

# **Ageing Evaluation of Certain Reduced Sensitivity RDXs in a Formulation Based on a Wax Binder System.**

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## **ABSTRACT**

*ARDEC pursued an effort to develop melt cast formulations based on RDX that would possess improved IM characteristics. One element in this development was to minimize shock sensitivity. To this effect, some effort was made to incorporate some of the reduced sensitivity RDX products that had become available at the time. Of two products evaluated, both reduced sensitivity initially, but one seemed to change sensitivity with aging. This work was initiated specifically to measure the aging characteristic of the material that became more sensitive.*

*The two RS-RDX's considered were two separate lots of U.S. Bachmann process material, with its usual HMX impurity, as reprocessed by SNPE (now Eurenco, France, group SNPE) and one lot of the Woolwich process (no impurity) I-RDX<sup>®</sup> material from SNPE. A specific formulation was chosen, and all three RS-RDXs used to produce test charges. Equivalent, conventional U.S. Holston RDX based charges were also included. The primary test employed was the NOL Large Scale Gap Test (LSGT), as it is in this test that the shock insensitivity shows up. Typical aging studies were performed, and additionally, separate LSGT specimens were made from time-to-time that provided useful compliments to the data. Thus various aging data accumulated both for the RDX's in the formulation and with RDX stored in the box until just prior to formulation. Mostly actual testing occurred shortly after casting LSGT tubes or according to a rigorous aging schedule, but a few data used leftover formulation from casting earlier tests that was saved and stored awhile. The two lots of reprocessed material were produced about a year apart. So changes in these could be studied as a function of time since reprocessing; their timeline progression over some 1000 days is compared. Results will be presented and data showing the long-term shock sensitivity trend of these formulations will be shown.*

## **INTRODUCTION**

During 2000, the US Army ARDEC investigated various RDX based explosive formulations as potential replacements for artillery rounds, which were typically loaded with TNT. One family of formulations selected for more advanced development were formulations that consisted of RDX mixed with various wax binder systems. These formulations did not contain any TNT; the binder system was entirely inert. They had the advantage that they could be melt cast loaded using existing TNT loading facilities.

During development, the I-RDX<sup>®</sup> low shock sensitivity RDX from SNPE (now part of Eurenco) was evaluated to determine its benefit in these formulations. The insensitiveness of I-RDX<sup>®</sup> is explained as resulting from a proprietary final recrystallization step, where parameters are very carefully controlled to produce high quality crystals. No claim has been made that I-RDX<sup>®</sup> has different properties with respect to types of initiation other than shock-to-detonation transition (SDT). So its special property is primarily observed when it is incorporated into a formulation and shock initiation studies performed. It has been reported elsewhere (ref.1) that not every formulation brings out its special shock insensitiveness. In some cases it acts no different from other RDX products. The feedstock for I-RDX<sup>®</sup> is RDX made using the Woolwich process (Type I); there is no HMX impurity in the feedstock. Part of the evaluation was to see if the proprietary processing could make a kind of "I-RDX", later designated

HIRDX, using typical U.S. Bachmann process RDX (Type II) as feedstock. Bachmann process RDX always has HMX impurity from 5 to 17 wt%. An advantage of the Bachmann process is it lends itself to high volume production and is facilitated in the U.S.

A particular experimental formulation (not developed for production) utilizing 83 wt.% RDX was selected for the RDX evaluation. It was produced in three versions, nominally designated as PAX/AFX 194, with -194A signifying use of HRDX (the normal Holston product), -194B use of I-RDX<sup>®</sup> and plain -194 use of HIRDX. Ratios of Class I and Class V RDX were essentially the same in -194A and 194 (i.e. 3/1), while there were 5% more fines in -194B (i.e. 2/1). This small change helped with casting for the -194B and the rationale was I-RDX<sup>®</sup> had slightly more fines in its Class I than the others, even though it met specifications. It turns out that the I-RDX<sup>®</sup> in the PAX/AFX 194B nearly doubled the LSGT shock insensitivity from that of PAX/AFX 194A and the same was nearly true for PAX/AFX 194 (made with HIRDX). Thus, PAX/AFX 194 development was pursued further. However, after a while it became apparent the initial good insensitivity results disappeared, and shock sensitivity degenerated to become similar to PAX/AFX 194A. The work reported here was performed to quantify this deterioration and attempt to identify the causes.

