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Ageing of DNAN Based Melt-Cast Explosives

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ABSTRACT

DNAN or 2,4-dinitroanisole is a new melt-cast matrix that replaces traditional TNT based melt-cast explosives. Aside from sensitiveness improvements, the use of DNAN allows for the continued operation of existing melt-cast facilities (for example the Australian Munitions plants located at Mulwala and Benalla) without the need for major plant modifications. Weapons and Combat Systems Division (WCSD) has developed several DNAN-based formulations that have been extensively characterised and in an effort to better understand the ageing properties of these formulations, an accelerated ageing program was undertaken. Testing was conducted under two different ageing conditions; the first test condition was conducted at a constant 60 °C with ambient humidity and the second test condition was the A2 diurnal cycle (representative of the MEAO climate). Analysis of the ingredient composition, sensitiveness, mechanical and thermal properties was made at 3-month intervals for a period of 12 months and results compared with conventional explosives similarly aged.

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Ageing of Insensitive DNAN Based Melt-Cast Explosives

Executive Summary

An accelerated ageing program was undertaken to assess the ageing characteristics of Weapons and Combat Systems Division (WCSD) developed DNAN (2,4-dinitroanisole) formulations as replacements for TNT based melt cast explosive fills. WCSD has developed three DNAN formulations: ARX-4027 consisting of 60% RDX and 40% DNAN (analogous to Composition B, that is direct replacement of TNT by DNAN), ARX-4028 (70% NTO and 30% DNAN) designed as an insensitive explosive fill and ARX-4029 (65% NTO, 30% DNAN and 5% RDX) also designed as an insensitive explosive however with improved explosive performance characteristics over ARX-4028 (due to the presence of RDX).

Testing was conducted utilising two different ageing conditions: first a constant 60 ºC with ambient humidity and the second an A2 diurnal cycle, which represents the Middle East Areas of Operation (MEAO) climate. The first test condition was chosen to provide a worst-case scenario. Analysis of the ingredient composition, theoretical maximum density, sensitiveness, mechanical and thermal properties was made at 3 month intervals for a period of 12 months and the results compared to those of conventional TNT-based explosives similarly aged.

Results showed that accelerated ageing of DNAN samples showed no adverse effects compared to similarly aged Composition B samples. For both testing cycles, it was observed that there was slight degradation in component ratios and mechanical strength. It is also clear that the A2 cycle ages the formulations less harshly than for the 60 ºC ageing cycle. This can be attributed to the lower ageing temperatures for the A2 cycle as compared to the dry and harsh conditions encountered for the 60 ºC cycle.

It was observed that there was negligible change in impact, friction, electrostatic discharge and thermal testing (both differential scanning calorimetry (DSC) and temperature of ignition) over the entire testing time for all DNAN-based formulations.

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Abbreviations

ADF	Australian Defence Force
ARX	Australian Research Explosive
BAM	Bundensassanstalt für Materialprüfung (friction testing)
Comp. B	RDX/TNT (60/40)
DG	Dangerous Goods class
DoD	Department of Defence
DNAN	2, 4-Dinitroanisole
DSC	Differential scanning calorimetry
DSTO	Defence Science and Technology Organisation
ELSGT	Expanded Large Scale Gap Test
ESD	Eletrostatic discharge
F of I	Figure of Insensitiveness
GPa	Gigapascal
HD	Hazard Division
HEFC	High explosive firing chamber
IM	Insensitive munitions
LSGT	Large Scale Gap Test
MEAO	Middle East Areas of Operation
MNA	N-methyl-4-nitroaniline
NEQ	Net equivalent weight
NTO	3-Nitro-1,2,4-triazol-5-one
PBX	Polymer (or plastic) bonded explosive
PETN	Pentaerythritol tetranitrate
RDX	Cyclotrimethylenetrinitramine
T of I	Temperature of ignition
TGA	Thermogravimetric Analysis
TNT	2,4,6-Trinitrotoluene
VoD	Velocity of detonation
WCSD	Weapons Combat Systems Division

1. Introduction

Insensitive munitions (IM) or low vulnerability munitions are a key mandate underpinning modern explosives research in the ADF [1]. In an effort to increase IM technology options, the search for alternative ingredients has driven WCSD, DSTO to explore new insensitive explosive formulations that contain 2,4-dinitroanisole (DNAN). DNAN has recently found use in main charge fills as a melt-cast replacement explosive for TNT-based melt cast fills. TNT fills are traditionally known to suffer sensitivity problems to shock and have resulted in numerous catastrophic events world-wide with the concomitant loss of life and platforms. Thus the replacement of TNT with DNAN as the explosive matrix binder is seen as an integral step in moving towards IM melt-cast explosive fills.

Three model DNAN-based formulations developed at WCSD have now been extensively tested and offer good explosive performance with concomitant increases in IM behaviour. A number of reports detailing the formulation and explosive performance [2], shock sensitivity [3] and thermal testing of these new formulations [4] compliments this work. Further analysis was undertaken to examine their ageing characteristics and is the focus of this report.

The accelerated ageing program was designed to study and asses the suitability of the three candidate DNAN-based melt cast fills as a replacement for TNT-based melt cast fills in Australian munitions. Testing was carried out via two different ageing conditions:

> *Ageing Condition 1*: Heating at a constant 60 ºC with no humidity control and represents the most severe temperatures conditions possible whilst still maintaining safe test conditions.
>
> *Ageing Condition 2*: The A2 diurnal cycle for the A2 climatic category (Hot Dry) as specified in the DEF(AUST)5168 [5]. This cycle offers a severe temperature and humidity cycle (*sinusoidal* in nature) that the DNAN formulations could be expected to experience for a reasonable period of time within its service life. This cycle represents the harsh conditions found in places such as the Orzugan province in Afghanistan where the ADF has recently been deployed for a significant period of time.

In this work, accelerating ageing was conducted on three DNAN-based melt cast fills (ARX-4027, 4028 and 4029) and Composition B as a reference explosive. For each explosive formulation; analysis of the ingredient composition, mass loss, bulk density, sensitiveness, thermal properties and mechanical strength was performed at 3 monthly intervals over a period of 12 months at both ageing conditions.

2. Background

An accelerated ageing program was conducted on three melt-cast formulations that have been recently developed at DSTO (ARX-4027, ARX-4028 and ARX-4029). Two of the formulations (ARX-4028 and ARX-4029) contain the melt-cast binder matrix 2,4-dinitroanisole, DNAN coupled with the insensitive explosive 3-nitro-1,2,4-triazol-5-one, NTO (ARX-4028). In the case of ARX-4029, the addition of 5% RDX is provided to increase explosive performance. ARX-4027 contains DNAN and RDX only and was formulated to be the DNAN analogue of Composition B (i.e. the direct replacement of TNT with DNAN).

To make pellets for ageing, DNAN (supplied by BAE Systems, Army Ammunition Plant, Holston, Tennessee) was freeze dried for up to 24 hrs until constant mass was reached to remove water. Bimodal 3-nitro-1,2,4-triazol-5-one (NTO, supplied by Dyno, Norway) comprising 85% fine grade (30-110 µm) and 15% medium grade (200-400 µm) particle size distribution was used to optimise casting density, processability, explosive performance and shock sensitivity. RDX Type 1 Class 1 (Grade A) from Australian Munitions was used after drying at 60 ºC. N-methyl-4-nitroaniline (MNA) was used as received from Aldrich Chemicals. Note that MNA is added as a processing aid (melt-soluble additive) to lower the melting point of DNAN (94-95 ºC) to facilitate melting. Further additional processing and formulation details can be found in [2].

For comparative purposes, the standard melt-cast explosive Composition B was chosen as a reference composition. Explosive properties for all formulations are listed in Table 1.

All charges to be aged were cast as cylinders (sticks) at 25.4 mm diameter x 300 mm long and sectioned into one-inch pellets. Identical sized sticks of Composition B were also cast and sectioned accordingly.

2.1.1 ARX-4027

ARX-4027 was formulated to match Composition B with direct replacement of the TNT by DNAN. The formulation contains 39.75% DNAN, 60% RDX and 0.25% MNA as processing aid.

2.1.2 ARX-4028

This formulation ARX-4028 is formulated in-house at WCSD, DSTO and consists of 29.75% DNAN, 70% NTO and 0.25% MNA. ARX-4028 was formulated to provide maximum insensitive properties.

2.1.3 ARX-4029

ARX-4029 is an insensitive melt-cast composition designed to provide increased explosive performance over ARX-4028. The composition comprises a 29.75% DNAN matrix with 65% solids loading of the insensitive explosive (NTO), 5% RDX and 0.25% MNA.

2.1.4 Composition B

Composition B or more accurately known as RT 60/40 [3] was formulated in-house at WCSD, DSTO using Australian Munitions procured Type 1 RDX (60%) and Australian Munitions TNT (40%) + 1% wax.

