC. The project received endorsements from the Program Executive Office-Ammunition (PEO-AMMO) and the Joint Insensitive Munitions Technology Program (JIMTP) and collaborated with a class of key researchers and user community stakeholders.*

(U) At the 2017 SERDP-ESTCP Symposium, Washington, DC, there were several presentations and discussions that touched upon the high cost of environmental risk factors built into emerging technologies, particularly munitions systems. Questions abounded: Who is supposed to pay for the high cost of environmental testing? When in the R&D life cycle should the DoD invest for environmental protection? Should major funding be applied in early product development with greater up front risk? Or should funding agencies cherry pick innovations with the hopes that no future environmental risks will emerge? Many questions from different perspectives. The answer to these questions is that the DoD should expect to pay a premium for environmental protection, sooner rather than later.

(U) The same reasoning is applied to the demonstration technology where the PAX-52 product is designed to be much less toxic to the environment than the current C4, but comes at a price premium to the product managers. The several important characteristics that the alternate technology provides in contrast to C4 (cold-weather moldability, initiation reliability, and continuous processing, etc) do not rank ahead of its higher cost. At some future point the DoD may use a different calculus that monetizes these several benefits and then decide to pay the premium for the superior product.

(U) Researchers at the Defense Research and Development Canada (DRDC) Valcartier Research Centre, are currently working with the director of Land Environment of the Department of National Defence to explore suitable RDX-free replacements for Composition C4. By their account, PAX-52 appears to Canada as a very good option for an RDX-free replacement of Composition C4. They further state DRDC hopes that this product will be supported and transitioned into a commercial option in the near future in the United States (6). If Canadian Armed Forces ultimately decide PAX-52 is the best candidate for C4 replacement and are willing to pay the price for the environmental benefits, then possibly the US will agree to transition the technology into production.

(U) The DoD would not have to "re-invent the wheel" to stand up a TSE production line. Twin screw extrusion technology is widely used by various industries. There are several providers of production-sized platforms and many more supplies of ancillary feed and handling systems. As well, Programmable Logic Control (PLC) and Supervisory Control and Data Acquisition (SCADA) computer programs can be readily purchased and customized to support PAX-52 production on any TSE production platform. The science of compounding and die forming high solids suspension, specifically energetics, is well-founded due to the work in the field by Stevens Institute of Technology. Various branches of the DoD have worked primarily with Stevens Institute of Technology since the mid-1980s in the development of the science and technology base of continuous manufacturing of energetic formulations using twin screw extrusion.

2 (U) INTRODUCTION

(U) The Composition C4 (C4) and M112 hand-moldable demolition blocks are used by all Department of Defense (DoD) services and consumed at the rate of several hundred thousand pounds per year. C4 is produced in a multi-step water slurry process that requires an organic, solvent-based lacquer to mix a cohesive binder and plasticizer with energetic RDX nitramine crystals. This multi-step process, developed in 1948, has been largely successful owing to the availability of cheap labor, cheap energy, and lower environmental protection standards; however, six decades later it is still time, energy, and labor intensive and generates 1.5 million gallons of aqueous waste per year (**Figure 9**). The output of the C4 production process is a loose powder (or cake) that is boxed and transported to an entirely different packaging facility where it is further processed into M112 demolition blocks via two additional processing steps.

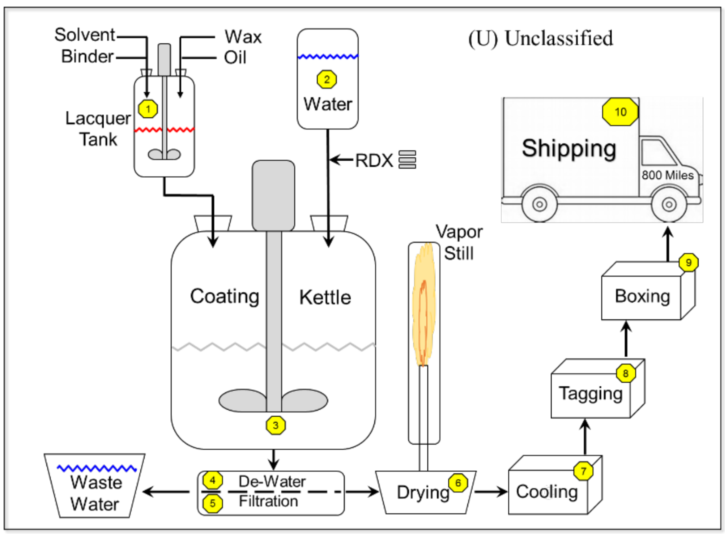


Figure 9 (U) Diagram of the Multi-step Process for Composition C4 Manufacturing. *The process requires large quantities of resources including energy for process heating and cooling, distilled water, waste water treatment, and solvent reclamation. Not depicted here, the process actually spans across several production plants and requires at least six consecutive labor shifts to complete a batch. The C4 is packaged in boxes and shipped 800 miles to be post-processed into the M112 demolition block configuration. It is there that the boxed C4 gets its moniker "cake" because it has a tendency to settle and partially consolidate during the long transport, becoming an agglomerate, cake-like formation.*

(U) This project's original principle investigator proposed a manufacturing process that would replace the combined 12-steps of manufacturing C4 and M112 demolition blocks with a single step by using a continuous twin-screw mixing and extrusion process (**Figure 10**). In conjunction, a new "green" formulation would be developed to replace the current C4 binder system with a silicone polymer, eliminate the use of an organic solvent, and preclude any waste-water streams. The green formulation would provide another environmental benefit gained by replacing the RDX with the more environmentally-friendly HMX in the new C4, presently named PAX-52.

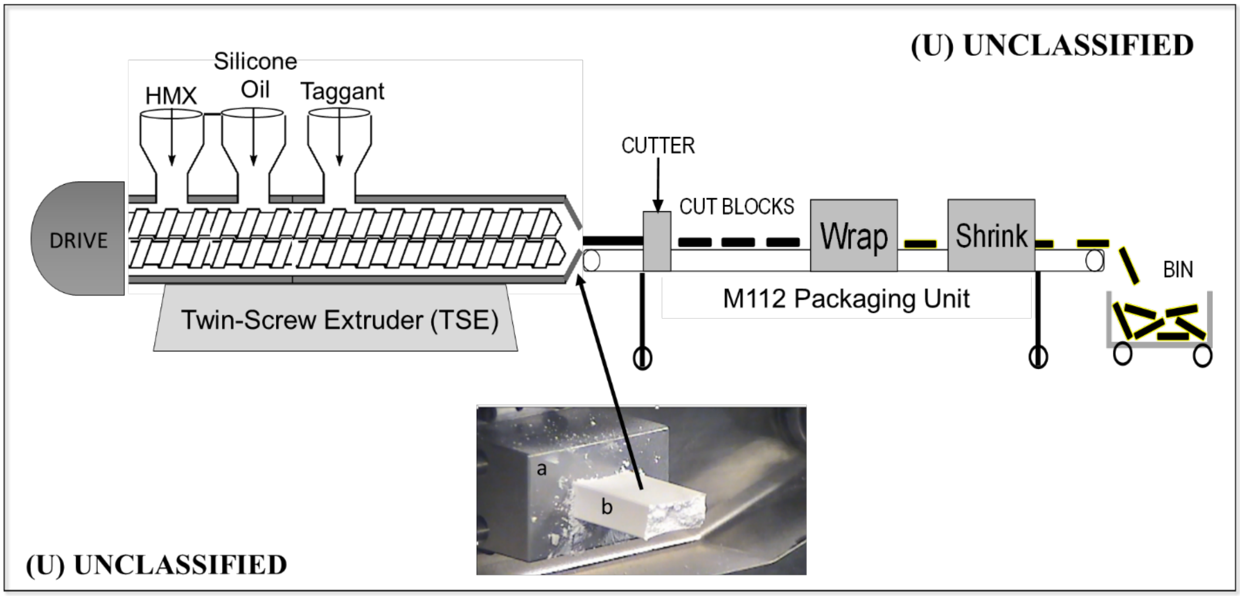


Figure 10 (U) Diagram of the Proposed Single-Step Manufacturing Process. *The single step production method would be demonstrated using a fully-intermeshing, co-rotating twin screw extruder (TSE) at Picatinny Arsenal to validate a continuous process for the manufacture of both PAX-52 and the M112 demolition block on a single production platform. The two ingredients; HMX, Silicone, (and Taggant;) are metered into the TSE, homogeneously mixed by co-rotating screws, and finally extruded through a forming die (a) into the cross-section of the M112 demolition block (b.) The downstream packaging of the extrudate would not be validated by this project; however, it is depicted here to emphasize the efficiency gained by bundling all elements of M112 production into a single package.*

(U) Silicone polymers are environmentally-inert and exhibit low surface tension for highly efficient wetting and coating of high explosives such as RDX and HMX. The silicone polymer type and the particle size distribution of the energetic particles can be tailored to generate an easily processed PAX-52 formulation that can be mixed, deaerated, and pressurized in any twin screw extruder. With a specially designed forming die directly attached to the twin-screw discharge port, the mixed material can be shaped to any desired profile, here a simple rectangular cross-section specified for the M112. Though the continuous TSE process is fairly straightforward there was required much research that brought the project to the point of a demonstration.

2.1 (U) BACKGROUND

(U) Composition C4 (simply, C4) is currently produced in a multi-step water slurry process that requires an organic solvent-based lacquer binder that is applied to RDX explosive. The solvent used by this lacquer process is n-Octane, a normal, straight-chained molecule that is highly flammable (flash point of 13 °C) and very toxic to aquatic life (3). The current process generates a water waste stream contaminated with this lacquer solvent and residual formulation ingredients. During the C4 manufacturing process, a portion of the solvent is distilled for recovery while the remainder is evaporated and vented to the atmosphere followed by collection of contaminated process water that is directed to water treatment. After the RDX is coated, the n-octane remains in the process water until it is partially removed by distillation and evaporation during a subsequent process step. The remainder of the n-octane and residual binder ingredients and uncoated RDX in the process water are transferred to water treatment. In this project it would be demonstrated that a silicone polymer can be used as the solvent-free binder to eliminate the toxic organic solvent as well as the contaminated water streams from the manufacture of C4 and M112. The use of twin screw extrusion would enable the waterless and solvent-less production of PAX-52 and the forming of M112 blocks within a single process.

(U) Past efforts to improve C4 manufacturing technique, reliability, or performance have focused on formulation adjustment and modification. The DoD has invested in capital upgrades to production facilities and equipment over the decades to ensure the longevity of the process to guarantee the supply of C4 to its services. None of the upgrades or investments have seriously questioned nor addressed the production water-slurry coating process that can generate sixteen-hundred gallons of contaminated water waste per batch of material. There currently exists no approach that eliminates or mitigates the waste generated from the C4 manufacturing process other than the legacy on-site waste-water treatment plant, which alone burdens the environment and adds an additional cost burden to the production of C4. In this project it would be demonstrated that a continuous twin screw mixing process eliminates the use of water in a slurry technique, whereby HMX crystals are held in suspension until fully coated. It would be shown that the low viscosity silicone polymer mixes with and coats the dry crystals to form a colloid without solvent or water. This single-step approach provides the DoD with a solution to eliminate the use of the large quantities of process water and the need for water waste treatment.

(U) C4 is approximately 90 percent RDX by weight. Though the RDX nitramine content in the formulation provides sufficient energy to effectively defeat intended targets, its use comes with a risk to the environment especially at test ranges where there are continual and concentrated practice missions using M112 demolition blocks. RDX is stable, "persistent in the environment, water soluble, and it moves relatively rapidly towards surface and groundwater bodies in ranges and training areas (RTAs.) Many studies were conducted both in Canada and in the USA to better understand the nature and extent of contamination in RTA's. In fact, RDX is the key contaminant that has triggered the closure of a large RTA in the USA." (4) (7) (8) RDX is considered an environmental and potential human health hazard by the EPA; (9) furthermore, "the US environmental Protection Agency (EPA) has assigned RDX as a possible human carcinogen. It targets the nervous system and can cause seizures in humans and animals when large amounts are inhaled or ingested. RDX eco-toxicity has also been extensively studied in Canada and it showed toxicity to various receptors. The North-American RDX guidance value and threshold in water bodies are low, in the parts per billion magnitudes. This means that whenever RDX is detected in a water bodies, it triggers a potential risk. Recently RDX levels approaching threshold levels were detected in water bodies of interest in Canada, triggering a moratorium on the use of RDX-based items in certain areas." (4) As of 2007, RDX had been identified at more than 30 sites on the EPA National Priorities List (NPL.) (14) It follows that the DoD will have to get ahead of the environmental hazard of RDX in munitions or be faced with similar moratoria on ranges all across the US and in allied countries.

(U) The project would demonstrate a greener formulation produced by TSE that is based on the high explosive HMX instead of RDX. HMX provides high energy as well as exhibiting low transport potential in the environment. "In comparison to RDX, HMX offers over one order of magnitude reduction in carcinogenic potency and an order of magnitude increase in clean-up threshold in soil. HMX is a nitramine and has a molecular structure very similar to RDX but it is much less soluble and toxic. EPA drinking water threshold for RDX is 2 ppb while the HMX threshold is 400 ppb or 200 times less stringent. The water solubility of HMX is also ten times lower than that for RDX. Therefore, using HMX instead of RDX would reduce the associated risk by at least three orders of magnitude, as the impact of a contaminant is equivalent to its effect, exposure and fate." (4) The immediate benefits gained by DoD by the availability of HMX-based PAX-52 are the elimination of a toxicity risk to end-users and mitigation of a hazard to ecosystems that suffer test ranges.

2.2 (U) OBJECTIVES OF THE DEMONSTRATION

1. (U) The purpose of this project demonstration was to show that an analogue composition C4 explosive, called PAX-52, can be produced by compounding only two ingredients[2] in a twin-screw, continuous mixing process. Furthermore, that the compounded material can be extruded through an inverted forming die to the cross-section dimensions specified for the Army's M112 demolition block and that the material, PAX-52, would meet C4/M112 quality specifications for plasticity and performance (MIL-DTL-50523A.) (2) (1) See Performance Objectives **Table 2 Page 28**.

2. (U) The demonstration would have shown that PAX-52 meets or exceeds sensitivity and performance characteristics of C4. The demonstration would have shown that the TSE PAX-52 product would not undergo a detonation response to heat ignition while confined.

3. (U) The demonstration would show that there are no by-products created by the TSE process. Only two ingredients (and a taggant) are required to produce PAX-52. All the energetic solids (HMX) and binder oil (PDMS) are metered into the TSE mixing process and compounded as the final product, PAX-52. No chemical reactions are required to achieve a chemically stable product. Though it is required by law that a taggant (Ex. DMDNB), which is designed to volatize as agent of detection, be added to all plastic explosives; here the taggant would not considered as part of the formulation, nor would it contribute to the generation of by-products.

4. (U) The demonstration would show that there is no waste stream once the process achieves steady-state production. Some scrap material is produced initially to ramp up the process to steady-state and some is produced during process shut down. The TSE barrel has to be purged of material anytime there is an interruption in production to prevent material seizure in the mixing sections. For the demonstration, these small quantities of material associated with the start-and-stop of the process would not considered a waste stream.

5. (U) The demonstration would show that there would be no process aids used in the manufacture of PAX-52. Use of a solvent in the mix to keep viscosity low through the mixing sections is not required in this process. PDMS is used at a significant weight percent to allow thorough mixing without the inherent rise in temperature and pressure within the TSE barrel. Silicone has a low surface tension which causes it to mix freely into a mix of solid particles.

[2] Though it is required by law that a taggant (Ex. DMDNB), which is designed to volatize as agent of detection, be added to all plastic explosives; here the taggant would not considered an ingredient of the formulation.

6. (U) The demonstration would show that the PAX-52 process does not require external heating or cooling to maintain a steady-state level of production. Little heat is generated by shear friction due to low viscosity within the mixing section. Once the system equilibrates to the TSE platform, any process heat generated by mixing is carried away by product or dissipated through the mass of the equipment. The formulation would prove to be flexible through a range of operational temperatures owing to the properties of silicone and a facile mixing method.

7. (U) The demonstration would show that the TSE PAX-52 product would be homogeneous and would consist of the appropriate weight percent of HMX and PDMS as required by the M112 military specification. The product would be shown to have a mixedness index greater than 0.99 (10). The material would behave similar to C4 through hand-moldability, friability, tackiness, and transferability.

8. (U) The project proposed to demonstrate that PAX-52 material can be packaged in the M112 configuration using an alternate method, with an allowance for off-site transportation. Due to cost and schedule constraints, the proposed one-step process whereby PAX-52 M112 demolition blocks are extruded and packaged on a single production line, could not be demonstrated. Market research has shown that an M112 packaging unit would be impossible to implement on site. Employing an existing GOCO packaging line would present unmanageable risk to both the Project and the GOCO.

9. (U) The project would demonstrate that PAX-52 in bulk form and in the M112 configuration is comparable to C4, as evaluated by the user community. The material would be used in a variety of qualitative tests conducted by various military units that are familiar with the characteristics of C4, both good and bad. The demonstration would show that PAX-52 meets or exceeds the physical qualities and performance of C4 in a direct comparison.

10. (U) The demonstration would show that PAX-52 has an environmental impact three orders of magnitude less than C4 by virtue of the use of nitramine HMX energetic instead of nitramine RDX. Furthermore, it would be demonstrated that the production and handling of PAX-52 is less toxic to humans.

11. (U) The project would demonstrate cost savings as a result of the high rate of the one-step continuous processing method and the elimination of process aids and water waste. It was envisioned that the qualities of PAX-52 and M112 demolition block would also be improved. Processing under ambient temperature conditions minimizes the heat degradation of product and improves consistency. Furthermore, the well-demonstrated distributive and dispersive mixing and devolatilization capabilities of the TSE should lead to improved homogeneity and quality (11). Less handling, packaging, and transportation would further improve process safety while introducing additional cost savings and minimizing the potential for product contamination.

12. (U) There would be several benefits gained by the use of continuous processing technology for the production of PAX-52 and M112 demolition blocks. Not all of the gains could be measured

directly due to restricted access to proprietary manufacturer's information, but would be stated in general terms and estimated by percentages.

Table 2 (U) Performance Objectives *These presented in the demonstration plan were based mostly on the specifications for quality and performance of C4. The technology demonstration had to meet at a minimum all C4 standards for it to have any chance of being accepted as a feasible production method to produce a military grade explosive. The project had high confidence in some of the objectives due to earlier experiments and previous experience with explosives production using TSE. A couple qualitative objectives were included in the demonstration but there was not a clear plan for how they would be tested. The proposed user-evaluations presented significant risk without commitments from individuals from the user communities (combat engineers and explosive ordnance disposal EOD) to manage those efforts, which alone were fairly significant tasks and completely outside the scope of this project.*

(U) Quantitative Performance Objectives			
Performance Objective	**Section Ref**	**Data Requirements**	**Success Criteria**
PAX-52 shall be produced continuously by a steady state process for at least three hours.	2.2-1	1. Time record. 2. Temperature and pressure records.	Recorded production run exceeding three hours. Recorded feedback data within three standard deviations for pressure, temperature, and torque.
PAX-52 consists of only two ingredients. Taggant not accounted for in the formulation.	2.2-1	Composition analysis	Detection of HMX at appropriate percent by weight.
Demolition charge cross-section	2.2-1	MIL-DTL-50523B, drawing 9204248.	Measurement of 2"x1" with side concavity allowance.
M112 Physical properties - Plasticity	2.2-1	Penetrometer MIL-DTL-50523B 3.2.1.1	Force resistance of not less than 2.0 pounds and not greater than 8.0 pounds
M112 Specific gravity	2.2-1	Archimedes' water method MIL-DTL-50523B 4.4.2	Specific gravity greater than 1.50
Energy output	2.2-2	Plate-cutting test MIL-DTL-50523B 4.4.3	Cut a steel witness plate completely into two separate sections.
Physical sensitivity	2.2-2	ESD (AOP-7201.03.001) BAM Friction (AOP-7 201.02.006) ERL Impact (AOP-7 201.01.001)	ESD: No Go < 0.25 Joules BAM Friction: 10 No Go greater than 200 Newtons ERL Impact: 50% reaction height greater than 75 cm Cap: Detonation reaction
Shock sensitivity	2.2-2	Cap Sensitivity (TB700-2 Para 5.6a). IHE Gap Test (AOP 201.04.005)	Sustained Detonation Response Attenuation Gap between 161-171 cards.
Detonation velocity Energetic performance	2.2-2	Detonation velocity test Plate dent test	Velocity greater than 7.5 km/sec At 99% TMD Pressure greater than 24 kBar at 99% TMD

Product response to ignition	2.2-2	1. VCCT (AOP-7 202.01.002) 2. Small Scale Burning Test (TB700-2 Para. 5.4a)	1. Pass: No detonation response 2. Pass: No detonation response
PAX-52 chemically stable	2.2-2	1. Vacuum Thermal Stability (40 h @ 100 ºC - AOP-7; US/202.01.022) 2. Thermal Stability (48 h @ 75 ºC - AOP-7; US/202.01.013). 3. Aging Study	1. Pass: no significant decomposition or off-gassing. 2. Pass: no exothermic reactions. 3. No accumulated exudation of oil.
Homogeneity	2.2-7	Composition analysis	Mixedness index greater than 0.99
Environmental impact	2.2-10	Deposition rate test	Less than 1% by weight of RDX accumulation.
HMX workplace exposure	2.2-10	OSHA Arbitrary Target Concentration[3]	Less than 0.2 mg/m^3
Qualitative Performance Objectives			
Performance Objective		**Data Requirements**	**Success Criteria**
Packaging	2.2-8	Contractor Inspection Report	MIL-DTL-50523A, drawing 9204248.
User evaluation	2.2-9	Color Manual Moldability Tackiness Surface oil transfer.	No noticeable change in the quality of the product during any field demonstration conditions.

2.3 (U) ENVIRONMENTAL BENEFITS:

a. (U) The technology reduces overall energy consumption by eliminating multiple steps of the manufacturing process. Far fewer facilities, logistics, and process steps have to be energy managed because the technology brings together both elements of C4 and M112 production into one facility.

b. (U) The use of this technology eliminates at least 1.5 million gallons of process water and the need to treat the contaminated waste stream. It is estimated that there are significant energy savings achieved by removing the need to heat, cool, filter, vacuum, distill, and treat the waste process water, and other constituents of C4 (**Table 3**).

Table 3 (U) Estimated Energy Required to Heat a Batch of C4. *Estimates were made based on information taken from the Composition C4 specification and some reasonable assumptions*

[3] There is no official Occupational Safety and Health Administration (OSHA) Personnel Exposure Limit (PEL) established for HMX. OSHA recommended an arbitrary concentration target of 0.2 milligrams per cubic meter (mg/m^3). Also, there is no published American Conference of Governmental Industrial Hygienists (ACGIH) threshold limit values (TLV) for a time weighted average (TWA) for HMX (Octogen)

were made to calculate the estimated total energy required just to heat a batch of C4 in a slurry kettle (steps 1-3, Figure 9.) The total heat energy amounts to about a quarter-ton of coal or about 35 gallons of No-2 heating oil. This estimate for heating of the components in a single batch is only a fraction of the total energy requirements through the entire 12-step process.

(U) UNCLASSIFIED

(U) Batch Process Energy Consumption

Process	Component	Mass (g)	Specific Heat ($J*g^{-1}C^{-1}$)	Temp 1 (C^o)	Temp 2 (C^o)	Energy E (J)	Combustion Efficiency η_C Q_{out}/Q_{in}	Process Steam Efficiency (η_p) (approximate)	Total Efficiency (η_T) ($\eta_C \eta_p$)	Total Energy (J) E/η_T	Btu	Boiler HP	Btu/H	Time to Temp (h)
Lacquer Process 25°C to 65°C	Component 1		2.15	25	65	172965516	0.85	0.9	0.8	2.79E+08	264837	300	7.63E+05	0.347
	Component 2		1.95	25	65	11109375.3								
	Component 3		1.75	25	65	23559568.5								
	Component 4		1.67	25	65	6120589.01								
		Sub-Total				213755049								
Lacquer Process 65°C to 100°C	Component 1		2.15	65	100	151344826	0.85	0.9	0.8	2.44E+08	231733	300	7.63E+05	0.304
	Component 2		1.95	65	100	9720703.36								
	Component 3		1.75	65	100	20614622.4								
	Component 4		1.67	65	100	5355515.38								
		Sub-Total				187035667								
Coating Process	Component 1		4.184	25	100	1927665740	0.85	0.9	0.8	2.78E+09	2633240	300	7.63E+05	3.450
	Component 2		0.966	25	100	74335518.8								
	Component 3		0.966	25	100	73941165.9								
	Component 4		0.966	25	100	49392698.8								
		Sub-Total				2125335124								
Distillation Process	Component 1		4.184	100	150	1285110494	0.85	0.9	0.8	1.85E+09	1755493	300	7.63E+05	2.300
	Component 2		0.966	100	150	49557012.5					$E = \int_{°C0}^{°C1} g \cdot \frac{J}{g \cdot °C} \cdot dT°C$			
	Component 3		0.966	100	150	49294110.6								
	Component 4		0.966	100	150	32928465.9								
		Sub-Total				1416890083	Total Estimated Heating Energy (J)			5.15E+09				

c. (U) The technology eliminates the need for an organic solvent required by the batch process to solvate a polymer binder into a lacquer to enhance coating of RDX crystals. The silicone polymer (PDMS) used by this technology is environmentally friendly with no known toxicity hazard to humans (12). The solvent used by this lacquer process is n-Octane, a normal, straight-chained molecule that is highly flammable (flash point of 13 °C) and very toxic to aquatic life (3). The current process generates a water waste stream contaminated with this lacquer solvent and residual formulation ingredients. During the C4 manufacturing process, a portion of the solvent is distilled for recovery while the remainder is evaporated and vented to the atmosphere followed by collection of contaminated process water that is directed to water treatment. After the RDX is coated, the n-octane remains in the process water until it is partially removed by distillation and evaporation during a subsequent process step. The remainder of the n-octane and residual binder ingredients and uncoated RDX in the process water are transferred to water treatment. In this project it would have been demonstrated that a silicone polymer can be used as the solvent-free binder to eliminate the toxic organic solvent as well as the contaminated water streams from the manufacture of C4 and M112. The use of twin screw extrusion would enable the waterless and solvent-less production of PAX-52 and the forming of M112 blocks within a single process.

d. (U) The technology replaces RDX with the less toxic HMX nitramine. Environmental benefits gained by the green formulation have international ramifications (4) wherever the DoD operates RTA's near aqueous ecosystems. **Figure 11** In addition, since HMX is more energy dense, the new greener (U) PAX-52 formulation has the added benefit of requiring less energetic solids to achieve performance results equivalent to or better than the C4.

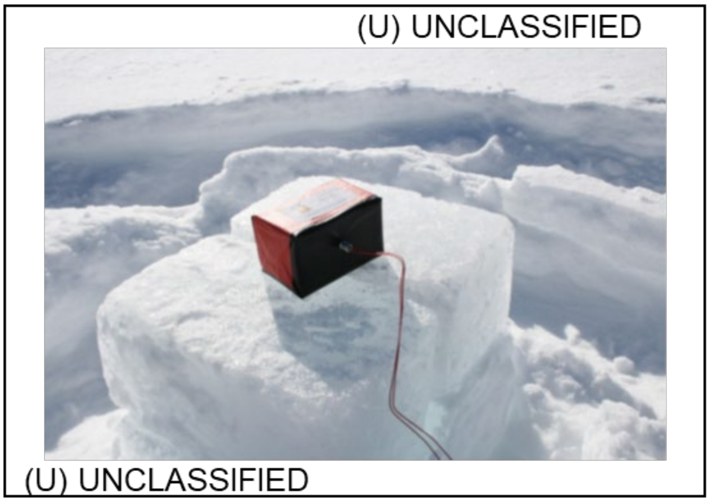


Figure 11 (U) Environment Deposition Rate Experiments. *The Canadians have found that RDX in Composition C4 has a deposition rate ten times greater than found in other munitions. Researchers at the DRDC use a test method where RDX deposits can be collected from pristine snow pack after detonation. The project would provide the DRDC with PAX-52 for candidacy trials for a C4 replacement in Canadian armed forces.*

e. (U) The technology eliminates the need to ship C4 cake material 800 miles by ground transit, thereby saving fuel and vehicle emission to the environment.

2.4 (U) REGULATORY DRIVERS

(U) The US government has known of the environmental impact of RDX for decades. The DoD included RDX as having a probable high impact and ranked it as an actionable contaminant in its Emerging Contaminants Program, (13) which brings agency auditing and prohibitions to bear on contaminated sites. Other regulatory agencies are increasingly aware of contamination of waterways due to RDX released by production waste and expended munitions.

(U) "The Army requires the capability to sustain Warfighter training and the testing, production, storage and demilitarization of munitions by preventing or controlling their environment, safety and occupational health (ESOH) impacts. Hazardous materials contained in ordnance may affect human health and the environment at various points throughout their lifecycle (research, development, test and evaluation, manufacturing, storage, use, demilitarization and cleanup of unexploded ordnance (UXO) or munitions constituents on ranges). This impacts all Army ammunition plants, live-fire ranges, static test facilities and most arsenals and depots. The technical approach to this requirement is to synthesize, characterize, test and scale-up secondary explosives as alternatives to RDX and TNT." (14). This technology demonstration directly supports the Army initiative by replacing RDX in the M112 demolition block with a less toxic nitramine energetic material.

(U) The EPA determined RDX to be a possible human carcinogen based on animal studies which found adenomas and carcinomas in female mice. Exposure of the general population is minimal but workers in DoD manufacturing and loading plants who work with RDX can potentially breathe RDX dust or get it on their skin. It is known that soldiers can also breathe fumes of burning RDX (sometimes by burning C4 for heating coffee or meals). Since 1988, no further data has been added to increase the RDX carcinogenicity classification; however, a toxicological review of RDX in support of the EPA's Integrated Risk Information System (IRIS) (15) was completed in August 2018 and the EPA plans to update its toxicity benchmarks and health risk assessment for RDX.

(U) The EPA reports other findings from different studies which indicate that RDX may bio accumulate in plants and could be a potential exposure route to herbivorous wildlife; that low soil sorption coefficient (Koc) values indicate that RDX is not significantly retained by most soils and can migrate to groundwater; that RDX can migrate through the vadose zone and contaminate underlying groundwater aquifers; and that RDX does not evaporate from water readily as a result of its low vapor pressure (9). RDX has been found in water and soil near some ammunition plants and storage areas. Those who live near these areas can be exposed by drinking contaminated water or touching contaminated soil in the area. People who eat crops grown in or animals raised on contaminated soils may be exposed to RDX (**Figure 12**).

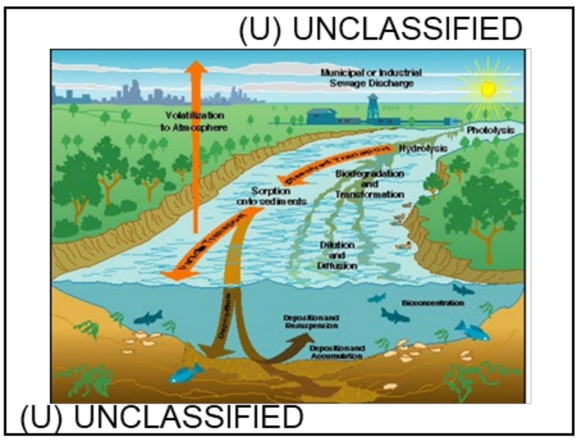


Figure 12 (U) Fate of Contaminants in a River

(U) An ongoing waste water treatment program at Holston AAP is an expansion of the existing Industrial Waste Water Treatment Facility (IWWTF), which implements biological degradation of explosives contaminated process wastewater resulting from the production of explosives. The objective of the program is to enhance treatability of the waste water to keep Holston AAP in compliance with the National Pollution Discharge Elimination System NPDES permit, which contains permit limits on a variety of chemical constituents, including RDX. The project has evolved over increased scrutiny of RDX discharge. In 2006 the EPA released a human health advisory limit of 2 ppb in drinking water per 70 kg adult for exposure over the course of a lifetime. Then, in May 2007, the Tennessee Department of Environment and Conservation (TDEC) modified Holston's NPDES discharge permit with a 12.2 pound per day discharge limit and a 5-year compliance schedule to be completed in May 2012, as a domestic drinking water source would be coming online downstream from HSAAP's outfall. Following investments in R&D studies and pilot testing, the Army chose to pre-treat the RDX waste water with Reverse Osmosis at Holston

AAP. However, originally unforeseen fugitive RDX sources proved to circumvent the RDX pretreatment system, and Holston continued to exceed the 12.2 pound per day RDX outfall limit by May 2012. Consequently, the Army determined it would be best to treat all the water at the existing IWWTF, through enhanced treatment capacity. In AUG 2014, TDEC required the Army and BAE enter a Compliance Agreement identifying 14 tasks for IWWTF expansion with the last to complete by FEB 2020 (16).

(U) Our neighbors in Canada have a different stance on RDX contamination of their waterways. "Although not a regulated substance in Canada, published guidance values for RDX in drinking water and for the protection of surface water resources are low, in the parts per billion range. Recently, RDX concentrations approaching published guidance values were detected in water bodies of interest in Canada triggering a decision to restrict the use of RDX-based items in certain sensitive areas of a Canadian Army RTA. Thus, there is a pressing need for studying potential alternatives to replace RDX based explosives, as the continued use of such ordnance will exacerbate the contamination of water bodies both in and around CAF RTA's. As the plastic explosive C4 is widely used and as it was demonstrated to lead to the accumulation of RDX in the environment, alternative options were studied to replace it in the Canadian inventory with a non-RDX formulation. Many options were considered and two options are presently under study, one based on pentaerythritol tetranitrate (PETN) and one based on octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine (HMX)." (4). Here PAX-52 directly satisfies the Canadians need for replacing RDX with HMX and it is envisioned that the DoD would follow suit when presented with this viable technology for producing a C4 replacement.

3 (U) TECHNOLOGY

3.1 (U) TECHNOLOGY DESCRIPTION[4]

(U) The technology demonstrated for the solvent-less production of C4 is the continuous manufacturing via the twin screw extrusion process. Prior to 1980s there was one important demonstration of the continuous processing of energetic materials [1]. This continuous processing effort relied on the use of a single screw/kneader (Baker-Perkins co-kneader, UL-200) and could process ANB-3254 propellant at a rate of up to 4,000 lb/hour. The twin screw extrusion process represents a more difficult challenge than the single screw kneader and its implementation for the processing of energetic materials required the development of multiple important scientific and technical capabilities prior to its implementation at relatively large scale. Various branches of the DoD and DoD contractors have worked primarily with Stevens Institute of Technology, that was funded initially by the Strategic Defense Initiative Innovative Science and Technology Office and then the Ballistic Missile Defense Organization since the mid-1980s in the development of the science and technology base of continuous manufacturing of energetic formulations using twin screw extrusion (over 180 contracts and grants). The key accomplishments in the development of the science and technology base for the processing of energetic using the twin screw extrusion process were the following (some of the pertinent key publications are included):

a. (U) Development of on-line and off-line rheometers and rheological characterization methods for the assessment of the flow and deformation behavior and, hence, the processability of energetic formulation. The key deliverable was the development of the abilities to determine the parameters of wall slip and shear viscosity for highly filled energetic suspensions and gels [2-35] so that processability could be assessed and mathematical modeling and simulation enabled.

b. (U) Installation of rheological characterization facilities at the US Army, NSWC and DoD contractor sites [14-18].

c. (U) Development of methods for the characterization and simulation of the goodness of mixing of the ingredients of the energetic formulation upon continuous mixing in TSEs [36-57].

d. (U) Development of methods for the preparation of processing simulants

e. (U) Development of analytical and Finite Element Method based simulation source codes for the prediction of the thermo-mechanical history in extrusion and die flows [36, 37, 44-46, 56-76].

f. (U) Development of specialized extrusion hardware for the flexible processing of energetic formulations including the Universal Extrusion platform and the mini TSE that are both installed at Picatinny Arsenal US Army base [71].