## BACKGROUND

This aging study involved single Class I and Class V lots of HRDX and I-RDX<sup>®</sup>, but two different lots of HIRDX. HIRDX Lot #1 was received at ARDEC in December 2000 and HIRDX Lot #2 received in January 2002. Some of the Class I HRDX was used to make the Lot #1 HIRDX. The Lot #2 HIRDX was made from a different source of HRDX approximately a year later. We assume that the ARDEC receipt date can be used as time zero when measuring the age of the HIRDXs. Significance of this will become apparent later. The ages of the “program standard” HRDX (Class I → 1993, Class V → 1994) was much older than the I-RDX<sup>®</sup>, which dated from 2000.

## NEED TO COMBINE LSGT RESULTS FROM TWO LABORATORIES

The formulation of interest was developed at AFRL, Eglin AFB, Florida sponsored by an ARDEC program (ref. 1). AFRL only worked with HIRDX Lot #1. Subsequent formulating and testing at ARDEC involved both HIRDX lots. Shock sensitivity was always measured using the NOL Large Scale Gap Test (LSGT, MIL-STD-1751A, Method 1041). Testing of HIRDX Lot #1 occurred sequentially, first at AFRL and then at ARDEC. Thus it became necessary to compare results between both laboratories, and identify any inconsistencies. A reference standard was needed. The single lot of I-RDX<sup>®</sup> used in this work, tested to be an extremely stable material in this formulation, without any hint of aging. Also, the PAX/AFX 194B formulation cast very well, making it easy to produce high quality LSGT tubes. Importantly, the Gap Test results between labs were quite comparable and consistent (figure 1). PAX/AFX -194B reliability displayed the same LSGT value at both AFRL and ARDEC. This is evidence that Gap Test values from both laboratories can be directly compared, at least with formulations that process easily.

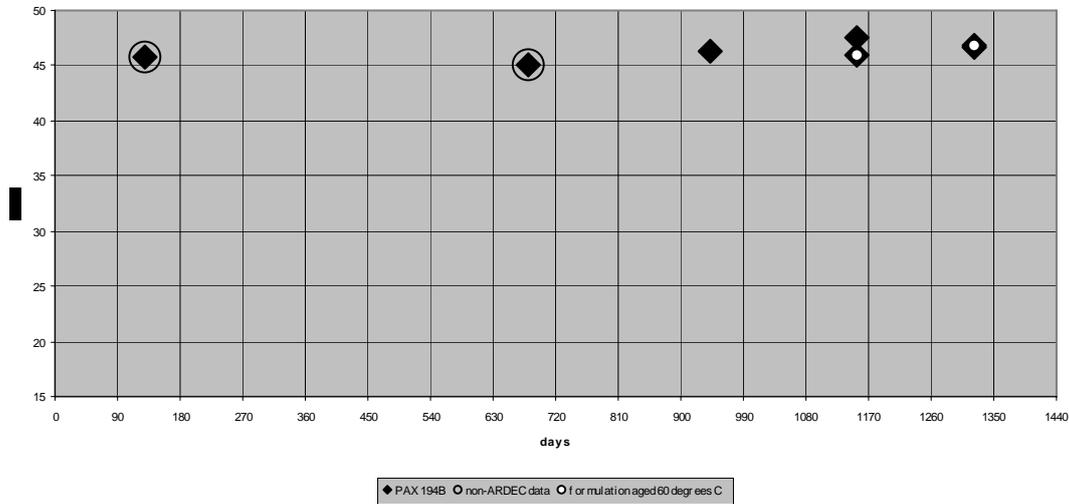


Figure 1. LSGT results for PAX/AFX 194B.

The comparison using PAX/AFX 194A was not as good. Both laboratories stated there were difficulties in producing LSGT tubes, because the melt would not pour very well. ARDEC had problems in getting desired densities, and densities were, in fact, low. Gap Test results are shown in figure 2. ARDEC values, while very consistent amongst themselves, run about 7~9 kilobars higher than AFRL values, where the same “project standard” HRDX was used. When PAX/AFX 194A was made using another HRDX lot at AFRL, it was within the range of the “project standard” version. Thus, for PAX/AFX 194A, results between laboratories are not equivalent and PAX194A always tests sensitive. A review did not find any explanation for these variations between laboratories, except possibly the difficulties in casting this material. It is significant that there was essentially no overlap of values between ARDEC and AFRL, indicating a systemic problem, not present with PAX/AFX 194B.