Table 1: Explosive, Thermal and Sensitiveness Properties of DNAN, ARX-4027-9 & Comp. B

Ingredients	**DNAN^**	**ARX-4027^**	**ARX-4028^**	**ARX-4029^**	**Comp. B**
DNAN	100.0%	39.75%	29.75%	29.75%	–
MNA	–	0.25%	0.25%	0.25%	–
RDX	–	60.0%	–	5.0%	60.0%
NTO	–	–	70.0%	65.0%	–
TNT	–	–	–	–	40.0%
Property					
VoD (m/s)	5344	7398	7179	7487	7843
Rel. Det. Pressure (GPa)	9.49	22.47	20.84	22.01	24.5
D_{crit} (mm)	47.5 – 50.8	9.3-11.8	44.0 – 50.8	38.1 – 44.0	3.0 – 4.0
Density (g/cm^3)	1.54	1.68	1.76	1.77	1.72
Shock sensitivity (GPa)	7.02	2.62	8.14	6.21	2.69
Gap (mm)*	18	47	13.5	21.4	45.9
Thermal (DSC)					
Melting point (ºC)	95.1	91.07	95.8	90.86	80.9
Decomposition point (max., ºC)	350	236	262	258	220
Sensitiveness					
Impact (F of I)	>220	160	200	200	140
Friction (N)	160	288	324	288	108
T of I (ºC)	347	220	227	205	212
ESD (ignition/J)	4.5	4.5	4.5	4.5	4.5
Vac. Thermal Stability (mL/g)	0.02	0.2	0.37	0.33	0.15

^ Data taken from [2], [4-6].
* Gap material is polymethyl methacrylate.

Of the three ARX DNAN-based formulations, the two that contain NTO show considerable reduction in shock sensitivity over both Composition B and ARX-4027 (note that ARX-4027 was formulated to be the DNAN analogue of Composition B). Nevertheless, from a thermal performance point of view, all three DNAN-based formulations display advantages over Composition B in terms of higher thermal decomposition properties. Furthermore, all three DNAN-based formulations have significantly improved sensitiveness properties over Composition B and this gives these formulations the potential of being IM compliant.

3. Ageing Conditions

Ageing samples were prepared by sectioning one-inch (25.4 mm) diameter sticks into one-inch long pellets. From each stick, the bottom three pellets were used for mechanical testing, the next pellet was used for sensitiveness and thermal testing and the final pellet for ingredient analysis. Testing was carried out on a three monthly basis over a period of twelve months. At each time period of ageing, pellets in identical positions were compared. If required, additional pellets from the remains of the longer sticks were used for retesting. Typically one stick would supply the pellets required for one set of testing per each time period.

Un-aged pellets were accurately pre-weighed, measured for height and diameter (at 0^o and 90^o orientation) by digital callipers, and photographed for later comparison against aged samples. Pellets were then placed in the oven on trays and ageing commenced. No attempt to confine or cover the pellets was made.

Pellets were subjected to both testing conditions. One set of pellets was held at 60 °C in an oven at DSTO Edinburgh with no humidity control. This represents the most severe temperature conditions possible while still maintaining safe test conditions. The second testing condition was performed for the A2 diurnal cycle (Hot Dry climate) according to DEF(AUST)5168 [7]. The A2 diurnal cycle consists of offset temperature and humidity cycles over 24 hours and is representative of typical southern Afghanistan regions[1]. The A2 diurnal cycle involves temperature cycles between 30 and 44 °C with humidity cycling between 14 and 44% (Fig. 1).

[1] This assignation is based on DEF(AUST)5168 [7], Category A2 which applies to areas which experience high temperatures accompanied by moderately low humidity, namely, the most southerly parts of Europe, part of the Australian continent, south central Asia, northern and eastern Africa, coastal regions of North Africa, southern parts of USA and most of Mexico. Differentiation between A1, A2 & A3 diurnal cycles according to region is made on the map found in [7]. See Appendix A for the map.

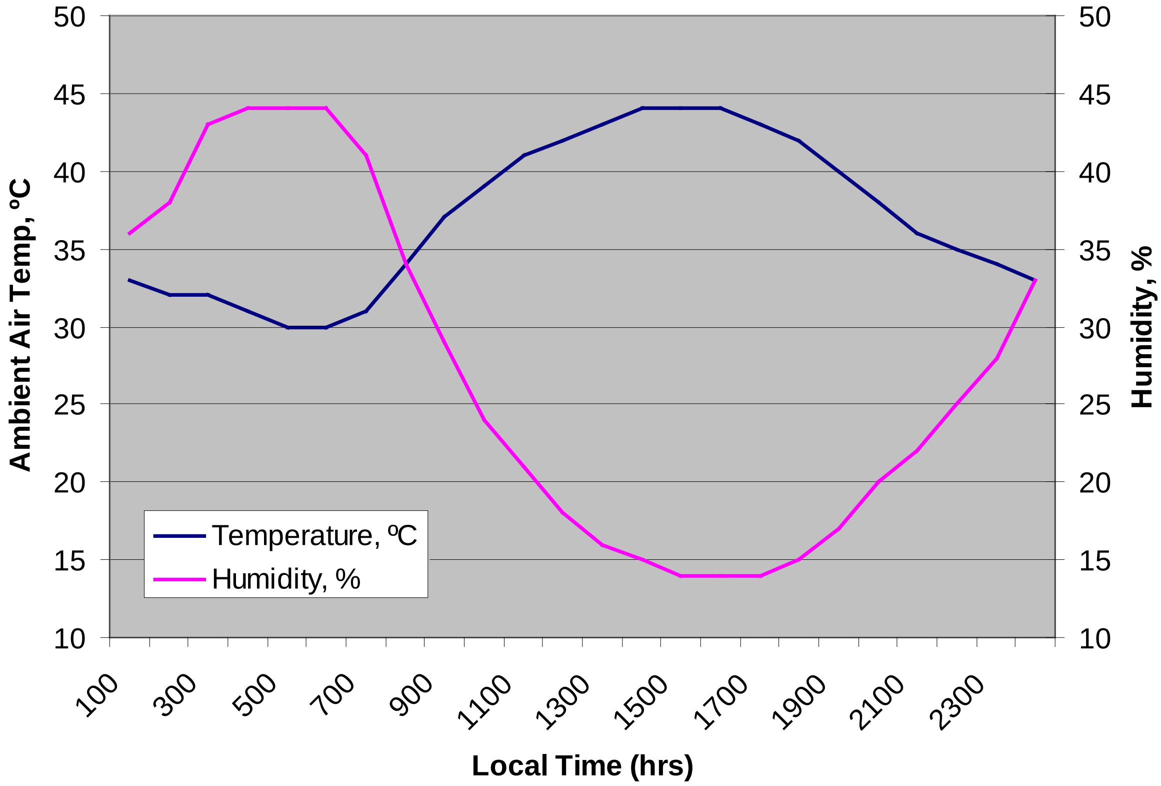

Figure 1: A2 Diurnal Cycle [7]

4. Experimental

4.1 Materials

In an effort to ensure that the contents of each stick did not deviate more than expected, ingredient analysis was undertaken for each formulation. In all cases, the 5th pellet (from the bottom of the stick upwards) for each stick was utilised for the analysis. Note that due to the small amount of MNA (0.25%) per formulation, a calculation for its total content was not conducted. All consumables used were analytical reagent grade.

4.1.1 ARX-4027 Ingredient Analysis

The 5th pellet of ARX-4027 (NEQ of approximately 20 g) was dissolved in benzene (100 mL) and stirred for an hour. The mother liquor was then decanted through filter paper and collected. Further washing with 20 mL aliquots of benzene was then applied to adequately remove the DNAN.

The mother liquor was then placed in a rotary evaporator to remove solvent and then dried under vacuum. The material was weighed (to give % DNAN) with the dried contents from the filter paper to give the total amount of RDX and DNAN.

4.1.2 ARX-4028 Ingredient Analysis

The 5th pellet of ARX-4028 (NEQ of approx. 20 g) was dissolved in benzene (100 mL) and stirred for an hour. The mother liquor was then decanted through filter paper and collected. Further washing with 20 mL aliquots of benzene was then applied.

The mother liquor was then placed in a rotary evaporator to remove solvent and then dried under vacuum. The material was weighed to give the total DNAN content for the pellet. The filter paper contents were then dried under vacuum and weighed to give the total NTO content.

4.1.3 ARX-4029 Ingredient Analysis

The 5th pellet of ARX-4029 (NEQ of approx. 20 g) was dissolved in benzene (100 mL) and stirred for an hour. The mother liquor was then decanted through filter paper and collected. Further washing with 20 mL aliquots of benzene was then applied.

The mother liquor was then placed in a rotary evaporator to remove solvent and then dried under vacuum. The material was weighed to give the DNAN content for the pellet.

The filter paper contents were then placed in a beaker and water (100 mL) was added and solution stirred for an hour. The material was then filtered and washed with a further two aliquots of water (20 mL each). The solids remaining in the filter paper were then dried under vacuum and weighed to give the total RDX content. The mother liquor was then placed in a rotary evaporator to remove the water and then dried under vacuum. This provides the total NTO content for ARX-4029.

4.1.4 Composition B Ingredient Analysis

The 5th pellet of Composition B (NEQ of approx. 20 g) was dissolved in benzene (100 mL) and stirred for an hour. The mother liquor was then decanted through filter paper and collected. Further washing with 20 mL aliquots of benzene was then applied.

The mother liquor was then placed in a rotary evaporator to remove solvent and then dried under vacuum. The material was weighed to give the total TNT content for the pellet. The solids collected on the filter paper were then dried under vacuum and weighed to give the RDX content.

4.2 Instrumentation

4.2.1 Differential Scanning Calorimetry

Differential scanning calorimetry (DSC) analyses were performed on a TA Instruments Q10 DSC, under nitrogen purge in closed aluminium pans (sample weights of approximately 1 mg). High temperature decomposition behaviour was measured by heating from 25 - 500 ºC at 10 ºC per minute with Indium metal as reference (156.6 ºC).

4.2.2 Thermogravimetric Analysis

Thermogravimetric analysis (TGA) analyses were performed on a TA Instruments Q500 TGA, under nitrogen purge in open aluminium pans (sample weights of approx. 1-2 mg). Weight loss with time was measured by heating from 25 to 60 ºC at 10 ºC per minute and isothermally heating for 180 mins. Nickel metal was used as the temperature reference.

