Development and Evaluation of Granular Insensitive Melt Pour Explosives

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Topics of Discussion

- Insensitive Melt Pour Explosives
- Thermal Evaluations of DNAN in Water
- Water Based Slurry Coating Trials
- Alternate Fluid Based Slurry Trials
- Pressing Evaluation
- Scale Up of Baseline Process
- Performance Testing
- Conclusions and Future Work

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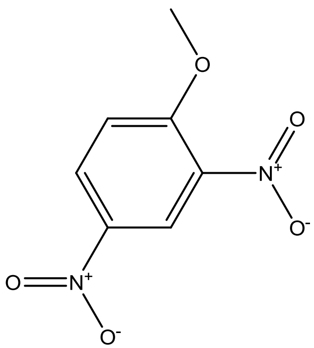
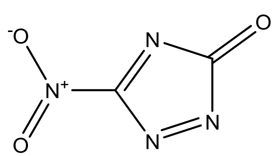
Insensitive Melt Pour Explosives

- 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

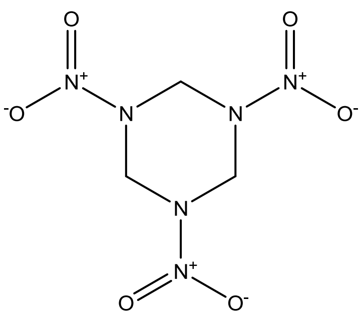
- Contain non-traditional ingredients



2,4-dinitroanisole
(DNAN)

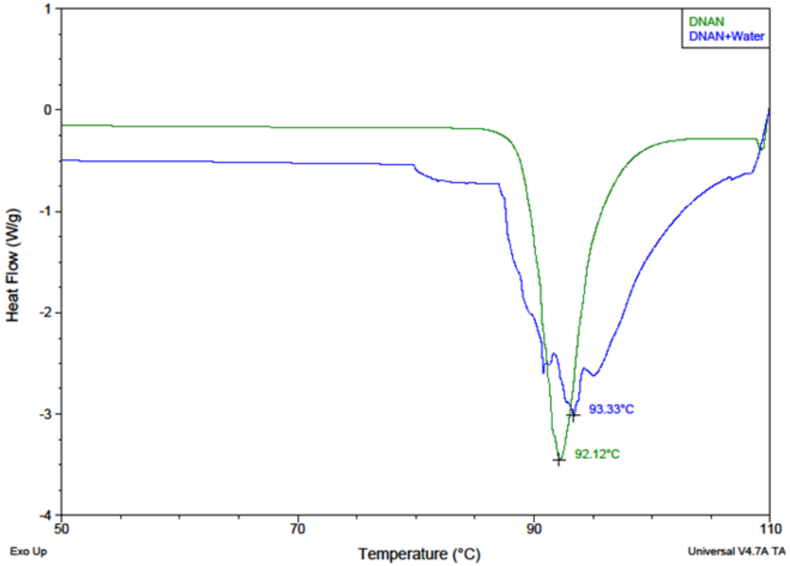


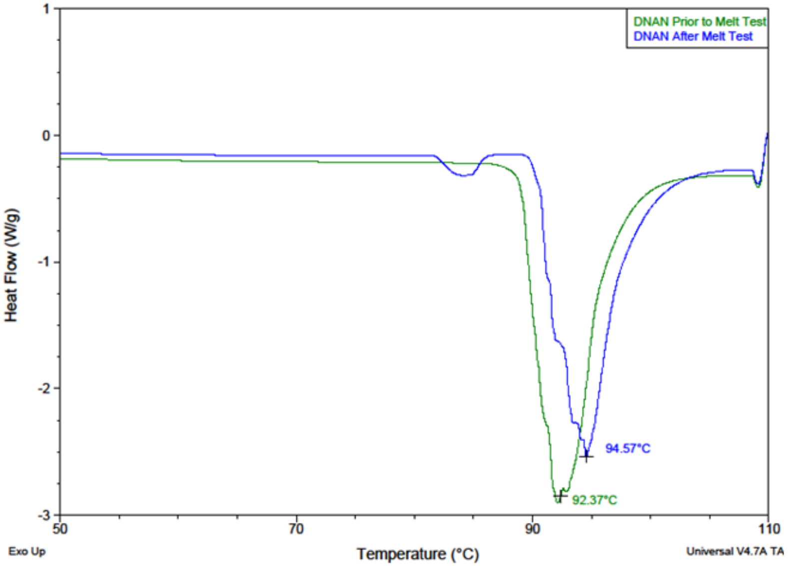
3-nitro-1,2,4-triazol-5-one
(NTO)



