Journal of Energetic Materials
Publication details, including instructions for authors and subscription information:
http://www.informaworld.com/smpp/title~content=t713770432

Composition Choice and Formulation of Three HMX-Based Research Explosives

G. T. Sutherland[a]; L. J. Brousard[a]; J. F. Leahy[a]; S. Prickett[a]; J. S. Deiter[a]
[a] Indian Head Division, Naval Surface Warfare Center, MD, USA

Online publication date: 17 August 2010

To cite this Article Sutherland, G. T. , Brousard, L. J. , Leahy, J. F. , Prickett, S. and Deiter, J. S.(2004) 'Composition Choice and Formulation of Three HMX-Based Research Explosives', Journal of Energetic Materials, 22: 4, 181 — 197
To link to this Article: DOI: 10.1080/07370650490893009
URL: http://dx.doi.org/10.1080/07370650490893009

Energetic Materials, 22: 181–197, 2004

ISSN: 0737-0652 print
DOI: 10.1080/07370650490893009

Taylor & Francis
Taylor & Francis Group

Composition Choice and Formulation of Three HMX-Based Research Explosives

G. T. SUTHERLAND
L. J. BROUSARD*
J. F. LEAHY+
S. PRICKETT
J. S. DEITER

Indian Head Division, Naval Surface Warfare Center, Indian Head, MD, USA

The composition and formulation for three research explosives having similarities to military explosives are described. The primary energetic ingredient in each is cyclotetramethylene-tetranitramine (HMX), whose particle size is limited to a range of 125–210 μm to reduce variations in shock reactivity and performance. The binder in each explosive is hydroxy-terminated polybutadiene (HTPB). The first composition contains only these two components. Aluminum with a nominal particle size of 5 μm is incorporated into the second composition. The third composition contains ammonium perchlorate (AP) with a nominal particle size of 200 μm in addition to the aluminum. The explosives are designed with features to

Address correspondence to G. T. Sutherland, Naval Surface Warfare Center, Code 920 Building 600, Indian Head, MD 20640, USA. E-mail: sutherlandgt@in.navy.mil
*Present address: Navy Medical Command, 8901 Wisconsin Ave., Bethesda, MD 20899
+Present address: US Air Force Research Laboratory, Munitions Directorate (AFRL/MNME) 2306 Perimeter Road, Eglin, AFB, FL 32542

allow for comparisons in shock reactivity and performance and to elucidate the roles of HMX, Al, and AP.

Keywords: HMX, ammonium perchlorate, metallized explosive

Introduction

Researchers have formulated explosives with changes in composition and ingredients to assist in shock reactivity and detonation property studies. Finger and coworkers [1] conducted cylinder expansion tests on 24 explosives based on cyclotetramethylene-tetranitramine (HMX). They determined the effect from the addition of a fuel, oxidizer, and in one case both a fuel and oxidizer on the metal acceleration. They found that only a lithium perchlorate/HMX composition outperformed an explosive highly loaded with HMX. Chunhua and Jing [2] conducted a study to determine the roles of various propellant components for shock reactivity. Materials tested included ammonium perchlorate (AP), mixtures of aluminum (Al) and AP, mixtures of AP and hydroxy-terminated polybutadiene (HTPB) binder, and a propellant containing Al, AP, and HTPB binder. From comparison of embedded gauge records, conclusions were drawn about the rate and amount of reaction from each binder component.

We have manufactured three research explosives, designated IRXs, that are similar to three types of Navy explosives. Like the work of Finger et al. [1] our compositions contained a nitramine and were geared to explosives rather than propellants as in the work of Chunhua and Jing [2]. IRX-1 explosive simply contains a nitramine, chosen to be HMX, in a polyurethane binder. IRX-3A adds Al, which is a fuel, to the simple composition. IRX-4 adds both the fuel and an oxidizer, chosen to be AP, to the simple composition.

Since the HMX is the principal detonating component in these explosives, each was manufactured from the same batch. This approach eliminated variations in particle size, shape, and defect concentration, which have been shown to affect detonation and shock reactivity properties. We define shock reactivity as the

rate of chemical reaction in an explosive caused by a shock wave. The reaction at each point in the explosive will depend on the local pressure, energy imparted by the shock wave.