[4] **TECHNOLOGY DEVELOPMENT** was written by Dr. Dilhan Kalyon, SIT. All references [in brackets] in this section are located in **Section 10.1**

(U) Twin screw extrusion is a continuous manufacturing platform relying on screw elements assembled on two parallel shafts, with the two screws enclosed in a figure-8 barrel with openings for feeding of the solid and liquid ingredients, devolatilization via the application of vacuum and a die for the shaping of the energetic mixture. **Figure 13** shows the different types of TSEs, including fully-intermeshing co-rotating, fully-intermeshing counter-rotating and counter-rotating tangential TSEs [69, 72, 76, 77]. Some of the possible screw elements including fully-flighted screw sections and kneading disks staggered in reverse and forwarding modes are shown in **Figure 14.** For the processing of energetic formulations the commonly employed mode is the fully-intermeshing and co-rotating mode. In this mode the screws rotate in the same direction and the flank of one screw wipes off the root of the second screw. This eliminates the possibility of dead zones, i.e., stagnant energetic material in the flow channel. The following are the general advantages of the twin screw extrusion process.

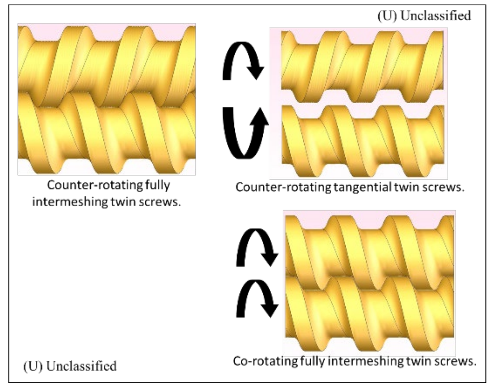


Figure 13 (U) Types of Twin Screw Extruder Configurations.

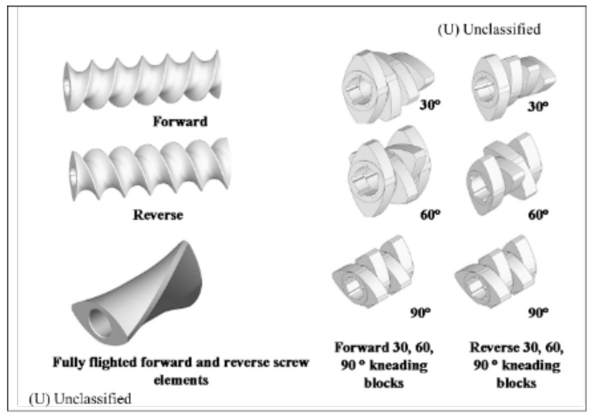


Figure 14 (U) Modular TSE Elements

a. (U) **Flexibility:** The twin screw extrusion process is flexible because the screw elements can be selected and assembled on a shaft to generate a specific screw configuration targeting a given processing task [38, 45, 46, 55, 69, 71, 72, 73, 77]. Any time a new screw configuration is assembled, the extruder is a new mixer and continuous processor. A screw configuration is shown in **Figure 15** with the screw sections assembled prior to the extrusion run. Mathematical modeling is used in the selection of the screw configuration and the associated die and operating conditions so that the processing of the energetic material occurs under safe conditions [71]. **Figure 16** shows typical degree of fill and the pressure distributions as a function of the flow rate.

Figure 15 (U) Assembly of Twin Screws before Production

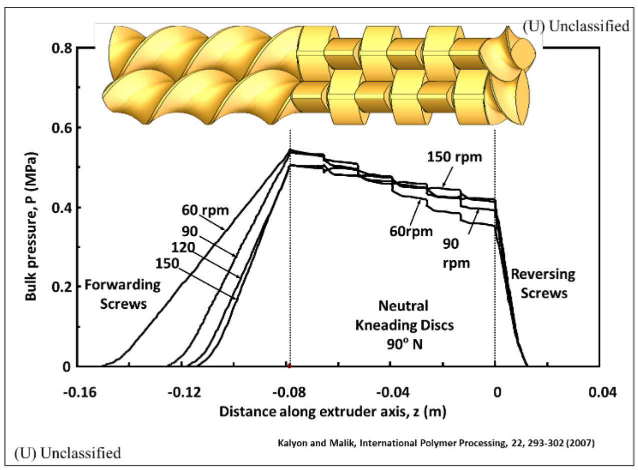


Figure 16 (U) Pressure along Screw Sections.

(U) One particular uniquely flexible extrusion technology is the Universal Extrusion Platform shown in **Figure 17** [71]. This extrusion platform is unique because with a single machine it is possible to have single screw extrusion, twin screw extrusion with the screws co-rotating, twin screw extrusion with the screws counter-rotating, screws which are fully-intermeshed, screws which are not intermeshed, i.e., the "tangential configuration" or any degree of intermesh. The sensor and feed locations and the die type are interchangeable. During operation of the Universal Extrusion System, the die and the barrel can be detached fast to allow water impingement from an overhead deluge system.

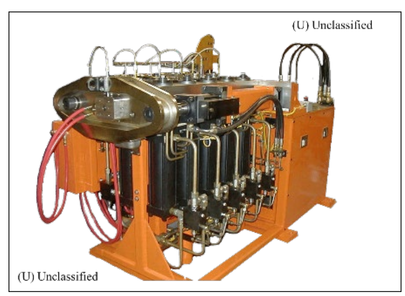


Figure 17 (U) UTSE

b. (U) Inherent safety of twin screw extrusion in comparison to batch processing: The residence time of the energetic time in a batch mixer is generally relatively long. For example, a typical mixing run in a vertical batch mixer can be 6 hours to mix a batch of 600 lb of propellant. This generates a production rate of less than 100 lb/hour. If there is an incident all of the 600 lb are involved. On the other hand, a similar production rate of 100 lb/hour can be achieved in a TSE with only about 15 lb of propellant in the extruder at any given time, with the possible damage of an incident limited to that of 15 lb.

c. (U) Surface to volume ratio of the extruder is much higher than batch mixers producing at the same production rate. With the above example, the typical surface to volume ratio of the TSE would be conservatively at least one order of magnitude greater than that of the batch mixer. This provides much better temperature control in the TSE in comparison to a batch mixer.

d. (U) Mixing capability of TSE is much better than batch mixers- Superb distributive and dispersive mixing can be achieved with the twin screw extrusion process due to the much greater surface to volume ratio of the TSE. This superior mixing capability can be demonstrated experimentally [48, 51, 53, 55] and theoretically [45, 46, 59, 67, 77].

e. (U) Twin screw extrusion can process highly viscous suspensions in comparison to batch mixers (the behavior of which approaches solid like behavior, i.e., viscoplasticity) whereas the batch mixing would likely result in stagnant regions and unmixed material when the shear viscosity of the suspension is relatively high.

f. (U) Twin screw extrusion provides ease of process control and reproducibility in comparison to batch mixers: With a much greater surface to volume ratio and much better distributive and dispersive mixing capabilities [51, 53, 55, 77] twin screw extrusion provides better temperature control and a more homogeneous energetic product.

g. (U) Exposure of production staff to process streams and the process, thus the vulnerability, is much greater with batch processes in comparison to twin screw extrusion: The batch process invariably involves the physical removing of the energetic material from the batch mixer by the operators followed by the manual cleaning of the batch mixer. The twin screw extrusion process can be run without stopping. Furthermore, in case of an interruption of the process, the TSE can be purged with an inert first to remove all energetic residues.

(U) The fully-intermeshing co-rotating twin screw extrusion process is preferable over single screw extrusion and the single screw based "ko-kneaders" because of the self-wiping action of the two screws plus the much better distributive and dispersive mixing capability of the co-rotating fully intermeshing twin screw extrusion process [77]. In single screw extrusion the material moves in closed streamlines with little intermixing (limited distributive mixing) and is not forced repetitively into tight gaps (the basis for effective dispersive mixing). With the ko-kneaders there is no flexibility in geometry, i.e., the same geometries of the screw and the barrel are used.

(U) The typical schematics of the twin screw extrusion process is shown in **Figure 18a.** The extrusion process consists of multiple feeders (typically loss-in-weight) for feeding of the solids and liquids at accurate rates, feeding locations, screws/barrel combination for melting of the polymeric phase if necessary, and mixing of the ingredients, a devolatilization section at which vacuum is pulled to remove the air content of the energetic suspension, and a die for the shaping of the energetic grain that is extruded out of the die under pressure. The extruder has facilities for temperature control and monitoring and for the monitoring of the pressure distribution in the extruder and the torque that is generated. A typical screw configuration is so shown **Figure 18b**.

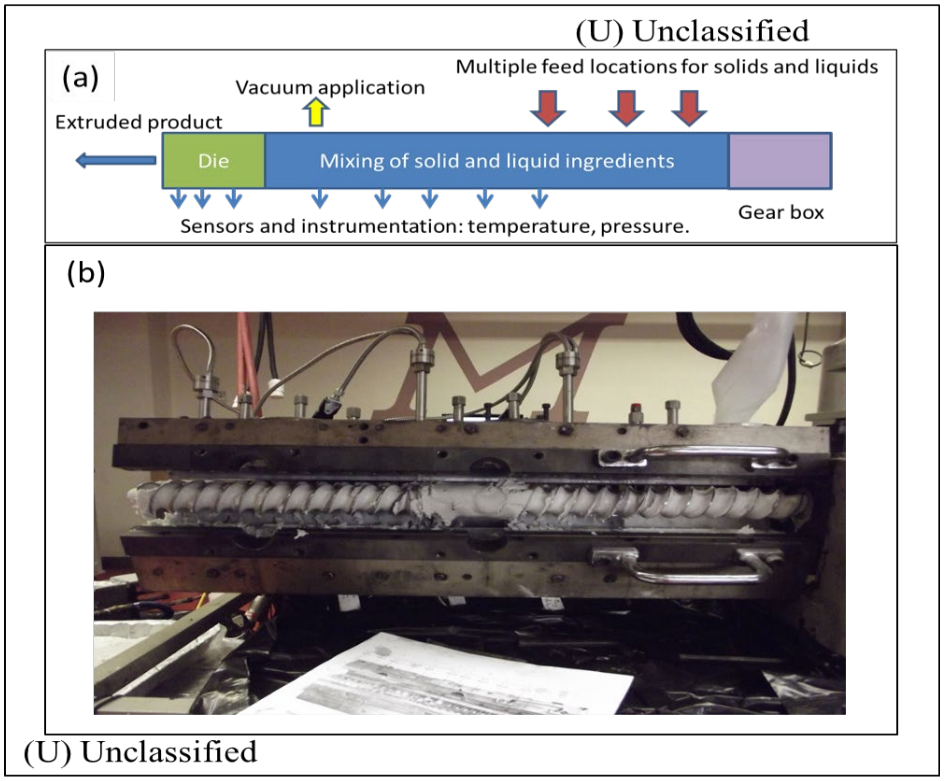


Figure 18 (U) Process Schematic (a) and Image of a TSE (b)

(U) With this typical configuration parts of the extruder remain only partially full (with the degree of fill principally dependent on the geometry, the rotational speed of the screws, and the flow rate of the energetic formulation. The solid and liquid ingredients are fed via loss-in-weight feeders into the extruder at precisely controlled rates. The degree of fill is reduced below 1 (partially full) over a certain length of the extruder adjacent to the pressurization section preceding the die to allow the pulling of vacuum **Figure 19.**

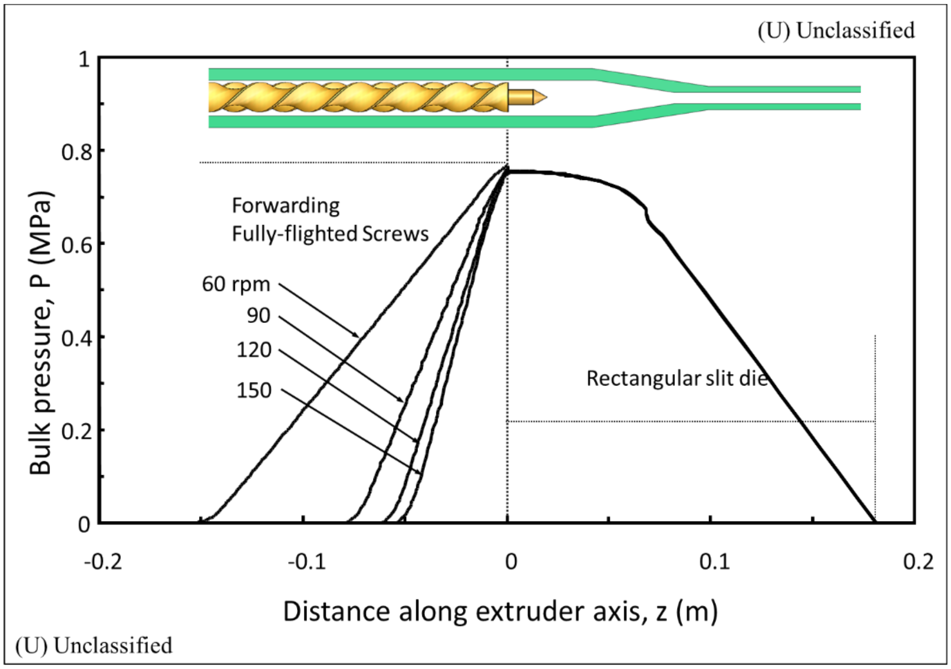


Figure 19 (U) Bulk Pressure along TSE Axis at Different RPM. *The graph shows the distance over which the screw remains partially full as a function of the operating conditions. The degree of fill is shown to decrease with increasing rotational speed of the two screws. Similarly, the degree of fill would decrease with decreasing mass flow rate [38, 72].*

(U) The feeding locations and the sensor locations for a typical application are shown in **Figure 20**. The feed locations need to coincide with partially full sections. Various sensors for torque, pressure and temperature are provided at the correct locations (temperature and pressure of the energetic material can only be monitored and thus used for process control at sections of the extruder which are completely full) to allow accurate process and product quality control on an in-line basis. The pressure sensors are placed only at locations at which the screw is completely full [72]. It should be understood that the degree of fill and hence the pressure distribution in the extruder would depend on the material, the geometry of the screw, the feed locations and the operating conditions.

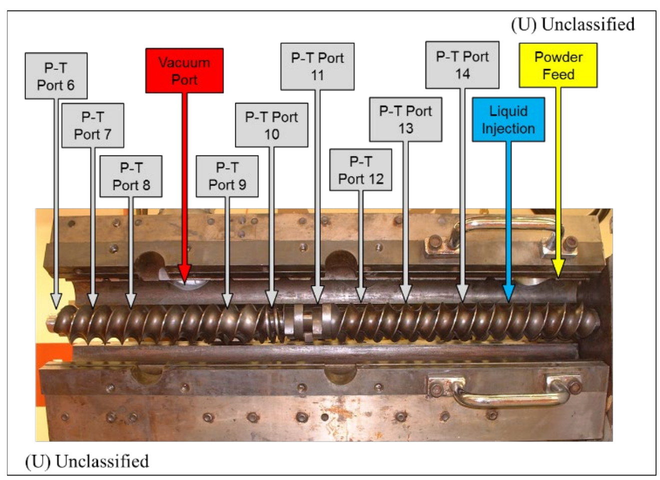
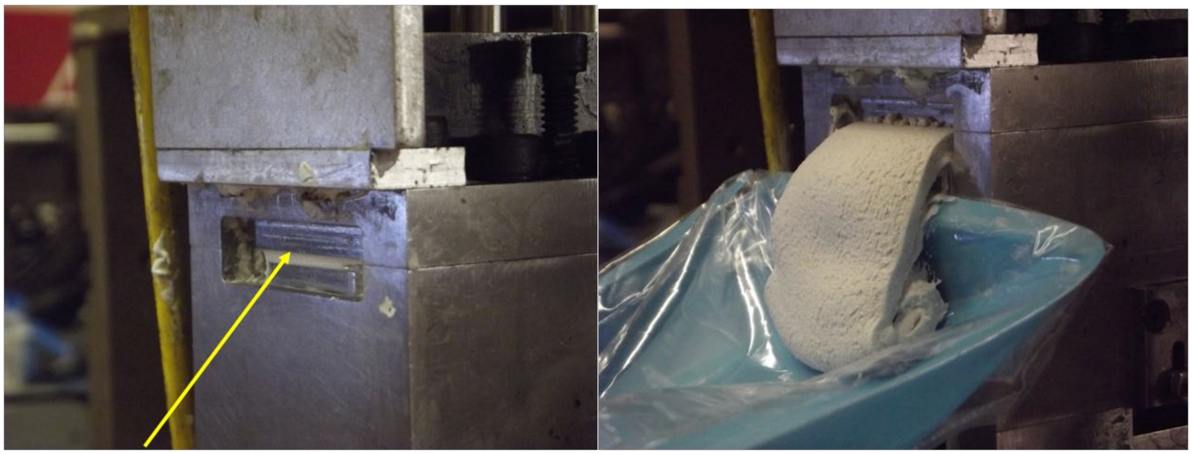


Figure 20 (U) TSE Feeder and Sensor Locations

(U) A die is provided to allow the shaping of the energetic material. A typical rectangular slit die is shown in **Figure 21a**, with a strand, i.e., the extrudate, emerging out of the die **Figure 21b**. There is a coupling between the die and the extruder. This is because the pressure drop at the die determines the pressurization rate in the extruder and the related degree of fill in the pressurization section of the extruder that precedes the die. In a two mixing zone screw configuration the energetic material is mixed at two consecutive zones. Both fully-flighted screw sections and kneading disks are provided to enable effective distributive and dispersive mixing actions.

(U) Unclassified



(a) Rectangular slit die attached to a twin screw extruder. (b) Extrudate emerging out of a slit die.

(U) Unclassified

Figure 21 (U) TSE Rectangular Slit Die

The following steps need to be taken for the development of the twin screw extrusion process.

a. (U) Characterization of the rheological binder of the energetic formulation: It is essential to understand the rheological behavior of the binder of the formulation first since the behavior of the binder significantly affects the rheological behavior and, hence, the processability behavior of the full formulation. Typically, the dynamic properties (linear viscoelastic material functions) of the binder need to be characterized to understand whether the behavior of the binder approaches the behavior of a Newtonian fluid (limited elasticity, shear viscosity not significantly dependent on the rate of deformation) or whether the binder is a non-Newtonian fluid with significant elasticity and shear rate/stress dependent shear viscosity. The temperature dependence of the rheological behavior of the binder (shear viscosity and elasticity) also needs to be understood via rheological characterization as a function of temperature. If the binder of the suspension is a gel, the shear viscosity and elasticity of the gel needs to be characterized using specific characterization methods associated with visco-plasticity.

b. (U) Characterization of the particle size and shape distributions: The particle size and shape distributions of the solid phase of the energetic formulation (the fuel and oxidizer particles) need to be characterized using methods like scanning electron microscopy, SEM, or diffraction methods. If the particles exhibit relatively high aspect ratios, continuous processing via twin screw extrusion becomes a significant challenge. The particle size and shape distributions define the maximum packing fraction of the solid phase as described next.

c. (U) Determination of the maximum packing fraction: The rheological and, hence, the processability behavior of the energetic formulation depend on the ability of the solid phase of the formulation to pack, as described with the maximum packing fraction of the solid phase

[78]. Both experimental and theoretical means can be utilized for the determination of the maximum packing fraction of the solid phase [78]. The maximum packing fraction represents the maximum concentration of the solid phase that can be incorporated into a binder. With increasing maximum packing fraction (which can be generated by increasing the modality of the particle size distribution, i.e., going to bimodal from unimodal or going to trimodal from bimodal etc., would increase the maximum packing fraction of the solid phase and enable better processability).

d. (U) Rheological characterization of the suspension: There are a number of on-line and off-line rheometers and rheological characterization methods developed for the characterization of the flow and deformation behavior and hence the processability of energetic formulation. The key deliverables were the development of the abilities to determine the parameters of wall slip and shear viscosity for highly filled energetic suspensions and gels [2-35] so that processability could be assessed and mathematical modeling and simulation of the twin screw extrusion process could be enabled.

e. (U) Mathematical modeling of the flow and heat transfer at the die for the shaping of the formulation: The rheological characterization data for the energetic suspension or gel can be used in the mathematical modeling and the simulation of the flow and heat transfer occurring in the die. Such calculations are very important for the design of the die and the optimization of the shaping conditions. The flow and deformation and heat transfer occurring in the die directly affect the flow and deformation and the coupled heat transfer occurring in the extruder section that is preceding the die as explained next.

f. (U) Mathematical modeling of the twin screw extrusion process: The die and the extruder are two components of the extrusion process that are integral to each other. What happens on one affects the other and the calculations need to be carried out on both geometries simultaneously. The objectives of modeling also include the determination of the distributions of stress, deformation rate, velocity and temperature in the extruder **Figure 22.** Some typical results can be found in [65, 69, 71, 72, 76].

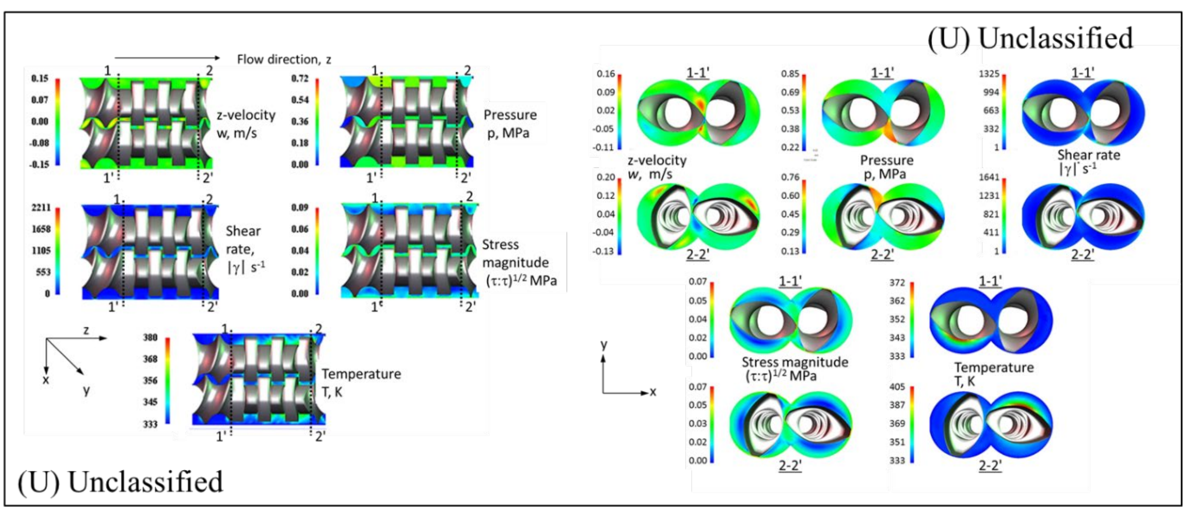


Figure 22 (U) Mathematical Model of TSE Elements. *A model predicts changes in critical variables within the material and against boundaries through a single section of screw element.*

g. (U) Application of a degree of mixing measure (mixing index) for the formulation: The rheological behavior and the processability (flow ability in the die and the extruder) depend on the mixing efficacies that can be achieved. It is very important to be able to characterize the goodness of the mixing via mixing indices. Such mixing indices can be generated using various experimental means including wide-angle x-ray diffraction and can also be simulated for [36-57]. Samples are collected from twin screw extrusion process and they are tested for their degree of mixing (mixing index values) [48, 49]. **Figure 23** shows how the mixing indices are calculated and provide some data obtained from the processing of a plasticized cellulose acetate butyrate binder with RDX using two types of twin screw extrusion (TSE-1 and 2) and a solvent dissolution method (SOLV-EXT) and a batch processing method (BATCH 1) **Figure 24a** and the distributions and mixing indices of burn rate modifiers etc. and ammonium perchlorate in an elastomeric binder (HyTemp) **Figure 24b**

(U) Unclassified

$$s^2 = \frac{1}{(N-1)} \sum_{i=1}^{N} \left(c_i - \bar{c}\right)^2$$

S is the standard deviation, C_i is the concentration, C is the average, N is the number of specimens

$$s_0^2 = \bar{c}\left(1 - \bar{c}\right)$$

Variance for the completely segregated sample

Mixing Index, MI = 1 – s/s_o

(U) Unclassified

Figure 23 (U) Mixing Index Formulas

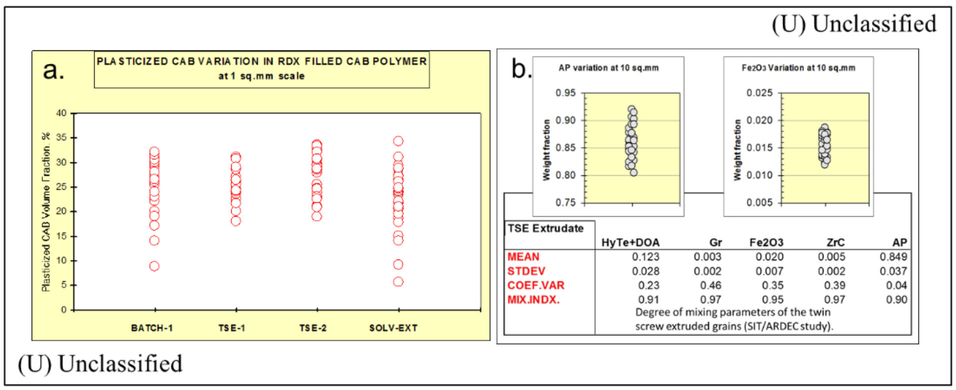


Figure 24 (U) Data Collected Distributions (a), and Mixing Indices (b).

h. (U) Processing of the simulant and the live formulations in the twin screw extrusion process: It is always a good idea to develop a processing simulant first and to test the extrudability of the formulation using the processing simulant. The development of the processing simulant is based on the matching of the volume fractions and the maximum packing fractions of the solid phase and the wettability of the solids by the binder. During the processing operation the data are collected and the control of the process parameters achieved via programmable logic controllers, PLCs **Figure 25a** via the sensors installed to the extruder and the die and all data collected via a human machine interface **[18b]** and control and historian workstations **[18c]**.

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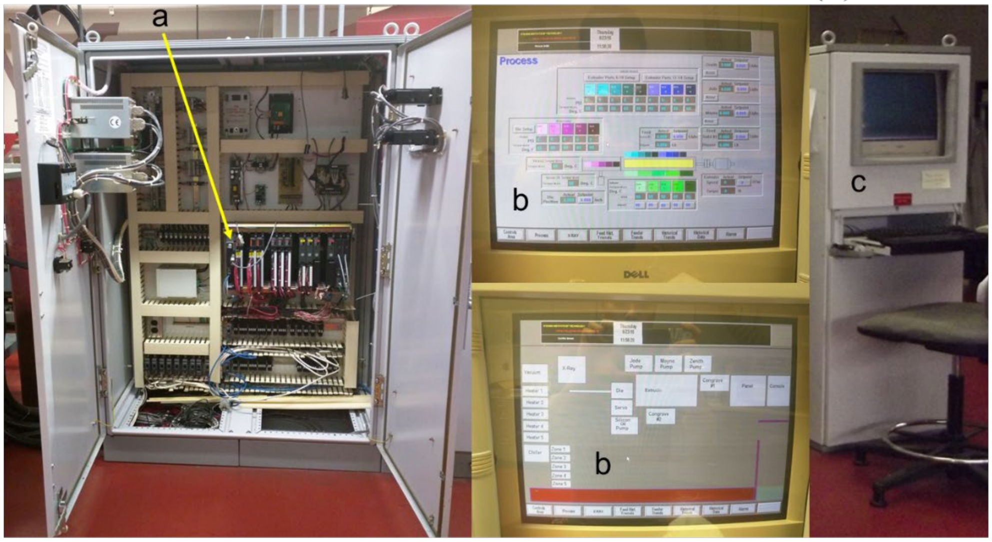

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Figure 25 (U) TSE Supervisory Control and Data Acquisition (SCADA) Equipment

(U) Expected applications of the technology: Continuous processing via the twin screw extrusion process is applicable to all processes that are currently applied on the batch mode. The current demonstration project is in the area of manufacturing of C4. The application of the technology to other energetic formulations – especially those which involve significant concentrations of solvents would introduce significant savings and environmental benefits at improved quality.

The following is a chronological summary of the development of the twin screw extrusion process for PAX-52:

1. (U) Characterization of the rheological binder of the energetic formulation
2. (U) Characterization of the particle size and shape distributions
3. (U) Determination of the maximum packing fraction
4. (U) Rheological characterization of the suspension
5. (U) Mathematical modeling of the flow and heat transfer at the die for the shaping of the formulation
6. (U) Mathematical modeling of the twin screw extrusion process
7. (U) Application of a degree of mixing measure (mixing index) for the formulation
8. (U) Processing of the simulant and the live formulations in the twin screw extrusion process

3.2 (U) TECHNOLOGY DEVELOPMENT

(U) Use of silicone as a binder system for high explosive material is not novel to this project. There are several global defense agencies working with silicone binders:

> (U) "The DoE and DoD have had a class of explosives available for 40 years based on silicone as the binder. The past explosives include the LANL and Navy, both of which were PETN and thermoset silicone. The LANL and Navy formulations employ a silicone compound that is a combination of a siloxane, silica (SiO_2) filler, and a curative for cross-linking of the siloxane.

> (U) Other silicone based explosives include Israeli **LBR** series that are Army qualified but do not have C4 performance and a Czech formulation that employs a non-traditional HMX like ingredient. A CL-20/HMX and silicone binder C4 analog was reported in a US patent where general silicone based formulations were produced to generate hand-moldable explosives that were easily blended, stable, and had excellent low temperature flexibility. Finally, hand-moldable silicone based explosive formulations were prepared using resonant acoustic mixing (RAM) and were characterized for low temperature flexibility, hazard sensitivity, cap sensitivity, and detonation velocity.
>
> (U) Recently, the US Army ARDEC conducted a program for PM Close Combat Systems to replace C4 with an IM compliant version. That formulation contained an extrusion enabling binder but, among other deficiencies, did not conform to the M112 specification. Rafael Advanced Defense Systems, Ltd has a long history with the extrusion and injection loading of silicone based explosives. Eurenco has recently produced a twin screw extruded plastic explosive known as Hexomax that is an analogue of C4. Finally, the US Army ARDEC FREEDM technology development program currently supports a task called "Improved Green C4" to formulate and characterize an environmentally friendly C4 analogue with improved reliability. Much of the small scale explosive formulation work has been completed and a viable silicone polymer candidate has been identified and used for advanced characterization; however, there is room in the formulation design space to increase the energetic output, modify the extrudability of the paste, and modify the physical properties like tackiness and stiffness (17)." [Passage edited by this author]

(U) Further R&D of silicone-based C4 formulations, which became the basis for this technology demonstration, focused on mixing different classes of HMX with silicone oil. In one trial, two lots of a bimodal formulation (18) were mixed by a vertical Baker-Perkins sigma blade mixer. Investigators used two classes of HMX powder (Class I and V) to enhance the volume fraction of solids in the formulation for more energy output. The results were good. The energy output performance of these two lots held promise for the HMX and silicone formulation (**Figure 26**).

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(U) Sensitivity and Performance Comparative Tests			
Test	PAX-52	Comp C-4	RDX Type II, Class 5
Density (g/cm3)	1.63 (95% TMD)	1.62 (99% TMD)	1.81 (100% TMD)
DSC (Exotherm Onset, °C) (AOP-7; US/202.01.020)	279	220	210
ERL Impact (50%, cm) (AOP-7; US/201.01.001)	100	64	18
ABL Friction (10 no go, N) (AOP-7; US/201.02.005)	3330	8000	1742
ESD (go/no go, 0.25 J) (AOP-7; US/201.03.001)	No Go	No Go	Go
Cap Sensitivity (Y,N) (UN Test 5(a))	Y	Y	Y
Detonation Pressure (GPa) (Plate Dent)	(95% TMD)	(99% TMD)	(88% TMD)
Detonation Velocity (km/s) (AOP-7; US/302.01.002)	(95% TMD)	(99% TMD)	(88% TMD)
Vacuum Thermal Stability (40 h, 100 °C) (AOP-7; US/202.01.022)	Pass	Pass	Pass
Thermal Stability (48 h, 75 °C) (AOP-7; US/202.01.013)	Pass	Pass	Pass
Small Scale Burning Test (UN Test 3(d))	Pass	Pass	Pass
VCCT (AOP-7; US/202.01.002)	T-30; Press Rupt T-60; Deflag T-90; Deflag		

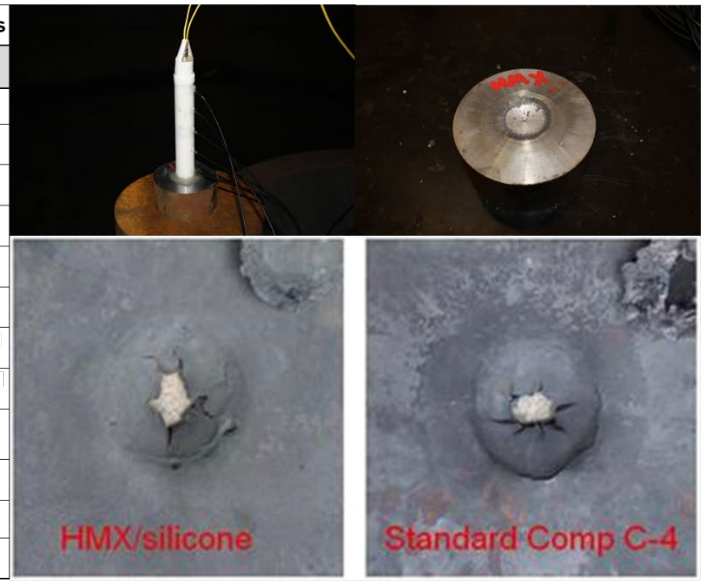

Figure 26 (U) Test Results of Early Formulations of a New C4. *Some performance tests were conducted on early HMX/Silicone formulations though not all the tests procedures were documented for publication. The table in the image shows that early blade-mixed PAX-52 had sensitivity and performance results comparable to Composition C4. The image on the top-right shows results from a detonation velocity and plate dent test, while the images on the bottom show another plate dent test with results also comparable to C4.*

(U) At the same time production of M112 demolition blocks was conducted using the ARDEC 40 mm co-rotating TSE. One of the HMX/silicone formulations that was produced at the 2500 g scale by the RAM method mentioned previously was set up for TSE trials. Two weight loss feeders were used to feed HMX Class I and V powder while a diaphragm pump injected PDMS oil. A forming die was installed so the mixed material could be extruded into M112 demolition blocks. Vacuum was used to enhance product density, but no heat was introduced into the system. Soon into the TSE trial a feeder problem, caused by bridging of Class V HMX finely ground powder within a feed hopper, terminated an extended run and none of the first-run product was retained for testing. The Class V feeder was removed from the trial and the M112 blocks were generated at 87% solids loading using only Class 1 HMX. Over 50 kg of M112 demolition blocks were produced, packaged, and shipped to Ft. Polk, Louisiana for wall breaching evaluation and one block was shipped to Boulder, Colorado, for sensitivity and performance characterization (**Figure 27**). Characterization of that material was never conducted; however, the PI visited Rocky Mountain Scientific Laboratory in Boulder to get a firsthand look at that material and judge its general qualitative properties (**Table 4**). That single block remained the only proof of success for the single-mode formulation that has brought the project to this demonstration.

