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TECHNICAL REPORT

WHITE OAK LABORATORY

A CHARACTERIZATION STUDY OF SEVERAL HEAT RESISTANT EXPLOSIVES IN MILD DETONATING CORDS

BY
E. Eugene Kilmer

30 AUGUST 1976

NAVAL SURFACE WEAPONS CENTER
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20. ABSTRACT *(Continue on reverse side if necessary and identify by block number)*

A study was made of the performance of three new heat resistant explosives fabricated into mild detonating cords. Cords were exposed to temperatures from 280°C to 326°C in sealed copper tubes. After exposure to these temperatures, the cord detonation velocity was measured. The dynamic compressibility and heat of formation of TPT were also determined. TPT was judged to be the best explosive for heat resistant detonating cord and was characterized to generate data for a procurement specification.

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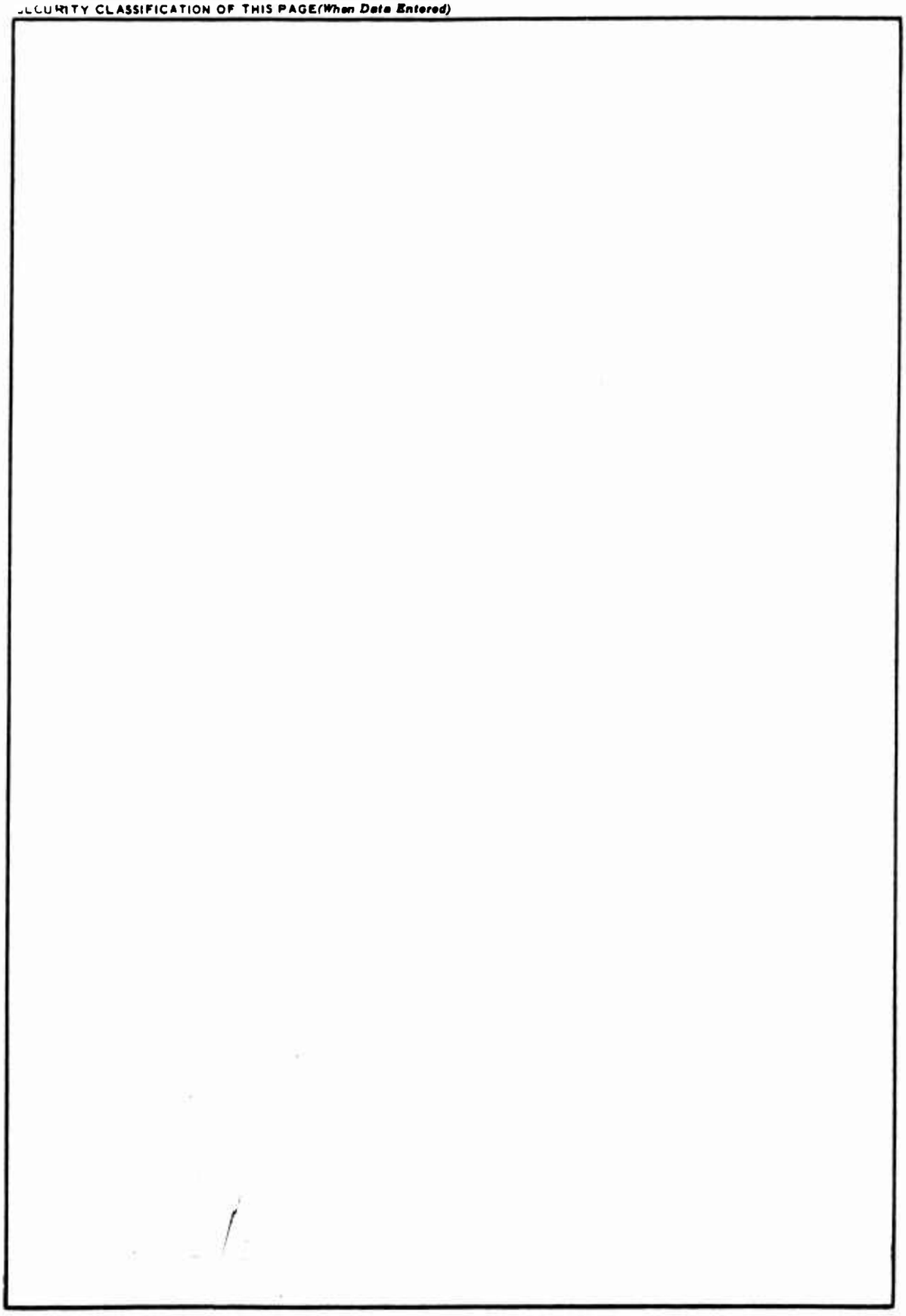

NSWC/WOL/TR 76-43 30 August 1976

A CHARACTERIZATION STUDY OF SEVERAL HEAT RESISTANT EXPLOSIVES IN MILD DETONATING CORDS

This is a final report on work conducted for the Lyndon B. Johnson Spacecraft Center at Houston, Texas, under Task NSWC-0841/NASA-T-8917E, which covered the period 1 July 1974 - 31 December 1975.

