



# Large Scale Manufacture of Granular IMX-104 Melt Pour Explosives

## NDIA Insensitive Munitions & Energetic Materials Technology Symposium 2013

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# Topics of Discussion

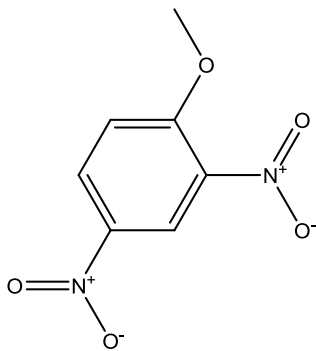
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- Insensitive Melt Pour Explosives
- Laboratory Development Work
- Intermediate Scale Up of Baseline Process
- Large Scale Manufacture
- Conclusions and Future Work

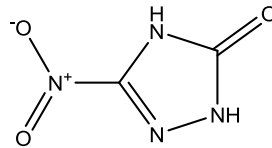


# Insensitive Melt Pour Explosives

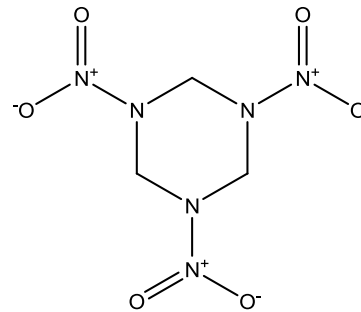
- Provide Insensitive replacements for TNT based explosives with similar performance
  - IMX-101 – Insensitive replacement with comparable performance to TNT
  - IMX-104 – Insensitive replacement with comparable performance to Composition B
  - Both currently qualified for use as main fill explosives
- Contain non-traditional ingredients



DNAN



NTO



RDX



# Laboratory Development Work – Overview

- Established process developed using Alternate Fluid
  - Alternate Fluid - A fluorinated hydrocarbon which has similar properties to water and can be used in a place of water in multiple applications
  - Successfully used in the manufacture of aluminized PBX's at HSAAP
  - Utilized laboratory coating still and standard slurry coating techniques
  - Varied agitation rate to observe changes in particle size distribution
    - Ranged from 37.5% of maximum to 100% of maximum
- Analysis of resulting batches is promising
  - Composition, and thermal properties unchanged; bulk density is high



