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Chemical and Physical Characterization of Comp A-3 Type II Prills

by Rose A. Pesce-Rodriguez and Stephanie M. Piraino

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14. ABSTRACT

Prills of Composition A-3, Type II (Comp A-3) were characterized to provide information on the composition and properties of the explosive for implementation into modeling and simulation tools as part of the Multi-scale Response of Energetic Materials (MREM) program. Optical and scanning electron microscopy (SEM) were used to study the interaction between the RDX fill and polyethylene binder. The components of the formulation as well as residual solvent from the RDX and several impurities carried over from the polyethylene emulsion were identified using desorption-gas chromatography/mass spectroscopy (D-GC-MS) and Fourier transform infrared (FTIR) spectroscopy. Quantitative analysis of nitramines and morpholine were performed using high performance liquid chromatography (HPLC). The reduced indentation modulus and hardness values of RDX crystals of different orientations were also measured. Thermal behavior was investigated by differential scanning calorimetry (DSC), from which the percent crystallinity was estimated. The chemical and physical characterization of Comp A-3 prills is the first step toward understanding the microstructure of the pressed explosive and provides critical information for the development of a high fidelity particle-based course-grain model for predicting energetic material response.

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1. Introduction

This report summarizes experimental efforts to characterize Composition A-3, Type II (Comp A-3) explosive as a part of the Multi-scale Response of Energetic Materials (MREM) program. This program is a collaborative theoretical and experimental effort being led by the U.S. Army Research Laboratory (ARL). Insensitive Munitions (IM) requirements are a fundamental and primary consideration in the design and implementation of new energetic materials (EMs). High fidelity modeling and simulation (M&S) tools to facilitate the design criteria and estimate the sensitivity and performance of advanced EMs are sought to enable predictive capabilities at the forefront of IM technologies. As part of this effort, experimental data regarding the microstructure, defects, and heterogeneity of an EM are necessary to build accurate models that can predict response to shock and thermal stimuli.

In an effort to simplify M&S efforts, it was decided to choose a relatively simple explosive formulation. The composition of Comp A-3 Type II is usually given as being 91 wt.% RDX and 9 wt.% polyethylene (PE), and appears to meet the requirement for a simple formulation. Type II RDX, i.e., RDX produced by the acetic anhydride process (*1*), is used. Unfortunately, it also contains HMX impurity and residual cyclohexanone solvent. The PE in Comp A-3 is actually an oxidized polyethylene (OPE), which has carboxylic acid groups on approximately 15% of its chain ends and a relatively low molecular weight (M_n ~1200) (*2*), and as reported herein, brings several trace impurities along with it to the explosive formulation. OPE is also sometimes called "polyethylene wax." Comp A-3 Type II is produced by BAE Systems. Although few formal records about the manufacturing of Comp A3 are readily available, limited information is available in reference 3 (reproduced in appendix A). Additional information about the formulation and processing was graciously provided by BAE (*4*):

- The RDX used in Comp A-3 Type II is Class 1 RDX, which typically has a broad particle size distribution. Particle size analysis for various batches of Class 1 RDX used to make Comp A-3 confirmed that the majority of RDX particles were roughly 200 μm but that there was a broad distribution of sizes.
- RDX is recrystallized from cyclohexanone but not milled. It is then made into a water slurry before the coating process.
- An emulsion of OPE is used to bind the RDX under heavy agitation at 93–95 °C and atmospheric pressure. Magnesium chloride is used to break the emulsion.
- For the most part, emulsifiers and other additives are washed away with the water from the slurry and the emulsion.

- During the coating procedure, the RDX agglomerates and OPE adheres to the surface; however, the OPE does not provide uniform coating of the RDX and can be considered more of a binder than a true coating.

This process makes small prills that are subsequently pressed together into charges of varying densities and dimensions depending on the intended application. Of particular interest for M&S efforts is how impurities might affect both particle/binder adhesion and RDX particle size in the pressed explosive. The experimental efforts described herein focused on the chemical and physical characterization of Comp A-3 prills and the identification of impurities that might affect binder-filler adhesion. Characterization of the pressed explosive (before and after shock testing) will be the subject of a future investigation.

2. Experimental

Neat A-C 656 oxidized polyethylene homopolymer (CAS 68441-17-8) was obtained from Honeywell (material information sheet given in appendix B). Emulsified polyethylene and Comp A-3 prills were obtained by BAE Systems (Holston, material data safety sheet given in appendix C).

A Zeiss Stemi 2000-C microscope equipped with a digital camera was used to capture images of the tested samples using AxioVision software (Release 4.8.1).

An ISI-SS40 scanning electron microscope was used to analyze the Comp A-3 Type II prills. The prills were mounted on conductive (aluminum) studs using either a conductive adhesive paint or adhesive disc. The samples were coated with a thin conductive film via “cold sputtering.”(*5*) The sample was placed in the instrument chamber, and a vacuum was applied. Various images were taken using different working distances and voltages to determine the optimum image quality. The working distance was set at 20 mm, and the voltage was 10 kV. Images were taken at approximate magnifications ranging from 50× to 2000×.

Analysis of desorbable species was performed using an Agilent gas chromatography-mass spectroscopy (GC-MS) (Model 6890N GC and Model 5973N MSD) system fitted with a Pyroprobe 2000 (CDS Analytical, Oxford, PA). The GC column used was a HP-5 capillary column (0.25 mm × 30 m, 0.25-mm film). The injector temperature was 200 °C and the Pyroprobe interface temperature was 175 °C. The GC oven temperature program was as follows: 50 °C isothermal for 1 min, 50–250 °C at 40 °C/min, and 250 °C isothermal for 1 min. The Pyroprobe was programmed to give a 20-s desorption pulse at 175 °C (heating rate 1000 °C/s). The sample was held within the coil of the Pyroprobe by first placing it in a quartz tube containing a small plug of glass wool, and then inserting the entire tube into the coil.

All Fourier transform infrared (FTIR) spectra were obtained using a ThermoFisher (formerly Nicolet) Nexus 870FTIR spectrometer fitted with a Thermo Nicolet OmniSampler (attenuated total reflectance [ATR]) accessory. A deuterated triglycine sulfate (DTGS) and potassium bromide (KBr) beamsplitter were used. For each spectrum, 64 scans were collected at a resolution of 4 cm^{-1}.

Differential scanning calorimetry (DSC) analyses were carried out using a TA Instruments (New Castle, DE) Q2000 instrument operating under TA Universal Analysis software. Samples were run in hermetic aluminum pans with "pin-hole" lids (TA Instruments, p/n 900860.901). The heating rate was 10 °C/min, and analyses were run from 40 to 110 °C under a nitrogen flow of 50 mL/min.

High performance liquid chromatography (HPLC) analysis was performed with an Agilent Technologies (Santa Clara CA) 1200 Series HPLC. An isocratic elution of a 60% acetonitrile and 40% water mobile phase at a flow rate of 1 mL/min was used. An Agilent Technologies Pinnacle II C18 5-mm (250 x 4.6mm) column was kept at a constant temperature of 30 °C. An injection volume of 10 mL was used. Samples were analyzed at 254 nm. Standard samples of RDX and HMX were prepared in acetonitrile. Comp A-3 samples were also prepared in acetonitrile.

Nanoindentation of RDX crystals was completed using a Hysitron TI950 Triboindenter equipped with TriboScan Software (9.2.12.1). A standard Berkovich probe was used to evaluate RDX crystals (R021 and R210) made under controlled conditions at Los Alamos National Laboratory (LANL).

3. Results and Discussion

To characterize the Comp A-3 Type II formulation, optical and scanning electron microscopy (SEM) were used to examine the physical appearance of the material. Figure 1 is an optical microscopic image of the sample as received. Each prill is approximately 1 mm in size, and areas of the crystalline RDX are visible as the polyethylene does not provide a complete covering. Further evidence of the crystalline RDX particles is observed when the prill is "smeared" with a small spatula (figure 2). The prill was observed to be relatively soft, and broke apart with little applied force.

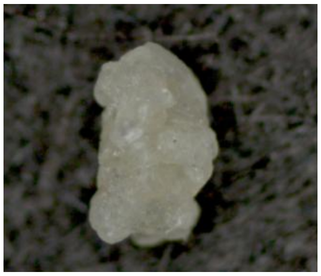

Figure 1. Image of Comp A-3 (as received) taken with an optical microscope.

Figure 2. Image of “smeared” Comp A-3 prill taken with an optical microscope.

The prills were also analyzed by SEM to examine the microstructure of the material and identify any characteristics related to the adhesion of the polyethylene binder on the RDX crystals (figure 3). At the lower magnifications, the inhomogeneity of the shape of the prills was readily observed. Each prill consists of many smaller RDX particles held together by OPE binder. No uncoated RDX particles were observed at the surface of the prills. At higher magnifications (figure 3d), very small particles were seen on the surface of the prill, which could possibly be excess binder from the processing.

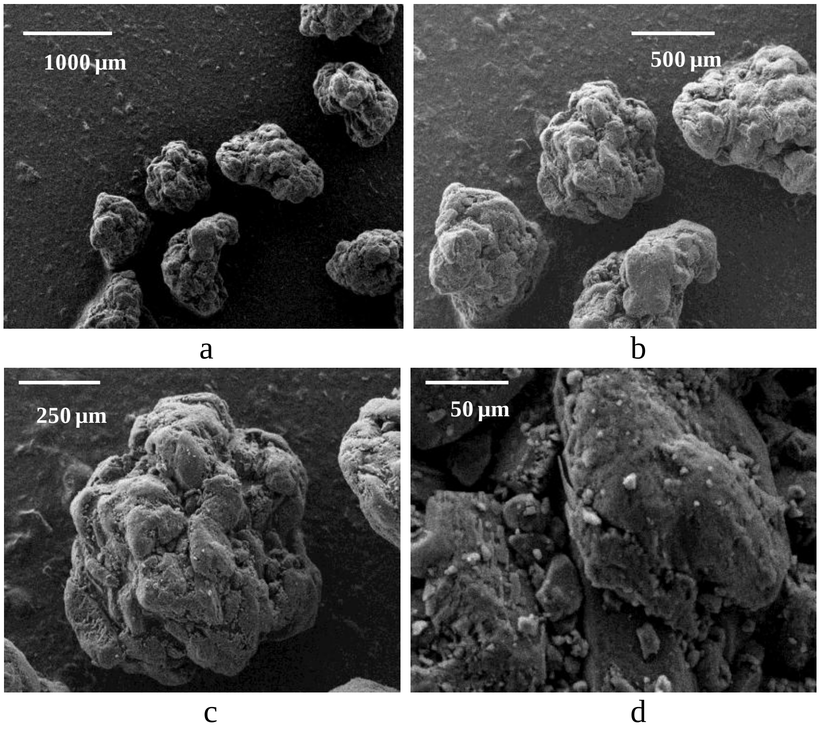

Figure 3. SEM images of Comp A-3 Type 2 prills at various magnifications: (a) 27×, (b) 50×, (c) 100×, and (d) 500×.

One of the prills was cut in half using a razor blade to expose the inner portion of the prill (figure 4). Figure 4b shows the one half of the prill with the “inner” portion of the prill facing up. Other than being flat because of the cut, the inner portion of the prill resembled the outer surface. However, once the prill was cut, some of the crystalline material was exposed (figure 4c). The red arrows in figure 4c point to small voids that might be inhomogeneities on the surface of an exposed RDX crystal. In this SEM image, the lighter portion on top of the prill is the OPE binder. The OPE did not appear uniformly distributed around the RDX particles, and some chunks of polyethylene, similar to that observed in figure 3d, were visible.

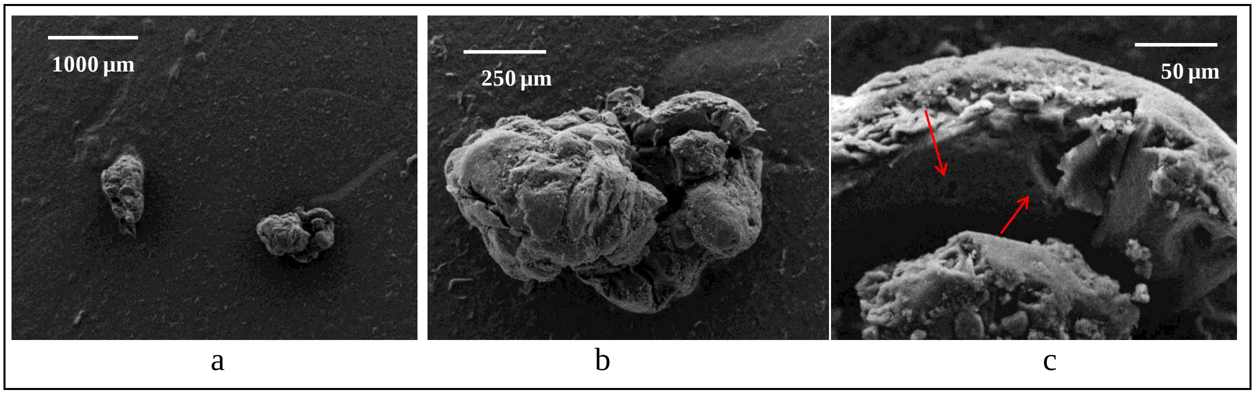

Figure 4. SEM images of Comp A-3 prill at various magnifications: (a) single prill cut in half (27×), (b) half of a prill (100×), and (c) close-up image (100×) with red arrows pointing to small voids.

According to the information obtained from BAE Systems regarding the formulation process, the average particle size of the Class 1 RDX used for the Comp A-3 formulation is about 200 µm (*4*). Given that the size of each prill appears to be roughly 1000 µm, as observed in the SEM images, a prill could contain up to five RDX crystals in one direction bonded together by the OPE. The SEM images of the prills are consistent with what has been observed for other wax-coated RDX crystals (*6*), i.e., the distinct faces of the RDX crystals are obscured by the polyethylene binder. The neat RDX used in the actual formulation process was not available for characterization; however, SEM images of Class 1 RDX provided by Holston (not shown) are consistent with an RDX particle size on the order of 200 µm. The SEM images of the prills will serve as a baseline for comparing the interface of the energetic and binder materials once the prills are pressed into explosives of varying densities (subject of future investigation).

HPLC analysis was performed to determine both the overall nitramine content in Comp A3 prills and the relative percentages of RDX and HMX in the nitramine. The analysis revealed a total nitramine content of 91.0 wt.%, as expected. The nitramine was found to be composed of 92.2 wt.% RDX and 7.8 wt.% HMX. Given that the RDX used in Comp A-3 is Type II RDX and is manufactured by the acetic anhydride process (also known as the Bachmann process), an HMX content of 4–17 wt.% is expected.

Desorption-gas chromatography-mass spectrometry (D-GC-MS) was used to probe the chemical composition of the prills, specifically as related to desorbable species. The chromatogram in figure 5 (top) revealed the presence of cyclohexanone, 4-nitroso morpholine, RDX, and a range of hydrocarbons (presumably from the oxidized polyethylene binder) in the prills.