Particle size effects have been studied in nitramine compositions based on both HMX and cyclotrimethylene-trinitramine (RDX). Simpson, et al. [3] performed gas gun experiments and wedge tests using fine (6 μm), course (60 μm), and very course (1700 μm) HMX/water compositions. For input pressures of 50 kbar in his gun tests, much less reaction was observed for the fine HMX/water composition. Bernecker [4] found (see his Figure 4) from comparing his run distance to detonation experiments for HMX/water compositions with run distance (wedge) tests of Simpson that HMX/water compositions composed of 60 μm particles would show the most shock reactivity for run distances of 12.5 mm. Particle size effects were studied by Moulard and coworkers [5, 6] in a variety of experiments on several research explosives having the same weight percentage of RDX in HTPB binder. The explosive with very coarse particles (428 μm) was more shock reactive at pressures below 75 kbar and had a larger failure diameter than an explosive containing fine particles (6 μm) and coarse particles (134 μm). Also, an explosive with coarse particles (134 μm) was more shock reactive at pressures below 95 kbar and had a larger failure diameter than the fine particles.

Particle shape was found by Van der Steen and coworkers [7] to affect the shock reactivity of bimodal RDX/HTPB explosives. The explosives contained 85% RDX and were subjected to both NOL large-scale gap tests (LSGTs) and run distance to detonation experiments. Their LSGT measurements show the minimum pressure required for detonation is 32 kbar for the explosive containing irregularly shaped particles and 37 and 39 kbar for explosives containing regularly shaped particles. Their run distance to detonation experiments showed that regularly shaped particles were less reactive than the irregular particles for pressures below 45 kbar.

Particle defects were found by Borne [8] to affect the shock reactivity. RDX was separated into two subbatches by flotation and used to make two 70/30 RDX/wax compositions. The less

dense RDX had an increase in both the volume and size of intragranular cavities. Explosive samples were impacted with a flyer plate, and the transit time was measured for the shock wave to travel through the sample. A faster wave speed implies that reaction of the sample was adding energy to the shock wave. For input pressures of 47 and 57 kbar, the transit times were lower for the explosive containing RDX with more cavities.

Composition Choice

General Requirements

The IRXs should employ ingredients used in making existing Navy explosives. This allows for comparisons of the shock and detonation properties of the research and Navy explosives. Also, the techniques for manufacturing the explosives with existing ingredients are well established.

The research explosives need to be free from very large (>500 μm) and fine (<10 μm) HMX particles. Large particles may disturb pressure measurements in light gas gun experiments, as the gauge pressure sensing area is about 1.5 mm wide. The gauge should measure pressure over an area containing many HMX particles so that macroscopic rather than mesoscopic (granular) properties are measured. Fine HMX particles are not a main constituent in most Navy HMX-based explosives [9]. For the IRXs the sieved range of HMX particle size was about 120–215 μm. This particle size also permits reactive growth studies at shock pressures below 50 kbar and run distances less than 12 mm, which is within the capability of our light gas gun experiments. Work of Simpson et al. [3] implies a mean HMX particle size greater than 60 μm is required to obtain the required shock reactivity properties.

For the gas gun experiments, the explosives need to be hard enough to machine into thin disks. For example, some samples were ∼70 mm in diameter and ∼6 mm thick.

Our explosives should be free from large binder voids. At present, the relationship of void contact or void size on shock reactivity is unknown for this type cast explosive [10]. In addition, voids will perturb gauge measurements much as large HMX particles

do. For gas gun experiments, radiographic and visual inspection was used to determine the amount and location of the voids.

The binder system should be one that has been characterized and used in other Navy explosives. The selected HTPB binder system is used in many cast explosives manufactured at our center [9]. The shock wave properties of the HTPB binder system were measured by Gupta and Gupta [11] and Chunhua and Jing [2].

Specific Compositions

The compositions of the IRXs in Table 1 are in terms of both weight percentage and volume percentage. We chose the composition of IRX-1, which is 70 weight percent HMX with HTPB, to be similar in nitramine loading and particle size to a RDX/HTPB composition studied by Moulard et al. [5, 6]. In addition, IRX-1 is similar to the Navy explosive PBXN-110. PBXN-110 [9, 12] which contains mostly large and some small HMX particles and allows for a larger nitramine loading than IRX-1. IRX-1 was formulated without plasticizer to maximize the hardness of the explosive.