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Figure 27 (U) Twin Screw Extruded PAX-52 M112 Demolition Blocks *The image on the left shows PAX-52 extrudate with a near net shape of the M112 demolition block. Side concavities were designed into the inverted die to compensate for material swell upon release from the die constraint. The middle image shows a breached wall target that was defeated with PAX-52 M112 demolition blocks. No formal test plan or report of the test was provided. The image on the right shows the only remaining PAX-52 material from the TSE trial. The PI opened the M112 packaging to find the material very smooth and hand-moldable.*

Table 4 (U) Qualitative Assessment of TSE PAX-52 Demolition Block *The PI found the material to be in excellent condition. Very light and spotty deposits of oil were observed when the specimen was held to a light, but no accumulation that would suggest that silicone oil migrated out of the material over time. Moldability of the material was superior to any other PAX-52 formulation previously produced by the RAM method. By the same comparison the sample did exhibit a more than expected amount of transfer to a gloved-hand, but not with a buildup of either bare particles or free oil. This condition indicated more design space for higher packing of solids into future formulations.*

(U) PAX-52 M112 (RDD14K046-001) Inspection at ARTI		
Line	**Condition Inspected**	**Comment**
1	Date	6-Apr-16
2	Inspector	John Centrella, ARDEC
3	Transients	Travis Swanson, RMSL and Don New, ARTI
4	Location	ARTI test range, Aurora, CO
5	LOT Number etc	RDD14K046-001
6	NEW of Specimen	1.48 lb
7	Density	TBD
8	General Appearance of Specimen	Good
9	Color of PAX-52	Pure White
10	Shape	M112 Rectangular
11	Dimensions (in.) Approximately	10Lx2Wx1H
12	Odor upon opening shipping containe	None
13	Odor upon opening package	None
14	Taggant Crystals Evident	None
15	Condition of Package	Excellent
16	Evidence of Tamper	None
17	Temperature of Material	Cool Approximately 50 deg F.
18	What Environment Stored	Uncontrolled 8x8x8 Storage Igloo
19	Duration of Storage	16 months
20	Firmness in package - finger firmnes	Medium
21	Evidence of Oil in Package	None
22	Discolored Package	None
23	Degraded Package	None
24	Elastic	None
25	Plastic	High
26	Friable	None
27	Moldability by Hand	High
28	Tension Strength	Medium by hand
29	Transfer to Hand	Medium without buildup on glove surface.

(U) Another PAX-52 effort was directed toward further formulation optimization by employing a special HMX grind with a mono-modal particle size distribution of slightly greater than 10 microns (**Figure 28**). The intent for that formulation was to increase the energetic solids content for greater cap sensitivity and energy output. All of the PAX-52 material for a new regimen of tests were produced by RAM and then fully characterized for hazard and performance measures. The shock sensitivity of the explosive was measured by NOL Large Scale Gap Test. The energy output was measured by the plate cut test as described in the military specification for the M112 demolition block (19) (**Figure 29**). Sensitivity and energy output results were as expected for the formulation with a very promising outlook for mix-ability of HMX with silicon. At that point in the development, PAX-52 formulations had been successfully mixed with blade and acoustic technologies.

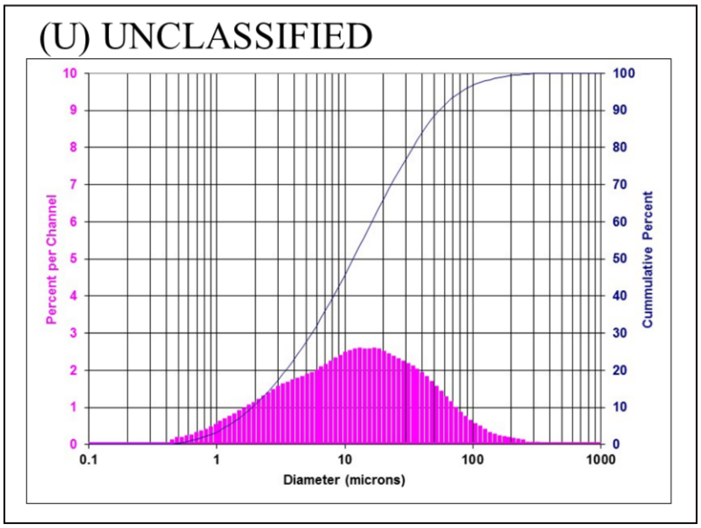

Figure 28 (U) Particle Size Distribution of a Special Grind Class V HMX *A broad range of particle sizes around 10-11 microns was used for this series of test. The powder could not be mixed greater than 83 weight percent due to the high fraction of small particles but resulted in a smooth consistency similar to Composition C4. Commodity Class V HMX available from the DoD consists of a size distribution around 1 micron, which would preclude its sole use in a standard formulation of PAX-52.*

Figure 29 (U) Results of Plate Cutting Test *To evaluate the energy output of PAX-52, a plate cut test was performed on 1 in. thick ASTM A36 grade 1018 steel plate. The charges for this test were cut from hand-packed M112 size charges to the dimensions of 0.5 in. x 1.5 in. x10.0 in. The charge was placed in the center of the plate and secured with tape at each end (top right.) Five of five shots with PAX-52 completely cut the steel witness plate comparable to two baseline shots using Composition C4* (20).

(U) An experimental batch of PAX-52 consisting of class-3 HMX and silicone oil was mixed in a 1kg horizontal sigma mixer to determine if an acceptable formulation could be made in a reasonable amount of time. At each interval during the mixing process, definite improvements to the mix quality were observed and, over the course of three hours, the HMX eventually homogenized to form a single lump of moldable material with acceptable consistency and flexibility. The mix procedure was very controlled and yielded steady improvements in mix quality over time with no anomalous results. The experiment showed that a formulation of Class-3 HMX crystals could be homogenized and sufficiently coated with silicone oil to form a substance consistent with C4, where it becomes moldable without being tacky or friable. The formulation held out promise for using straight Class III HMX and the ability to batch-mix PAX-52 using a sigma-blade mixer (**Figure 30**). The material had excellent sensitivity qualities but was not cap sensitive. The Class III HMX particles, though larger than the Class I type, are less sensitive to shock impulse due to the morphology of the crystals. Fewer defects and inclusions are apparent on the larger particles, which contributes to a dampening of shock impulse in the mass.

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Figure 30 (U) Blade-mixed PAX-52 *Some features of the mixed material: fissures (a) and crumbled edges (b) made it look like it was still friable; however, a sample kneaded by hand smoothed out the roughness (c). The PI pressed his thumb into the sample leaving a molded print with unbroken edges (d) and a smooth surface (e.) Very minor transfer of material to the glove surface (f) indicates a good homogenization of the crystals and full take-up of the silicone oil.*

(U) A follow-up production run of TSE PAX-52 duplicated an earlier TSE trial so the material could be characterized for sensitivity and energy output performance. The earlier product was never actually tested for small scale characterization, so it was critical for the project to verify that the formulation at least met basic performance criteria to be reliably compared to C4. Sensitivity and performance results showed that this formulation of PAX-52 was comparable to C4, more importantly it was cap sensitive (**Table 5**). Ten samples of the material were analyzed using High Performance Liquid Chromatography (HPLC) for composition in order to calculate a mixing index, which determines the homogeneity of the mix. The mixing index was calculated to be 0.995 (10) (**Figure 31**).

Table 5 (U) Sensitivity and Performance of TSE PAX-52

(U) PAX-52 LOT RDD16E046-134 Sensitivity and Performance Comparative Tests			
Test	**PAX-52**	**Comp C-4**	**RDX Type II, Class 5**
Density (g/cm3)	1.67 (99% TMD)	1.62 (99% TMD)	1.81 (100% TMD)
DSC (Exotherm Onset, ºC) (AOP-7; US/202.01.020)	277	220	210
ERL Impact (50%, cm) (AOP-7; US/201.01.001)	100	64	18
ABL Friction (10 no go, N) (AOP-7; US/201.02.005)	360	8000	1742
ESD (go/no go, 0.16 J) (AOP-7; US/201.03.001)	No Go	No Go	Go
Cap Sensitivity (Y,N) (UN Test 5(a))	Y	Y	Y
Detonation Pressure (GPa) (Plate Dent)	(99% TMD)	(99% TMD)	(88% TMD)
Detonation Velocity (km/s) (AOP-7; US/302.01.002)	(99% TMD)	(99% TMD)	(88% TMD)

Mixing Index Calculations

$$\overline{c} = \frac{1}{N}\sum_{i=0}^{N} c_i$$

$$s^2 = \frac{1}{(N-1)}\sum_{i=1}^{N}(c_i - \overline{c})^2$$

$$s_0^2 = \overline{c}(1-\overline{c})$$

$$MI = 1 - \frac{s}{s_0}$$

Figure 31 (U) Mixing Index Calculations *High Performance Liquid Chromatography (HPLC) analysis was performed to give the percent organic compounds in the matrix. HPLC is a means for qualitative and quantitative analysis of nonvolatile organic compounds. Samples are separated into individual components by partitioning between a flowing solution and a stationary phase consisting of coated particles packed into a column. The HPLC used has a diode array absorbance pdetector that provides UV-VIS spectra for the eluted compounds. Sample analyzed for % weight HMX by HPLC, silicone by difference. Quantitative analysis was performed by preparing greater than 99% pure HMX as standards at different concentrations and obtaining an optimal* R^2 *(correlation coefficient) value. The standard values are then incorporated in the instrument software (Waters Empower) for analysis.*

3.3 (U) ADVANTAGES AND LIMITATIONS OF THE TECHNOLOGY

(U) Several key advantages of twin screw extrusion (TSE) mixing technology over the conventional batch-mixed, blade-mixing method are described previously in section (U) Technology Description 3.1 page 16 and are listed here:

a. (U) Flexibility
b. (U) Inherent safety
c. (U) Surface to volume ratio
d. (U) Mixing capability
e. (U) Can process highly viscous suspensions
f. (U) Ease of process control and reproducibility.
g. (U) Limited exposure to process streams

(U) Some disadvantages related to the TSE process technology especially when processing energetics where initiation hazards have high risks:

a. (U) Process development requires a comprehensive understanding of the rheological properties of the particular binder and mixed emulsion. Guesswork or rule-of-thumb techniques do not work well with TSE under any circumstances when mixing energetic material. The mixing process can run out of control in short time possibly leading to a hazardous condition. A steady state throughput of mixed ingredients is manage by the use of computer control of an array of sensors that monitor critical process parameter (feed rates, pressure and temperature along several points of the mixing protocol, and drive torque.)

b. (U) Requires feeding of dry, well blended powder to gain a consistency of product density and composition. This is a disadvantage as compared to the batch method where powder feed rate and blend are not critical to product quality. As such, the TSE operator has to manage yet another risk category working with dry energetic powders in an environment fraught with initiation modes (static charge, pinch point, dust plumes, etc.), so methods of wetting or coating the energetic powders must be introduced into the process. This risk mitigation adds another ingredient to the mix and more variables to the overall process. This demonstration was conducted by the use of dry powder; however, the author knows of no other TSE operation that is willing to feed dry powders.

c. (U) Decontamination of internal segments of all energetic residue is tedious and time consuming. It requires large amounts of organic solvent to dissolve a particular binder but not a particular energetic crystal, which could lead to further hazards. Disposal of contaminated solvent presents an additional cost to the operation. This disadvantage is minimized if the TSE mixing platform is dedicated to a single class of ingredient where cross-contamination has no safety risk.

(U) Several key advantages of the formulation PAX-52 over conventional C4 are derived from its silicone binder system (PDMS.) Silicone has an affinity for energetic crystals that causes them to be efficiently coated and desensitized to stimuli. The oil coats the surface thinly and is not easily removed by aging or heating so the PAX-52 is not prone to delamination voids or exposure of crystals. This quality yields improvements in product shelf life, initiation reliability, and product consistency in all environments. Other advantages of PAX-52 over C4 as an explosive are the following:

a. (U) PAX-52 maintains constant viscosity through an operational range of temperatures. The most outstanding quality of PAX-52 that attracts the combat engineers and EOD technicians is its ability to stay soft and moldable at temperature extremes. Where C4 becomes very hard in cool weather, around 40^{o}F, and extremely hard at freezing, PAX-52 remains easily pliable; and conversely, where C4 loosens, weakens and exudes oil in hot temperatures (desert missions or in containers soaking in the sun) PAX-52 keeps its form, strength and consistency as when it was extremely cold. Siliconize formulations have shown excellent flexibilty, where the material shows a glass transition step function at -38^{o}C **Figure 32** (19)

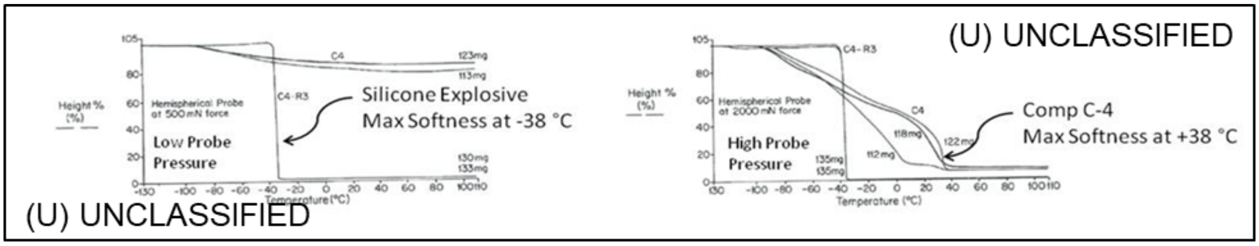


Figure 32 (U) Softness of Silicone Explosives vs. C4. *The silicone explosives will remain fully soft at temperatures normally experienced by combat engineers and SOF. In contrast, C4 undergoes gradual hardening as the viscosity of its binder changes inversely with temperature.*

b. (U) PAX-52 is easily formed into small crevices and through small ports. This quality has benefits to the soldiers who must load demolition charges and pack warhead cases. PAX-52 requires much less force than C4 to pack into a case so larger chunks of material can be used, requiring less time to hand pack demolition devices. This advantage was observed first hand at a user evaluation conducted at Fort Leonard Wood, MO. Soldiers of the 5th Engineering Battalion found the PAX-52 easier and quicker to load a series of demolition devices in side-by-side comparisons with standard C4. (21)

c. (U) PAX-52 has a lower risk to the environment and human toxicity due to the use of nitramine HMX as oppossed to RDX used in C4. RDX is considered an environmental and potential human health hazard by the EPA (9). "Relatively high acute oral doses of RDX are known to cause seizures in humans and can cause seizures in animals from low-level chronic oral exposure. The effects of long-term, low level exposure to humans is unknown, although the USEPA has determined RDX to be a suggestive human carcinogen. In terms of environmental transport, RDX can enter groundwater through a low affinity to organic carbon and limited water solubility (its solubility in water at 20 °C is between 38 and 60 mg/L), which suggests a moderate to high soil mobility in combination with a very slow biodegradation process. HMX provides high energy as well as exhibiting low transport potential in the environment. In comparison to RDX, HMX offers over one order of magnitude reduction in carcinogenic potency and an order of magnitude increase in clean-up threshold in soil. HMX is a nitramine and has a molecular structure very similar to RDX but it is much less soluble and toxic. EPA drinking water threshold for RDX is 2 ppb while the HMX threshold is 400 ppb or 200 times less stringent. The water solubility of HMX is also ten times lower than that for RDX. Therefore, using HMX instead of RDX would reduce the associated risk by at least three orders of magnitude, as the impact of a contaminant is equivalent to its effect, exposure and fate" (4).

d. (U) The PAX-52 formulation has only one non-energetic ingredient. Silicone is a widely available polymer manufactured by numerous US chemical companies. This is an advantage compared to the C4 formulation that contains a plasticizer Polyisobutylene (PIB) that has a sole US source. In the recent decades C4 has had to be re-qualified due to difficulty maintaining a US source for PIB.

e. (U) PAX-52 can stick to moist, soiled surfaces. Silicone oil will readily coat wet surfaces by displacement of surface moisture and adhesion to the surface. C4 will not readily stick to wet surfaces without some preparation. It was observed by this PI that a lump of PAX-52 stuck fast to a slime coated surface submerged in water. The lump remained securely attached to the surface for several hours and could not be easily dislodged. This quality will give special operations soldiers the advantage when in mining operation with PAX-52 as compared to C4.

(U) The key disadvantage of PAX-52 is its high unit cost as compared to C4. In a time of budgetary constraints, Army program and product managers are reluctant to invest in a new technology when there exists a qualified system that meets their mission requirements. Even though the proposed PAX-52 has numerous advantages and superior qualities compared to the standard C4, its price differential is a deterrent to capital investments in equipment, qualification of a new Army explosive material and re-qualification of the entire C4 product line. Then on top of that budget risk program managers will still be paying twice as much for PAX-52. In a cost-benefit analysis of PAX-52 TSE production and conventional C4 batch production, TSE production of PAX-52 seems like a winning alternative. There are measurable cost reductions related to manufacturing efficiencies and product benefits related to the PAX-52 formulation that figure into an analysis and there are benefits of PAX-52 related to its superior qualities above C4 that cannot be easily monetized but also figure into the analysis. All measured, the high unit cost of the alternative PAX-52/M112 product cannot match the current C4 cost benefit due to the high cost of HMX as compared to RDX.

(U) PAX-52 has no limitations in quality, performance, sensitivity or application as a demolition explosive. Some minor quality issues were raised during user evaluation. For instance, one soldier felt that the PAX-52 was too soft and not strong enough to firmly hold an embedded primer device. Another felt it was too sticky while he packed a demolition charge with a wood dowel. Both issues are legitimate and will be considered for future formulations of PAX-52. A slightly higher ratio of solids to silicone binder will likely satisfy both issues.

4 (U) PERFORMANCE OBJECTIVES

Table 6 (U) Performance Objectives

Performance Objective	Data Requirements	Success Criteria	Results
PAX-52 shall be produced continuously by a steady state process for at least three hours.	1. Time record. 2. Temperature and pressure records.	Recorded production run exceeding three hours. Recorded feedback data within three standard deviations for pressure, temperature, and torque.	Partial Success
PAX-52 consists of only two ingredients. Taggant not accounted for in the formulation.	Composition analysis	Detection of HMX at 85 percent by weight.	Success
Demolition charge cross-section	MIL-DTL-50523B, drawing 9204248.	Measurement of 2"x1" with side concavity allowance.	Success
M112 Physical properties - Plasticity	Penetrometer MIL-DTL-50523B 3.2.1.1	Force resistance of not less than 2.0 pounds and not greater than 8.0 pounds	No Test
M112 Specific gravity	Archimedes' water method MIL-DTL-50523B 4.4.2	Specific gravity greater than 1.50	Success
Energy output	Plate-cutting test MIL-DTL-50523B 4.4.3	Cut a steel witness plate completely into two separate sections.	Success
Physical sensitivity	ESD (AOP-7201.03.001) BAM Friction (AOP-7 201.02.006) ERL Impact (AOP-7 201.01.001)	ESD: No Go < 0.25 Joules BAM Friction: 10 No Go greater than 200 Newtons ERL Impact: 50% reaction height greater than 75 cm	Success Success
Shock sensitivity	Cap Sensitivity (TB700-2 Para 5.6a). IHE Gap Test (AOP 201.04.005)	Sustained Detonation Response Attenuation Gap between 161-171 cards.	Success
Detonation velocity Energetic performance	Detonation velocity test Plate dent test	Velocity greater than 7.5 km/sec At 99% TMD Pressure greater than 25.0 GPa at 99% TMD	Success
Product response to ignition	1. VCCT (AOP-7 202.01.002) 2. Small Scale Burning Test (TB700-2 Para. 5.4a)	1. Pass: No detonation response 2. Pass: No detonation response	Success
PAX-52 chemically stable	1. Vacuum Thermal Stability (40 h @ 100 ºC - AOP-7; US/202.01.022) 2. Thermal Stability (48	1. Pass: no significant decomposition or off-gassing. 2. Pass: no exothermic reactions. 3. No accumulated exudation of oil.	Success

	h @ 75 ºC - AOP-7; US/202.01.013). 3. Aging Study		
Homogeneity	Composition analysis	Mixedness index greater than 0.99	Success
Environmental impact	Deposition rate test	Less than 1% by weight of RDX accumulation.	Success
HMX workplace exposure	OSHA Arbitrary Target Concentration	HMX TWA-TLV 0.2 mg/m3	Success
Performance Objective	**Data Requirements**	**Success Criteria**	
Packaging	Contractor Inspection Report	MIL-DTL-50523A, drawing 9204248.	Success
User evaluation	Color Manual Moldability Tackiness Surface oil transfer.	No noticeable change in the quality of the product during any field demonstration conditions.	Success

4.1 (U) STEADY-STATE PROCESSING OF PAX-52

(U) The technology demonstration required a 3-hour continuous production of PAX-52. Success was defined as the control of the process to within three standard deviations of the sample mean for each of the variables sampled by the PLC at evenly divided intervals. During the demonstration temperature of the PAX-52 product, which was one of three variables defined as success criteria, was not recorded; however, several other process parameters were recorded: pressure, powder and liquid feed rates and drive torque. This particular demonstration objective was deemed only a **partial success** due to missing data requirements, a slight loss of process control and an unmet run time requirement; however, the technology demonstration provided validity to the proposed continuous production method.

4.2 (U) PAX-52 CONSISTS OF ONLY TWO INGREDIENTS.

(U) The PAX-52 produced during the 3-hour demonstration was sampled by a random selection of ten 50-gram samples and analyzed by HPLC for HMX content. Success criteria for this objective was a measured minimum percentage of HMX composition of the formulation. This demonstration objective was deemed a **success** as the batch contained only two ingredients in the proper ratios: HMX and Silicone.

(U) Demolition Charge Cross-section A representative sample of extruded demolition block was measured with a caliper to determine its dimensions. To achieve success, the block was to measure no greater than 2.00 inches wide by 1.00 inches thick, allowing for a shallow concavity on each side. Cross-section measurements taken from the belt of PAX-52 produced in this demonstration achieved the objective at 2.00 inches by 1.00 inches.

4.3 (U) PHYSICAL PROPERTIES

(U) A quantity of PAX-52 was packaged in accordance with MIL-DTL-50523B but was not tested for resistance to force in the M112 configuration. See Appendix D 11.3**.** Force resistance is a measure of a key property that affects the end-user's ability to use the product in IAW technical bulletins. (Relates to User Evaluations: See Appendix **D21**) Though the moldability of the material was not measured because the project did not have access to a penetrometer, several qualitative assessments were made by explosives technicians and engineers that found the material to be superior to C4; nevertheless, this demonstration objective was deemed a **No-Test**

4.4 (U) M112 SPECIFIC GRAVITY

(U) A representative sample of extruded demolition block was analyzed for specific gravity (SG) in accordance with MIL-DTL-50523B. (See: Appendix **D19**.) SG relates directly to its physical strength and energy density. There is a direct correlation between an energetic material density and its detonation pressure. Though the specification for C4 SG is 1.50, for PAX-52 demonstration to be a success, it must have been greater than 1.65 so that achieves comparable detonation pressure. This demonstration objective was a **success**, with the PAX-52 exhibiting a specific gravity of 1.688 using a gas pycnometer and 1.666 by the Archimedes method.

4.5 (U) ENERGY OUTPUT

(U) A quantity of PAX-52 was packaged in accordance with MIL-DTL-50523B and tested for energy output in the M112 configuration to measure its effectiveness against a steel plate. (See Appendix **D20**.) The performance objective for energy output of PAX-52 was considered a **success** in this demonstration as the plate was cut in two sections.

4.6 (U) PHYSICAL SENSITIVITY

A regimen of tests were required to verify the PAX-52 hazard classification for handling and shipping safety. See Appendix **D1-D4**

(U) ERL Impact: The ERL, Type 12 Impact Tester, utilizing a 2 ½ kg drop weight, was used to determine the impact sensitivity of the PAX-52 sample. Results greater than the impact sensitivity of HMX Cl-5 T2 (28 cm) were deemed acceptable for transport and handling. PAX-52 was **successful** in this demonstration as results indicated an impact sensitivity of 100 cm.

(U) BAM Friction: The result is positive (+) if the lowest friction load at which one explosion occurs in six trials is less than 18 lb_f (80 N). For this assessment, PAX-52 was be verified to be greater than 100 N, which indicates **success**.

(U) Electrostatic Discharge. The ESD Test was used to determine the sensitivity of a material to electrostatic discharge. Results greater than the ESD sensitivity of RDX Cl-5 T2 (.063J) were deemed **acceptable** for transport and handling.

(U) Thermal Stability. This test evaluated the thermal stability of PAX-52 when subjected to elevated temperature. Several criteria were used to evaluate the results of the test. IAW TB700-2: explosion, ignition, substance exudation, or a temperature rise exceeding 5°F (3°C). For this demonstration, the PAX-52 was assessed as **successful** as none of these conditions resulted from heating.

4.7 (U) SHOCK SENSITIVITY

(U) PAX-52 was tested for shock sensitivity in two different tests. Cap sensitivity verified the material was sensitive enough to shock to initiate with a blast cap while the IHE test quantified the shock sensitivity of PAX-52. Appendix **D13**

(U) The cap sensitivity test was required to verify whether the energetic material transfers initiation shock of a typical blasting cap into a mass detonation event. PAX-52 was **verified** as cap sensitive from this test.

(U) PAX-52 was also characterized for shock sensitivity using the insensitive high explosives (IHE) test, which measured the sensitivity to detonation of an explosive exposed to an explosive induced shock. Results are not assessed as a pass/fail. Results would verify the 50% shock pressure point of the PAX-52. For this assessment, PAX-52 slightly **outperformed** the objective.. Appendix **D14.**

4.8 (U) ENERGETIC PERFORMANCE: DETONATION VELOCITY AND PRESSURE.

(U) PAX-52 was characterized for detonation velocity, which success defined as having a detonation velocity ≥ 7.5 km/sec (Appendix **D10**). This demonstration objective of PAX-52 was deemed a **success**.

(U) PAX-52 was characterized for detonation pressure, with success defined as a detonation pressure ≥ 24.0 GPa (Appendix **D11**). PAX-52 **succeeded** in this objective.

4.9 (U) PRODUCT RESPONSE TO IGNITION

(U) PAX-52 was characterized for its response to heating in a Variable Close Confinement Test VCCT. From this demonstration, the material was deemed **acceptable** as no detonation occurred. Appendix **D15**

(U) PAX-52 was characterized for its response to heating in a small scale burning test. From this demonstration, the material has been deemed **acceptable** as no detonation occurred. Appendix **D16**

4.10 (U) CHEMICAL STABLILITY

(U) PAX-52 was characterized for chemical stability under steady and variable heating conditions and deemed a **success**, as in the thermal stability test for Performance Objective. Appendix **D8, D9, D17**

4.11 (U) HOMOGENEITY

(U) PAX-52 mixing index (measure of homogeneity) was calculated to be 0.995 based on mean and standard deviation of ten samples and verified to **exceed** the performance objective. See Appendix **D6.**

4.12 (U) ENVIRONMENTAL IMPACT

(U) PAX-52 was studied by the DRDC to determine the deposition rate of RDX and HMX. By using HMX instead of RDX in the formulation would reduce the associated risk by at least three orders of magnitude, as the impact of a contaminant is equivalent to its effect, exposure and fate. (4) The testing for deposition rates was performed this winter and demonstrated a good behaviour for PAX-52 during the detonation of blocks and while used as a donor charge for 40mm grenades. Acceptable deposition rates were measured. So far, PAX-52 appears to Canada as a very good option for an RDX-free replacement of Composition C4. The assessment is deemed **very positive** with nearly a three orders (2.91) of magnitude reduction in environmental risk associated with RDX nitramine explosive as determined by the Canadian test results. Environmental risk associated with HMX contained in the PAX-52 was not assessed by the project but is considered an acceptable risk by Canadian researchers (6)**.** The demonstration meets the performance objective of less than 1% RDX deposition. **Success**. See Appendix **D23**

4.13 (U) HMX WORKPLACE EXPOSURE

(U) There is no official Occupational Safety and Health Administration (OSHA) Personnel Exposure Limit (PEL) established for HMX. OSHA recommended an arbitrary concentration target of 0.2 milligrams per cubic meter (mg/m^3). Also, there is no published American Conference of Governmental Industrial Hygienists (ACGIH) threshold limit values (TLV) for a time weighted average (TWA) for HMX (Octogen). For an 8-hr TLV-TWA there were only 0.005 mg/m^3 of HMX detected during the technology demonstration which **meets** the performance objective. See Appendix **D24.**

4.14 (U) PACKAGING

(U) The project proposed to demonstrate that PAX-52 material could be packaged in the M112 configuration using an alternate method, with an allowance for off-site transportation. Due to cost and schedule constraints, the proposed one-step process whereby PAX-52 M112 demolition blocks are extruded and packaged on a single production line, could not be demonstrated. Market research has shown that an M112 packaging unit would not be feasible to implement on the demonstration site. Employing an existing GOCO packaging line would present unmanageable risk to both the Project and the GOCO; however, there was a non-Government contract packager that was willing to help package bulk PAX-52 into the M112 configuration.

(U) PAX-52 was readily formed into M112 demolition blocks by being pressed into a closed mold and then inserted into a specified container. More than 250 M112 demo blocks were produced thereby meeting this particular objective. **Success**

4.15 (U) USER EVALUATION

At the time of submitting the project demonstration plan it was not certain how or if any user groups would be able to support any kind of informal evaluation of the PAX-52 demolition explosive. As time passed, both the Combat Engineering School at the Maneuver Support Center of Excellence (MSCoE) at Fort Leonard Wood, Missouri and the Explosives Ordnance Disposal

Technology Division at US Army ARDEC, Picatinny Arsenal, New Jersey agreed to participate in well-planned events that yielded excellent feedback from combat engineers and ordnance disposal technicians. Countless hours of planning and execution, a good amount of military assets and supplies, and a wealth of test participants who wear the uniforms of the Army, Air Force, and Marines; were generously provided at no cost to the project. This particular objective was met with resounding **success**.

(U) Samples of the PAX-52/M112 demolition blocks were evaluated by the DoD C4-user community for a hands-on comparison to the C4/M112. Mostly positive feedback was received from the subjective, qualitative evaluations, but the clearest and loudest signal from the user community was their enthusiasm for the cold-weather moldability of PAX-52 (**Figure 33**). The 595 Sapper Company, 5th Engineer Battalion at Fort Leonard Wood evaluated PAX-52 by loading devices and demolition charges in a live training exercise. In addition, the Explosives Ordnance Disposal (EOD) Division, ARDEC, in cooperation with the Ordnance Procedures Development Team, Indian Head, MD, conducted subjective handling assessments of the PAX-52 energetic material and its employment in various shaped charge containers and disposal procedures by representatives of three Armed Services (Army, Air Force and Marine Corps.)

Figure 33 (U) PAX-52 Moldable Explosive. *The PAX-52 was evaluated by soldiers accustomed to working with C4. They spent hours comparing the merits of each composition while packing a variety of demolition charges. Their overall assessment of the new moldable explosive was expressed plainly by one soldier's comment, "This $%!# makes the soldier's job a hell of a lot easier."*

5 (U) SITES/PLATFORM DESCRIPTION

5.1 (U) TEST PLATFORMS/FACILITIES

(U) This technology demonstration was conducted at Picatinny Arsenal in Rockaway, New Jersey, at the Universal Twin Screw Extrusion (TSE) Facility. The complex is home to numerous energetics laboratories, operation facilities, and a waste processing complex, all staffed by numerous personnel including scientists, engineers, technicians, and energetics operators. The facility benefits from a large resource of labor from organizational management, first line supervision, and certified energetic technicians to ensure TSE processes are in compliance with all workplace, explosive, and environmental regulations. A complex infrastructure that includes storage magazines, blast containment barriers, access and egress roads, and warning systems provide safe and efficient means to conduct simultaneous energetic operations without jeopardy to the surrounding community.

(U) The TSE platform was placed toward one end of an operation bay with ancillary equipment conveniently located for easy access (**Figure 34**). In the facility there is ample room to conduct work necessary for operation and maintenance of the system. The TSE facility is considered a pilot-scale production environment, licensed by regulation to process energetic material. Workflow in and out of the facility can only be accomplished by an interruption of the extrusion process while sub-tasks are completed. Once the facility is cleared of personnel then the extrusion process can resume. The TSE platform was perfectly suited for this demonstration of low-rate production of PAX-52.

(U) The choice to demonstrate the technology at the TSE facility greatly reduced risk for the project on several levels. The lead engineer of the TSE facility and technical advisor on this project is an Army subject matter expert in twin screw extrusion technology, particularly with gun propellants and high explosives. He brought a large body of knowledge to the demonstration gained by years of experience, most notably his work with the development of blast explosives. He also developed processes to extrude solvent-less explosives and pyrotechnic flare compositions. His team of engineering technicians have the ability to set up and operate all the TSE equipment, as well as provide him with feedback commensurate with the technical complexity of each different process. The TSE team has the knowledge and experience this project required to mitigate technical risk and ensure success.

(U) The TSE provided all the equipment needed for this technology demonstration. Other than minor expenses for small implements, the project carried no risk for a major budgetary expense for equipment. There were weight loss feeders, metering pumps; multiple feedback sensors for temperature, pressure, and torque; and, overall PLC process control already in place on the platform. The human machine interface (HMI), written specifically for the TSE process, is a well-proven program that provides the operators discrete process data in real time and manages data acquisition in the background. Success of the project technology demonstration depended in good measure on the reliability of the TSE system and supervisory and control programs, all of which were in good operational readiness at the TSE facility.

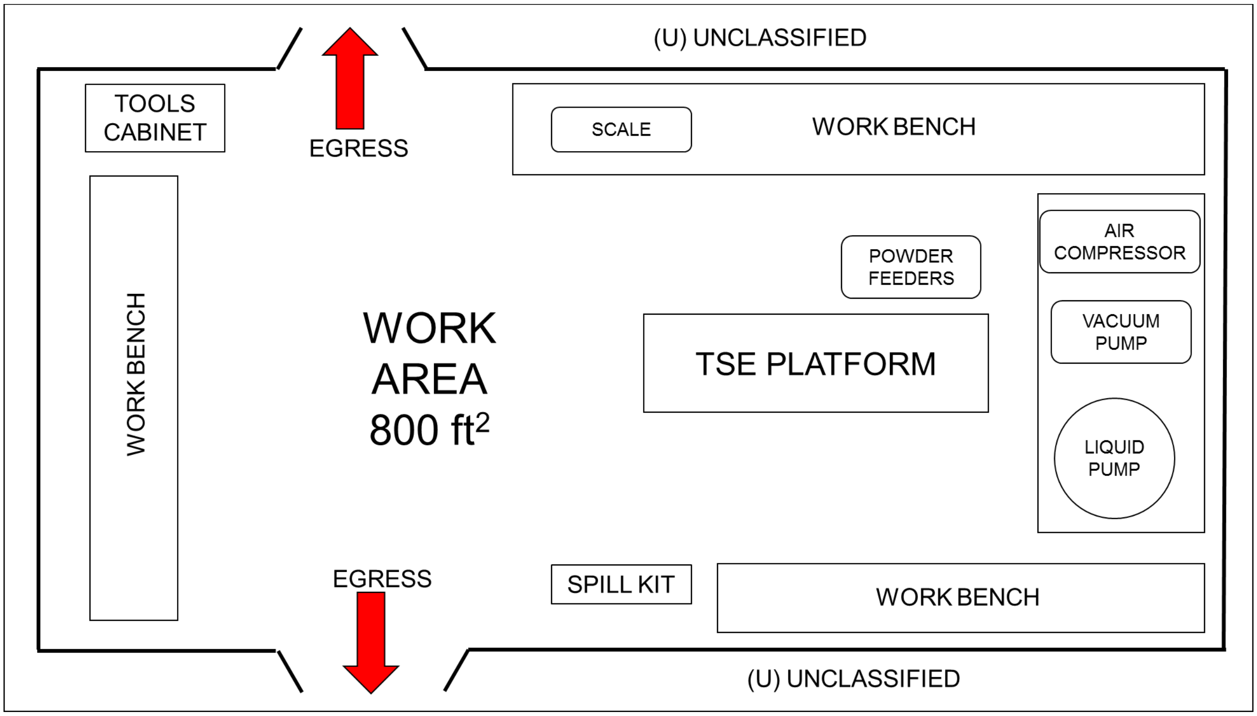


Figure 34 (U) Layout of the TSE Facility. *Depicted is a general layout of the TSE production facility. Multiple subsystems in the facility are designed to protect life and property to a risk level mandated by Army regulation. Every electric device, including primary power and data transmission cables, must be specially installed for explosion proofing. Metallic objects are bonded and grounded to prevent static discharge, and the flooring is coated with a carbon epoxy that conducts static charge off persons wearing special static dissipative footwear. The facility and personnel are protected by fire suppression and high-speed deluge systems in the event of an initiation, while walls and roof system are of frangible structure to relieve resultant blast overpressure.*

(U) The TSE has been used to successfully produced thousands of pounds of a high explosive formulation used mostly for munitions. The effort demanded attention to precise feed rates of powders and solvent to find the zone of operation between low quality and high hazard. The problem was studied by 3D computational modeling to understand fluid dynamics of the mixture and to head off disastrous results triggered by precipitous changes in temperature or pressure within the mixing barrel of the TSE.