When evaluating the mass spectra of the peaks labeled as “unknown” in figure 5 (top), a dominant peak for m/z = 59 was observed. These peaks are more clearly visible in the selected ion chromatogram (SIC) for m/z = 59 (figure 5, bottom). A search of the mass spectra for these peaks against a library of mass spectra suggested that they are secondary alcohols, but no exact matches were found. An example of a search result that is similar, but not identical to the spectra of one of the peaks (8.2 min) is shown in figure 6. The peaks in green are a good match, but the ones in red do not match at all. The green peaks are characteristic of the class of compound (i.e., secondary alcohols), while the red peaks are characteristic of the overall molecular weight of the compound. Based on this, it is concluded that the peaks appearing in the m/z = 59 SIC are secondary alcohols with molecular weights larger than any of those in the mass spectral library. Analysis of the mass spectra of secondary alcohols available in the library revealed that there is an excellent correlation between the largest mass in the spectrum and the overall molecular weight of the compound. Using this relationship, the masses of the secondary alcohols in the Comp A-3 spectra were extrapolated and found to be in the range of C_{15}–C_{23}.

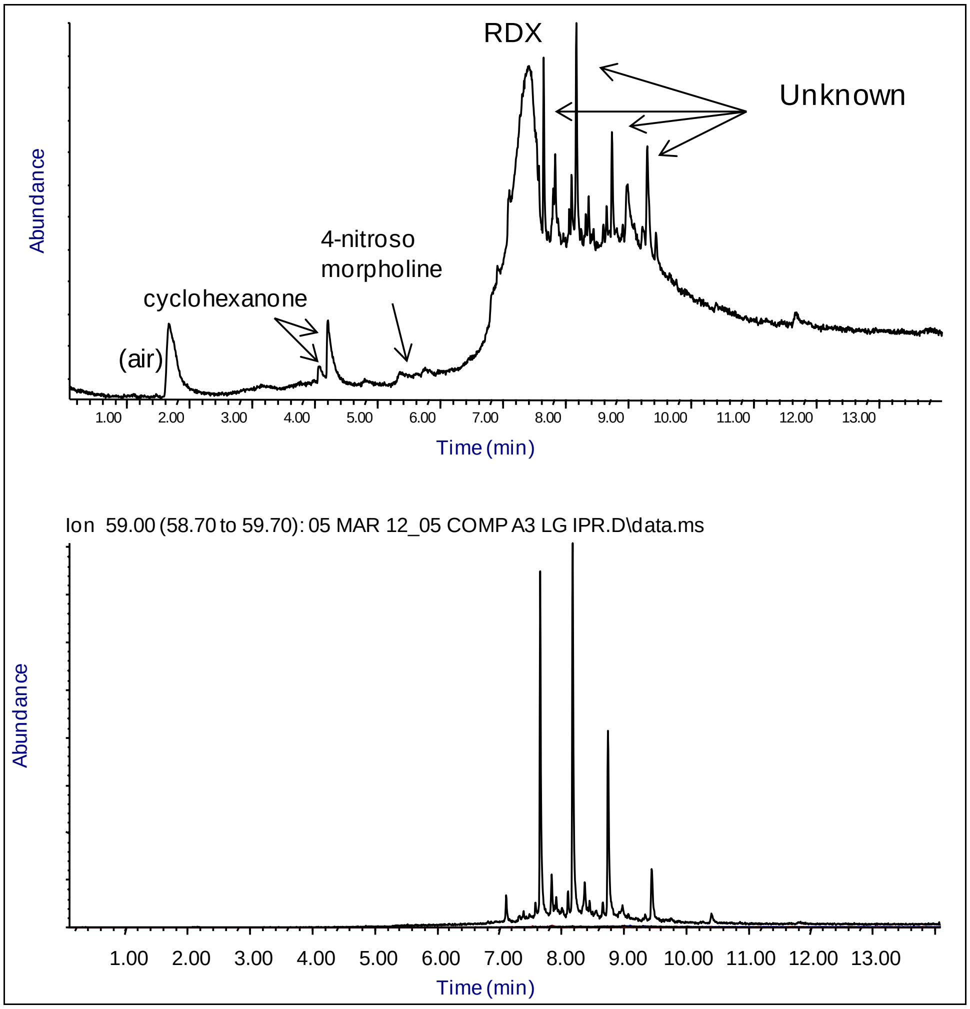

Figure 5. D-GC-MS results: (top) total ion chromatogram (TIC) for desorption of 15 mg sample of Comp A-3 at 175 °C and (bottom) SIC of same for m/z=59.

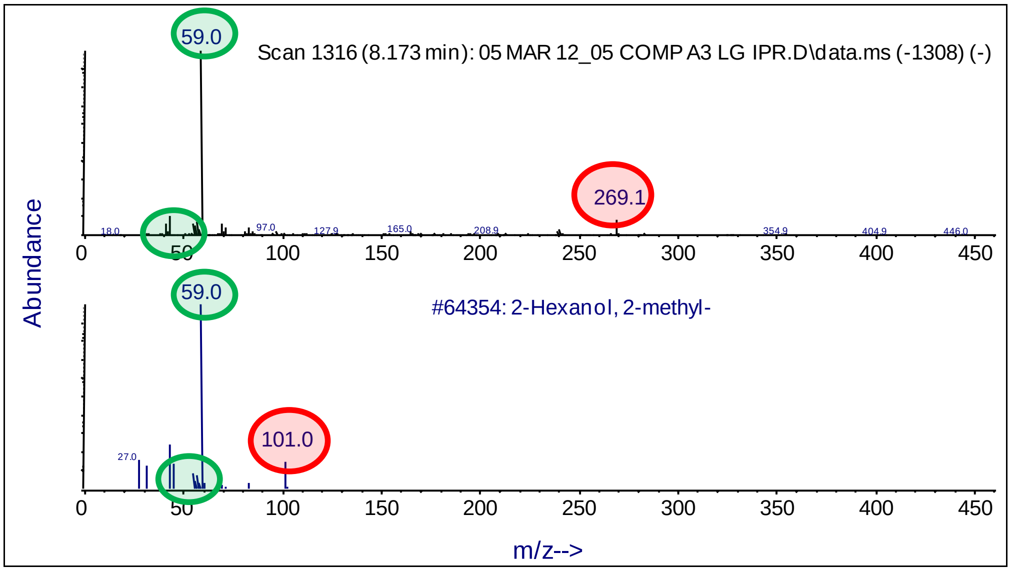

Figure 6. (Top) Mass spectrum for a secondary alcohol impurity and (bottom) library search result.

The mass spectra of the peaks at both 4.15 and 4.25 min in figure 5 (top) were identified as cyclohexanone. Two prominent ions indicative of cyclohexanone are m/z= 55 and 98 (figure 7). When these ions are extracted, the SIC indicates the gradual evolution of cyclohexanone from the RDX inclusions throughout the course of the run (figure 8).

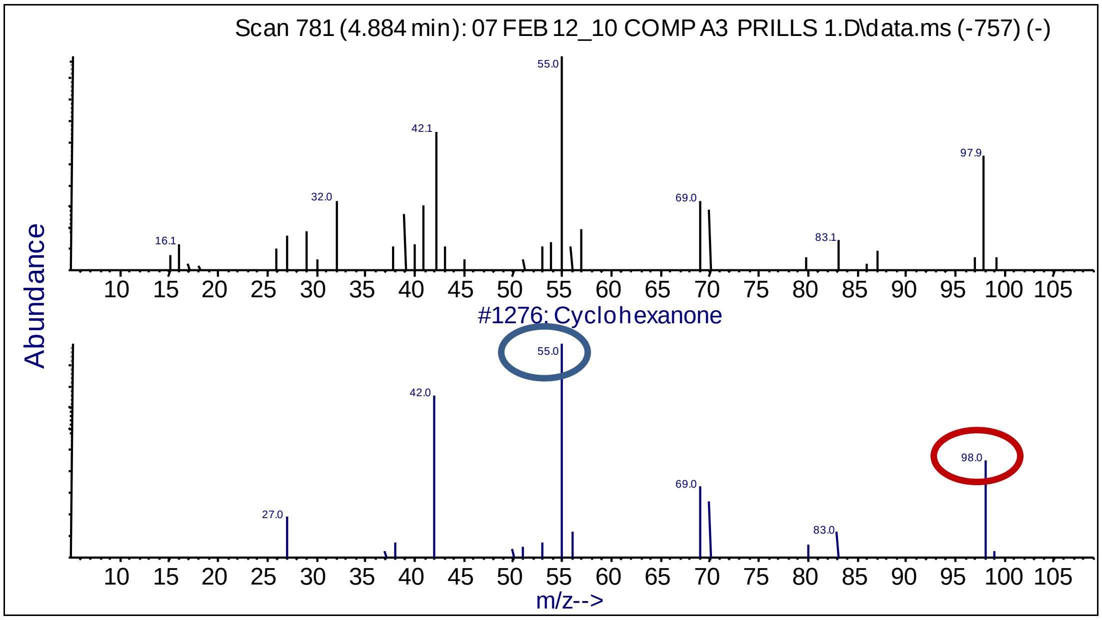

Figure 7. (Top) Mass spectra of peak at 4.88 min and (bottom) library match for cyclohexanone.

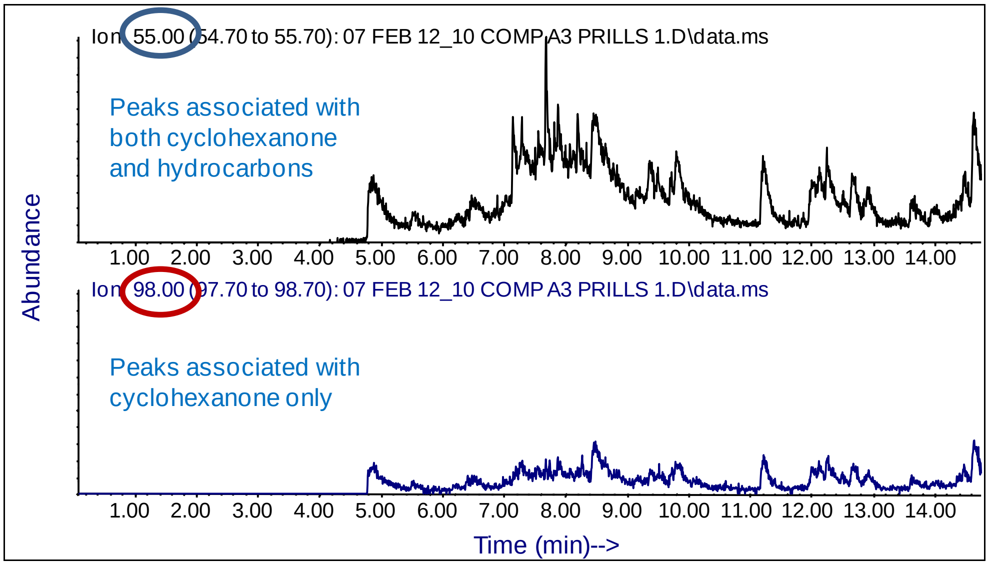

Figure 8. D-GC-MS results: SIC for m/z=55 (top) and m/z=98 (bottom) indicating evolution of cyclohexanone from RDX inclusions.

After the Comp A-3 Type II prill was heated at 175 °C for the GC-MS analysis, the crystalline portion of the formulation was still observed, yet the prill appeared brown in color (figure 9). The discoloration could be attributed in part to the decomposition of polyethylene binder as a result of interaction with RDX and/or its decomposition products. Untreated prills are very soft and waxy, as evidenced by the ease with which they break apart when gently pressed with a spatula. The number average molecular weight (M_n) of neat OPE is known to be approximately 1200 (*2*), meaning that the polymer has approximately 40 ethylene repeat units per chain.

Figure 9. Prill after being heated at 175 °C
for the GC-MS analysis.

The emulsion used in the processing of Comp A-3 prills contains morpholine (emulsifier and solubility aid). Other additives to the emulsion include tall oil acids, stearic acid, and ammonium hydride. Tall oil acids are composed primarily of oleic acid; however, the commercial-grade product can also contain stearic acid (the saturated form of oleic acid), linoleic acid, and palmitic acid. At atmospheric pressure, oleic acid decomposes when heated to 80–100 °C. Upon exposure to air, it oxidizes and acquires a yellow to brown color and rancid odor. While it is tempting to attribute the color of the heated prills to the oxidation of oleic acid, analysis of Comp A-3 prills by D-GC-MS does not indicate the presence of either oleic or linoleic acids or any of their decomposition products.

Samples of both the OPE and the polyethylene/ammonium stearate emulsion were obtained and analyzed by D-GC-MS. Prior to analysis, the emulsion was allowed to dry for several hours at room temperature. This resulted in the loss of most of the water from the emulsion but not the loss of morpholine and other additives that are mostly driven off during the actual processing of Comp A-3 prills. Evaluation of the chromatograms of the OPE and a “dried” sample of the emulsion (figure 10 and 11, respectively) confirmed that the source of the secondary alcohols in the chromatogram of the prills (figure 5) is the A-C 656 OPE itself. The large peak at approximately 3.5 min in the chromatogram of the dried emulsion was identified as morpholine.

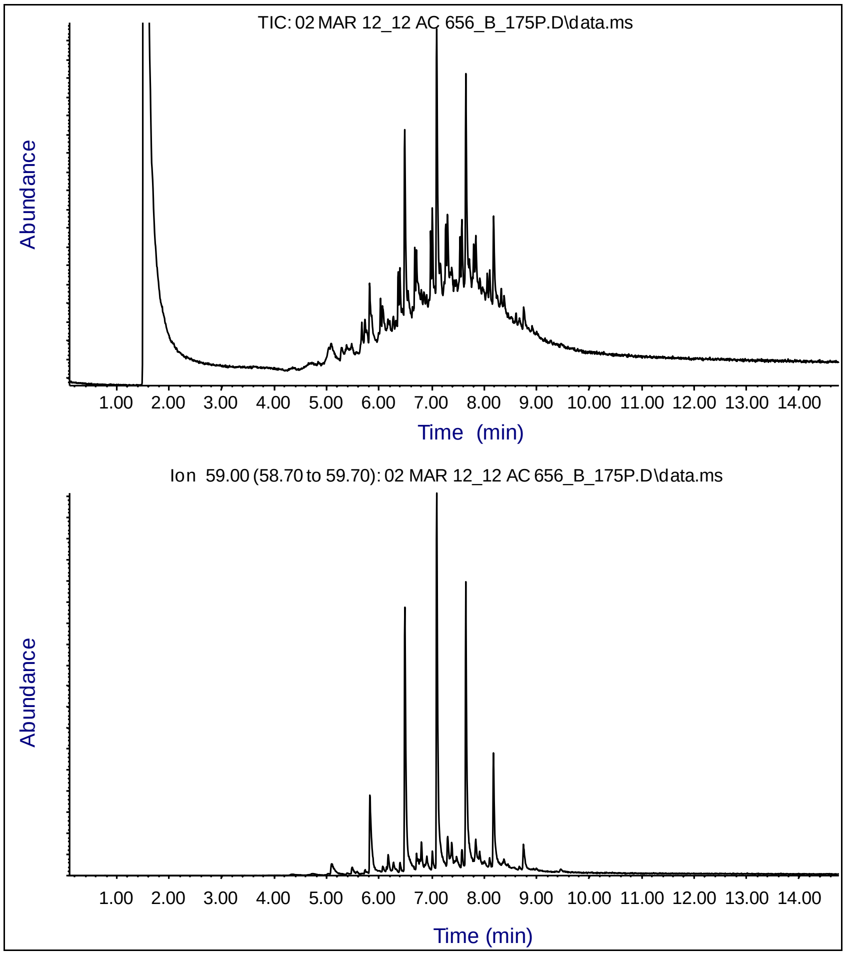

Figure 10. D-GC-MS results for neat OPE (175 °C pulse): (top) TIC and (bottom) SIC (m/z = 59).