4.3 Hazards and Mechanical Properties Testing

4.3.1 Rotter Impact Sensitiveness

Impact sensitiveness was determined on a Rotter apparatus [8]. Samples were prepared from melt-cast sticks of the material under investigation, using a mortar and pestle to crush the sample and passed through an 850 micron sieve. The powder was weighed into brass caps (30 mg per cap), allowed to stand overnight in a desiccator, then fitted over a polished steel anvil and impacted by a 5 kg weight falling from a pre-set height.

Go / No Go was determined by the evolution of gas, a positive result being recorded for > 0.5 mL. Impact height was varied in a Bruceton procedure with a total of 25 caps tested. The resulting figure of insensitiveness (F of I) is quoted relative to RDX, F of I = 80 and gas evolution represents the average of all positive results.

4.3.2 Friction - BAM Test

Friction was determined on a Julius Peter BAM tester which measures values by applying a thin film of sample to a ceramic tile and lowering the friction peg [8]. A maximum loading is placed on the tester and the peg dragged across the sample to create friction. The sample was observed for any sign of ignition, spark, crack or smoke. The test was repeated with decreasing loadings until six repetitions with the same loading provided no evidence of ignition. The result is reported as the limiting load (N), the minimum load required for ignition.

4.3.3 Temperature of Ignition

Temperature of ignition (T of I) was determined on an instrument built to specification [8]. Duplicate samples of 200 mg in glass test tubes were heated at 5 ºC/min until a reaction occurred, as defined by first visible signals such as smoke/flame or audible hiss/bang. The T of I is the lowest temperature at which this event occurred.

4.3.4 Electrostatic Discharge, ESD

This test is performed according to the established procedure in [8]. Five cavities in a polypropylene annulus are filled with the sample and each hole individually covered with a small piece of copper foil to assist conductivity through the sample. A capacitor is charged to a defined potential, giving a stored energy of 4.5 J, 0.45 J or 0.045 J. The test is initiated by applying a pulse that causes the potential of the selected capacitor to form across the sample spark gap and is tested 25 times.

4.3.5 Tensile Strength Testing

Mechanical strength testing (compression) was undertaken on an Instron 5500 (R1185) Universal Testing Machine with a 100 kN load cell set to a crosshead rate of 1 mm/min. Testing was undertaken at 23 °C (room temperature) on machined sections (25.4 mm x 25.4 mm). The following parameters were recorded: maximum load, modulus and compressive stress at the yield point (failure point).

5. Results & Discussion

5.1 Ingredient Analysis

For each ARX explosive, examination of the individual ingredients percentage data is presented in Table 2. The underlying trend reveals that mass losses for all formulations at the 60 ºC ageing cycle percentage levels are higher than for the corresponding A2 ageing cycles. The highest mass loss at the A2 ageing cycle was 0.8% mass loss after 12 months for ARX-4029, whereas for the 60 ºC ageing cycle after 12 months, the highest mass loss was observed for the ARX-4027 formulation (4.5% mass loss). Such differences are more than likely attributed to the sublimation of DNAN at elevated temperatures. This is also apparent for Composition B where there is also a minor loss of TNT due to its sublimation at elevated temperatures (between 1.2 – 1.4% loss of TNT at both ageing cycles).

Table 2: Ageing Ingredient Analysis Data for ARX Explosives (Values Represent Percentage of Ingredient Mass)

	0 Months	**3 Months**		**6 Months**		**9 Months**		**12 Months**	
		60 °C	**A2**	**60 °C**	**A2**	**60 °C**	**A2**	**60 °C**	**A2**
ARX-4027									
RDX	60.1	60.12	60.09	59.92	60.02	59.54	59.82	59.2	59.5
DNAN	39.66	39.01	39.73	38.11	39.68	37.05	39.62	36.05	39.65
% Δ @ 12 months								4.5	0.6
ARX-4028									
NTO	70.29	70.28	70.22	70.25	70.25	70.25	70.26	70.2	70.24
DNAN	29.76	28.95	29.75	28.32	29.73	27.98	29.75	27.7	29.74
% Δ @ 12 months								2.2	0.1
ARX-4029									
NTO	65.9	65.8	65.9	65.7	65.8	65.5	65.8	65.5	65.9
RDX	4.95	4.8	4.82	4.44	4.55	4.21	4.35	4.07	4.14
DNAN	29.61	29.23	29.65	29.1	29.59	28.95	29.6	28.71	29.6
% Δ @ 12 months								2.2	0.8
Composition B									
RDX	58.97	59.57	59.65	58.48	58.55	58.47	58.92	58.07	58.09
TNT	41	40.38	40.41	40.32	40.11	40.1	40.75	40.68	40.5
% Δ @ 12 months								1.2	1.4

5.2 Density and Mass Analysis

For each pellet, measurements were taken of the length, height and mass at both pre- and post-ageing cycle. As a general rule, for both ageing cycles progressing from 0 to 12 months, the densities of the ARX (and Composition B) formulations decrease accordingly (Table 3).

For the A2 ageing cycle, after 3 months ageing only minor decreases in density are observed; with prolonged ageing to 12 months the highest % density decrease is 1.42% (for ARX-4029). This is in contrast to the 60 ºC ageing cycle where larger decreases in density are observed of up to 3.21% (for ARX-4027). Composition B gives the largest decrease in density of 3.49% of any formulation tested after 12 months at the 60 ºC ageing cycle.

Table 3: Pre- and Post-density Values for both Ageing Cycles

	Ageing Cycle					
	60 ºC			A2		
	Density		%	Density		%
	Pre-Test	Post-Test	Diff.	Pre-Test	Post-Test	Diff.
ARX-4027						
3 months	0.9738	0.9599	1.36	0.9691	0.9607	0.84
6 months	0.9686	0.9487	1.99	0.9745	0.9642	1.03
9 months	0.955	0.9229	2.62	0.9686	0.9552	1.29
12 months	0.9539	0.9276	3.21	0.9697	0.9567	1.34
ARX-4028						
3 months	0.9777	0.9713	0.64	0.9720	0.9675	0.45
6 months	0.9807	0.9661	1.46	0.9765	0.9695	0.69
9 months	0.9763	0.9542	1.33	0.9777	0.9665	0.65
12 months	0.97645	0.9632	2.21	0.9778	0.9713	1.11
ARX-4029						
3 months	0.9770	0.9720	0.49	0.9711	0.9749	0.22
6 months	0.9779	0.9624	1.55	0.9825	0.9721	1.04
9 months	0.9709	0.9548	1.61	0.9813	0.9671	0.89
12 months	0.9757	0.9584	1.73	0.9815	0.9725	1.42
Comp. B						
3 months	0.9754	0.9703	0.51	0.9749	0.9699	0.50
6 months	0.9787	0.9654	1.33	0.9886	0.9759	1.30
9 months	0.9742	0.9499	2.43	0.9799	0.9651	1.48
12 months	0.9752	0.9403	3.49	0.9887	0.9633	2.54

Comparison between the two ageing conditions highlights an increase in % change of density for the harsher ageing test (60 ºC constant heating with no humidity control) over the A2 cycle. This is due to the fact that the constant heating rate confers extra heat and low humidity on melt-cast DNAN systems over the A2 cycle which only has a maximum temperature of 44 ºC with humidity (14 – 44%). Thus the 60 ºC aged pellets experience harsher environmental conditions and as a consequence degrade more than the A2 aged pellets. Nevertheless, the largest % decrease in density values obtained at the 60 ºC ageing cycle (3.21% for ARX-4027) implies that all three DNAN-based formulations are capable of withstanding prolonged ageing with only minor reductions in density. They also show marginally better ageing characteristics than the reference explosive Composition B (density of 3.49%).

In an effort to correlate the density with the mass observations of each pellet, a table of mass loss is provided below (Table 4). It was found that values correlate well; for the harsher ageing test (60 ºC constant heating with no humidity control) the mass loss is greater than for the A2 cycle pellets and there is also little difference between the DNAN formulations and Composition B. These results imply that the density decreases with ageing are due to the mass loss of each pellet.

Table 4: Pre- and Post- Mass Values for both Ageing Cycles

	Ageing Cycle					
	60 ºC			**A2**		
	Mass, g		**% Diff.**	**Mass, g**		**% Diff.**
	Pre-Test	**Post-Test**		**Pre-Test**	**Post-Test**	
ARX-4027						
3 months	21.632	21.284	0.348	21.497	21.398	0.099
6 months	21.462	20.911	0.551	21.646	21.558	0.088
9 months	21.219	20.547	0.672	21.536	21.476	0.060
12 months	21.265	20.402	0.863	21.529	21.438	0.091
ARX-4028						
3 months	22.878	22.609	0.269	22.636	22.611	0.025
6 months	22.944	22.507	0.437	22.802	22.739	0.063
9 months	22.812	22.278	0.534	22.784	22.717	0.067
12 months	22.833	22.222	0.611	22.846	22.744	0.102
ARX-4029						
3 months	22.891	22.71	0.181	22.751	22.733	0.018
6 months	22.861	22.469	0.392	22.858	22.789	0.069
9 months	22.644	22.193	0.451	22.829	22.765	0.064
12 months	22.827	22.308	0.519	22.813	22.699	0.114
Comp. B						
3 months	22.349	22.165	0.184	22.357	22.268	0.089
6 months	22.892	22.471	0.421	22.947	22.859	0.088
9 months	22.476	21.909	0.567	22.056	21.959	0.097
12 months	22.859	22.237	0.622	22.874	22.723	0.151

5.3 Sensitiveness Testing

For all formulations the effects of hazardous stimuli at each ageing period were examined by sensitiveness testing. Tests utilised were Rotter Impact (Table 4), BAM Friction (Table 5), Temperature of Ignition (Table 6) and Electrostatic Discharge (ESD). Importantly, only minor variances were noted for any of the tests; with no discernible change noted for the ESD test, where in all samples ignition at 4.5 J but not at 0.45 J was observed.