(U) Each new formulation comes with a different set of variables and challenges. Black powder was compounded on the TSE with moderate results. In 2003, a study was undertaken by the Twin Screw Extruder TSE team, funded by Life Cycle Pilot Process (LCPP) to demonstrate a viable means of producing black powder with characteristics similar that of GOEX black powder. The results of the effort produced two different black powder substitute materials comparable to GOEX BP (commercial producer of black powder.) The study showed that a new alternative black powder substitute could be made in a safer and environmentally friendly manner while still being comparable to GOEX BP (23).

5.2 (U) PRESENT OPERATIONS

(U) Composition C4 is produced in a multi-step water slurry process that requires an organic, solvent-based lacquer to mix a cohesive binder and plasticizer with energetic RDX nitramine crystals. This multi-step process, developed in 1958, has been largely successful owing to the availability of cheap labor, cheap energy, and lower environmental protection standards; however, six decades later it is still time, energy, and labor intensive and generates 1.5 million gallons of aqueous waste per year. The output of the C4 production process is a loose powder (or cake) that is boxed and transported to an entirely different packaging facility where it is further processed into M112 demolition blocks via two additional processing steps (**Figure 35**).

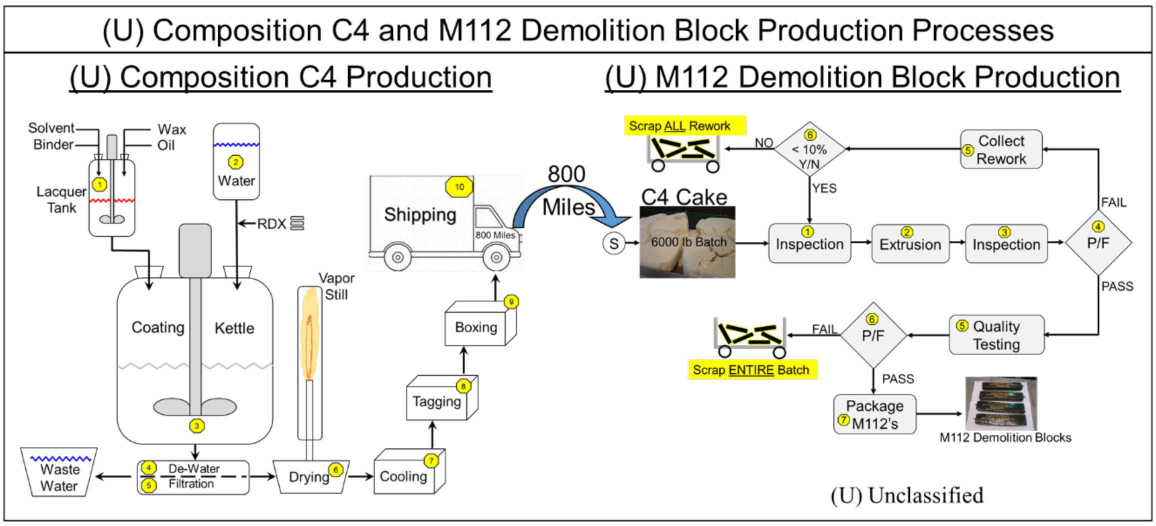


Figure 35 (U) Current Processes for C4 and M112 Production

The diagram shows individual steps for the manufacture of C4 bulk powder and M112 demolition block. In the C4 process, lacquer ingredients are weighed, pulverized, pumped and hand-loaded, heated, and agitated in a dissolver (1) for a minimum time, then drop-loaded into wagons and transferred to a coating kettle. Meantime water and nitramine powder are charged into the coating kettle (2) and heated. The lacquer is added to the kettle to begin a coating process as the slurry is agitated and heated (3.) After the slurry is distilled and cooled it is dumped into nutsches for de-watering (4) and filtration (5), weighed and transferred to a drying building, while the waste water is pumped off to a waste treatment facility. The entire batch is divided into small batches and dried in a kettle (6) while the vapors are distilled and partially reclaimed. The kettle-dried batches are transferred to another facility to cool (7) and be sampled for quality control. After the entire batch cools it is transferred to another facility to be mixed with a taggant (8) and dumped into fiberboard boxes (50 lb net explosive weight) (9), shrink-wrapped and eventually shipped 800 miles by truck (10) to another loading facility to be post-processed into the M112 configuration.
In M112 process, the C4 "cake" is received and warehoused until there is enough material to commence a new production campaign (12 consecutive labor shifts.) The "cake" is inspected (1) for different qualities by the judgment of one experienced operator and sorted into batches to be pressed into a screw extruder (2.) Unlike a TSE, where raw ingredients are compounded before die extrusion, here the screw extruder is used primarily to condition C4 powder and die-shape the final product. Heat and vacuum are applied to the C4 in order to increase processability and product density, causing an inadvertent volatilization and hardening of the material as it passes through a forming die. The M112 product is inspected (3) for quality features including dimension,

density, and plasticity. Rejected product (<10%) can be reintroduced back into the extrusion feeder to be reprocessed (4-6.) The acceptable product continues through to acceptance testing and packaging (4-7.)

(U) The demonstration technology took advantage of TSE continuous processing on a very small production platform (**Figure 36** & **Figure 37**). Raw materials were precisely metered into rotating screws within a barrel. The ingredients were efficiently compounded (see Section 3.1) as they moved through different sections of the screws. Process heat was not used in this demonstration due to the wall slip behavior of the silicone binder; however, air was evacuated from the product to drive up density before extrusion through a forming die. Since this technology is a continuous process, all reject product was scrapped. The demonstration envisioned minimal scrap once the process reached a steady state. As long as all ancillary systems were tuned to the desired production rate, the TSE process was controlled. Once an acceptable M112 block was extruded from the forming die, both the current manufacturing and the demonstration technology ceased to be contrasted. After the forming die, both products were packaged and evaluated according to military specification. The demonstration evaluated PAX-52 demolition blocks packaged to specification at a contractor facility using a different packaging method.

(U) Twin Screw Extrusion Process

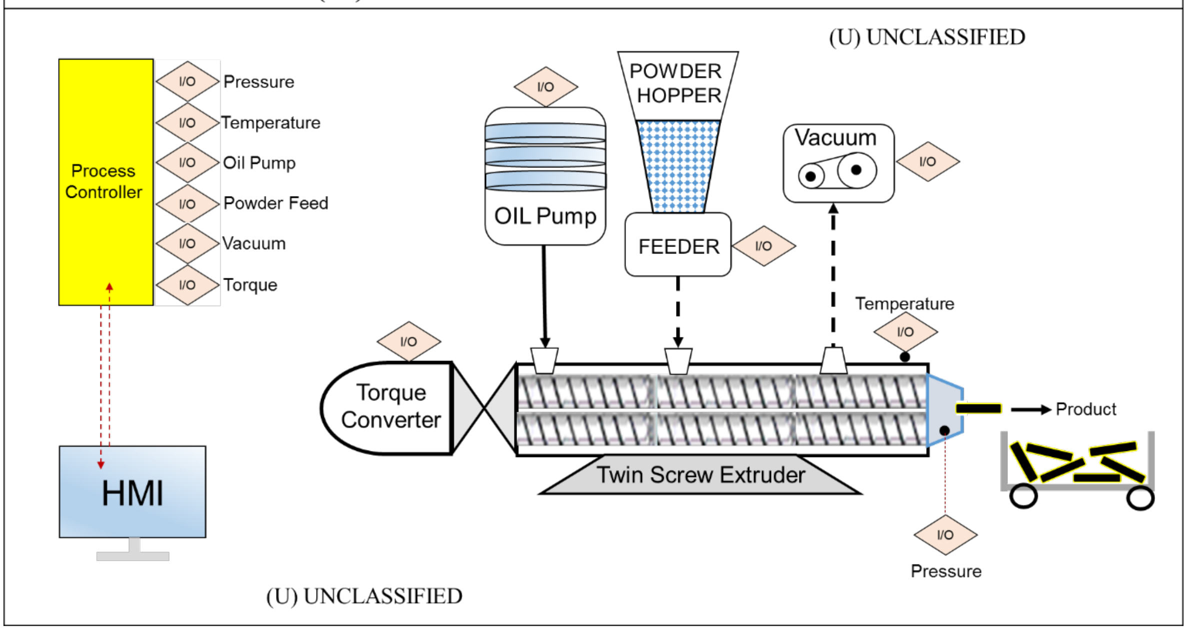


Figure 36 (U) Schematic of the Twin Screw Extruder System *The TSE stands about five feet high, eight feet long, and four feet wide. It is a rather compact system with multiple feed ports for powder and liquids. It is equipped with an array of temperature and pressure transducers along its length that feedback critical process data in real time to a logic controller. Each mechanical component in the system has an I/O reporting loop back to the controller. The process controller and Human Machine Interface (HMI) are remotely located in a reinforced concrete bunker to protect operators in the event of an initiation of energetic material.*

(U) Features of the Twin Screw Extruder

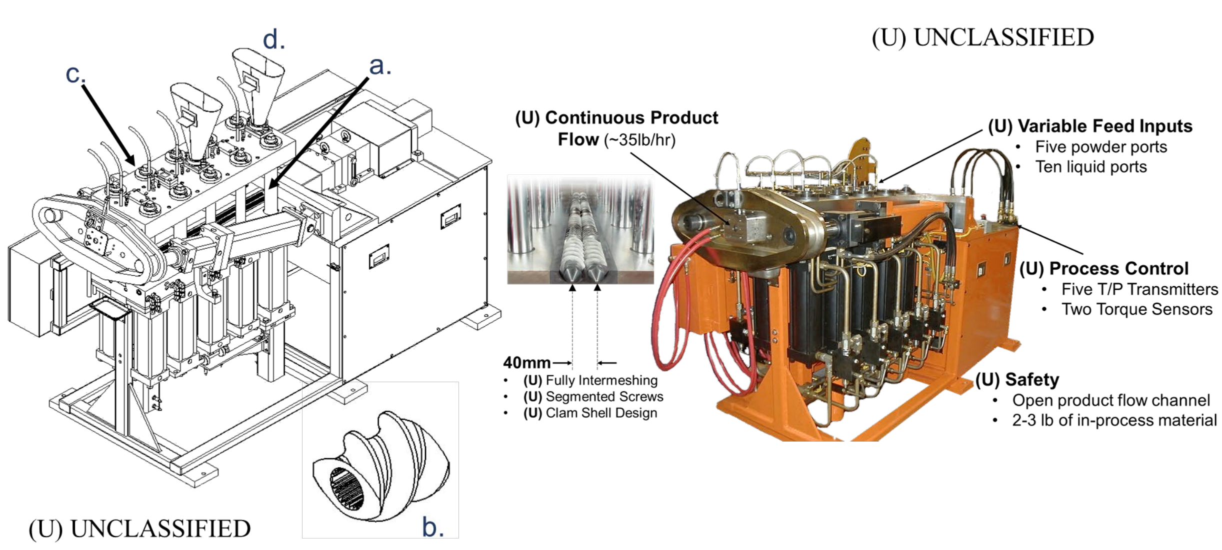


Figure 37 (U) Features of the Twin Screw Extruder(23) *The Universal Twin Screw Mixer / Extruder (UTSE) is a 40mm TSE capable of operating in single screw and twin screw; co or counter rotating modes. The twin screw mode can be either fully intermeshing or tangential. The UTSE is a clamshell design that is fixed on the bottom half while top half raises 12" vertically (a). The USTE is powered by a remote 15hp hydraulic motor which provides an opening and clamping (5000psi at die) of the barrel and the rotation of the screws (maximum of 300rpm). The segmented screw design (b) of the UTSE allows for numerous configurations using standard screw elements to obtain the desired feeding and mixing properties. The screw elements are positioned on a shaft with a 24 tooth spine. The UTSE has a full operating length of 25:1 (L: D). Screw clearance is 0.0125" between the tip of the element and the internal bore. The USTE is equipped with five solid loading ports and 10 liquid / temperature pressure transmitters (TPT) ports along the top barrel half (c). Powder material (feed stock) is fed into the USTE through stainless steel funnels which are covered by a Velostat top and connected to a feeder through a Velostat sock (d). The UTSE is also equipped with a vacuum port to collect excess solvents if needed. The UTSE also has the ability to provide torque measurements which are read directly from both shafts. This allows the operator to see if the material is building up on one shaft, or if material is blocked from entering into the extruder. The complete system is controlled remotely through a PLC-5/25 located in an explosive free environment. The PLC provides both instrumentation input and control output. All functions of the PLC are continuously data logged in both a real time graph and excel spread sheet for further analyses.*

The standard UTSE is designed with maximum safety in mind. A quick open system is designed to open the top barrel half, in a fraction of a sec to a length of 1". The quick open allows for venting the extruder (minimize pressure build up). The following safety considerations are integrated into the PLC for a quick open:

- *Maximum pressure of 1000psi*
- *Maximum temperature of 150^{o}F*
- *Maximum shaft torque of 50ft-lb*
- *An e-stop (emergency stop) is located on both the PLC control panel and on the UTSE*

The basic design of the UTSE provides also provides the following safety features:

- *A 5500psi shear washer on each hydraulic piston*

- *The hydraulic drive unit is mechanically set to release clamping pressure at 3000psi*
- *2-5lbs of in-process material*
- *A continuous open channel in co-rotating mode*
- *Designed to blow out toward the die end (away from operators)*
- *Proximity sensors prevent the barrel or die from closing without operator input*
- *Complete grounding and bonding system that is connected to building grounding system*
- *Rapid response deluge is positioned on top of extruder and feeders*
- *Microphone*
- *Closed circuit TV*

5.3 (U) SITE-RELATED PERMITS AND REGULATIONS

a. **(U) Picatinny Environmental Management System.** Picatinny Arsenal is committed to an active policy of protecting our environment for all activities. The installation environmental policy applies to all government and contractor employees, to include tenants that work at Picatinny. In this demonstration plan, ARDEC was considered a tenant of Picatinny Arsenal and was required to abide by all federal, state, and local regulations for environment protection in accordance with the installation environmental management system. Picatinny's Environmental Affairs Division was responsible for providing guidance and ensuring compliance with all environmental laws across multiple jurisdictions. The Division is the contact point for governing agencies seeking compliance and reporting standards and provided a web-based portal that aids in coordinating all installation activities that can potentially impact the environment. The Installation Commander, had approved the Environmental Management System in an Official Policy Statement 200-1, 5-July-2016 (24).

b. **(U) Picatinny Explosives Safety Management Program (ESMP)** This policy complies with the requirements set forth in Department of Defense (DoD) Directive 6055.9E, DoD Instruction 6055.16, DoD Manual 6055.09–M, Army Regulation (AR) 385–10, Department of Army Pamphlet (DA PAM) 385-64, DA PAM 385-65, AMC-R 385-10, and ARDEC Regulation 385-10. This ESMP identifies the roles and responsibilities of all ARDEC organizations with an Ammunition and Explosives (A&E) mission including any service components and contractors. It provides the policy and framework for addressing the sixteen elements (organization and staffing, site planning, facilities conformance, emergency response, tenants, master planning, ranges, contractors, accident prevention program, facility maintenance, demilitarization/destruction, risk management, explosives safety issuances, records management, inspections/evaluations/audits and training) required by the Army and DoD (25).

c. **(U) ARDEC-METC Environmental Safety and Occupational Health Plan.** This policy detailed the specific responsibilities for the ARDEC Munitions, Engineering and Technology Center (METC) in implementing an ESOH Program. The TSE operates in the jurisdiction of METC and benefits from the provisions of this program. The program's principle directives are to provide personnel with a safe work environment, support all federal laws and regulation related to ESOH, mitigate workplace risk through proactive management of personnel, property, and the environment, and take appropriate actions to correct deficiencies (26).

d. **(U) Local Hazard Familiarization Training.** All workers and trainees are required to receive hazard familiarization training for each operation they conduct to prevent inadvertent and potentially catastrophic impact on their safety.

e. **(U) Hazards Material Training and Certification.** All workers and trainees are required to take semi-annual Resource Conservation and Recovery Act (RCRA) training and be certified by Picatinny's Environmental Affairs Division to conduct operations involving hazardous materials. Building managers are also required to report on a monthly basis to the division on the status of RCRA training certifications and the conditions of their facilities.

f. **(U) Standing Operating Procedures (SOP).** The TSE facility uses an SOP to describe the operations for explosives, propellants, and pyrotechnic processed by the twin-screw extruder. In addition, the SOP covers auxiliary processing equipment: loss-in-weight solid and liquid feeders, solvent recovery system, in-line granulator and conveyors (27).

g. **(U) Other Local Regulations, Plans, and Permits**. There are other facility-specific documents written for the TSE facility and associated processes. All are derived from authorities leading back to the DoD. They include:

- **a.** Safety Site Plan
- **b.** Facility Explosives License
- **c.** Air Management Plan IS024 for Propellant Operations
- **d.** TSE Facility Storm Water Management Plan
- **e.** TSE Facility Spill Prevention and Containment (SPCC) Management Plan for energetic waste disposal (Red-Can), solvents and contaminated waste (Green Can)
- **f.** Emergency Response and Contingency Plan.

6 (U) TEST DESIGN

6.1 (U) CONCEPTUAL EXPERIMENTAL DESIGN

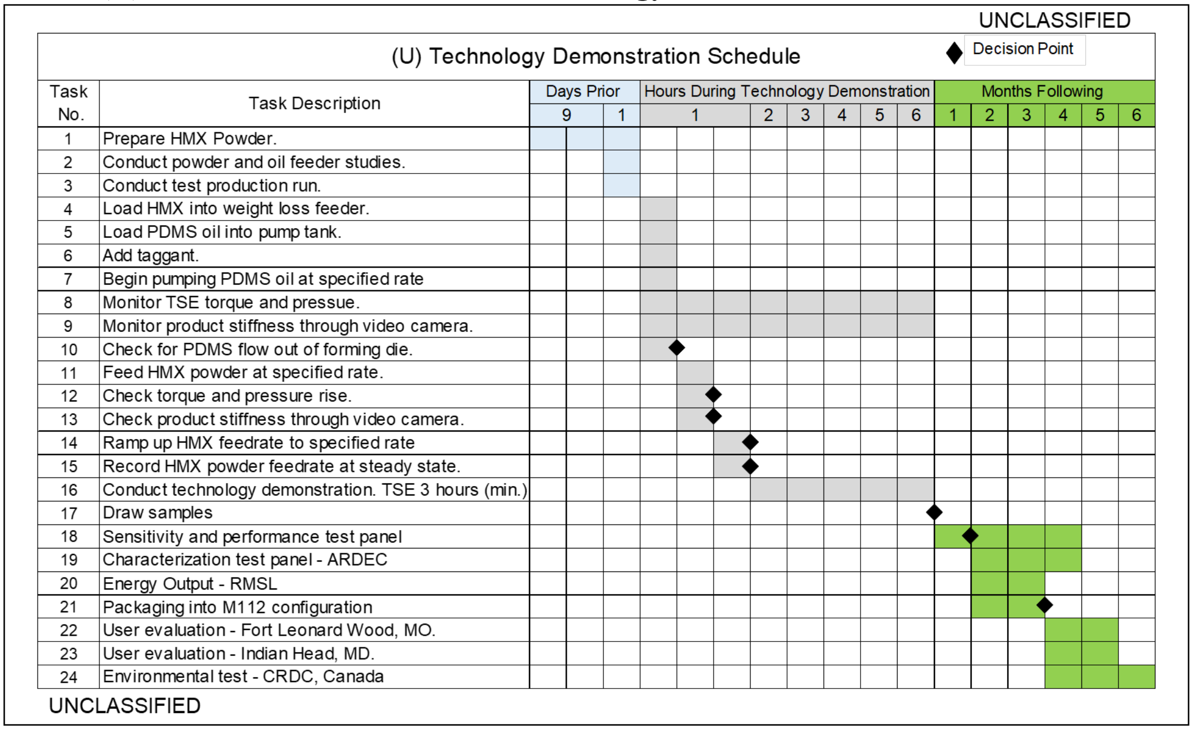
(U) The technology demonstration was an event that took place in a single day with several months of follow-up test and evaluation of PAX-52 that was produced during a continuous 3-hour production run. Operation workers prepared HMX Type II Class I powder by drying and sieving so it flowed freely through a USSS #20 sieve. All the energetic material was inspected for foreign object debris (FOD), particularly metal FOD, and kept in dry containers in preparation for the demonstration. The powder LOT number was recorded and a certificate of acceptance kept on file.

(U) Prior to the scheduled demonstration, loss-in-weight feeder and liquid feeder studies were conducted to verify accurate feed rates for both systems. Verified feed rates were recorded. A trial production run was conducted using verified feed rates to produce 15-20 lb of PAX-52. The extrudate was collected in a plastic-lined container and examined for smooth consistency, strength, friability, color, odor, and oil transfer. The material was assigned a LOT number and stored in a service magazine for future reference.

(U) On the day of the demonstration, a specified amount of the prepared HMX was loaded into a feeder hopper and covered. Several pounds of PDMS silicone oil was loaded into a pump reservoir without taggant. The oil solution was continuously mixed during production. Production began by feeding the oil solution into the rotating TSE to ensure all interior surfaces were wetted with oil and that the oil bled out of the forming die. Throughout the production process torque and pressure level were monitored for controlled conditions. The lead engineer began to slowly increase the powder feed rate, allowing the TSE dynamic conditions to stabilize after each increase. The product stiffness was visually accessed through a video camera while the powder feed rate was increased to a set point determined through the feeder studies. When the process reached a steady-state for pressure and torque the demonstration began. The process variables were monitored through at least three hours of continuous processing, or longer. The production run ended when the lead engineer dialed back the HMX feed to zero and confirmed that pressure and torque values dropped to zero and a loose, oily mixture extruded from the die face.

(U) The material was collected, weighed, packaged, issued a LOT number of RDD17B046-E001, and transported to a service magazine. From there a small sample of the TSE PAX-52 was tested for small scale physical sensitivity. Positive sensitivity data was a decision point that allowed the material to be transported in accordance with an interim hazard classification specific to PAX-52. Some material was sent for energetic characterization at ARDEC, Picatinny Arsenal. A sample was sent for energy output performance tests at Rocky Mountain Scientific Laboratory (RMSL), Boulder, CO. Some was sent for production of 245 (ea) M112 demolition blocks at Accurate Energetic Systems (AES), Tennessee, which was then forwarded to Fort Leonard Wood (FLW), MO for user evaluation. Lastly, a sample was sent to CRDC, Canada for RDX deposition testing. A Gantt chart for the schedule of tasks is provided in **Table 7.**

Table 7 (U) Gantt Chart Schedule for Technology Demonstration



6.2 (U) PRE-DEMONSTRATION TESTING

(U) Twin Screw Extrusion of the simulants of PAX 52 with different particle size distributions and maximum packing fractions. See Section 3.1

(U) Mathematical Modeling of the flow and deformation occurring in the slit die for used for the processing of the live PAX 52 formulation. See Section 3.1

(U) Mixing Index determination from the HPLC data collected on PAX 52 samples that were twin screw extruded at ARDEC. See Section 3.1and Section 11.3

(U) Development of Processing Simulants for PAX 52 and rheological behavior of simulant suspensions. See Section 3.1

(U) Early development of PAX-52 in 2014 showed good progress in compounding nitramine HMX with PDMS oil by twin screw extrusion. The work was completed before this ESTCP project was launched and never written into a tech report. The work investigated bi-modal particle size distributions to achieve an optimal packing fraction in candidate formulations. Lab scale formulations indicated that a bi-modal distribution of Class I and Class V HMX in a 4:1 ratio would yield a high solids product with excellent physical properties. The work culminated in a pilot scale demonstration of a candidate formulation by the extrusion of 50kg of M112 demolition blocks. The demonstration was halted due to problems feeding Class V HMX into the TSE. Rather than abort the pilot scale production, the small particle HMX was eliminated from the formulation and continued feeding only larger particle Class I HMX powder with silicone oil into the TSE.

(U) The modified process produced excellent quality product extruded into a 1” x 2” continuous belt, which was hand-cut and packaged into the M112 configuration. Over 50 kg of M112 blocks was produced and prepared for testing. 40 kg of the TSE M112 blocks were shipped to Ft. Polk, LA for a wall-breach target demonstration conducted by the Demolitions and Special Munitions Branch, ARDEC. The remaining 10 kg was shipped to Applied Research and Technology Institute (ARTI) to be used for material qualification tests conducted by RMSL the auspices of a follow-on R&D effort funded by the current ESTCP project.

(U) There were some details of an earlier production PAX-52 M112 blocks that were sent to ARTI (LOT RDD14K046-001) for performance testing and held since December 2014. Only two short video clips of the die-extrusion and wall-breach demonstrations exist as documentation that the material was actually produced; however, no records of any material sensitivity or performance tests could be found. The PI thought it prudent to visit the ARTI in person to inspect the material and document its physical characteristics. A random M112 specimen was inspected for a variety of physical characteristics. It is reported here that the material was found to be in excellent condition. Very light and spotty deposits of oil were observed when the specimen was held to a light, but no accumulation that would suggest that the silicone oil migrated out of the material over time (**Figure 38**). The moldability of the material was superior to any other PAX-52 formulation previously produced by the RAM or blade method. By the same comparison, the sample did exhibit a more than expected amount of transfer to a gloved-hand, but not with a buildup of either bare particles or free oil. This condition indicates more design space for higher packing of solids into future formulations. The PAX-52 was continually stored in one of ARTI outdoor magazines and weathered one and a half weather cycles since it arrived in December 2014, but showed no signs of aging degradation or decomposition.

Figure 38 (U) PAX-52 with very light oil deposits.

(U) Since no documented testing was ever conducted on the TSE PAX-52 (LOT RDD14K046-001), it was incumbent upon the PI to order the reproduction of the same formulation by the same TSE process parameters used in the original production run. The project had to document that the candidate formulation was reproducible by the TSE method and could meet acceptable performance criteria; otherwise, previous PAX-52 performance claims made to stakeholders would be undermined and the project would suffer a loss of credibility. A 35-pound batch of material (RDD16E046-134) was produced and sent for a panel of energetic material characterization tests. The TSE production run was successful and the test results were all positive (**Appendix A: Section 11.1**). This hurdle past, the project was able to look ahead to the technology demonstration with a high degree of confidence for success. The results obtained for this lot of

PAX-52 is compared to that obtained from the current demonstration in the following sections of this report.

6.3 (U) DESIGN AND LAYOUT OF TECHNOLOGY COMPONENTS

(U) The demonstration technology took advantage of TSE continuous processing on a very small production platform (**Figure 36 & Figure 37**). Raw materials were precisely metered into rotating screws within a clam-shell barrel. HMX powder was delivered by a loss-in-weight feeder (details provided in **Section 11.2)** that continuously reported the powder feed rate to a PLC controller. Silicone oil was delivered by a precision diaphragm pump that continuously reported the fluid feed rate to a PLC controller. The ingredients were efficiently compounded as they moved through different sections of the screws. Process heat was not used in this demonstration due to the wall slip behavior of the silicone binder; however, air was evacuated from the product to drive up density before extrusion through a forming die. Temperature, drive torque, and product pressure values were reported to the controller and monitored real-time by the lead engineer. Feedback data were generated every second for each variable controlled by the PLC and stored on a computer hard drive for post-production analysis.

(U) Product extruded from the TSE forming die was not conveyed or packaged in the M112 demolition block configuration. It fell freely into a collection bin lined with a double Velostat bag. The product was weighed and samples were drawn for small-scale sensitivity tests before it was transported to a service magazine. All scrap material was sent to a NJDEP regulated incineration facility located at ARDEC.

(U) The demonstration was a live energetic operation which required all operators to be remotely located in a secure, reinforced control bunker equipped with audio and visual monitors. The lead engineer had the capability to adjust powder and liquid feed rates from the control room. The operation was expected to run continuously for three hours without stopping. No operator was permitted to re-enter the operation bay once the demonstration began. In the event of an unintended interruption of production, due to safety or security issues, the demonstration would be aborted until another demonstration is allowed. If the interruption was process related, the conditions would be fully documented before the demonstration was to be allowed to continue. The demonstration platform and all ancillary systems, equipment and controls are part of an existing pilot-scale production facility and remained in place after the technology demonstration.

6.4 (U) FIELD TESTING

(U) This section describes details of the various tasks involved with the technology demonstration as planned in **Table 7**. All tasks and procedures related to processing energetics on the UTSE were conducted in accordance with the facility Standing Operating Procedure (27). Other tasks involving test and evaluation followed explosives safety regulations in accordance with DA-PAM 385-64 (28) and local safety standards. The following are task-specific details, descriptions, and method references:

(U) **Task 1.** HMX powder was prepared. Energetic powders were shipped from the manufacturer in drums and completely submerged in water/isopropyl alcohol (IPA) to desensitize the material for shipping and storage. The wet powder was removed from the bath, leaving excess liquid, but still was required to be dried and sieved several days prior to the actual demonstration. Large plastic static-dissipative scoops were used to transfer the wet powder onto fiber-reinforced phenolic trays. There the thick, mud-like powder mass was manually spread across the tray to enhance the drying process. Multiple drying trays were laid up in an explosion-proofed drying oven and heated for several days to drive off all moisture. The dried powder was collected and processed through a metal detector to ensure no foreign objects (packaging staples, fasteners, pen clips, etc.) were present in the powder. The final step in preparing the powder was to sieve out all agglomerates that may have formed during the shipping and storage. The prepared powder was sealed in 50-pound increments until ready for production.

(U) **Task 2.** Powder and oil feeder studies were conducted. For both ingredients it was necessary to verify the nominal feed rate for a given set of variables associated with the planned production. Ambient conditions, particle size, viscosity, feeder accuracy were some of the variables that changed from one product to the next during a given production campaign. For the technology demonstration it was not expected that feed rates for either ingredient were to remain constant throughout. The nominal feed rate was determined by a sample of 30 feed rates over duration of 60 seconds. The feed rates were required to be within 1% of each other to determine the nominal feed rate. Feed rates were verified by using an external scale with sampling rate of every second, which were averaged over 10 seconds. Once the feeders were calibrated, the lead engineer began planning a trial production run based on a known weight percentage for each component of the PAX-52 formulation. Based on previous PAX-52 experiments good quality material was extruded at a specified powder feed rate with a specified oil feed rate, the total undisclosed here. Using a target production weight, the lead engineer established feeder set points for the demonstration PAX-52 formulation.

(U) **Task 3.** Trial production run was conducted. TSE was started-up in accordance with SOP.

Step 1. HMX powder was loaded: The material was brought from the temporary storage area and connected to the required grounding straps. The cover of the feed hopper to be filled was removed. Enough of the solid material was transferred to cover the feeder screw. The operator either poured or scooped the material into the hopper. Clamps were secured on the feed hopper lid.
Step 2. Oil hopper was loaded: The flanged cover on loss-in-weight feed hopper of the liquid feeder was removed. The grounding strap was attached. The lid from the raw material container was removed. The amount of pre-made material specified by the lead engineer was transferred into the holding tank. The operator either poured or scooped the material into the hoppers. The flanged cover was placed on the feed hopper and nuts and bolts were laid on the cover.
Step 3. A collection container was placed at the discharge port of the extruder. This container collected the initial unacceptable material that was processed as energetic waste. A second properly grounded collection container was also placed under the discharge port of the extruder. Once a quality product was being produced the operator initiated a diverter, and the product was collected in the second grounded/bonded container.
Step 4. TSE was started: The operator checked to ensure the screw speed was set below 200 RPM. It was visually confirmed that screws were turning from TV monitor. Funnel vibrators were started (as required per the lead engineer) using the controls area. This was confirmed using camera audio.
Step 5. Oil flow began: Oil was pumped into the screw system at the feed rate determined by the feeder study. Flow was confirmed. Oil was required to have bled out from the die face.

Step 6. Powder feed began: The powder feed was started at 75 percent of the rate determined by the feeder study.
Step 7. Powder feed rate was ramped up. Powder feed rate was slowly increased in small increments. System torque, pressure, and condition of the extrudate were monitored. Both pressure and torque began to rise as the material became packed with higher solids content. The lead engineer determined by the conditions and variables of the system whether the trial run was able to attain the desired feed rates.
Step 8. Continuous process at steady state was run. The process ran continuously for 30 minutes without deviation outside the control limits to ensure that the trial was a success. If the trial run would have been aborted for any reason, it would have been deemed not valid and rerun the next day.
Step 9. Production was stopped. Feeding of HMX powder into the TSE was stopped. Feeding of the Oil was continued until a loosely formed extrudate began to flow from the die. This oil flush ensured that no accumulations of solidly mixed PAX-52 were latent in the screw to barrel clearances. Feeding of the oil was then stopped. TSE was cleaned out and shut down in accordance with the facility SOP. All scrap and clean-out material was processed as energetic waste.

(U) **Tasks 4-15.** TSE was set up for technology demonstration. Steps 1-7 in previous task 3 were followed.

(U) **Tasks 16-17.** Technology demonstration of continuous processing of PAX-52 was conducted. Step 8 in the previous task was followed, but for a minimum of three hours. Once the demonstration was complete, production was stopped in accordance with step 9 in the previous task.
(U) A LOT number was assigned to the batch and 30 random samples were drawn of approximately 20 grams each using a 20 mL plastic scoop. Bulk material was packaged in fiberboard boxes and sent to a service magazine before further handling and shipping.

(U) **Task 18.** PAX-52 sample was sent for sensitivity and performance testing. Tests for ESD, Impact, Friction, and Thermal Decomposition were to have passed before further testing or transport were completed. Appendix **D 11.3:1-4.**

(U) **Task 19.** PAX-52 was sent for a panel of characterization testing. Appendix **D: 11.3:5-15**.

(U) **Task 20.** Bulk quantity of PAX-52 was shipped to RMSL for Energy Output Test. Appendix **D: 20**

(U) **Task 21.** Quantity was shipped to contractor for off-site packaging into M112 configuration. Appendix **D 11.3:18**.