This work was carried out to characterize several new thermally stable explosives in detonating cords and to determine their usefulness as explosive components for possible application in the Space Shuttle Program. The latest findings on the performance of these cords after exposure to elevated temperature are discussed.

The author wishes to acknowledge the TPT synthesis by Dr. Joseph Dacons, the dynamic compressibility tests by Dr. Jerry Forbes both of the Naval Surface Weapons Center and the determination of heat of formation of TPT by E. E. Baroody of the Naval Ordnance Station, Indian Head, Maryland. The identification of commercial materials implies no criticism or endorsement by the Naval Surface Weapons Center.

J. Kabik

I. KABIK
By direction

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THE DEVELOPMENT OF SPACECRAFT EXPLOSIVES FOR THE LYNDON B. JOHNSON SPACE CENTER

1. INTRODUCTION

1.1 This is a Naval Surface Weapons Center (NAVSURFWPNCEN) report on work done on the "Space Shuttle Explosives" task for the National Aeronautics and Space Agency (NASA), Lyndon B. Johnson Space Center at Houston, Texas. The purpose of the work was to characterize several heat resistant secondary explosives suitable for use in spacecraft hardware and select a prime candidate for further study. The design goal criterion was "no significant degradation of the explosive after exposure to 315°C (600°F) for one hour".

1.2 The final selection of a prime candidate explosive for further study was made from three candidates, PYX (2,6-bis (picrylamino) -3,5-dinitropyridine)[1,2], ONT (octanitro-m-terphenyl)[3,4] and TPT (2,4,6 Tripicryl-s-triazine)[5], following performance tests in detonating cords. TPT was chosen, and was further characterized and evaluated in order to obtain data for a procurement specification.

2. EXPOSURE OF PYX DETONATING CORDS TO ELEVATED TEMPERATURES

2.1 PYX, one of the three heat resistant explosives being investigated, was procured from the Los Alamos Scientific Laboratory (LASL). Scanning Electron Microscope (SEM) photomicrographs of this PYX (ID 1817) are shown in Figure 1. The literature publication[1] on PYX reveals the product was washed with concentrated nitric acid, water, and methanol, and then dried at 140°C. There were no published vacuum thermal stability results for the laboratory or pilot plant synthesized explosive. Test results[2] from NAVSURFWPNCEN show a large quantity of gas for the initial 20 minutes (about three times the acceptable maximum amount) of the vacuum thermal stability test. In

(1) Coburn, M. D. and Singleton, J. L., J. Heter. Chem., 9, 1039, 1972
(2) Kilmer, E., "A Characterization Study of Several Heat Resistant Explosives," NOLTR 74-177, 4 Oct 1974
(3) Dacons, J. C., "Heat Resistant Explosives XXIII. The Preparation and Properties of 2,2';4,4';4';6,6';6" - Octanitro-m-terphenyl, ONT," NOLTR 66-179, 5 Oct 1966
(4) Dacons, J. C., US Patent #3,592,860, "Octanitroterphenyl," 13 Jul 1971 patented
(5) Dacons, J. C., US Patent #3,755,321, "2,4,6-tripicryl-s-triazine," 28 Aug 1973 patented

250 X ID 1817 1 CM = 40μ

500 X ID 1817 1 CM = 20μ

500 X ID 1817 1 CM = 20μ

FIG. 1 SEM PHOTOMICROGRAPHS OF PYX (ID1817) "AS REC'D" FROM LASL

view of these results, LASL (Dr. M. Coburn) was contacted by NAVSURFWPNCEN (Dr. J. Dacons). Dr. Coburn recommended that the explosive received from the LASL pilot plant be "cleaned-up" by dissolving the PYX in red fuming nitric acid and recrystallizing it. This was done and the PYX was resubmitted for vacuum stability testing. The surge value was reduced to an acceptable quantity. The recrystallized PYX (ID 1854) is shown in Figure 2.

2.2 A quantity of PYX was furnished to the Ensign Bickford Company to be fabricated into detonating cords. Cords were loaded with 2-3 gr/ft and 10-15 gr/ft explosive in aluminum and silver sheaths. These cords were sealed into copper tubes by cold welding and then exposed to elevated temperatures. The assemblies were exposed to 315° and 326°C temperatures for 1/2, 1, and 2 hours time.