The IRX-3 explosives were designed to be an aluminized version of IRX-1, containing the same 70 weight percent coarse HMX with 10 weight percent Al. IRX-3 was found to have many voids after casting; to limit these voids IRX-3A was formulated with additional plasticizer. IRX-3 explosives are similar to the Navy explosive PBXW-114 [9, 12], which contains large and small HMX particles, small Al particles, and HTPB binder.

We chose the composition of IRX-4 to contain 30 weight percent HMX, 16 weight percent Al, and 24 weight percent AP. The AP particles had a nominal particle size of 200 μm. An HMX loading of 30 weight percent was chosen, instead of a lower HMX loading, to obtain a failure diameter less than 5 cm. IRX-4 is similar to the Navy explosive PBXN-111 [9, 12], which contains large and small RDX particles, small Al particles, and AP particles. To maximize explosive hardness, IRX-4 like IRX-1 was formulated without a plasticizer.

Table 1
IRX explosive composition

Ingredient	Function	Weight/volume percentage			
		IRX-1	IRX-3	IRX-3A	IRX-4
Sieved HMX	Explosive	70.00/52.79	70.00/58.64	70.00/58.48	30.00/23.56
Al	Metal fuel		10.00/5.91	10.00/5.89	16.00/8.86
AP	Oxidizer				24.00/18.41
Polybutadiene, linear hydroxyl-terminated (R45 HT)	Binder polymer	27.39/43.67	13.69/24.25	10.89/19.23	27.40/45.49
Isodecyl perlargonate	Plasticizer		5.0/9.24	8.0/14.74	
Triphenylbismuth	Catalyst	0.01/0.01	0.01/0.01	0.01/0.01	0.01/0.01
Isophorone diisocyanate	Cross linking agent	2.60/3.53	1.30/1.96	1.10/1.65	2.60/3.68
HTPB (Total)		30.00/47.21	20.00/35.45	20.00/35.63	30.00/49.17

Explosive Preparation

Ingredients

To ensure constant HMX characteristics, Class A material from the Holston Army Ammunition Plant was sieved to remove very large and small particles. A 210 μm sieve, 125 μm sieve, and metal pan were stacked on each other and mechanically shaken. Only the HMX retained in the 125 μm sieve was used to make the research explosives. IRX-3 and an initial mix of IRX-1 were made from one lot of HMX. Samples from these mixes were used only to determine shock sensitivity from modified gap testing [13]. All other mixes to be used in subsequent tests were obtained from a different lot.

For this second lot a set of three specimens was taken from top, middle, and bottom of the container holding sieved HMX to make both IRX-3A and more IRX-1. Particle size for each specimen was obtained by scanning electron microscopy. The weight and number distributions displayed in Figures 1–2 are consistent for the two explosives, and the ranges and averages of particle size for each sampling are listed in Table 2. A photomicrograph of one specimen is shown in Figure 3. The particle size distributions show some HMX finer than 125 μm

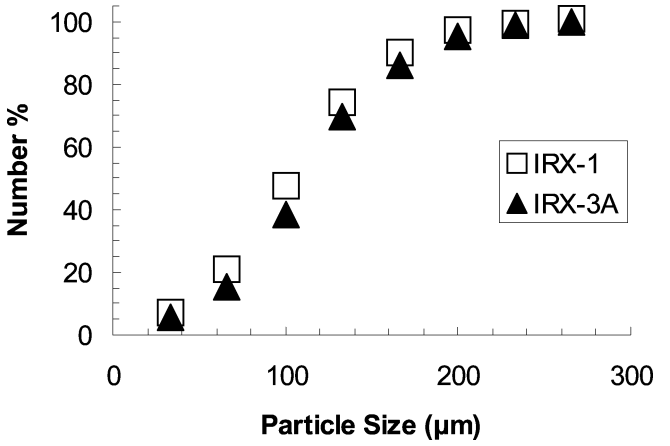

Figure 1. Histogram of particle size vs. number percentage.

