



#### DEVELOPMENT & OPTIMIZATION OF A PRODUCTION METHOD FOR MANUFACTURING PAX-41

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#### **Briefing Objectives**

- Background
- Program Objectives
- Engineering Study
- Confirmation Batches
- Conclusion





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#### Background

- PAX-41 Formulation
  - Developed at Picatinny Arsenal as a derivative of PAX-21
  - Melt-pour formulation containing DNAN, RDX & MNA
  - Designed with a small critical diameter tailored for optimum performance (Miniature Grenade) in the SPIDER Area Denial System
  - OSI was contracted to manufacture PAX-41 in full production scale



#### **Program Objectives**

- OSI to establish a reproducible manufacturing process of PAX-41 at HSAAP under optimum operating conditions
  - Design of Experiment (DOE) approach used to optimize processing parameters
  - Conduct experiments in production facility and compare results against specification requirements
  - Manufacture confirmation batches using optimized processing parameters
- Produce SOP and Manufacturing Instruction for the manufacture of PAX-41



Holston Army Ammunition Plant



## PAX-41 Manufacturing Process

#### Load Ingredients (DNAN, RDX etc.)



Molten PAX-41



Molten PAX-41

3. Cool/solidify and break-up



PAX-41 flakes





Images shown are from the PAX-21 production

## **Engineering Study**

- Test Plan Development (DOE)
  - 3 processing parameters studied

Batch Size	Small		Standard		
Agitator Speed	Low		High		
Residence Time	Short	Medium		Long	

- Other processing parameters remained constant
- Mix temperature = 100°C
- Spec. compliance testing carried out on each sample
  - Composition
  - Melting Point and Exotherm Onset
  - Efflux Viscosity
  - Moisture
  - Flake Size & Density
  - Impact and Friction Sensitivity
  - Shock sensitivity (LSGT)





# **Engineering Study**

• Engineering Study Test Plan



Batch Sample ID	Residence Time (minutes)	Batch size (LBS)	Agitator Speed (RPM)		
1	High				
1A	Medium	Std	High		
1B	Low				
2	High				
2A	Medium Small		Low		
2B	Low				
3	High				
3A	Medium	Std	Low		
3B	Low				
4	High		High		
4A	Medium	Small			
4B	Low				

Dip samples were taken at various durations after the addition of all ingredients

# **Engineering Study**

- Customer Purchase Description requirement for PAX-41
  - (Purchase Description Document AR-PD-138, dated 6/21/2005)



AR-PD-138 Requirements	Value			
Composition Analysis Total RDX DNAN MNA	Undisclosed			
Melting Point (°C) DSC method, @ 20°C/min	$90\pm5$			
Exotherm Onset Temp (°C) DSC method, @ 20°C/min	$232\pm12$			
Viscosity - Brookfield, kP (max) @ 205°F ± 5°F - Efflux Viscosity, sec (max) @ 205°F	$5.5\pm0.5$			
Moisture (% max)	0.04			
Flake Thickness, inches (max)	0.25			
Flake Density (gm/cm³)	$1.71 \pm 0.5$			
Impact Sensitivity - ERL, Type 2, cm (min) - ABL Method, cm (min)	49 36			
Friction Sensitivity ABL Method, Ib @ 8 fps	680 ± 240			
NOL Large Scale Gap Test, cards 50% max	185			

## Engineering Study Result

- 1. Batch Size (Small / Standard LBS)
  - For small batch sizes, efflux viscosities were relatively higher (both agitator speeds)
  - For small batch sizes, and high agitator speed, the efflux viscosities were above PD requirement (> 6.0 seconds) by a large margin
    - Not sure whether the batch size is the contributing factor!
  - LSGT > 185 cards for both batch sizes
  - No other significant difference found
  - The standard batch size is more economical for large scale production
- 2. Agitator Speed (low / high RPM)
  - At the standard batch size, the efflux viscosities were relatively lower at a higher agitator speed (3.2 second @ high speed; 5.1 seconds @ low speed)
  - No other significant difference found
  - Low speed is preferred as the higher agitator speed may lead to spillage out of kettle (safety) & higher energy consumption (cost)