Table 4: Rotter Impact Results (Figure of Insensitiveness)

Formulation & Ageing Cycle	0 months	3 months	6 months	9 months	12 months
ARX-4027					
A2	160	150	150	170	170
60 °C	160	140	130	140	140
ARX-4028					
A2	200	>200	>200	>200	>200
60 °C	200	>200	>200	170	170
ARX-4029					
A2	200	>200	>200	>200	>200
60 °C	200	>200	>200	200	200
Comp. B					
A2	110	170	170	170	180
60 °C	110	130	150	150	140

The impact test results of the DNAN-based formulations show consistent trends with respect to each individual formulation and its respective cycle condition. It can be observed there is an overall trend to become slightly more insensitive to impact for A2 conditions and slightly more sensitive for 60 °C conditions. In the case of the 60 °C ageing cycle, the increase in sensitiveness (maxima of 30 units) is minor considering the aggressive ageing conditions that the pellets are experiencing. On the other hand, Composition B becomes more insensitive to both ageing conditions and reasons as to why this occurs cannot be readily explained.

Table 5: BAM Friction Results (N)

Formulation & Test	0 months	3 months	6 months	9 months	12 months
ARX-4027					
A2	288	288	252	252	288
60 °C	288	288	288	288	324
ARX-4028					
A2	324	324	360	360	360
60 °C	324	360	324	324	360
ARX-4029					
A2	288	324	324	288	360
60 °C	288	324	288	324	360
Comp. B					
A2	108	80	80	108	108
60 °C	108	80	80	84	84

Friction sensitiveness for all DNAN-based formulations has become marginally more insensitive with time for both ageing conditions; Composition B has not followed this trend becoming more sensitive with time for the 60 °C cycle while sensitiveness remains unchanged for the A2 cycle. This can be explained by the aggressive nature of the 60 ºC cycle which renders the Composition B more friction sensitive. The A2 cycle (for

Composition B) imparts lower temperatures and humidity which provide a less aggressive regime to keep the sensitiveness unchanged.

Table 6: Temperature of Ignition (°C)

Formulation & Test	0 months	3 months	6 months	9 months	12 months
ARX-4027					
A2	205	205	207	207	206
60 °C	205	206	206	208	209
ARX-4028					
A2	237	239	238	237	236
60 °C	237	239	238	239	240
ARX-4029					
A2	220	218	218	218	216
60 °C	220	218	216	216	217
Comp. B					
A2	212	212	211	212	213
60 °C	212	212	210	210	211

The results for the Temperature of Ignition test show ARX-4027 from the 60 °C cycle had an overall change of +4 °C between zero and twelve months and ARX-4029 from the A2 cycle a difference of -4 °C, these two results were the largest changes observed. These results are not considered significant, if we consider the high temperature of ignition values are still retained and that the data from the other sensitiveness testing are not deviating by large factors.

Results from the sensitiveness tests on the accelerated ageing data gathered suggests that the DNAN-based melt cast formulations do not become more sensitive to hazardous stimuli upon ageing. Sensitiveness remains relatively constant regardless of ageing conditions and time duration, and compare more favourably to those of Composition B.

5.4 Thermal Properties

Thermal properties (onset of melt and maximum peak decomposition) were measured by DSC (see Appendix B). From time 0 to 12 months only minor variation was observed in onset and decomposition temperature for both 60 °C and A2 ageing cycles. Some characteristic features observed from the DSC thermograms include a broadening of the decomposition peak at time 12 months, and this could be indicative of degradation of the explosive formulation. Interestingly this may be only occurring for the RDX, as both RDX containing formulations (ARX-4027 and ARX-4029) display such behaviour (Appendix B) while ARX-4028 which contains only DNAN and NTO does not show this degradation behaviour in the DSC traces. Such degradation behaviour is also more pronounced for samples at 60 °C than for samples aged via the A2 cycle and is consistent with other results such as density and sensitiveness testing.

Table 7: DSC Thermal Properties

Formulation & Test	Onset Temperature, °C					Decomposition Temperature, °C (Maximum)				
Months	0	3	6	9	12	0	3	6	9	12
ARX-4027										
A2	87.4	87.8	88.5	91.0	90.8	235.7	236.9	235.2	234.2	235.5
60 °C	87.4	87.8	88.6	90.7	90.6	235.7	237.2	237.2	236.9	240.4
ARX-4028										
A2	89.6	90.7	92.4	94.2	94.0	262.1	260.7	267.9	261.6	260.9
60 °C	89.6	90.6	92.1	93.9	94.4	262.1	259.6	263.0	266.5	268.2
ARX-4029										
A2	87.0	87.5	88.2	90.0	90.3	257.9	257.0	263.3	252.9	263.4
60 °C	87.0	87.7	88.2	90.0	90.5	257.9	253.1	262.5	262.5	256.9

5.5 Mechanical Strength

Three pellets from each formulation were analysed for mechanical strength properties at three monthly ageing periods. Mechanical properties data are presented for both ageing cycles in tabular format (Tables 8 and 9) and data is also compared for each mechanical property (maximum load, modulus and compressive stress at yield) against each ageing cycle (Figs. 4–8). Standard deviation for each property is also given.

With compressive mechanical testing of cylinders (pellets), the forces are acting to shorten the pellet and thus the maximum load is simply the maximum amount of load that the pellet experiences under test (kN). The modulus is the stress experienced by the pellet over the strain (in MPa) and the compressive stress at yield is the stress experienced by the pellet at the yield point (or failure point, in MPa).

For the DNAN-based ARX formulations, the mechanical properties tested were observed to have suffered adversely under the 60 °C cycle when compared to the A2 ageing cycle. This was directly attributed to the more severe and harsh environment of the constant 60 °C test cycle.

Further, all ARX formulations have maintained mechanical superiority over Composition B for the duration of both ageing cycles. Indeed at 12 months, all ARX formulations were at equal or greater mechanical properties than Composition B. At both testing cycles, the maximum load, modulus and compressive stress are all observed to follow a downwards trend with time (Figs. 4-8).

Other trends noted include formulations that contain NTO (ARX-4028 & 4029) display higher mechanical properties than those formulations which do not contain any NTO (ARX-4027 & Composition B). This has been noted previously for both TNT and DNAN melt cast binders and has been directly attributed to the increased mechanical properties of the matrix between NTO with DNAN [2] and NTO with TNT [10].

Table 8: 60 °C Mechanical Testing

Formulation & Test	0 months	3 months	6 months	9 months	12 months
ARX-4027					
Max Load (kN)	11.6	9.6	7.8	7.6	7.2
Standard Deviation	0.5	0.4	0.7	0.2	0.3
Modulus (MPa)	1878	2091	1709	1596	1530
Standard Deviation	32	13	23	29	32
Stress at Yield (MPa)*	22.89	18.97	12.21	14.73	14.17
Standard Deviation	0.01	0.05	0.01	0.01	0.01
ARX-4028					
Max Load (kN)	16.4	14.6	13.3	11.8	10.5
Standard Deviation	0.5	0.3	0.4	0.5	0.5
Modulus (MPa)	2277	2426	1929	1854	1841
Standard Deviation	11	19	23	44	45
Stress at Yield (MPa)	32.00	28.89	26.34	23.35	20.77
Standard Deviation	0.01	0.18	0.02	0.02	0.01
ARX-4029					
Max Load (kN)	17.5	15.4	14.8	13.5	13.5
Standard Deviation	0.2	0.2	0.2	0.3	0.2
Modulus (MPa)	2364	2393	1955	1906	1946
Standard Deviation	9	9	19	5	13
Stress at Yield (MPa)	34.1	30.43	29.17	26.56	26.6
Standard Deviation	0.01	0.31	0.01	0.02	0.01
Composition B					
Max Load (kN)	7.2	7.2	7.6	6.8	6.0
Standard Deviation	0.1	0.4	0.8	1.0	0.9
Modulus (MPa)	1602	1526	1287	1260	1146
Standard Deviation	23	61	113	63	84
Stress at Yield (MPa)	18.99	17.96	16.33	14.74	14.52
Standard Deviation	0.01	0.01	0.02	0.01	0.01

* Compressive stress at yield

The visual appearance of both the 60 °C and A2 cycle samples are markedly different. In the case of the 60 °C samples, all ARX formulations that are yellow in colour (due in part to DNAN and to the MNA) begin to fade and become paler with ageing. Also for the constant 60 °C condition, all formulations containing NTO (ARX-4028 and 4029) exhibit a powdery white chalky coating (Fig. 2) whereas this discolouration of the pellets tested via the A2 cycle was not as pronounced (Fig. 3).

It is believed that the white colour is related to NTO as it is not as evident in the ARX-4027 formulation which contains no NTO. To further confirm the presence of NTO, some of the white material was scraped off and FT-IR spectroscopic analysis conducted to reveal the presence of NTO with DNAN. Furthermore, there is no correlation of this phenomenon with thermal analysis techniques such as DSC.