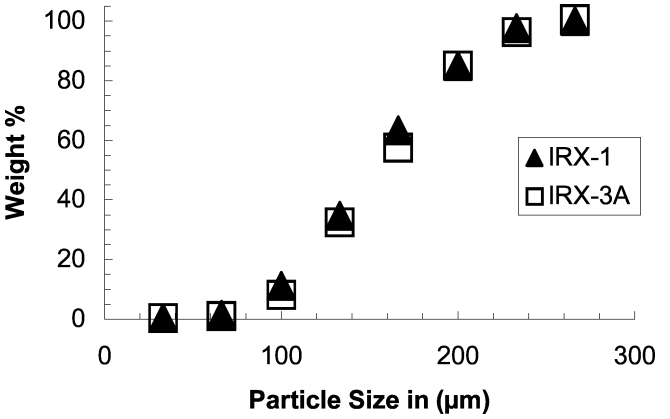

Figure 2. Histogram of particle size vs. weight percentage.

is present, indicating that the sieve runs were not performed long enough to eliminate all the fine particles. While no measurements were made for the HMX used to make the IRX-4, no inconsistencies were expected.

H5 Al with a particle size of ∼11 μm was used in IRX-3A and IRX-4. The AP used in IRX-4 has a particle size of ∼200 μm.

The HTPB binder (see Table 1) was composed of a binder polymer, cross-linking agent, and catalyst. The composition was chosen to minimize the voids present in the cast charges. More plasticizer was added to the binder for IRX-3A. No agents were added to retard oxidation by oxygen or ozone.

Mixing and Casting Procedure

IRX-1, IRX-3A, and IRX-4 were made at the former facility in White Oak, MD, USA. The explosive was mixed in a 5 gallon Baker-Perkins high-shear vertical mixer. The procedure for mixing ∼12 kg of IRX-3A is described below; the procedures for IRX-1 and IRX-4 were similar. The first step is to mix the HTPB R45HT, isodecyl perlargonate, triphenylbismuth, and Al for 15 minutes. One-third of the HMX is then added; the resulting mixture is mixed for 20 minutes. This step is repeated two more times. The isophorone diisocynate is added, and the mixture is

Table 2
HMX particle size distribution

Sample location	Number of particles measured	Range of diameter measured (μm)	Average diameter by number (μm)	Average diameter by weight (μm)
IRX-1 top	600	27–264	120	138
IRX-1 middle	50	30–250	130	–
IRX-1 bottom	50	30–250	130	–
IRX-3A top	600	29–259	129	145
IRX-3A middle	50	30–250	130	–
IRX-3A bottom	50	30–250	130	–

Location refers to the part of the container HMX sample came from. This container contained the HMX used to make each mix.

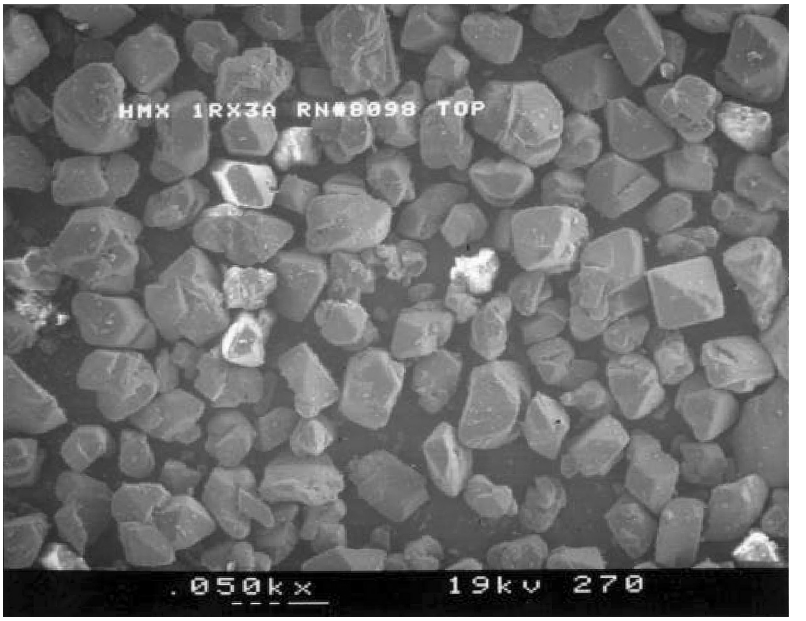

Figure 3. Photomicrograph of HMX used in IRX explosives.

mixed for an additional 15 minutes. The temperature of the mix was raised from 33.8 to 61.6°C. Viscosity measurements appear in Table 3 and were measured one half hour after the isophorone diisocyanate was added.

After mixing, the material is cast under vacuum, to remove or limit voids, into various cylindrical molds. The molds are placed in an oven to for curing the explosive. The charges were then machined to sizes required for a particular test as needed.

