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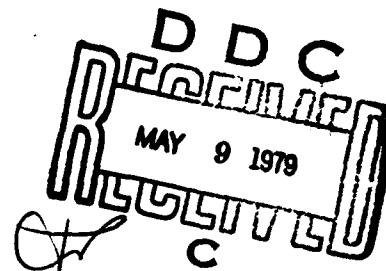
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EFFECT OF HNS ON PHYSICAL PROPERTIES
OF TNT EXPLOSIVES:
SURVEILLANCE EVALUATION

March 1979



NWS

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20. ABSTRACT

A laboratory scale study on the effects of small percentages of the anticracking explosive additive, 2,2',4,4',6,6' hexanitrostilbene (HNS), on the physical characteristics (i.e., cracking, density, exudation, crystal pattern, and dimensional growth) of small billets of TNT and TNT-based explosive compositions was conducted by subjecting them to an extended JAN temperature cycle of 6 to 21 months. An evaluation of the Swedish Bofors processing technique for the incorporation of HNS was included.

The immediate improvement in the handling qualities of the billets was not significantly reflected in an improvement in long-term irreversible growth; in fact, growth resulted in crumbling with time. The Bofors two-stage method and the minimum of 30 minutes required to achieve maximum solubility at 100°C of the HNS in TNT resulted in increasing processing time by a considerable factor. The close temperature control, 83° to 86°C, on remelt also is a disadvantage in a production process.

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F O R E W O R D

1. This report describes the effects of small percentages of the anticracking explosive additive, 2,2',4,4',6,6' hexanitrostilbene (HNS), on the physical characteristics of small billets of trinitrotoluene (TNT) and TNT-based explosive compositions.
2. The effort reported herein was authorized and funded under the Naval Surface Weapons Center, White Oak Laboratory, Silver Spring, Maryland Work Request WR-W-0005 of 3 October 1977.

Released by

W. McBride

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Naval Explosives Development
Engineering Department
March 1979

Under authority of
LEO A. HIBSON, JR.
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EFFECT OF HNS ON PHYSICAL PROPERTIES OF TNT EXPLOSIVES:

SURVEILLANCE EVALUATION

I. OBJECTIVE AND PLAN

The objective was to examine, on a laboratory scale, the effects of small percentages of the anticracking explosive additive, 2,2',4,4',6,6' hexanitrostilbene (HNS), on the physical characteristics (i.e., cracking, density, exudation, crystal pattern, and dimensional growth) of small billets of trinitrotoluene (TNT) and TNT-based explosive compositions by subjecting them to an extended WR-50¹ JAN temperature cycle. The regular 28-day cycle is a major requirement in the qualification tests for final warhead acceptance. The TNT was to be selected from both the older batch method and the more recent continuous process method of manufacture. At the end of one year, the optimum amount of HNS was to be determined and a second phase of study using larger billets would be initiated based on this optimum HNS percentage. The study was to include an evaluation of the Bofors² processing technique for the incorporation of the HNS.

II. DISCUSSION

A. Background

HNS is a high temperature, heat resistant material developed by the Naval Surface Weapons Center (NAVSWC), White Oak (formerly the Naval Ordnance Laboratory).^{3 4 5} It has been produced commercially by United Technologies, Teledyne, and Chemtronics. The material is

¹NAVSEA WR-50, *Warhead Safety Tests, Minimum for Air, Surface and Underwater Launched Weapons (Excluding Mine and Nuclear Warheads)*, 13 Feb 1964.

²J.S. Back, J.L. Soderberg and C.L. Hakanson, Patent Specification 1249038, Bofors Company, Sweden, filed 22 Apr 1969, complete spec published 6 Oct 1971.

³K.G. Shipp, NOLTR 64-34, *Heat Resistant Explosives, XVI: A New Synthesis of 2,2',4,4',6,6' Hexanitrostilbene, HNS (C)*, 22 Apr 1964 CONF - Declassified.

⁴E. Eugene Kilmer, NOLTR 65-170, *Development of a High Temperature Resistant Mild Detonating Fuse V (U)*, 10 Jun 1966 CONF - Declassified.

⁵K.G. Shipp, NOLTR 62-107, *Heat Resistant Explosives, XIV: Synthesis and Properties of 2,2',4,4',6,6' Hexanitrostilbene, HNS (C)*, 15 Jun 1962 CONF - Declassified.

manufactured in the crude form, HNS I, and a purified form, HNS II. The crude form crystallizes in fine particles characterized by a cluster of needles which are highly twinned. This crystal structure has been reported as orthorhombic⁵ or it may be monoclinic.⁶ The HNS II produced by the acetonitrile-toluene recrystallization⁶ gives large particles, nominal 50 to 100 microns, with a lower l/d ratio.

A Swedish patent specification by Bofors Company of Apr 1968 described HNS as an additive for TNT and the cyclotol, Comp B. The recommended concentration of HNS was from 1/2% to 2%. The patent described a better way of obtaining a microcrystalline charge with a random crystal structure. It stated that a microcrystalline structure was less prone to cracking than that normally obtained in TNT castings. The Swedish patent also reported the selection of HNS as an additive would not cause significant changes in composition and performance of the explosive, would not form large quantities of low melting eutectics, and would be satisfactory for forming nuclei for TNT crystals.

TNT, upon solidifying from a melt, can be expected to crystallize in the monoclinic system above 75 degrees Celsius (°C) and in an orthorhombic form below that temperature with transitions from one form to the other and between variants within the same crystal class.^{7 8} It exhibits a strong tendency to supercool. The crystals formed have a coefficient of longitudinal expansion along the shortest axis which is approximately four times that of the longest axis.^{7 9} The results are friable castings and cracking which follows the longer crystal axis.

TNT is frequently used as the castable matrix for the high energy explosive, RDX. The term cyclotol is applied to this family of explosives. Common cyclotols are Comp B and its aluminized version, H-6. As both RDX and TNT crystallize in the highly anisotropic orthorhombic system, crystallization takes place without systematic orientation.

⁶Francis Taylor, Jr. and R.E. Oesterling, NOLTR 65-142, *Heat Resistant Explosives, XX: Production of Grade II HNS (C)*, 26 Aug 1965 CONF - Declassified.

⁷F.C. Rauch and R.B. Wainwright, *Studies on Composition B, Final Report*, Contract No. DAAA21-68-C-0334, American Cyanamid Company for Picatinny Arsenal, Dover, N.J., Feb 1969.

⁸F.C. Rauch and W.P. Colman, *Studies on Composition B, Final Report*, Contract No. DAAA21-68-C-0334, American Cyanamid Company for Picatinny Arsenal, Dover, N.J., Mar 1970.

⁹W. Kegler and A. Baumann, *Studies on Changes in Density of Cast TNT-RDX Charges Resulting from Cyclic Heat Treatment*, *Explosivstoffe*, Vol 13, No. 7, Jul 1965.

This becomes a major factor in irreversible growth of cyclotols when subjected to temperature cycling.⁸ RDX solubility in TNT ranges from ~3% at 79°C to ~7% at 100°C.¹⁰

Military grade TNT produced by either the batch or the continuous process for explosive loadings contains a number of impurities including isomers and homologs which form low melting eutectics. The continuous process results in a TNT with more exudate and with 2,4 dinitrotoluene as the major impurity. Information at length has been published^{9 10 11 12 13 14} on TNT crystallization, with a description of the behavior of additives such as 2,4,6 trinitrostilbene and alpha nitronaphthalene (α NN), and irreversible growth and eutectic formation of conventional TNT and cyclotols.

As a result of the interest in HNS as an additive to improve the resistance to cracking of TNT and TNT-based production explosive loads, and to evaluate the Bofors process, an investigation was undertaken to determine the effect of HNS overall on physical properties of these type explosives at the optimum percentage under long-range storage conditions. The emphasis was on military grade TNT produced by the two types of manufacturing processes, batch and continuous. The storage conditions were based on the conventional WR-50 warhead acceptance test JAN temperature cycle of -54° to 71°C. The physical characteristics evaluated were exudation as weight loss, dimensional billet growth (diameter and length), density, cracking, friability and crystal pattern of small billets of TNT and cyclotols.

The initial study, designated Phase I, was centered on determining the optimum percentage of HNS to improve the physical characteristics of right cylinder billets of TNT and Comp B when subjected to the JAN temperature cycle. The billets, 1 inch in diameter by 2 inches long, were cycled for 21 months. On the basis of the data after one year of cycling, the most favorable percentage of HNS would be formulated into TNT, Comp B, and H-6 compositions cast to 2.0

¹⁰R.P. Parker and B.W. Thorpe, Tech Note 140, *The Phase Diagram of the RDX/TNT System*, DS, ADSSDSL, Australia, Jan 1970.

¹¹W.A. Gey, E.R. Dalbey, A.F. Teague, and R.W. VanDolah, NAVORD 1335, *Investigation of TNT Crystallization*, 7 Aug 1951.

¹²James E. Abalard, NAVSEA-03-TR-058, *Composition B: A Very Useful Explosive*, 1 Jul 1977.

¹³M.C. Chick, W. Connick, and B.W. Thorpe, *Microscope Observations of TNT Crystallization*, Journal of Crystal Growth, 7 (1970) 317-326, North-Holland Publishing Company, Netherlands.

¹⁴H.W. Voigt, TM 2188, *Pressed Explosive-Coated RDX for Shock Resistance: 91/9 RDX/Modified TNT*, Picatinny Arsenal, Dover, N.J., Nov 1975.

inches in diameter by 2.7 inches long. This would retain the right cylinder format at double the diameter, thereby increasing stress on the billets. This aspect of the study is described in Phase II.

B. Phase I

1. Introduction

As growth, exudation, and crystal formation would be expected to vary with the impurities present, four series of billets were prepared to give data on this variable: (1) TNT manufactured by the batch method, (2) TNT manufactured by the continuous process which has replaced the batch method, (3) Comp B which is a cyclotol containing 39.5% TNT, 59.5% RDX and 1% wax, and (4) purified TNT with a set point of 80.8°C. (Military grade TNT set point ranges from 80.2° to 80.6°C.) Each series contained billets with 0%, 1/2%, 1% and 2% HNS. This was the range given in the Swedish patent. Included as a control, because of its use as a cyclotol anticracking agent in past explosive formulations,^{12 15} were billets with 1% aNN in lieu of HNS.

2. Experimental

A description of batch materials for the test billets is given in Table I. The TNT used for the recrystallization was from a lot of Weldon Springs manufacture which is a purer TNT than the regular run of military grade TNT. The solvent used for the recrystallization was ethyl alcohol. Vacuum thermal stability (VTS) and impact sensitivity (IS) of TNT containing varying percentages of HNS were determined, Table II.

The HNS additive used in this surveillance study was HNS, Type IIB, Lot 66-12, obtained from Chemtronics. It was made under specification WS 5003.¹⁶ Type II signifies recrystallized and controlled bulk density and B signifies that it was not tested for gap sensitivity and output requirements of the specification. It was a purified form of Type I HNS with a melting point of 318°C and an assay better than 98%. The average nominal particle size was 50 to 100 microns which is considered a medium granulation. These values were determined by differential thermal analysis (DTA), high performance liquid chromatography (HPLC), and photomicrography, respectively. See Figures 1 thru 4. The HPLC procedure was a variation of that of C. L. Schaffer, described in Appendix 3 of Stull's

¹⁵WS 1726E, *Warhead, Guided Missile, Mk 51 Mod 0, Explosive Loaded Assembly*, 4 Apr 1968.

¹⁶WS 5003E, *HNS Explosive*, 3 Mar 1969.

MHSMP report.¹⁷ Standards for the HPLC analysis, with the exception of the TNT, were supplied by NAVSWC White Oak. These standards were HNS II, ID No. 2299, and the major impurity of HNS, hexanitrobibenzyl.¹⁸

Explosive batches of 1/2 kilogram were prepared in a manner similar to that described in the Swedish patent. These were heated at 95° to 100°C for a short time to allow some of the HNS, when present, to dissolve in the molten TNT. The melt was then poured into trays to a depth of a quarter of an inch for quick solidification. Later the material was broken into pieces and remelted with agitation. During the remelt, the temperature was held between 83° and 86°C. As soon as melting was completed, the explosive was cast into preheated aluminum molds, 1 inch in diameter by 4 inches high, fitted with aluminum risers. This two-step procedure was required to change the HNS to a small particle size. Fine HNS is required to provide nucleating centers for the TNT which is prone to supercool. The solubility of HNS in TNT is ~0.2% near the set point.²

The explosive loaded molds were transferred immediately after casting to a steam oven at 90°C. Subsequently, the heat was turned off to allow slow cooling overnight to simulate the cooling patterns that occur in large castings.

After removal from the molds, the right cylinder billets were sawed into two billets, each 1 inch in diameter by 2 inches long, ranging in weight from 40 to 60 grams. The sawed ends were faced with sandpaper and crocus cloth to give a smooth surface, and wiped lightly with an acetone damp cloth to facilitate observation of the crystal patterns. Three billets were selected for each test condition and were photographed, weighed, measured (length and diameter), and density calculated. Visual observations were made with respect to type of crystal formation, cracks, and friability.

The billets were then placed in glass jars fitted with screw caps and a layer of filter paper discs to absorb exudate. Closed containers were used to minimize weight loss resulting from sublimation. The billets were oriented in an upright position that corresponded to their original mold position.