RDX

Thermal Evaluations of DNAN in Water





- DNAN and 1:1 DNAN:Water analyzed via DSC to compare melting point
- No effect seen other than a broadening of the melt peak

- DNAN melted in water and then allowed to solidify in granular form
- Analyzed using DSC for melting point comparisons
- No effect seen on melt temperature or peak

Water Based Slurry Coating Trials

Percent Deviation from Target – Water Based Trials				
Ingredient	**Trial 1**	**Trial 2**	**Trial 3**	**Trial 4**
DNAN	**-79.49**	**85.17**	**89.27**	**104.42**
RDX	**-75.16**	**101.96**	**91.50**	**105.88**
NTO	**74.34**	**-76.42**	**-80.94**	**-93.77**

Percent Deviation from Target – Screen Fractions				
Batch ID	**Trial 3**		**Trial 4**	
Screen	**#8**	**#325**	**#8**	**#325**
DNAN	**99.05**	**-0.03**	**104.73**	**-65.30**
RDX	**103.27**	**-24.84**	**113.07**	**-64.70**
NTO	**-92.08**	**-2.64**	**-98.11**	**57.36**

- Utilized excess NTO or NTO Saturated water to overcome solubility of NTO in water
- Trial 1 – DNAN stuck to vessel – High NTO
- Trial 2 – Segregated materials – Low NTO
- Trial 3 – Segregated materials – Low NTO
- Trial 4 – Segregated materials – Low NTO
- NTO re-crystallizes after DNAN solidification in granules

- Screen fractions confirm segregation of material
- #8 – Coarse – High DNAN\Low NTO
- #325 – Fine – Low DNAN\High NTO
- Due to delayed re-crystallization of dissolved NTO water based trials were put on hold in favor of alternate processes

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Alternate Fluid A Based Slurry Coating Trials – Overview

- Alternate Fluid A is 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
  - Allows for use of existing manufacturing infrastructure for scale up

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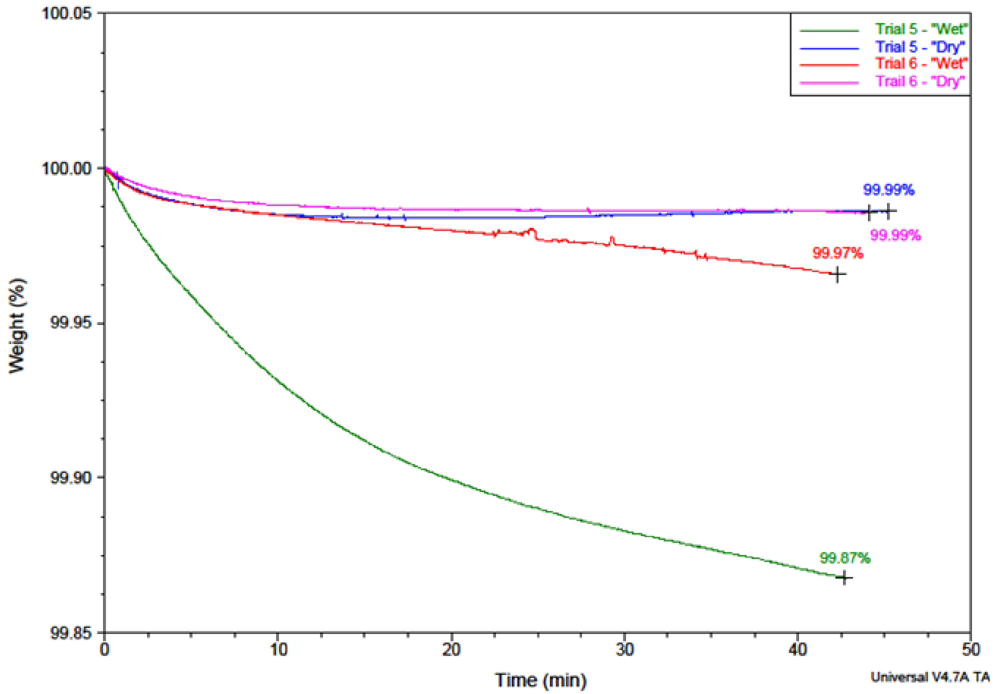
Alternate Fluid A Based Slurry Coating Trials – Analysis