(U) **Task 22-23.** M112 blocks were shipped from AES to FLW and Indian Head for user evaluation. Appendix **D 11.3: 21 - 22.**

(U) **Task 24.** Quantity of material was shipped to CRDC, Canada for characterization and environmental test. Appendix **D 11.3: 23**

6.5 (U) MEASUREMENT AND MONITORING PLAN

Table 8 (U) Process Parameters

Parameter	*Method*	*Method Description*
HMX powder feed rate.	Loss in weight over time. Pounds per hour.	• Feeder controller monitors weight loss over time. • Feedback each second.
PDMS/taggant feed rate	Loss in weight over time. Pounds per hour.	• Feeder controller monitors weight loss over time. • Feedback each second.
Die pressure	Transducer at die port	• 4-20 mA current input to controller
Temperature	Thermocouple at die	• mV thermocouple voltage converted to mA current input to controller.
Drive speed	Revolutions per minute.	• Magnetic pickup on motor shaft. • Induced current pulses counted per minute by controller.
Drive torque	Direct from strain gage on drive shaft. Foot-pounds (ft-lb)	• Voltage resistance converted to 4-20 mA current input controller. • Gage calibrated using a torque wrench.
Production rate	Adding powder and oil feed rates. lb/hr.	Controller continuously calculates and reports.
Total product	Mass by weight	Digital scale. Army Calibrated
Total waste	Mass by weight	Digital scale. Army Calibrated
Time	Clock time.	Controller internal clock.
HMX exposure	XAD-2 tubes	• Tubes placed at location to detect levels of HMX exposure. • Sampling rate: 1 liter per minute. • Total sample: 40-60 liters.
PAX-52 Analysis and Performance Characterization	Standard AOP-7 Tests	Appendix C, Section 11.3 11.3 Page 118
Physical properties: Force resistance	• Penetrometer • MLD-DTL 50523B paragraph 4.4.1	• Cone penetration method ASTM D217
Specific gravity	• Archimedes' method • MLD-DTL 50523B paragraph 4.4.2	• Appendix C, Section 19 Page 120
Physical Features	Qualitative assessment of PAX-52 by combat engineers.	• User evaluation survey Appendix 11.3.19 • Technical manuals for medium and conical warheads. TM-5-1375-238-10
Performance	• Qualitative assessment of PAX-52 • EOD render-safe procedures.	Internal report

6.6 (U) LABORATORY MATERIAL TESTING

Table 9 (U) Laboratory Testing Requirements

Engineering Requirement	Test	Acceptance Criteria	References
Impact Sensitivity	ERL Impact	Impact sensitivity of Comp C4 50% ≥ 64 cm.	Appendix D **Section 1**
Friction Sensitivity	BAM Friction	Friction sensitivity of Comp C4 50% > 360 N	Appendix D **Section 2**
ESD Sensitivity	ESD	ESD > HMX Class I -2 >0.051J	Appendix D **Section 3**
Exothermic Onset	DSC	Onset temperature: > 220°C	Appendix D **Section 4**
Composition	HPLC	Minimum HMX content: xx% by weight	Appendix D **Section 5**
Homogeneity	HPLC	Mixedness Index: ≥ 0.99	Appendix D **Section 6**
Chemical Stability	VTS	Gas evolved per gram of explosive: <2 ml 48 hours at 100 ºC	Appendix D **Section 8**
Detonation Velocity	Detonation Velocity	Detonation velocity of Comp C4: >7.50km/sec at 99% TMD	Appendix D **Section 10**
Detonation Pressure	Plate Dent Test	Detonation pressure of C4: ≥ 24 Gpa	Appendix D **Section 11**
Sensitivity to shock	Cap Test	Sustained Detonation Clear cut witness plate	Appendix D **Section13**
Sensitivity to shock	Insensitive High Explosive (IHE) Gap Test	Card Gap: 161-171 cards	Appendix D **Section: 14**
Thermal Response	1. VCCT (AOP-7 202.01.002) 2. Small Scale Burning Test (TB700-2 Para. 5.4a)	1. Pass: No detonation response 2. Pass: No detonation response	Appendix D **Section 9**
Age stability	Exudation	No accumulation of oil No decomposition of material	Appendix D **Section 17**
Specific gravity	Archimedes' method	Specific gravity: ≥ 1.50	Appendix D **Section 19**
Force resistance Plasticity	Penetrometer	Force resistance: ≥ 2.0 lb, ≤ 8.0 lb	Appendix C **Section 18**
Energy Output	Plate Cutting test	Plate cut cleanly into two sections.	Appendix D **Section 20** ASTM A36 grade 1018 steel
HMX exposure	Air sampling	6 times < RDX inhalation limit 10- hour time weighted average: 0.25 mg/m^3	NIOSH 2010 Appendix D **Section 24**

7 (U) PERFORMANCE ASSESSMENT

7.1 (U) STEADY-STATE PROCESSING OF PAX-52

(U) The technology demonstration required a 3-hour continuous production of PAX-52. Success was defined as the control of the process to within three standard deviations of the sample mean for each of the variables sampled by the PLC at evenly divided intervals. During the demonstration temperature of the PAX-52 product, which was one of three variables defined as success criteria, was not recorded; however, several other process parameters were recorded: pressure, powder and liquid feed rates, and drive torque (**Figure 39**). This particular demonstration objective was deemed only a partial success due to missing data requirements, a slight loss of process control and an unmet run time requirement; however, the technology demonstration provided validity to the proposed continuous production method.

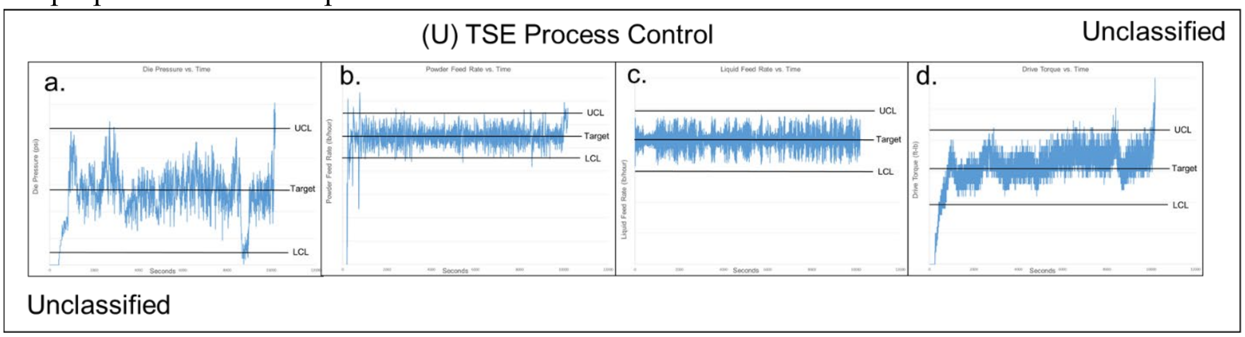


Figure 39 (U) TSE Steady-state Process Control *Process feedback data was recorded for 2.5 hours of steady-state production of PAX-52. Upper and lower control limits, defined by three standard deviations, were a good starting point to determine whether the TSE process was controlled. Die pressure (a.) data shows one excursion of low pressure toward the end of the run. There was no explanation for the sudden drop in pressure. Powder and liquid feed rates were largely steady for the duration (b., c.). The drive torque tended to increase toward the upper control limit with minor excursions for short intervals (d.) Most of the outside excursions are considered spikes attributed to feedback noise and could be filtered by decreasing the frequency of sampling.*

7.2 (U) PAX-52 CONSISTS OF ONLY TWO INGREDIENTS.

(U) Due to the sensitive nature of the PAX-52 formulation, some values were omitted in this section.

(U) The target composition of PAX-52, not including taggant was xx% HMX and xx% Silicone. Analysis of the composition was conducted via HPLC to obtain the percent HMX by weight. As there are only two components in the formulation, the percentage of silicone was calculated by difference. Each analysis was performed in triplicate to ensure accuracy. Previous lots of prepared PAX-52 mixed at Rocky Mountain Scientific Laboratory exhibited a range of X-X% HMX, with higher solids loading proving to be difficult to achieve. The current PAX-52 lot produced contained xx% HMX and xx% Silicone. Though only two ingredients were fed into the TSE during the production run, it is important to show that there are no by products or process aids detected in the material composition. The weight percentages of the composition derived by the feed rates of solids and liquids were significantly different than the analysis. These figures were consistent, with a standard deviation of 0.3% for the measurements in triplicate. Per the guidelines set forth, this demonstration is deemed a **success** as the samples met the goal of consistently showing an HMX content greater than xx% by weight.

7.3 (U) DEMOLITION CHARGE CROSS-SECTION

(U) A representative sample of extruded demolition block was measured with a caliper and found to be 2.00 inches wide by 1.00 inches thick, allowing for a shallow concavity on each side. This measurement agrees with the **success** criteria.

7.4 (U) M112 SPECIFIC GRAVITY

(U) The specific gravity of PAX-52 was determined to be 1.6880 g/cm^3 according to Archimedes' water method. This result is considered a **success** as it exceeded the specification of 1.50 g/cm^3.

7.5 (U) PHYSICAL PROPERTIES

(U) The project was unable to assess the plasticity of the PAX-52 in the M112 configuration due to safety restrictions. The equipment required to accomplish this test was not located in an area that could accommodate Class I energetic material. When the material was packaged, the contractor stated that, in his experience, the bulk material and packaged M112s were the same shape and stiffness as a C4 block. This is a measure of a key property that affects the end-user's ability to use the product in IAW technical bulletins. Feedback from the user community affirmed that the PAX-52 M112 blocks showed no apparent difference in plasticity; however, one note from a user evaluation described the material as too soft to hold an initiator device in place. This objective is considered a **no-test**. Future development will address making the formulation stiffer by adding more solid components.

7.6 (U) ENERGY OUTPUT

(U) The Plate Cutting Energy Output test was performed to measure the effectiveness of PAX-52 against a steel witness plate. The alternate method was implemented, where the thickness of the cut M112 block varied and was modified up/down by a 0.003 inch spacer. The tests were conducted per the Bruceton 50% analysis using an approximate 5 shot pre-test to establish the Go/No Go Threshold. With a thickness closer to 0.5 inches, the M112 demolition block with PAX-52 effectively cut the steel plate in 2 sections. This test establishes that PAX-52 has enough energy output to exceed the requirements for C4 acceptance. **Success.**

(U) To evaluate the energy output of PAX-52, a plate cut test was performed on 1 in. thick ASTM A36 grade 1018 steel plate. (29) The charges for this test were cut from hand packed M112 size charges to the dimensions of 0.5 in. x 1.5 in. x10.0 in. The charge was placed in the center of the plate. All charges were primed with a RP-83 detonator in the middle of the charge. The end of the blasting cap was covered with a strip of explosive with the dimensions of 0.5 in. x 1.0 in. x 4.0 in. The steel witness plates were supported along opposite sides by 2 in. thick steel supports.

Figure 40 provides an illustration of the test setup.

(U) A modified plate cutting test was used in the exact procedure in MIL-DTL-50523B with the exception of the 0.5 inch dimension of the cut M112 block. This thickness dimension was a variable in the alternate test and modified up/down by a 0.03 inch spacer based on the preceding test (**Table 10**). Tests were conducted per the Bruceton 50% analysis using an approximate 5 shot pre-test to establish the GO/NOGO threshold.

(U) For this test Rocky Mountain Scientific Laboratory (RMSL) hand-packed PAX-52 into a demolition block form, the thickness of which was varied through the use of calibrated spacers. Forming the blocks in this way may have led to low or inconsistent densities, potentially contributing to some to the variance observed in the cut threshold thickness. The charges used in this testing had a density of 1.68 ± 0.03 g/cc based on the charge mass divided by physical measurements of volume.

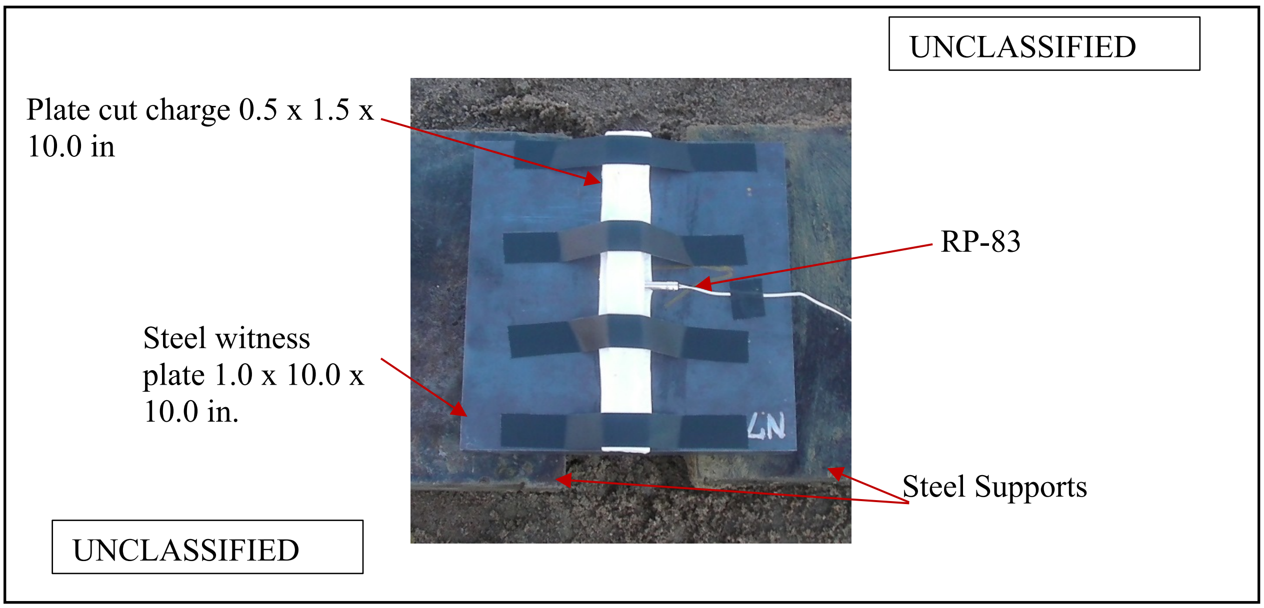


Figure 40 (U) Plate Cut Test Setup

(U) RMSL has used the SenTest numerical analysis software to help in statistical analysis of the modified plate cut test. Plate cut tests showed a 50% cut threshold block thickness of 0.463 ± 0.027”. A plot detailing the probability of cut as a function of block thickness along with the corresponding statistical confidence levels can be seen in Appendix **F1**

Table 10 (U) Plate Cutting Energy Output Test Results.

(U) UNCLASSIFIED

PAX-52 Plate Cutting Energy Output Test					
Test #	Charge Thickness (in.)	Charge Mass (g)	Charge Density (g/cc)	Primer Mass (g)	Plate Cut
1	0.491	224	--	59.2	N
2	0.491	221	--	56.8	Y
3	0.306	138.4	--	57.6	N
4	0.401	191.4	--	60.2	N
5	0.431	176.8	1.67	56.6	N
6	0.461	193.4	1.71	58.1	N
7	0.491	199.8	1.66	56.2	Y
8	0.491	204.8	1.70	59.5	Y
9	0.461	191.9	1.69	60.5	Y
10	0.431	172.3	1.63	59.4	N
11	0.461	191.6	1.69	60.2	Y

(U) UNCLASSIFIED

7.7 (U) PHYSICAL SENSITIVITY

(U) The physical sensitivity of PAX-52 was determined through the performance of four (4) tests: ERL Type 12 Impact Test, BAM Friction Test, ABL Electrostatic Discharge Test, and Thermal Stability.

(U) **The ERL, Type 12 Impact Tester** utilized a 2 ½ kg drop weight to determine the impact sensitivity of the PAX-52 sample. The Bruceton method of statistical analysis was implemented to determine a 50% drop height corresponding to the 50% probability of initiation used to measure the impact sensitivity. As indicated in **Table 11**, PAX-52 displayed 4 reactions out of 10 trials at the maximum drop height of 100 cm, which is an identical result to previous samples produced. This result is much less sensitive than that of Class 1 RDX and HMX Cl-5 T2, **(success)** which react at an impact heights of 26.1 and 28 cm, respectively. Per impact sensitivity guidelines, PAX-52 shall be deemed **acceptable** for transport and handling as the results are greater than HMX Cl-5 T2.

Table 11 (U) ERL, Type 12 Impact Test Results

(U) UNCLASSIFIED

ERL, Type 12 Impact Test	
Sample	50% Impact Height (cm)
Class 1 RDX	26.1
PAX-52	100 (4 reactions out of 10 trials)

(U) UNCLASSIFIED

(U) **The BAM Friction** tester was used to determine the friction sensitivity of the PAX-52 sample. The results were reported as a reaction if flash, smoke, or audible signal are observed. An iterative procedure was used to determine the highest load at which no positive results were obtained in 10 trials, which value is considered the Threshold of Initiation Level (TIL). Testing began at the maximum load of 360 N and the load reduced until no reactions are observed in ten trials. This demonstration was **successful** as PAX-52 showed no reaction in 10 trials at 324 N. **Table 12** Compared to RDX which reacts at 168 N, PAX-52 is much less sensitive. This result is comparable to the older samples of PAX-52 produced, which exhibited identical friction sensitivity.

Table 12 (U) BAM Friction Test Results

(U) UNCLASSIFIED

(U) BAM Friction		
Sample	Energy (N)	
	Reacted	No Reaction in 10 Trials
Class 1 RDX	168	160
PAX-52	360	324

(U) UNCLASSIFIED

(U) The ABL ESD Test was used to determine the sensitivity of the PAX-52 to electrostatic discharge. In this test, an approaching electrode assembly is used, in which the upper electrode is lowered to a preset distance of 0.015 inches above the lower electrode containing the sample and immediately raised to its initial position. A positive result is defined as a flash, spark, burn, or noise upon discharge of the upper electrode through the material in the lower electrode. If a reaction is observed at any point, an iterative process is used to determine the highest energy level at which no positive result occurs in 20 trials. The testing began at 0.25 joules, with a starting capacitance of 0.02 microfarad and starting voltage of 5.0 kV. The energy is reduced to the next lowest level if a reaction occurs. Results for PAX-52 as compared to Class 1 RDX are depicted in **Table 13**. PAX-52 exhibited low ESD sensitivity, though not as low as that of RDX. **Success**

Table 13 (U) Electrostatic Discharge Test Results

(U) UNCLASSIFIED

(U) Electrostatic Discharge		
Sample	Energy (J)	
	Reacted	No Reaction in 20 Trials
Class 1 RDX	0.025	0.020
PAX-52	0.063	0.051

(U) UNCLASSIFIED

(U) Thermal Stability: PAX-52 was subjected to an elevated temperature (75°C) for a period of 48 hours to evaluate its chemical stability. The material was weighed before and after heating. A -0.05% change in mass occurred according to the measurements, meaning that there was no decomposition or off-gassing, no exothermic reactions, and no accumulated exudation of oil. As such, PAX-52 **passes** the 48-hour thermal stability test, indicating that there is no evidence of instability.

(U) DSC testing and analysis was performed by RMSL personnel utilizing a TA Q2000 instrument. The tests were conducted in accordance with (IAW) AOP-7 (US/202.01.022) and run at 5°C/min ramp. Three DSC parameters were measured and recorded: exotherm onset temperatures, peak temperatures, and heats of reaction. The exotherm onset temperature of the sample is the lowest temperature at which positive deflection from the baseline is first observed, while peak temperatures correspond to the maximum deflection of the DSC curve. Heat of reaction is the energy released or absorbed by the sample (the area under the curve).

(U) Small samples (1-2 mg) of the subject material were placed into hermetically sealed pans with pinholes laser cut into the top. The sample container was then pressed closed with a die. The sealed sample was then visually inspected and weighed before analysis. The DSC was calibrated with indium and test samples were run under nitrogen purge gas flow (50 mL/min). The sample temperature was increased from an ambient temperature of 40°C to 400°C at a linear ramp rate. **Figure 41** provides a representative DSC curve collected during this effort and shows a slight endothermic phase change ca. 175°C and then a large exothermic reaction at the critical temperature.

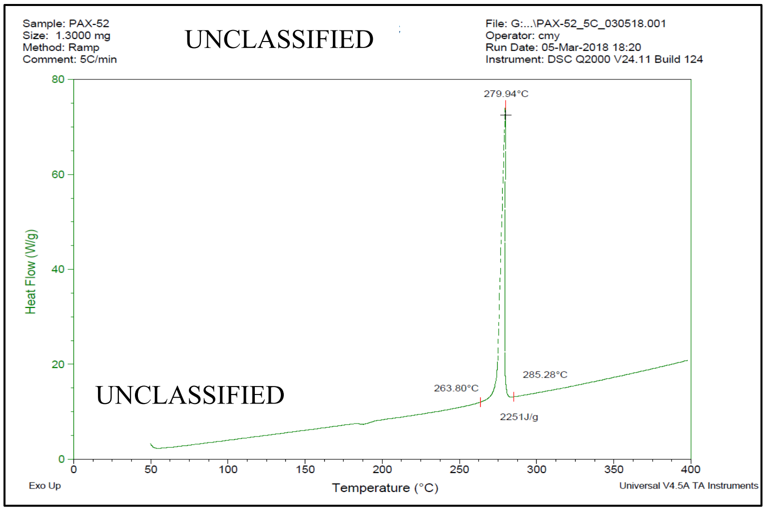


Figure 41 (U) PAX-52 DSC Plot (5°C/min)

(U) RMSL conducted variable ramp rate testing on samples as part of determining the Arrhenius kinetics of PAX-52 to assist in predicting the critical temperature at larger charge scales. Linear ramp testing was performed IAW AOP-7 (US/202.01.022) at linear ramp rates of 3, 5, 7, 10, and 20 °C/min. Measurements were made of exotherm/endotherm onset temperatures, peak temperatures, and heat of reaction to evaluate the overall thermal stability of the sample

(U) The Arrhenius kinetics of the decomposition reaction can be determined by measuring the shift of the exothermic peak as a function of ramp rate, allowing prediction of the critical temperature as a function of charge size for PAX-52 (**Table 14**). Plots for decomposition kinetics Appendix **F2** and predictions of critical temperature as a function of charge scale can be seen in Appendix **F3. Success**

Table 14 (U) Exotherm Peak Temperatures for PAX-52 vs. Linear Ramp Rate

(U) UNCLASSIFIED

Ramp Rate (°C/min)	Peak Temperatures (°C)				
	20	10	7	5	3
Exotherm Peak Temperature (°C)	292.64	284.98	281.62	279.94	275.00

(U) UNCLASSIFIED

7.8 (U) SHOCK SENSITIVITY

PAX-52 was tested for shock sensitivity in two different tests. Cap sensitivity verifies the material is sensitive enough to shock to initiate with a blast cap while the IHE test quantifies the shock sensitivity of PAX-52. **Success**

(U) In order to determine the shock sensitivity of PAX-52, both Extremely Insensitive Substance (EIS) Cap Sensitivity and IHE Gap Tests were performed. The purpose in completing the Cap Sensitivity Test is to determine whether there is a mass explosion of the contents from accidental ignition or initiation by a No. 8 blasting cap. It was performed by placing a cardboard tube containing the sample on top of a steel witness plate with a No. 8 cap inserted at the top of the tube. Detonation was determined by examining the witness plate for tears or penetration. PAX-52 failed this test (therefore, not an EIS), as the use of 1664.2 grams of material resulted in a **positive** detonation reaction. The test shot completely destroyed the witness plate and standoff steel ring leaving only the base plate that was located below (**Figure 42**). This verifies that PAX-3 is cap sensitive. This result is consistent with previous lots of PAX-52, where the material had also completely destroyed the steel witness plate and standoff steel ring. **Success**

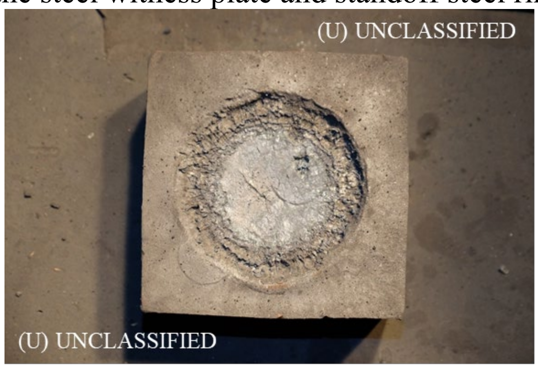


Figure 42 (U) Witness Plate *Post-test picture of the base plate located under the witness plate and standoff steel ring during the Cap Sensitivity Test. A positive reaction occurred, where the witness plate and ring were completely destroyed.*

(U) IHE Gap test was also performed on PAX-52, with results depicted in **Table 15**. Samples of PAX-52 were pressed to the density of 1.77 g/cc and drop-loaded into steel tubes. A total of eleven shots were fired and determined a shock attenuation gap of approximately 160 cards, which is slightly less than the goal of 161-171 cards. Better less than more - **success**.

Table 15 (U) PAX-52 IHE Gap Test results.

(U) UNCLASSIFIED

(U) IHE Gap Test

Test #	Cards	Charge Density (g/cc)	Detonation
2	0	1.62	Y
1	150	1.61	Y
5	155.1	1.65	Y
6	158.2	1.66	Y
8	159.4	1.61	Y
10	159.6	1.63	Y
11	159.7	1.63	N
9	159.8	1.64	N
7	160.2	1.66	N
4	162.6	1.61	N
3	175.4	1.62	N

(U) UNCLASSIFIED

7.9 (U) ENERGETIC PERFORMANCE: DETONATION VELOCITY AND PRESSURE

(U) The detonation velocity and detonation pressure of PAX-52 was calculated and found to be nearly identical to the previous samples of PAX-52 produced. The acceptable parameters were defined to be >7.5 km/sec and > 24.0 GPa, respectively. Since PAX-52 exceeds these parameters, it is deemed **acceptable**.

7.10 (U) PRODUCT RESPONSE TO IGNITION

(U) A Variable Close Confinement Test (VCCT) was performed on the demonstration sample of PAX-52. This test was considered a **success** as only non-violent burning reactions were observed. No detonations occurred.

(U) A small-scale burn test was performed on PAX-52, where several trials were performed on a 10 gram sample and on a 100 gram sample. The 10 gram sample burned for 40 to 60 seconds, with nonviolent burn reactions. The larger sample burned for longer at approximately 105 seconds, but maintaining nonviolent burn reactions. PAX-52 is deemed **acceptable** with no detonation, passing the response to ignition test.

7.11 (U) CHEMICAL STABLILITY

PAX-52 was characterized for chemical stability under steady and variable heating conditions as for Thermal Stability. Modified Vacuum Thermal Stability (MVTS) testing was performed by using a TA instruments TAM III micro-calorimeter. Undried test samples of approximately 0.2g were placed into stainless steel vessels and held at 100°C for 48 hours. Pressure and heat flow graphs were continuous and constant, showing no sharp peaks, indicating sample stability. **Success**. Data can be seen in Table **16**.

Table 16 (U) MVTS Data

(U) PAX-52 MVTS (U) UNCLASSIFIED

Test #	Mass (g)	Volume Gas Evolved (STP) (cm^3)	Gas Evolved per Mass (STP) (cm^3/g)
Test 1	0.202	0.58	2.87
Test 2	0.203	0.39	1.92
Test 3	0.206	0.53	2.57
Average	0.204	0.50	2.46
STDEV	0.002	0.1	0.5

(U) UNCLASSIFIED

Thermal stability was also examined by conducting testing in accordance with (IAW) AOP-7 (US/202.01.013). A test sample of ~20g of PAX-52 was placed in a tared beaker, covered with a watch glass, and placed into a programmable, digital oven at 75°C for 48 hours. This system was monitored by thermocouple and a data acquisition system for the length of the test. Upon completion, the sample and beaker were cooled in a desiccator and weighed to determine mass loss. The sample did not explode, burn, or decompose, meeting passing standards. Mass loss was 0.22%.

7.12 (U) HOMOGENEITY

(U) The calculation of the mixing index from the HPLC data collected samples that were twin screw extruded was used to determine homogeneity of PAX-52. The mixing index is defined on the basis of 1 minus the standard deviation of the distribution of the concentration of the solid particles over the standard deviation of the completely segregated particles. This value would approach 1 for a completely random distribution of the concentration of solid particles and would approach 0 for completely segregated particles. Ten samples from a pre-production batch were analyzed to derive a mixing index of 0.995 for PAX-52, indicating that the mixture of particles is randomly distributed throughout the mix. **Success**

(U) UNCLASSIFIED

Mixing Index Calculations

$$\bar{c} = \frac{1}{N}\sum_{i=0}^{N} c_i \qquad \text{Eq (1)}$$

$$s^2 = \frac{1}{(N-1)}\sum_{i=1}^{N}(c_i - \bar{c})^2 \qquad \text{Eq (2)}$$

$$s_0^2 = \bar{c}(1-\bar{c}) \qquad \text{Eq (3)}$$

$$MI = 1 - \frac{s}{s_0} \qquad \text{Eq (4)}$$

(U) UNCLASSIFIED

Figure 43 (U) Mixing Index Calculations. *The mixing index calculation procedure begins with the determination of N measurements of concentration, c_i of the solid ingredient/binder system. Here 10 HPLC based measurements of the HMX wt% concentration values were available. The mean, c and the variance, s^2 of the concentration distribution of the HMX are calculated from equations (1) and (2). The maximum variance for a completely segregated system is defined by assuming that samples are collected from either HMX or from silicone polymer without any diffusion through the boundary. For the completely segregated system the maximum variance can be determined from Equation (3). We have generally elected to work with a mixing index that is defined on the basis of 1 minus the standard deviation of the distribution of the concentration of the solid particles over the standard deviation of the completely segregated state of the solid particles, i.e., Equation (4). This mixing index would be zero for completely segregated particles and its value would approach one for a completely random distribution of the concentration of the solid particles.* (10)

7.13 (U) ENVIRONMENTAL IMPACT

(U) PAX-52 was studied by the DRDC to determine the deposition rate of RDX and HMX. By using HMX instead of RDX in the formulation would reduce the associated risk by at least three orders of magnitude, as the impact of a contaminant is equivalent to its effect, exposure and fate (4). The assessment is deemed a **success** with nearly a three orders of magnitude (2.91) reduction in environmental risk as determined by the Canadian test results. There was an expectation that the HMX used in the demonstration had 0% RDX according to the certificate of acceptance received from the supplier; however, it was learned that contents measured by weight are rounded when inspected. This might explain why RDX was detected at all during the deposition studies. See **Appendix D23.**

(U) The testing for deposition rates was performed this winter (2018) at the Valcartier testing range. It was a significant undertaking and provided important results, because, in the end, the search for a new RDX-free C4 was triggered by environmental concerns.

(U) One of the advantages of those winter tests was that it was possible to get a qualitative assessment of the blocks at cold temperatures. The EOD personnel really appreciated the cold temperature malleability of the product.

(U) In order to assess the detonation efficiency of the candidate plastic explosives, a detailed methodology was applied to precisely measure the mass of HMX left-over, post-blast. This protocol was developed in collaboration between the United State Cold Regions Research and Engineering Laboratory and DRDC to quantifies post-blast explosive residues with high sensitivity and precision and it was further adapted to the Canadian needs. The methodology entails that the detonations are conducted over a bed of snow, and the detonation plumes are defined by walking around the perceived terminus of the residues and the corresponding plume is sampled using a systematic method, as illustrated in **Figure 44**. This allowed the measurement of the detonation's efficiency (DETEF) of the plastic explosive, or to the HMX mass deposition.

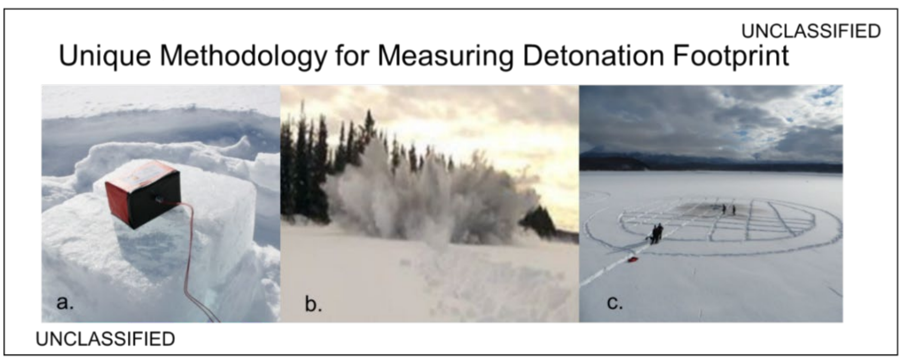


Figure 44 (U) Deposition Rate Test Set-up. *This unique method can capture combustion residues from an explosive charge detonated upon a pristine snow pack. A known mass of material is primed for initiation on blocks of ice centered in a field of freshly fallen snow (a.) The detonation creates a plume that spreads across the field (b) then multiple samples are collected within a patterned area (c) to be analyzed in a lab.*

(U) Three replicates of each detonation were performed and plumes were sampled twice to ensure data reproducibility and validity.

(U) Three blocks of each formulation were remotely detonated as standalone, as shown in **Figure 45** for PAX-52. The candidates, as well as C4 as a comparison, were also tested as donor charges against a 40 mm RDX-based grenade. This allowed the measurement of their relative efficiency as donor charge for Blow in Place (BIP) operations in comparison with C4. The setup used for the BIP was a half block detonated on the side of the grenade. Initiation was done remotely using an electric detonator. All the blocks had the same weight as C4 although the dimension varied. The residual mass of explosives deposited upon the detonation of the four plastic explosives are presented in **Table 17**. As described earlier, C4 leaves around 65 mg of RDX at detonation point, which is considered non-sustainable. PAX-52 shows a deposition of 47 mg of HMX and 0.08 mg of RDX. This is considered near to acceptable, as HMX is much less soluble and toxic than RDX and as the RDX deposition rate is near three (2.91) orders of magnitude less when compared to the RDX deposition rate of C4.

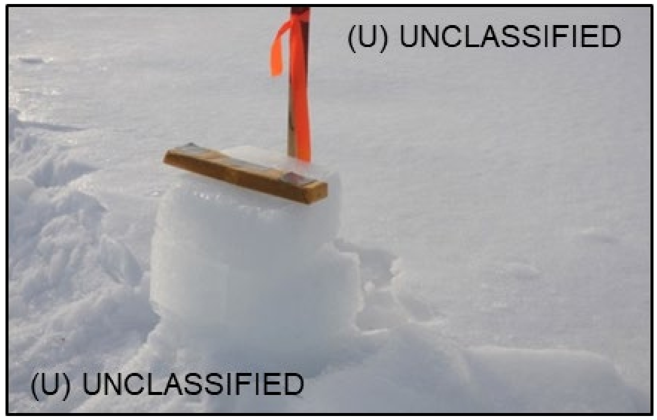


Figure 45 (U) PAX-52 block prior to detonation.

Table 17 (U) Mass of Residual RDX and HMX *Deposits of residual explosive nitramines when detonated as standalone charges.*

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Mass of Residual RDX and HMX		
Plastic Explosive	**Mass of RDX Deposited (mg)**	**Mass of HMX Deposited (mg)**
C-4	65	1
PAX-52	0.08	47

UNCLASSIFIED

(U) **Table 18** presents the RDX and HMX residual mass post-detonation, after using the plastic explosives as donor charges against a 40 mm RDX-based grenade. The use of C4 led to the deposition of 3 mg of RDX at the detonation point. The RDX remaining post-blast is in the same order of magnitude for all options under study, with between 0.1 mg and 0.2 mg of RDX deposited, for a ten-fold increase in detonation efficiency when compared to C4. The HMX deposited in C4 comes from its presence as an impurity in commercially available RDX, and does not represent a significant contribution. PAX-52 led to the deposition of around 45 mg of HMX, which would be acceptable. This shows that all the options would behave as acceptable donor charges from a deposition rate point of view in this specific case.

Table 18 (U) Mass of Residual RDX and HMX *Deposits of residual explosive nitramines when detonated as donor charges and RDX based 40 mm grenade.*

UNCLASSIFIED

Mass of Residual RDX and HMX		
Explosive Donor Charge	Mass of RDX Deposited (mg)	Mass of HMX Deposited (mg)
C4	3	0.005
PAX-52	0.1	41

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(U) The testing for deposition rates was performed this winter (2018) and demonstrated a good behaviour for PAX-52 during the detonation of blocks and while used as a donor charge for 40mm grenades. Acceptable deposition rates were measured. So far, PAX-52 appears to Canada as a very good option for an RDX-free replacement of Composition C4.

7.14 (U) HMX WORKPLACE EXPOSURE (30)

(U) Occupational Safety and Health Administration (OSHA) arbitrary target concentration for HMX is 0.2 mg/m^3.(32) The HMX concentration results on 21 Mar 17 (technology demonstration) were well below the target concentration. (In addition, employees were in the control room most of the sampling period). For the purpose of this demonstration only HMX levels within the TSE operation bay were measured. For an 8-hr TLV-TWA there were only 0.005 mg/m^3 of HMX detected during the technology demonstration which meets the performance objective.

Table 19 (U) HMX Air Sampling Results. *The purpose of the test was to determine the HMX concentration during the operation of the TSE during the HMX extrusion process. An air sampling line was attached at the TSE die (extrusion area.) The air sampling results were well below the OSHA arbitrary target concentrations and it should be noted that the employees were not present during the process.*

UNCLASSIFIED Twin Screw Extruder, HMX Air Sampling Results

Bldg 1403, Twin Screw Extruder, HMX (Octogen) Air Sampling Results				
Twin Screw Extruder (TSE)	Sample Time (min)	TWA Results for Sampling Period (mg/m3)	8-hr TLV-TWA (mg/m3)	Exceeds OSHA Arbitrary Target Concentration? (0.2 mg/m3)
	211	0.011	0.005	No

UNCLASSIFIED

(U) Threshold Limit Value-Time-Weighted Average (TLV-TWA) Definition: The TLV-TWA concentration for a conventional 8-hr workday and a 40-hr workweek, to which it is believed that nearly all workers may be repeatedly exposed, day after day, for a working lifetime without adverse effect.

(U) Air sampling for HMX was conducted using MSA Escort Elf medium flow sampling pumps. Sampling media, sample volume, and flow rates were determined by the Army Public Health Center laboratory IAW SOP Number: DLS 810, effective date APR 2011. The air sampling pumps were pre- and post-calibrated using The Gilibrator Primary Flow Calibrator Control Unit, B/C OH4322, Serial Number 002096-B, and the Gillian Flow Cell Assembly, Model D-800266, Serial Number 20658-S, (both TMDE calibrated 14 Jul 16). The samples were analyzed by the Army Public Health Center.