The billets were cycled in an oven at $71.0^{\circ} \pm 0.5^{\circ}\text{C}$ and in the freezer at $-54.0^{\circ} \pm 0.5^{\circ}\text{C}$ over the test period. They were held for 24 hours at one temperature then transferred to the other. Data

¹⁷T.W. Stull, MHSMP-75-37, *Synthesis of High Purity Hexanitrostilbene*, Sep 1975.

¹⁸Eleonore G. Kayser, NSWC/WOL TR 77-154, *Analysis of 2,2',4,4',6,6' Hexanitrostilbene (HNS) by High Performance Liquid Chromatography*, 14 Mar 1978.

was obtained frequently at the beginning of the testing period then reduced as the rate of change slowed. The data points for the graphs are the average of three billets of each series for the 28-day cycle. Those for the 21-month cycle represent the best case survivor billet.

3. Results

a. Before cycling

Stability of the TNT/HNS was satisfactory and sensitivities were intermediate between the two components with no indication of incompatibility. The highly macrocrystalline nature of the TNT did not give way to the randomly oriented microcrystalline condition until 1% to 2% HNS was present. The macrocrystalline state was prone to chipping. The α NN did not reduce the formation of large crystals. By contrast, the Comp B specimens were similar to the TNT:HNS 2% condition. See Photos 1 and 2. The top views show the elongated habit of the TNT crystals.

b. 28-day cycle

Weight loss was primarily from exudation. The major portion occurred within the first week. The loss was related to the amount of TNT and the impurities present, i.e., continuous TNT > batch TNT > Comp B > recrystallized TNT. The overall losses for the two TNT's and the Comp B series ranged from 0.5% to 1.5% with the HNS additive generally increasing the loss by a small amount. The billets containing α NN lost more than 4% by weight for the three types of TNT and better than 3% with Comp B. The recrystallized TNT losses were less than 0.1% except in the presence of the α NN. A data plot is given in Figure 5.

Percentage growth for military grade TNT billets averaged 0.7% for both diameter and length. As expected, minimal growth was detected in the purified TNT without additives present. Comp B growth diameter was about double that of military grade TNT, ~1.5% versus 0.7%. The adverse increase caused by the α NN was quite apparent. Graphs are presented in Figures 6 and 7. Growth characteristics were reflected also in the decreasing density values, Figure 8.

Neat billets of batch TNT, recrystallized TNT, and Comp B had begun to exhibit hairline cracks. The highly crystalline billets of recrystallized TNT with 1/2% HNS or less and the TNT with 1% α NN were extremely fragile to handle, more so than the equivalent military grade TNT. The presence of 1% to 2% HNS gave improved handling characteristics, similar to that associated with neat Comp B.

c. 12-month cycle

Exudation had ceased to be a significant factor. For the military grades of TNT, with and without HNS, it took one week for the weight loss to reach 1% but another 6 months to approach 2%. By the end of the year it was ranging from 1.8% to 2.5%. Total Comp B weight loss was less than 1%. The TNT billets with α NN were in such poor condition from exudation and chipping that testing was discontinued after 9 months. See Figure 9 for the graph.

Diameter and length of the neat and HNS billets continued to increase with percentage of growth larger for the former dimension. The smallest change occurred with the purified TNT free of any additive, the largest for Comp B, Figures 10 and 11. The continued irreversible expansion in both directions was reflected in decreasing density, Figure 12. Density had decreased by 0.15 grams per milliliter (g/ml) or more for the military grade TNT's and by more than 0.20 g/ml for Comp B. Density differences greater than 0.02 g/ml are considered significant.

Continuous, batch, and recrystallized TNT billets, neat and with 1/2% HNS additive, had developed cracks along the elongated crystal faults. They were prone to crushing at the center faces and the edges chipped easily. When the concentration of the HNS was high enough (1% to 2%) to have caused a microcrystalline structure, the cracking pattern and handling characteristic changed to that normally exhibited by Comp B. This horizontal cracking across the face of the billet was most severe for the 2% HNS level. Two out of three of the continuous TNT series at the 2% HNS level cracked into three pieces by the end of the year. The Comp B:HNS 1/2% and 1% billets were the only conditions that were crack-free. Photo 3 represents the groups with at least one surviving billet of the original three and for which cycling was continued; Photo 4, those which had no survivors for that parameter. Photo 5 is a comparison of the continuous TNT and Comp B series with 0%, 1/2%, 1% and 2% HNS, respectively. The billets shown in Photo 5 were the ones in the poorest condition of the original three.

d. 21-month cycle

All of the TNT billets with and without HNS were in poor physical condition by this time. Exudation had leveled off after one year. Late differences seen in the plots of Figure 9 were primarily a result of weight loss from chipping of the billets during handling. Comp B billets at the 1/2% level were free of surface cracks but density continued downward at a measurable rate. Data is presented in Figures 9 thru 12; Photo 6 records the appearance of the billets at this time.

4. Summary

After 21 months of thermal cycling at -54° to 71°C, an HNS concentration between 1% and 2% was the most beneficial, i.e., resulted in the least cracking from the largest density decrease; it was the least amount of HNS to give a microcrystalline pattern to the TNT. This level of HNS was necessary to prevent friability which was manifested by dusting of crystals, crushed surface centers, chipping of edges, cracking longitudinally along the elongated crystal axis. Unfortunately, the 2% level was prone to horizontal cracking of the type experienced with neat Comp B. Irreversible growth continued unabated in both the TNT and Comp B billets. The HNS did not cause an excessive increase in TNT exudation as occurred with the α NN.

C. Phase II

1. Introduction

This phase centered on the study of the effect of the optimum percentage of 1-1/2% HNS nucleating agent on TNT and TNT-based compositions. The HNS level selected gave the best trade-off of physicals for the 1-inch diameter TNT and Comp B billets after one year temperature cycling. Billet size was increased. The diameter was doubled in a right cylinder configuration to increase the dimensional stress on the system. The cyclotol, Comp B, and its aluminized version, H-6, were selected as representing conventional TNT compositions. Also included were TNT billets which were partially confined by the right cylinder aluminum molds in which they were cast. Only the top of the molds was open. Additive-free compositions were included as controls for each set of conditions. For comparison, a set was added containing 7% RDX as the nucleating agent. The higher solubility of RDX in TNT as compared to HNS required this percentage to ensure fine RDX crystallites would be available when the TNT reached its true set point.

2. Experimental

The HNS was from the same lot that had been used in Phase I. The percentage of HNS was calculated on the basis of the weight of TNT in the composition. The TNT was restricted to that from the continuous process of manufacturing. The Comp B and H-6 were prepared with TNT from the same 50-pound box to eliminate any variables that would be introduced by TNT with different impurities. The Comp B and H-6 were prepared from Class A RDX which would be classified as a medium grist. Class A is the type most likely to be present in production grade Comp B. Class E RDX, which has a particle size of less than 44 microns, was used for the TNT:RDX 7%

condition. The batch materials are described in Table I. Their stability and that of the explosive batches as measured by IS, DTA, and VTS are tabulated in Tables III and IV.

Explosive preparation, including batching, casting, cooling and surface finishing, were the same as for Phase I except that batch sizes were 1.5 kilograms. Batch cast densities are given in Table III. The billets which were to be partially confined were removed temporarily from their molds for surface refinishing. Two billets, 2.0 inches in diameter by 2.7 inches long, were poured from each batch for each condition. Temperature cycling was the same as for Phase I. The billets were weighed, measured, and photographed prior to cycling. The billets in the molds were measured from the top of the mold to the surface of the billet.

3. Results

a. Before cycling

The effect of HNS on VTS, melting point, and ignition temperature was negligible. The small increase in sensitivity to impact where HNS was present would be expected, considering the value for neat HNS. TNT changed from a highly macrocrystalline pattern to a microcrystalline structure in the presence of 1-1/2% HNS, while the 7% RDX billet was intermediate between the two types. The Comp B reflected the same trend as the TNT billets but to a lesser degree, as the large percentage of RDX present reduced the size of the TNT crystals. The H-6 billets with and without HNS were similar to the TNT billets with 1-1/2% of HNS. Photos 7, 8 and 9 illustrate these changes.

b. 28-day cycle

The exudation paralleled that for the Phase I smaller billets. The major portion occurred within the first week and the HNS caused a small increase. At the end of 4 weeks the percentages were 1.2 for neat TNT and 1.5 for TNT with HNS. The presence of the 7% fine, Class E RDX in the TNT reduced the exudation by one-half. The exudation figures for H-6 were in the same range as TNT but this was partially due to the Comp D-2 in the formulation. The Comp B value was 75% lower than that for neat TNT, reflecting its lower TNT content. The liquid fraction from the confined samples was retained to some degree at the top surface; when HNS was present, considerable darkening of the liquid fraction occurred. Weight loss data is plotted in Figure 13.

Irreversible growth took place in the billets for all conditions. The difference between TNT and the cyclotols was

markedly larger than that between TNT and TNT:HNS. Increase in diameter was about double for H-6 and Comp B as compared to TNT, 2% versus 1%; whereas, TNT:HNS was one-third less than the TNT. The most noteworthy difference was the reduction of the growth of the H-6 to that of TNT when 1-1/2% HNS was present. The same trend was found for longitudinal billet growth. The partially confined TNT and TNT:HNS height increase reversed the preceding; neat TNT increased by 1/2% and the TNT:HNS by 2%. The opening up of the structure of all the billets is reflected in the decreasing density values. See Figures 14, 15 and 16.

At the end of the 4 weeks, the neat TNT and the TNT:RDX 7% had begun to crack; the Comp B series had a few hairline cracks around the sides for the neat billets and cracks across the top and bottom for 1-1/2% HNS. All of the billets of H-6, TNT:HNS, and the partially confined TNT with and without HNS were crack-free. Photos 10 and 11, taken at the end of 3 months cycling, give an indication of the appearance of the billets (except for the development of the fine crack pattern for TNT:HNS and the worsening of the TNT:RDX 7% fault in the intervening 2 months).

c. 6-month cycle

Exudation continued at a slow rate for all conditions with that for Comp B being the lowest as it had less TNT and/or wax than the others. See Figure 13.

The percentage of longitudinal growth for TNT was approximately 1% less than that in the transverse direction. For Comp B and H-6 it was about the same. This held true whether the 1-1/2% HNS was present or not. Irreversible growth continued for all compositions, ranging from 2% to 4% over the initial values. Density values decreased markedly. The partially confined billets of TNT:HNS were growing in the unconfined direction at approximately double the rate of their TNT counterparts. The dimensional and density data is plotted in Figures 14, 15 and 16, with Figure 15 containing values for the partial confinement parameter.

All of the unconfined TNT and Comp B billets with and without 1-1/2% HNS were in poor condition by the end of this time period. The cracking pattern of the Comp B and the TNT:HNS were similar, as can be seen in Photos 12 and 13. The Comp B:HNS billet was placed in an inverted position to enable it to be photographed with the others. The TNT:HNS billet selected for photographing was still in one piece but the other billet of the same composition was in three pieces. The H-6 billets were the only ones which gave no outward indication of cracking though density continued downward.

The TNT and TNT:HNS partially confined billets did not exhibit cracking across the exposed surface. The ones with HNS developed an unevenness across this surface which is not readily detectable in Photo 14. This was thought to be related to the vertical growth taking place in this direction.

4. Summary

The same pattern obtained in Phase I, with respect to exudation, friability, growth, and orientation of cracks, was realized with the large right cylinder billets of TNT and its cyclotol versions. HNS was present at 1-1/2% based on the weight of TNT in the formulation.

The cracking resulting from the irreversible growth occurred sooner, i.e., the 2-inch diameter billets were in the same state of physical deterioration by the end of 6 months as the 1-inch diameter billets at the end of one year.

The H-6 and the partially confined TNT billets did not have an outward appearance of deterioration but the percentage longitudinal growth and significant decrease in density testified that the overall quality was poor.

D. Addendum

In the course of the evaluation of the two-step Bofors process, it was learned that the British were obtaining a microcrystalline TNT by using 1/2% HNS as a TNT additive. The first step of HNS incorporation occurred at 110°C instead of 95° to 100°C used in this work. The 1-inch diameter TNT billets cycled for the present study did not exhibit this type of structure until the level of HNS was 1% or higher.

To evaluate this apparent solubility factor, TNT:HNS 1/2% melts were held for 3 hours at 110°C and for 3 hours at 100°C. Both conditions gave the preferred structure. When the time was reduced to 30 minutes at 100°C, the structure was still fine grained; at 15 minutes, the TNT once again gave the elongated macrocrystalline structure associated with military grade TNT solidification. A correlation was found between the density and the microcrystalline structure, the higher density being associated with the microcrystalline billets. Photo 15 presents these time-temperature effects. Density values are given in Table V.

Representative billets were subjected to the JAN temperature cycle for 28 days. These were the 110°C melts with and without HNS held 3 hours, and the TNT:HNS 1/2% melts held at 100°C for 3 hours and for 0.1 hour, respectively.

By the end of one week, the TNT:HNS 1/2% billets that had the fine grain pattern (heated 3 hours) gave double the amount of exudation (2% versus 1%) when compared to the neat TNT controls. In the macrocrystalline TNT:HNS 1/2% (heated 0.1 hour), exudation was at an intermediate level. Density values of the microcrystalline billets decreased at a more rapid rate than the TNT and TNT:HNS 1/2% with the macro structure.