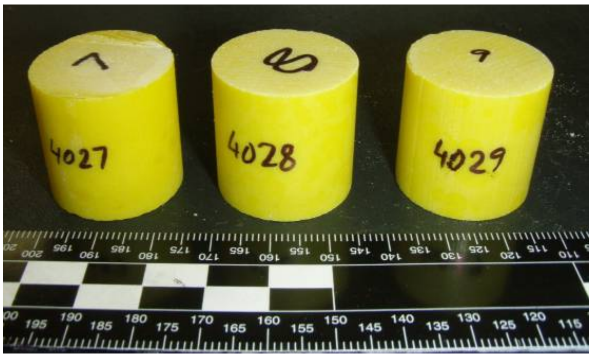

Figure 2: Photographs of 60 °C cycled ARX-4027-9 pellets (starting at t = 0 months on the top to 12 months on the bottom)

The most likely explanation for this discolouration is that at the 60 °C ageing condition with no humidity control, DNAN (and concomitantly the MNA) is subliming at a much greater rate over samples tested via the A2 cycle. This sublimation has the effect of removing the DNAN and MNA yellow colour from the pellet to leave behind the white NTO (for ARX-4028 & ARX-4029). The sublimation of DNAN is also apparent on the ARX-4027 pellet, with a fine coating of RDX apparent on all surfaces (Fig. 3). The minimal white colour for ARX-4027 is due to the fact that ARX-4027 does not contain any NTO, and also contains 10% more DNAN than 4028 or 4029.

Earlier studies to determine the DNAN sublimation rate showed that the rate of sublimation increases at higher temperatures [6]. The DNAN sublimation rate was determined by use of isothermal TGA (Thermogravimetric Analysis) by heating to temperatures between 95 to 125 °C and holding isothermally for three hours [6]. DNAN mass loss as a rate over time was then determined and extrapolated to provide a sublimation rate at each temperature.

DNAN was found to sublime at a faster rate at the higher temperatures (22 mg/min at 95 °C *vs.* 34 mg/min at 125 °C) and extrapolating backwards to 60 °C, it can be concluded that the sublimation rate for DNAN will be slower. An attempt at determining the DNAN sublimation rate at 60 °C by isothermal TGA was conducted, however due to the limits of the analyser there was no observed negligible differences in weight over the 3 hours isothermal time at 60 °C. What these experiments indicate, is that at high temperatures DNAN will sublime considerably and as a consequence, ageing at a constant 60 °C over 12 months; then loss of DNAN by sublimation is likely to lead to the white appearance for ARX-4028 & ARX-4029 and the pale yellow colour observed in ARX-4027.

Mechanical properties values were also typically lower for the pellets tested at 60 °C cycle over pellets tested at the A2 cycle (Tables 8 and 9) and this could potentially be attributed to sublimation.

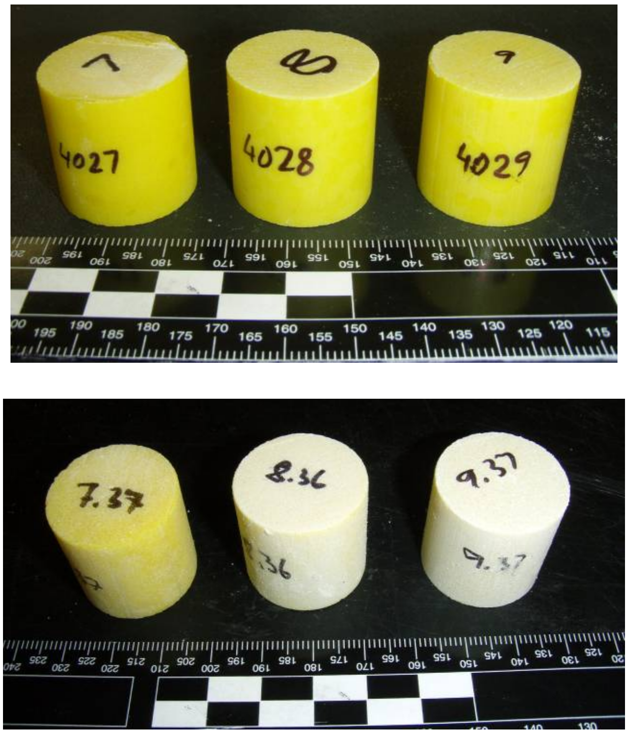

Figure 3: Photographs of A2 cycled ARX-4027-9 pellets (starting at t = 0 months on the top to 12 months on the bottom)

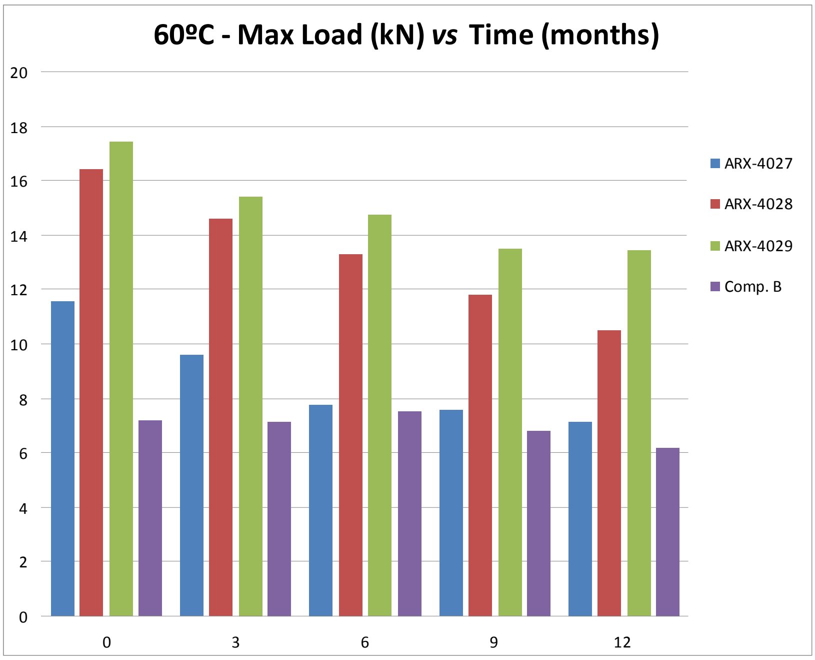

Figure 4: 60 °C ageing profile for maximum load (kN)

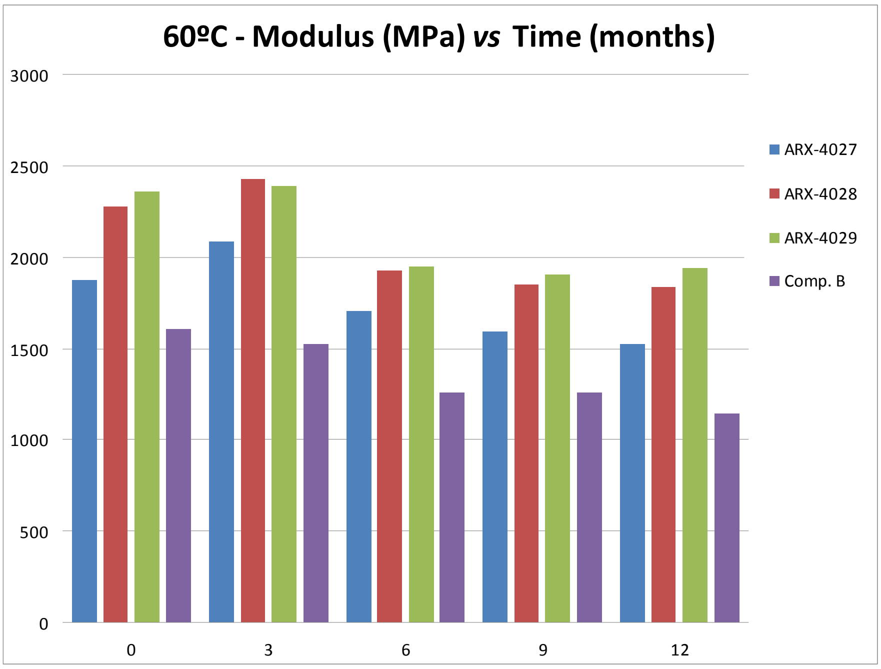

Figure 5: 60 °C ageing profile for modulus (MPa)

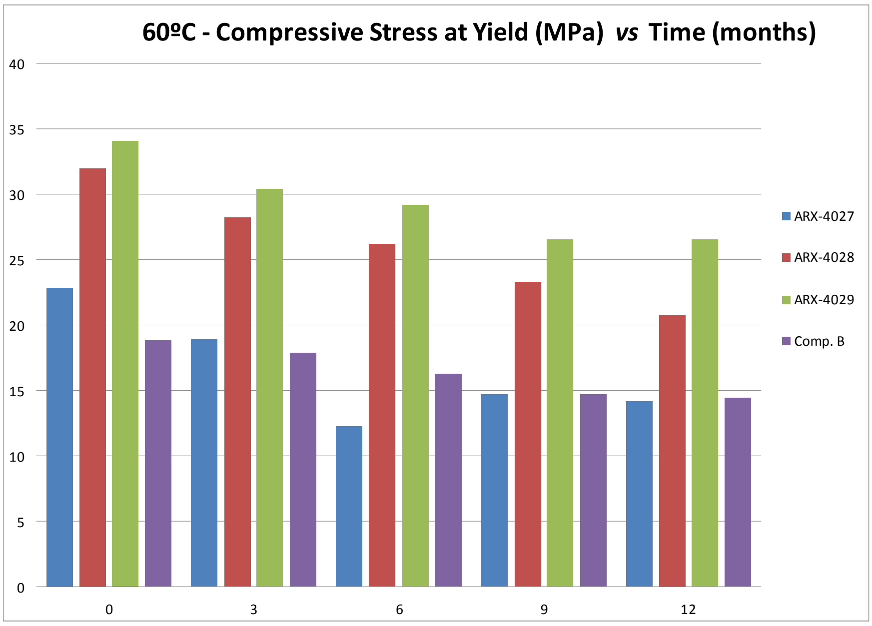

Figure 6: 60 °C ageing profile for compressive stress (MPa)

Table 9 displays the A2 results for all formulations at both testing cycles. As noted already, the most apparent result is that the A2 test is not as severe on the environmental condition of the pellets as is the 60 °C testing.