7.15 (U) PACKAGING

(U) Accurate Energetic Systems, LLC (AES) holds a contract with the Government for Rapid Fabrication Methods for Existing and Novel Explosive Formulations. The scope of the effort covers producing prototype hardware to support developmental testing and evaluation of novel explosives or energetic devices. When the Project was in need of pressed PAX-52 blocks to test as an alternate to the existing M112 Demolition Blocks, this initiative was selected as a good avenue to produce the PAX-52 M112 prototype demolition blocks.

(U) Standard M112 blocks are typically extruded, however, pressing M112 blocks is listed as an alternative production process in the military specification (2). There are several advantages to pressing an M112 block over the current extrusion technique. The first is smaller quantities can be manufactured with much less start-up time and waste. The second is the block is not formed under vacuum, therefore the resulting block is softer and more malleable with better ability to custom form in the field. Consequently, there are disadvantages, as well. Rate of production is much slower than extrusion and rejection rate for cavities and miss-formed corners is higher, however a cutting/slitting process is not required. The material used to line the die cavity during pressing also must be removed from the block before further processing.

(U) AES had two presses available for the pressing of M112 demolition blocks, a 60 ton 2-post press and a 300 ton 4-post press **Figure 46 a-b**. The press used for M112 block production was determined by work center loading and required throughput as either could be used interchangeably. To fully understand the pressing of PAX-52 material, both presses were a part of the experiment. The tooling used for M112 blocks was a 3-cavity, modular style mold. Each cavity was the length and width of a standard M112 block. An ejection plate was placed into the bottom of the cavity and in combination with the punch height and overall die height; the resulting cavity height was the thickness of an M112 block, therefore the die acted as a positive stop and controlled the height. Standard AES practice was to line the die cavities with waxed deli paper (**Figure 46 c-d**). This keeps the materials from extruding around the ejection plates and dies and allowed for removal of the block after pressing. The paper was either left intact or removed, depending on customer requirements.

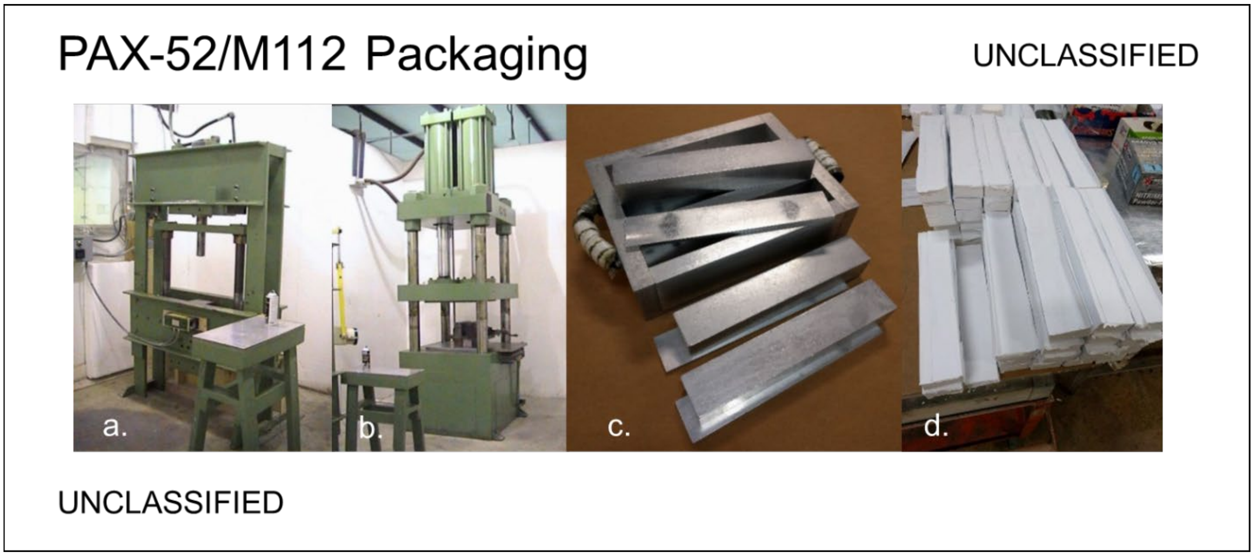

Figure 46 (U) PAX-52/M112 Packaging. *Two press platforms* ***(a.-b.)*** *were used to develop the correct process for pressing bulk PAX-52 into specialized tooling* ***(c.)*** *Formed blocks had to be unwrapped by hand before the block could be fitted into a specified container* ***(d.)*** *Once the process was debugged the production team were able to package 125 blocks per shift.*

7.16 (U) USER EVALUATION: COMBAT ENGINEERS.

(U) User evaluations were conducted at Fort Leonard Wood, MO, to demonstrate the capability of PAX-52 explosive as compared to C4 to provide developers with a qualitative assessment of the new moldable explosive. Soldiers from the 5th Engineers Battalion participated in the hands-on evaluations by conducting several tasks in loading and firing several demolition charges and warhead devices. In addition, the evaluation gathered feedback from soldiers on how PAX-52 compares to C4 with respect to hand moldability, tackiness, strength, other physical properties, and any other impressions gained from this demonstration. Soldiers prepared several live fire targets to qualitatively assess the blast performance of PAX-52 based on their familiarity with hand-moldable explosives. Appendix E: **11.4**

(U) During the planning meetings with range operators and 5th Engineers it was noted that a Safety Release would be required before the soldiers handle the PAX-52 material. The U.S. Army Evaluation Center (AEC) recommended that ARDEC convene a System Safety Working Group (SSWG) to review all safety and toxicology data and draw clear conclusions of support of the Soldier use of PAX-52 for within the tasks being required in the FLW evaluation.

(U) ARDEC System Safety brought together representatives from the Maneuver Support Center of Excellence (MSCoE), Army Public Health Center, ARDEC Energetics Development Branch, and ARDEC Demolition & Breaching Branch. Discussions and Q&A were fruitful in presenting the SSWG with conditional assurance of the safety of PAX-52, which ultimately lead to full safety release of PAX-52 for the evaluation.

(U) Soldiers were tasked with preparing the various demolition charges with C4 and PAX-52. An individual soldier was assigned a particular demolition charge and loaded one charge each with C4 and then PAX-52. The charges were prepared in accordance with their respective technical manual instructions. A survey was given to gage the soldier's impression of both explosives (**Table 20**.) Range Cadre personnel were on site to oversee and ensure compliance with range safety procedures.

(U) The evaluation exercise was burdened by unplanned delays and events that caused the agenda to be truncated. Completion of all the PAX-52 shots would have been something to see but the evaluation was never meant to be a quantitative, performance measure of the material. The soldiers had ample time and provided qualitative feedback as they packed the several charges. Since the new material has never been handled by soldiers in live demonstrations, their feedback was valuable information in the further development of the system. Generally, the soldiers found PAX-52 was easier to mold by hand, less affected by cold temperatures, and stickier in their hands as compared to C4.

Table 20 (U) Qualitative Survey of C4 vs PAX-52 *The survey was very informal since the soldiers were actually in training so there was not a time slot allowed for introducing and discussing the survey. Also, the soldiers each participated in the training in different ways, some were observing while others were demonstrating. The questions were very general and the ranking of each response was straightforward.*

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(U) Qualitative Survey C4 vs PAX-52

GENERAL	C4					PAX-52					Comments
DID THE DEMO BLOCK STAY TOGETHER WHEN UN-WRAPPED ?	YES	5	1	0	NO	YES	5	1	0	NO	
COULD THE DEMO BLOCK BE MOLDED AND SHAPED AS NEEDED?	YES	1	4	0	NO	YES	6	0	0	NO	
WAS THE MATERIAL STICKY IN YOUR HANDS?	YES	0	1	5	NO	YES	4	0	2	NO	
WAS THE MATERIAL SLIPPERY TO HANDLE?	YES	0	0	6	NO	YES	0	0	6	NO	
DID THE MATERIAL STICK TO SURFACES?	YES	0	2	4	NO	YES	4	1	1	NO	
DID COLD WEATHER AFFECT THE MATERIAL?	YES	3	2	1	NO	YES	0	0	6	NO	
DID THE MATERIAL SMELL OK?	YES	2	2	0	NO	YES	4	1	0	NO	
MEDIUM EFP	**C4**					**PAX-52**					**Comments**
WAS IT EASY/HARD TO SLICE THE DEMO BLOCK ?	EASY	1	3	1	HARD	EASY	5	0	0	HARD	
WAS IT EASY/HARD TO PACK THE MATERIAL INTO A CREVICE ?	EASY	0	1	4	HARD	EASY	5	0	0	HARD	
WAS IT EASY/HARD TO ELIMINATE AIR POCKETS WHILE PACKING ?	EASY	0	3	2	HARD	EASY	5	0	0	HARD	
WAS IT EASY/HARD TO PACK A HOUSING IN THE COLD ?	EASY	0	1	4	HARD	EASY	5	0	0	HARD	
WAS IT EASY/HARD TO CUT OFF EXCESS MATERIAL ?	EASY	0	2	3	HARD	EASY	5	0	0	HARD	
HOW GOOD/BAD DID EACH LAYER OF MATERIAL STICK TOGETHER ?	GOOD	0	3	1	BAD	GOOD	5	0	0	BAD	
HOW EASY/HARD WAS IT TO SCREW THE PLUNGER INTO BACK PLATE?	EASY	3	2	0	HARD	EASY	4	1	0	HARD	
CONICAL SHAPED CHARGE	**C4**					**PAX-52**					**Comments**
WAS IT EASY/HARD TO SLICE THE DEMO BLOCK ?	EASY	1	1	2	HARD	EASY	4	0	0	HARD	
WAS IT EASY/HARD TO PACK THE MATERIAL INTO A CREVICE ?	EASY	1	2	1	HARD	EASY	3	0	1	HARD	
WAS IT EASY/HARD TO ELIMINATE AIR POCKETS WHILE PACKING ?	EASY	1	3	0	HARD	EASY	3	0	1	HARD	
WAS IT EASY/HARD TO PACK A HOUSING IN THE COLD ?	EASY	1	1	2	HARD	EASY	4	0	0	HARD	
WAS IT EASY/HARD TO CUT OFF EXCESS MATERIAL ?	EASY	1	1	2	HARD	EASY	4	0	0	HARD	
HOW GOOD/BAD DID EACH LAYER OF MATERIAL STICK TOGETHER ?	GOOD	1	2	1	BAD	GOOD	4	0	0	BAD	
HOW EASY/HARD WAS IT TO FORM A DOME ?	EASY	1	3	0	HARD	EASY	3	1	0	HARD	

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7.17 (U) USER EVALUATION: EXPLOSIVE ORDNANCE DISPOSAL (EOD)

(U) Purpose: To have EOD technicians use PAX-52 in several test configurations and get their qualitative feedback. This particular objective was met with resounding success; however, due to the sensitivity of the data and the security of the forces, not much can be reported in this document.

8 (U) COST ASSESSMENT

(U) C4 is currently produced in a multi-step water slurry process that requires an organic solvent-based lacquer binder that is applied to RDX nitramine explosive. The use of twin screw extrusion demonstrated the waterless and solvent-less production of PAX-52 and the forming of M112 blocks within a single process. This project demonstrated that a silicone polymer, used as a solvent-free binder without a water slurry, efficiently coated HMX crystals in an emulsion with the consistency of C4. Each method has production costs that were assessed to the extent that was practical for this research.

(U) This assessment first established cost baselines for the existing technology (water slurry batch) and the demonstrated technology (continuous TSE) using reasonable estimates gathered from reliable data sets. Historical DoD procurement budgets for the existing technology were analyzed using time-value equations to set a baseline cost of \$35 per M112 demolition block, including process waste (section **8.1**.) Then an engineering model that used both empirical and theoretical standards was developed to estimate the baseline cost of the demonstrated process technology to be \$67/per M112 block. It was derived on a unit basis using retail prices for raw materials and energy with no consideration for economies of scale (section **8.2**.)

(U) Secondly, a cost assessment was done using standard accounting principles and outputs from the engineering model to estimate variable and fixed costs related specifically to a fixed annual production 80000 pounds of PAX-52 (64000 M112 blocks.) It calculated the cost of the M112 at \$62/per block; however, this assessment allows for the economies of scale. At a production rate equal to an 8-year average procurement of M112 blocks, the cost per block is extrapolated to \$40/block (section **8.3**.)

(U) The Office of the Assistant Secretary of Defense (Acquisition and Sustainment) (OASD(A&S)) partnered with the Army Armament Research, Development and Engineering Center (ARDEC) Explosives Development Branch (EDB) at Picatinny Arsenal on a pilot project to test DoD's Sustainability Analysis Methodology. This Sustainability Analysis evaluated PAX-52, a M112 demolition-block manufacturing process, as an alternative to the baseline C-4 process. That baseline process consists of C-4 production and M112 production processes, while the alternative system is a single-step process. (5) It was well-understood that replacing RDX with HMX would create a large cost differential between C4/M112 and PAX-52 production owing primarily to the cost the energetics. Plainly, HMX costs nearly three times RDX, which makes PAX-52 a pricey alternative to C4. The project looked past the obvious pound-for-pound HMX-to-RDX price differential to observe some over-shadowed cost burdens associated with the current technology that would be eliminated by the alternative technology.

8.1 (U) COST BASIS FOR C4/M112 PRODUCT – HISTORICAL BUDGETS

(U) The project Demonstration Plan provided details of the multi-step process for C4 production at BAE Holston (BAE), AAP, and details of the M112 production/packaging process at American Ordnance (AO) Iowa, AAP. Technical information was readily available to create process diagrams and descriptions of each process step (**Figure 47**); however, it was difficult to extract any relevant cost data from representatives of BAE, AO, or Government representatives due to the proprietary nature of the information.

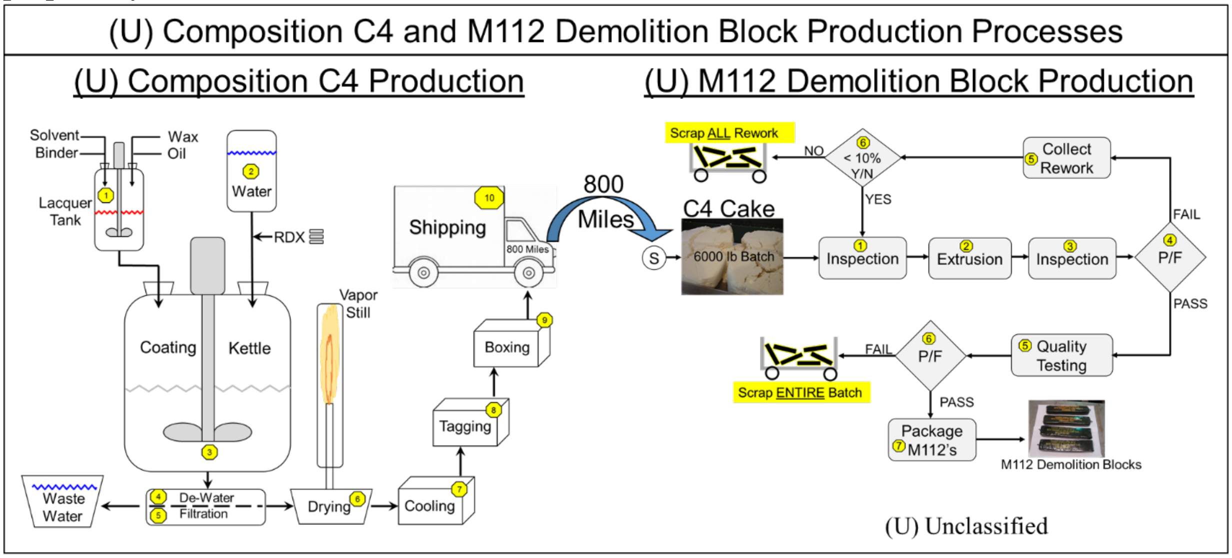

Figure 47 (U) Process Map *Composition C4 is produced in a multi-step water slurry process that requires an organic, solvent-based lacquer to mix a cohesive binder and plasticizer with energetic RDX nitramine crystals. This multi-step process, developed in 1958, has been largely successful owing to the availability of cheap labor, cheap energy, and lower environmental protection standards; however, six decades later it is still time, energy, and labor intensive and generates 1.5 million gallons of aqueous waste per year. The output of the C4 production process is a loose powder that is boxed and transported to an entirely different packaging facility where it is further processed into M112 demolition blocks via additional processing steps.*

(U) Without access to proprietary business accounting data related specifically to C4 and M112 production costs, it was difficult to accurately assess a cost basis for a realistic comparison to the demonstration technology. Assumptions were made based on a few known data points collected from industry standards, published reports and budgets, but ultimately the manufacturer's cost of production is most reliably represented by the price the DoD pays for these two products. The Army's procurement record is part of the President's Budget published each year, from which an average cost basis for C4/M112 was derived.

(U) Analysis of the past eight years of the Army's procurement of bulk C4 and M112 demolition blocks, as documented in the Army's budget line item justification (33) for the President's Budgets, shows that 5.6% more C4 is procured than is necessary to manufacture a given number of M112 demolition blocks (**Figure 48**). This margin provides for bulk C4 product usages and some waste associated with the M112 production. The Army's M112 product manager uses a 4.8% procurement factor to account for M112 production waste (34).

(U) The average procurement cost for bulk C4 from the presidents budgets FY2010-FY2017 accounted for in today's dollars is $19.30/lb. This includes the cost of waste product. To estimate the waste or scrap material associated with the bulk C4 manufacture process, a recent final article test (FAT) report for one C4 batch details the input weight of all materials used to produce a single batch and output measure of packaged product weight.

(U) Input weight of a single batch of C4: 6,644 lb.
(U) Output after final packaging: 6,105 pounds of C4 (35).

(U) Therefore, based on the FAT report, it can be estimated that the waste or scrap from C4 manufacturing process is about 8.8%. The waste comes from product residues that stick to kettles, nutzches, transfer containers, etc. The effective cost to the Army for bulk C4 is the price/yield = \$19.30/91% = **\$21.20/lb.**

(U) The average cost for M112 manufacture over the same eight years represented in today's dollars is **\$8.20/block**, which converts to \$6.56/lb, or effective cost of \$6.56/lb/95.2% = **\$6.89/lb**.

(U) Both figured, the average unit cost to procure M112 demolition blocks in today's dollars is \$28 /lb **(\$35** per M112 block.)

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Eight-year Average of Bulk C4 and M112 Unit Costs in Todays Dollars

Financial Year	M112			Bulk C4		
	Total Units	Unit Cost (\$/Unit)	Present Value (\$/Unit)	Total Pounds	Unit Cost (\$/lb)	Present Value (\$/Unit)
2010	487815	6.69	6.78	643915	13.68	13.87
2011	112159	10.35	10.47	148050	22.34	22.61
2012	1027889	4.19	4.23	1356803	15.64	15.80
2013	280058	9.59	9.67	369776	23.92	24.11
2014	320277	10.11	10.17	422716	21.93	22.06
2015	162947	6.46	6.49	216090	14.49	14.55
2016	116160	10.46	10.48	153318	23.05	23.10
2017	180101	7.3	7.30	237733	18.31	18.31
Average	**335926**	**8.14**	**8.20**	**443550**	**19.17**	**19.30**

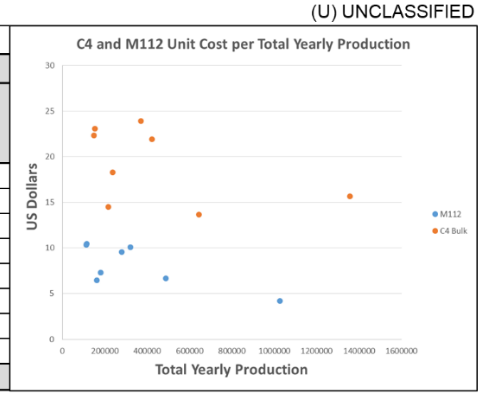

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Figure 48 (U) Cost Basis for C4/M112. *Orders for M112 demolition blocks varies according to a few factors, including: mission requirements in wartime, inventory levels and other military uses. Over the several years buying larger volumes of product did not always yield a lower unit price. Price differences can be attributed to many factors, e.g., volume, special requirements and timing of contracts. There are some variable cost factors used by the manufactures that are not known and therefore difficult to assess in a cost-benefit analysis. An average federal funds rate of 0.2% was used to calculate the present value of historic costs.*[5]

(U) Another data point that could be used to establish a DoD-wide cost basis for C4/M112 is to use a budgetary feedback mechanism called the Program Objective Memorandum, which details what each military branch plans to spend across several financial years. The line item for procurement of the M112 in the Army for 2018 shows **\$45.68** per block (**Figure 49**), but it does not detail any underlying formulation of the number. The POMs value here is to strike a boundary within which a cost comparison with PAX-52 and the demonstration technology can be constructed. **For this assessment the budget derived number of \$35 per block will be used as the baseline for comparison.**

[5] Data for the eight years was found on the Federal Reserve website and analyzed by the PI. website:https://www.federalreserve.gov/

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DEMO CHG BLK M112 1-1/4 LB COMP C-4 W/TAGGANT (013893854)								
POM Unit Price Budget Values (POM17)								
Service	2014	2015	2016	2017	2018	2019	2020	2021
ARMY	$0.00	$33.05	$43.80	$0.00	$45.68	$46.93	$50.87	$51.75
NSOF	$0.00	$27.07	$0.00	$0.00	$41.11	$0.00	$0.00	$0.00
USAF	$0.00	$26.57	$53.46	$44.96	$41.11	$42.23	$45.79	$46.58
USNS	$0.00	$0.00	$0.00	$44.96	$0.00	$42.23	$0.00	$0.00

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Figure 49 (U) Cost Basis of C4/M112 from POM Budget. *Values shown in this POM are derived by back-room budget planners and do not represent a strategic procurement policy, per se. The budgeted items could include engineering support, foreign military sales, training, inventory control, production waste, as well as, mission readiness.*

8.2 (U) COST BASIS FOR PAX-52 PRODUCT - ENGINEERING MODEL (36)

(U) Extrusion technology provides a cost effective solution to the mixing and compounding of energetic materials. Single and twin screw extrusion (TSE) technologies have been used with great success in the polymer, pharmaceutical and food industries as a way to not only provide increased control over the extrudate quality but also as economical alternatives to batch processes (37).

(U) This cost assessment is based on optimized operating conditions of a TSE located at the Highly Filled Materials Institute at Stevens Institute of Technology. These operating conditions may somewhat differ from those found in the final production environment, but it is expected that this model system provides a reasonable estimate of the operating costs in the production environment. This model does not consider the cost of packaging the extruded demolition blocks into the M112 configuration, which would require an addition process step.

(U) Mathematical models of the TSE process provide valuable insight into choosing optimal operating conditions and screw configuration. Detailed finite element calculations were performed for this purpose. These types of calculations include a comprehensive analysis of the relevant flow fields and boundary conditions (38). In addition to this, modeling is needed for predictive cost estimation of the entire process. In particular, the power demand of the overall process for a given residence time is required. When considering the final production process, simpler engineering models can be used given semi-empirical correlations used widely in extrusion engineering. The final production process is expected to be in a well-developed steady state which is an inherent assumption of these engineering models. Summaries of the applicable engineering models used in this cost assessment can be found in literature (39) (40).

(U) There are three categories of costs included in this estimate: raw material, operating labor, and utility costs. Capital and fixed costs are not included in this estimate.

(U) The raw material costs are derived from current supplier rates. The composition of PAX-52 and per pound costs of each ingredient are provided in **Table 21**.

Table 21 (U) Raw Material Costs of PAX-52 Ingredients. *Unit costs were gathered from material suppliers for low quantity orders. This model does not account for economy of scale in a full scale production environment. Mass fractions base on a nominal formulation for PAX-52 are hidden from view for information security.*

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Raw Material Costs of PAX-52 Ingredients		
Component Name	**Mass Fraction**	**Cost ($/lb)**
HMX	0.85	37.39
PDMS	0.1475	8.01
DMDNB	0.0025	89.01
PAX-52 Total		**33.19**

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(U) In this model a nominal labor rate of $100/hr is used for this estimate which includes overhead, fringe and G&A.

(U) Electrical power is the only utility required for the TSE process. The major components of the process drawing electrical power include the motor/gear-box driving the screws and five 3-resistor electrical cartridge heaters. Other electronic systems including controls and human machine interfaces are negligible in comparison and not included. The motor efficiency is calculated from industry standard NEMA tables (41).

(U) The retail price of electricity, set at $0.0674/kWh, is taken from the U.S. Energy Information Administration's Rolling 12-month average for the industrial sector ending in September 2016 (42).

(U) Under optimal operating conditions specified by the Stevens Institute of Technology study, engineering models provide several outputs. Primarily they predict a continuous extruder motor operating output of 9.36 kW or about 50% of its capacity. An under-loaded motor would operate under low power factor (30% is estimated here). For the final production process, an appropriately sized motor would run closer to capacity and with a much higher power factor (>90%) providing tangible cost savings in the final per pound cost of PAX-52. The five three-resistor cartridge heaters are assumed to run at capacity providing an upper bound on the electrical requirements of the process. The actual heating requirements will fluctuate with environmental factors at the production installation.

(U) The three contributions to the cost basis of PAX-52 are included in **Table 22** for a total of $53.34 per pound **($66.68 per block.)** Since the alternative technology eliminates many of the manufacturing steps required to post process bulk material into M112 blocks, here no additional cost burden is added for packaging extruded blocks.

Table 22 (U) Operating Cost Breakdown for PAX-52 TSE Process *For this engineering model electricity was included at a higher requirement than would be necessary for production of PAX-52 since there is no need to employ five three-resistor cartridge heaters to heat the product. Since this energy cost feeds into another corroborating cost assessment (next heading) it will be retained for clarity but noted as an over-estimation of the base cost of PAX-52.*

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(U) Operating Costs for PAX-52 TSE Process	
Category	**Cost ($/lb)**
Raw Materials	33.19
Labor	15.12
Utility (Electrical)	5.04 (over-estimated)
Price of PAX-52	**53.34**

(U) UNCLASSIFIED

8.3 (U) COST ASSESSMENT

(U) In this cost assessment (43) gross estimates of the fixed and operating costs associated with the continuous processing of PAX 52 are provided. The major assumptions include a production rate of 80,000 lb/year using the 40 mm Universal Extrusion System and raw material costs of about $40/lb for HMX, $10/lb for PDMS and $90/lb for DMDNB and nine year amortization of all items associated with fixed costs. On the basis of these assumptions the cost of the PAX 52 formulation is estimated to be $47/lb out of which the major portion is associated with raw material costs. Since the alternative technology eliminates many of the manufacturing steps required to post process bulk material into M112 blocks, no additional cost burden is added for packaging extruded blocks.

(U) The fixed total costs are calculated **Table 23**. The fixed costs include the TSE system, hardware, controls, data acquisition, deluge system, testing equipment for the product, and building and control systems. The total fixed cost is determined to be about 3M$.

Table 23 (U) PAX-52 Production Fixed Costs.*Based on analogous information collected from TSE operations at the Highly Filled Materials Institute at Stevens Institute of Technology, reasonable estimates for standing up a TSE production line are provided in the table. In this case of the alternative technology, the DoD would not be burdened with these fixed cost since there are existing TSE production lines capable of processing PAX-52 into M112 demolition blocks.*

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PAX-52 Production Fixed Costs	
Universal Twin Screw Extrusion System:	
Machine base, hydrostatic drive, housing assembly	
Barrel shell assembly, manifold assembly,	
Fully intermeshing co-rotating twin screw module	
Agitator shafts, agitator components, liners	
Figure 8 to "O" adopter, Feed portsa dn vent port	
Gear Box, 6 heating and cooling units,	
Feeder platform for up to four feeders	
Sub-Total	**$839,960**
Rectangular Slit Die	**$35,000**
Power, Controls and Data Acquisition System	**$175,835**
Delivery and Installation of the Universal Extrusion System	**$221,375**
Feeding Equipment:	
Feeder #1: Loss in weight feeder for HMX	$43,920
Feeder #2: Loss in weight feeder for HMX	$43,920
Feeder #3: Loss in weight for additives	$45,506
Feeder #4: Loss in weight for purge compound	$44,208
Feeder #5: Loss in weight feeder for silicone oil binder	$39,719
Enclosure and feeder controls	$24,194
Sub-Total	**$241,467**
Overhead Water Deluge System	**$250,000**
Squeeze Flow Rheometer	**$65,000**
Xray Diffraction Unit	**$200,000**
Ancillary Subsystems	**$275,558**
Production Facility Construction	**$750,000**
Total Fixed Costs	**$3,054,195**

(U) UNCLASSIFIED

(U) The fixed total costs are calculated in **Table 24**. The major costs are associated with the raw material costs. Some of the line item estimates were imported from an engineering model (36) in the previous section **8.2**. Labor and utilities and safety supplies are included. The total variable costs are estimated to be about 3.5M$ per year.

Table 24 (U) PAX-52 Annual Variable Costs *As discussed previously, the cost for utilities is overestimated in the engineering model* ***Section 8.2*** *but included here to avoid confusion. Water was included in this assessment but this is not necessary for the alternative technology. The estimates are reasonable to access the anticipated cost burden of PAX-52 production.*

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PAX-52 Annual Variable Costs

Line Item	Description	Cost	Quantity	Total
Production of PAX-52	Gross Production		80000	80000
Labor	Production Engineers	$90,000	2	$180,000
	Fringe Benefits (30%)	$27,000	2	$54,000
	Technicians	$65,000	2	$130,000
	Fringe Benefits (30%)	$19,500	2	$39,000
	Consultants	$600	5	$3,000
			Sub-Total	**$406,000**
Utilities	Electricity (per pound)	$5.04	80000	$403,200
	Water (per pound)	$0.03	80000	$2,400
			Sub-Total	**$405,600**
Supplies	Inert gases			$1,500
	Safety supplies			$2,000
			Sub-Total	**$3,500**
Raw Materials	HMX		80000	$2,542,520
	PDMS		80000	$94,518
	DMDNB		80000	$17,802
			Sub-Total	**$2,654,840**
Total Annual Variable Costs				**$3,469,940**

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(U) The amortization rates for buildings and equipment are included in the **Table 25** . The number of years for amortization is taken as 39 years for buildings and 5 years for equipment. These rates generate an annual fixed cost of 480K$ per year.

Table 25 (U) Amortized Fixed Costs. *Most of amortized annual costs would not be incurred by the DoD since there are existing TSE capabilities that could easily produce PAX-52. Some modernization and facilitization would be necessary accommodate continuous production of PAX-52 during a long term campaign.*

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Amortization Fixed Costs	
Amortization Rate of Fixed Costs	
Buildings	39 Years
Equipment	5 Years
Fixed Building Costs	$750,000
Building Amortization	**$19,231**
Fixed Equipment Cost	$2,304,195
Equipment Amortization	**$460,839**
Annual Amortized Fixed Costs	**$480,070**

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(U) It is assumed that the PAX-52/M112 TSE production plant will provide a production rate of 80,000 lb per year. On the basis of this production rate the cost of PAX 52 is estimated to be **$49.38/lb ($61.73/block**) **Table 26**. The main component of this cost is related to the cost of raw materials at $33/lb. Thus, the major driver for the cost is the cost of raw materials, specifically arising from the high cost of HMX at about $40/lb. If the cost of HMX could be reduced through production efficiencies or bulk purchase reductions, it would drive the cost of PAX-52 down significantly. Just a 10% per pound reduction in the cost of HMX would drive the cost of PAX-52 down by nearly 7%; a significant cost influence for a single component in a complex cost analysis..

Table 26 (U) Overall Cost Assessment of PAX-52 Production

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Annual Variable and Fixed Cost per Pound	
Annual variable costs:	**$3,469,940**
Annual Amortized Fixed Costs	**$480,070**
Production rate, pounds/year	**80000**
Cost per Pound	**$49.38**

(U) UNCLASSIFIED

8.4 (U) LIFE CYCLE ASSESSMENT (5)

(U) The Defense Acquisition Guidebook (DAG) requires program managers to consider sustainability during systems acquisition and select more sustainable systems that meet performance requirements; have fewer negative impacts on resource availability, human health, and ecosystem quality; and have a lower Total Ownership Cost (TOC). A Sustainability Analysis assesses life cycle costs (internal to DoD) and impacts on resource availability, human health, and environmental quality (external costs) of different alternatives for meeting a specific Department of Defense (DoD) requirement. It is used to reduce TOC and minimize external costs.

(U) The Office of the Assistant Secretary of Defense (Acquisition and Sustainment) (OASD(A&S)) partnered with the Army Armament Research, Development and Engineering Center (ARDEC) Explosives Development Branch (EDB) at Picatinny Arsenal on a pilot project to test DoD's Sustainability Analysis Methodology. This Sustainability Analysis evaluates PAX-52, a M112 demolition-block manufacturing process, as an alternative to the baseline C-4 process.[6] That baseline process consists of C-4 production and M112 production processes, while the alternative system is a single-step process.

(U) **Methods:** Sustainability Analysis integrates Life Cycle Cost (LCC) estimating with Life Cycle Analysis (LCA) to compare two or more systems, components, or processes that meet the same performance requirements. Direct process information from ARDEC-EDB was combined with an integrated hybrid LCA database developed for DoD, which includes environmental flows related to economic activity for industrial sectors across the U.S. economy with integrated hybrid processes for transportation, energy, and other defense activities. Information from the baseline C-4 and M112 processes was estimated using data provided by ARDEC-EDB. Use of the integrated hybrid LCA allows for quantification of both direct impacts to the Army and impacts along the associated DoD supply chain. Additionally, the LCC estimate quantifies internal costs to the Army over the life cycle on a per block basis.

(U) **Life Cycle Costs:** A preliminary estimate of annual operating costs was conducted. Costs are split into direct (labor) and indirect (electricity, transport, waste-treatment) costs. The largest driver of operating costs is labor for both alternatives (>50%; **Figure 50**). For the baseline C4 system, the remaining costs are driven by utilities and waste management. Labor costs for the baseline are based on engineering estimates and are higher than the alternative, given the difference in the number of processing steps. Given the magnitude of labor costs, the overall analysis is sensitive to the engineering estimates used to quantify labor cost, and these should be explored further. The alternative process (PAX-52) reduces operating costs per pound of M112 to 20-30 percent of the baseline. Further exploration of capital costs related to the installation and scale up of this process is warranted due to the potential savings to DoD. A bottom-up estimate of total cost to produce PAX-52 is calculated to be $23.1M annually. This production cost is based on per-pound estimates of raw materials, labor, and electricity[7]. For C4/M112, total costs are based on average procurement costs from 2010-2017 presidential budgets; this procurement cost is estimated to be $83.8M (not included in **Figure 50**).

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[6] Analysis presented here is from a preliminary report (5) submitted for inclusion in this ESTCP final report. A comprehensive analysis will augment this report as an addendum.

[7] Original estimates from Dr. Dilhan Kalyon, Stevens Institute of Technology **Section 8.3**; the per-pound price of HMX was adjusted to reflect full-scale production using the approach of Wright selected by Nagy et al (Nagy, B., Farmer, J.D., Bui, Q.M. & Trancik, J.E. 2013. "Statistical Basis for Predicting Technological Progress." PLOS ONE 8 (2): e52669.).