At the end of 28 days, exudation had leveled off, the irreversible dimensional growth in both directions was continuing as were the diminishing density values. Exudation, growth, and density curves of the billets before and after the 4 weeks of cycling are given in Figures 17 thru 20.

III. CONCLUSIONS

The benefits to be derived from the addition of a small percentage of HNS to TNT and related cast explosive compositions, such as Comp B and H-6, are not extensive enough to warrant its use on a routine production scale where time and simplicity of operation are major considerations. The immediate improvement in handling qualities is not significantly reflected in an improvement in irreversible growth. Where specialty items, i.e., TNT charges with the reduced friability of Comp B, are required, HNS at the 1-1/2% level will be useful.

A concentration of 1/2% to 2% HNS was required to produce a microcrystalline structure in TNT with the resultant major decrease in friability. The 1/2% level would give the preferred microcrystalline pattern only if incorporated for a minimum of 30 minutes at elevated temperatures. The 2% level accelerated crack development across the top of the billets on long-term temperature cycling.

TNT samples with small percentages of HNS (1/2% to 2%) exhibited irreversible growth when cycled with a decrease in density which eventually led to complete crumbling. The 1% HNS level gave a small measure of improvement in the life of the TNT billets.

The contrast in the growth pattern of batch versus continuous TNT series was not as great as that between Comp B and recrystallized TNT. The magnitude of growth was greatest for Comp B, negligible for the purified TNT.

Exudation ceased to be a major factor after the 28-day period was reached. The major portion of exudation occurred in the first week; loss was related to the amount of TNT and the impurities present.

Comparison samples with 1% of the anticracking agent, α NN, performed poorly from the beginning and had to be removed after 9 months cycling because of exudation and crumbling.

Larger test billets deteriorated faster than smaller ones. Loss of physical quality occurred at double the rate for TNT, Comp B, and H-6 formulated with 1-1/2% HNS based on TNT content when the diameter of the test billets was increased from 1 inch to 2 inches.

The H-6 and the partially confined TNT and TNT:HNS billets, i.e., top end of the mold open, did not exhibit surface cracking after 9 months but overall physical quality, as measured by longitudinal growth and decrease in density, was poor.

The Bofors method of incorporating HNS requires a two-stage processing technique, i.e., solidification of TNT:HNS melt with subsequent remelting before casting billets. This increases processing time by a considerable factor. Another disadvantage is the narrow temperature range, 83° to 86°C, on remelt, which is required to maintain the HNS in its optimum form to provide nucleating centers for the TNT.

TABLE I. BATCH MATERIALS

Material	Specification	Manufacturer	Notes
<u>PHASE I:</u>			
TNT	Contract W-Ord 482	Weldon Springs	Lot WLD 1102
TNT	JAN-T-248 Grade I, Flake	Volunteer	Lot VOL-1-1786 dtd Jun 1955 Batch Process
TNT	MIL-T-248A Type I, Flake	Volunteer	Lot VOL 76F014-230 Continuous Process
HNS IIB	WS 5003E	Chemtronics	Lot 66-12
Comp B	MIL-C-401E Type I, Grade A	Holston	Lot HOL 051-4 Batch 419834 dtd 2-1975
Alpha Nitro- naphthalene	MIL-A-23894	E. I. DuPont	Lot 7 dtd 10-20-64
<u>PHASE II:</u>			
TNT	MIL-T-248A Type I, Flake	Volunteer	Lot VOL 76F014-232 Continuous Process
HNS IIB	WS 5003E	Chemtronics	Lot 66-12
RDX	MIL-R-398C Class A	Holston	Lot HOL 21-40
RDX	MIL-R-398C Class E	Holston	Lot HOL 445-6
Comp D-2	MIL-C-18164A	Hercules	Lot 7-HIK-03732
Wax, R68097	MIL-W-20553B Grade A	Witco	Microcrystalline
Aluminum	MIL-A-512A Atomized, Type III Grade F, Class 7	Alcan Metal	Lot D-43
Calcium Chloride	MIL-C-13573A	Octagon Process, Inc.	Lot-1-OPI-0268 Anhydrous

TABLE II. STABILITY:TNT/HNS

Composition (%)		Impact sensitivity ^a (cm)	VTS ^b (ml/g)
TNT	HNS		
100	0	85	0.1
99	1	87	0
98	2	85	0
95	5	79	0
90	10	68	0.1
80	20	55	0
70	30	49	0
50	50	43	0
0	100	42	0.1
Control: Comp A-3		51	

^a50% ht: Bruceton Method/NOL Machine/Type 12 tools/2.5 kg wt/25 trials/35 $\pm$ 2 mg/5/0 sandpaper.

^bVacuum thermal stability: ml/g/48 hrs/100°C/STP.

TABLE III. STABILITY:EXPLOSIVE BATCHES:PHASE II

Material	Density ^a (g/ml)	VTS ^b (ml/g)	Impact sensitivity ^c (cm)	DTAd		
				Endotherm (°C)		Ignition (°C)
				TNT	RDX	
TNT	1.59	0	86	79	-	252
TNT:1-1/2% HNS	1.58	0	80	79	-	251
TNT:7% RDX, Class E	1.59	0.1	61	77	-	219
Comp B ^e	1.68	0.1	38	78	171	205
Comp B ^e :1-1/2% HNS ^f	1.68	0.2	43	78	173	204
H-6 ^e	1.70	0.2	84	77	-	210
H-6 ^e :1-1/2% HNS ^f	1.69	0.3	77	78	-	212
TNT ^g	1.57	0	94	80	-	253
TNT ^g :1-1/2% HNS	1.59	0	78	80	-	252
Class E RDX	-	-	21	-	-	-
Class A RDX	-	-	20	-	-	-

^aWater weight method.^bVacuum thermal stability: ml/g/48 hrs/100°C/STP.^c50% ht: Bruceton Method/NOL Machine/Type 12 tools/2.5 kg wt/
25 trials/35 ± 2 mg/ 5/0 sandpaper.^dDuPont 990; rate of heating: 10°C/min; sample wt: 20 mg.^eClass A RDX.^f1-1/2% HNS on basis of TNT weight.^gMelt for partially confined billets.

TABLE IV. STABILITY:RAW MATERIALS:PHASE II

Material	VTS ^a (ml/g)	DTA ^b	
		Endotherm (°C)	Ignition (°C)
TNT	0	80	253
HNS IIB	0	318	320
RDX:Class A	0.3	184	213
RDX:Class E	0.1	185	208
Wax:R68097	0	70	-
Comp D-2	0.6	64	196
Aluminum	0.2	-	-

^aVacuum thermal stability: ml/g/48 hrs/
100°C/STP.

^bDuPont 990; rate of heating: 10°C/min;
sample wt: 20 mg, except HNS IIB: 10 mg.

TABLE V. DENSITY VERSUS TNT:HNS^a CRYSTALLINE STRUCTURE

Sample	Crystalline ^b structure	Explosive melt		Billet density (g/ml)	Avg billet density (g/ml)
		Time (hr)	Temp (°C)		
TNT	Macro	3.0	110	1.554 1.561 1.559 1.554	1.56
TNT:HNS 1/2%	Micro	3.0	110	1.610 1.615 1.611 1.607	1.61
TNT:HNS 1/2%	Micro	3.0	100	1.587 1.588 1.584 1.579	1.58
TNT:HNS 1/2%	Micro ^c	0.5	100	1.586 1.583 1.579 1.576	1.58
TNT:HNS 1/2%	Macro	0.25	100	1.565 1.560 1.566 1.554	1.56
TNT:HNS 1/2%	Macro	0.1	100	1.558 1.554 1.540 1.545	1.55

^aTNT:continuous, Military Grade; HNS IIB:Lot 66-12.^bMicro gave a light color; macro gave a dark color.^cRind effect: Light color outside, darker inside.

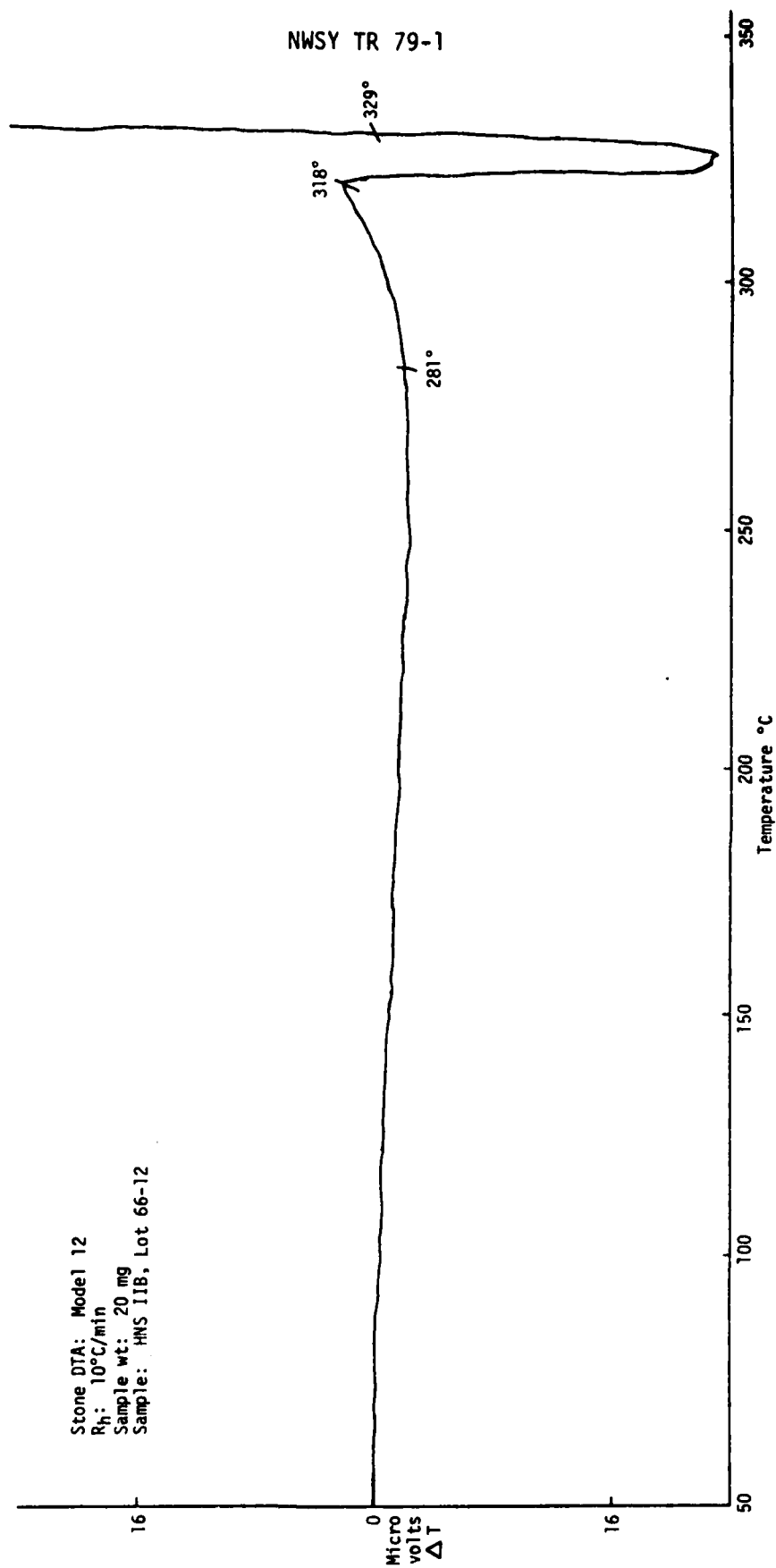


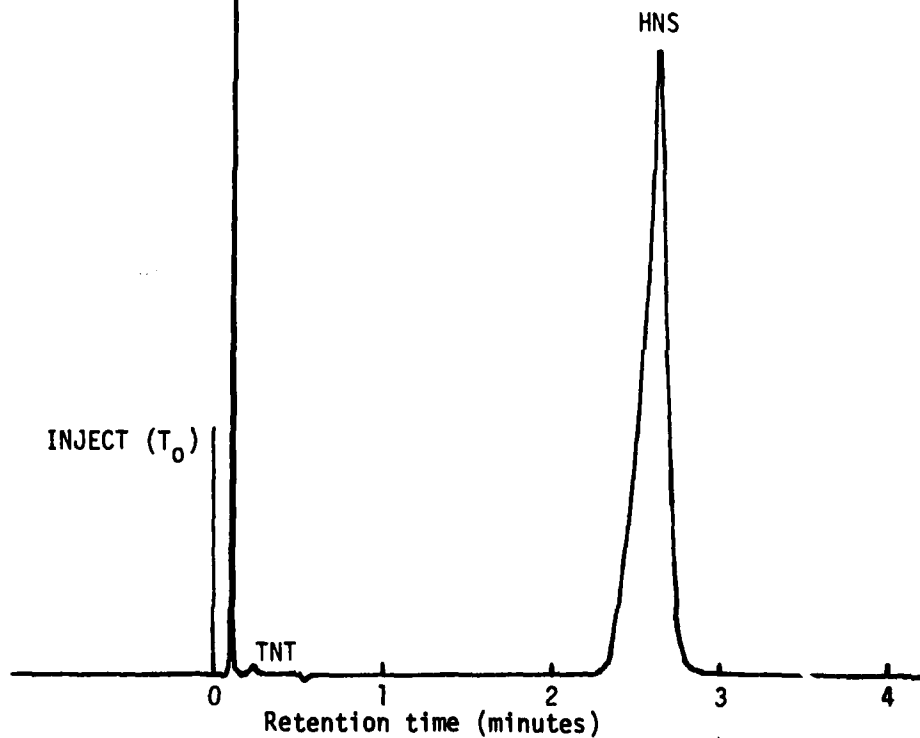
FIGURE 1. DIFFERENTIAL THERMAL ANALYSIS OF HNS IIB

DIOXANE

FIGURE 2. HPLC TRACE OF HNS IIB

CONDITIONS

Sample: HNS IIB, Lot 66-12, 50 ppm
Injected: 50 μ l
Column: μ Porasil: 4 mm ID x 30 cm
Pressure: ~1300 psig
Flow rate: 3.0 ml/min
Chart speed: 10 min/in.
Wavelength: 254 nm
Sensitivity: .05 abs
Solvent: 12.5/87.5 Dioxane/Heptane