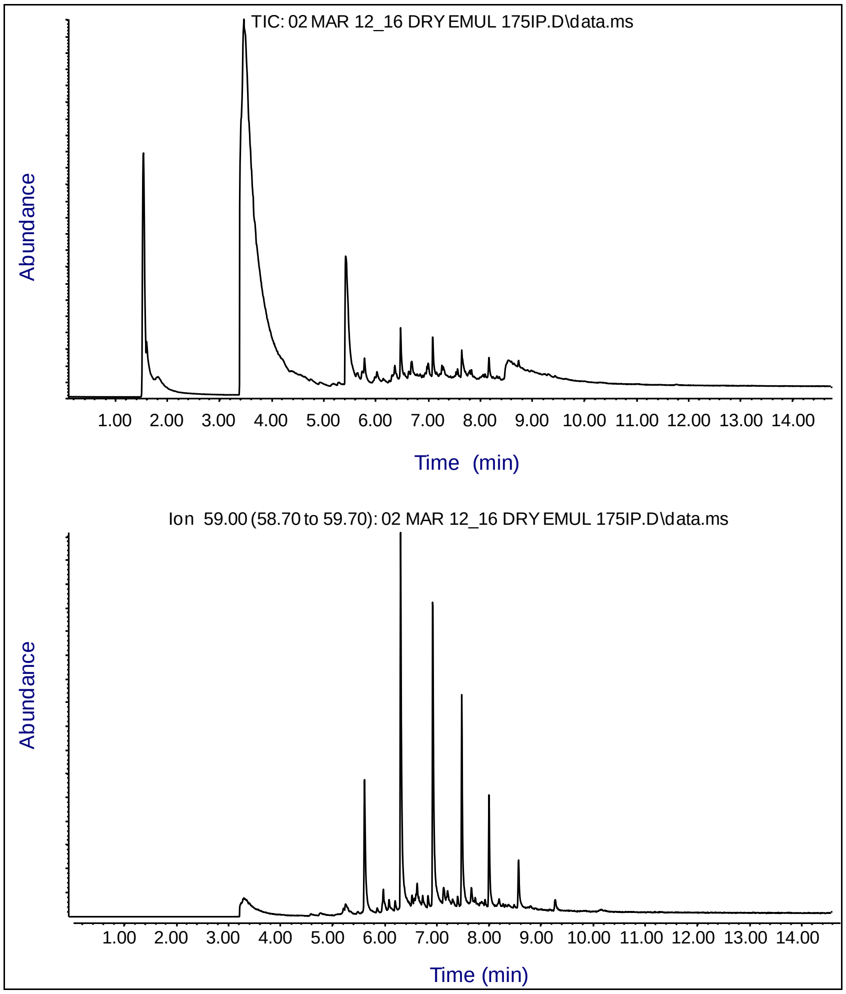

Figure 11. D-GC-MS results for dried emulsion (175 °C pulse): (top) TIC and (bottom) SIC (m/z = 59).

A closer look at the chromatograms for the A-C 656 (figure 10), as well as for the dried emulsion (figure 11) and Comp A-3 prills (not shown), revealed a set of much smaller peaks that are related to the secondary alcohols discussed above. In figure 12, several SICs have been generated. The SIC for m/z = 59 shows where each of the secondary alcohols elutes. For four of these peaks (5.8, 6.5, 7.2, and 7.7 min), SICs corresponding to the highest mass in the mass spectrum associated with each peak are also shown. For example, the peak at 7.2 min has a peak with m/z = 213, and the 7.7 min peak has a peak with m/z = 241. A much smaller peak is observed to the right of each of those peaks in the SIC. Examination of the mass spectra of these smaller peaks indicates that they are from a series of homologous ketones. While no calibration was performed to quantify the level of either the ketones or the secondary alcohols, it is assumed based on the sensitivity of GC-MS and the size of the peaks that both the secondary alcohols and ketones are present at the trace level.

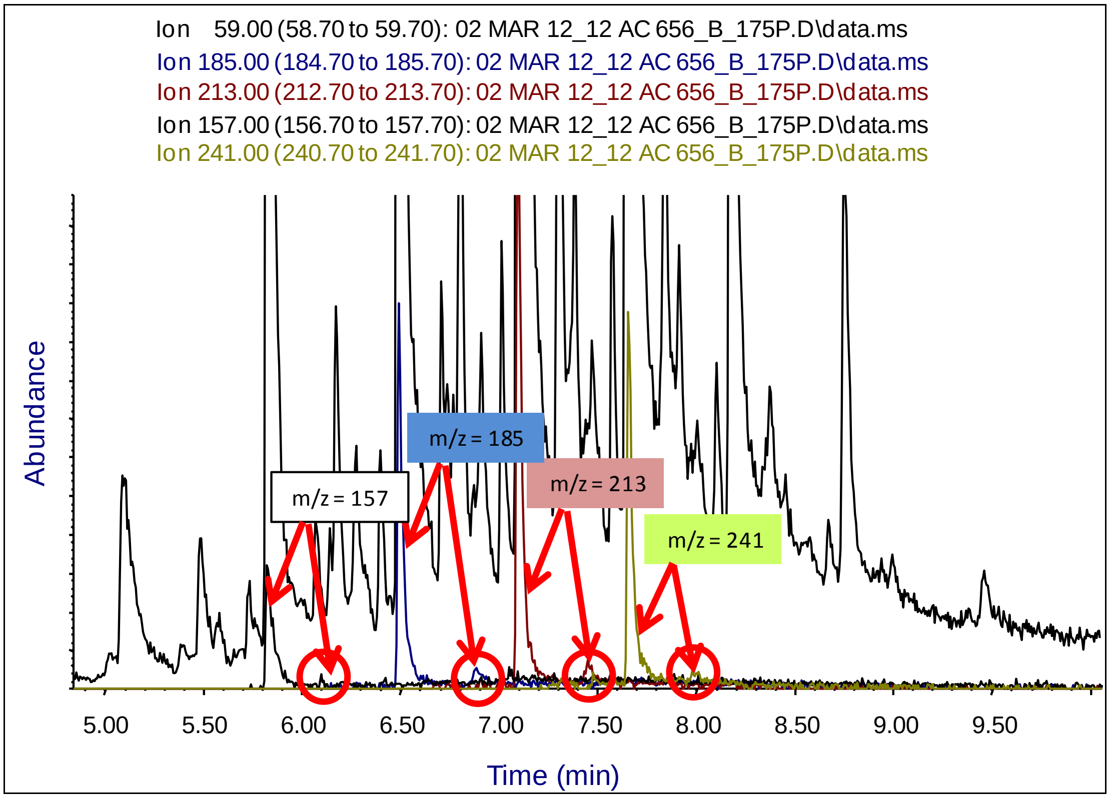

Figure 12. Several SICs for the 175 °C desorption-GC-MS analysis of A-C 656 oxidized polyethylene.

The Comp A-3 Type II prills, A-C 656 oxidized polyethylene, and a dried sample of the A-C 656/ammonium stearate emulsion were also qualitatively analyzed by FTIR. The spectra of neat RDX and Comp A-3 prills are shown in figure 13. Both RDX and OPE are clearly visible in the spectrum of the Comp A-3. The FTIR library match (not shown) for the neat OPE was confirmed in the spectral library. Comparison of the spectra for the neat OPE and dried emulsion (figure 14) revealed several peaks in the emulsion spectrum, most notably at 1600 cm^{-1}, that were not attributed to the oxidized polyethylene. Spectra for the various additives (morpholine, oleic acid, linoleic acid, and stearic acid) were found in the spectral library and compared to the spectra of the dried polyethylene emulsion, yet no matches were found. Since any additives present in the formulation are necessarily present at a relatively low level, it was concluded that the peaks were associated with a strong infrared (IR) absorber. Absorbance peaks in the region between 1500–1600 cm^{-1} are suggestive of carboxylic acid salts (CO_2^-). A successful match (not shown) for the unidentified peaks was found to be ammonium stearate, the salt formed by the ammonium hydroxide and stearic acid, which is consistent with the MSDS for the emulsion.

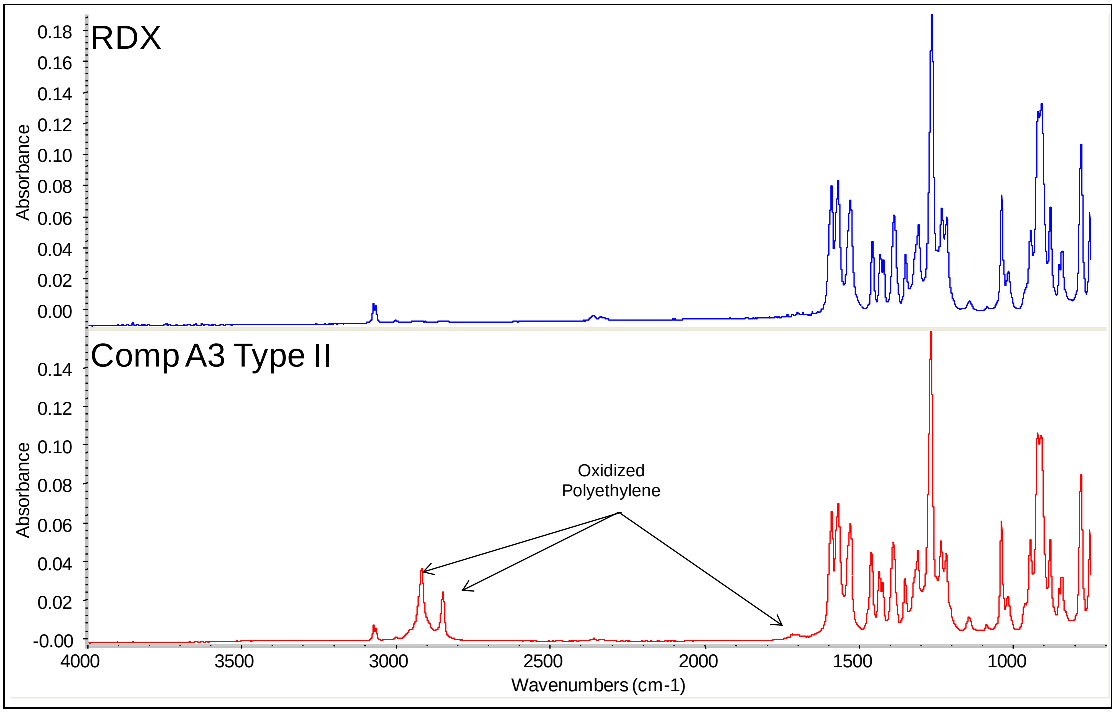

Figure 13. ATR-FTIR spectra of RDX and Comp A-3.

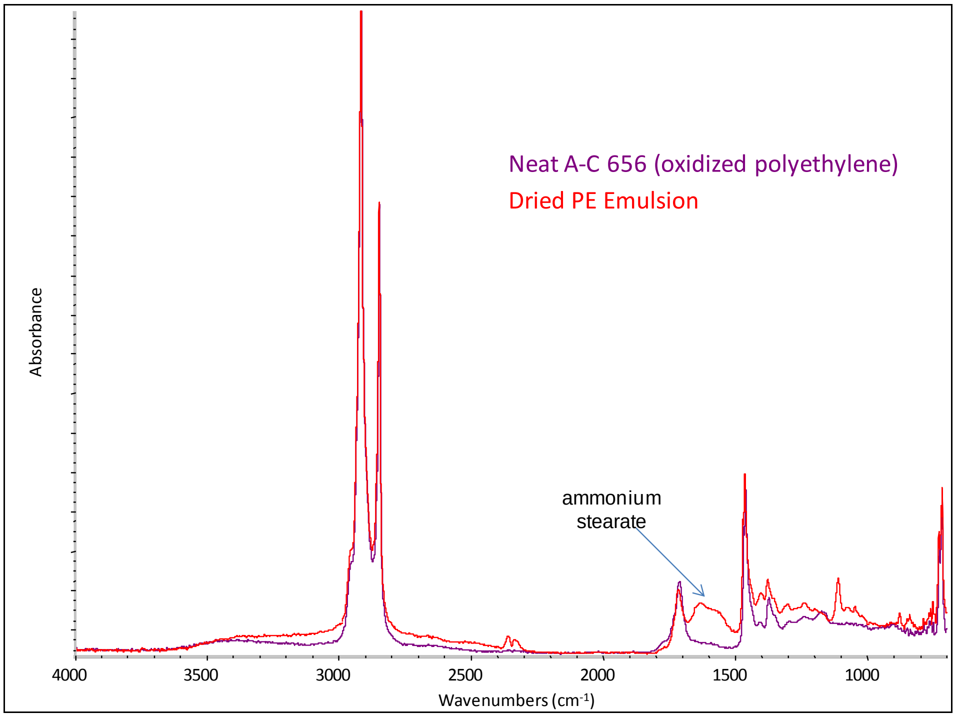

Figure 14. FTIR spectra of oxidized polyethylene and the dried polyethylene emulsion.

As discussed earlier, Comp A-3, while primarily composed of RDX and OPE, also contains trace levels of other materials that might affect the interface, and therefore the adhesion, between RDX and the OPE. Understanding the nature of that relationship is critical to the development of high fidelity physical models. A significant concern regarding possible impurities was the presence of morpholine from the emulsion and its possible effect on RDX solubility. Morpholine was readily detected in the chromatogram of the "dried" emulsion and was determined by HPLC to be present at a level of approximately 3% by weight in the emulsion. This weight percentage was consistent with other values reported in the literature (*7*). Analysis of Comp A-3 prills by GC-MS revealed a morpholine content of 0.0009 wt.%. Such a low content suggests that the presence of morpholine might not be a matter of concern, unless it happens to be concentrated at the interface.