**10 Liter Lab Vessel**



**500 Gallon Reactor**



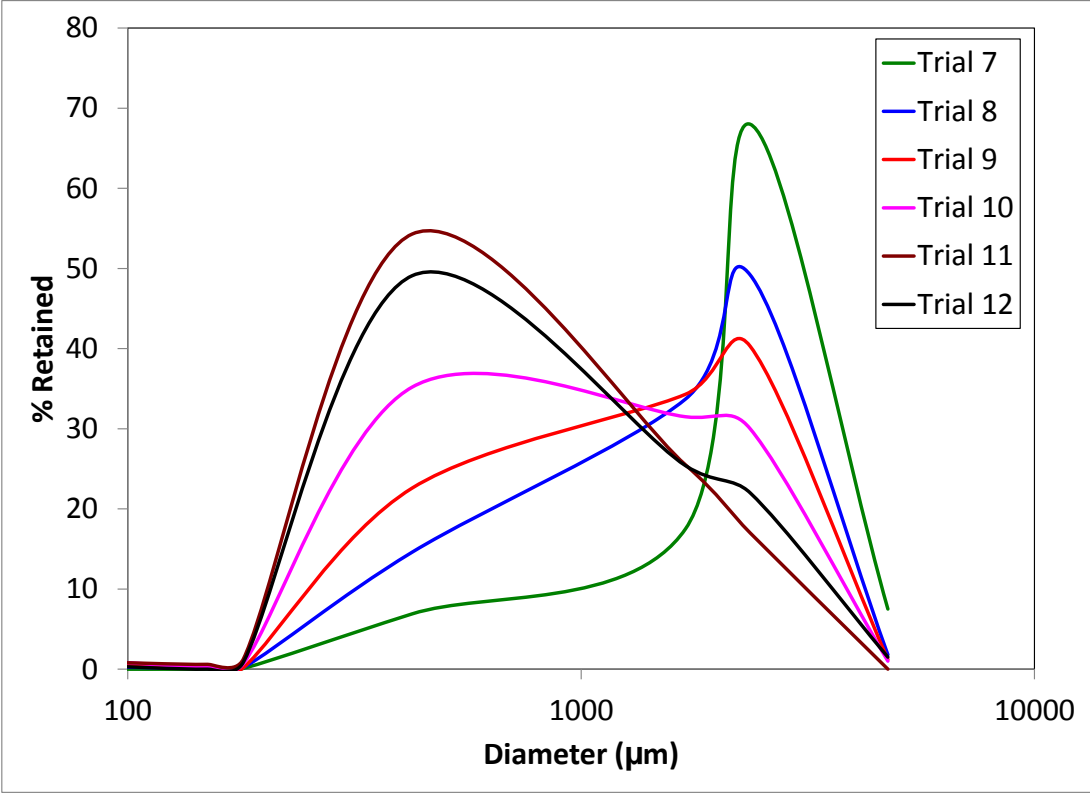
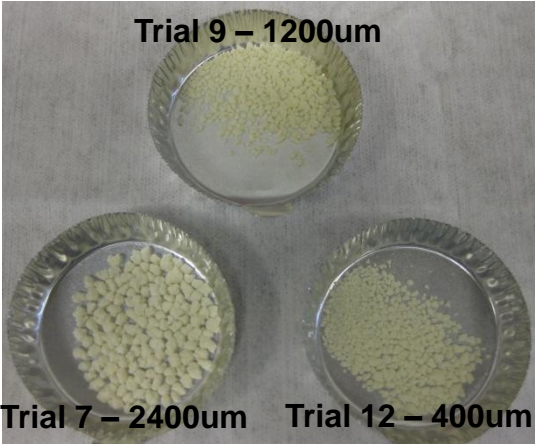
# Laboratory Development Work – Analysis

Batch ID		Trial 7	Trial 8	Trial 9	Trial 10	Trial 11	Trial 12
Agitation Rate (% of Maximum)		37.5	50	62.5	75	87.5	100
Yield (%)		59.78	61.02	68.30	67.58	75.02	70.38
DNAN		Nominal	Nominal	Nominal	Nominal	Nominal	Nominal
RDX		Nominal	Nominal	Nominal	Nominal	Nominal	Nominal
NTO		Nominal	Nominal	Nominal	Nominal	Nominal	Nominal
Melting Point (°C)		95.5	96.4	96.6	94.9	92.0	91.7
Exothermic Onset (°C)		203	207	210	219	204	220
Bulk Density (g/cc)		0.926	0.885	0.962	0.926	0.962	0.961
Screens (% Pass)	4	92.5	98.2	98.8	99.0	100	98.5
	8	24.5	49.0	58.5	69.0	83.0	76.5
	12	7.0	15.3	24.2	37.5	57.5	51.1
	40	0.1	0.7	1.6	2.3	3.2	2.0
	80	0.0	0.5	1.4	1.6	1.9	1.1
	100	0.0	0.4	1.2	1.4	1.3	1.1
	200	0.0	0.1	0.4	0.3	0.4	0.6
	325	0.0	0.0	0.0	0.0	0.0	0.3