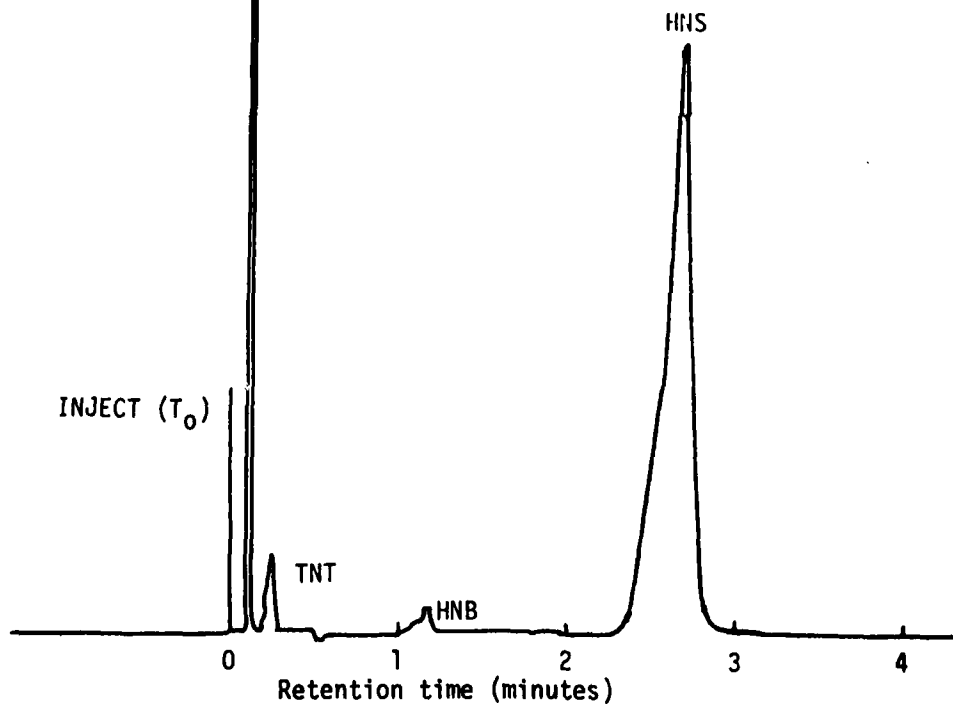


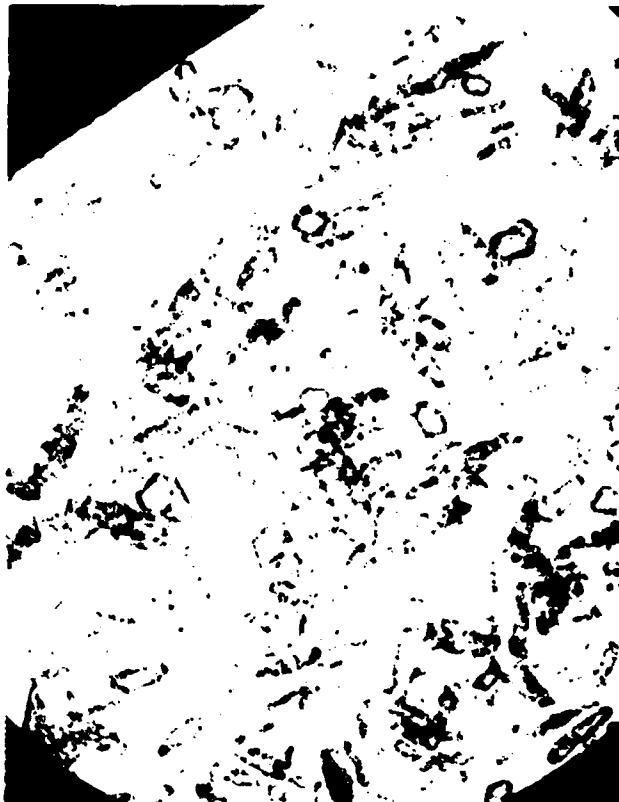
DIOXANE

FIGURE 3. HPLC TRACE OF HNS IIB (STANDARD)

CONDITIONS

Sample: HNS II, ID No. 2299, 48 ppm;
TNT, 1 ppm; HNB, 1 ppm
Injected: 50 μ l
Column: μ Porasil: 4 mm ID x 30 cm
Pressure: ~1300 psig
Flow rate: 3.0 ml/min
Chart speed: 10 min/in.
Wavelength: 254 nm
Sensitivity: .05 abs
Solvent: 12.5/87.5 Dioxane/Heptane





HNS IIB, LOT 66-12
(90 μ /division)

FIGURE 4. HNS IIB PHOTOMICROGRAPHY

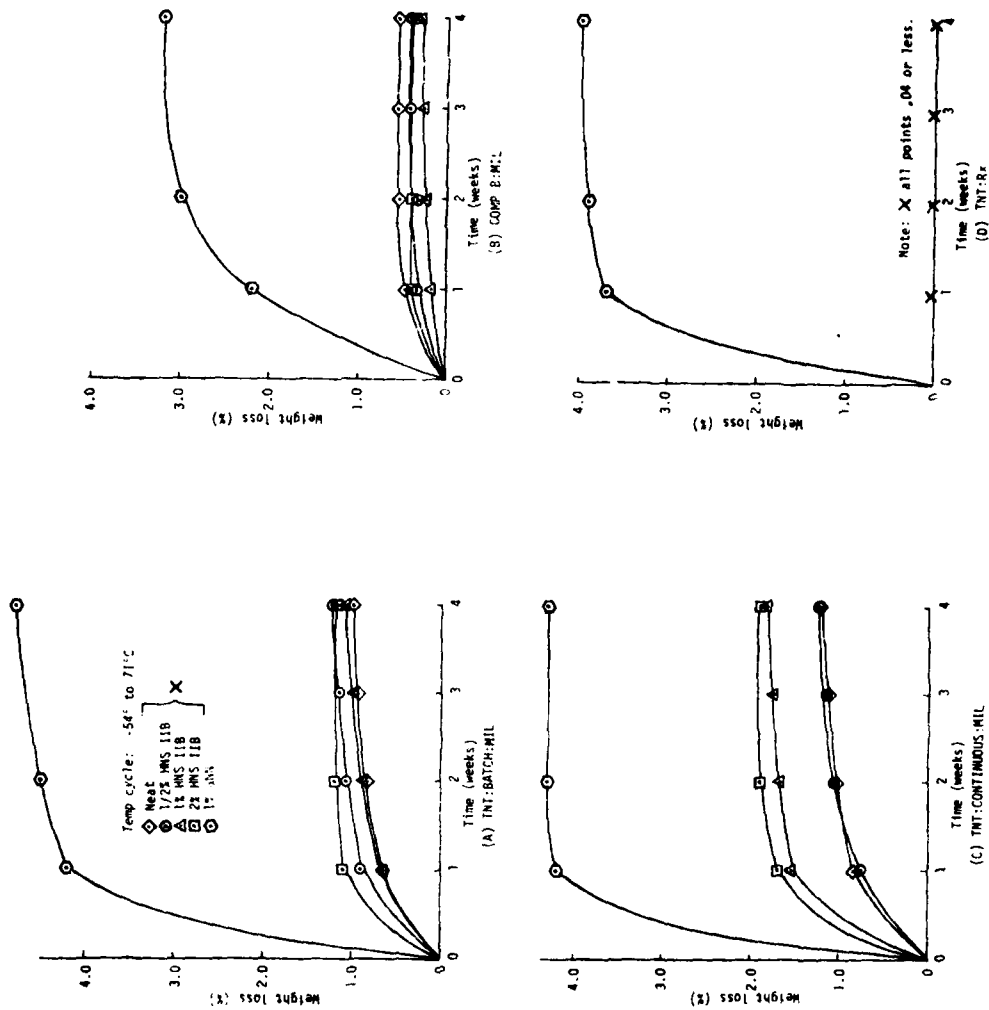


FIGURE 5. PHASE I EVALUATION (28 DAYS)

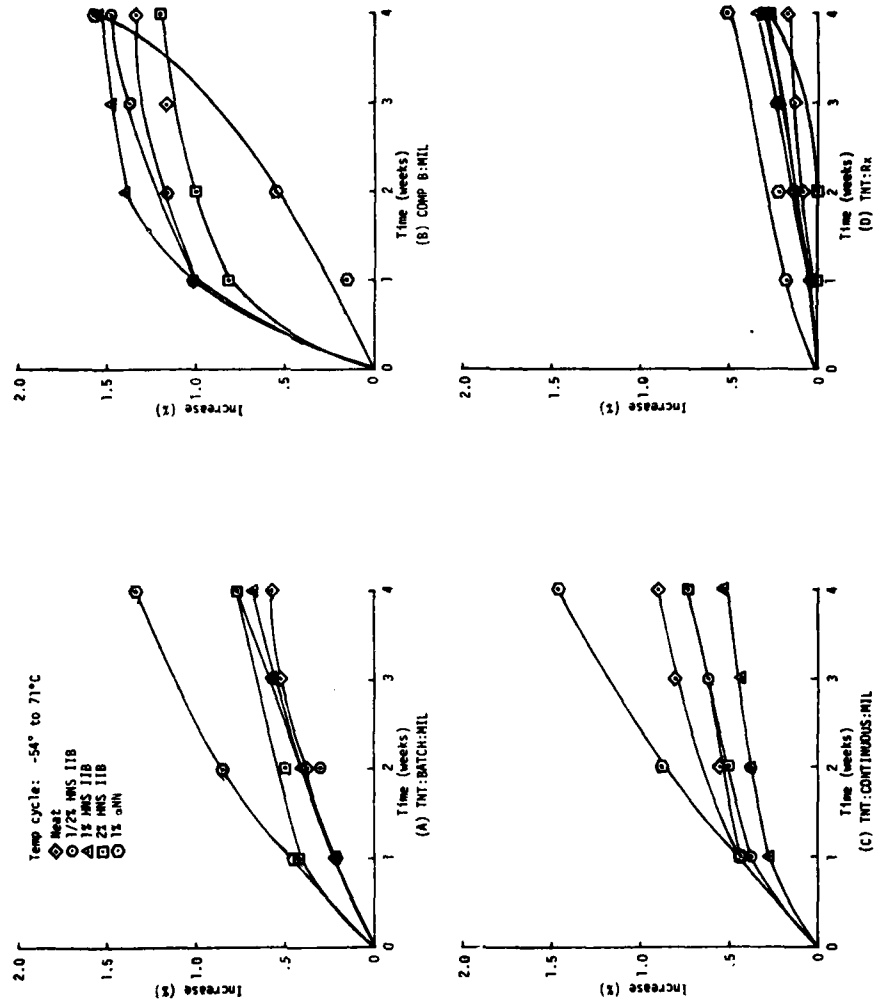


FIGURE 6. PHASE 1 GROWTH-DIAMETER (28 DAYS)

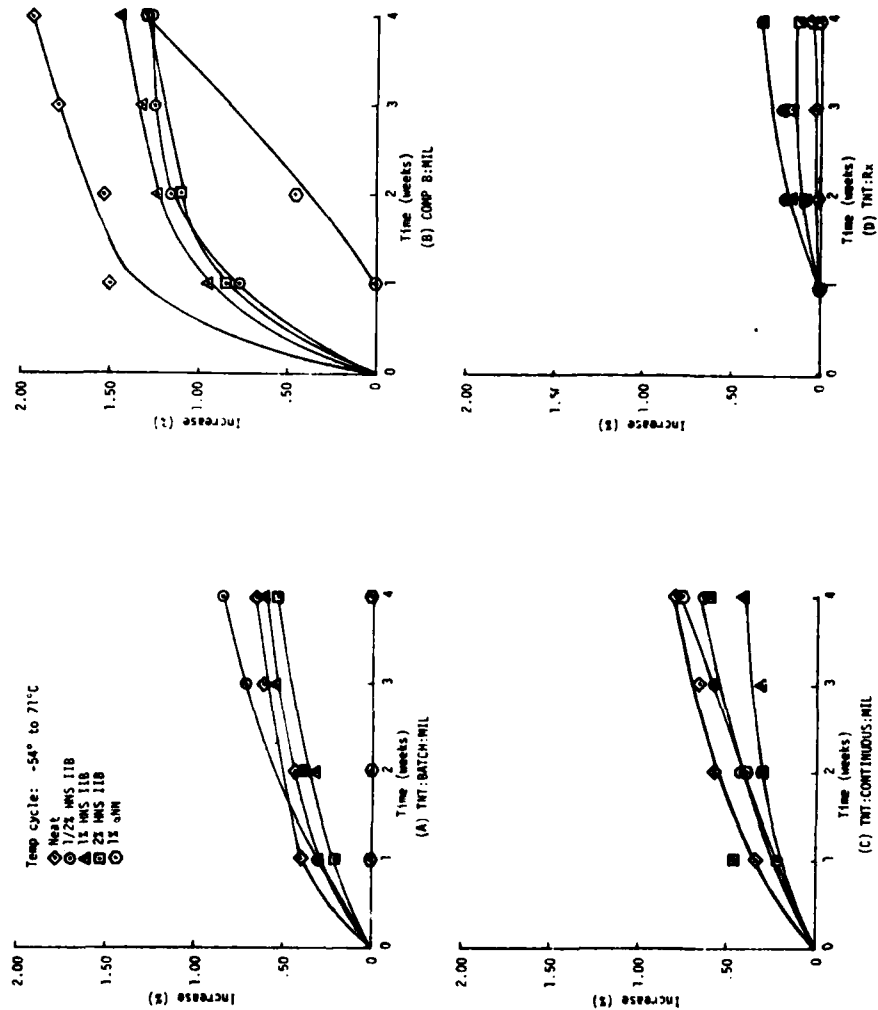


FIGURE 7. PHASE I GROWTH-LENGTH (ZB DAYS)