Parallel studies being conducted at LANL on the interfaces between several binders and crystalline fills (*8*) raised questions as to how the presence of morpholine might affect the solubility of RDX, and more importantly, result in a diffuse interface between the RDX and OPE binder; therefore, pertinent solubility measurements were made (table 1). For the determination of RDX solubility in pure morpholine and in a 3% aqueous solution (to model the emulsion), RDX was first allowed to dissolve in the solvent for approximately 3 h. While the solubility of RDX in pure morpholine was found to be relatively high (20.6 mg/mL), the solubility of RDX in a 3% morpholine solution (aqueous) was found to be on the same order of magnitude as in neat water. Considering the amount of the water from the RDX slurry contributed to the mix during processing, it is clear that the solubility of RDX would be even lower than in the 3% solution and would more closely approach that of neat water. If dissolution of RDX in the processing mix is of concern, the solvent to worry about is water, not morpholine.

Table 1. Solubility of RDX at 20 °C.

Sample	**Solubility (mg/mL)**
RDX in pure morpholine	20.6
RDX in 3% aqueous morpholine	0.0456
RDX in water[a]	0.038

[a] Literature value (*7*).

The solubilities in table 1 were all determined at 20 °C. Taking into account that the aqueous solubility of RDX nearly doubles as the temperature is raised from 20 to 30 °C (*9, 10*) and that the solubility increases by approximately a factor of five (*10*) at the processing temperatures of Comp A-3 prills (93-95 °C [*2*]; 100 °C [*3*]), the presence of water in the processing mix could very well affect the quality of the RDX/OPE interface by dissolving even a small fraction of the RDX.

Another consideration regarding changes in RDX particle size that may result in a diffuse interface is the effect of the OPE emulsion's relatively high pH. The as-received emulsion has a pH of approximately 10, and even when diluted to 1% (in water) the pH is still fairly high (approximately 9) and sufficient to result in hydrolysis of RDX. (*11*) For prolonged exposure, it would not be unreasonable to expect some degree of RDX decomposition as evidenced by pitting or particle size reduction. However, given the relatively short time that RDX and the emulsion are in contact with each other during processing and the fact that any hydrolysis products generated would likely evaporate from the prills along with water, there is probably no reason to be concerned about the effect of hydrolysis on quality or physical properties of RDX in Comp A-3 prills.

To further understand the nature of the interaction between OPE and RDX, the degree of crystallinity of the OPE in the prills was investigated. The DSC analysis of neat A-C 656 OPE (figure 15) revealed a broad endotherm between 40 and 100 °C that is due primarily to the melting of the OPE but also, to a smaller extent, due to the evaporation of relatively low molecular weight impurities in the OPE. Integration of the peak gave a heat of fusion of approximately 84 J/g. This value is converted to percent crystallinity by dividing by the heat of fusion for 100% crystalline polyethylene (i.e., 294 J/g) to give 28.6%, which is in good agreement with a value of 29% crystallinity quoted by the manufacturer (*2*). Figure 16 shows the results from an annealing experiment for A–C 656 OPE: the polymer was first heated to 150 °C (upper trace), allowed to cool to 40 °C, and then reheated (lower trace). While it was observed that the overall heat of fusion decreased from 89.70 to 73.73 J/g, likely due to the absence of the low molecular weight impurities that escaped the pan through the pinhole during the annealing process, the DSC trace also revealed the presence of new, more obvious features near 90 and 100 °C. These features suggest that annealing the polymer resulted in morphological changes (perhaps by extension of polymer chains resulting in thickening of the lamellae).

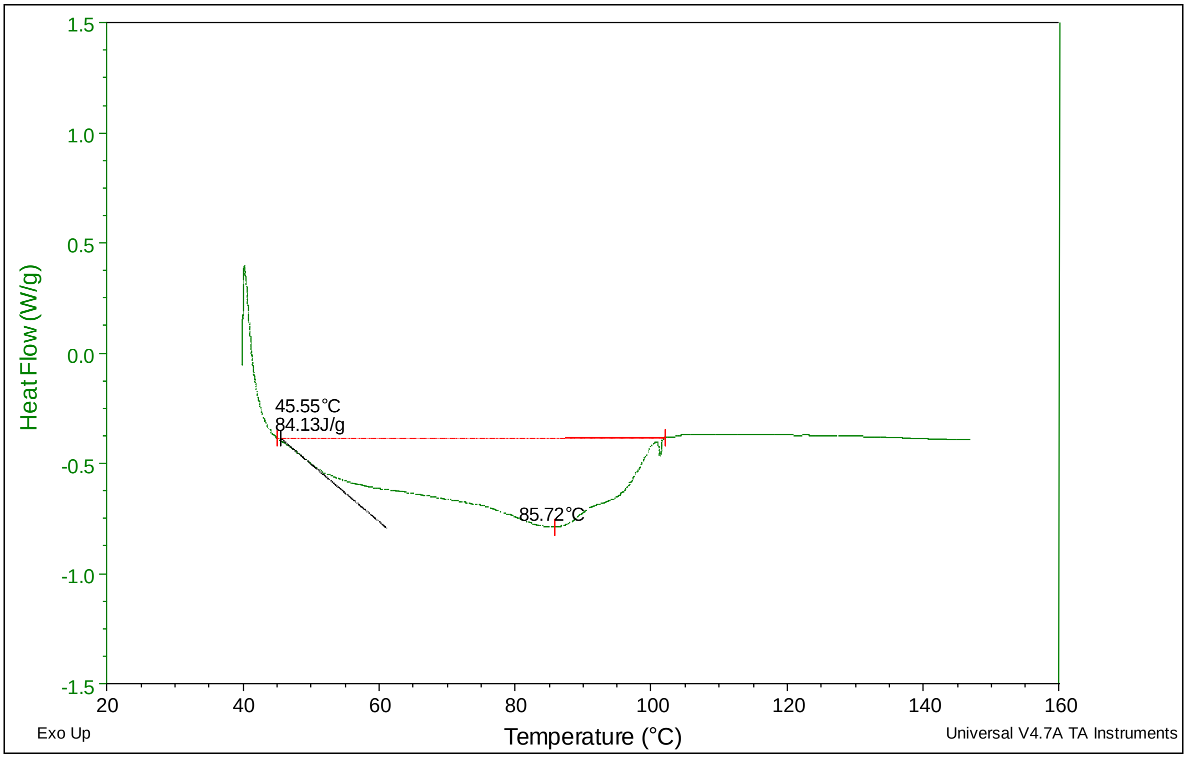

Figure 15. DSC of A-C 656 (oxidized polyethylene homopolymer).

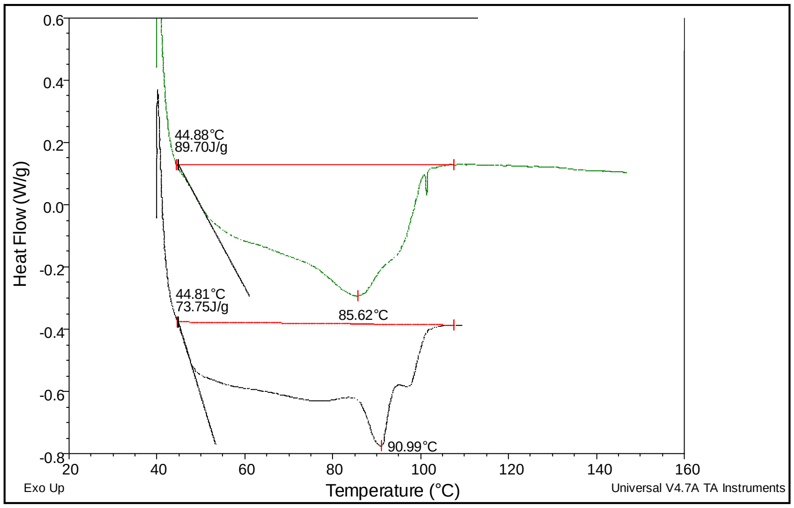

Figure 16. DSC annealing analysis of A-C 656 (oxidized polyethylene homopolymer). Top: As-received; Bottom: Annealed

Performing the same analysis on the “dried” emulsion (figure 17) initially yielded a very broad melting endotherm ranging from 40–100 °C (black trace in figure 17). When allowed to cool, and then reheated, the endotherm of the annealed sample became slightly narrower, and the same two peaks emerged between 90–100 °C (red trace in figure 17). The analysis was repeated a third time, and the resulting trace (green trace in figure 17) was nearly identical to that of the second analysis. Based on the appearance of the first trace, the changes observed in the later traces, and the results of the D-GC-MS analysis discussed previously, it is concluded that while the "dried" sample is probably mostly devoid of water, it still contains other additives from the emulsion (the most significant being morpholine and ammonium stearate) as well as the secondary alcohol and ketone impurities from the neat OPE. The presence of these additives, alone or in combination, might inhibit crystallization. It appears that as the sample is heated, the additives/impurities are at least partially excluded from the sample, leaving the oxidized polyethylene better able to organize without a significant change to the crystallinity.

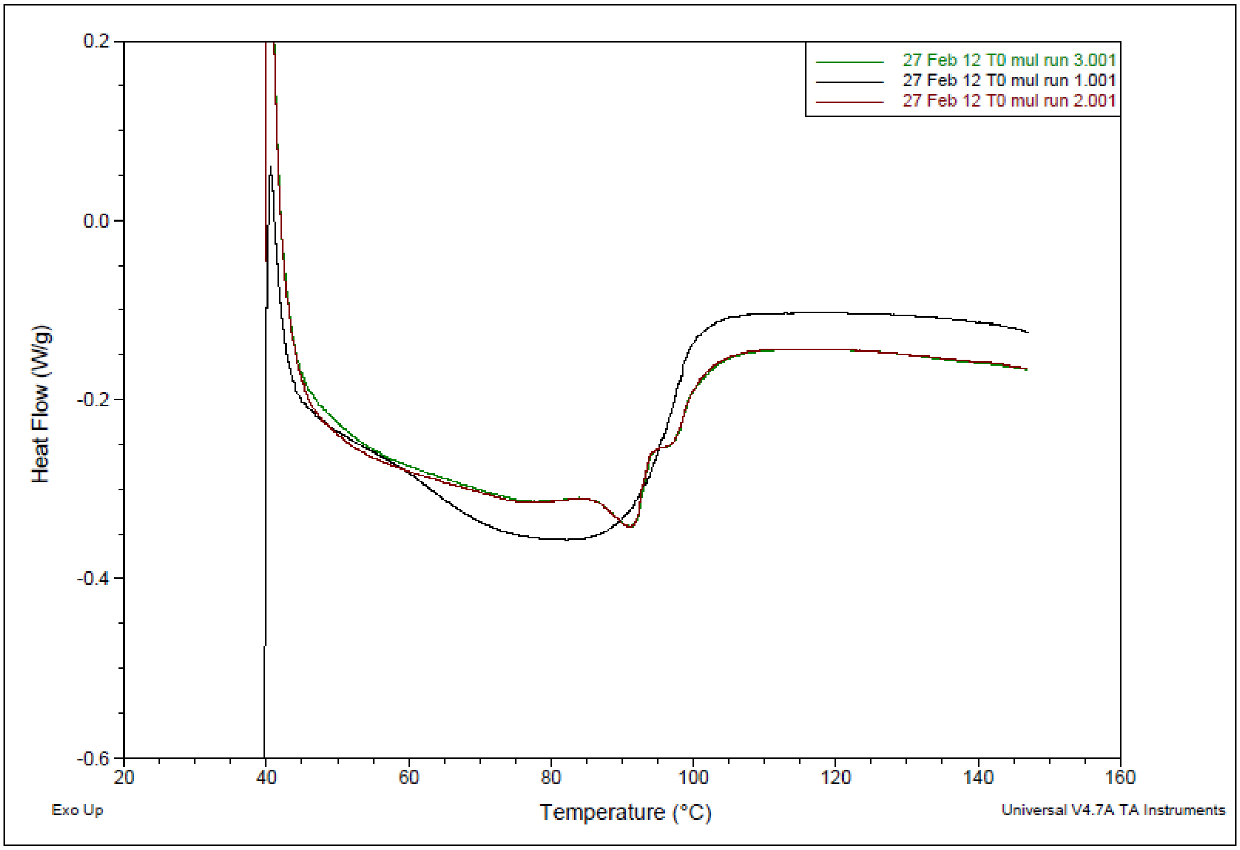

Figure 17. DSC traces for three analyses of a single sample of "dried" polyethylene/ammonium stearate emulsion (black trace: 1st analysis; red trace: 2nd analysis; green trace: 3rd analysis).

Since the secondary alcohol and ketone impurities were found to be C_{15}–C_{23} and the ammonium stearate is known to be C_{18}, it is expected that each would exert more or less the same effect on OPE crystallization, i.e., they could each fit within the OPE lamellae, which are typically on the order of 10–20 nm in thickness, which corresponds to 40–80 CH_2-CH_2 units (i.e., C_{80}–C_{160}), when grown from dilute solution.(*12*) Furthermore, given that the overall length of the typical OPE chain (i.e., approximately C_{40}) falls below the minimum length (i.e., C_{150}) thought to give stable folded chain lamellae (*13*), it is assumed that the best the OPE can do with respect to crystallization is to assemble chains "side by side" as indicated in figure 18, which shows two crystallization options: a low degree of order (left) and high degree of order (right). Given that the change in morphology of the OPE in the dried emulsion on annealing seems to be somewhat greater than that of the neat A–C 656 OPE, it is proposed that the additive/impurity with the greatest potential effect on OPE crystallinity is morpholine, since the neat OPE has the C_{15}–C_{23} species but no morpholine, and the dried emulsion has the C_{15}–C_{23} and C_{18} species and morpholine. Also, it is assumed that alcohol/ketone impurities are present at only trace levels, whereas morpholine was found to make up 3 wt.% of the as-received emulsion. Drying the emulsion at room temperature would do little to drive off morpholine, especially considering that morpholine can react to form salts with the carboxylic acid functional groups on the OPE endgroups and several additives in the emulsion (oleic, linoleic, and stearic acid). The salts should be relatively stable at room temperature but would break down on heating (during processing), allowing the morpholine to evaporate during Comp A-3 processing. One reference (*13*) claims that the dark tan color of OPE emulsions is the result of colored compounds formed by reaction of amines (in this case morpholine) with oxygen-containing functional groups in the OPE. Given that Comp A-3 prills retain the dark tan color of the emulsion, it is likely that morpholine-containing compounds or complexes are present in Comp A-3. It is not clear if the morpholine detected in Comp A-3 was from free morpholine or from morpholine containing compounds/complexes.