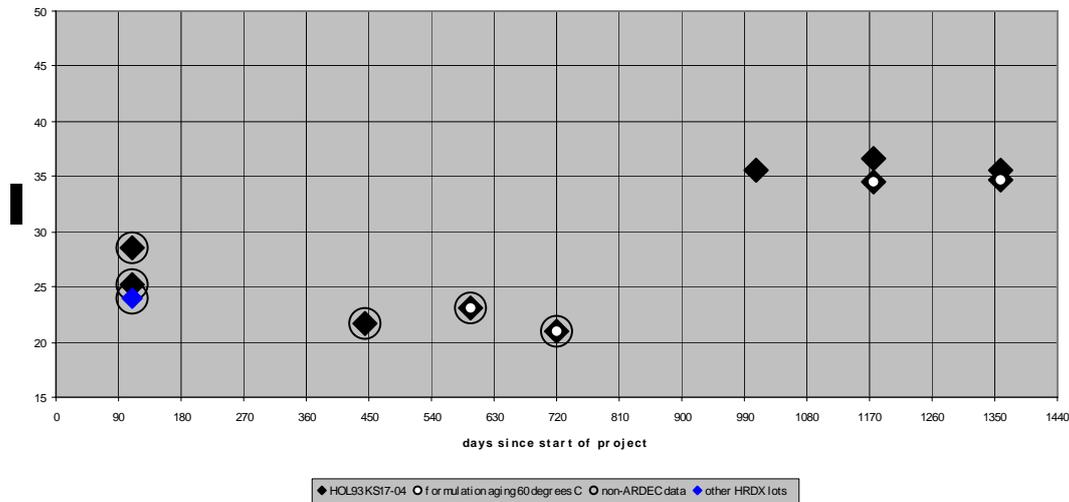


Figure 2. LSGT results for PAX/AFX 194A.

The justification for mingling AFRL and ARDEC data on the PAX/AFX 194, itself, is that this version fell into the “easy to cast” category and early values from Gap Tests where the RDX should not have yet been affected by age were very close between both laboratories. Rather like the PAX/AFX 194B, instead of the PAX/AFX 194A.

#### IDENTIFICATION OF THE PROBLEM

The various RDXs were assembled in December 2000 and formulation into various PAX/AFX 194s started shortly thereafter. Initially, LSGT values were PAX/AFX 194A, 28.9 kbars, PAX/AFX 194B, 45.6 kbars and PAX/AFX 194, 43.1 kbars. So there is a nominal 34% drop in shock sensitivity using insensitive RDX. Subsequently, 155 mm artillery projectiles were cast filled with all three variants of PAX 194 and subjected to a one-on-one SD test (ref. 2), an intermediate test that is often used to evaluate formulations in larger scale than the LSGT, but less than the full tactical configuration. In this test, subject projectiles are placed a fixed distance apart, and various thicknesses of standard barrier materials employed to find a threshold thickness for detonation transfer. Results showed a satisfactory linear correlation between barrier thickness and the LSGT results. Some time later, a new LSGT was performed using leftover material from the loading of the projectiles. The PAX/AFX 194 (HIRDX), at 24.7 kbars, now did not display any shock insensitivity better than PAX/AFX 194A. At this point other remaining supplies of HIRDX Lot #1 or leftover PAX/AFX 194 formulation were quickly tested and all showed the increased shock sensitivity. It appeared that HIRDX now was at least as sensitive as HRDX.

#### RDX CHARACTERIZATION

##### PARTICLE SIZE DISTRIBUTION

When the HIRDX shipments arrived at ARDEC, particle sizes were measured (ref. 2). One measurement for HIRDX Lot #1 displayed an unusually flat topped distribution, centered about 200~300  $\mu\text{m}$ , with a fairly small auxiliary peak off to the side at around

50  $\mu\text{m}$ . A companion measurement at another laboratory has a broad peak near 300  $\mu\text{m}$ , but data was cutoff where the auxiliary peak would have been. In the report, it was mentioned that the box from which the HIRDX Lot #1 sample came was suspect, and was later quarantined along with two other boxes from the shipment. Result for the “project standard” Class I HRDX had a typical curve peaking at 190  $\mu\text{m}$ , with auxiliary side peak around 50  $\mu\text{m}$ . The I-RDX<sup>®</sup> showed a very bimodal curve with peak at 185  $\mu\text{m}$  and auxiliary peak at 50  $\mu\text{m}$ . All these distributions have some particles greater than 500  $\mu\text{m}$ . The Class V HIRDX and I-RDX<sup>®</sup> have very similar, more typical looking curve centered about 11~15  $\mu\text{m}$ . Class V I-RDX<sup>®</sup> is ground Class I, as the propriety process does not produce Class V directly. The same is true for Class V HIRDX. The Class V HRDX has its peak at 22  $\mu\text{m}$  and lacks the very fine dust of the ground materials. At this time there was no HIRDX Lot #2.