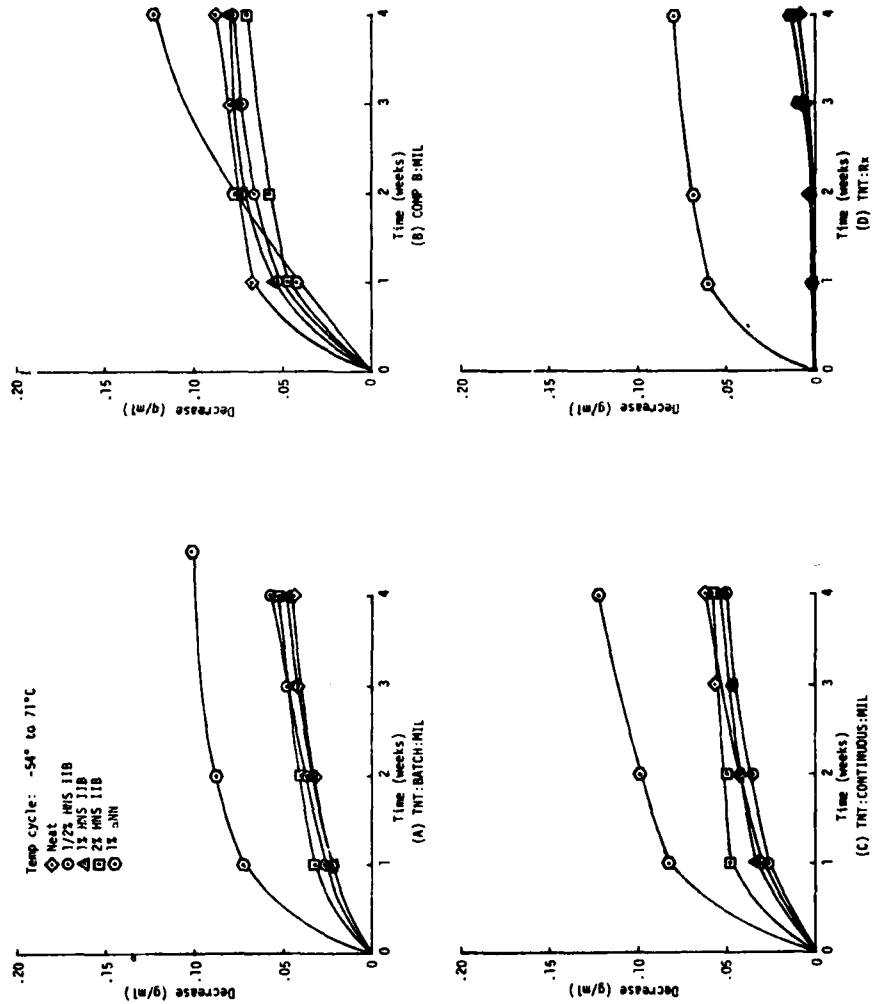


FIGURE 8. PHASE I DENSITY (28 DAYS)

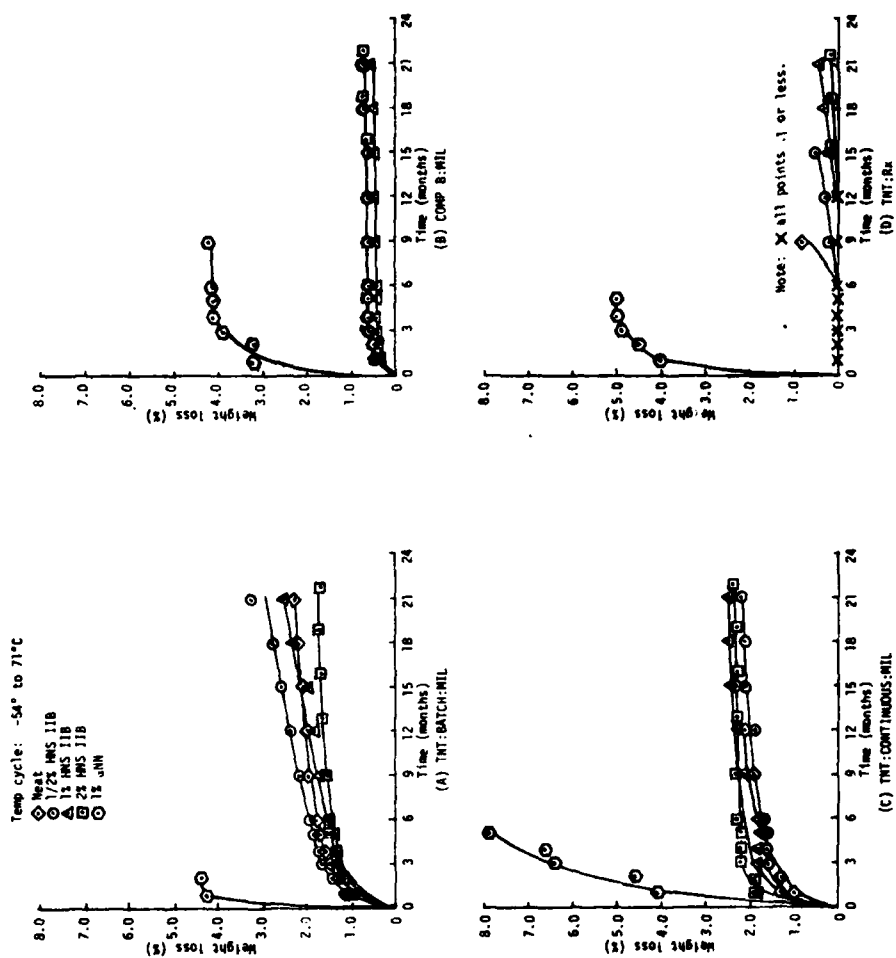


FIGURE 9. PHASE I EVALUATION (21 MONTHS)

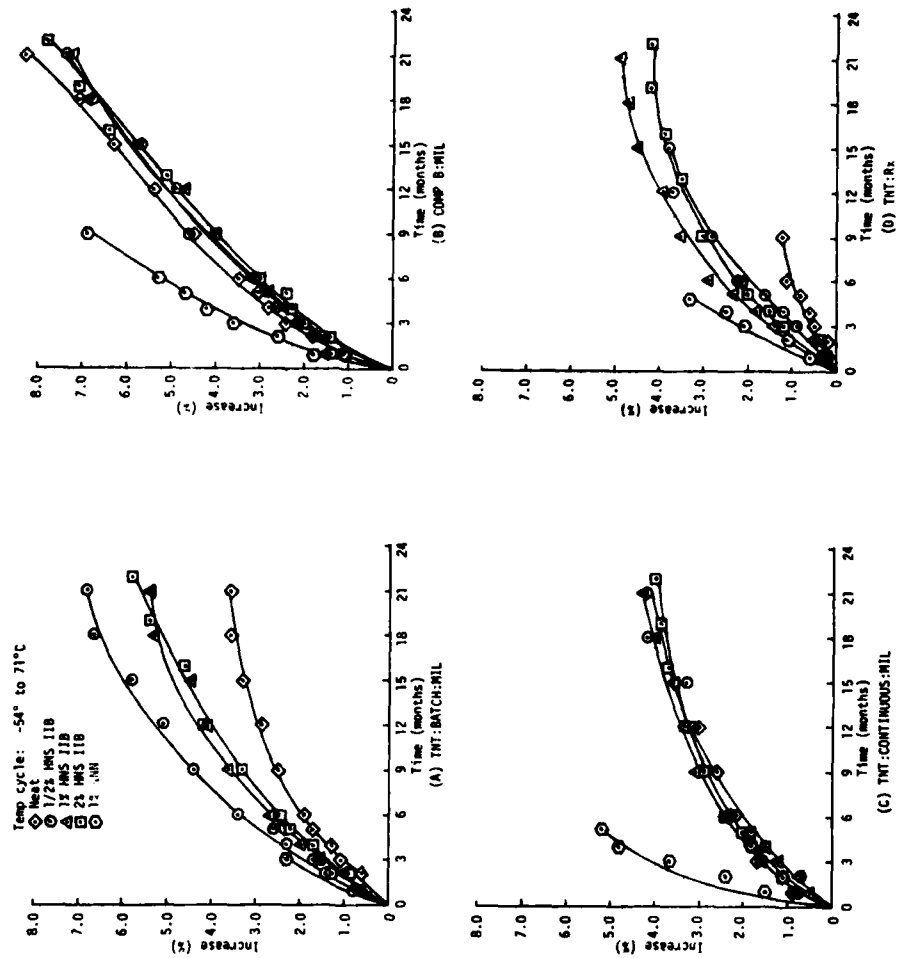


FIGURE 10. PHASE 1 GROWTH-DIAMETER (21 MONTHS)

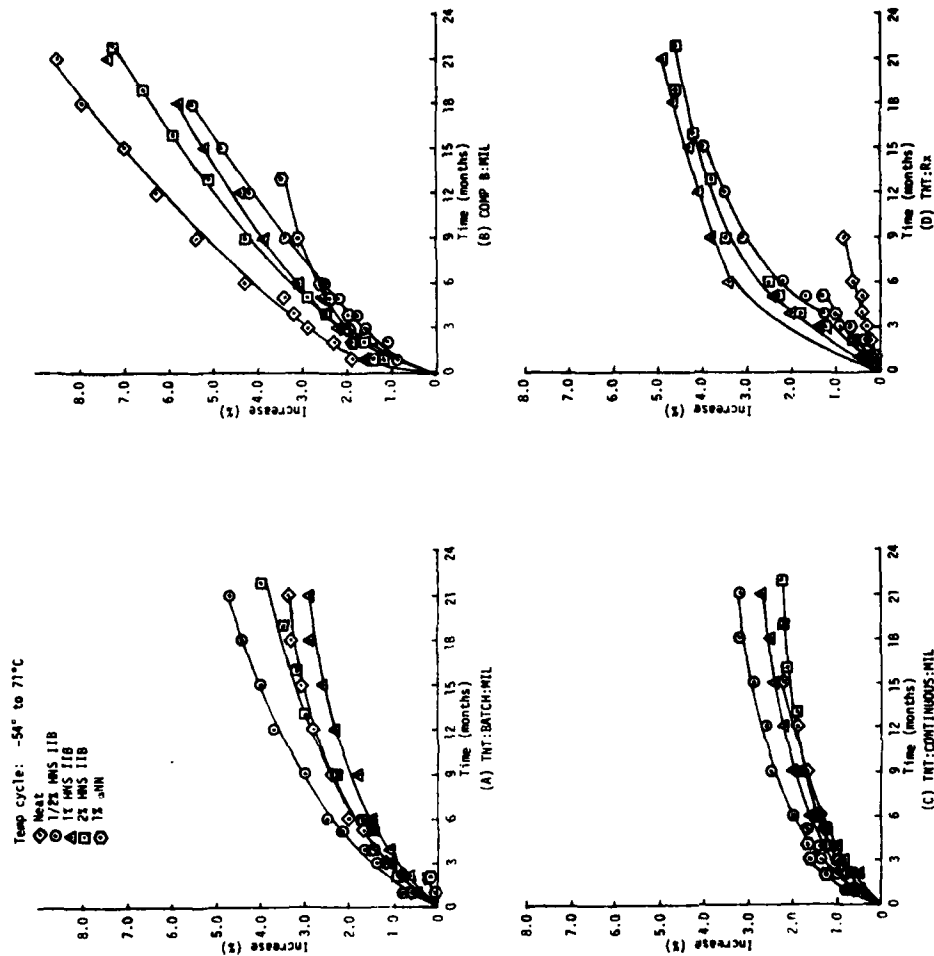


FIGURE 11. PHASE I GROWTH-LENGTH (21 MONTHS)