# Laboratory Development Work – Granulation

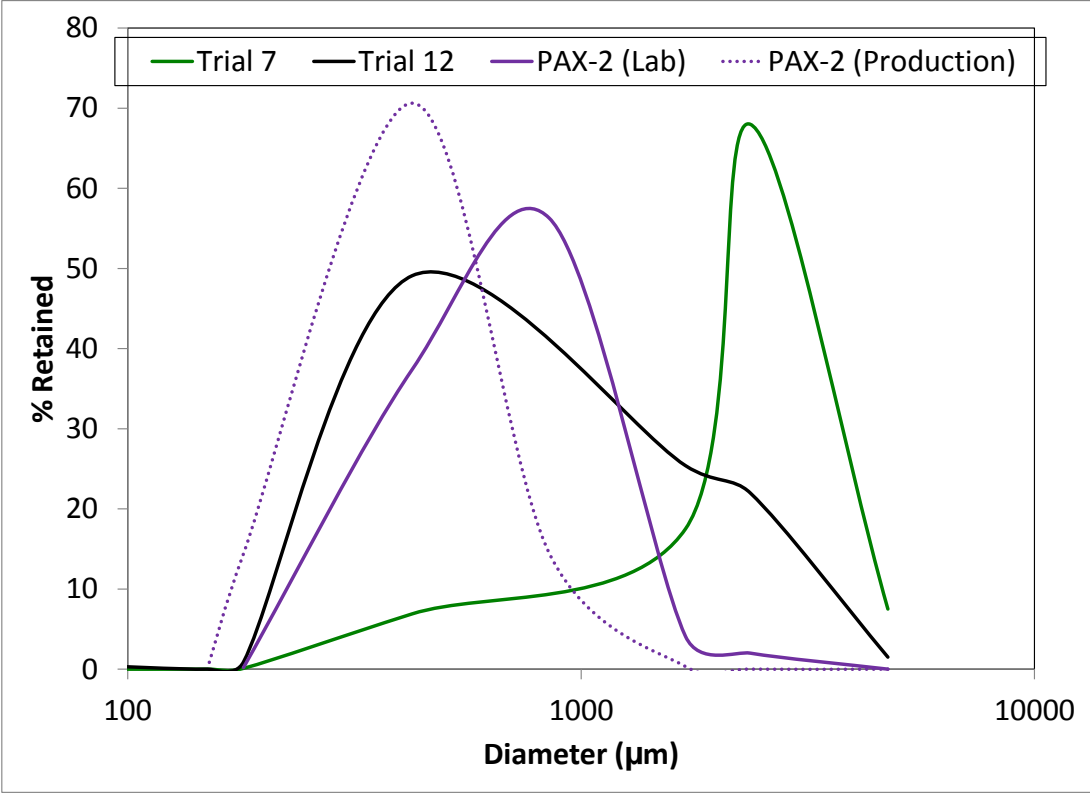
- Effect on granulation observed with changes in agitation
- Slower agitation rates result in larger particle sizes
  - Trial 7 (Slow) – 2400 $\mu\text{m}$
  - Trial 9 (Medium) – 1200 $\mu\text{m}$
  - Trial 12 (Fast) – 400 $\mu\text{m}$
- Further work is required to optimize granulation





# Laboratory Development Work – Granulation Comparison

- Comparison of Granular IMX-104 to typical granulated product at HSAAP
- Largest and Smallest distributions compared to PAX-2 (Laboratory and Production)
  - 37.5% – larger than PAX-2
  - 100% – similar to PAX-2
- Promising result
  - Can make granular melt pour at similar particle sizes to other granular explosives
- Intermediate Scale Conditions Selected from these results





# Intermediate Scale Up of Baseline Process – Overview

- Baseline process from trials was selected and scaled to a 50 pound scale
  - Tested for composition, thermal properties, bulk density, and granulation
  - Also tested for shock sensitivity via Large Scale Gap Test (LSGT)
- Analysis yielded promising results
  - Met specification for composition and thermal properties
  - Bulk density is high (greater than 0.9 g/cc)
  - Variation in granulation from small scale is negligible
  - Approximately 98% yield of product