- Composition compared using percent deviation from target
  - DNAN allowed ± 6.31% deviation
  - RDX allowed ± 12.74% deviation
  - NTO allowed ± 4.65% deviation
- Trial 5 and Trial 6 have acceptable composition, melting point, and exothermic onset values
  - Comparable to standard IMX-104
- Bulk Density greater than 0.9 g/cc
- Granulation is consistent between Trial 5 and Trail 6
- However poor yield due to low boiling point of Alternate Fluid A

Batch ID		Production IMX-104	Trial 5	Trial 6
DNAN (% Deviation from target)		5.04	-2.84	-0.95
RDX (% Deviation from target)		1.31	-3.27	-3.26
NTO (% Deviation from target)		-2.26	2.45	1.32
Melting Point (ºC)		92.1	93.4	92.9
Exothermic Onset (ºC)		208	210	207
Bulk Density (g/cc)			0.926	0.962
Screens (% Pass)	4		100	98.3
	8		76.7	74.8
	12		37.9	47.7
	40		0.8	5.6
	80		0.8	4.1
	100		0.6	3.7
	200		0.3	2.3
	325		0.1	1.3

Alternate Fluid A Based Slurry Coating Trials – TGA Scan

- Isothermal TGA scan performed on "wet" and "dry" materials
- Very little volatiles in "wet" materials
  - Trial 5 – 0.16%
  - Trial 6 – 0.03%
- Material is not really wet in traditional sense



Alternate Fluid A vs. Alternate Fluid B

Property	Units	Alternate Fluid A	Alternate Fluid B
Appearance		Clear; Colorless	Clear; Colorless
Average Molecular Weight	g/mole	438	521
Boiling Point	ºC	101	128
Vapor Pressure	Kpa	3.870	1.440
Liquid Density	g/ml	1.77	1.82
Absolute Viscosity	Cp		1.4

- Alternate Fluid A cannot exceed 92ºC which does not fully melt DNAN
- Alternate Fluid B selected due to higher literature value for boiling point
  - Similar in other physical properties

Alternate Fluid B Based Slurry Coating Trials – Overview

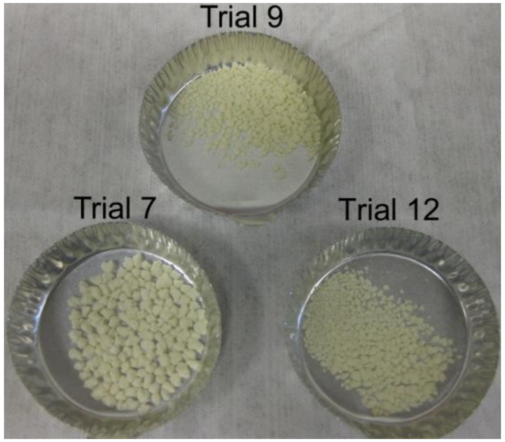
- Used established process from Alternate Fluid A based trials
  - Higher boiling point of Alternate Fluid B resulted in improved yield values
- Varied agitation rate to observe changes in particle size distribution
  - Ranged from 37.5% of maximum to 100% of maximum
- Analysis of resulting batches is promising
  - Meet specification for composition and thermal properties
  - Bulk density is high
  - Granulation did change with agitation rate