Characterization

Density and Hardness Measurements

Shortly after curing, hardness and density measurements were performed and appear in Table 3. The density was found by measuring the volume and mass of cast explosive pieces. Comparison of the density measurements with that of the theoretical maximum density show that we obtained good quality charges.

Table 3
Casting measurement and hardness measurements

Explosive	Viscosity (10^3 poise)	TMD (g/cm^3)	Initial density (g/cm^3)	Initial Shore A hardness	Aged Shore A hardness
IRX-1	2.3	1.437	1.43	66	88–92
	2.6	1.437	1.42	63	
IRX-3A	3.2	1.594	1.58	44	63–81
	4.3	1.594	1.59	48	
IRX-4	0.4	1.496	1.50	59	88–91
	0.5	1.496	1.50	61	

The hardness of 7 cm diameter disks of IRX-1, IRX-3A, and IRX-4 were measured about three years after casting. The values obtained are listed under the aged hardness in Table 3. We report the values obtained for measurements over various locations on the disk surface. The hardness has increased over time and is likely caused by postcuring of the binder and possible plasticizer migration out of the IRX-3A sample. The binder may also be degraded because no antioxidants or agents to retard oxidation by ozone were added because testing was to originally to be done shortly after casting. This may also explain discoloration observed on the surface of the explosive samples.

Safety Data

To ensure that large quantities of explosives could be safely handled and manufactured, small-scale safety tests were performed. The tests performed on small amounts of each explosive included drop weight impact, friction, vacuum thermal stability (VTS), and differential scanning calorimetry (DSC).

Drop weights tests [12, 14] give a preliminary indication of ignitability of the explosive. The tests were conducted with the ERL Bruceton apparatus. Impact sensitivities are given in Table 4. Results for both IRX-1 and IRX-3A indicate impact sensitivities comparable to similar existing explosives. Our laboratory characterizes these explosives as having a low sensitivity to impact. IRX-4, like similar Navy explosives containing AP, has high sensitivity to impact. These results mean that IRX-4 has a higher probability that it will react in any operation where the explosive is ground or undergoes shear deformation than does IRX-1 or IRX-3A. Note that drop height of HMX is higher than for IRX-4.

The Bundesanstalt für Material Forschung und Prüfung[1] (BAM) friction test indicated that our IRX series explosives had low friction sensitivity. Our laboratory defines a BAM measurement of more than 80 N as indicative of low friction sensitivity. Evidently

[1]Federal Institution for Material Testing.

Table 4
IRX explosive safety data

Explosive	Drop height (cm)	BAM friction (N)
IRX-1	71	>360
IRX-3A	66	324
IRX-4	17[a]	122
HMX class A	19	80
TNT	78	–
PETN	10–14	36–56

[a]The drop test of IRX-4 was done with bare tools, meaning ground IRX-4 was placed directly on the drop test anvil. If the sample were placed on sandpaper, like the IRX-1 and IRX-3A, the sample would likely have a value less than 10 cm.

the high volume percentage of binder used in our IRX series explosives reduces the friction sensitivity of these explosives. As a reference, a BAM value of 80 N was measured for Class A HMX.

The thermal stability of our explosives was measured in DSC or VTS tests. For IRX-3A, a VTS value of 0.261 cc/gm/48 hr at 100°C was obtained and indicates the explosive has good thermal stability characteristics. As a reference, Composition B has a measured value of 0.3 cc/gm/48 hr. The results for the DSC test appear in Figures 4–6. An exotherm appears at ≈ 280°C for all explosives and corresponds to the reaction of HMX. Examination of Figure 4 shows that the addition of aluminum does not affect the thermal behavior. Comparisons of the features of Figures 4 and 6 for heating rates of 5–10°C per minute would imply that the thermal behavior was not affected a great deal by the addition of AP.