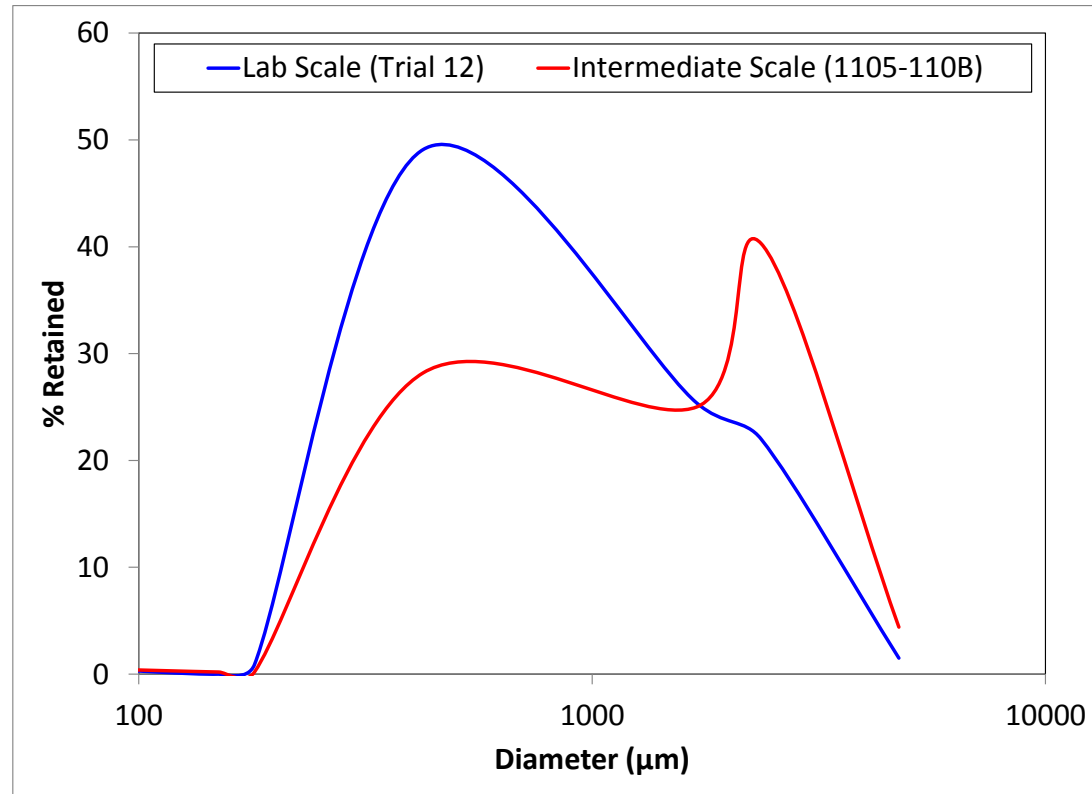
# Intermediate Scale Up of Baseline Process – Analysis

Batch ID	IMX-104 Specification		Trial 12	1105-110B
	Minimum	Maximum		
Yield (%)	70	100	70.38	98.41
DNAN	Nominal	Nominal	Nominal	Nominal
RDX	Nominal	Nominal	Nominal	Nominal
NTO	Nominal	Nominal	Nominal	Nominal
Melting Point (°C)	86	96	91.7	92.2
Exothermic Onset (°C)	193	217	217	200
Bulk Density (g/cc)	0.865		0.961	0.926
Impact (cm)	120			>200
Friction (N)	Exceed RDX CL5			231.7
Flowdex	10	20		16
Moisture (%)		0.2		0.0699
VTS (ml/g)		2		0.0846
Screens (% Pass)	4		98.5	95.6
	8		76.5	55.3
	12		51.1	30.3
	40		2.0	2.1
	80		1.1	1.9
	100		1.1	1.7
	200		0.6	1.2
	325		0.3	0.7



# Intermediate Scale Up of Baseline Process – Granulation

- Semi Bi-Modal distribution for larger scale batch
  - Still broad distribution and similar to baseline lab scale batch
- Still usable and processed well
  - Average particle size is ~1100 $\mu\text{m}$