Near the start of this aging study (early 2002), particle sizes of the RDX ingredients were again measured using a Microtrac analyzer (ref. 3). The mean particle sizes of all the Class I RDXs were similar, ranging from 230 to 270  $\mu\text{m}$ . These new measurements did not see any flat-topped nature to the HIRDX Lot #1 distribution. Both HRDX and I-RDX<sup>®</sup> had a noticeable auxiliary peak in the small particle region, about at 45  $\mu\text{m}$  for I-RDX<sup>®</sup> and 50~100  $\mu\text{m}$  for the HRDX. No such feature was visible for either HIRDX Lot. Overall, the HRDX particle size was just slightly larger than that of the others. There was similar variation with the Class V material, with HRDX being slightly larger. The mean size for HRDX was about 44  $\mu\text{m}$  while all the others were closer to 22  $\mu\text{m}$ . The Class V I-RDX<sup>®</sup> overlapped with the side peak seen the Class I I-RDX<sup>®</sup>, while there was no overlap with HRDX or the HIRDXs. There is not much difference between HIRDX Lot #1 or Lot #2 for either Class I or Class V.

The HIRDX Lot #1 particle size distribution can also be compared with that initially taken at SNPE during production of Lot #1, Class I HIRDX (ref. 4). The distribution peak occurred at 389  $\mu\text{m}$  compared to a 290  $\mu\text{m}$  peak from ARDEC measurements. The lack of any auxiliary small particle peak noted in ARDEC data was also evident in the original SNPE data. Both labs observed the presence of some large (around 500  $\mu\text{m}$ ) particles. For reference, SNPE measured a peak at 195  $\mu\text{m}$  for the incoming “project standard” HRDX compared to ARDEC measuring 230  $\mu\text{m}$ . Both laboratories observed the auxiliary small particle peak on the tail of the distribution of HRDX; but SNPE observed it around 18  $\mu\text{m}$ , while ARDEC saw it at around 50~100  $\mu\text{m}$ . From this, the HIRDX Lot #1 does not appear to have changed up to 2002, even though its shock sensitivity had changed by that date.

#### CRYSTAL MORPHOLOGY

SEM photos (refs. 2,3,4) of the various RDXs were done during manufacturing, early in the life of HIRDX Lot #1 and again at the initiation of this program (Spring 2002). All revealed general similarities between HIRDX and I-RDX<sup>®</sup> with only subtle differentiating characteristics, such as a more angular appearance to the HIRDX lots. Matching refractive index immersion microscopy was done in both refs 2 & 3 and the reduction of internal crystal defects of HIRDX Lot #1 over HRDX is documented.

Generally, HIRDX crystals are of superior quality, without so many internal voids as HRDX, though I-RDX<sup>®</sup> is somewhat superior to HIRDX in all respects. Even so, the initial SNPE report indicates HRDX density matches HIRDX Lot #1; one would have expected the better crystals would have caused higher density. The photos in reference 3 were performed at the beginning of the aging study in 2002, so the HIRDX Lot #1 had “gone bad”, but the Lot #2 was still “fresh.” The Lot #1 images, which could be compared with the earlier photo in reference 2, didn’t show as much surface pitting, but this artifice is somewhat variable over the collection of HIRDX crystals, so could just be due to the particular image section. Basically SEM did not distinguish any obvious differentiating features between the two HIRDX lots and they both resemble the I-RDX<sup>®</sup>. Immersion microscopy was not done as part of the aging study.

#### IMPACT SENSITIVITY

Impact sensitivity (ERL, MIL-STD-1751A, Method 1012) basically showed similarity between all the RDXs and aging them for 6 months at 60 degrees C made no difference. The value for HIRDX Lot #1 was still the same as ARDEC measured when Lot #1 was received. Ditto for the I-RDX<sup>®</sup>. There was no distinction between any of the RDXs even when kept in the formulation for 12 months at 60 degrees C. During this 12-month period, the HIRDX Lot #2 (not Lot #1) showed sensitization in the LSGT. BAM friction was equivalent between all the RDXs, and retests at 6 and 12 months, using both ambient and hot storage, indicated no change. This was true of the RDX powders, themselves, and when in the PAX/AFX 194 formulations.