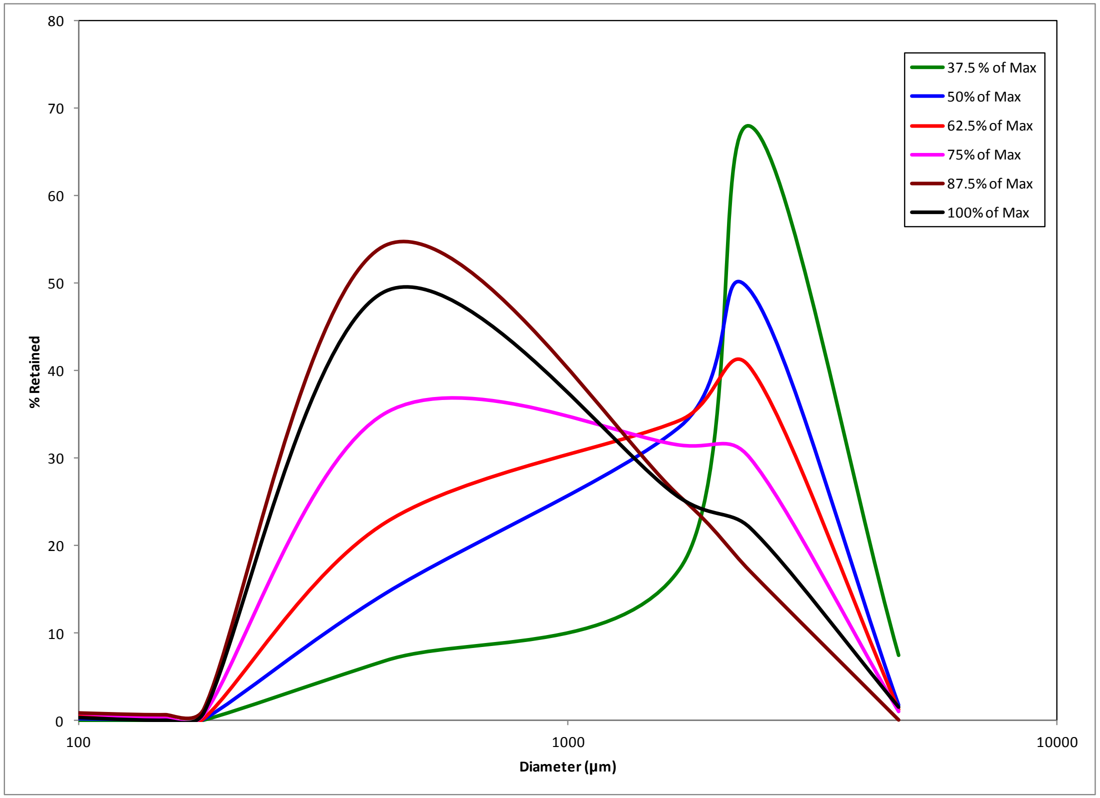
Alternate Fluid B Based Slurry Coating Trials – Analysis

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 (% Deviation from target)		-1.26	-3.78	-1.26	-1.89	-4.10	-3.78
RDX (% Deviation from target)		-2.61	-11.76	-8.49	-5.22	-3.92	-6.53
NTO (% Deviation from target)		-0.75	4.53	2.45	0.75	2.64	3.39
Melting Point (ºC)		95.5	96.4	96.6	94.9	92.0	91.7
Exothermic Onset (ºC)		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

Alternate Fluid B Based Slurry Coating Trials – Granulation

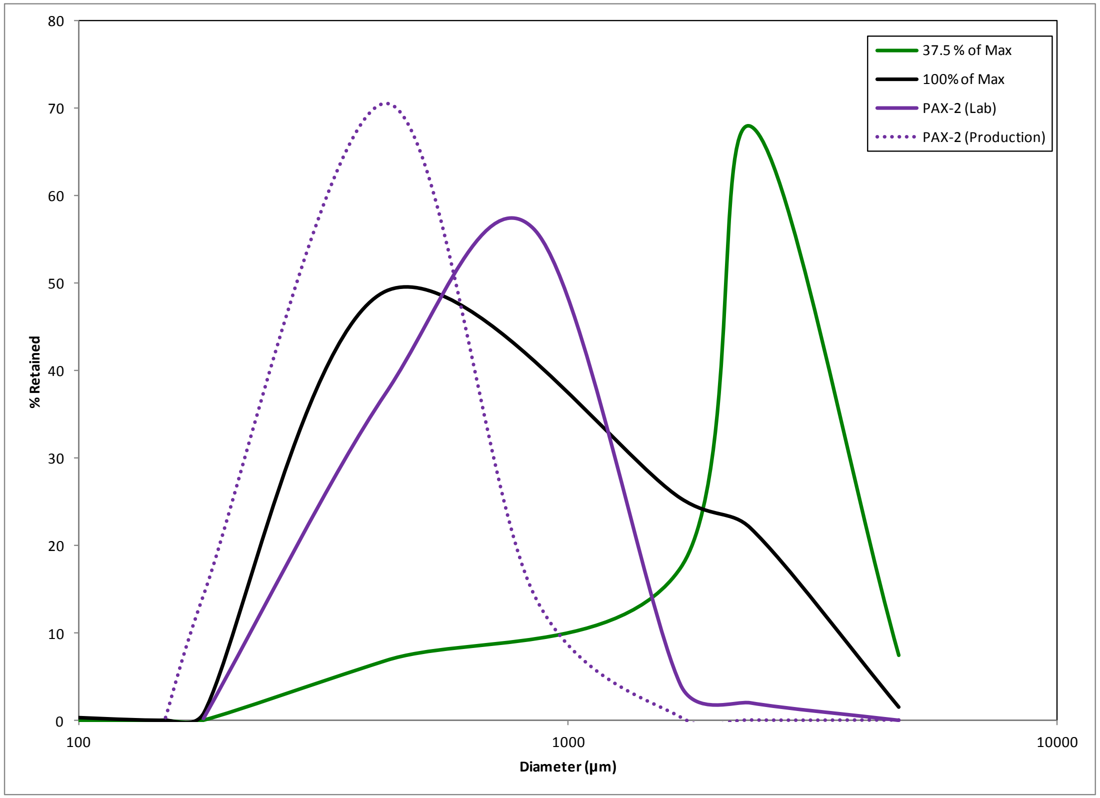
- Effect on granulation is observed with changes in agitation
- Slower agitation rates result in larger particle sizes
  - 37.5% – 2400µm
  - 100% – 400µm
- Distributions are still very broad
  - Further work is required to optimize granulation