Table 9: A2 Mechanical Testing

Formulation & Test	0 months	3 months	6 months	9 months	12 months
ARX-4027					
Max Load (kN)	11.6	9.1	7.8	6.7	6.9
Standard Deviation	0.5	1.3	1.0	0.1	0.9
Modulus (MPa)	1878	2164	1519	1590	1420
Standard Deviation	32	156	57	163	89
Stress at Yield (MPa)	22.89	17.94	13.51	13.21	14.1
Standard Deviation	0.01	0.01	0.01	0.14	0.01
ARX-4028					
Max Load (kN)	16.4	16.1	15.3	14.8	15.1
Standard Deviation	0.5	0.3	0.2	0.2	0.2
Modulus (MPa)	2277	2561	2188	2179	2172
Standard Deviation	11	40	45	88	101
Stress at Yield (MPa)	32.00	31.88	30.26	29.27	31.17
Standard Deviation	0.01	0.02	0.01	0.01	0.01
ARX-4029					
Max Load (kN)	17.5	16.7	16.2	15.2	14.7
Standard Deviation	0.2	0.4	0.3	0.6	0.6
Modulus (MPa)	2364	2604	2226	2173	2187
Standard Deviation	9	20	56	6	17
Stress at Yield (MPa)	34.10	32.80	32.07	30.05	29.09
Standard Deviation	0.01	0.01	0.01	0.01	0.02
Composition B					
Max Load (kN)	7.2	7.2	7.5	7.1	6.6
Standard Deviation	0.6	1.2	1.3	1.1	0.6
Modulus (MPa)	1605	1525	1287	1260	1151
Standard Deviation	89	45	79	29	38
Stress at Yield (MPa)	18.99	18.56	17.99	17.10	15.70
Standard Deviation	0.01	0.01	0.01	0.02	0.01

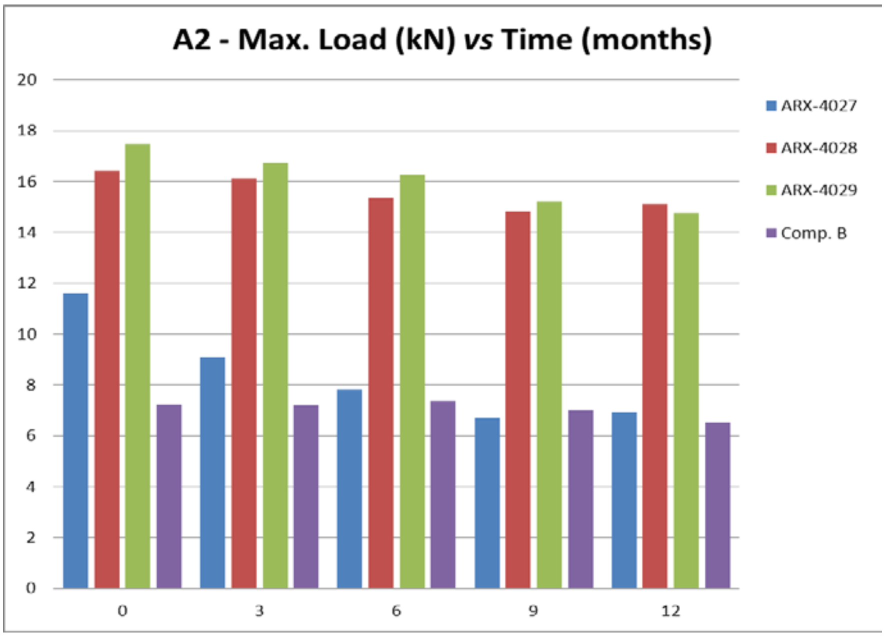

Figure 7: A2 ageing profile for maximum load (kN)

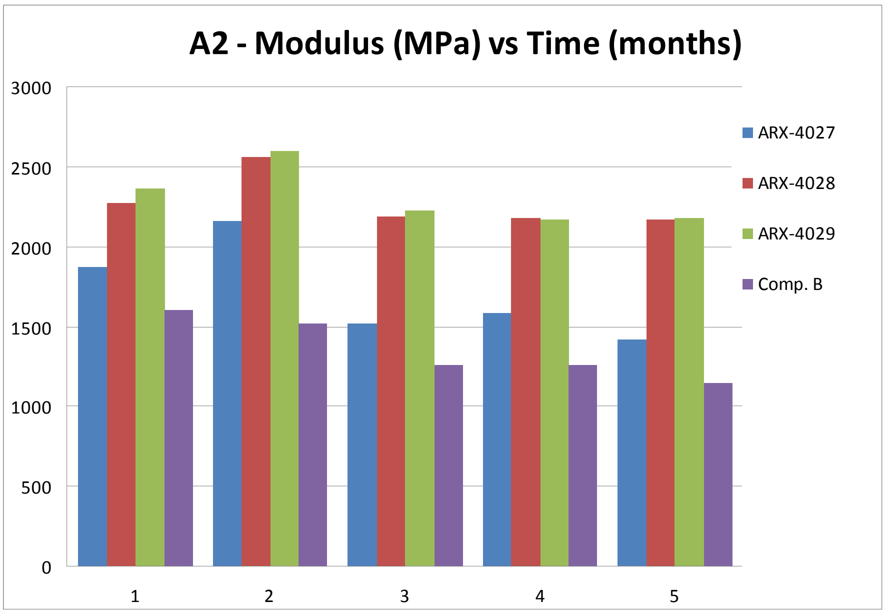

Figure 8: A2 ageing profile for modulus (MPa)

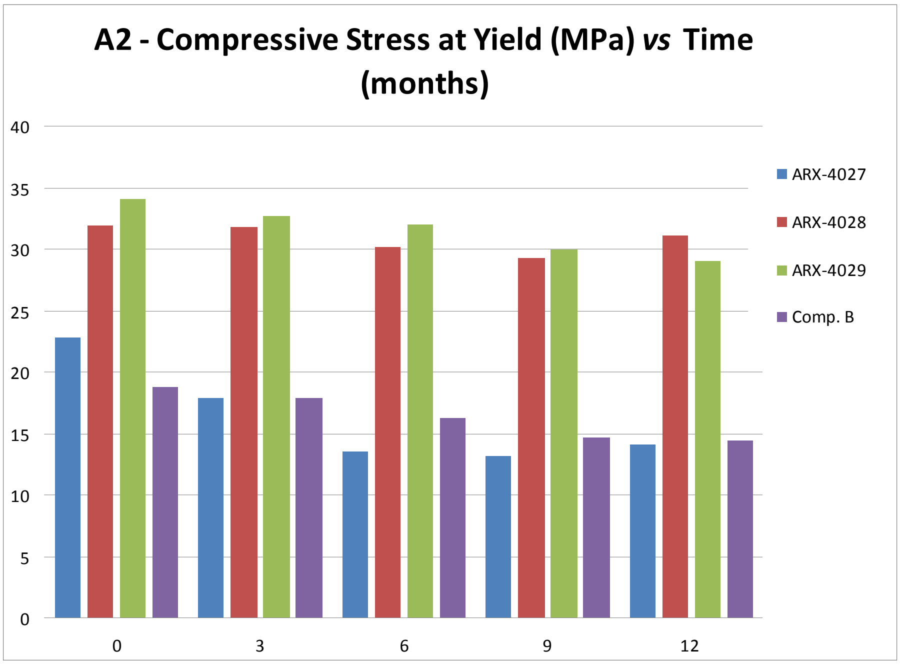

Figure 9: Comparison of compressive stress (MPa) for the A2 ageing profile

6. Conclusions & Further Work

Three DNAN-based melt cast formulations (ARX-4027, 4028 & 4029) were subjected to accelerated ageing conditions for 12 months. As a reference, Composition B samples were also aged similarly. The ageing conditions included two regimes: one set of pellets per formulation were aged at 60 ºC and ambient humidity, and another set of pellets according to the A2 diurnal cycle which involves a cyclic temperature and humidity profile and is defined in DEF(AUST)5168 [8].

For all DNAN-based ARX formulations and reference explosive (Composition B), ageing results were analysed by ingredient composition, density and mass loss, sensitiveness properties and mechanical properties (maximum load, modulus and compressive strength) at both ageing cycles. Based on the findings, an assessment was made on the suitability of these DNAN-based formulations as melt-cast explosive replacements.

Results from both the 60 ºC and A2 testing cycle showed that the DNAN-based melt cast ARX formulations aged better than when compared to the reference explosive, Composition B. Generally, there was slight degradation observed at both cycles in component ratios and mechanical strength. It is also evidently clear that the A2 cycle ages the formulations less harshly than the 60 ºC ageing cycle and this can be attributed to presence of both higher humidity and lower ageing temperatures for the A2 cycle compared to the dry conditions encountered for the 60 ºC cycle.

For all DNAN-based formulations there was negligible change in impact, friction, electrostatic discharge and thermal testing (both DSC and temperature of ignition) over time. These results highlight the ability of the ARX formulations to withstand temperature cycling and not become sensitive to various stimuli.