Conclusions and Suggestions for Future Work

Three research explosive compositions were formulated and cast. The explosives obtained were hard enough to be machined and had few voids. However, the lack of an antioxidant may have led to the dramatic increase in hardness (Shore A)

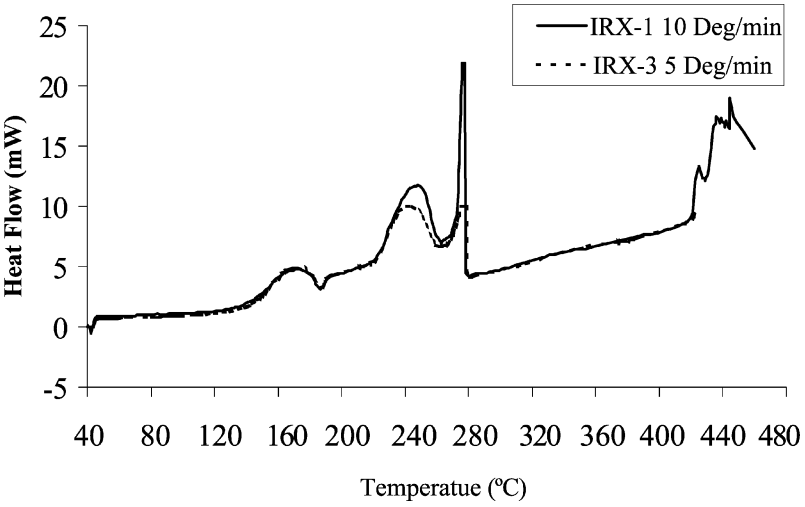

Figure 4. DSC results for IRX-1 and IRX-3. Traces digitized from a paper graph. Graph limits (data was "clipped") were ≈ 22 mW for IRX-1 and ≈ 8 mW for IRX-3.

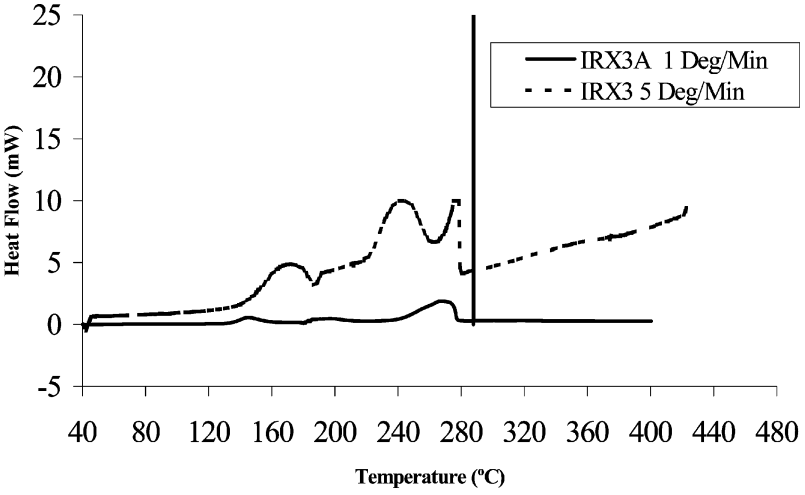

Figure 5. DSC results for IRX-3 and IRX-3A. Traces for IRX-3 digitized from a paper graph. Graph limit ≈ 8 mW for IRX-3.

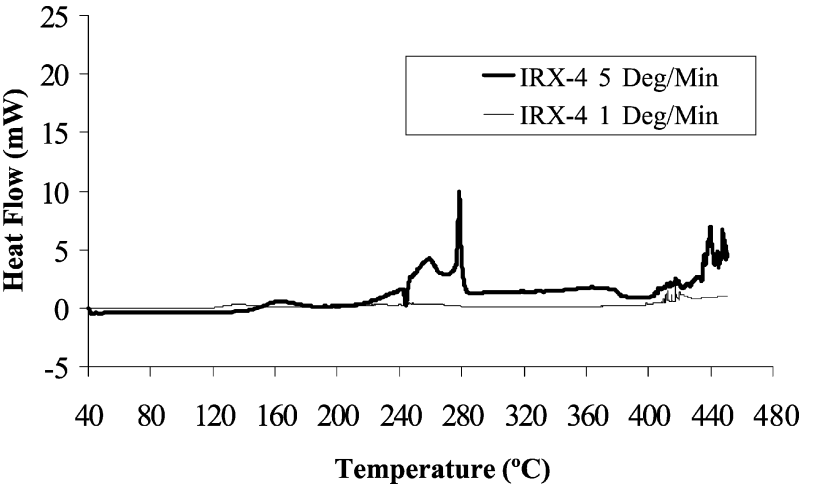

FIGURE 6. DSC results for IRX-4.

measured for samples after aging for about three years. We recommend that this additive be added if a similar formulation effort is performed in the future. The safety tests indicate that explosives in general pose no increased hazards in handling over most common types of explosives. However, IRX-4 was found to be sensitive in drop weight tests. This means that steps should be undertaken to prevent the sample from undergoing friction or being sheared.