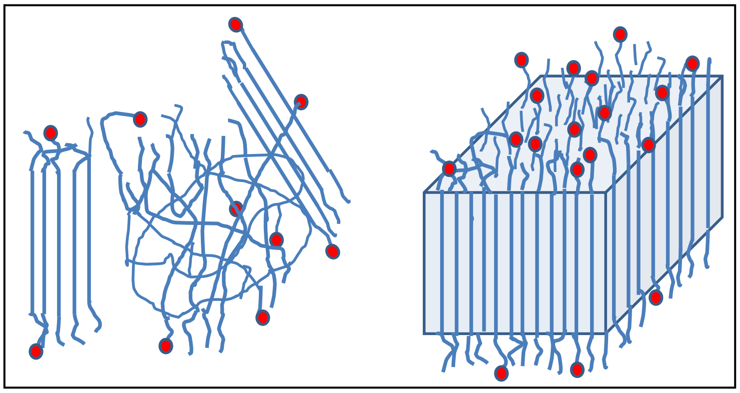

Figure 18. Simplified schematic representation of two OPE crystallization options: (left) low degree of order and (right) high degree of order (red balls represent carboxylic acid end groups).

Results from the DSC analysis of a relatively large sample of Comp A-3 prills are compared with results from analysis of smaller samples of A–C 656 and the dried emulsion (figure 19). The net amount of PE in the Comp A-3 was of the same order of magnitude as the PE in the other two samples. The trace for the Comp A-3 included the same two endothermic peaks observed for both the A–C 656 and the annealed dried emulsion, suggesting similar morphology. As already indicated, the A–C 656 was approximately 29% crystalline and the dried emulsion approximately 15% crystalline. Integration of the trace for the Comp A-3 revealed an OPE crystallinity of approximately 25%, and suggests that of the two options shown in figure 16, the one on the left is closer to the arrangement for the OPE in Comp A-3.

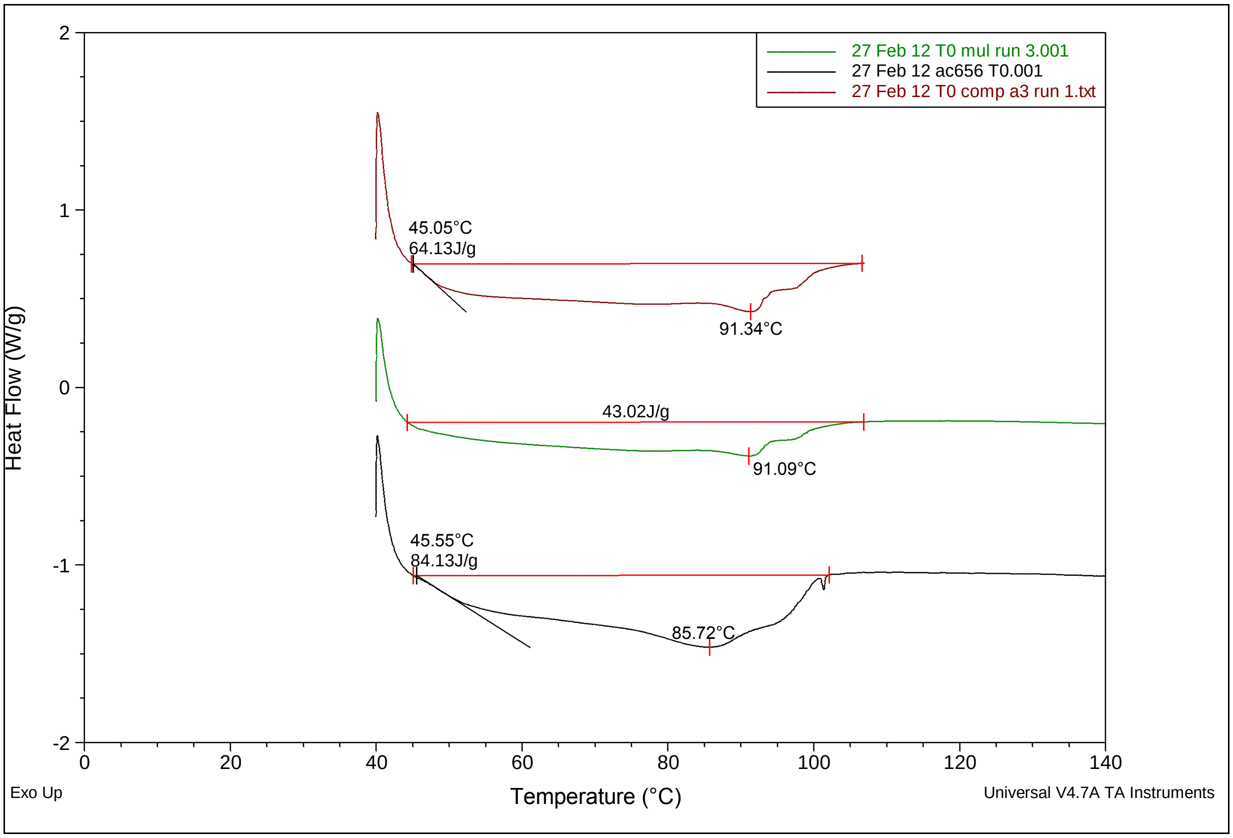

Figure 19. DSC traces for (top) Comp A prills (10.71 g; PE content = 1.07 g), (middle) "dried" annealed polyethylene/ammonium stearate emulsion (1.12 g), and (bottom) A-C 656 oxidized polyethylene homopolymer (1.54 g).

One of the goals of this study was to understand how impurities in the Comp A-3 formulation might affect the interaction between RDX and binder, and thereby provide information to support the modeling effort. A parallel effort to investigate the mechanical properties of RDX at the submicron scale (<1 μm^2) using nanoindentation was also conducted. The sensitivity, resolution, and relative forces applied with the nanoindenter warrant the examination of samples with smooth and flat surfaces. While the surface of Comp A-3 Type II prills is too complicated to evaluate using nanoindentation, the surface of neat RDX crystals can be well suited for analysis by nanoindentation. For the purposes of this study, two different RDX crystal orientations (021 and 210) were grown at the High Explosive Crystal Laboratory at LANL and provided for indentation characterization at ARL. Scanning probe microscopy (SPM) images taken with the instrument prior to indentation did not reveal any surface or roughness issues associated with the RDX crystals that might impede accurate and reproducible determination of the reduced indentation modulus (E_r) and hardness (H) of the sample. The reduced indentation modulus is a function of Young's moduli and Poisson's ratios for both the specimen and the indenter. Additional experimentation and mathematical manipulations would be required to get the directional material moduli of the RDX crystals.

Indentations were made on the RDX crystals with a Berkovich probe to a maximum load of 500 µN at an indent rate of 100 µN/s and a hold time of 5 s between the loading and unloading segments. The indentations resulted in elastic and plastic deformation. The reduced indentation modulus (measure of elasticity) and hardness (measure of resistance to plastic deformation) of the RDX crystals were calculated using the indentation load-displacement response and the traditional Oliver-Pharr nanoindentation analysis (*15*). The modulus and hardness values reported in table 2 are the average of 10 indentations. The different crystal orientations exhibited similar modulus and hardness values which were on the same order of magnitude with previously reported values (*16*). Differences between the values obtained in this study and previously reported values may be attributed to the different contact conditions (i.e., probe geometry, indent depths, etc.) and/or conditions for crystal growth and surface preparation.

To examine whether there was any dependence of indentation modulus and hardness on indentation depth, a set of partial unloading experiments with a beginning force of 50 µN and a peak force of 1000 µN (20 loading segments) was also conducted. According to the results of this study, only a very small material dependence on depth was observed at low contact depths.

Table 2. Reduced indentation modulus (E_r) and hardness (H) values for RDX crystals.

	E_r (GPa)		**H (GPa)**	
	Average	**Std dev**	**Average**	**Std dev**
(021)	23.96	0.36	0.803	0.024
(210)	23.89	0.37	0.806	0.030

4. Conclusions

The physical and chemical characterization of Comp A-3 prills reported here provides fundamental understanding of some of the subtleties related to what was thought to be the "simple" Comp A-3 formulation that might prove critical to the generation of robust mesoscale models to predict the sensitivity of the explosive. Considerations that were identified include the following:

- The evolution of cyclohexanone (processing solvent) was observed in the GC/MS data of the prills.
- The RDX used in the Comp A-3 prills examined contained nearly 8 wt.% HMX.
- A number of relatively short-chain (C_{15}–C_{23}) secondary alcohols and ketones were detected in the GC/MS data of the neat OPE, the emulsion, and Comp A-3 Type II prills.

- The binder used in Comp A-3 is not “polyethylene,” but an OPE with carboxyl groups on approximately 15% of its chain ends and a number average molecular weight (M_n) of approximately 1200. The binder brings along with it a suite of secondary alcohols and ketones that are produced during the oxidation of polyethylene to produce OPE.
- While morpholine is present in the emulsion at a level of approximately 3 wt.%, and while RDX was found to be very soluble in neat morpholine, it was determined that the solubility of RDX in a 3% aqueous solution of morpholine is nearly identical to that in pure water.
- It was proposed that morpholine would only be a matter of concern to modelers if it tended to concentrate at the binder-filler interface. While there is no firm evidence that this is the case, suspected changes in the morphology of OPE observed in the annealed “dried” emulsion suggest that it may be possible and should be considered further.
- If there is any material that should give rise to concern related to RDX solubility and the possible effect on the generation of a diffuse interface with the OPE binder, it is the water that comes from the RDX slurry and the OPE emulsion, not morpholine or other trace impurities.
- The percent crystallinity of the OPE in a Comp A-3 prill (25%) is lower than that of the neat polymer (29%) but higher than that of the emulsion (15%) when allowed to dry at ambient conditions for several hours. Annealing of neat OPE slightly lowers the overall crystallinity, yet gives rise to new peaks likely resulting from morphological changes of the polymeric network.
- Reduced indentation modulus and hardness values for both RDX crystal orientations were the same, and these values were within the same order of magnitude as previously reported values.

This work serves as the foundation for upcoming studies of pressed Comp A-3 Type II and will be the subject of a future report.

5. References

1. Ledgard, J. *The Preparatory Manual of Explosives*; 3rd ed., Jared Ledgard, 2007.
2. Jablon, M.; Sung, H.-N. Personal communication. Honeywell, 26 July 2012.
3. Army Materiel Command, *Engineering Design Handbook: Explosive Series, Properties of Explosives of Military Interest.* Headquarters, U.S. Army Materiel Command, AMCP 706-177, Washington, D.C. January 1971. AD0764340
4. Alexander, B. Personal communication. BAE Systems, 26 July 2012.
5. Kaste, P. J.; Ceaser, J.; Lieb, R. J. *Scanning Electron Microscopy (SEM) to Probe Propellant Morphology*; ARL-TR-230; U.S. Army Research Laboratory: Aberdeen Proving Ground, MD, 1993.
6. Cowey, K.; Day, S.; Fryer, R. Examination of Wax-Coated RDX by Scanning Electron Microscopy and X-Ray Photoelectron Spectroscopy. *Propellants, Explosives, Pyrotechnics* **1985**, 10, 61–64. (ref for SEM of prills)
7. Hargreaves, A. E. Chemical Formulation: An Overview of Surfactant-based Preparations Used in Everyday Life. *Royal Society of Chemistry 2003*, 40.
8. Yeager, J. D.; Ramos, K. J.; Pesce-Rodriguez, R. A.; Piraino, S. M. Microstructural Effects of Processing in the Explosive Composition A-3. Submitted to *Materials Chemistry and Physics*, Sept. 2012.
9. Spanggord, R. J., et al. *Environmental Fate Studies on Certain Munitions Wastewater Constituents: Phase IV—Lagoon Model Studies*; U.S. Army Medical Research and Development Command: Frederick, MD, ADA138550, 23.
10. Phlen, J. M.; Romero, J. V.; Barnett, J. L.; Parker, D. R. *Solubility and Dissolution Kinetics of Composition B Explosive in Water*; SAND2002-2420; July 2002, 10.
11. Balakrishnan, V.; Halaz, A.; Harwari, J. Alkaline Hydrolysis of the Cyclic Nitramine Explosives RDX, HMX, and CL-20: New Insights into Degradation Pathways Obtained By the Observation of Novel Intermediates. *Environ. Sci. Technol.* **2003**, *37*, 1838–1843
12. Yashiro, K.; Furuta, A.; Tomita, Y. Molecular Dynamics Simulations of Nanoindentation on Folded Chain Crystal of Polyethylene. *Key Engineering Materials* **2005**, *297–300*, 2247–2252.

13. Sundararajan, P. R.; Kavassalis, T. A. Molecular Dynamics Study of Polyethylene Chain Folding: the Effects of Chain Length and the Tortional Barrier. *J. Chem. Soc. Faraday trans.* **1995**, *91* (16), 2541–2549.

14. Mirabile F. A., et al. Color Stabilized Oxidized Polyethylene Emulsions. U.S. Patent number: 3565840, Filing date: May 21, 1968, Issue date: 1971.

15. Oliver, W. C.; Pharr, G. M. An Improved Technique for Determining Hardness and Elastic Modulus Using Load and Displacement Sensing Indentation Experiments. *J. Mater. Res.* **1992**, 7, 1564–1583.

16. Ramos, K. J.; Hooks, D. E.; Bahr, D. F. Direct Observation of Plasticity and Quantitative Hardness Measurements in Single Crystal Cyclotrimethylene Trinitramine by Nanoindentation. *Philosophical Magazine* **2009**, *89*, 2381–2402.