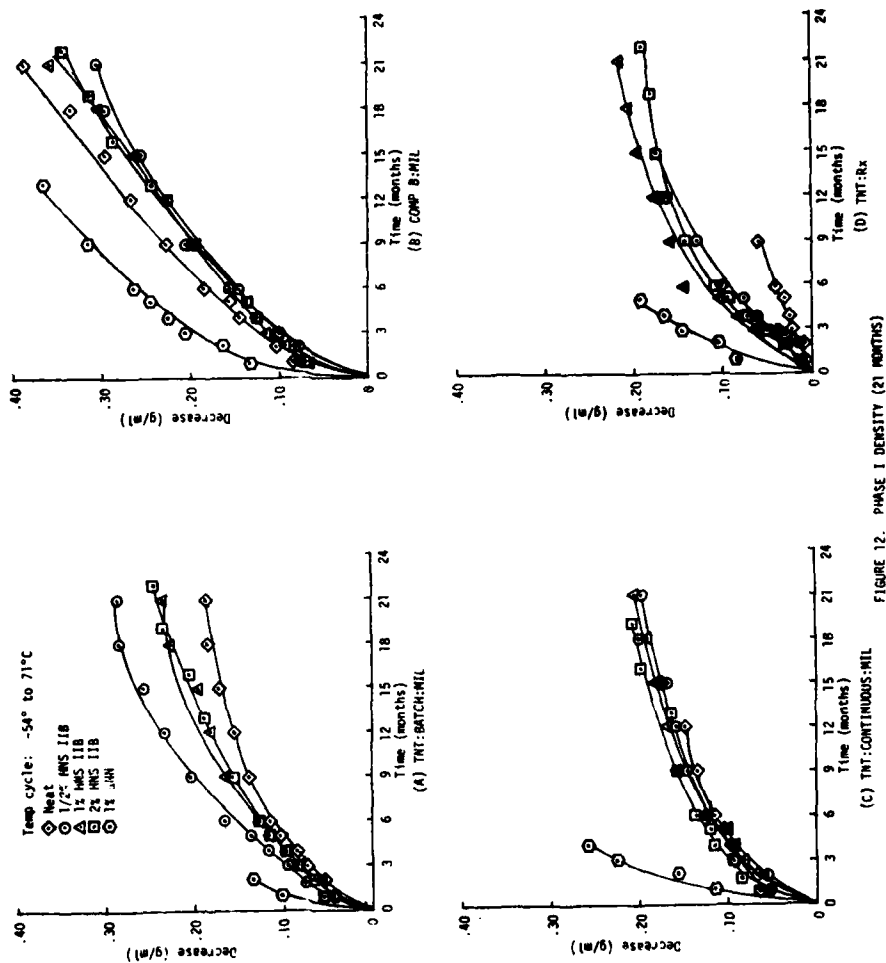


FIGURE 12. PHASE I DENSITY (21 MONTHS)