#### ENERGETIC FRACTION

The aging study performed analysis to determine energetic fraction in each formulation. Indicated proportions of ingredients met the specification. In particular, all were very close to the required 83 wt. % RDX (reported values ranged between 80.86 to 83.65 wt.% RDX).

#### MELTING POINT

Melting point data are in Tables 1 & 2. Data is taken from references 3 & 4 and from Prof. Oxley’s results (the “URL” column) from reference 5. URL values are from DSC thermograms, so they are not exactly comparable to the other data, which was taken using the method from MIL-DTL-389D. The melting points for both HIRDXs were higher than for HRDX, and, although not shown, lower than the HMX free I-RDX<sup>®</sup>. Lot #2 seemed to have its Class I melting point drop 4 degrees over the 12 month observation period in the aging study, while its Class V increased by 3 degrees. Lot #1 didn’t show as much change. Only the Lot #2 had sensitization in the LSGT during the period over which melting points were measured.

Table I  
Melting Point  
Class I

	Initial at SNPE 2000	Received at ARDEC 2001	ARDEC Start of aging program 2003	URL Start of aging	6 month ambient	6 month 60 °C	12 month ambient	12 month 60 °C
HRDX	197	191	192	189~192	190~191	190~191	190~191	190~191
HIRDX/#1	198	198 (also 196 from different box)	194		194~195	194~195	192	192
HIRDX/#2			195	201~201	193~195	193~195	190	191
I-RDX <sup>®</sup>		203	203		203~204	203~204	203	203

Table 2  
Melting Point  
Class V

CLASS V	Initial at SNPE 2000	Received at ARDEC 2001	Start of aging program 2003	6 month ambient	6 month 60 °C	12 month ambient	12 month 60 °C
HRDX		193	194	192~193	192~193	192~193	192~193
HIRDX/#1		191 (also 195)	193	192~193	192~193	191~192	191~192
HIRDX/#2			194	194~196	194~196	196	197
I-RDX <sup>®</sup>	203		204	203~204	204	201~202	201~202

According to reference 6.5 wt.% HMX in RDX should drop the melting temperature by 2.71 °C, while 15 wt.% HMX drops it by 8.33 °C. Significant HMX impurity in all but the I-RDX<sup>®</sup> is indicated by their lower melting points. And both Class I HIRDX lots seem to melt at lower temperature with aging. But, only the Lot #2 displayed increased sensitization over the same time period, which was well beyond where Lot #1 had become sensitive and Class V had an opposite trend. No obvious conclusion seems justifiable, except that the reprocessed HIRDX lots do not have consistent melting temperature compared to HRDX or I-RDX<sup>®</sup>. This could indicate nonhomogeneous distribution of HMX impurity within the HIRDX lots.

#### DETERMINATION OF HMX IMPURITY

The most obvious difference between HIRDX and I-RDX<sup>®</sup> is that HIRDX retains some HMX impurity because it is made from Type II, Bachmann RDX. The HMX content of

the RDXs have been measured multiple times during their history. Initially, SNPE (ref. 4) measured the HMX impurity with HPLC just before and just after reprocessing part of HIRDX Lot #1. After reprocessing, the HMX content was reduced (10.3 % compared to the original 13.6%), but there was still significant impurity. Note these are only Class I, although the HIRDX Lot #1 Class V was made by grinding the Class I.

Initial ARDEC analysis obtained values of only 2.9+/-0.1 wt.% for Class I HIRDX Lot #1, but 10.6+/-0.1 wt.% for Class V HIRDX Lot #1 compared with 9.3+/-0.6 % for the Class I and 10.6 % for the Class V “project standard” HRDX. The large difference between Class I and Class V HIRDX Lot #1 is surprising because the Class V consisted of ground Class I. The deviation from SNPE’s “project standard” HRDX Class I value is 4.3 wt.%.

When HPLC testing was done again, at a later time, for this aging program, only the HMX content for the formulation, itself, was recorded. When corrected to allow for the binder weight, baseline values for HIRDX Lot #1 RDX was 11.9 wt.% HMX compared to HRDX value of 11.75 wt.%. No difference. These values are global values for the 3/1 blend of Class I and Class V that is in the formulation. Values can be compared to the initial ARDEC values by combining the initial values for Class I and Class V in the 3/1 ratio to arrive at 4.8 wt.% for HIRDX Lot #1 and 9.625 +/- 0.45 wt.% for the HRDX. There is considerable disparity over the HIRDX Lot #1 HMX level.