2.3 The detonating cords were removed and cooled to room temperature. Detonation velocities were measured on each piece of detonating cord. The detonation velocity is plotted as a function of exposure time to elevated temperature in Figures 3, 4, 5, and 6. Each point represents a detonation velocity for a single cord exposure to the elevated temperature.

2.4 The degradation of the detonation velocity is a measure of the performance of the cords over the time period indicated. Some degradation of the PYX is taking place at 315°C but should not affect its performance in aluminum or silver sheath detonating cords over the two hour exposure period. However, the degradation is taking place at a greater rate when the explosive is subjected to a higher penalization temperature of 326°C. The rate of degradation of PYX in the lower core load (2-1/2 gr/ft) material appears to be greater than with the 10-12 gr/ft cords at 326°C.

3. EXPOSURE OF ONT DETONATING CORDS TO ELEVATED TEMPERATURES

3.1 ONT, one of the heat resistant explosives of the group of materials investigated for NASA, was fabricated into detonating cords and subjected to elevated temperatures. ONT was synthesized in 1966 by Dacons[3,4]. Some of the first work on studying the performance in detonating cords was done by Kilmer[5] in 1970 under a NASA "High and Low Temperature Resistant Devices" task.

3.2 As a part of the present task, it was decided (because of ONT's good thermal properties) to investigate ONT at higher temperatures than considered in the past. ONT loaded aluminum and silver sheathed cords were sealed into copper tubes by cold welding. They were exposed to elevated temperatures of 280°C, 300°C, and 315°C in the same way as were the PYX cords. SEM photomicrographs of the ONT explosive are shown in Figure 7.

3.3 Cords containing ONT were fired at a room temperature after exposure to elevated temperatures. The detonation velocities of the cords are shown as a function of exposure time at elevated temperatures in Figures 8 through 19. It should be noted that the characterization studies on ONT indicate considerable gassing at