Accelerated ageing tests reveal that the DNAN melt-cast explosives offer improved ageing characteristic profiles compared to the traditional TNT-based explosive fills such as Composition B. Coupled with their insensitive properties to various stimuli (shock, thermal, etc.) these formulations would make good candidates for IM fills.

One potential area that needs to be assessed for these formulations involves testing for irreversible growth; a phenomenon exhibited by DNAN itself as well as other DNAN-based melt cast explosive fills such as IMX-101 and IMX-104. IMX-101 is a BAE (US) DNAN-based IM melt cast explosive fill designed to meet TNT performance specifications and contains DNAN, NTO and nitroguanidine (NQ) [11]. IMX-104 is also produced by BAE (US) and was formulated to have near or equivalent performance to the traditional Composition B legacy explosive with concomitant improvements in IM benefits [12]. IMX-104 contains DNAN, NTO and RDX.

Both of the above formulations exhibit irreversible growth of the fill; and is characterised by a volumetric expansion of the explosive fill (growth of DNAN) that could potentially pose problems in the fuze wells of artillery/mortars rounds[2]. Furthermore, the IMX formulations have also been found to form a powdery, chalky coat on pellet surfaces after 40 thermal cycles [14]. The ARX formulations discussed within this report should be tested for any irreversible growth and compared against the IMX explosive fills.

Other future work could include testing the hazards properties of aged DNAN based melt cast formulations for shock sensitivity via the Large Scale Gap Test (LSGT). This could provide knowledge towards the potential for aged DNAN based formulations to withstand shock. It should be noted that the US have tested shock sensitivities by the Expanded LSGT (ELSGT) for both IMX-101 and TNT after thermal cycling and that IMX-101 was found to maintain superior shock sensitivity over aged TNT explosives [14].

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[2] The Irreversible Growth Test involves thermal cycling 1″ x 1″ bare pellets between -54 ºC to +71 ºC according to AOP-7 [13] in an oven. The advisory criteria in AOP-7 states that the amount of growth be no more than 1%. Samuels [14] lists the following figures: IMX-101 has 8.00% average volume change, IMX-104 5.26%, Comp. B 8.46% & TNT 3.10%. The fact that Comp. B has a greater volume change than either IMX-101 or IMX-104 may suggest that there is likely to be no inherent problem with the IMX formulations.

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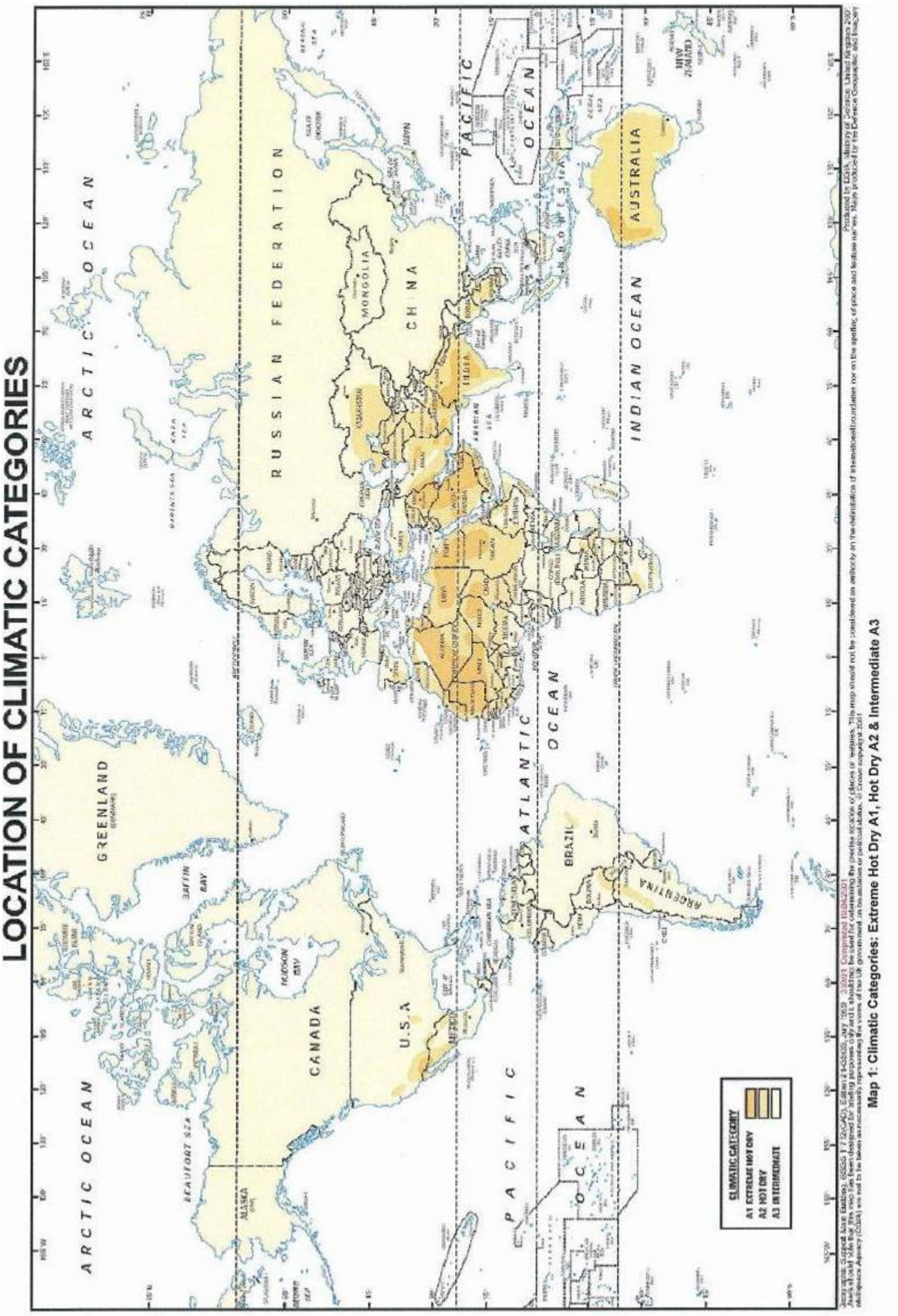
Appendix A: Location of Climatic Categories A

A.1. View of World Map with A1-3 Climatic Category [8]



Appendix B: DSC Thermograms

Figure 10: DSC Trace of ARX-4027 at 60 °C Ageing Cycle for 0, 3, 6, 9 & 12 months

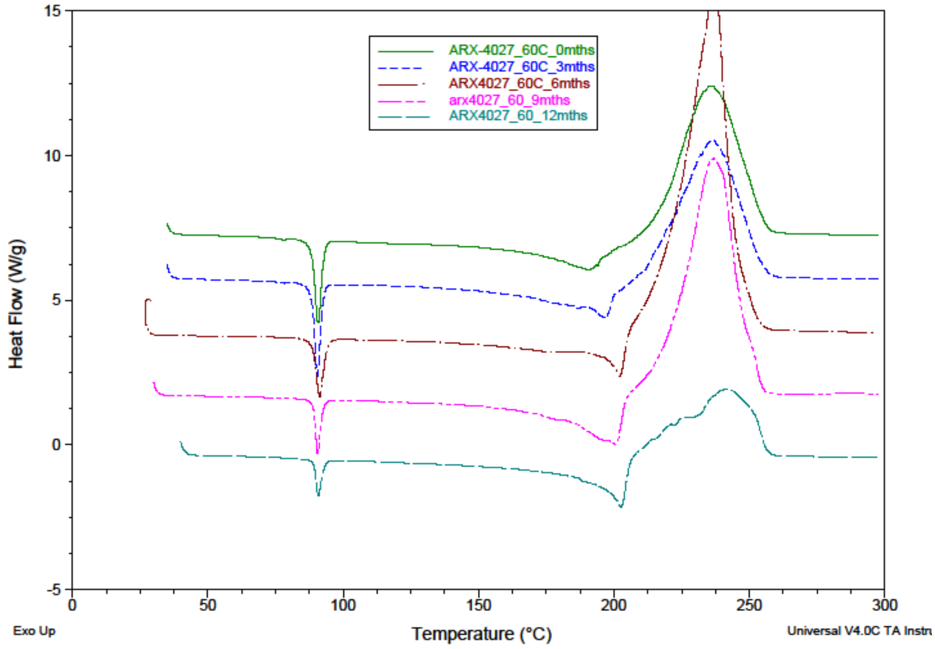


Figure 11: DSC Trace of ARX-4027 at A2 Ageing Cycle for 0, 3, 6, 9 & 12 months

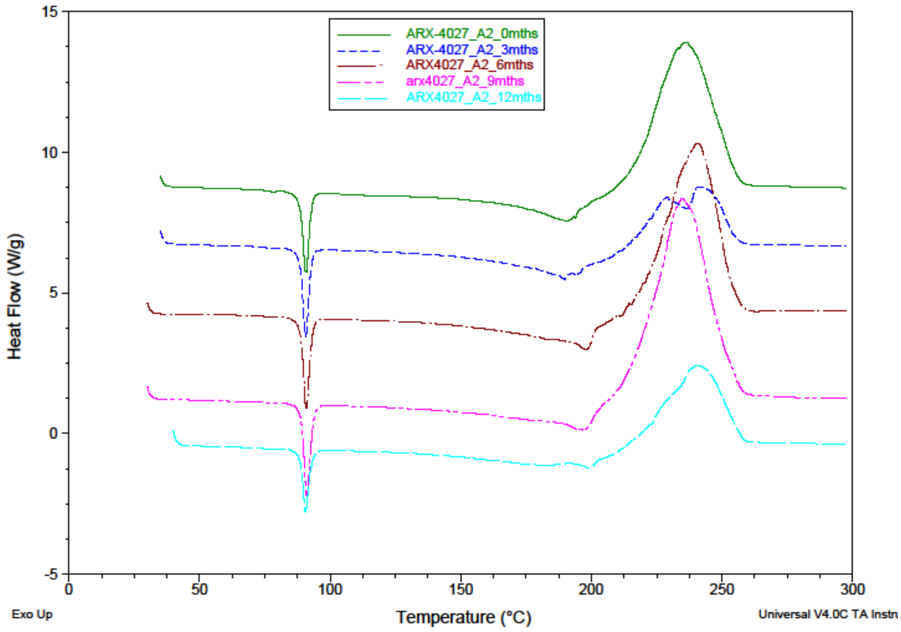


Figure 12: DSC Trace of ARX-4028 at 60 °C Ageing Cycle for 0, 3, 6, 9 & 12 months