Simultaneous to measurements done at ARDEC for the aging study, HPLC measurements were made at the University of Rhode Island (ref. 5) on selected samples (only Class I) split from those being used at ARDEC. There the baseline values were 2.9 wt.% for Class I HIRDX Lot #1 compared to 15.5 wt.% for “project standard” Class I HRDX. The 2.9% value agrees with that initially measured at ARDEC, but doesn’t seem to fit with the later value obtained with material removed from the PAX194 formulation. The value for Class I HRDX is a few percent high.

Complete aging study measurements are given in Table 3. Class I RDX samples were supplied to URI already labeled “6 months at 60 °C”, etc. Actual storage and heating was at ARDEC. ARDEC values from the formulation really are very steady. Values measured at URI indicate elevated HMX impurity in all 60 °C aged samples.

Table 3  
Weight percent HMX impurity from HPLC

	ARDEC From formulation time=0	URL Class I only time=0	ARDEC 6 month 60°C	URL 6 month 60°C	ARDEC 12 month ambient	ARDEC 12 month 60°C	URL 12 month 60°C
HIRDX Lot #1	11.88	2.9	11.89	6.6	11.88	11.46	5.4
HIRDX Lot #2	7.0	1.9	7.0	6.9	7.70	6.99	4.7
HRDX	11.75	15.5	10.64	14.5	12.14	11.72	15.6

Obviously there is a big change in the URL results between time = 0 and later. Complementary IR spectroscopy of these same samples also detected significantly higher HMX impurity in both the 6 month and 12 month samples, verifying the difference. In addition, the melting point of the time=0 HIRDX Lot #2 sample was very close to HMX-free RDX, indicating very little HMX contamination. This is in agreement with the HPLC value of only 1.9 %.

To summarize these impurity level measurements, from the beginning, measurements for HIRDX Lot #1 had large variations (+/- 61%), measuring less than 3 % impurity on two occasions. The HIRDX Lot #2 also had a wide range of impurity level. Measurements of standard HRDX didn't have such large variations (but still +/- 25%). When viewed all together, the data do not indicate obvious systematic changes in HMX impurity. A more likely explanation is that RDX lots are really not homogeneous in HMX impurity, and as samples are taken they end up with different amounts. The HIRDX lots seem to have noticeably more variation than is typical. This is consistent with the variations of melting point described above.

#### FORM OF THE HMX IMPURITY

X-ray diffraction indicates  $\beta$ -HMX crystals and RDX crystals. No odd crystal forms are evidenced. The HMX contamination seems to exist as  $\beta$ -HMX crystals. It was observed that melting of RDX with (a large amount) of HMX produces a certain amount of amorphous material upon subsequent cooling. But the evidence supports the conclusion that normal recrystallization does not produce odd forms.

#### HMX AS SOURCE OF SHOCK SENSITIZATION

It remains to consider on how presence of HMX might cause shock sensitization. There have been attempts to observe HMX sensitization of "insensitive" RDX. In reference 7, HMX was added to RDX both in PBX109 (64 wt.% RDX) and an Australian equivalent made with reduced sensitivity Australian RDX (shown to act like I-RDX<sup>®</sup> in shock sensitivity) at the 5% containment level, and there was no affect on sensitivity. That is, the more sensitive PBXN-109 kept its higher sensitivity, while the equivalent ARX-2014/M1 kept its lower sensitivity. But, in this case the HMX was simply blended in with RDX powder before mixing the formulation. Although the x-ray diffraction result

indicates HMX and RDX keep their identity, it is possible co-precipitated RDX with HMX is different.

Work was done during PAX/AFX 194 development to see if HMX was the sensitizing factor. AFRL used Holston Class I HRDXs that were selected to have various amounts of HMX impurity. None had zero HMX, but values were 5.9 wt.%, 7.3 wt.%, 8.7 wt.%, 10.5 wt.% and 16.9 wt%. These all were obtained by co-precipitation in the final recrystallization, not blended afterwards. The PAX/AFX 194 binder system was used to keep compatibility with other data, but because of the single Class of RDX, only 70 wt.% solids was feasible. All these formulations had nearly the same LSGT values; no trend with fraction of HMX was detected. However, these all were fairly insensitive, around the value typically expected for a quality formulation having 70 % solid (RDX or HMX). Clearly the binder dominated the LSGT. Thus the conclusion is that large amounts of HMX impurity didn't change things enough to alter the effect of excess binder.