Alternate Fluid B Based Slurry Coating Trials – Granulation

- 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



Pressing Evaluation – Overview

- Performed a small screening effects designed experiment (DOE)
  - Two different diameter die sets
    - 1” and 1.5”
  - Varied pressing force and sample mass
    - 3000 pounds to 20000 pounds
    - 1” Die – 20 grams to 30 grams
    - 1.5” Die – 20 grams to 50 grams

- Want to observe the density of the resulting pellet using granular IMX-104
  - IMX-104 melt casted
    - Density values from 1.70 g/cc to 1.72 g/cc
  - 1” Die Set
    - Density values from 1.66 g/cc to 1.72 g/cc
  - 1.5” Die Set
    - Density values from 1.63 g/cc to 1.70 g/cc

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Pressing Evaluation – Results

- 1” Die Set
- Density is independent of mass completely dependent on load
    - 3000 lbs = 1.66 g/cc
    - 115000 lbs = 1.70 g/cc
    - 20000 lbs = 1.72 g/cc

- 1.5” Die Set
- Maximum density at higher load but dependent on mass
    - Less mass results in higher density
    - 20 grams = 1.71 g/cc
    - 50 grams = 1.69 g/cc

Scale Up of Baseline Process – Overview

- Baseline process from trials in Alternate Fluid B was selected and scaled to a two pound scale
  - 5 replicates prepared and blended for use in performance testing
  - Each replicate tested individually for composition, thermal properties, bulk density, and granulation

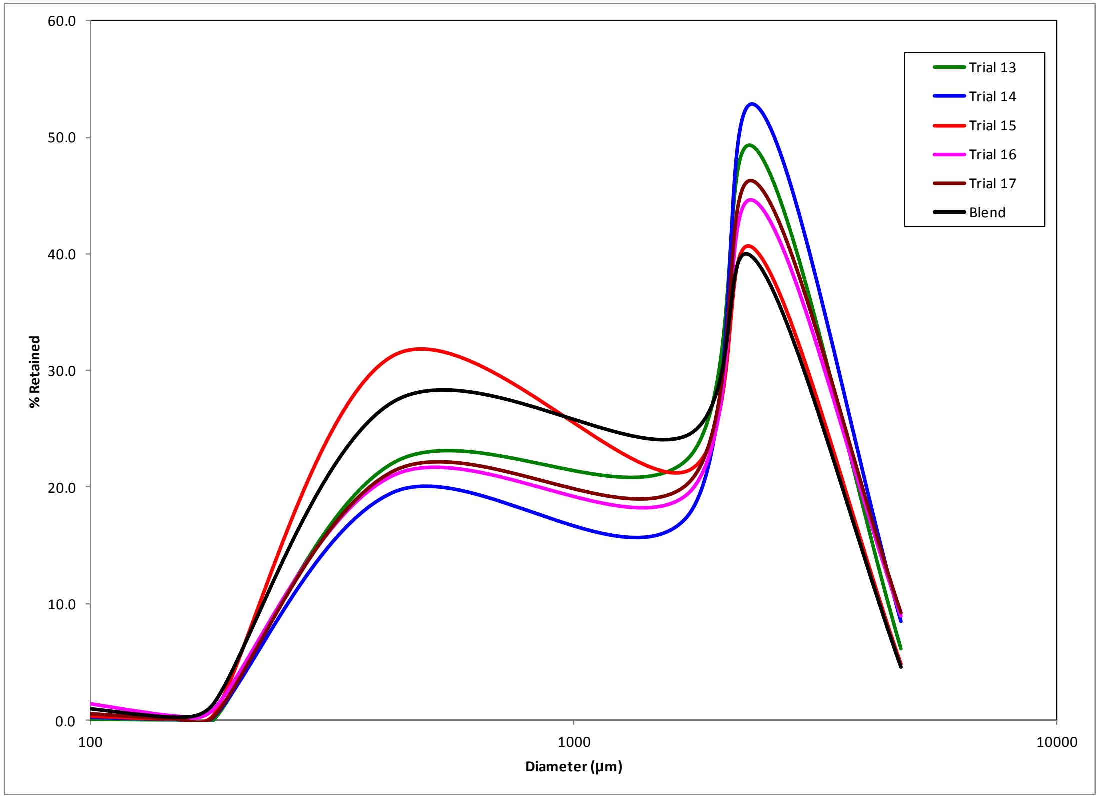
- Analysis yielded promising results
  - Replicates all meet specification for composition and thermal properties
  - Bulk densities are similar (greater than 0.9 g/cc)
  - Variation in granulation from batch to batch is small
  - Approximately 90% yield of product overall