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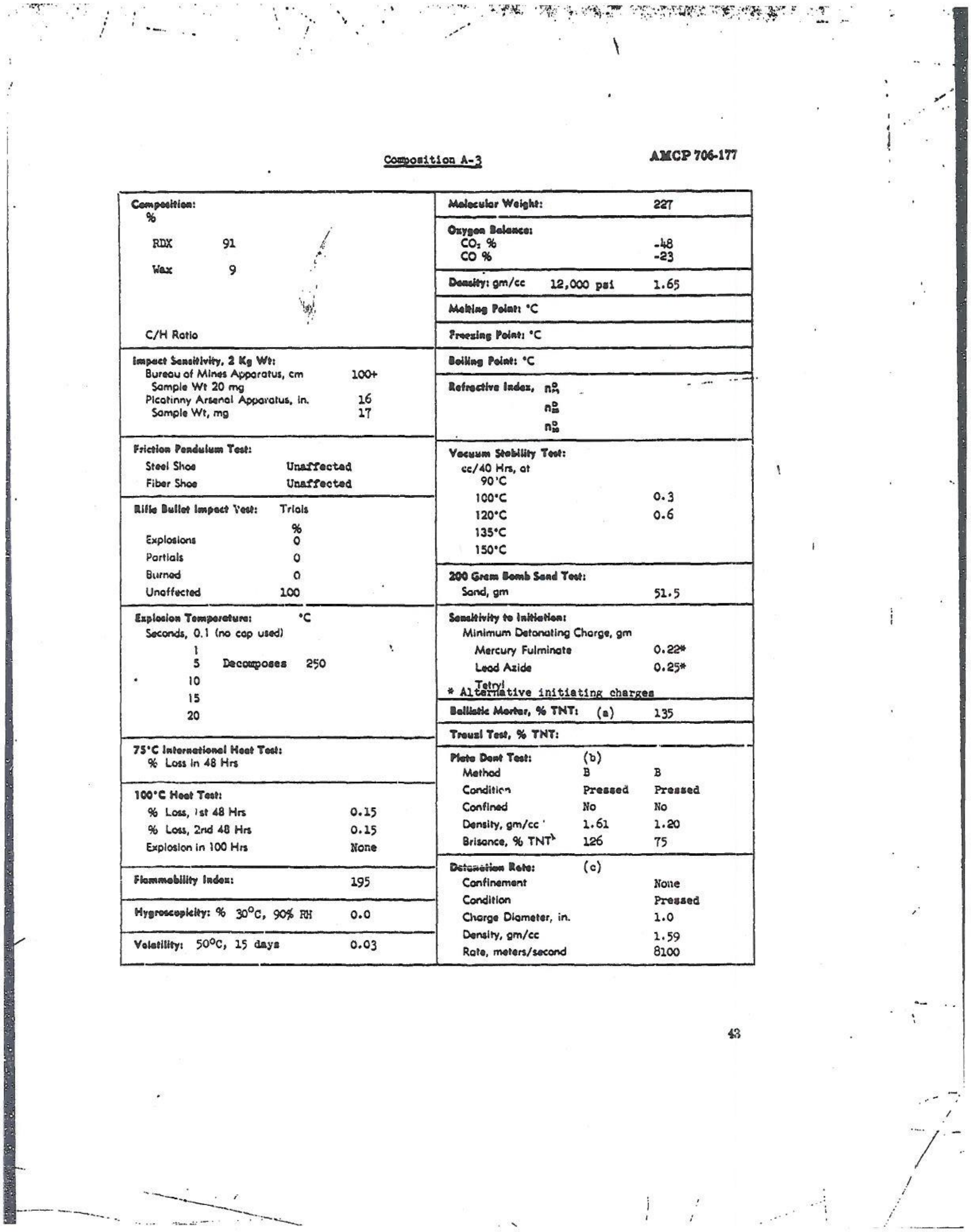
Appendix A. Specifications for Composition A-3

Note that in the attached, the composition of Comp A-3 is listed as 91% RDX and 9% polyethylene. This is not strictly true. As discussed in this ARL technical report, the polyethylene is actually oxidized polyethylene and contains residual impurities from the emulsion used to produce Comp A-3. The RDX also contains impurities (HMX, cyclohexanone).



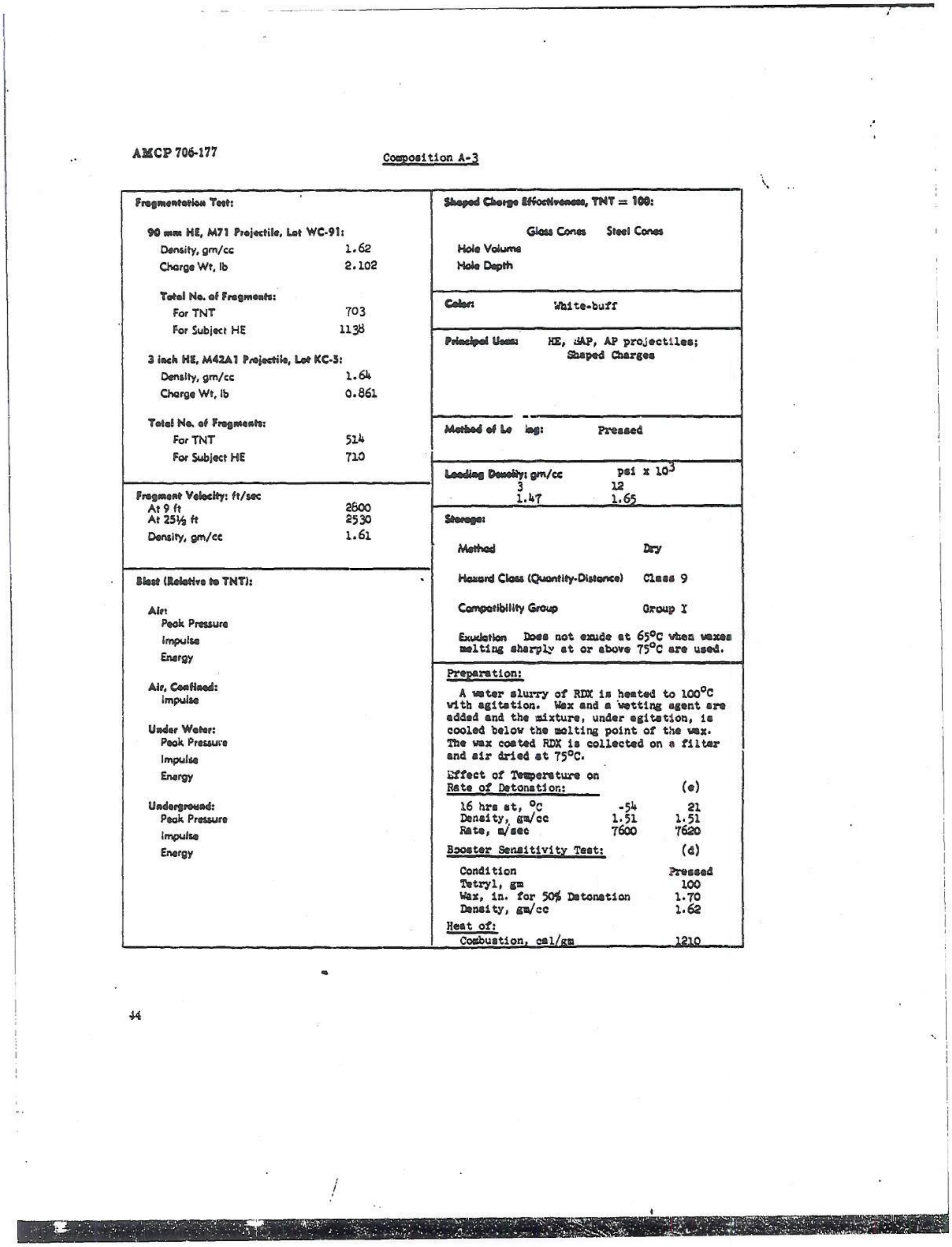
Composition A-3

AMCP 706-177

Composition:		
%		
RDX	91	
Wax	9	
C/H Ratio		
Impact Sensitivity, 2 Kg Wt:		
Bureau of Mines Apparatus, cm		100+
Sample Wt 20 mg		
Picatinny Arsenal Apparatus, in.		16
Sample Wt, mg		17
Friction Pendulum Test:		
Steel Shoe	Unaffected	
Fiber Shoe	Unaffected	
Rifle Bullet Impact Test:	Trials	
	%	
Explosions	0	
Partials	0	
Burned	0	
Unaffected	100	
Explosion Temperature:	°C	
Seconds, 0.1 (no cap used)		
1		
5 Decomposes	250	
10		
15		
20		
75°C International Heat Test:		
% Loss in 48 Hrs		
100°C Heat Test:		
% Loss, 1st 48 Hrs		0.15
% Loss, 2nd 48 Hrs		0.15
Explosion in 100 Hrs		None
Flammability Index:		195
Hygroscopicity: % 30°C, 90% RH		0.0
Volatility: 50°C, 15 days		0.03

Molecular Weight:		227
Oxygen Balance:		
CO_2 %		-48
CO %		-23
Density: gm/cc	12,000 psi	1.65
Melting Point: °C		
Freezing Point: °C		
Boiling Point: °C		
Refractive Index, n^{D}_{20}		
n^{D}_{25}		
n^{D}_{30}		
Vacuum Stability Test:		
cc/40 Hrs, at		
90°C		
100°C		0.3
120°C		0.6
135°C		
150°C		
200 Gram Bomb Sand Test:		
Sand, gm		51.5
Sensitivity to Initiation:		
Minimum Detonating Charge, gm		
Mercury Fulminate		0.22*
Lead Azide		0.25*
Tetryl		
* Alternative initiating charges		
Ballistic Mortar, % TNT:	(a)	135
Trauzl Test, % TNT:		
Plate Dent Test:	(b)	
Method	B	B
Condition	Pressed	Pressed
Confined	No	No
Density, gm/cc	1.61	1.20
Brisance, % TNT	126	75
Detonation Rate:	(c)	
Confinement		None
Condition		Pressed
Charge Diameter, in.		1.0
Density, gm/cc		1.59
Rate, meters/second		8100

43



Composition A-3

Fragmentation Test:

90 mm HE, M71 Projectile, Lot WC-91:

Density, gm/cc	1.62
Charge Wt, lb	2.102
Total No. of Fragments:	
For TNT	703
For Subject HE	1138

3 inch HE, M42A1 Projectile, Lot KC-5:

Density, gm/cc	1.64
Charge Wt, lb	0.861
Total No. of Fragments:	
For TNT	514
For Subject HE	710

Fragment Velocity: ft/sec

At 9 ft	2800
At 25½ ft	2530
Density, gm/cc	1.61

Blast (Relative to TNT):

Air:
Peak Pressure
Impulse
Energy

Air, Confined:
Impulse

Under Water:
Peak Pressure
Impulse
Energy

Underground:
Peak Pressure
Impulse
Energy

Shaped Charge Effectiveness, TNT = 100:

	Glass Cones	Steel Cones
Hole Volume		
Hole Depth		

Color: White-buff

Principal Uses: HE, SAP, AP projectiles; Shaped Charges

Method of Loading: Pressed

Loading Density: gm/cc

psi x 10^3	3	12
gm/cc	1.47	1.65

Storage:

Method	Dry
Hazard Class (Quantity-Distance)	Class 9
Compatibility Group	Group I

Exudation Does not exude at 65°C when waxes melting sharply at or above 75°C are used.

Preparation:

A water slurry of RDX is heated to 100°C with agitation. Wax and a wetting agent are added and the mixture, under agitation, is cooled below the melting point of the wax. The wax coated RDX is collected on a filter and air dried at 75°C.

Effect of Temperature on Rate of Detonation: (e)

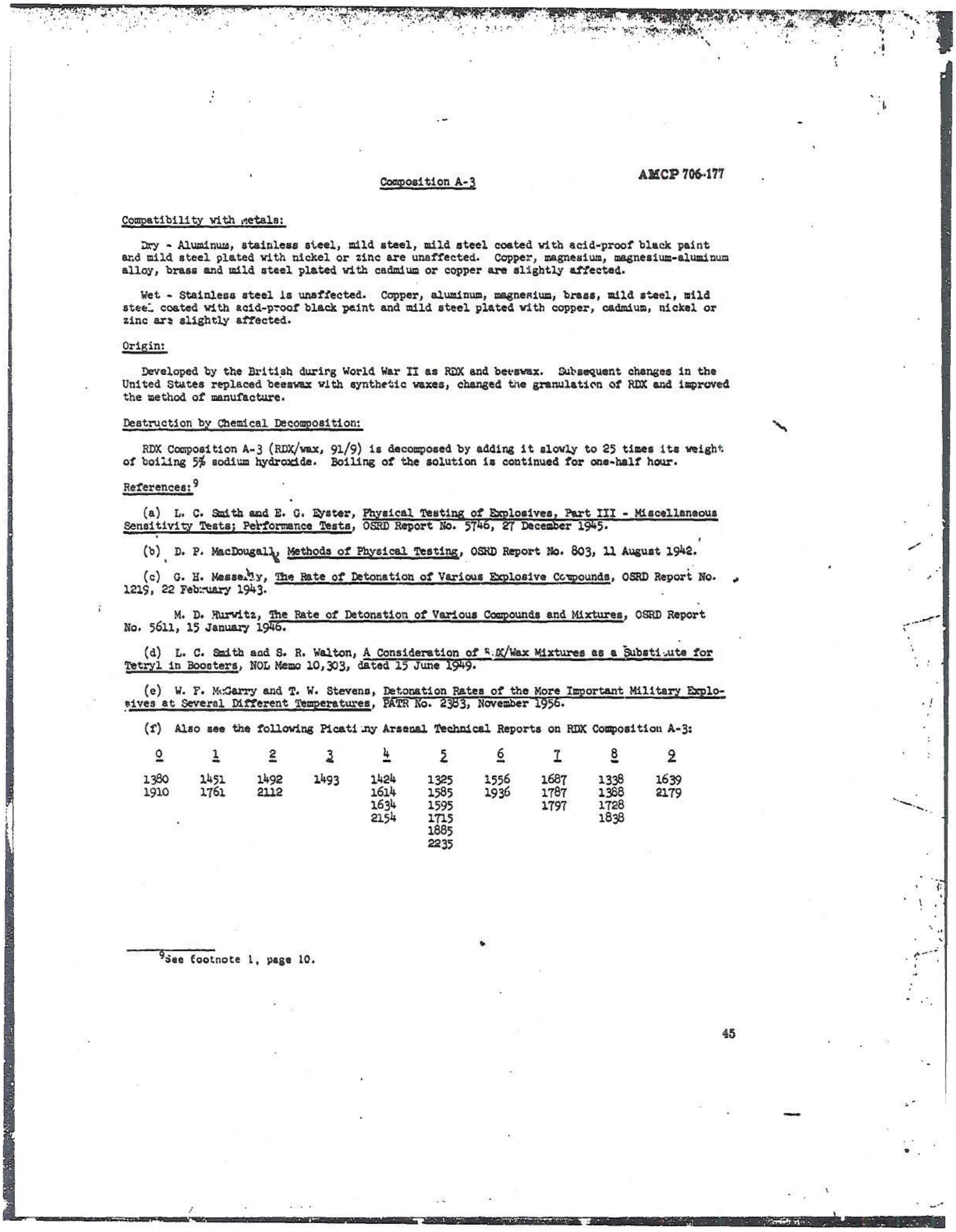
16 hrs at, °C	-54	21
Density, gm/cc	1.51	1.51
Rate, m/sec	7600	7620

Booster Sensitivity Test: (d)

Condition	Pressed
Tetryl, gm	100
Wax, in. for 50% Detonation	1.70
Density, gm/cc	1.62

Heat of:

Combustion, cal/gm	1210



Composition A-3

Compatibility with metals:

Dry - Aluminum, stainless steel, mild steel, mild steel coated with acid-proof black paint and mild steel plated with nickel or zinc are unaffected. Copper, magnesium, magnesium-aluminum alloy, brass and mild steel plated with cadmium or copper are slightly affected.

Wet - Stainless steel is unaffected. Copper, aluminum, magnesium, brass, mild steel, mild steel coated with acid-proof black paint and mild steel plated with copper, cadmium, nickel or zinc are slightly affected.

Origin:

Developed by the British during World War II as RDX and beeswax. Subsequent changes in the United States replaced beeswax with synthetic waxes, changed the granulation of RDX and improved the method of manufacture.

Destruction by Chemical Decomposition:

RDX Composition A-3 (RDX/wax, 91/9) is decomposed by adding it slowly to 25 times its weight of boiling 5% sodium hydroxide. Boiling of the solution is continued for one-half hour.