# Intermediate Scale Up of Baseline Process – LSGT

- Material from scale up trials tested in LSGT configuration against poured charges of standard IMX-104

Sample ID	IMX-104 (Flake)	IMX-104 (Granular)		
		Intermediate Scale		Lab Scale Baseline
Manufacture Process	Large Scale (Melt cast)	Intermediate Scale		Lab Scale Baseline
Charge Density (%TMD)	98	98	95	95
50% Point (card gap)	125	126	158	156
Pressure (kbar)	48.8	48.5	35.4	36.3

- No difference in sensitivity observed between melt cast and granular charges
- No difference in sensitivity observed between intermediate scale and lab scale material
- Composition B (201 – 220 cards; 16.9 – 20.5 kbar at 99%TMD)



# Large Scale Manufacture – Overview

- Process scaled to a 500 pound scale
  - Tested for multiple properties
  - Final product to be delivered to customer
- Required Properties
  - Composition, thermal properties, and moisture
- Informational Properties
  - Impact, friction, flowdex, VTS, granulation, and bulk density



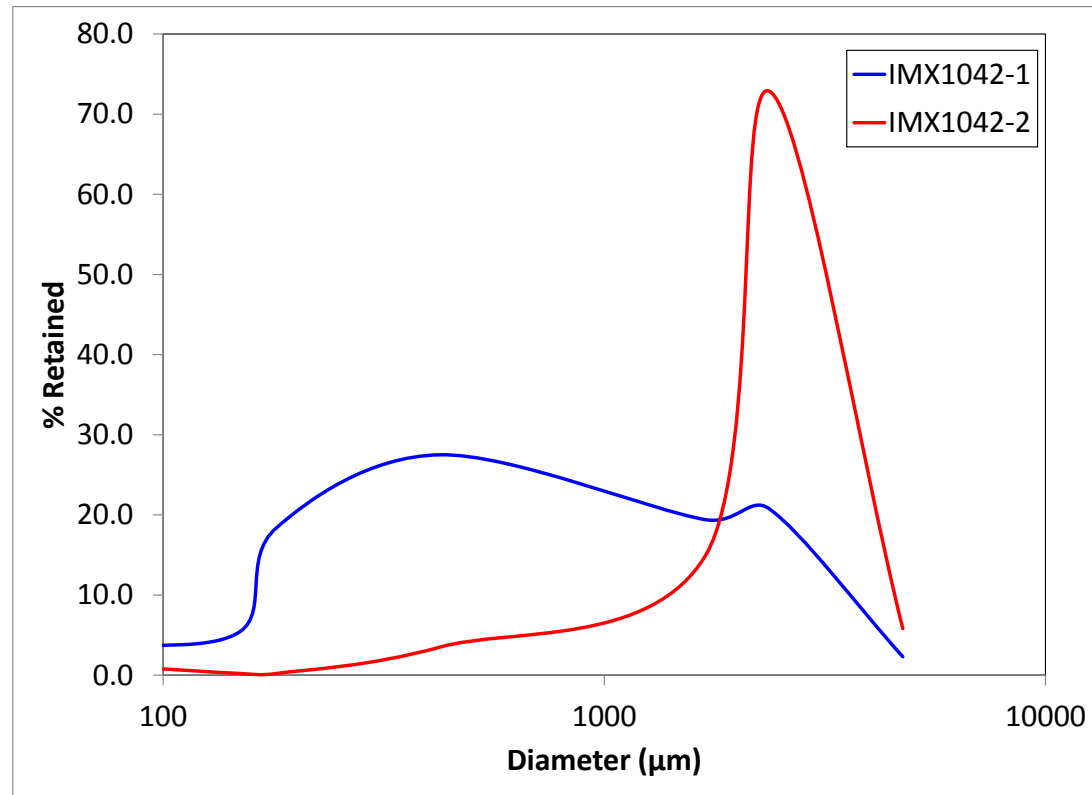
# Large Scale Manufacture – Initial Run

Batch ID	IMX-104 Specification		IMX1042-1	IMX1042-2