Scale Up of Baseline Process – Analysis

Batch ID		Trial 13	Trial 14	Trial 15	Trial 16	Trial 17	Blend
Yield (%)		83.30	97.73	85.63	94.06	95.44	90.25
DNAN (% Deviation from target)		-2.08	1.97	-3.40	-1.07	-0.87	-0.42
RDX (% Deviation from target)		0.64	-4.56	-3.59	-0.95	-6.06	-5.71
NTO (% Deviation from target)		0.47	-0.89	2.87	0.42	1.98	1.98
Melting Point (ºC)		91.87	91.36	91.93	91.72	91.71	92.01
Exothermic Onset (ºC)		198	206	198	209	213	216
Bulk Density (g/cc)		0.9091	0.9259	0.9434	0.9615	0.9259	0.961
Screens (% Pass)	4	93.9	91.5	95.2	91.1	90.8	95.4
	8	44.8	38.7	54.8	46.6	44.6	55.8
	12	22.6	21.4	33.5	27.4	24.5	31.4
	40	0.5	1.8	2.2	6.4	3.1	4.0
	80	0.4	1.4	1.6	5.4	2.7	2.4
	100	0.4	1.3	1.5	5.1	2.6	2.1
	200	0.3	0.9	1.0	3.1	1.8	0.7
	325	0.1	0.5	0.5	1.0	0.9	0.2

Scale Up of Baseline Process – Granulation

- Each replicate manufactured in the scale up exercise is similar in granulation
- Larger than desired but usable
  - Average particle size is 1000µm
  - Still broad distribution
- Further work is required to optimize granulation



Performance Testing

- Material from scale up trials pressed into pellet form for use in LSGT evaluation against poured charges of standard IMX-104

Sample ID	Units	IMX-104 (Standard)	IMX-104 (Granular)
Charge Density	g/cc	1.72	1.69
Number of Shots		9	8
50% Point	cards	125	156
Kilobar Value	kbar	48.8	36.3

  - Average charge density of 1.69 g/cc (95% TMD)
  - Average charge density for poured material of 1.72 g/cc (97% TMD)
  - Difference in sensitivity partially due to difference in charge density
  - Still less sensitive than Composition B (201 – 220 cards; 16.9 – 20.5 kbar)

- Evaluation of detonation velocity and shockwave pressure scheduled for week of April 30, 2012

Conclusions and Future Work

- Water based process not viable due to solubility of ingredients in water
  - Disparity in temperature that NTO re-crystallizes from water causes segregation of NTO from other components
- Process based in alternate fluids has been successful in manufacturing granular versions of IMX-104
  - Meet specification for composition with no change in thermal properties
  - Some evidence that granulation can be controlled but requires additional evaluation
  - Pressing evaluation indicates that equivalent pressed densities to poured material can be achieved
  - LSGT value not significantly effected by granulation process
  - Utilizes existing manufacturing infrastructure at HSAAP
  - Additional work is required to determine scale up to manufacturing scale

Acknowledgements

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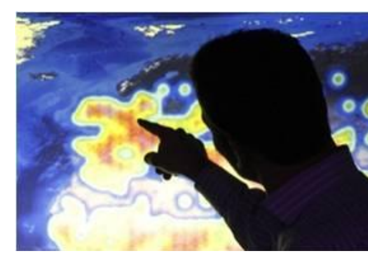

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Questions?