References:[9]

(a) L. C. Smith and E. G. Eyster, Physical Testing of Explosives, Part III - Miscellaneous Sensitivity Tests; Performance Tests, OSRD Report No. 5746, 27 December 1945.

(b) D. P. MacDougall, Methods of Physical Testing, OSRD Report No. 803, 11 August 1942.

(c) G. H. Messerly, The Rate of Detonation of Various Explosive Compounds, OSRD Report No. 1219, 22 February 1943.

M. D. Hurwitz, The Rate of Detonation of Various Compounds and Mixtures, OSRD Report No. 5611, 15 January 1946.

(d) L. C. Smith and S. R. Walton, A Consideration of RDX/Wax Mixtures as a Substitute for Tetryl in Boosters, NOL Memo 10,303, dated 15 June 1949.

(e) W. F. McGarry and T. W. Stevens, Detonation Rates of the More Important Military Explosives at Several Different Temperatures, PATR No. 2383, November 1956.

(f) Also see the following Picatinny Arsenal Technical Reports on RDX Composition A-3:

0	1	2	3	4	5	6	7	8	9
1380	1451	1492	1493	1424	1325	1556	1687	1338	1639
1910	1761	2112		1614	1585	1936	1787	1368	2179
				1634	1595		1797	1728	
				2154	1715			1838	
					1885				
					2235				

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[9] See footnote 1, page 10.

Appendix B. Specifications for Oxidized Polyethylene Homopolymer

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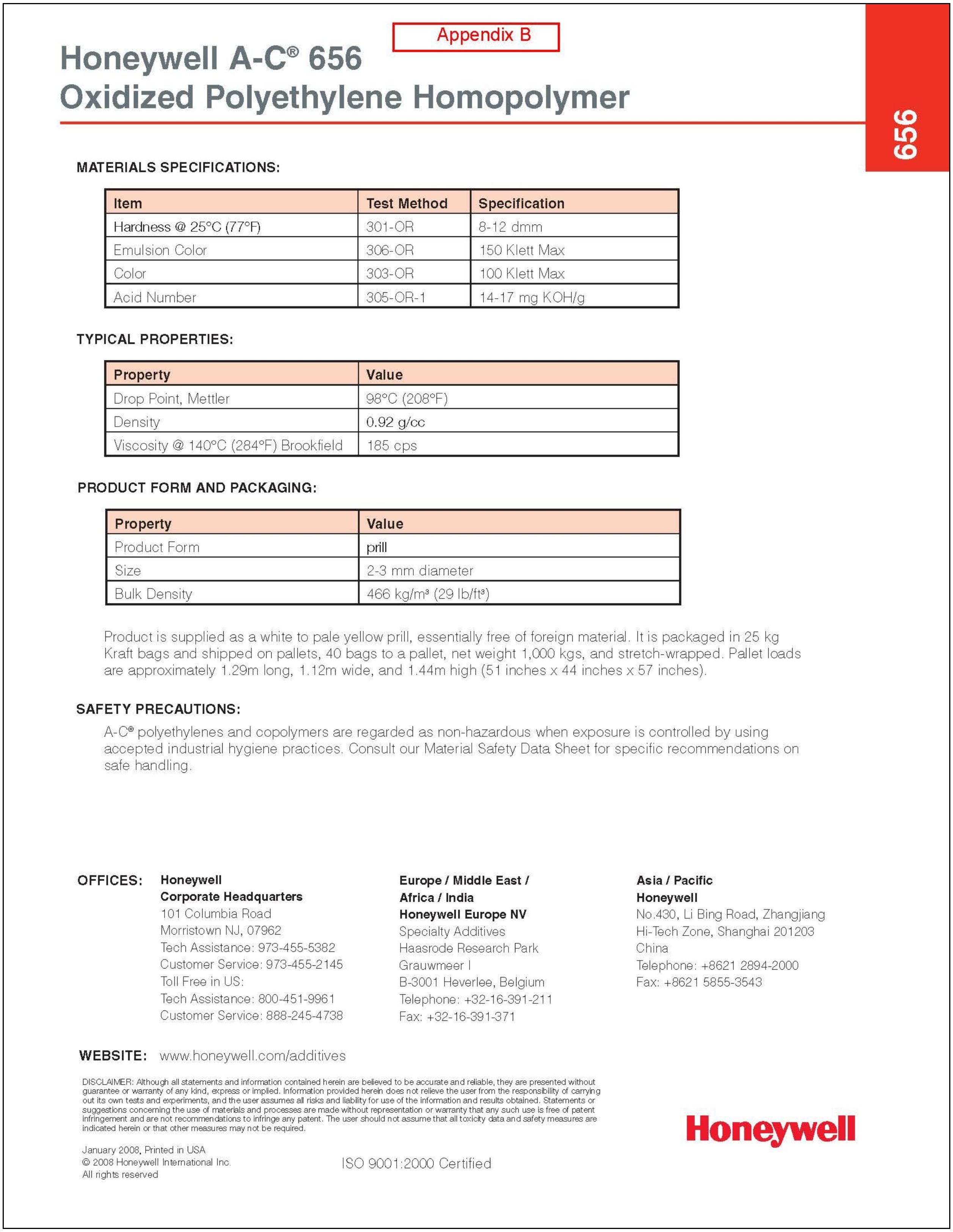
The following is an information sheet provided by Honeywell, the manufacturer of the oxidized polyethylene homopolymer (A-C 656) used in Comp A-3.



Appendix B

656

Honeywell A-C® 656 Oxidized Polyethylene Homopolymer

MATERIALS SPECIFICATIONS:

Item	Test Method	Specification
Hardness @ 25°C (77°F)	301-OR	8-12 dmm
Emulsion Color	306-OR	150 Klett Max
Color	303-OR	100 Klett Max
Acid Number	305-OR-1	14-17 mg KOH/g

TYPICAL PROPERTIES:

Property	Value
Drop Point, Mettler	98°C (208°F)
Density	0.92 g/cc
Viscosity @ 140°C (284°F) Brookfield	185 cps

PRODUCT FORM AND PACKAGING:

Property	Value
Product Form	prill
Size	2-3 mm diameter
Bulk Density	466 kg/m³ (29 lb/ft³)

Product is supplied as a white to pale yellow prill, essentially free of foreign material. It is packaged in 25 kg Kraft bags and shipped on pallets, 40 bags to a pallet, net weight 1,000 kgs, and stretch-wrapped. Pallet loads are approximately 1.29m long, 1.12m wide, and 1.44m high (51 inches x 44 inches x 57 inches).

SAFETY PRECAUTIONS:

A-C® polyethylenes and copolymers are regarded as non-hazardous when exposure is controlled by using accepted industrial hygiene practices. Consult our Material Safety Data Sheet for specific recommendations on safe handling.

OFFICES:

Honeywell
Corporate Headquarters
101 Columbia Road
Morristown NJ, 07962
Tech Assistance: 973-455-5382
Customer Service: 973-455-2145
Toll Free in US:
Tech Assistance: 800-451-9961
Customer Service: 888-245-4738

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Telephone: +8621 2894-2000
Fax: +8621 5855-3543

WEBSITE: www.honeywell.com/additives

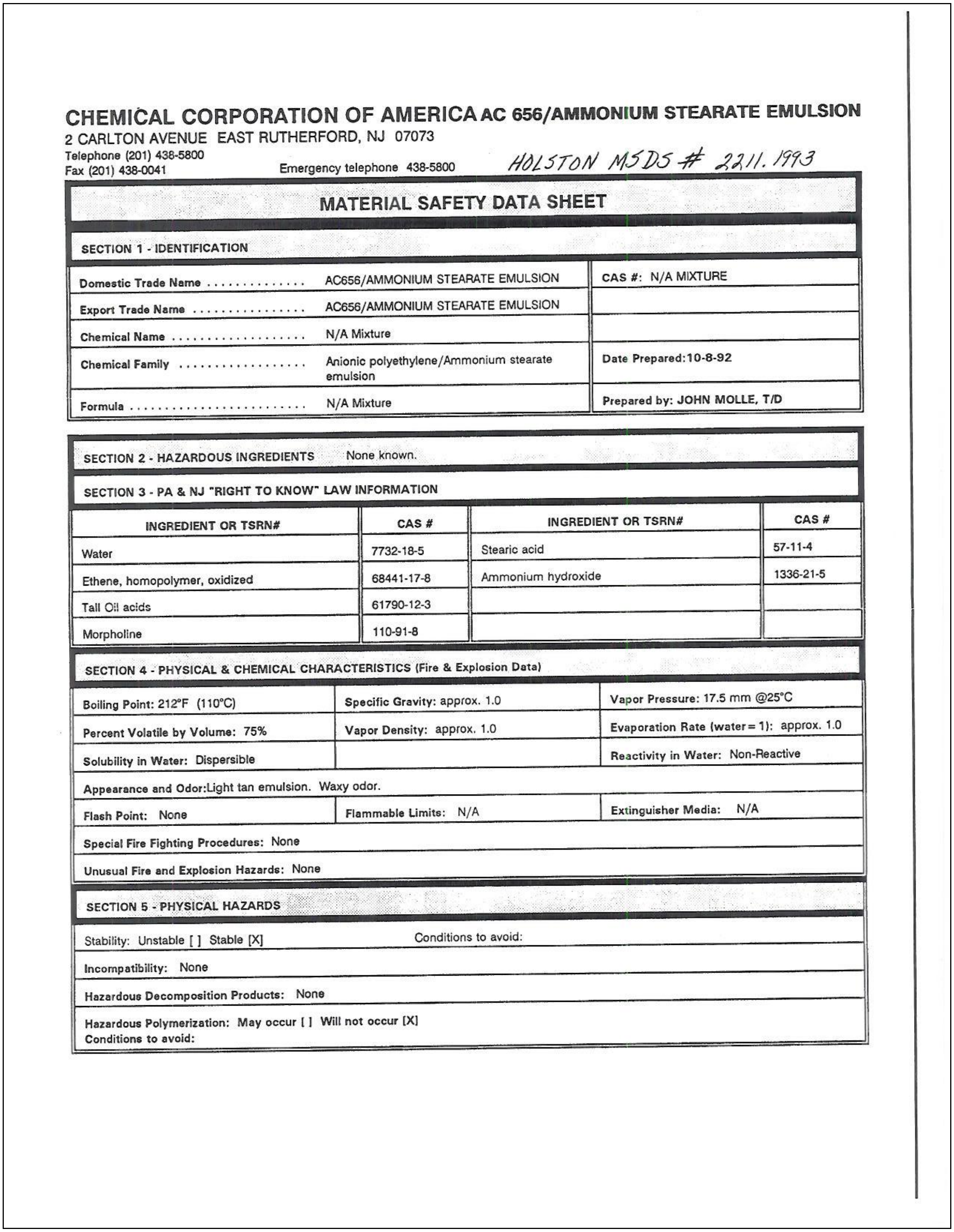
Honeywell

January 2008, Printed in USA

ISO 9001:2000 Certified

Appendix C. MSDS Analysis and Inspection Report, and Product quality Control Report for AC 656- Ammonium Sterate Emulsion

The following are documents provided by BAE Systems-Holston for the OPE emulsion used to formulate Comp A-3.



CHEMICAL CORPORATION OF AMERICA **AC 656/AMMONIUM STEARATE EMULSION**

2 CARLTON AVENUE EAST RUTHERFORD, NJ 07073
Telephone (201) 438-5800
Fax (201) 438-0041
Emergency telephone 438-5800

HOLSTON MSDS # 2211.1993

MATERIAL SAFETY DATA SHEET

SECTION 1 - IDENTIFICATION

Domestic Trade Name	AC656/AMMONIUM STEARATE EMULSION	**CAS #:** N/A MIXTURE
Export Trade Name	AC656/AMMONIUM STEARATE EMULSION	
Chemical Name	N/A Mixture	
Chemical Family	Anionic polyethylene/Ammonium stearate emulsion	**Date Prepared:10-8-92**
Formula	N/A Mixture	**Prepared by: JOHN MOLLE, T/D**

SECTION 2 - HAZARDOUS INGREDIENTS None known.

SECTION 3 - PA & NJ "RIGHT TO KNOW" LAW INFORMATION

INGREDIENT OR TSRN#	CAS #	INGREDIENT OR TSRN#	CAS #
Water	7732-18-5	Stearic acid	57-11-4
Ethene, homopolymer, oxidized	68441-17-8	Ammonium hydroxide	1336-21-5
Tall Oil acids	61790-12-3		
Morpholine	110-91-8		

SECTION 4 - PHYSICAL & CHEMICAL CHARACTERISTICS (Fire & Explosion Data)

Boiling Point: 212°F (110°C)	**Specific Gravity:** approx. 1.0	**Vapor Pressure:** 17.5 mm @25°C
Percent Volatile by Volume: 75%	**Vapor Density:** approx. 1.0	**Evaporation Rate (water = 1):** approx. 1.0
Solubility in Water: Dispersible		**Reactivity in Water:** Non-Reactive
Appearance and Odor: Light tan emulsion. Waxy odor.		
Flash Point: None	**Flammable Limits:** N/A	**Extinguisher Media:** N/A
Special Fire Fighting Procedures: None		
Unusual Fire and Explosion Hazards: None		