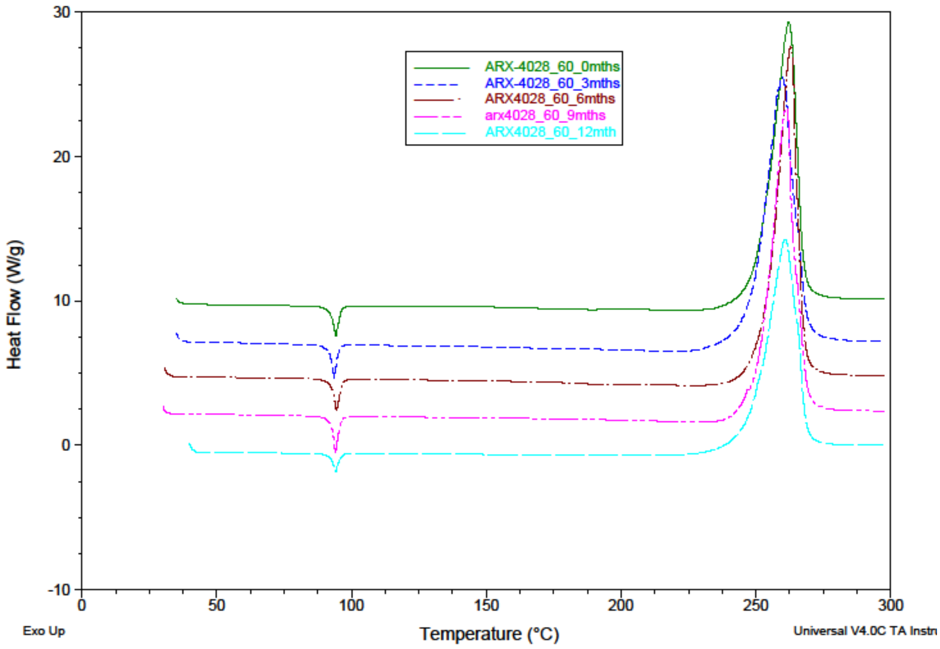


Figure 13: DSC Trace of ARX-4028 at A2 Ageing Cycle for 0, 3, 6, 9 & 12 months

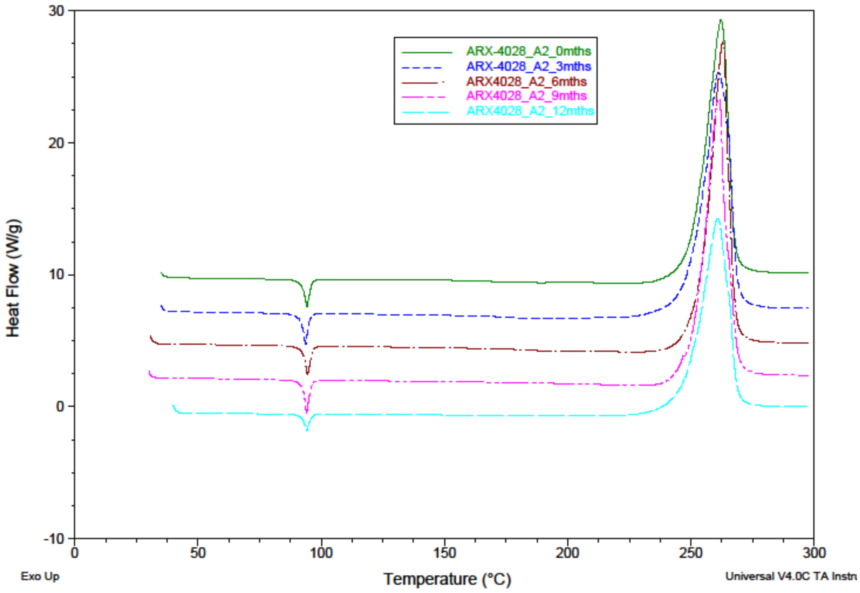


Figure 14: DSC Trace of ARX-4029 at 60 °C Ageing Cycle for 0, 3, 6, 9 & 12 months

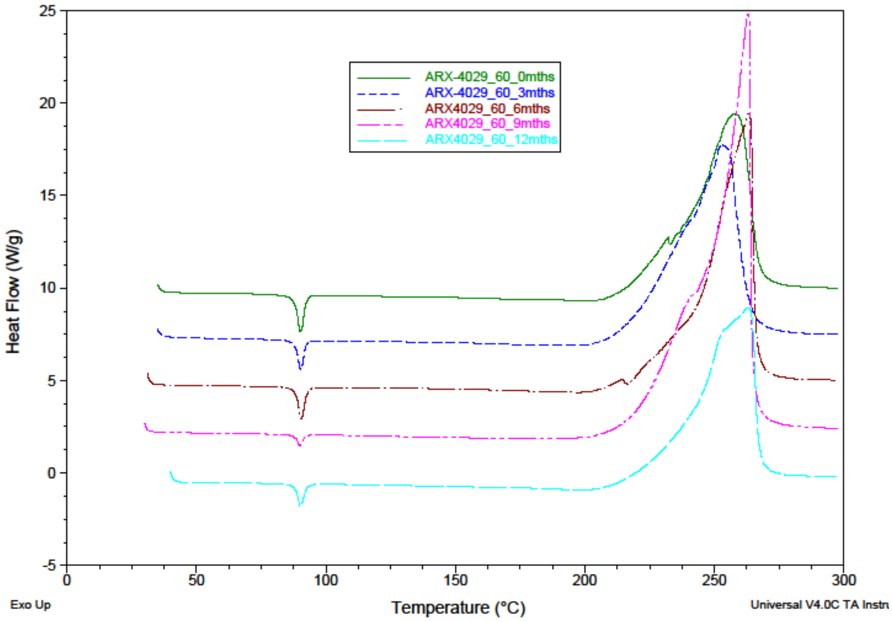
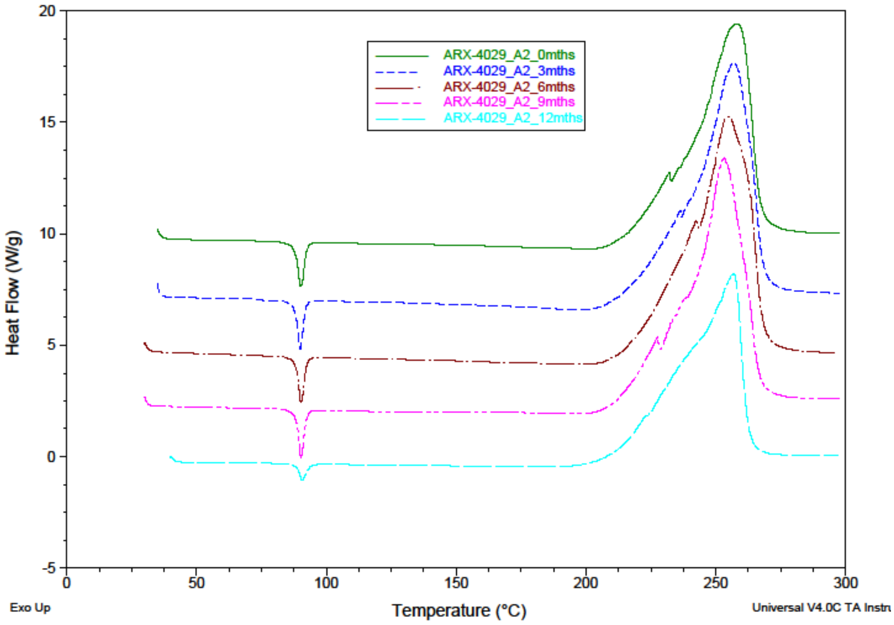


Figure 15: DSC Trace of ARX-4029 at A2 Ageing Cycle for 0, 3, 6, 9 & 12 months



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19. ABSTRACT

DNAN or 2,4-dinitroanisole is a new melt-cast matrix that replaces traditional TNT based melt-cast explosives. Aside from sensitiveness improvements, the use of DNAN allows for the continued operation of existing melt-cast facilities (for example the Australian Munitions plants located at Mulwala and Benalla) without the need for major plant modifications. Weapons and Combat Systems Division (WCSD) has developed several DNAN based formulations that have been extensively characterised and in an effort to better understand the ageing properties of these formulations, an accelerated ageing program was undertaken. Testing was conducted under two different ageing conditions; the first test condition was conducted at a constant 60 °C with ambient humidity and the second test condition was the A2 diurnal cycle (representative of the MEAO climate). Analysis of the ingredient composition, sensitiveness, mechanical and thermal properties was made at 3-month intervals for a period of 12 months and results compared with conventional explosives similarly aged.

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