So far, there is no evidence that HMX impurity sensitizes charges made with RDX, but the studies are limited.

#### GAP TEST RESULTS

The gap test results, the primary product from this study, are shown in figure 3, plotted in chronological sequence. Data from material aged at 60 °C are marked with a small white dot in the center. Obviously, the 6-month result is that which precedes the 12-month result on the plot; two companion points 90 days apart are part of a single aging test. Circles around the points are data from AFRL. A point is included to represent the one-on-one SD test on Lot #1, since this correlated nicely with the LSGT values then available. A point is included for a single Gap Test tube fired at a gap chosen to verify no sensitization drift had occurred from when its siblings were used to get a 50% point. The original design of experiments was set up to observe monotonic degradation of the HIRDXs with time and temperature. That wasn't what happened. As can be seen from figure 3, sensitization of PAX/AFX 194 made from Lot #1 was observed at AFRL, but not at ARDEC. However, the Lot #2 material did sensitize at ARDEC, and then seems to revert to its original properties later. There are no obvious trends from the 60 °C aging tests and material kept at ambient condition approximately undergoes the same process. Unfortunately, there is no data for the situation where stored bulk RDX is kept at 60 degree C and then used to make charges. Bulk storage was always at ambient

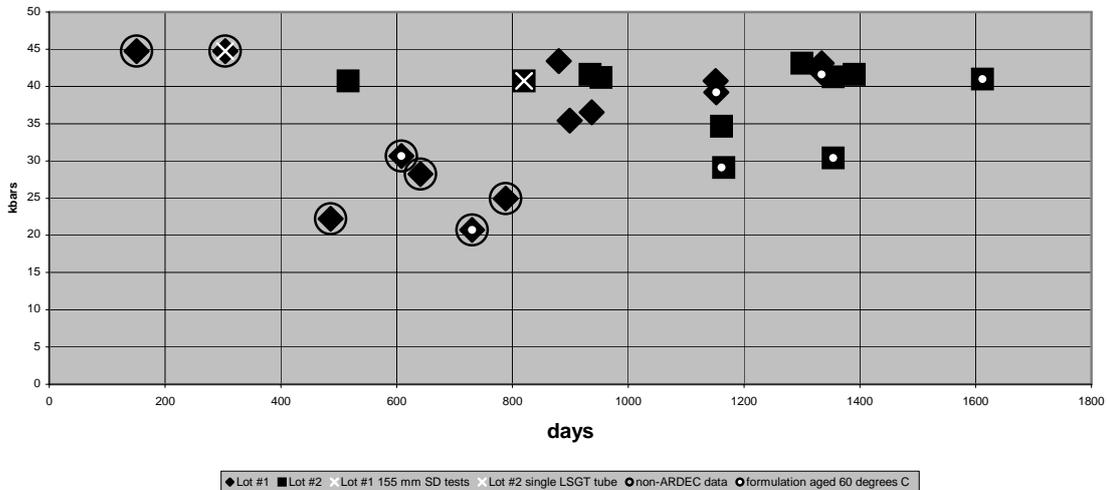


Figure 3. LSGT results for PAX/AFX 194 in chronological order.