	Minimum	Maximum		
DNAN	Nominal	Nominal	High	High
RDX	Nominal	Nominal	Low	Nominal
NTO	Nominal	Nominal	Low	Low
Melting Point (°C)	86	96	90.3	90.2
Exothermic Onset (°C)	193	217	198	196
Bulk Density (g/cc)	0.865		0.99	1.00
Impact (cm)	120		>200	>200
Friction (N)	Exceed RDX CL5		156.3	242.2
	RDX CL5		182.8	182.8
Flowdex	10	20	32	18
Moisture (%)		0.2	0.019	0.006
VTS (ml/g)		2	0.08	0.04
Screens (% Pass)	4		97.7	94.2
	8		76.9	21.3
	12		57.5	6.3
	40		30.0	2.8
	80		11.7	2.6
	100		6.2	2.4
	200		2.7	1.3
	325		0.4	0.3



# Large Scale Manufacture – Initial Run Granulation

- IMX1042-1
  - Very fine and powder like
  - Conditions adjusted for second batch to make material coarser
  - Analysis of powder resulted in high NTO values
- IMX1042-2
  - Better granulation with narrow distribution
  - Coarser than intermediate batch
  - However still did not meet composition
- Water present in DNAN (~15%)
  - NTO lost in water as it is removed
  - High ratio of fluid to water during removal step (~10:1)





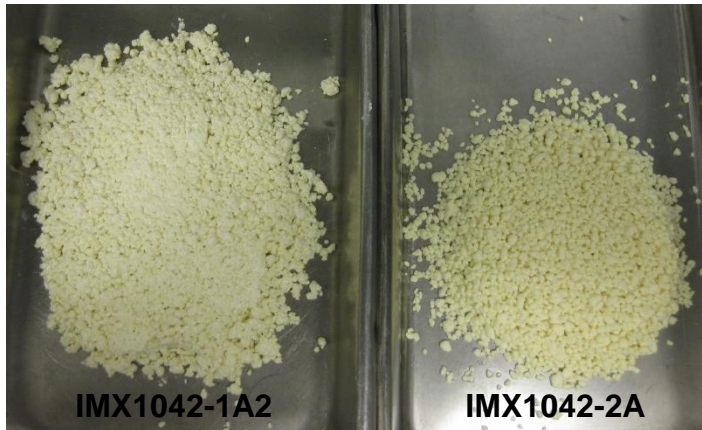
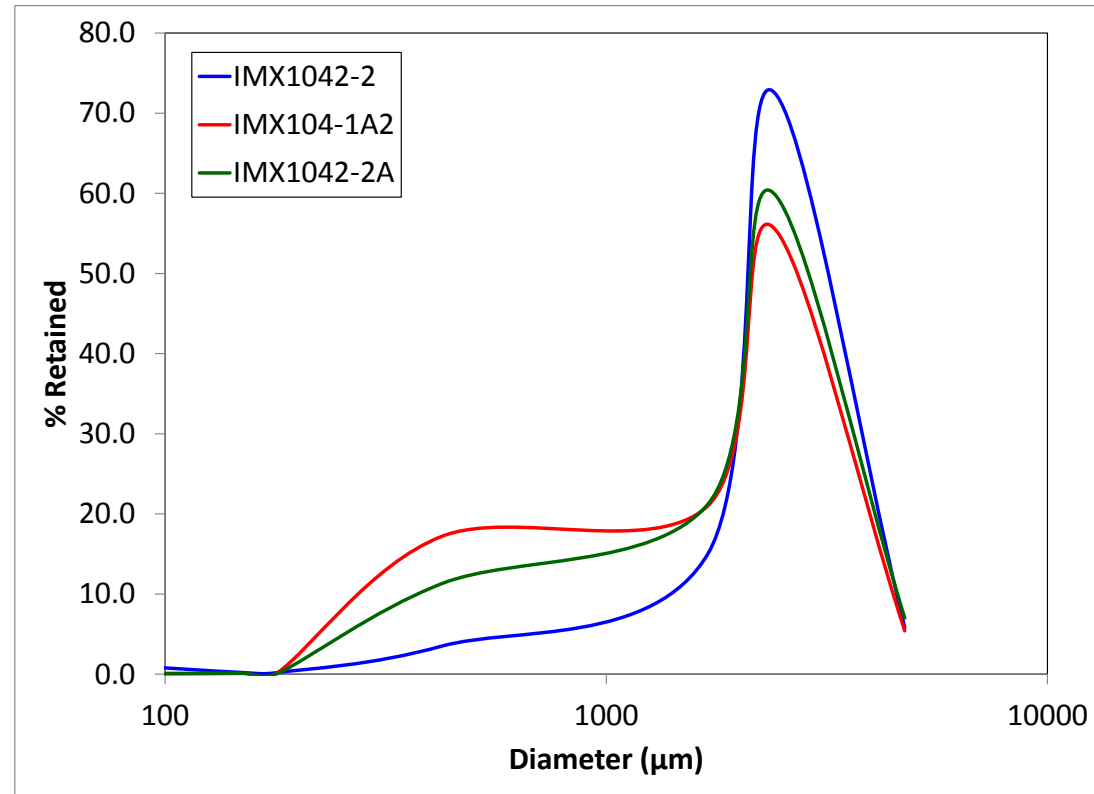
# Large Scale Manufacture – Rework