250 X ID 1854

800 X ID 1854

1000 X ID 1854

FIG. 2 SEM PHOTOMICROGRAPHS OF PYX (ID1854) RECRYSTALLIZED FROM RED FUMING NITRIC ACID

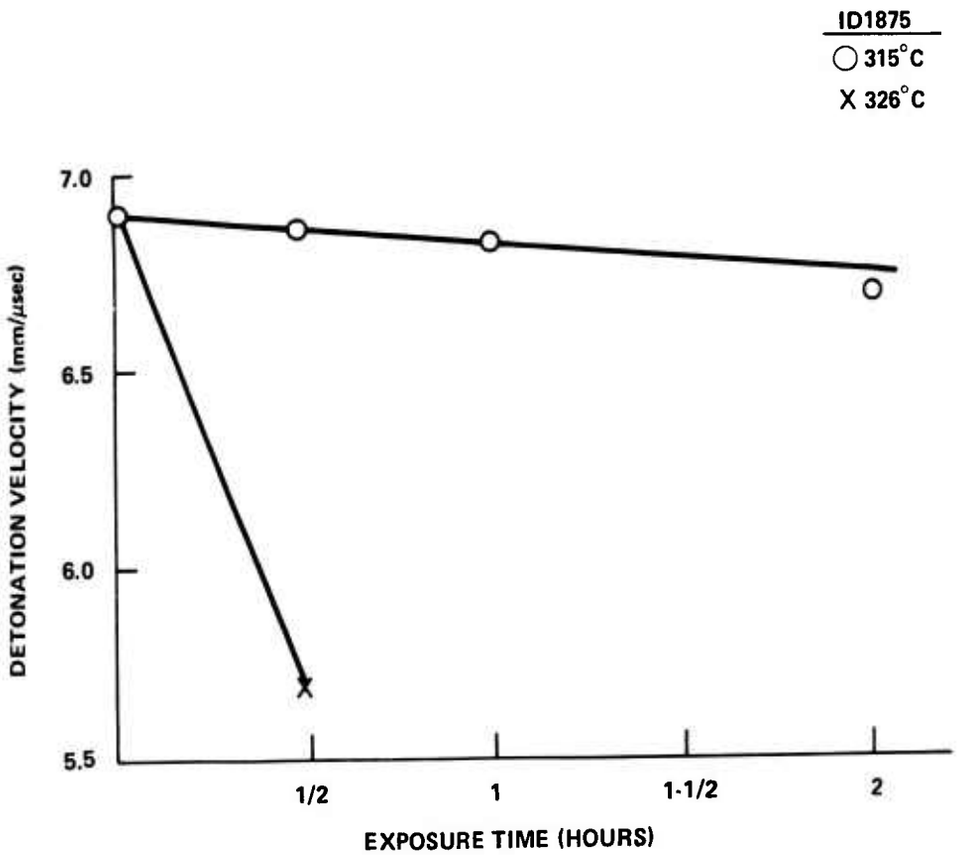


FIG. 3 THE DETONATION VELOCITY OF PYX IN ALUMINUM MDC (2-1/2gr/ft) AFTER EXPOSURE TO ELEVATED TEMPERATURES

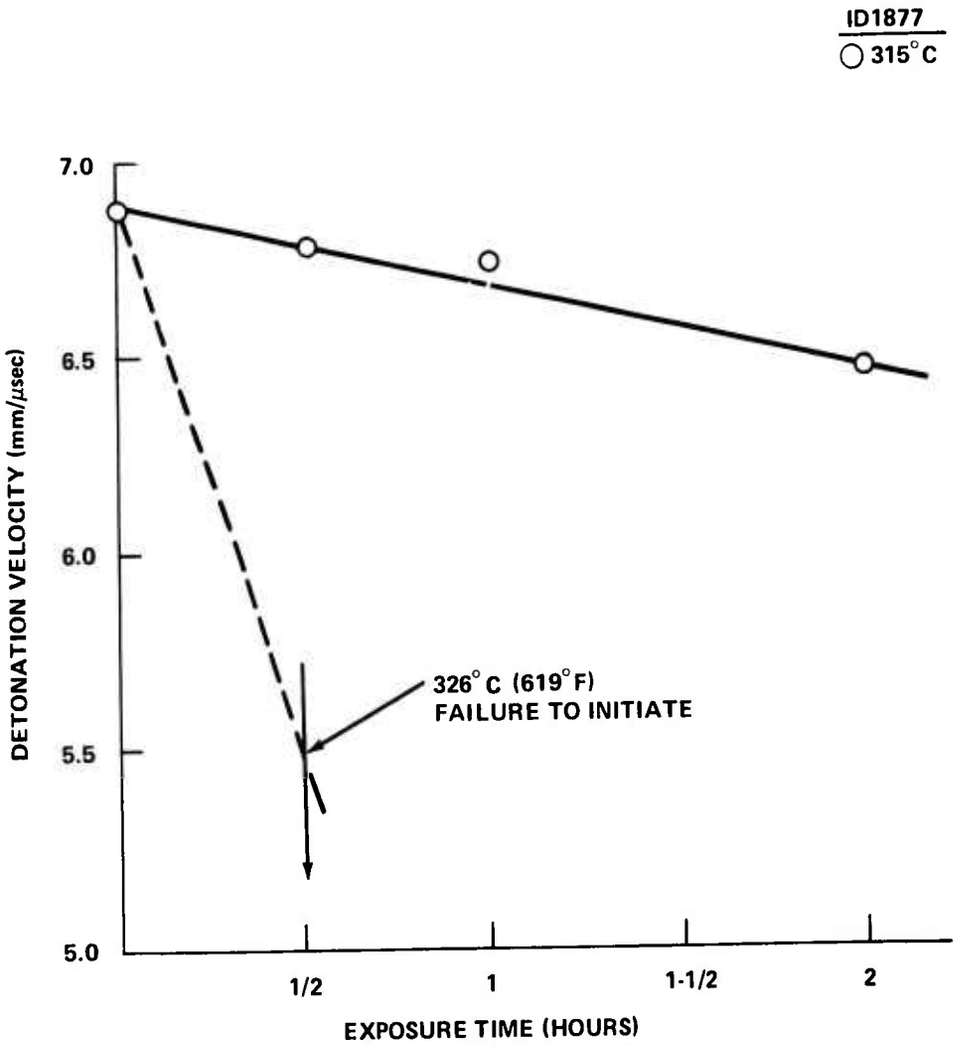


FIG. 4 THE DETONATION VELOCITY OF PYX IN SILVER MDC (2-1/2gr/ft) AFTER EXPOSURE TO ELEVATED TEMPERATURES

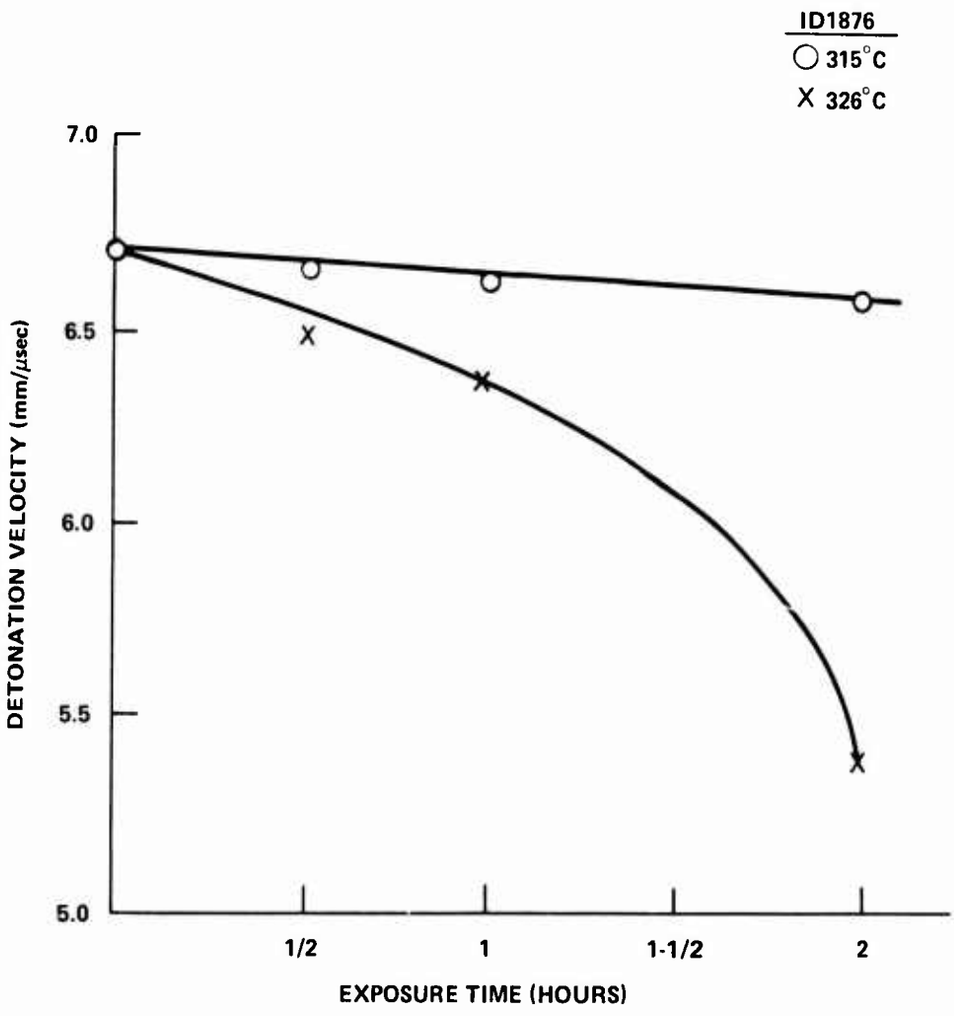


FIG. 5 THE DETONATION VELOCITY OF PYX IN ALUMINUM MDC (12gr/ft) AFTER EXPOSURE TO ELEVATED TEMPERATURES

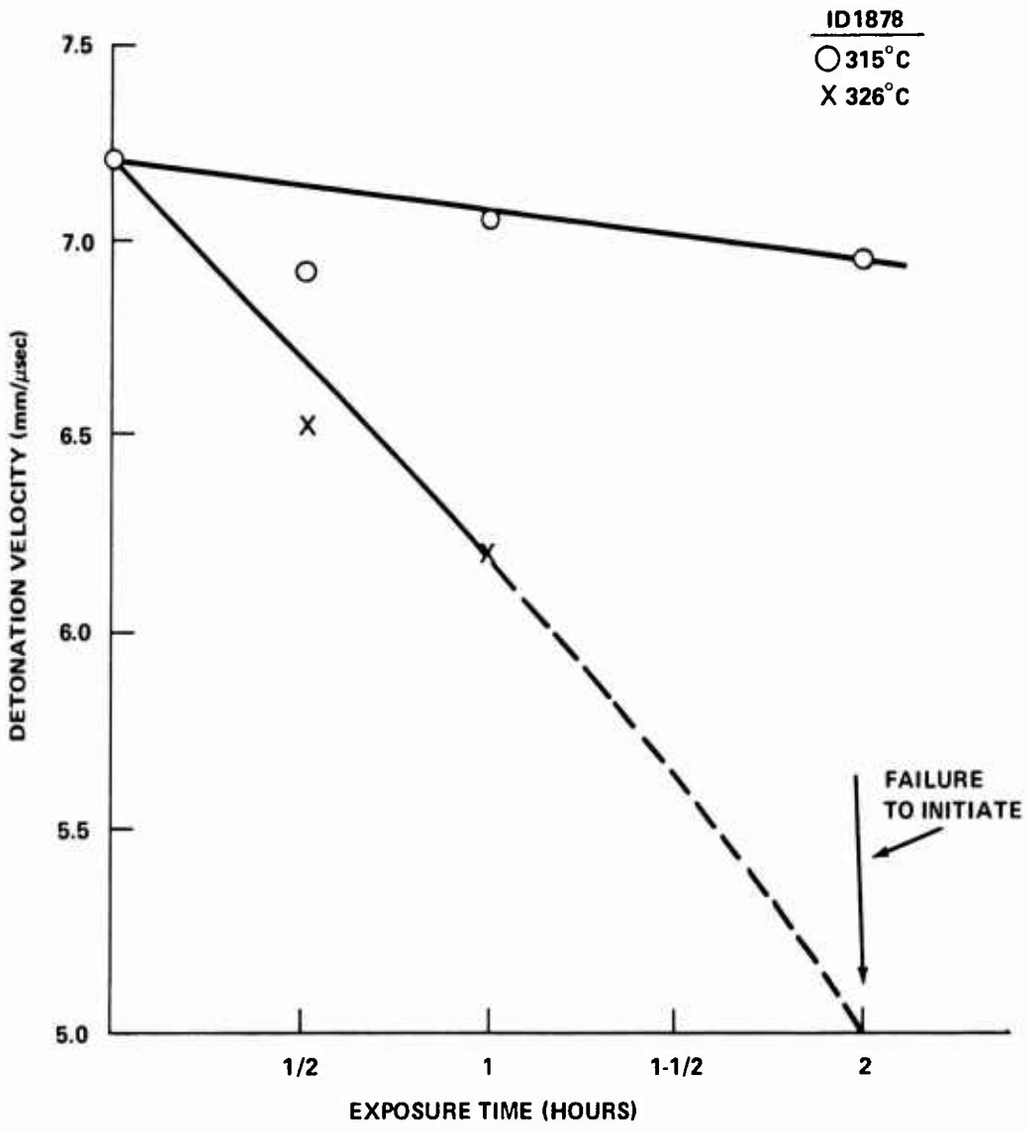


FIG. 6 THE DETONATION VELOCITY OF PYX IN SILVER MDC(10gr/ft) AFTER EXPOSURE TO ELEVATED TEMPERATURES

50 X ID 1803

100 X ID 1803

250 X ID 1803

FIG. 7 SEM PHOTOMICROGRAPHS OF ONT (ID1803)

300°C in the vacuum thermal stability test. Therefore, it was expected that performance would probably be limited to temperatures below 300°C.