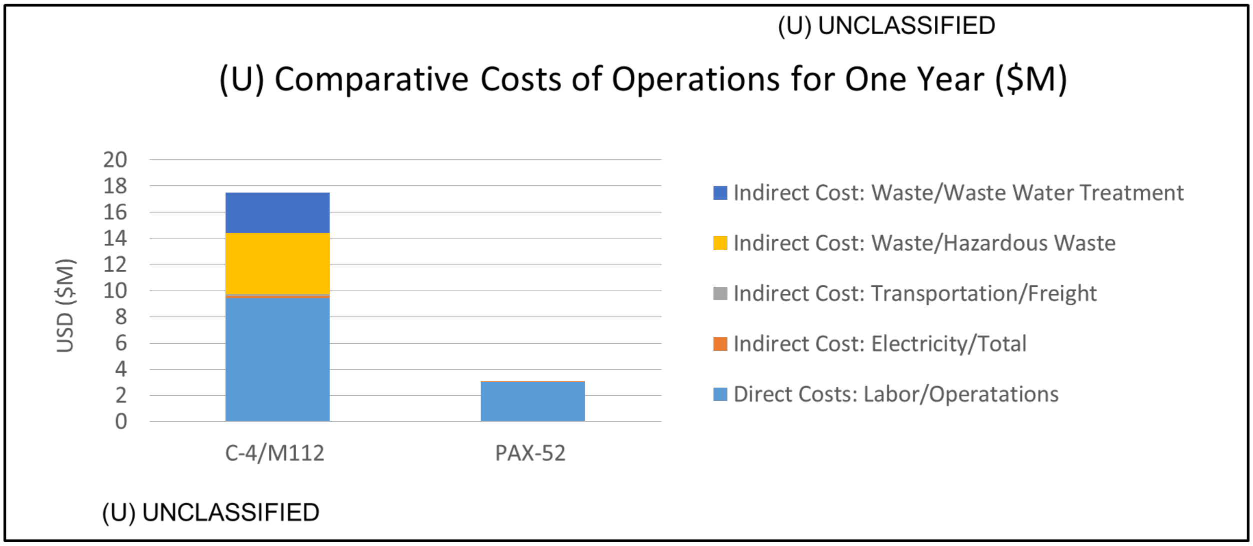


Figure 50 (U) Comparative Costs of Operations for One Year *Estimate of selected annual operating costs for M112 production (both systems produce M112 blocks, but the baseline system is identified as C-4/M112 to reflect that it includes two steps).*

(U) **External Costs:** The first step of the sustainability analysis creates an inventory of material and energy inputs and outputs throughout the production of explosives and manufacture of M112 blocks, including the upstream supply chains. These inputs and outputs are translated to impacts using a suite of embedded models in a Defense Input-Output database (DIO) model. For example, the use of electricity in C4 or PAX-52 facilities requires the production of electricity; the DIO has state-specific models of electricity production that represent state mixes of energy sources (coal, hydro, etc.). The combustion of coal emits, among other substances, particulate matter (PM) to the surrounding population. The DIO impact models translate that PM emission into human- health impacts. These human-health impacts, along with effects on climate, ecosystem quality, and the availability of energy resources, are converted to economic damages. These damages are an indicator of potential future liability, rather than a quantitative estimate of costs borne outside of DoD.

(U) Results of the external cost analysis are shown in **Figure 51**. Across the four areas of environmental concern, the baseline C4 system has higher external costs, indicating higher potential future liability. Among the four areas, human health has the largest absolute cost (ranging from $1.8M for C4 to $0.6M for PAX-52), while ecosystem quality has the smallest (< $0.2M annually). When interpreting the results, note that the differences between C4 and PAX-52 for climate and human health are factors of ~2.5 and 3, respectively, which are considered to be significant. Differences for ecosystem and resources are considered to be negligible. Again, costs shown are indicators of potential harm, rather than quantitative estimates of costs borne outside of DoD.

(U) Impacts on the two largest areas of concern, climate and human health, are driven by the electricity required for wastewater treatment and production-line machinery. While C4 requires wastewater treatment, PAX-52 does not; while C4/M112 has multiple steps and pieces of equipment in the production line, PAX-52 does not. The difference in complexity between the two production systems translates into higher electricity demands for the C4/M112 system, which drives increased external impacts.

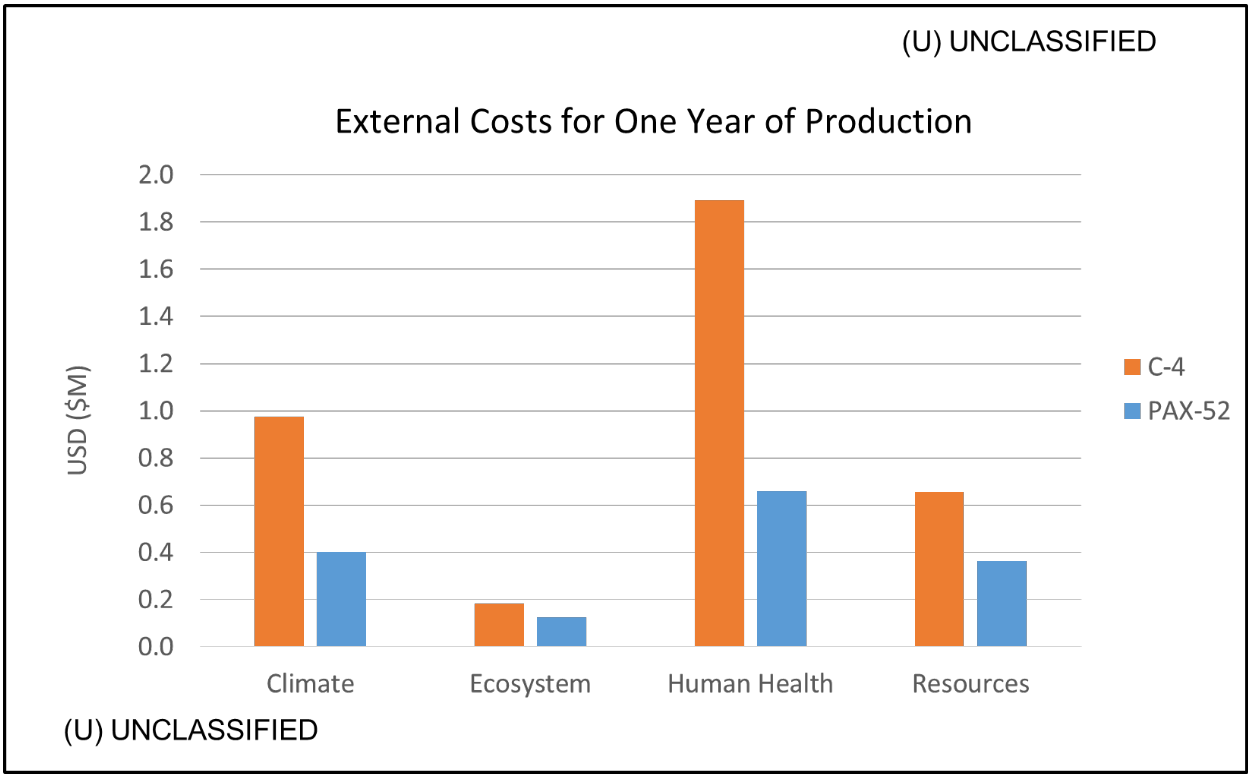


Figure 51 (U) External Costs for One Year of Production. *Estimate of external costs per year of production across four areas of environmental concern.*

(U) Though the single-step production demonstration validated manufacturing cost savings through reductions in labor, energy and process waste, and the several non-tangible cost benefits of PAX-52 related to hand-moldability, mission effectiveness, initiation reliability, manufacturing flexibility and other "….ilities"; the unit cost of nitramine HMX was found to be the overwhelming cost driver in the manufacture of PAX-52. HMX costs nearly three times RDX which makes PAX-52 a pricey alternative for the C4 product managers; however; the project looked past the obvious HMX-to-RDX price differential to investigate several over-shadowed cost impacts associated with the current technology that would be greatly reduced by the alternative.

9 (U) IMPLEMENTATION ISSUES

9.1 (U) STAKEHOLDER/END-USER ISSUES

(U) This project has received endorsements from several stakeholders in the development of an alternate C4 product. Each of the following have a role in bringing the development of PAX-52 to a manufacturing readiness level that is attractive to the C4 product owner, PEO-AMMO. The strategy is to gain endorsements from the current C4 user community including; the combat engineering school, an EOD demolition and demil unit, insensitive munition planners, and future munitions developers. The technology demonstration would highlight the manufacturing feasibility of a one-step M112 production process and present the product for first-hand evaluation.

(U) The Program Executive Officer Ammunition chief scientist endorsed this ESTCP program (44) PEO Product Manager for Close Combat System (PM-CCS) manages the Composition C4 product line. A PM-CCS officer attended a project stakeholders meeting and agreed that the development of PAX-52 had much potential as a C4 alternate product. The PM-CCS derives engineering and production support from ARDEC to maintain the C4 production and C4 product line. This project's PI has a good working relationship with several members of the Comp C4 work group within ARDEC, who are the gate keepers for engineering change proposals to any C4 or M112 products. This team interfaced with PM-CCS to transition PAX-52 into qualification and production and participated in a stakeholder's meeting and witnessed first-hand the superior hand-moldability of the HMX/Silicone formulation, but were particularly interested in the performance of PAX-52 in a standard energy output test, called the plate-cutting test. For them, that single test would make or break the chances for PAX-52 to be considered for transition to the C4 product portfolio within PM-CCS.

(U) This PI had numerous conversations with all the above-mentioned associates of the PEO/PM-CCS in regard to the on-going work with the PAX-52 development. Almost all their feedback for the technical, performance and physical characteristics of the material was positive, but all the conversations wound down to the cost issue. No matter how the cost differential was framed in the discussions, everyone deferred their full acceptance of the product until it matched the price of C4.

(U) Maneuver Support Center of Excellence is the US Army's training center for combat engineers and soldier technologies. ARDEC's liaison to the MSCoE, worked closely with this PI in planning a demonstration and user evaluation of PAX-52 M112 demolition blocks (**see 7.16**). The PM-CCS C4 working group participated in the user evaluation. Feedback from this evaluation was generally positive and thought to be the key to a Technology Transfer Agreement (TTA) with a good chance for PAX-52 to be fielded. Reports of the successful user evaluation at Fort Leonard Wood were recommended to the highest level within Army Materiel Command (45). No further feedback or solicitation for PAX-52 was received from Army command.

(U) Explosive Ordnance Disposal (EOD) division within ARDEC coordinated an evaluation of PAX-52 M112 demolition blocks. EOD is a large consumer of the M112 around the world. EOD has render-safe procedures written for nearly every munition ever produced globally. If the EOD demolitions team would document performance advantages by using PAX-52 in one of their procedures, the project hoped to gain a valuable endorsement of the product to add impetus for PAX-52 transition. The evaluation yielded positive results (46) but no endorsements for PAX-52 were forthcoming from the EOD.

(U) The Program Manager of the Joint Insensitive Munitions Technology Program (JIMTP) endorsed the project and provided project funding support in testing and outside research. The JIMTP funded an effort to evaluate the same silicone binder material for a different type of formulation. The PM views the effort to define the manufacture of an environmentally responsible C4 to be of great benefit to the DoD and EOD (47). He continues to be an advocate of PAX-52, liaised with the Canadian DRDC on behalf of the project, and remains a proponent to endorse future qualification efforts should there be a transition path.

(U) Researcher at the Defense Research and Development Canada (DRDC) Valcartier Research Centre, are currently working with the director of Land Environment of the Department of National Defence to explore suitable RDX-free replacements for Composition C4. By their account, PAX-52 appears to Canada as a very good option for an RDX-free replacement of Composition C4. They further state DRDC hopes that this product will be supported and transitioned into a commercial option in the near future in the United States (6). If Canadian Armed Forces ultimately decide PAX-52 is the best candidate for C4 replacement and are willing to pay the price for the environmental benefits, then possibly the US will agree to transition the technology into production.

9.2 (U) ENVIRONMENTAL ISSUES

(U) At the 2018 SERDP-ESTCP Symposium, Washington, DC, there were several presentations and discussions that touched upon the high cost of environmental risk factors built into emerging technologies, particularly munitions systems. Questions abounded: Who is supposed to pay for the high cost of environmental testing? When in the R&D life cycle should the DoD invest for environmental protection? Should major funding be applied in early product development with greater up front risk? Or should funding agencies cherry pick innovations with the hopes no future environmental risks will emerge? Many questions from different perspectives. The answer to these questions is that the DoD should expect to pay a premium for environmental protection, sooner rather than later.

(U) The same reasoning is applied to the demonstration technology where the PAX-52 product is designed to be much less toxic to the environment than the current C4, but comes at a price premium to the product managers. The several important characteristics that the alternate technology provides in contrast to C4 (cold-weather moldability, initiation reliability, and continuous processing, etc) do not rank ahead of its higher cost. At some future point the DoD may use a different calculus that monetizes these several benefits and then decide to pay the premium for the superior product.

(U) As stated in the previous section, the Canadian's search for a C4 replacement could have import to the political and legislative process in the US. If our allies choose US developed PAX-52 as their best candidate then that might be the impetus to qualify and field the product to American armed forces. Also, findings from Canadian research may trigger the US EPA to legislate restrictions on RDX thereby compelling the DoD to find an RDX-free alternative. The DoD could implement the demonstration technology into existing industrial-sized load plants without incurring excessive cost and schedule risk.

9.3 (U) PROCUREMENT ISSUES

(U) The DoD would not have to "re-invent the wheel" to stand up a TSE production line. Twin screw extrusion technology is widely used by various industries. There are several providers of production-sized platforms and many more supplies of ancillary feed and handling systems. As well, PLC and SCADA computer programs can be readily purchased and customized to support PAX-52 production on any TSE production platform. The science of compounding and die forming high solids suspension, specifically energetics, is well-founded due to the work in the field by Stevens Institute of Technology. Various branches of the DoD have worked primarily with Stevens Institute of Technology since the mid-1980s in the development of the science and technology base of continuous manufacturing of energetic formulations using twin screw extrusion.

(U) Currently, BAE Ordnance Systems, Inc. (OSI) at Holston Army Ammunition Plant, TN operates an 86mm twin screw extrusion production platform capable of producing PAX-52 to meet all the DoDs demolition procurement requirements. Incidentally, BAE-OSI is the sole producer and supplier of bulk HMX and DMDNT taggant to the DoD. Implementation of the demonstration technology at BAE would carry little risk for the DoD.

9.4 (U) POLITICAL ISSUES

(U) Implementation of the PAX-52 TSE continuous process technology may pose a political challenge within the DoD and among representative members of the Government. The single-step production of PAX-52 M112 demolition blocks would threaten shut-down of an existing C4 M112 production line at the Iowa AAP. This decision could result in the loss of jobs and tax revenue for the particular congressional district in Iowa, which could have ramifications for defense appropriations for the Army.

(U) There may also be an underlying resistance by the decision makers in the DoD to shift program funds away from Iowa AAP to avoid the effects of the Base Realignment and Closure (BRAC) when DoD facilities face activity reductions. Solutions to this scenario are outside the scope of this project.

9.5 (U) FUTURE OPPORTUNITIES

(U) The PI will continue to present project information at technical and scientific forums and remain ARDEC's point of contact for any future solicitation for the technology. If the Canadians choose PAX-52 as their RDX-free demolition explosive there may result a sure path to US explosives qualification, but not necessarily a transition to a US fielded system. If qualification is warranted, this PI would manage the effort.

(U) With increased capability for extrusion/injection loading being developed at ARDEC, PAX-52 is a viable candidate explosive with wide applications. Again, US explosives qualification would be warranted before any down-selection process is conducted for a particular munition. All the research and test data available for PAX-52 would make it a very desirable candidate for any product manager.

(U) There could arise a cold weather mission requirement in regions of conflict around the world that could be met by PAX-52, whether in block form or in bulk. The Army could qualify the PAX-52 material, as described, and then follow this project's production plan for pilot scale quantities to meet mission requirements. With minor investment, ARDEC's TSE facility could ramp up to produce 500 M112 demolition blocks per day.

10 (U) REFERENCES

1. **US Government.** Detail Specification Composition C-4 MIL-DTL-45010B. 2014.
2. —. Detail Specification CHARGE, DEMOLITION - M112, MIL-DTL-50523A, DWG 9404248. 2011.
3. **Sigma-Aldrich.** "Octane", Safety Data Sheet 412230, Version 5.3. 2014.
4. **Thiboutot, S, et al.** *Alternative Options to C4 Plastic Explosive to Mitigate RDX Dispersion in Ranges and Training Areas STO-MP-AVT-224 Paper 18-1.* s.l. : NATO, 2015. STO-MP-AVT-224 Paper 18-1.
5. **Bruckner, Michael, et al.** *OSD-AS Summary PAX-52 Sustainability Analysis.* 2018.
6. **Brousseau, Patrick and Thiboutot, Sonia.** *Summary of the Work Performed on PAX-52.* DRDC Valcartier Research Centre. 2018. Final report will be submitted upon publishing.
7. **Assessment, EPA-National Center for Environmental.** *In Support of Summary Information on the Integrated Risk Information System (IRIS).* 2016.
8. **Daniels, J.I., and Knezovich, J. P.** *"Human Health Risks from TNT, RDX, and HMX in Environmental Media and Consideration of the U.S. Regulatory Environment".* s.l. : UCRL-JC-119715, 1994.
9. **EPA.** *"Technical Fact Sheet – Hexahydro-1,3,5-trinitro-1,3,5-triazine (RDX)".* s.l. : EPA 505-F-14-008, 2014.
10. **Kalyon, Dilhan.** *Mixing index determination from the HPLC data collected on PAX 52 samples that were twin screw extruded at ARDEC.* 2016.
11. **Erol, M and Kalyon, D.** *"Assessment of the Degree of Mixedness of Filled Polymers: Effects of Processing Histories in Batch Mixer and Co-Rotating and Counter-rotating Twin Screw Extruders", Int. Polym. Proc., 20, 228-237* . 2005.
12. **Gelest, Inc.** Safety Data Sheet DMS-T35 POLYDIMETHYLSILOXANE, TRIMETHYLSILOXY TERMINATED. November 2014.
13. **Defense, Department of.** *DoD Emerging Contaminants Program.* 2011.
14. **Hangeland, Erik B and Watts, Kimberly.** Ordnance Environment Program, Environmental Quality Technology Pollution Prevention Program. 2016.
15. **EPA.** *Chemical Assessment Summary of Hexahydro-1,3,5-trinitro-1,3,5-triazine (RDX); CASRN 121-82-4.* 1988.
16. **Smolinski, Benjamin L and Conway, Matthew J.** 2016.
17. **Lee, Kenneth E.** *One Step No Waste Composition C4 Production.* 2014.
18. **Lee, Kenneth and Beckel, Eric.** Formulation Sheet for C4 LOT HM_HMX01 Mixed in B-252. 2013.
19. —. *Composition C-4 Explosive Hazard and Performance Characterization.* s.l. : Unpublished, 2015.
20. **Fallt, James and Swanson, Travis J.** *Physical and Thermal Sensitivity Evaluation and Performance Evaluation of HMX-Silicone Based C-4.* 2015.
21. **Centrella, John M.** *PAX-52 Evaluation at Fort Leonard Wood, MO.* 2018.
22. **EPA.** *Technical Fact Sheet Hexahydro-1,3,5-trinitro- 1,3,5-triazine (RDX).* 2014.
23. **Fair, Michael, et al.** *Continuous Processing of Sodium Carboxymethylcellulose Black Powder and.* Picatinny Arsenal : ARDEC, 2009.
24. **Department of the Army.** Official Policy Statement 200-1, Installation Environmental Management. 2016.
25. **ARDEC.** *Explosives Safety Management Program (ESMP).* 2015.

26. **ARDEC-METC.** *METC Explosives and Ammunition Research, Engineering and Test Operations Environmental Safety and Occupational Health (ESOH) Responsibilities.* 2009.
27. **Bolognini, John and Fair, Michael.** Standing Operating Procedures for Building 1403 (AR-XAEE-P-122). 2015.
28. **Department of the Army.** DA-PAM 385-64 Ammunition and Explosives Safety. 2009.
29. **Rocky Mountain Scientific Laboratory.** *PERFORMANCE AND SENSITIVITY CHARACTERIZATION OF PAX-52- LOT RDD17B046E001.* 2018.
30. **Calvin, Christa, J.** *Bldg 1403, Twin Screw Extruder, HMX (Octogen) Air Sampling Results.* 2017.
31. **American Conference of Governmental Industrial Hygienists.** 2016.
32. **Occupational Safety and Health Administration.** OSHA . [Online] https://www.osha.gov/dts/sltc/methods/partial/pv2032/2032.html .
33. **Office of Management and Budget.** Exhibit P-40, Budget Item Justification Sheet: PB Amended 2014 Army. 2014.
34. **SFAE-AMO-CCS.** M112 Cost Factors. 2017. We buy based on a procurement factor of 1.31 LBS/ block in order to produce a 1.25 approx block. AO uses the scrap for demil.
35. **Ordnance Systems Inc.** *First Article Testing (FAT) Report of Composition C-4 Class 3 Manufactured at Building G-7.* Kingsport : s.n., 2015.
36. **Costa, Louis.** PAX-52 Cost Estimate v1. 2016.
37. *Twin Screw Extruders as Continuous Mixers for Thermal Processing: a Technical and Historical Perspective.* **Martin, Charlie.** 2016, AAPS PharmSciTech, Vol. 17, No. 1, pp. 3-19.
38. **Rauwendaal, Chris.** *Polymer Extrusion.* Cincinnati : Hanser, 2014.
39. **Repka, M.A., Langley, Nigel and DiNunzio, James.** *Melt Extrusion.* New York : Springer, 2013.
40. **White, James L. and Kim, Eung K.** *Twin Screw Extrusion.* Cincinnati : Hanser, 2010.
41. **AMO Premium Efficiency Motor Selection and Application Guide. *U.S. Department of Energy Website.* [Online] December 1, 2016. http://energy.gov/sites/prod/files/2014/04/f15/amo_motors_handbook_web.pdf.**
42. Average Price of Electricity to Ultimate Customers by End-Use Sector. *U.S. Energy Information Administration.* [Online] November 20, 2016. https://www.eia.gov/electricity/monthly/pdf/epm.pdf.
43. Kalyon, Dilhan M. *Estimation of fixed and operating costs for continuous processing of C-4 replacement (PAX 52).* 2018.
44. Program Executive Office - Ammunition. Endorsemnt of Environmental Security Technology Certification Program. 2015.
45. Wins, Cedric F GEN USARMY RDECOM. RDECOM Weekly SITREP for week ending 04 May 2018. 2018. email trail sent to Gen Gustave Perna USARMY USAMC.
46. Explosives Ordnace Disposal. *PAX-52 USER HANDLING AND EMPLOYMENT.* 2018.
47. Di Stasio, Anthony R. Endorsement of the Environmental Security Technology Certification Program. 2015.
48. Anderson, Paul E, et al. *The ARDEC Half Inch Detonation Velocity Test.* 2014.
49. Black, Brian, et al. PAX-52 Test Plan and Assessment at Fort Leonard Wood. 2016.
50. Kase, James. EOD Evaluation of PAX-52. 2016.
51. *Deposition testing results of PAX-52 and ARDEC shaped charges.* Thiboutot, Sonia, Brouseau, Patrick and Brochu, David. 2018. US/CA DEA-1753 meeting, Quebec, July 2018.

52. USA Public Health Command. Analysis of the Components of IMX-101 by Liquid Chromatography and Gas Chromatography . ***SOP Number DLS-810.*** **2011.**
53. Spicer, Daniel and Preuett, Stanley. ***M112 Demolition Block Pack-out Line Modernization, ARMET-TR-09062.*** **Picatinny : US Army ARDEC, 2010.**
54. Army, US. ***AR 40-5, Preventive Medicine.***
55. HMX (Octogen) Safety Data Sheet, Ordnance Systems, Inc.
56. Threshold Limit Values & Biological Exposure Indices. ***2016.***
57. Accurate Energetic Systems. ***15-01-INIT307 Milestone 3, Task 1.*** **2017.**
58. ***Statistical Basis for Predicting Technological Progress.*** **Nagy, B, et al. e52669, 2013 : PLOS ONE, Vol. 8(2).**

10.1 (U) SECTION 2.1 REFERENCES:

1. W. L. Lambert, "Facility modification for full-length 260"diameter motor processing and test", report of Aerojet Solid Propulsion Company submitted to NASA Lewis Research Center (contract number NAS3-12041, report designation NASA CR 72729), August (1970).
2. U. Yilmazer and D. M. Kalyon, "Slip effects in capillary and parallel disk torsional flows of highly filled suspensions," Journal of Rheology, 33, 1197-1212, 1989.
3. U. Yilmazer, C. G. Gogos, and D. M. Kalyon, "Mat formation and unstable flows of highly filled suspensions in capillaries and continuous processors," Polymer Composites, 10, 242-248, 1989.
4. D. M. Kalyon and U. Yilmazer, "Rheological behavior of highly filled suspensions which exhibit slip at the wall," in *Polymer Rheology and Processing*, p. 241-77, 1990.
5. U. Yilmazer and D. M. Kalyon, "Dilatancy of concentrated suspensions with Newtonian matrices," Polymer Composites, 12, 226-232, 1991.
6. D. M. Kalyon, R. Yazici, C. Jacob, B. Aral, and S. W. Sinton, "Effects of air entrainment on the rheology of concentrated suspensions during continuous processing," Polymer Engineering & Science, 31, 1386-1392, 1991.
7. D. M. Kalyon and H. S. Gokturk, "Adjustable gap rheometer," US Patent 5277058 A, 1992.
8. D. M. Kalyon, P. Yaras, B. Aral, and U. Yilmazer, "Rheological behavior of a concentrated suspension: A solid rocket fuel simulant," Journal of Rheology, 37, 35-53, 1993.
9. D. M. Kalyon, "Rheological Behavior of Solid Rocket Fuel Simulants," 1993 JANNAF Propellant Development and Characterization Subcommittee Meeting, Lawrence Livermore National Laboratory, Livermore, CA, April 29, 1993.
10. B. K. Aral and D. M. Kalyon, "Effects of temperature and surface roughness on time-dependent development of wall slip in steady torsional flow of concentrated suspensions," Journal of Rheology, 38, 957-972, 1994.

11. P. Yaras, D. Kalyon, and U. Yilmazer, "Flow instabilities in capillary flow of concentrated suspensions," Rheologica Acta, 33, 48-59, 1994.
12. M. Michienzi, C. Murphy and D. M. Kalyon, "Using Instrumented Capillary Dies to Characterize the Rheology of Extruded Composite Propellant," Proceedings of JANNAF Conference, April, (1996).
13. B. K. Aral and D. M. Kalyon, "Viscoelastic material functions of noncolloidal suspensions with spherical particles," Journal of Rheology, 41, 599-620, 1997.
14. D. M. Kalyon, "Characterization of Poly(BAMO/AMMO)", Thermoplastic Elastomer (TPE) Processing Workshop at JANNAF Propellant Development Subcommittee Meeting, NASA AMES Center, Sunnyvale, California, 17 March, (1997)
15. S. E. Prickett, D. M. Kalyon and S. Railkar, " Temperature Dependent Shear Viscosity Material Function of AA-6 Propellant" JANNAF Propellant Development and Characterization Subcommittee Meeting, NASA AMES Center , Sunnyvale, California, April (1997)
16. M.A. Michienzi, C.M. Murphy, D. M. Kalyon and S. Railkar, Wall Slip and Capillary Flow Behavior of Extrudable Composite Propellant, Proceedings of JANNAF Conference Propellant Development and Characterization Subcommittee Meeting, Sunnyvale, California, April (1997).
17. D. M. Kalyon, S. Railkar, S.E. Prickett, M.A. Michienzi, C.M. Murphy and F. M. Gallant, " Capillary and On-Line Slit Rheometry of an HE LOVA Gun Propellant Formulation," Proceedings of JANNAF Conference Propellant Development and Characterization Subcommittee Meeting, Sunnyvale, California (1997).
18. E. Kucukpinar and D. M. Kalyon "Viscoelastic Material Function and Extrudability of BAMO/AMMO TPE", Proceedings of 9th Continuous Mixer and Extruder Users Group Meeting, Weehawken, NJ, 2-4 June, (1997)
19. A. Lawal and D. M. Kalyon, "Squeezing flow of viscoplastic fluids subject to wall slip," Polymer Engineering & Science, 38, 1793-1804, 1998.
20. A. Lawal and D. Kalyon, "Compressive squeeze flow of generalized Newtonian fluids with apparent wall slip," International Polymer Processing, 15, 63-71, 2000.
21. M. Allende and D. M. Kalyon, "Assessment of particle-migration effects in pressure-driven viscometric flows," Journal of Rheology, 44, 79-90, 2000.
22. H. S. Tang and D. M. Kalyon, "Estimation of the parameters of Herschel-Bulkley fluid under wall slip using a combination of capillary and squeeze flow viscometers," Rheologica acta, 43, 80-88, 2004.
23. D. M. Kalyon, "Apparent slip and viscoplasticity of concentrated suspensions," Journal of Rheology, 49, 621-640, 2005.
24. D. Kalyon, H. Tang, and B. Karuv, "Squeeze flow rheometry for rheological characterization of energetic formulations," Journal of Energetic Materials, 24, 195-212, 2006.

25. D. M. Kalyon, H. Gevgilili, J. E. Kowalczyk, S. E. Prickett, and C. M. Murphy, "Use of adjustable-gap on-line and off-line slit rheometers for the characterization of the wall slip and shear viscosity behavior of energetic formulations," Journal of Energetic Materials, 24, 175-193, 2006.
26. D. M. Kalyon, "An overview of the rheological behavior and characterization of energetic formulations: Ramifications on safety and product quality," Journal of Energetic Materials, 24, 213-245, 2006.
27. E. Birinci, H. Gevgilili, D. M. Kalyon, B. Greenberg, D. F. Fair, and A. Perich, "Rheological characterization of nitrocellulose gels," Journal of Energetic Materials, 24, 247-269, 2006.
28. D. M. Kalyon, D. Dalwadi, M. Erol, E. Birinci, and C. Tsenoglu, "Rheological behavior of concentrated suspensions as affected by the dynamics of the mixing process," Rheologica Acta, 45, 641-658, 2006.
29. D. M. Kalyon and H. Tang, "Inverse problem solution of squeeze flow for parameters of generalized Newtonian fluid and wall slip," Journal of Non-Newtonian Fluid Mechanics, 143, 133-140, 2007.
30. H. Tang and D. M. Kalyon, "Unsteady circular tube flow of compressible polymeric liquids subject to pressure-dependent wall slip," Journal of Rheology, 52, 507-525, 2008.
31. H. Tang and D. M. Kalyon, "Time-dependent tube flow of compressible suspensions subject to pressure dependent wall slip: ramifications on development of flow instabilities," Journal of Rheology, 52, 1069-1090, 2008.
32. J. Pérez-González, J. J. López-Durán, B. M. Marín-Santibáñez, and F. Rodríguez-González, "Rheo-PIV of a yield-stress fluid in a capillary with slip at the wall," Rheologica Acta, 51, 937-946, 2012.
33. S. Aktas, D. M. Kalyon, B. M. Marín-Santibáñez, and J. Pérez-González, "Shear viscosity and wall slip behavior of a viscoplastic hydrogel," Journal of Rheology, 58, 513-535, 2014.
34. D. M. Kalyon and S. Aktas, "Factors affecting the rheology and processability of highly filled suspensions," Annual Review of Chemical and Biomolecular Engineering, 5, 229-254, 2014.
35. J. F. Ortega-Avila, J. Pérez-González, B. M. Marín-Santibáñez, F. Rodríguez-González, S. Aktas, M. Malik, et al., "Axial annular flow of a viscoplastic microgel with wall slip," Journal of Rheology, vol. 60, pp. 503-515, 2016.
36. D. M. Kalyon, A. D. Gotsis, U. Yilmazer, C. G. Gogos, H. Sangani, B. Aral, et al., "Development of experimental techniques and simulation methods to analyze mixing in co-rotating twin screw extrusion," Advances in Polymer Technology, 8, 337-353, 1988.

37. D. M. Kalyon, A. Gotsis, C. Gogos and C. Tsenoglou, "Simulation of the Mixing of Highly Filled Suspensions in the Co-rotating Twin Screw Extrusion Process," Innovative Science and Technology Symposium of SPIE, Propulsion , 71-78 (1988).
38. D. M. Kalyon and H. N. Sangani, "An experimental study of distributive mixing in fully intermeshing, co-rotating twin screw extruders," Polymer Engineering & Science, 29, 1018-1026, 1989.
39. J. Sinton, J. Crowley, G. Lo, D. Kalyon, and C. Jacob, "Nuclear magnetic resonance imaging studies of mixing in a twin-screw extruder," ANTEC : Society of Plastics Engineers Annual Technical Papers, 36, 116-119, 1990.
40. D. M. Kalyon, R. Yazici, C. Jacob, B. Aral, and S. W. Sinton, "Effects of air entrainment on the rheology of concentrated suspensions during continuous processing," Polymer Engineering & Science, 31, 1386-1392, 1991.
41. D. M. Kalyon, C. Jacob, and P. Yaras, "An experimental study of the degree of fill and melt densification in fully-intermeshing, co-rotating twin screw extruders," Plastics Ccomposites processing and Applications, 16, 193-200, 1991.
42. R. Yazici and D. Kalyon, "Degree of mixing analyses of concentrated suspensions by electron probe and x-ray diffraction," Rubber Chemistry and Technology, 66, 527-537, 1993.
43. D. M. Kalyon, R. Yazici and A. Lawal, "Techniques to Analyze Goodness of Mixing of Concentrated Suspensions and Simulation of Mixing in Extrusion Flows," Proceedings of 1993 JANNAF Propellant Development and Characterization Subcommittee Meeting, Livermore, CA, April 28 (1993).
44. A. Lawal, D. M. Kalyon, and Z. Ji, "Computational study of chaotic mixing in co-rotating two-tipped kneading paddles: Two-dimensional approach," Polymer Engineering & Science, 33, 140-148, 1993.
45. A. Lawal and D. M. Kalyon, "Mechanisms of mixing in single and co-rotating twin screw extruders," Polym. Eng. Sci., 35, 1325-1338, 1995.
46. A. Lawal and D. M. Kalyon, "Simulation of intensity of segregation distributions using three-dimensional fem analysis: Application to corotating twin screw extrusion processing," J. Applied Polymer Sci., 58, 1501-1507, 1995.
47. B. K. Aral and D. Kalyon, "Rheology and extrudability of very concentrated suspensions: Effects of vacuum imposition," Plastics, Rubber and Composites Processing and Applications, 24, 201-10, 1995.
48. R. Yazici and D. M. Kalyon, "Quantitative characterization of degree of mixedness of LOVA grains," Journal of Energetic Materials, 14, 57-73, 1996.
49. R. Yazici and D. M. Kalyon, " Degree of Mixing Analysis in LOVA Propellants by Wide-Angle x-Ray Diffraction," US Army Report, ARPBM-CR-95001, 33 pages, April, (1996)

50. R. Yazici and D. M. Kalyon, "Analysis of degree of mixing in filled polymers by wide-angle X-ray diffraction," ANTEC : Society of Plastics Engineers Annual Technical Papers, 2, 2076-2080, 1997.
51. R. Yazici and D. M. Kalyon, “On-Line and Off-line Analysis of the Extruded and Cast Energetic Materials for Microstructural Distributions”, Proceedings of the 10th Joint Ordnance Commander’s Group, Continuous Mixer and Extruder Users Group Meeting, Thiokol Center, Brigham City, Utah, May 10-12, pages 61-69 (1999).
52. D. M. Kalyon, E. Birinci, R. Yazici, B. Karuv, and S. Walsh, "Electrical properties of composites as affected by the degree of mixedness of the conductive filler in the polymer matrix," Polym. Eng. Sci, 42, 1609-1617, 2002.
53. M. Erol and D. Kalyon, "Assessment of the degree of mixedness of filled polymers: Effects of processing histories in batch mixer and co-rotating and counter-rotating twin screw extruders," Int. Polym. Proc., 20, 228-237, 2005.
54. C. Feger, J. D. Gelorme, M. McGlashan-Powell and D. Kalyon, “Mixing, Rheology and Stability of Highly Filled Thermal Pastes”, IBM Journal of Research and Development, 49, 4/5, 699-707 (2005).
55. D. M. Kalyon, D. Dalwadi, M. Erol, E. Birinci, and C. Tsenoglu, "Rheological behavior of concentrated suspensions as affected by the dynamics of the mixing process," Rheologica acta, 45, 641-658, 2006.
56. M. Allende, D. F. Fair, D. M. Kalyon, D. Chiu, and S. Moy, "Development of particle concentration distributions and burn rate gradients upon shear-induced particle migration during processing of energetic suspensions," Journal of Energetic Materials, vol. 25, pp. 49-67, 2007.
57. D. Fair, D. M. Kalyon, S. Moy, and L. R. Manole, "Cross-sectional functionally graded propellants and method of manufacture," US Patent 7,896,989, 2011.
58. A. Gotsis, Z. Ji, and D. Kalyon, "3-D analysis of the flow in co-rotating twin screw extruders," ANTEC : Society of Plastics Engineers Annual Technical Papers, 36, 139-142, 1990.
59. Z. Ji and D. M. Kalyon, "Two Dimensional Computational Study of Chaotic Mixing in Two-tipped Kneading Paddles of Co-rotating Twin Screw Extruders," ANTEC : Society of Plastics Engineers Annual Technical Papers, 1, 1323-1327, 1992.
60. A. Lawal, D. M. Kalyon, and U. Yilmazer, "Extrusion and lubrication flows of viscoplastic fluids with wall slip," Chemical Engineering Communications, 122, 127-150, 1993.
61. A. Lawal and D. M. Kalyon, "Non-isothermal model of single screw extrusion of generalized Newtonian fluids," Numerical Heat Transfer, Part A Applications, 26, 103-121, 1994.
62. A. Lawal and D. M. Kalyon, "Single screw extrusion of viscoplastic fluids subject to different slip coefficients at screw and barrel surfaces," Poly. Eng. Sci., vol. 34, pp. 1471-1479, 1994.