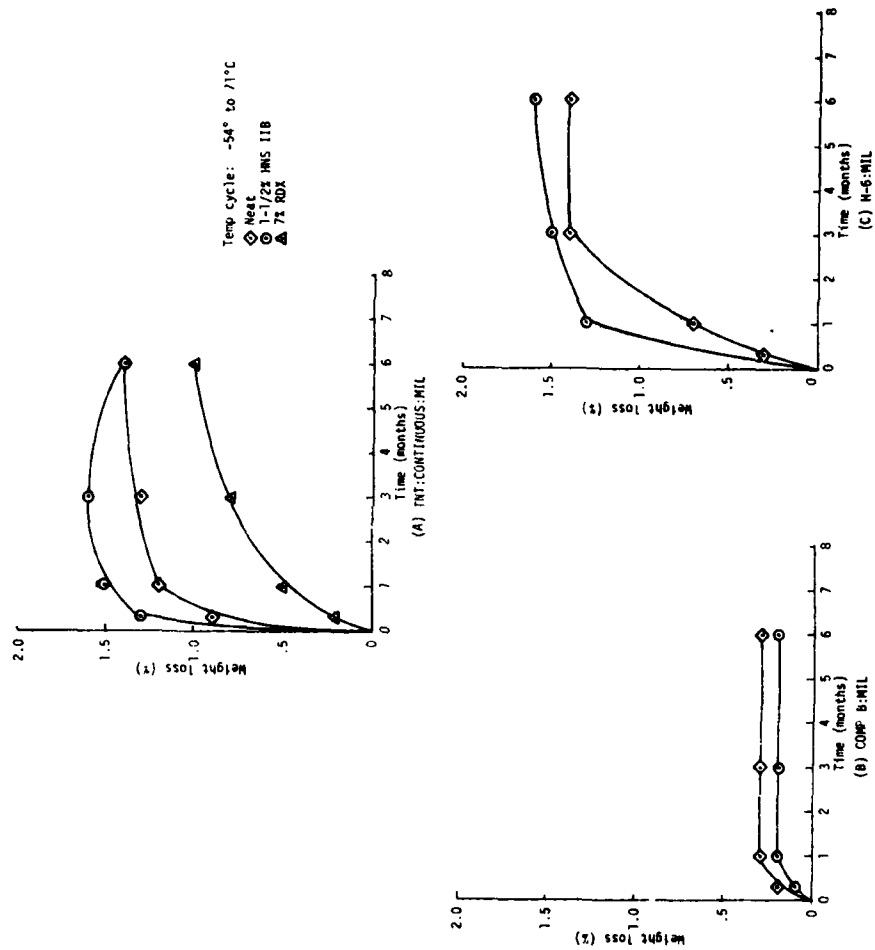


FIGURE 13. PHASE II EXUDATION

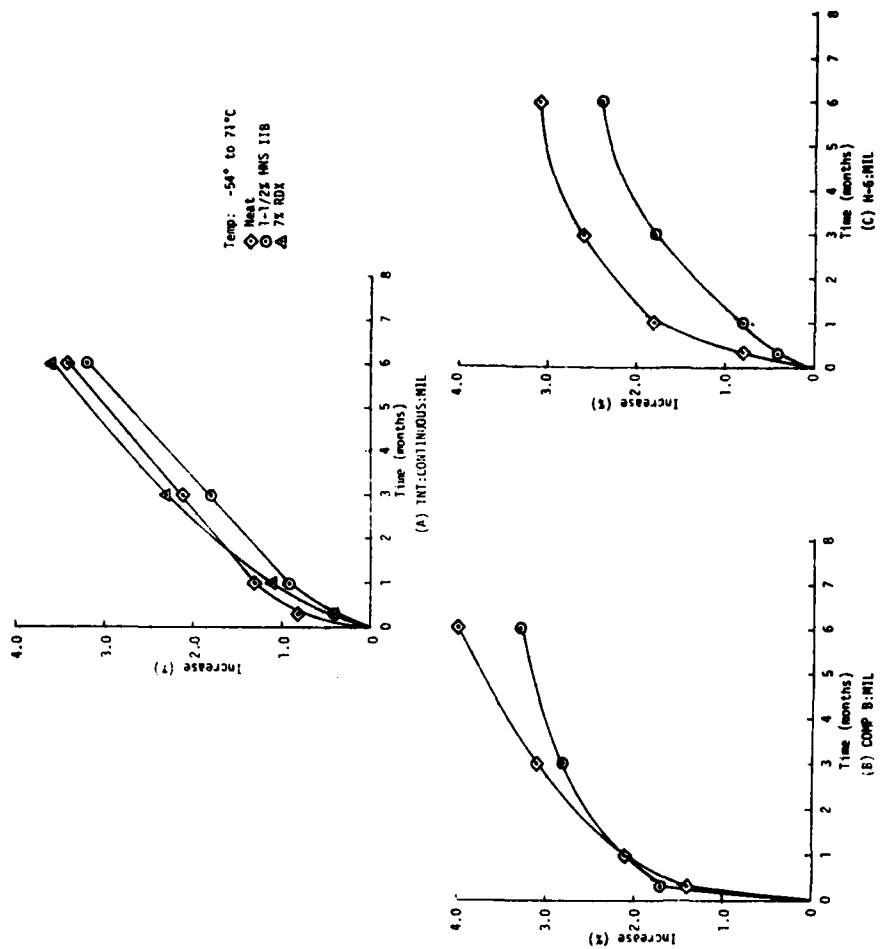


FIGURE 14. PHASE II GROWTH-DIAMETER

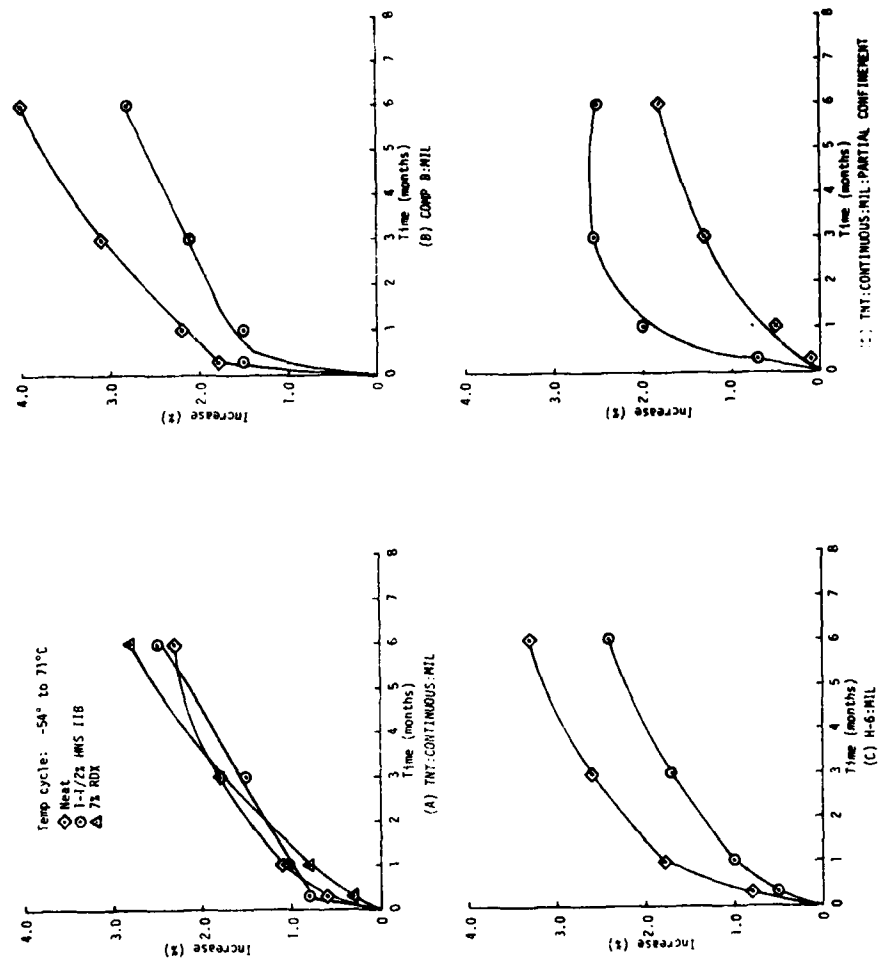


FIGURE 15. PHASE II GROWTH-LENGTH

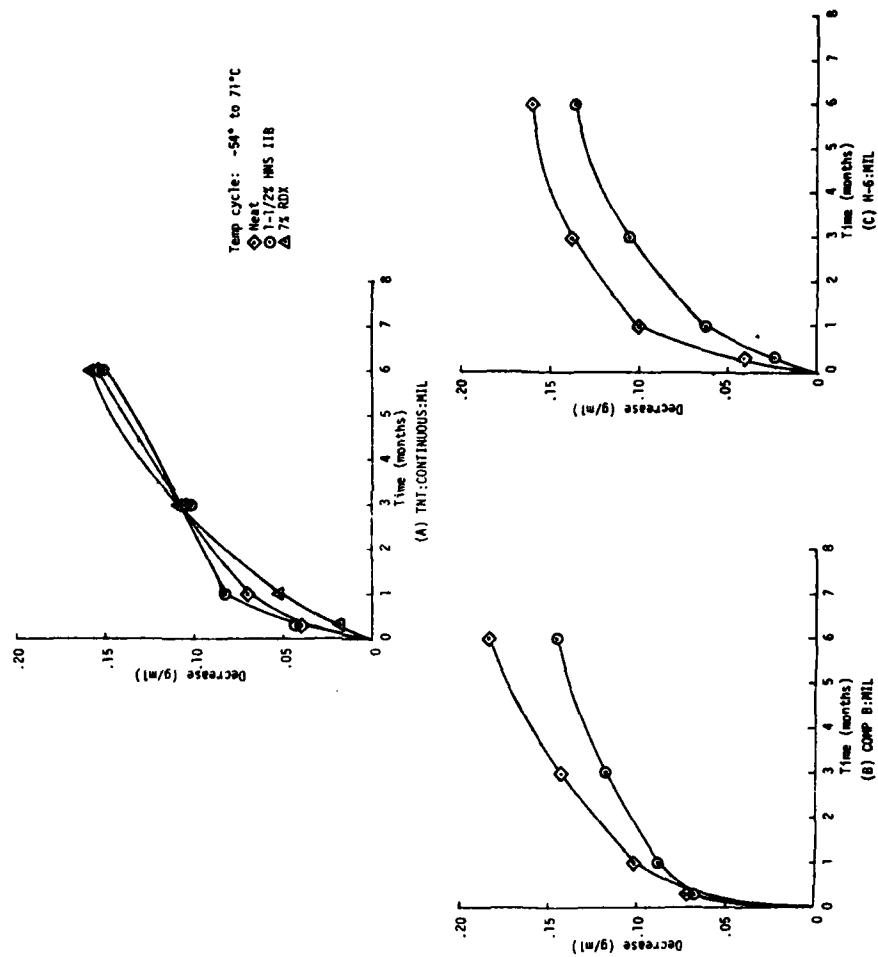


FIGURE 16. PHASE II DENSITY

ADDENDUM EXUDATION

TNT:CONTINUOUS:MIL
Temp cycle: -54° to 71°C

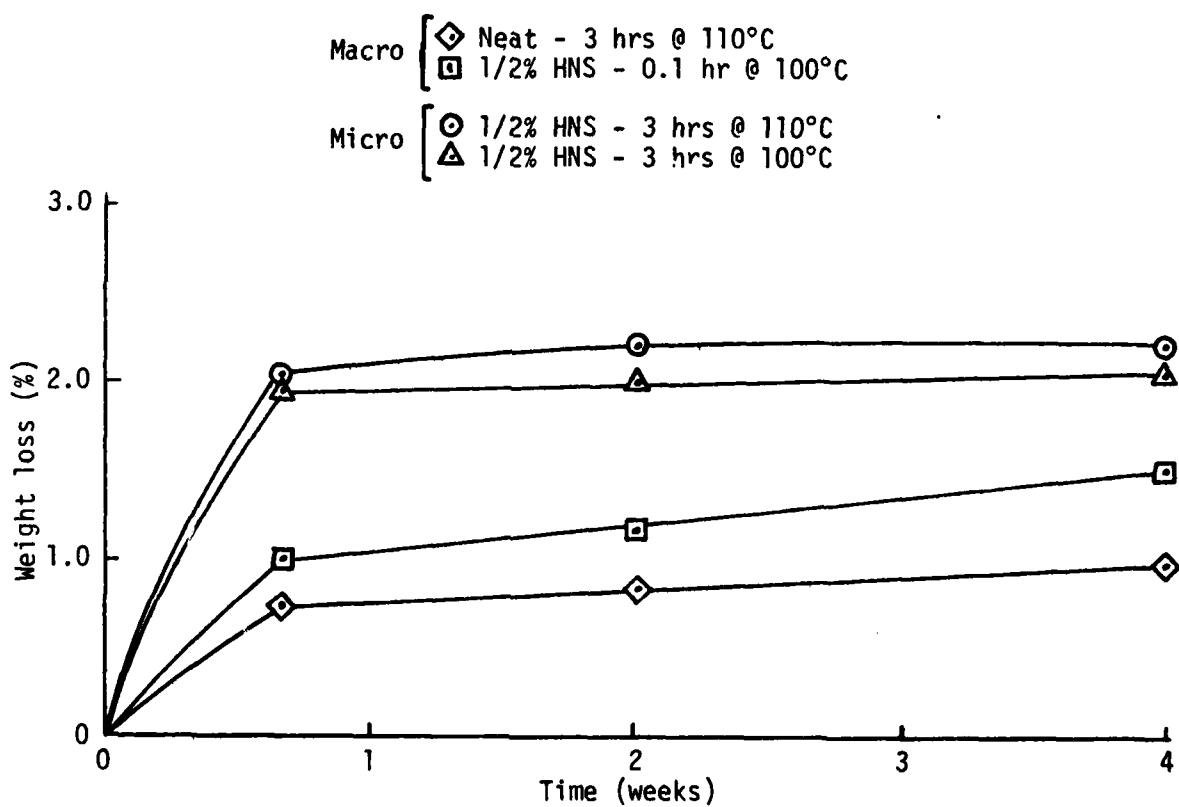


FIGURE 17

ADDENDUM GROWTH-DIAMETER

TNT:CONTINUOUS:MIL
Temp cycle: -54° to 71°C

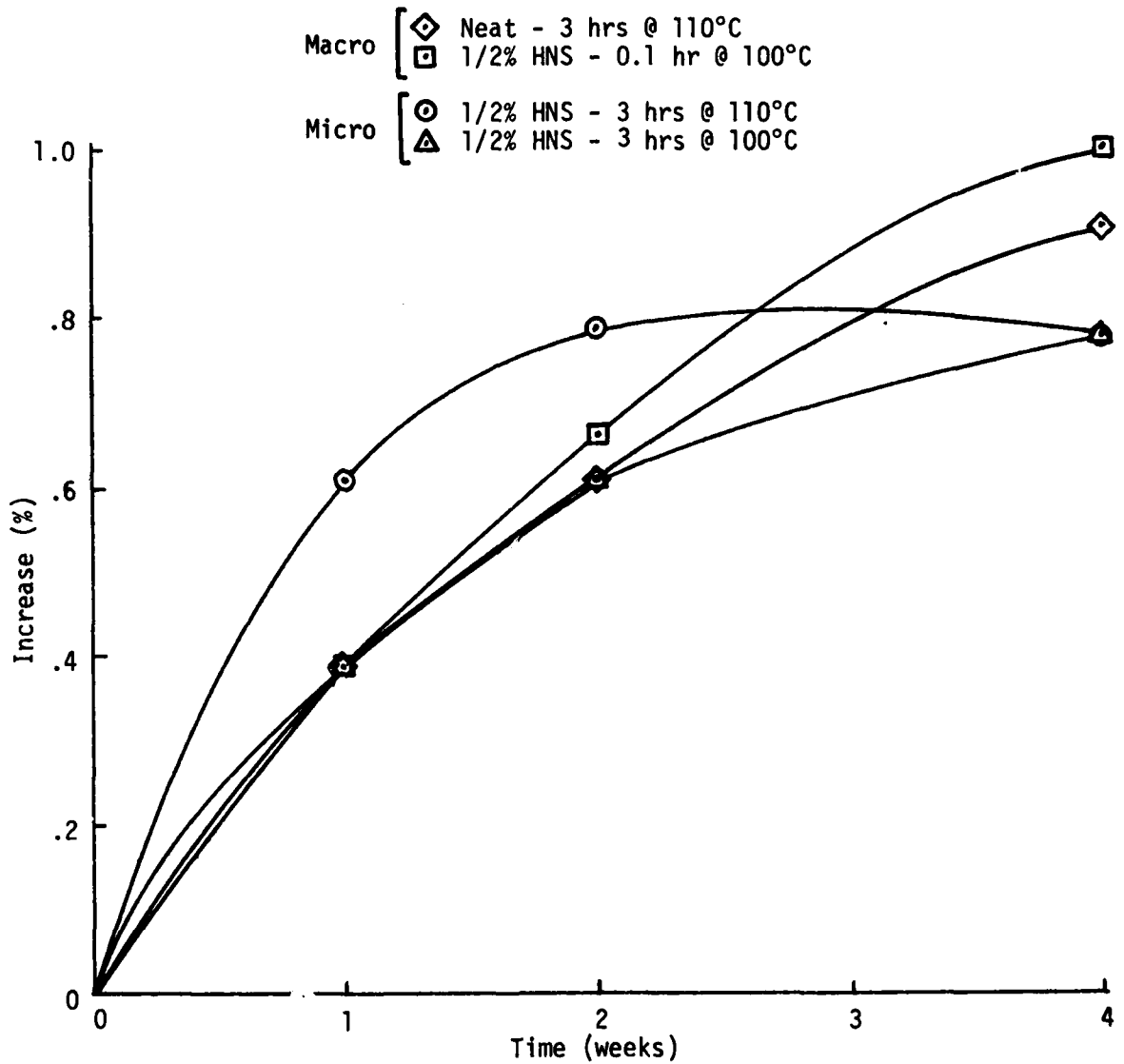


FIGURE 18

ADDENDUM GROWTH-LENGTH

TNT:CONTINUOUS:MIL
Temp cycle: -54° to 71°C

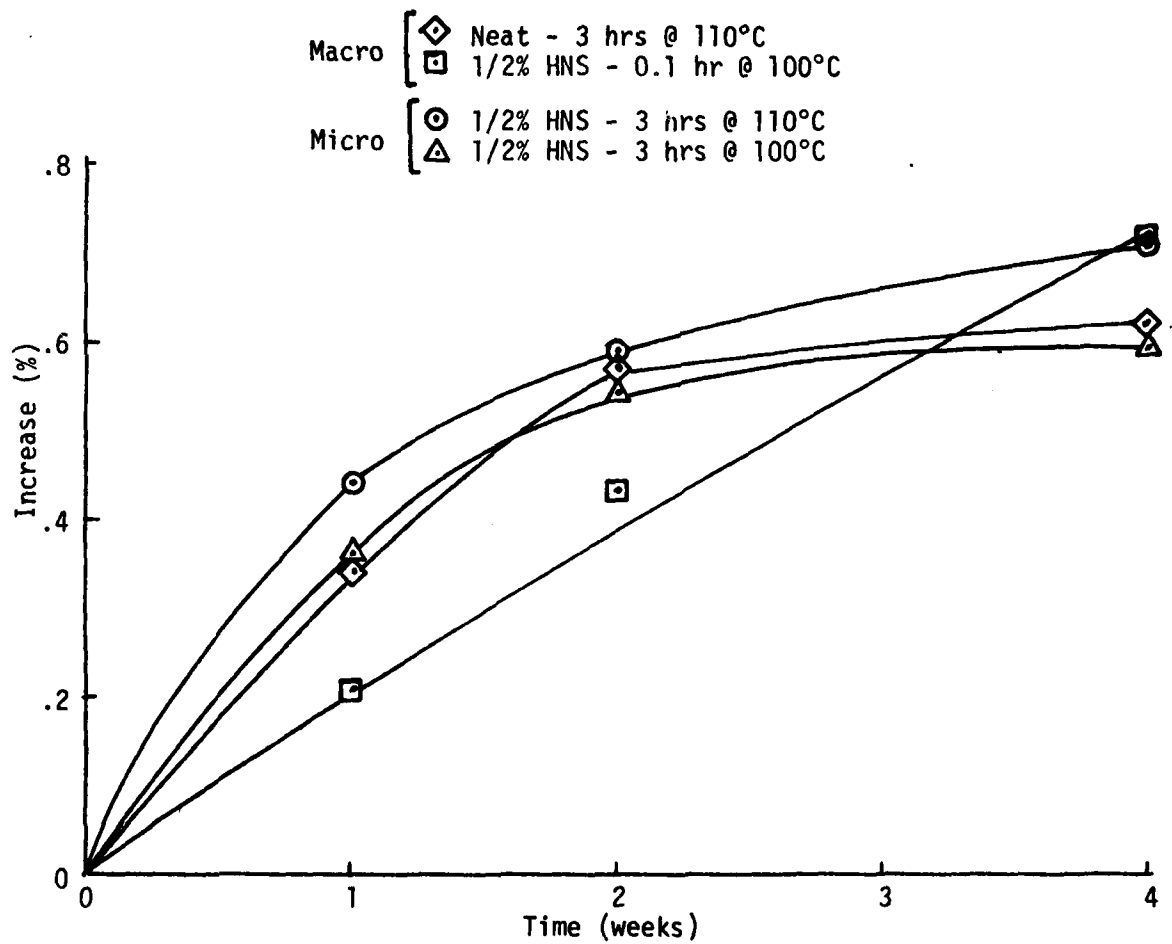


FIGURE 19

ADDENDUM DENSITY

TNT:CONTINUOUS:MIL
Temp cycle: -54° to 71°C

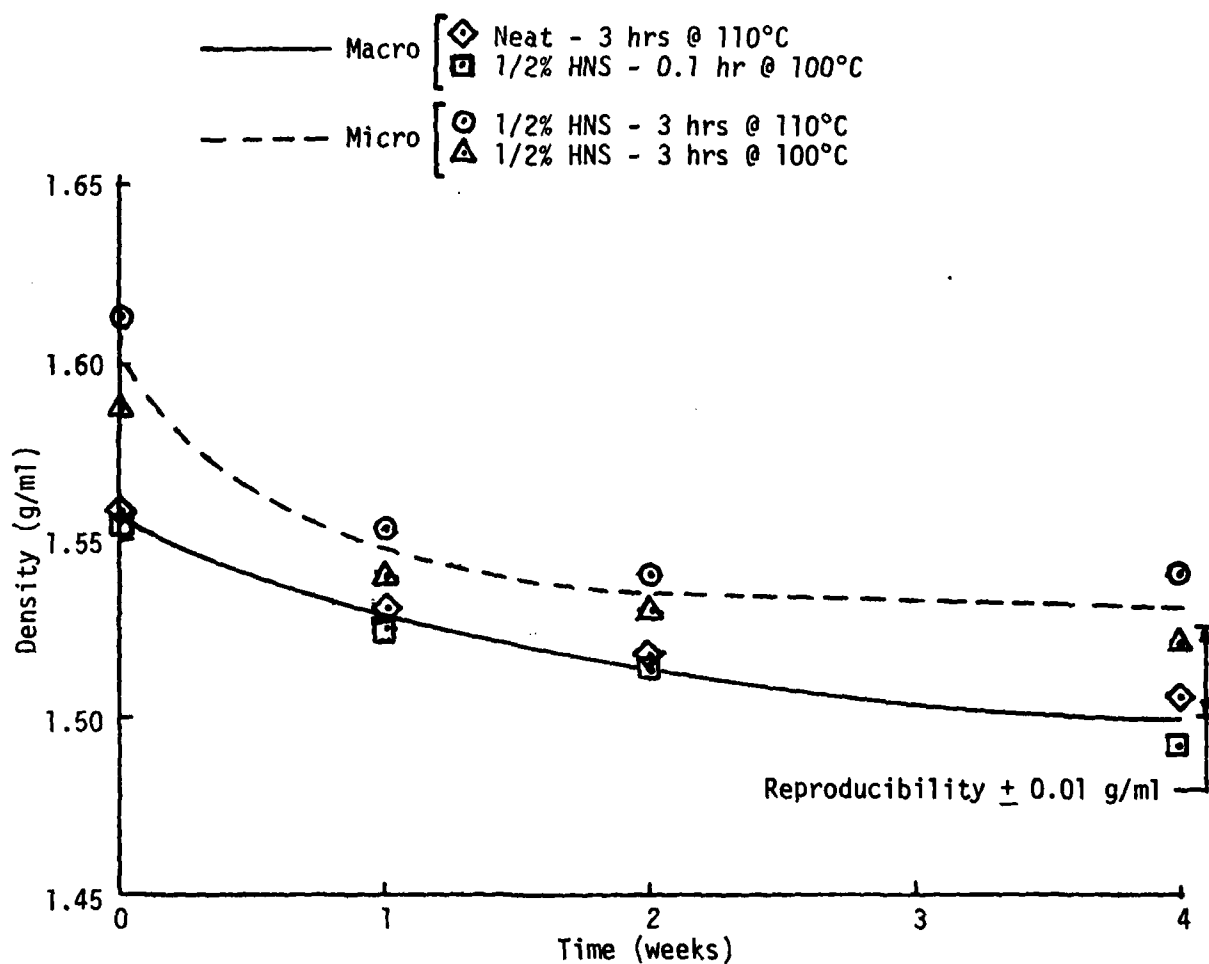
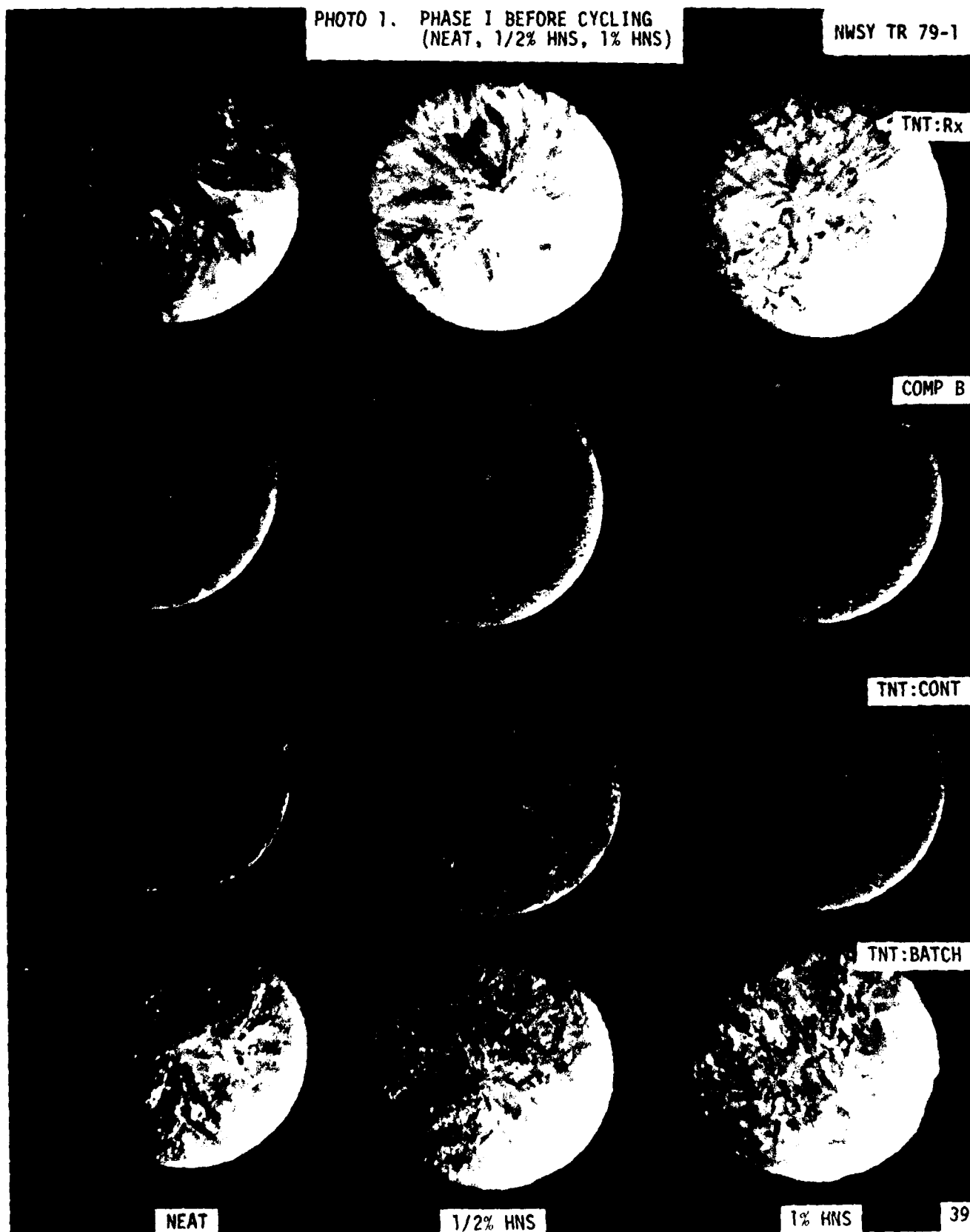


FIGURE 20

PHOTO 1. PHASE I BEFORE CYCLING
(NEAT, 1/2% HNS, 1% HNS)

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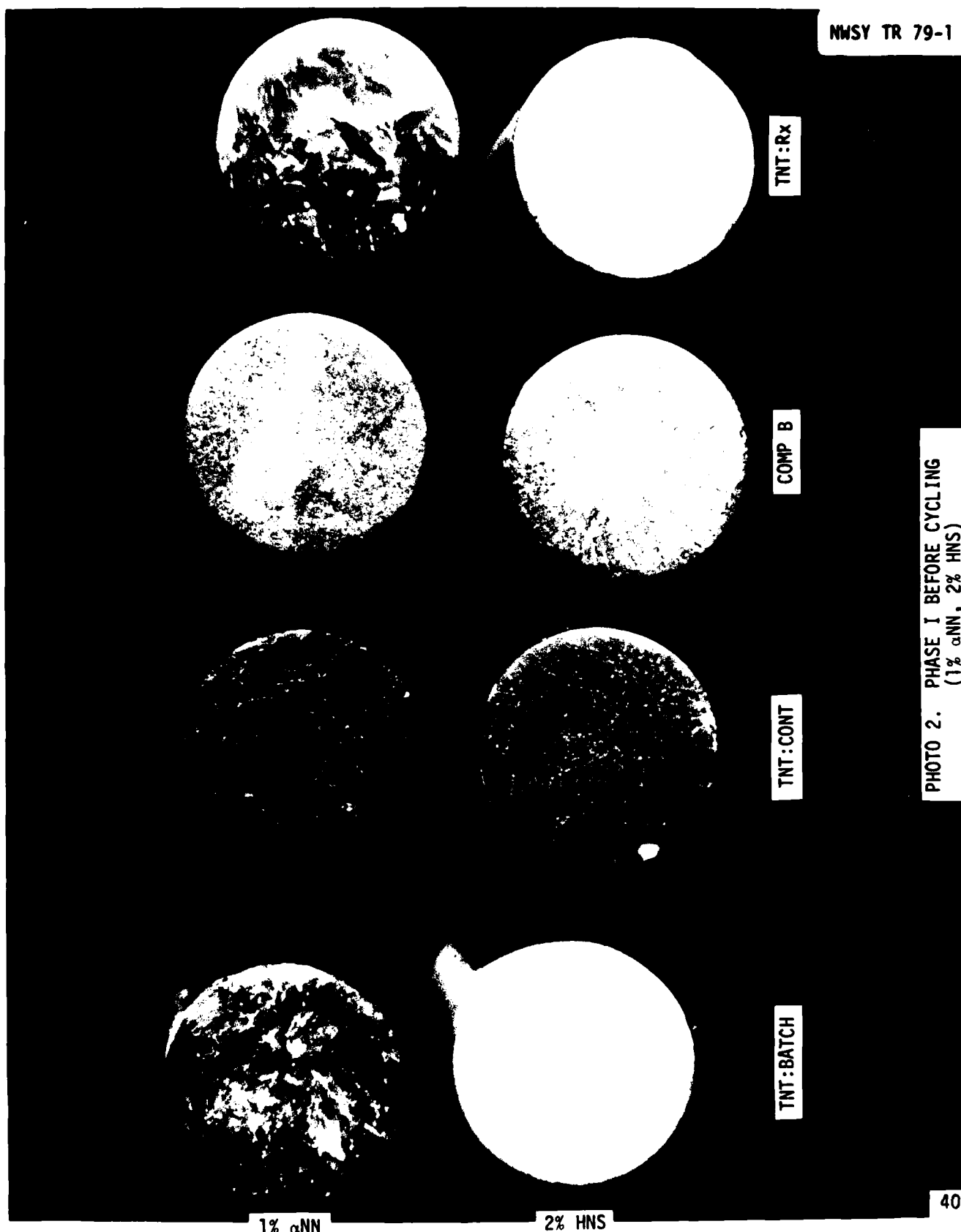


PHOTO 2. PHASE I BEFORE CYCLING
(1% aNN, 2% HNS)

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TNT:Rx

TNT:CONT

TNT:BATCH



1% dNN

2% HNS

1% HNS

1/2% HNS

NEAT

PHOTO 3. PHASE I 12-MO CYCLE
SURVIVING BILLETS

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TNT: BATCH

TNT: CONT

TNT: Rx

TNT: Rx

NEAT



PHOTO 4. PHASE I 12-MO CYCLE
WITHDRAWN BILLETS

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COMP B

TNT:CONT

2% HNS

1% HNS

1/2% HNS

NEAT

FISHER SCIENTIFIC

PHOTO 5. PHASE I 12-MO CYCLE
COMP B VS TNT:CONT

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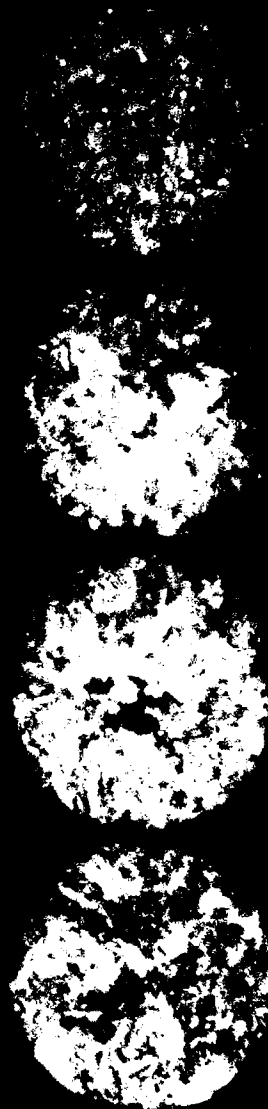
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TNT:Rx

COMP B

TNT:CONT

TNT:BATCH



2% HNS

1% HNS

1/2% HNS

NEAT

PHOTO 6. PHASE I 21-MO CYCLE