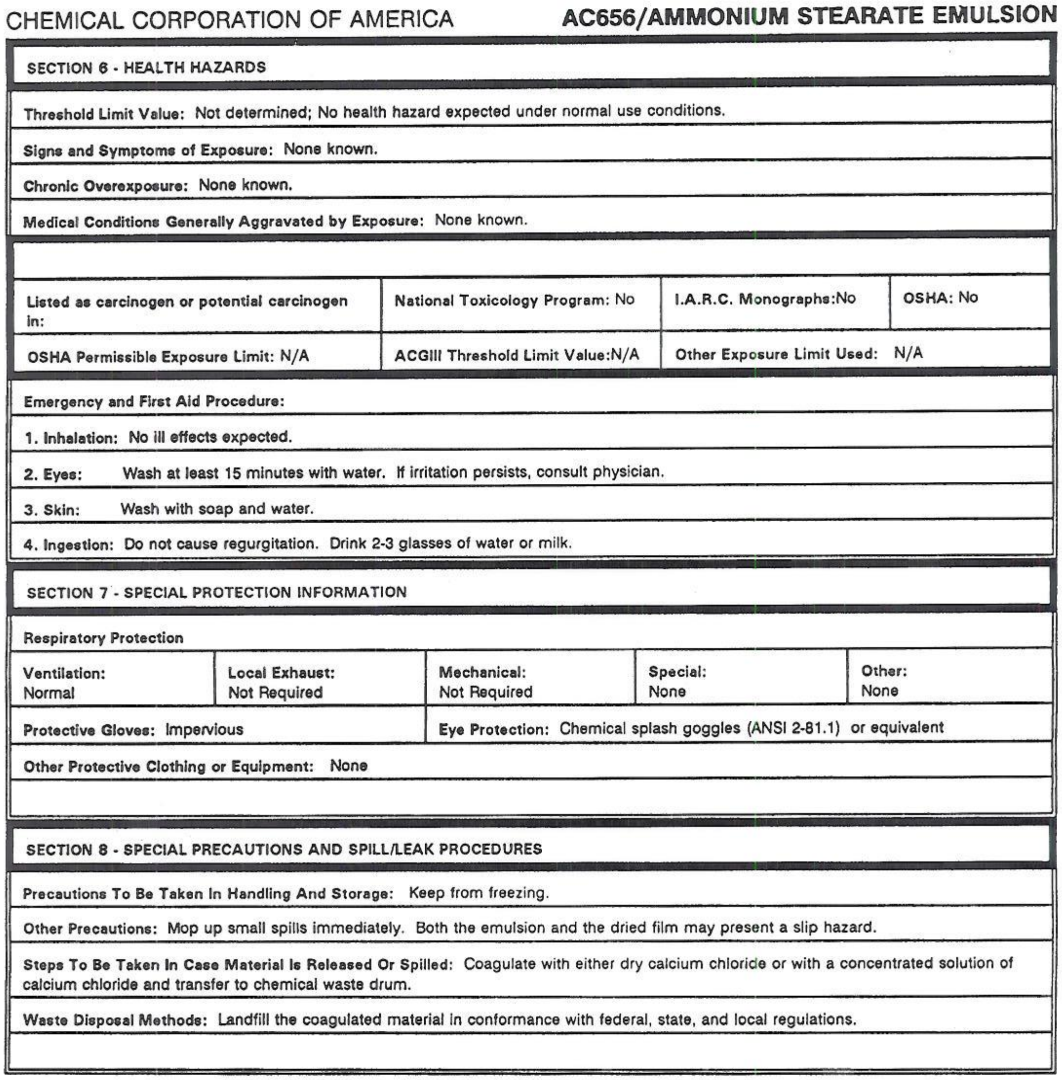
SECTION 5 - PHYSICAL HAZARDS

Stability: Unstable [ ] Stable [X] Conditions to avoid:

Incompatibility: None

Hazardous Decomposition Products: None

Hazardous Polymerization: **May occur [ ] Will not occur [X]**
Conditions to avoid:



CHEMICAL CORPORATION OF AMERICA **AC656/AMMONIUM STEARATE EMULSION**

SECTION 6 - HEALTH HAZARDS

Threshold Limit Value: Not determined; No health hazard expected under normal use conditions.

Signs and Symptoms of Exposure: None known.

Chronic Overexposure: None known.

Medical Conditions Generally Aggravated by Exposure: None known.

Listed as carcinogen or potential carcinogen in:	**National Toxicology Program:** No	**I.A.R.C. Monographs:**No	**OSHA:** No
OSHA Permissible Exposure Limit: N/A	**ACGIH Threshold Limit Value:**N/A	**Other Exposure Limit Used:** N/A	

Emergency and First Aid Procedure:

1. Inhalation: No ill effects expected.

2. Eyes: Wash at least 15 minutes with water. If irritation persists, consult physician.

3. Skin: Wash with soap and water.

4. Ingestion: Do not cause regurgitation. Drink 2-3 glasses of water or milk.

SECTION 7 - SPECIAL PROTECTION INFORMATION

Respiratory Protection

Ventilation: Normal	**Local Exhaust:** Not Required	**Mechanical:** Not Required	**Special:** None	**Other:** None

Protective Gloves: Impervious

Eye Protection: Chemical splash goggles (ANSI 2-81.1) or equivalent

Other Protective Clothing or Equipment: None

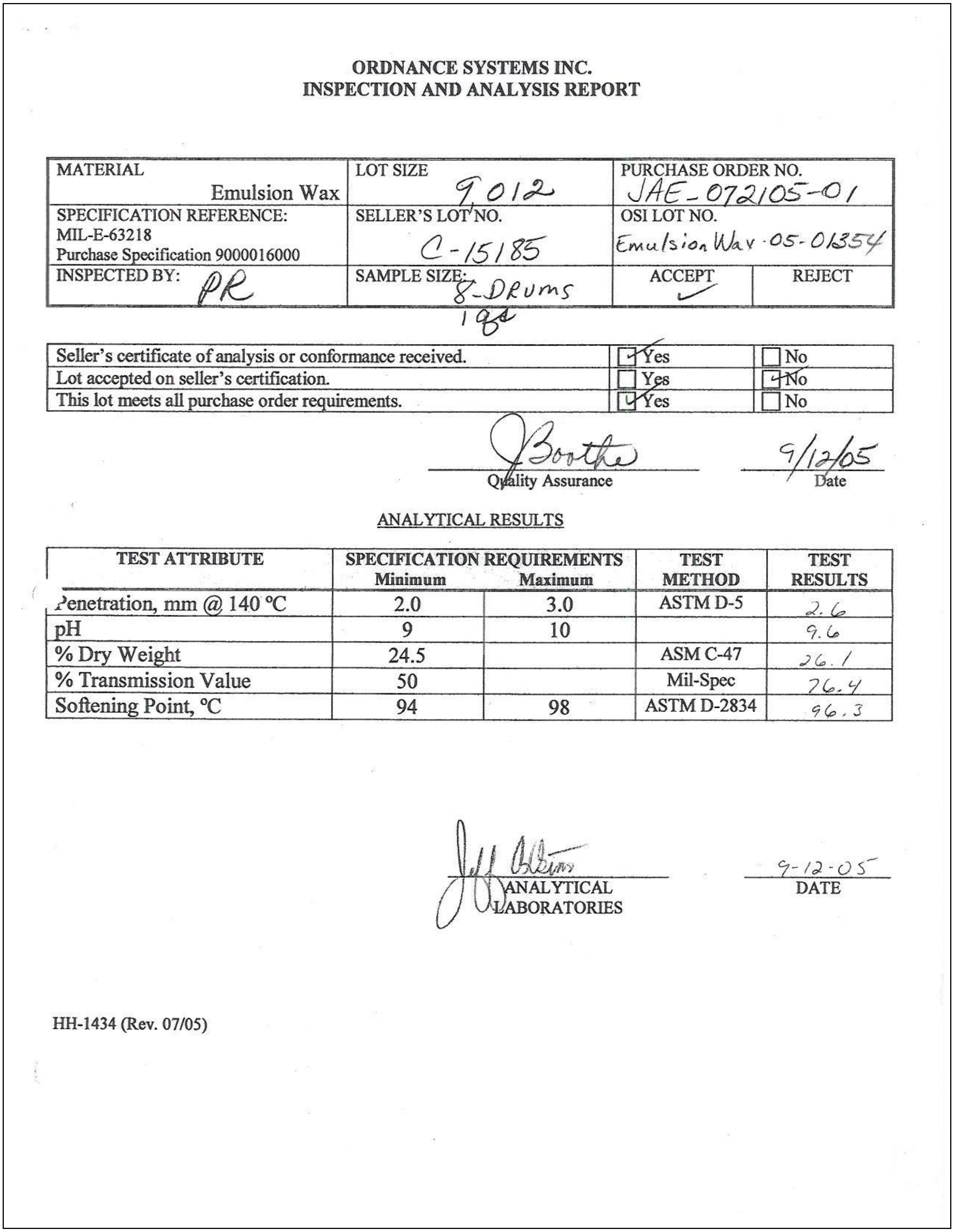
SECTION 8 - SPECIAL PRECAUTIONS AND SPILL/LEAK PROCEDURES

Precautions To Be Taken In Handling And Storage: Keep from freezing.

Other Precautions: Mop up small spills immediately. Both the emulsion and the dried film may present a slip hazard.

Steps To Be Taken In Case Material Is Released Or Spilled: Coagulate with either dry calcium chloride or with a concentrated solution of calcium chloride and transfer to chemical waste drum.

Waste Disposal Methods: Landfill the coagulated material in conformance with federal, state, and local regulations.



ORDNANCE SYSTEMS INC.
INSPECTION AND ANALYSIS REPORT

MATERIAL Emulsion Wax	LOT SIZE 9,012	PURCHASE ORDER NO. JAE-072105-01
SPECIFICATION REFERENCE: MIL-E-63218 Purchase Specification 9000016000	SELLER'S LOT NO. C-15185	OSI LOT NO. Emulsion Wax-05-01354
INSPECTED BY: PR	SAMPLE SIZE: 8-DRUMS 1 qt	ACCEPT ✓ / REJECT

Seller's certificate of analysis or conformance received.	☑ Yes	☐ No
Lot accepted on seller's certification.	☐ Yes	☑ No
This lot meets all purchase order requirements.	☑ Yes	☐ No

Boothe
Quality Assurance

9/12/05
Date

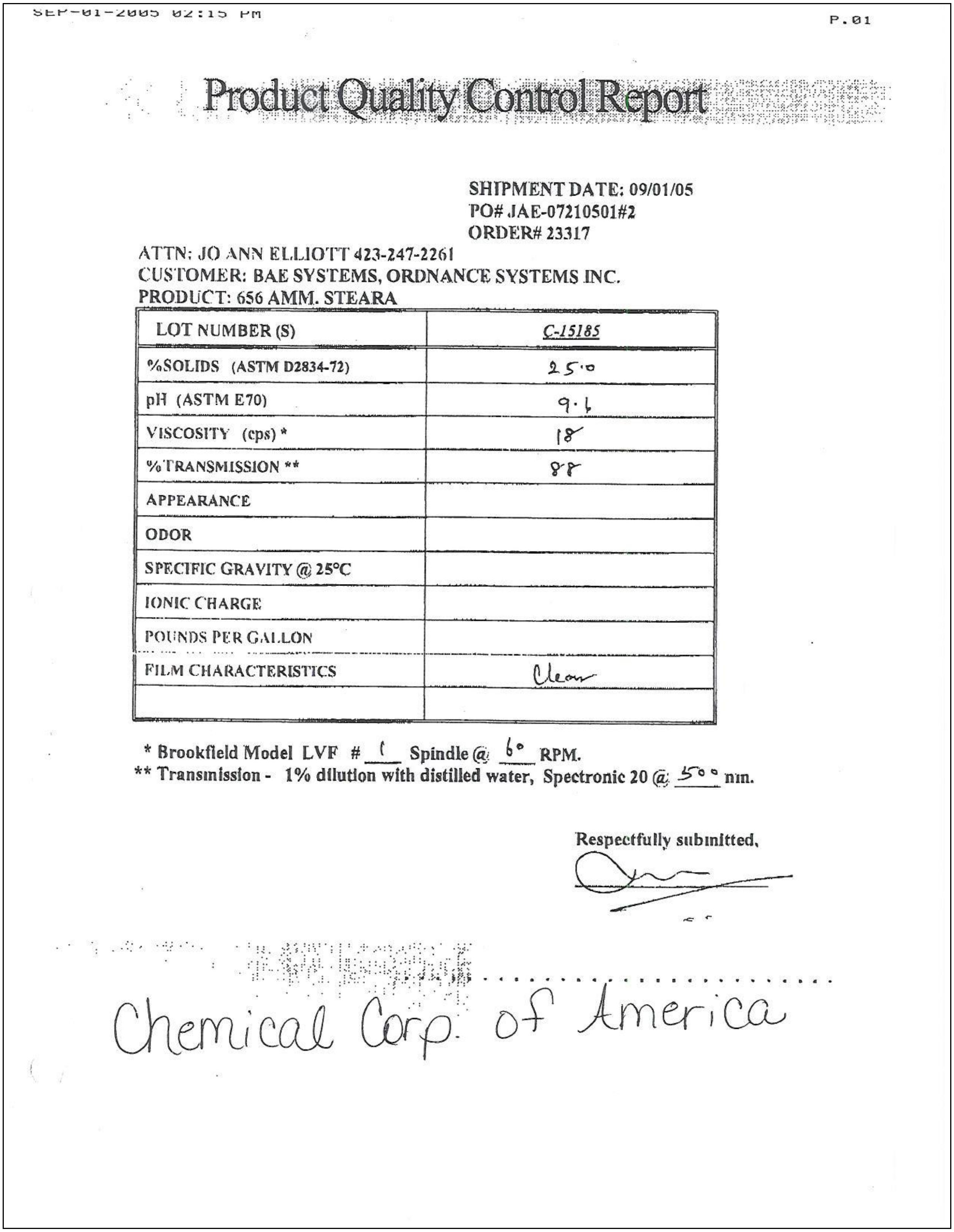
ANALYTICAL RESULTS

TEST ATTRIBUTE	SPECIFICATION REQUIREMENTS Minimum	SPECIFICATION REQUIREMENTS Maximum	TEST METHOD	TEST RESULTS
Penetration, mm @ 140 °C	2.0	3.0	ASTM D-5	2.6
pH	9	10		9.6
% Dry Weight	24.5		ASM C-47	26.1
% Transmission Value	50		Mil-Spec	76.4
Softening Point, °C	94	98	ASTM D-2834	96.3

Jeff
ANALYTICAL LABORATORIES

9-12-05
DATE

HH-1434 (Rev. 07/05)



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Product Quality Control Report

SHIPMENT DATE: 09/01/05
PO# JAE-07210501#2
ORDER# 23317

ATTN: JO ANN ELLIOTT 423-247-2261
CUSTOMER: BAE SYSTEMS, ORDNANCE SYSTEMS INC.
PRODUCT: 656 AMM. STEARA

LOT NUMBER (S)	*C-15185*
%SOLIDS (ASTM D2834-72)	25.0
pH (ASTM E70)	9.6
VISCOSITY (cps) *	18
% TRANSMISSION **	88
APPEARANCE	
ODOR	
SPECIFIC GRAVITY @ 25°C	
IONIC CHARGE	
POUNDS PER GALLON	
FILM CHARACTERISTICS	Clear

* Brookfield Model LVF # 1 Spindle @ 60 RPM.
** Transmission - 1% dilution with distilled water, Spectronic 20 @ 500 nm.

Respectfully submitted,

Chemical Corp. of America

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List of Symbols, Abbreviations, and Acronyms

ARL	U.S. Army Research Laboratory
ATR	attenuated total reflectance
Comp A-3	Composition A-3
D-GC-MS	desorption-gas chromatography-mass spectrometry
DSC	differential scanning calorimetry
DTGS	deuterated triglycine sulfate
EM	energetic materials
FTIR	Fourier transform infrared
GC-MS	gas chromatography-mass spectrometry
HPLC	high performance liquid chromatography
IM	Insensitive Munitions
IR	infrared
KBr	potassium bromide
LANL	Los Alamos National Laboratory
M&S	modeling and simulation
MREM	Multi-scale Response of Energetic Materials
OPE	oxidized polyethylene
PE	polyethylene
SEM	scanning electron microscopy
SIC	selected ion chromatogram
SPM	scanning probe microscopy
TIC	total ion chromatogram

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