Batch ID	IMX-104 Specification		IMX1042-1A2	IMX1042-2A
	Minimum	Maximum		
DNAN	Nominal	Nominal	Nominal	Nominal
RDX	Nominal	Nominal	Nominal	Nominal
NTO	Nominal	Nominal	Nominal	Nominal
Melting Point (°C)	86	96	90.4	90.5
Exothermic Onset (°C)	193	217	206	204
Bulk Density (g/cc)	0.865		1.02	1.02
Impact (cm)	120		>200	>200
Friction (N)	Exceed RDX CL5		254.6	209.1
	RDX CL5		164.5	164.5
Flowdex	10	20	18	20
Moisture (%)		0.2	0.03	0.04
VTS (ml/g)		2	0.07	0.09
Screens (% Pass)	4		94.6	93.0
	8		38.6	32.7
	12		17.6	11.5
	40		0.4	0.2
	80		0.2	0.1
	100		0.1	0.0
	200		0.0	0.0
	325		0.0	0.0



# Large Scale Manufacture – Rework Granulation

- IMX1042-1A2
  - Now similar to IMX1042-2
  - Meets composition requirement
- IMX1042-2A
  - No major change to granulation
  - Meets composition requirement
- Material delivered for further evaluation and testing







## Conclusions and Future Work

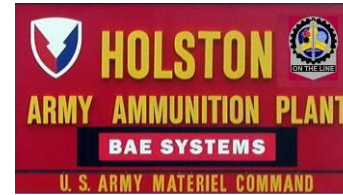
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- Process using alternate fluid has been successful in manufacturing granular versions of IMX-104
  - Laboratory process successfully scaled to 50 pound increment
    - Meet specification for composition with no change in thermal properties
    - Granulation can be controlled to obtain useable material
    - LSGT value not significantly effected by granulation process
  - Process further scaled to 500 pound batch size
    - Meets specification for composition with no change in thermal properties
    - Evidence that granulation can be controlled but requires additional work to optimize
    - Utilizes existing manufacturing infrastructure at HSAAP
    - Additional work is required to determine best method for removing water and minimizing NTO loss



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# Questions?