SURVIVING BILLETS

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H-6

COMP B

TNT:CONT



NEAT

1-1/2% HNS

7% RDX

PHOTO 7. PHASE II BEFORE CYCLING
(TOP VIEW)

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DATE

NEGATIVE NUMBER

H-6

COMP B

PHOTO 8. PHASE II BEFORE CYCLING
(SIDE VIEW)

TNT:CONT



NEAT

1/2% HNS

7% RDX

46

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NEGATIVE NUMBER

TNT:CONT



1-1/2% HNS

NEAT

PHOTO 9. PHASE II BEFORE CYCLING
(PARTIALLY CONFINED)

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DMM

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DATE

NEGATIVE NUMBER

FISHER SCIENTIFIC

H-6

COMP B

TNT:CONT

NEAT

1-1/2% HNS

7% RDX

PHOTO 10. PHASE II 3-MO CYCLE

COPY PHOTOGRAPH

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NEGATIVE NUMBER

TNT:CONT



PHOTO 11. PHASE II 3-MO CYCLE
(PARTIALLY CONFINED)

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NEGATIVE NUMBER

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H-6

COMP B

TNT:CONT

NEAT

1-1/2% HNS

7% RDX

PHOTO 12. PHASE II 6-MO CYCLE
(TOP VIEW)

SUBJECT _____

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H-6

COMP B

PHOTO 13. PHASE II 6-MO CYCLE
(SIDE VIEW)

TNT:CONT

NEAT

1-1/2% HNS

7% RDX

51

25 APR 1979

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NEGATIVE NUMBER

TNT:CONT



1-1/2% HNS

NEAT

PHOTO 14. PHASE II 6-MO CYCLE
(PARTIALLY CONFINED)

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1 2 3
SPEC. DATE

TNT
3 HRS 110°C

TNT:HNS 1/2%
0.1 HR 100°C

TNT:HNS 1/2%
0.25 HR 100°C

TNT:HNS 1/2%
0.5 HR 100°C

TNT:HNS 1/2%
3 HRS 100°C

TNT:HNS 1/2%
3 HRS 110°C

PHOTO 15. ADDENDUM: PROCESSING
TIME VS TEMPERATURE

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