## Engineering Study Result

- 3. Residence Time (Low / medium / high mins.)
  - At low rpm agitator speed:
    - the efflux viscosity failed to meet PD requirement of < 6 seconds at low residence time (small and standard batch size)
    - The efflux viscosity of the standard size batch material was still out of spec even after the medium mixing time
    - After mixing for the high residence time, both batches met PD requirement for efflux viscosity
  - Clearly, the longer residence time is required to ensure the efflux viscosity reaches the required level

#### OVERALL FINDINGS

• From the Engineering Study, it was found that the optimum processing parameters in terms of batch size, agitator speed and residence time are standard batch size, low agitator speed and high residence time respectively

## **Additional Findings**

- Large Scale Gap Test Requirement
  - The original LSGT 50% Card Gap requirement was 185 cards maximum
  - Such level was never reached throughout the engineering study
  - After consulting with Picatinny Arsenal, it was agreed that the LSGT 50% Card Gap for PAX-41 shall be amended to 210 cards maximum
- Exotherm Onset Temperature Requirement
  - Original requirement was 232 ± 12°C
  - Exotherm onset temperature analyzed throughout the engineering study ~ 225°C
  - The requirement was lowered to 225 ± 12°C
- Moisture Content Requirement
  - Original moisture content requirement was 0.04% maximum
  - Results obtained from the engineering study ~ 0.03-0.04%
  - The moisture content requirement was raised to 0.10%
- The above changes have been included in an Engineering Change Proposal to the PAX-41 purchase description document

#### **Confirmation Batches**

- 11 batches of PAX-41 were produced adapting the findings from the Engineering Study
  - Standard batch size, low agitator speed, long residence / mixing time
- All 11 batches met all the purchase description requirements
  - LSGT and Friction tested in the first 4 batches
  - High level of consistency achieved

Batch No	Total RDX	DNAN	MNA	Flake Size	Density	Moisture	Melting Point	Exotherm	lmpact, min	Friction Sensitivity	LSGT	Viscosity
Max		undisclosed		0.25	2.21	0.10	95	238	n∕a	920	210	6.0
Min		undisclosed		n/a	1.21	n/a	85	213	36	640	n/a	n/a
11	PASS	PASS	PASS	0.12	1.60	0.02	88	215	60	OK	190.5	3.4
12	PASS	PASS	PASS	0.11	1.60	0.03	88	214	60	OK	191.5	3.9
13	PASS	PASS	PASS	0.11	1.60	0.02	n/a	n/a	51	OK	192.5	3.2
14	PASS	PASS	PASS	0.10	1.61	0.02	88	215	57	OK	190.5	3.3
15	PASS	PASS	PASS	0.11	1.61	0.02	88	213	53	n/a	n/a	3.2
16	PASS	PASS	PASS	0.11	1.61	0.04	89	213	52	n/a	n/a	3.0
17	PASS	PASS	PASS	0.10	1.60	0.02	88	213	46	n/a	n/a	3.1
18	PASS	PASS	PASS	0.11	1.60	0.01	89	213	56	n/a	n/a	3.1
19	PASS	PASS	PASS	0.11	1.61	0.02	88	213	51	n/a	n/a	3.2
20	PASS	PASS	PASS	0.11	1.61	0.02	88	213	57	n/a	n/a	3.0
21	PASS	PASS	PASS	0.11	1.61	0.02	89	214	47	n/a	n/a	3.0

#### Conclusion

- A robust production method for the manufacturing of PAX-41 has been developed and optimized using a DOE approach
- Optimized processing parameters include batch size, agitator speed and residence time
- 11 large-scale production batches of PAX-41 produced following the optimized production method; all purchase description requirements were met
- Analytical data/results obtained from the DOE were used to modify the PAX-41 purchase description requirements