3.4 At the outset of the exposure to elevated temperature, i.e., the first one-half to one hour, the aluminum cords decrease a few hundred meters/sec in detonation velocity relative to the "as received" cords, but the change in velocity decreases with increased exposure time over a 7-1/2 hour period at 280°C and 300°C. This loss of detonation velocity is typical of other heat resistant explosives, such as HNS, when fabricated into cords. However, the change in detonation velocity is significant at 315°C (600°F) (see Figures 12 and 13), and the ONT is degrading fast enough so as to make its use questionable in hardware at this temperature.

4. EXPOSURE OF TPT DETONATING CORDS TO ELEVATED TEMPERATURES

4.1 TPT was fabricated into detonation cords. The core loadings were 2-3 gr/ft and 10-15 gr/ft in aluminum and silver sheaths. Lengths of detonating cords (10.2 cm or 4.0 inches), containing TPT, were sealed into copper tubes by cold welding the ends of the tubes together, exposed to elevated temperatures of 135°C, and 326°C, removed, and fired at room temperature. The detonation velocities of these cords are shown as a function of the exposure time in Figures 20 and 21.

4.2 One must consider the scatter in the data in the small number of tests reported but it appears from the plot of values in Figures 20 and 21 that the degradation of the performance of the explosive is slightly greater for the low core load material. This may be a function of the core load or quantity of explosive available or it may be caused by the difference in the final densities in the cords. This type of degradation caused by a combination of high density/high temperature exposure has been shown before with detonating cords containing HNS (Hexanitrostilbene)[6]. TPT shows the same degradation at the higher penalization temperature of 326°C.

5. EXPOSURE OF TPT AND PYX DETONATING CORDS TO ELEVATED TEMPERATURES FOR AN EXTENDED PERIOD OF TIME

5.1 In the past, the NAVSURFWPNCEN developed dipicramide (DIPAM) and hexanitrostilbene (HNS) (Figures 22 and 23) have been exposed to temperatures of 238°C (460°F) for relatively long periods of time[7]. Degradation occurred of such a magnitude as to cause performance problems after about three days at this temperature.