An alternative formulation for the research explosive IRX-3A would have been to hold the volume percentage of solids to that of IRX-1. This explosive may have resulted in better sensitivity and performance experiments to determine the role of aluminum but would have not allowed investigation of the role of HMX loading. Another formulation would be to formulate an explosive that contains HMX, AP, and HTPB. This would allow us to perform performance and sensitivity experiments for an explosive with excess oxygen.

The IRXs have been used in experiments [13] to elucidate the roles of HMX, Al, and AP in both shock reactivity and performance.

Acknowledgments

We thank V. Ringbloom, B. Baker, D. Ashwell, T. Fillman, L. Johnson, and R. Simkins for sieving the HMX and casting the compositions; P. Thomas and D. Remmers for performing the safety testing; L. Brown for his help in obtaining the HMX; Drs. J. Forbes, H. Sandusky, and R. Doherty for their cheerful reviews of this manuscript; and Drs. C. Dickinson and R. Bernecker and Messrs. E. Anderson and C. Gotzmer for valuable discussions concerning this project. This work was supported by the Independent Research Program at the Naval Surface Warfare Center.

References

[1] Finger, M., H. C. Hornig, E. L. Lee, and J. W. Kury. 1972. *Proceedings of the Fifth (International) Symposium on Detonation*, ACR-184, pp. 137–151. Arlington, VA: Office of Naval Research.

[2] Chunhua, B. and Ding Jing. 1991. *Proceedings of the Ninth Symposium (International) on Detonation*, OCNR 113291-71, pp. 879–885. Arlington, VA: Office of the Chief of Naval Research.

[3] Simpson, R. L., F. H. Helm, P. C, Crawford, and J. W. Kury. 1991. *Proceedings of the Ninth Symposium (International) on Detonation*, OCNR 113291-71, pp. 25–38. Arlington, VA: Office of the Chief of Naval Research.

[4] Bernecker, R. R. 1991. D. Schmidt and T. Forbes (eds.), *Shock Compression of Condensed Matter*, pp. 695–698. Dordrecht: Elsevier.

[5] Moulard, H., J. W. Kury, and A. Delchos. 1986. *Proceedings of the Eighth Symposium (International) on Detonation*, NSWC MP 86–194, pp. 902–914. Naval Surface Weapons Center. Indian Head, MD.

[6] Moulard, H. 1991. *Proceedings of the Ninth Symposium (International) of Detonation*, OCNR 113291-71, pp. 18–24. Arlington, VA: Office of the Chief of Naval Research. Indian Head, MD.

[7] Van der Steen, A. C., J. J. Verbeek, and J. J. Meulenbrugge. 1991. *Proceedings of the Ninth Symposium (International) on Detonation*, OCNR 113291-71, pp. 83–88. Arlington, VA: Office of the Chief of Naval Research.

[8] Borne, L. 1995. *Proceedings of the Tenth Symposium (International) on Detonation*, OCNR 33395-12, pp. 286–293. Arlington, VA: Office of the Chief of Naval Research.

[9] Anderson, E. 1993. *Tactical Missile Warheads*. Vol. 155 of *Progress in Astronautics and Aeronautics*, pp. 104–105. Washington, DC: American Institute of Aeronautics and Astronautics.

[10] Anderson, E. W. Andercorp Inc. 14924 Perrywood Dr., Burtonsville, MD 20670, USA. (Private Communication).

[11] Gupta, S. C. and Y. M. Gupta. *High Pressure Research*, 19(10): 785–789.

[12] Hall, T. N. and J. R. Holden. *Navy Explosives Handbook*, NSWC MP 88-116, pp. 8-3–8-4. Dahlgren, VA: Naval Surface Warfare Center.

[13] Sutherland, G. T., E. R. Lemar, J. W., Forbes, E. Anderson, P. Miller, K. D. Ashwell, R. N. Baker, and T. P. Liddiard. 1993. S. Schmidt and R. Samara (eds.), *High-Pressure Science and Technology*, pp. 1413–1416. Woodbury, NY: American Institute of Physics.

[14] Eyster, E. H. and L. C. Smith. 1949. *Studies of the ERL Type 12 Drop-Weight Impact Machine at NOL*, NOLM 10,003, 25 January. Silver Spring, MD: Naval Ordinance Laboratory.