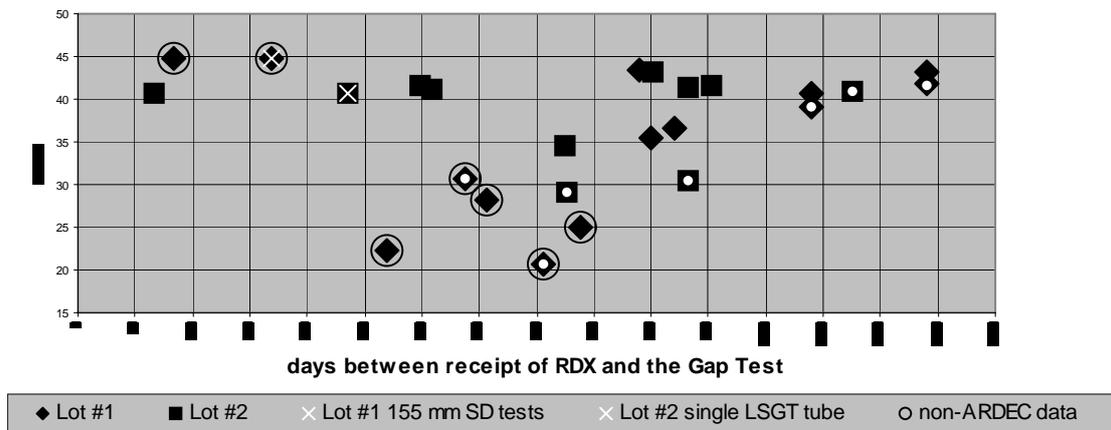


Figure 4. LSGT results versus date HIRDX was received at ARDEC

This data is more interesting when plotted using the age of the HIRDX (basically with Lot #2 data shifted by about a year) in figure 4. It appears the interval of sensitization for both Lot #1 and Lot #2 more-or-less overlap when measured from their individual time zero. Both lots seem to roughly go through a similar temporal sequence. The sensitization occurs after a certain time and lasts over some period, but eventually sensitivity returns to nearly the original state. This suggests the dichotomy between AFRL and ARDEC over Lot #1 occurred because AFRL stopped testing before the Lot

#1 recovered and ARDEC started testing after it had recovered. If this is correct, ARDEC completely missed the time during which Lot #1 was sensitive purely by chance.

Fortunately, there was enough variation in the times over which RDX was in the formulation to generate figure 5. Once in the formulation, the HIRDX is exposed to the various binder system components. The figure shows no obvious correlation of shock

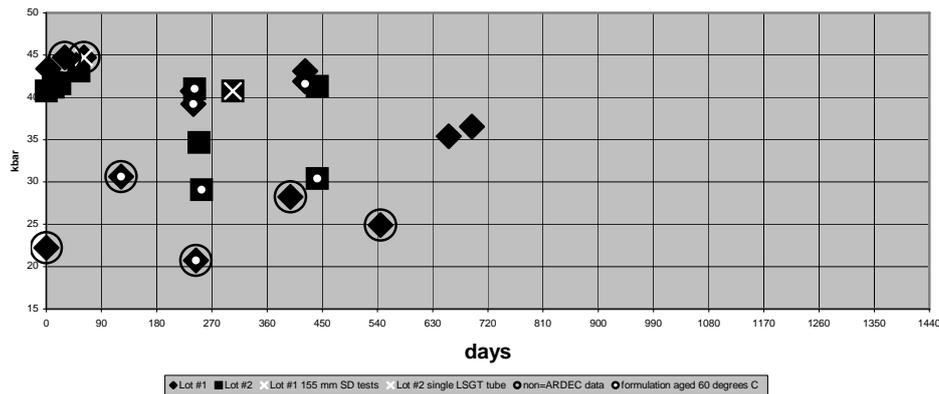


Figure 5. LSGT results versus time in the formulation.

sensitivity with how long the HIRDX is in contact with the binder. The same is true when data is plotted versus the time that the LSGT tubes were stored before testing. This includes all the intentional aging tests as well as incidental results because of scheduling changes and reclaiming previously cast material to make new LSGT tubes. Results show no seasonal effect, eliminating possible differences caused by winter weather. Also, there is no correlation between the age of the HIRDX and the time it was first cast into charges, which along with the result for time in formulation, implies the changes appear independent of whether the HIRDX is in the formulation or not. Apparently it is a process in the HIRDX, itself.

The seeming similarity of events when the year difference in manufacturing date is included argues against results just being random chance; say due to the demonstrated variations in HMX impurity. Having Lot #2 undergo its entire sequence at ARDEC suggests events were not caused just by having tests shift from AFRL to ARDEC. And the year difference in absolute dates (as opposed to relative age) argues against drift due to changes in operational procedures. However, the explanation for this sequence is not apparent. It is not known if the HIRDXs have reached some steady state or not. A more positive conclusion is that I-RDX<sup>®</sup> and other reduced sensitivity RDXs (Dyno-Nobel RS-RDX and probably also the ADI grade A RDX) don't show this behavior.

## CONCLUSION

Reprocessed Type II, Bachmann RDX is not stable, and undergoes shock sensitization and desensitization over time. The fact that sensitization was not permanent is a new observation, but there is no assurance further changes will not occur. None of the

ancillary laboratory testing surfaced any obvious distinctions, but unusual inhomogeneity with respect to HMX impurity in the reprocessed material was indicated. The suggestion of a temporal cycle to the sensitization greatly complicates the investigation, since the absolute time since manufacture becomes an important factor. Measurements must be done at the same time (within some leeway) to insure the same material is being tested. It is still strongly suspected the cause of this problem is the HMX impurity, but no mechanism is yet apparent.

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