---

(6) Kilmer, E., "Detonating Cords Loaded with Hexanitrostilbene (HNS) Recrystallized from Acid or Organic Solvents," NSWC/WOL/TR 75-142, 2 Sep 1975.

(7) Kilmer, E., "Annual Report on Investigation of High and Low Temperature Resistant Explosive Devices," NOLTR 67-1333, 18 Oct 1967.

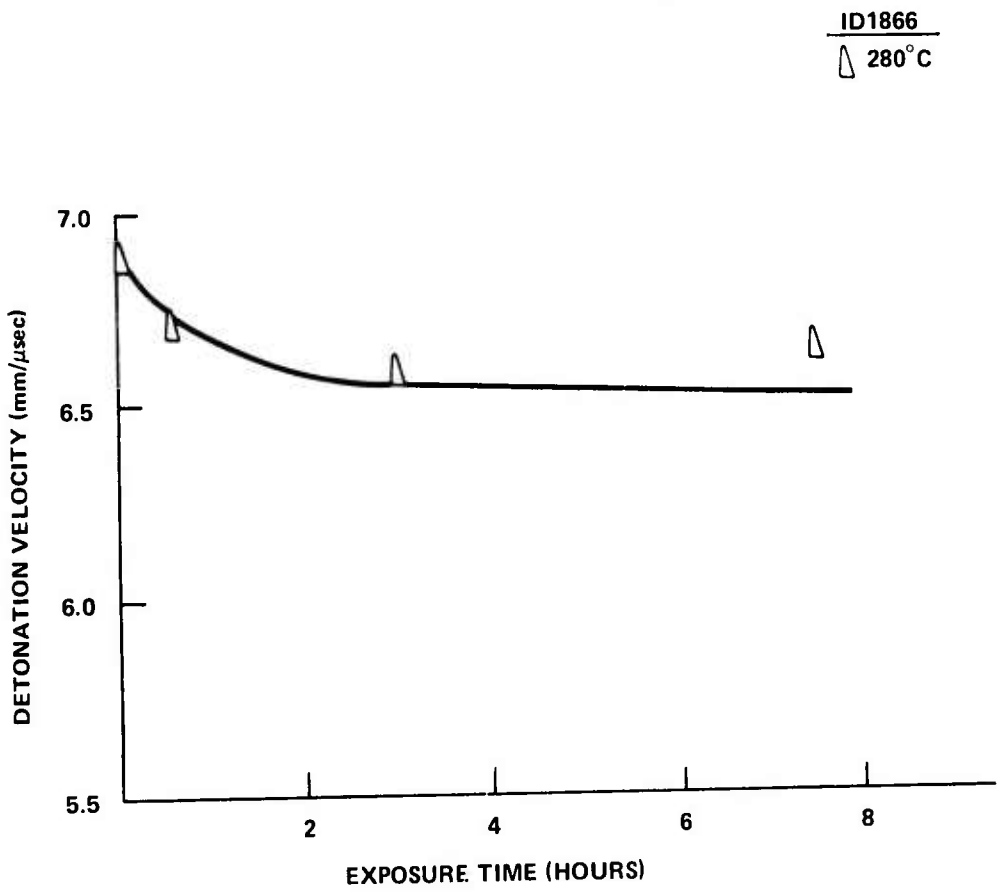