63. A. Lawal, S. Railkar, and D. Kalyon, "A new approach to simulation of die flow which incorporates the extruder and rotating screw tips in the analysis," International Polymer Processing, 12, 123-129, 1997.
64. A. Lawal and D. M. Kalyon, "Viscous heating in nonisothermal die flows of viscoplastic fluids with wall slip," Chemical Engineering Science, 52, 1323-1337, 1997.
65. D. M. Kalyon, A. Lawal, R. Yazici, P. Yaras, and S. Railkar, "Mathematical modeling and experimental studies of twin-screw extrusion of filled polymers," Polymer Eng. Sci., 39, 1139-1151, 1999.
66. A. Lawal and D. M. Kalyon, "Analysis of nonisothermal screw extrusion processing of viscoplastic fluids with significant back flow," Chemical Engineering Science, 54, 999-1013, 1999.
67. A. Lawal, S. Railkar, and D. M. Kalyon, "Mathematical modeling of three-dimensional die flows of viscoplastic fluids with wall slip," Journal of Reinforced Plastics and Composites, 19, 1483-1492, 2000.
68. D. M. Kalyon, H. Gevgilili, and A. Shah, "Detachment of the polymer melt from the roll surface: calendering analysis and data from a shear roll extruder," International Polymer Processing, 19, 129-138, 2004
69. M. Malik and D. Kalyon, "3-D finite element simulation of processing of generalized newtonian fluids in counter-rotating and tangential twin screw extruder and die combination," International Polymer Processing, 20, 398-409, 2005.
70. S. Ozkan, H. Gevgilili, D. Kalyon, J. Kowalczyk, and M. Mezger, "Twin-Screw Extrusion of Nano-Alumina–Based Simulants of Energetic Formulations Involving Gel-Based Binders," Journal of Energetic Materials, 25, 173-201, 2007.
71. J. Kowalczyk, M. Malik, D. Kalyon, H. Gevgilili, D. Fair, M. Mezger, et al., "Safety in design and manufacturing of extruders used for the continuous processing of energetic formulations," Journal of Energetic Materials, 25, 247-271, 2007.
72. D. M. Kalyon and M. Malik, "An integrated approach for numerical analysis of coupled flow and heat transfer in co-rotating twin screw extruders," International Polymer Processing, 22, 293-302, 2007.
73. H. Gevgilili, D. M. Kalyon, and A. Shah, "Processing of Energetics in Continuous Shear Roll Mills," Journal of Energetic Materials, 26, 29-51, 2008.
74. D. M. Kalyon, "An analytical model for steady coextrusion of viscoplastic fluids in thin slit dies with wall slip," Polym. Eng. Sci., 50, 652-664, 2010.
75. D. M. Kalyon and M. Malik, "Axial laminar flow of viscoplastic fluids in a concentric annulus subject to wall slip," Rheologica Acta, 51, 805-820, 2012.
76. M. Malik, D. Kalyon, and J. Golba Jr, "Simulation of co-rotating twin screw extrusion process subject to pressure-dependent wall slip at barrel and screw surfaces: 3D FEM Analysis for combinations of forward-and reverse-conveying screw elements," International Polymer Processing, 29, 51-62, 2014.

77. D. M. Kalyon, "Mixing in Continuous Processors," Encyclopedia of Fluid Mechanics, 7, Gulf Publishing (1988) Chapter 28, 887-926.
78. T. Fiske, S. Railkar and D. M. Kalyon, "Effects of Segregation on the Packing of Spherical and Non-Spherical Particles," Powder Technology, 81, 57-64 (1994).

11 (U) APPENDICES

11.1 (U) APPENDIX B: PAX-52 (RDD16E046-134) CHARACTERIZATION TEST DATA

Table 27 (U) Physical Sensitivity Data:

(U) UNCLASSIFIED

(U) Small Scale Sensitivity Test Summary
PAX-52 (RDD16E046E134) and HMX (BAE10K071-101)

Test	PAX-52	HMX
ERL Impact Sensitivity	<100cm	25.3 cm (50%)
BAM Friction Sensitivity	> 360 N	> 128 N
ESD Static Sensitivity	> 0.16 J	> 0.051 J

(U) UNCLASSIFIED

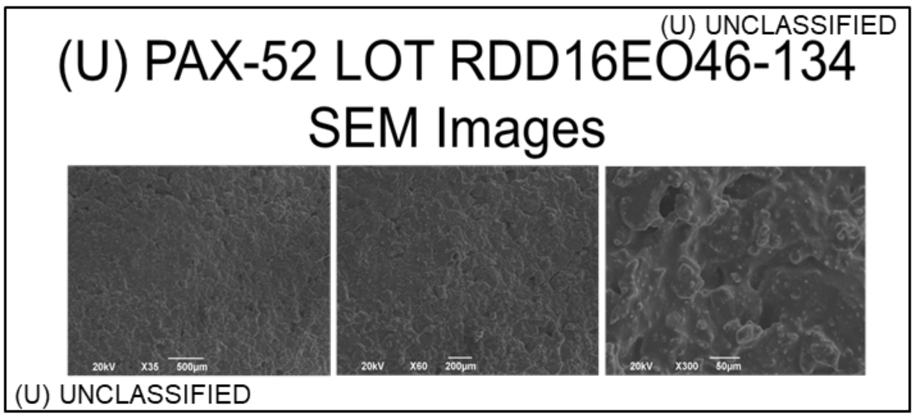


Figure 52 (U) Scanning Electron Micrography (SEM)

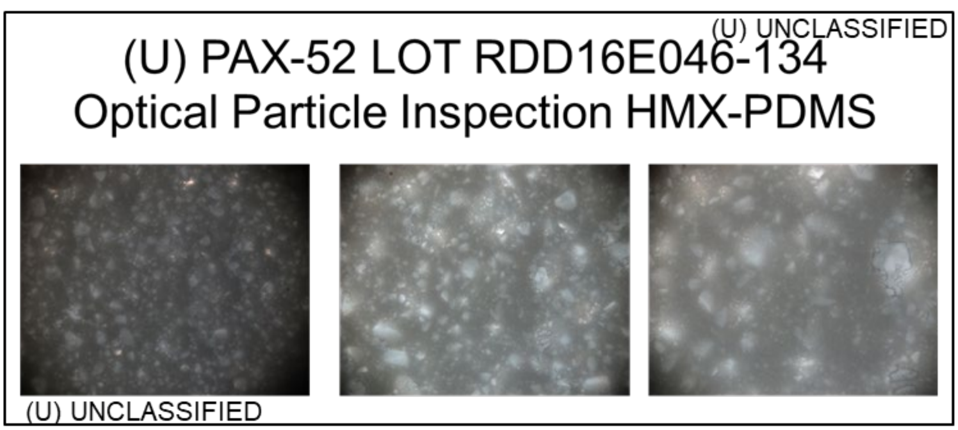


Figure 53 (U) Optical Particle Inspection

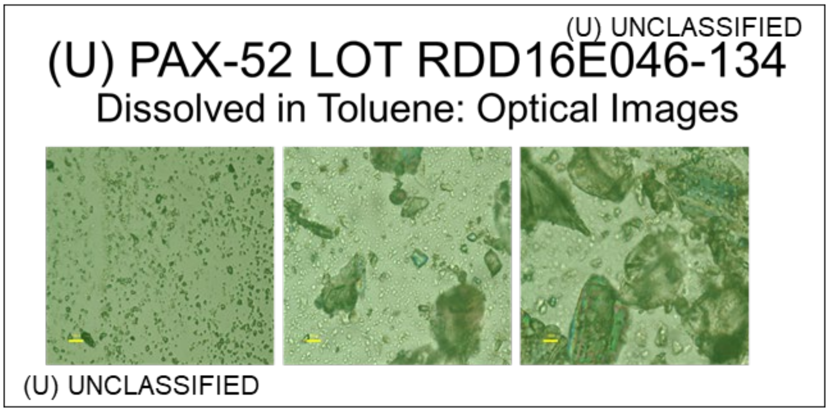


Figure 54 (U) Dissolved PAX-52 - Optical Images

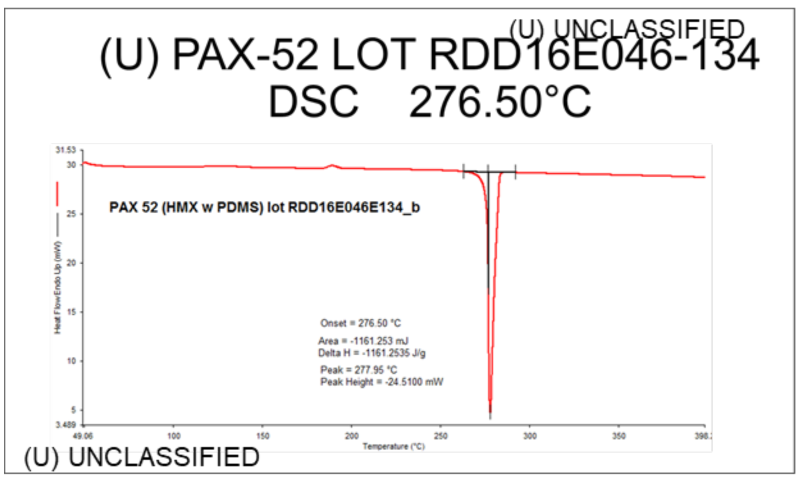


Figure 55 (U) Differential Scanning Calorimetry (DSC) Heat of Decomposition

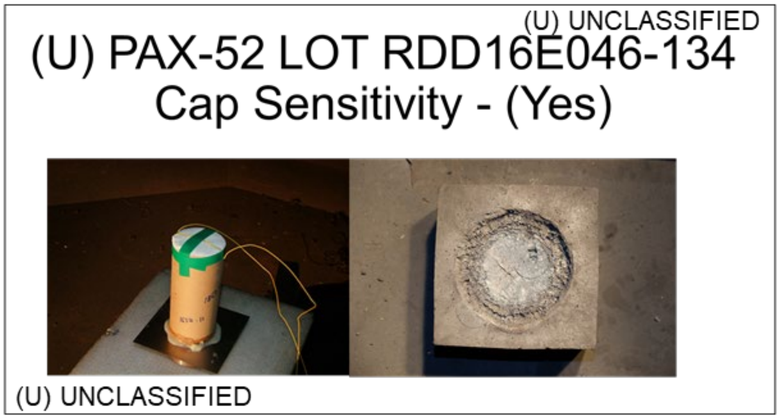


Figure 56 (U) Cap Sensitivity: Test Set-up and Witness Plate Result

Table 28 (U) Detonation Velocity and Detonation Pressure

(U) UNCLASSIFIED

(U) PAX-52 LOT RDD16E046-134
Detonation Velocity 7.83 km/s
CJ Pressure 242 kbar (24.2 GPa)

Detonation Velocity Data Table

Pin ID	Pin Locations (mm)	Distance (mm)	Time (µs)	Velocity (mm/µs)
DV 1 (1-2)	50.8	51.128	3.256	7.768
DV 2 (2-3)	76.2	76.419	3.259	7.777
DV 3 (3-4)	101.6	101.764	3.280	7.734
DV 4 (4-5)	127.0	127.131	3.164	8.022
DV 5 (1-5)	152.4	152.510	12.959	7.824
			Average	7.825

(U) UNCLASSIFIED

Table 29 (U) Insensitive High Explosive (IHE) Gap Test

(U) UNCLASSIFIED

(U) PAX-52 LOT RDD16E046-134
Insensitive High Explosive (IHE) Gap Test
166.5 Cards

Insensitive High Explosives Gap Test (IHE)
50% Point = N + 1/2 = 1.665" = 166.5 cards

Test #	Density (g/cc)	Gap (in.)	Dent (in.)	Result (GO/NO-GO)	Notes
1	1.66	none	0.027	GO	BASELINE
2	1.69	2.000	---	NO-GO	
3	1.68	1.700	---	NO-GO	
4	1.68	1.300	0.026	GO	
5	1.66	1.500	0.026	GO	
6	1.68	1.600	0.023	GO	
7	1.70	1.650	0.020	GO	
8	1.69	1.680	0.013	NO-GO	
9	1.68	1.670	0.008	NO-GO	N+1
10	1.68	1.660	0.017	GO	N

(U) UNCLASSIFIED

11.2 (U) APPENDIX C: SPECIFICATIONS FOR POWDER AND LIQUID FEEDERS

(U) Powder Feeder. The Accurate model 602 is a loss-in-weight powder feeder. It is outfitted with: explosion proof motors, a conductive lower hopper (bladder), a stainless steel extension hopper, continuously welded feed screw, and a stainless steel lid. The physical dimensions of the feeder including the scale are 36” x 22” x 22” (H x W x D). The feed screw is cantilevered out 8.5” from the feeder. The total capacity is 25.5US gal with an extension hopper and a maximum scale weight is 110lbf. The feed scale is capable of reading 0.001lbf. It is powered by two ¼hp, 90Vdc variable speed motors with a maximum of 1800rpm, one motor for the feed screw and one motor for the external paddle agitations. Motor speeds are geared down to allow for more accurate feeding; 24:9 and 24:15, feed screw and agitator gear ratio respectively. The agitator paddles are controlled manually through a potentiometer and set for 20% of maximum motor speed. The feed screw is an open helix–full pitch; with a diameter of 0.75”.

(U) Liquid Feeder. The liquid loss-in-weight feeder utilizes an Accurate scale and control system. It is outfitted with: an explosion proof motor, all stainless steel tank construction, and a stainless steel lid. The physical dimensions of the tank are 16” x 19” (Diameter x Height). The output of the feed pump is 10” below the outlet of the tank. The total capacity is 12US gal with a maximum scale weight of 110lbf. The scale is capable of reading 0.001lbf. It is powered by one ½hp, 90Vdc variable speed motor with a maximum of 1800rpm. This motor drives a Crane Chem/Meter double diaphragm pump (model number 2020-11-17sw), which feeds the material. This pump has maximum capacity of 10gal and maximum pressure of 2000psi.

11.3 (U) APPENDIX D: TEST AND EVALUATION STANDARDS

1. (U) Impact Sensitivity- ERL (Explosives Research Laboratory)/Bruceton Apparatus
Purpose: This test is designed to measure the sensitivity of an energetic material to impact.
Reference: AOP-7 Edition 2 Rev. 1 U.S. 201.01.001 and STANAG 4489 Ed. 1 "Explosives, Impact Sensitivity Tests".

2. (U) Friction Sensitivity - BAM Friction Test
Purpose: This test is used to determine the sensitivity of a substance when subjected to a sliding frictional force.
Reference: AOP-7 Edition 2 Rev. 1 U.S. 201.02.006

3. (U) Electrostatic Discharge (ARDEC (Picatinny Arsenal) Method)
Purpose: This test determines the energy threshold required to ignite explosives by electrostatic stimuli of varying intensities. Material response data obtained can then be used to characterize the probability of initiation due to electrostatic discharge (ESD) events.
Reference: AOP-7 Edition 2 Rev. 1 U.S. 201.03.001

4. (U) Thermal Stability - Determination of Critical Temperature and Self-Heating
Purpose: Predict and to experimentally determine the critical temperature and self-heating properties associated with a given energetic material.
Reference: AOP-7 Edition 2 Rev. 1 U.S. 202.01.012
Reference: DSC method: AOP-7 Edition 2 Rev. 1 U.S. 202.01.008

5. (U) Composition Analysis. High Performance Liquid Chromatography
Purpose: Qualitative and quantitative analysis of nonvolatile organic compounds.
Reference: NATO STANAG 4284 edition 1; Test Procedure: paragraph 2; RDX-Liquid Phase Chromatography Method

6. (U) Mixing Index
Purpose: The mixing index is a quantitative indicator of the degree of mixedness of a mixture.
Reference: R. Yazici and D. M. Kalyon, "Quantitative characterization of degree of mixedness of LOVA grains," Journal of Energetic Materials, 14, 57-73, 1996.

7. (U) Particle Size Distribution of HMX Class I Type B
Purpose: Determine particle size distribution of HMX with Laser Light Diffraction Analyzer.
Reference: Micromeritics Saturn DigiSizer 5200 laser light diffraction analyzer.

8. (U) Thermal Stability (Constant Temperature) - Vacuum Thermal Stability (VTS)
Purpose: This test measures the chemical stability of an explosive at an elevated temperature under vacuum.
Reference: AOP-7 Edition 2 Rev. 1 U.S. 202.01.001

9. (U) Thermal Stability

Purpose: This test is designed to measure the stability of the substance when subjected to elevated thermal conditions to determine if the substance is too hazardous to transport in the state in which it was tested.
Reference: AOP-7 Edition 2 Rev. 1 U.S. 202.01.013 and TB700-2 Para 5-4.b

10. (U) Detonation Velocity
Purpose: To determine the detonation velocity of an explosive and to characterize the explosive for application, performance, and safety.
AOP-7 Edition 2 Rev. 1 U.S. 302.01.001

11. (U) Plate Dent Test.
Purpose: To determine the detonation pressure of an explosive.
Reference: ARDEC test. (48)

12. (U) Scanning Electron Micrography (SEM)
Purpose: To evaluate how well the formulation is mixed and coated.
References: None.

13. (U) Cap Sensitivity
Purpose: The test is designed to determine the sensitivity of a substance to the shock from a standard detonator or blasting cap.
Reference: TB700-2 Para 5.7b

14. (U) Insensitive High Explosive (IHE) Gap Test
Purpose: This test measures the sensitivity to detonation of an explosive exposed to an explosive induced shock. This procedure is applicable to explosives with large critical diameters ranging up to 0.75 inches.
Reference: AOP Edition 2 Rev. 1 U.S. 201.04.005

15. (U) Variable Closed Confinement Test (VCCT)
Purpose: To measure an energetic response to heating.
Reference: AOP-7 202.01.002

16. (U) Small Scale Burning Test
Purpose: To measure an energetic response to heating.
Reference: TB700-2 Para. 5.4a

17. (U) Exudation Characteristics
Purpose: This test is designed to measure the exudation of energetic materials.
Reference: MIL-STD 1751A Method 1161

18. (U) M112 Demolition Block Physical Properties: Plasticity
Purpose: This test measures the force required for a cone point to penetrate the M112 demolition charge for a specified distance.
Reference: MIL-DTL-50523B paragraph 4.4.1

19. (U) Specific Gravity
Purpose: To determine the specific gravity of the M112 sample.
Reference: MIL-DTL-50523B paragraph 4.4.2

20. (U) Energy Output Test
Purpose: The demolition charge shall cut a 1.0 inch thick steel witness plate, ASTM A36 grade 1018, completely into two separate sections.
Reference: MIL-DTL-50523B paragraph 4.4.3

21. (U) User Evaluation at Fort Leonard Wood, MO.
Purpose: To have combat engineers use PAX-52 in several test configuration and get their qualitative feedback.
Reference: Evaluation Survey. Appendix D, (U) Appendix E: Qualitative Survey of C4 vs. PAX-52
Reference: Field Test: TM-5-1375-238-10 (Not available for distribution.)
Reference: Test Plan. (49) Reference: Target: MIL-DTL-12560C1-2 Appendix D, Section 11.4.1

(U) Target Plate *Target plate was erected at the edge of an earth berm to capture any stray projectiles during the tests. Soldiers painted a "happy face" on the target so they could aim at each eye. Calculations were made for how much the projectiles would drop as they traveled down range.*

22. (U) User Evaluation: Explosives Ordnance Disposal (EOD)
Purpose: To have EOD technicians use PAX-52 in several test configurations and get their qualitative feedback.
Reference: EOD Test Plan. (50) (Not available for distribution.)

23. (U) RDX Deposition Test (51)
Purpose: To measure the quantity of RDX deposited into the environment for a given mass of PAX-52 material.
(U) Deposition Rate (DR) and Detonation Efficiency (DE) Test Procedure:

1. Total mass (mg) in sample = aqueous + solid fractions
2. Area sampled = number of increments X 0.01 m2 (eg. 100 inc. = 1 m2)
3. Mass deposited = Mass for y m2 X Area of the plume
4. Total mass deposited = Mass deposited in ITP + mass deposited in OTP
5. DR = (Mass in plume/original mass in the round) X 100
6. Detonation efficiency = original mass in round – ((original mass in round – mass in plume) X 100)
7. Ranged from 1 x 10 -8 % (ng scale) to 100%. Directly related to combustion and detonation efficiencies.

(U) Estimation of Residual Energetic Mass:

1. Plume was measured. **Figure 57**
2. Samples were melted, filtrated and aqueous and solid fractions were analyzed
3. Concentration in liquid extract (mg/L) x volume of sample = Mass solubilized
4. Concentration in solid extract x volume of extraction solvent or = Mass solid

Figure 57 (U) Procedure for Analyzing Samples *Plume area is measured and methodically sampled (a.) The samples are melted, filtrated (b-c) then the aqueous and solid fractions are analyzed (d.)*

24. (U) HMX Workplace Exposure

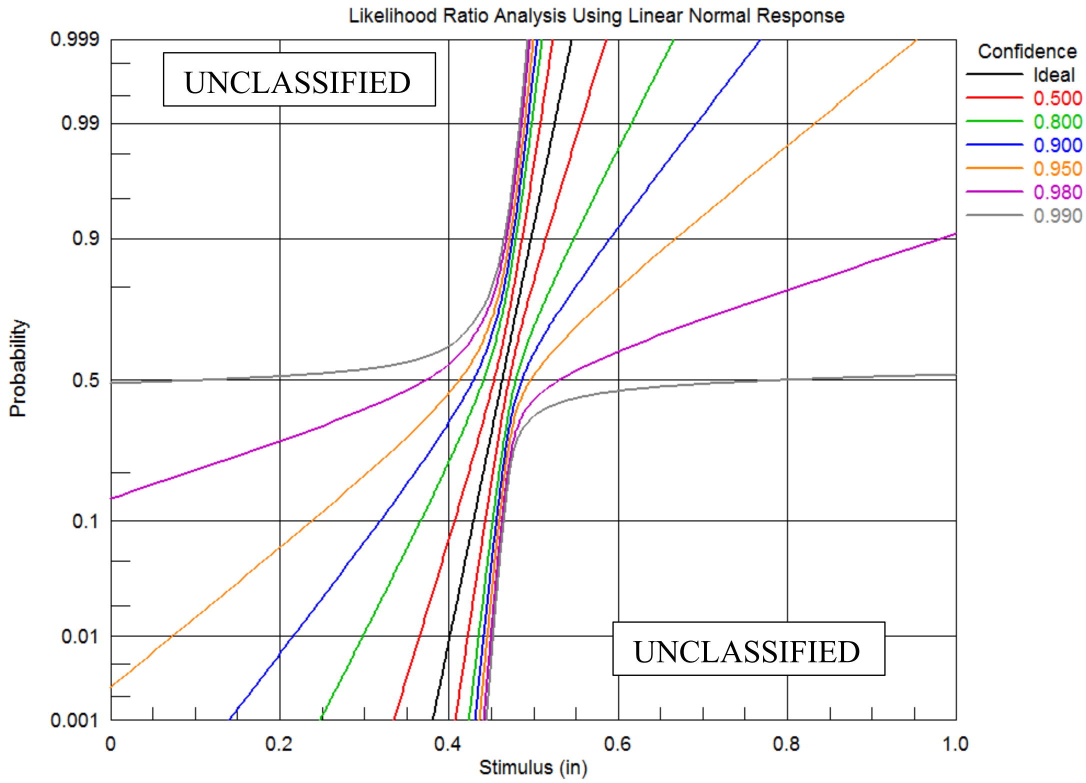
Purpose: To determine if the formulation or TSE process exposes workers to airborne HMX particles.
Reference: (22)
Reference: (52) SOP DLS-810
AR 40-5, Preventive Medicine.
HMX (Octogen) Safety Data Sheet, Ordnance Systems, Inc.,
http://www.petroexplo.com/catalog/files/HMX-MSDS.pdf
https://www.osha.gov/dts/sltc/methods/partial/pv2032/2032.html

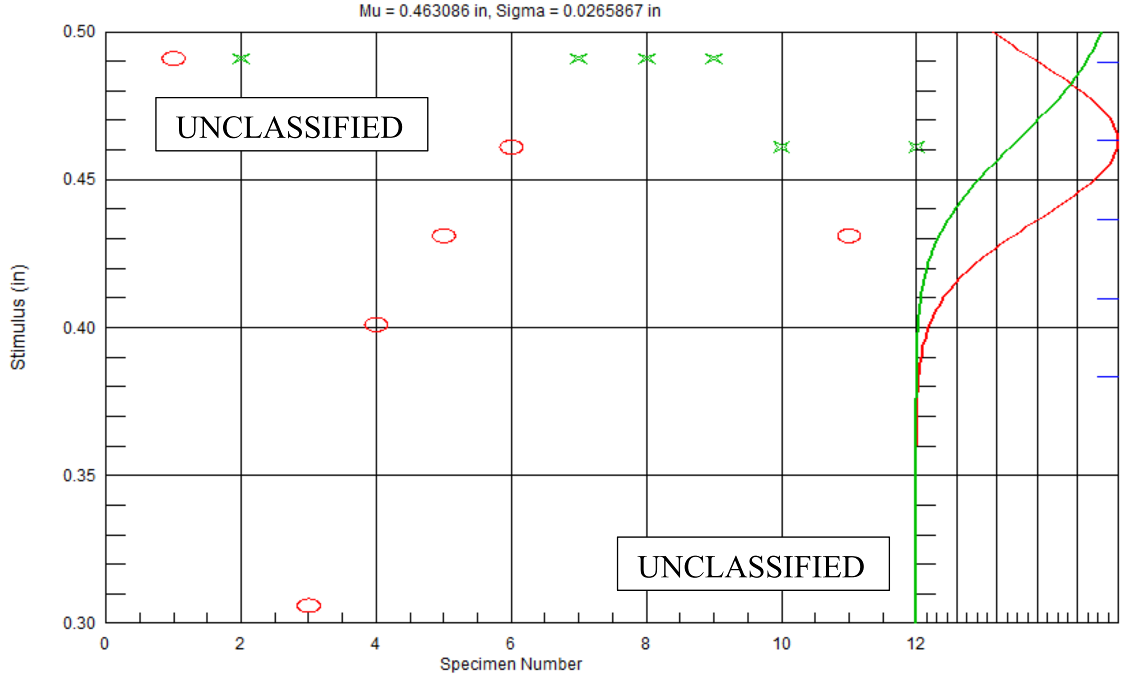
11.4 (U) APPENDIX E: QUALITATIVE SURVEY OF C4 VS. PAX-52

Activities and details of the evaluation event were organized by ARDEC liaison to the MSCoE at Fort Leonard Wood, MO. Logistics support to the evaluation was provided by several other engineers at ARDEC. The evaluation agenda and technical details are not available for release with this report.

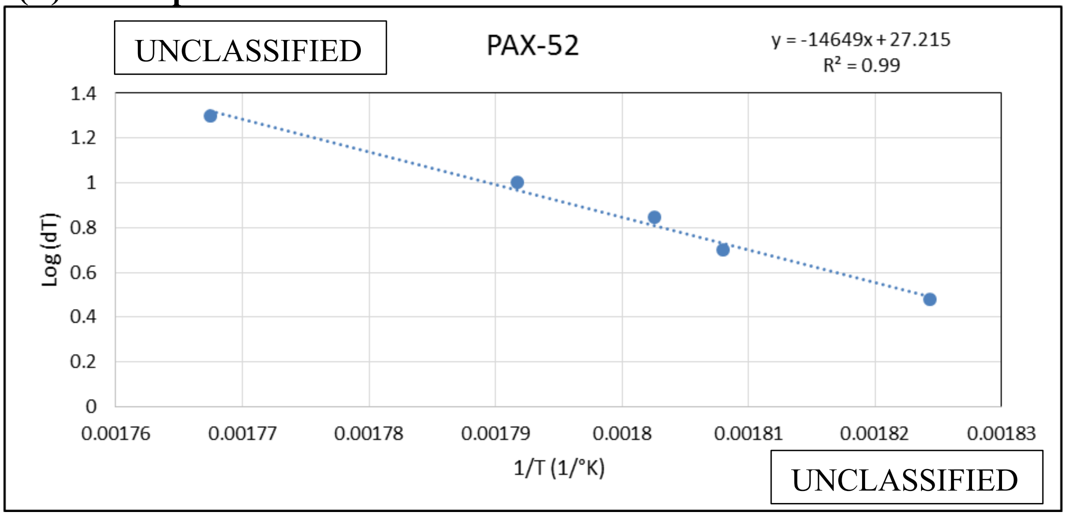
11.5 (U) APPENDIX F: TEST DATA AND ANALYSES

1. **(U) Plate Cutting** Probability, Confidence and Test History Plots

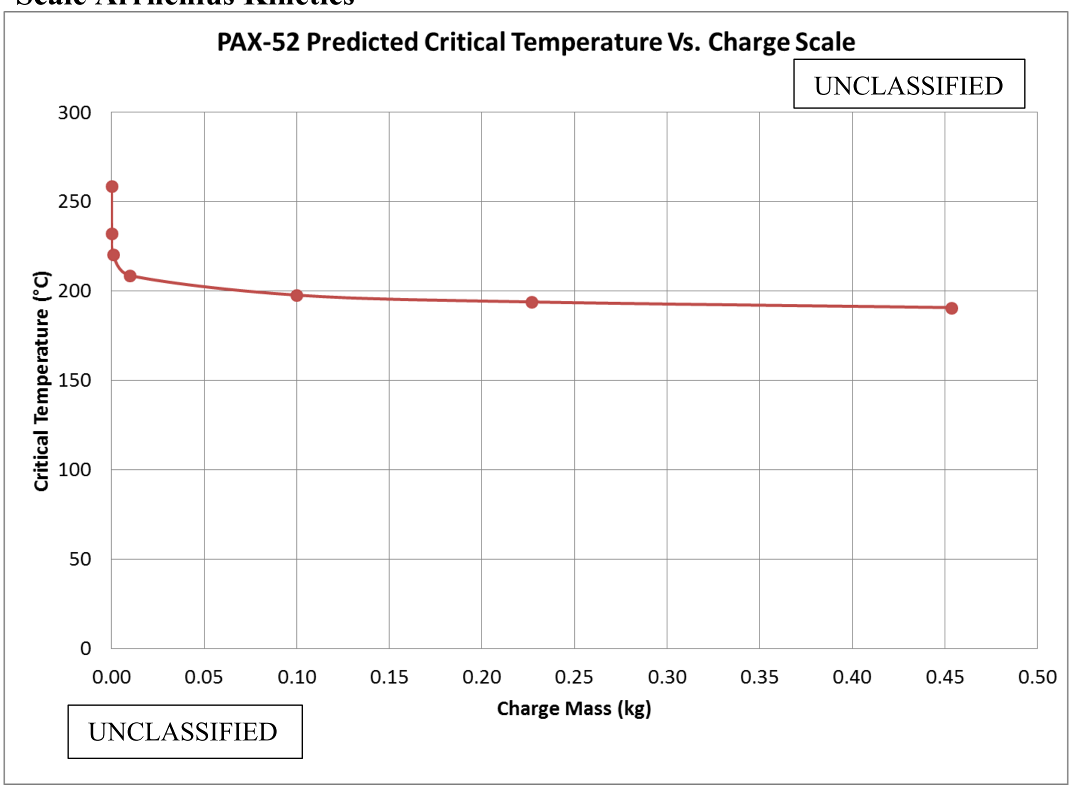




2. **(U) Decomposition Kinetics for PAX-52**



3. **(U) Predicted Critical Temperature as a Function of Charge Scale Based on Small Scale Arrhenius Kinetics**



11.6 (U) APPENDIX G: USER EVALUATION: EXPLOSIVES ORDNANCE DISPOSAL (EOD)

(U) Purpose: To have EOD technicians use PAX-52 in several test configurations and get their qualitative feedback.

(U) Reference: EOD Test Plan. (Kase, 2016) (Not available for distribution.)

(U) Reference: Report of Test "PAX-52 USER HANDLING AND EMPLOYMENT ASSESSMENT." 05-18-H-6005.00. MAY 2018. (Not available for distribution.)

11.7 (U) APPENDIX H: COST ASSESSMENT INPUT DATA

(U) Fixed Costs:

(U) Processing equipment:

1. Twin screw extruder to run 30-100 lb/hour (size 2" diameter) with hydraulic drive, with split barrels and die mechanisms, proximity sensors, 4 pressure transducers, 10 thermo- couples, torque sensors on both screws, heating/cooling capability, separate for the die and the barrel of the extruder, five zones for temperature control for the barrel- length over the diameter of the extruder = 15. The mode is to be fully- intermeshing co-rotating twin screw.
2. Screw parts to slip onto a hexagonal shaft, sufficient to cover flexibly the 15 length/diameter ratio (20 sets of kneading disks- lenticular elements, 40 sets of fully-flighted right and left handed screws, single and double channels).
3. A rectangular slit die is to be designed and fabricated.
4. Installation cost is estimated and provided.

(U) Auxiliaries:

1. Three silicone oil heating/circulation units for temperature control.
2. Four loss-in weight feeders for solids (20-60lb/hour), three need to be for energetics feeding and one for the inert feed.
3. One loss in weight feeder for silicone polymer.
4. One vacuum pump.
5. Take-off equipment to handle the extruded strands.
6. Cutting equipment
7. Tray dryer for HMX
8. Two bins/hoppers to handle larger quantities of HMX to be fed into the loss-in-weight feeders
9. Feeding equipment to transfer HMX from bin/hopper to the loss-in- weight feeders.

(U) Safety equipment:

1. A water deluge system to be placed above the extruder.
2. Tank for water holding for the deluge
3. 2 explosion proof video cameras and one thermal imaging camera
4. Uninterrupted power supply

(U) Process and product quality control:

1. An Allen-Bradley PLC with computer board and boards for 50 sensor inputs and 20 output signals.
2. Allen-Bradley software for the PLC.
3. A machine human interface- PC with WonderWare software.
4. A Rigaku mini-flex x-ray diffraction unit for the determination of the mass fraction of HMX and degree of mixing.
5. A squeeze flow rheometer for testing the rheological behavior of the extrudate.
6. A melt flow indexer for the silicone oil quality control (measurement of viscosity.)
7. Electronic balances (two) – to independently measure the flow rate

(U) Buildings:

1. One facility to house the extruder and the energetic materials (Class 1 facility)
2. A second facility to house the hydraulic drive and the operator's station with a tunnel in between for the cables and oil pipes.

(U) Operating costs:

(U) Labor:
1. Stipend and fringe benefits of two engineers.
2. Stipend and fringe benefits of two technician operators.
3. Consultant costs for occasional software and hardware modifications

(U) Utilities:
1. Electricity
2. Inert gases (maybe necessary for dryer operation and for the hoppers feeding HMX)
3. Water

(U) Waste disposal:

(U) Materials and supplies:
1. Safety gloves
2. Goggles
3. Conductive safety shoes
4. Conductive mats
5. Coveralls
6. Helmets

(U) Raw material costs:
1. HMX
2. Silicone oil
3. Taggant