FIG. 8 THE DETONATION VELOCITY OF ONT IN ALUMINUM MDC (2-1/2gr/ft) AFTER EXPOSURE TO ELEVATED TEMPERATURE

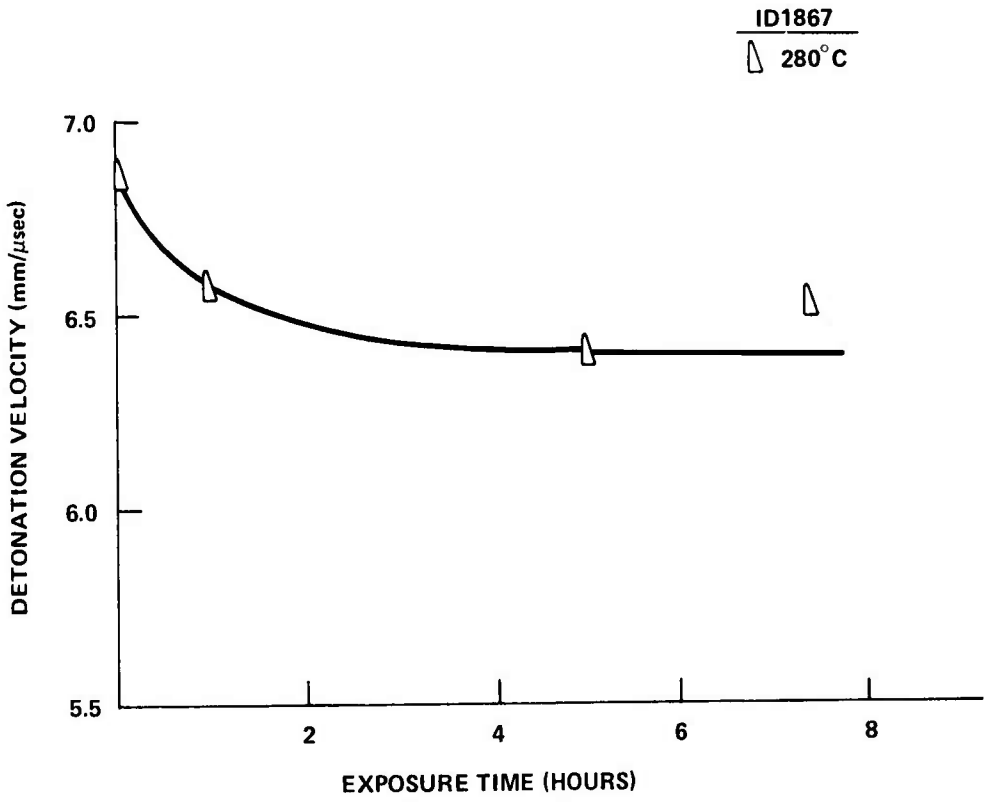


FIG. 9 THE DETONATION VELOCITY OF ONT IN ALUMINUM MDC (10gr/ft) AFTER EXPOSURE TO ELEVATED TEMPERATURE

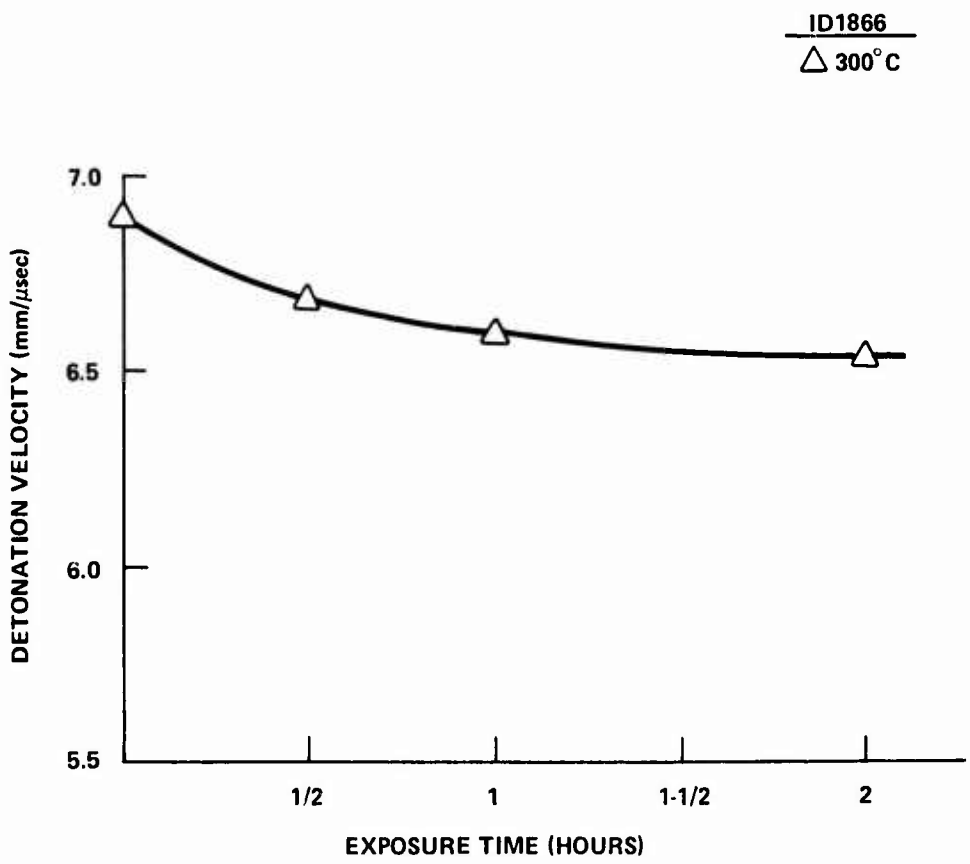


FIG. 10 THE DETONATION VELOCITY OF ONT IN ALUMINUM MDC (2-1/2gr/ft) AFTER EXPOSURE TO ELEVATED TEMPERATURE

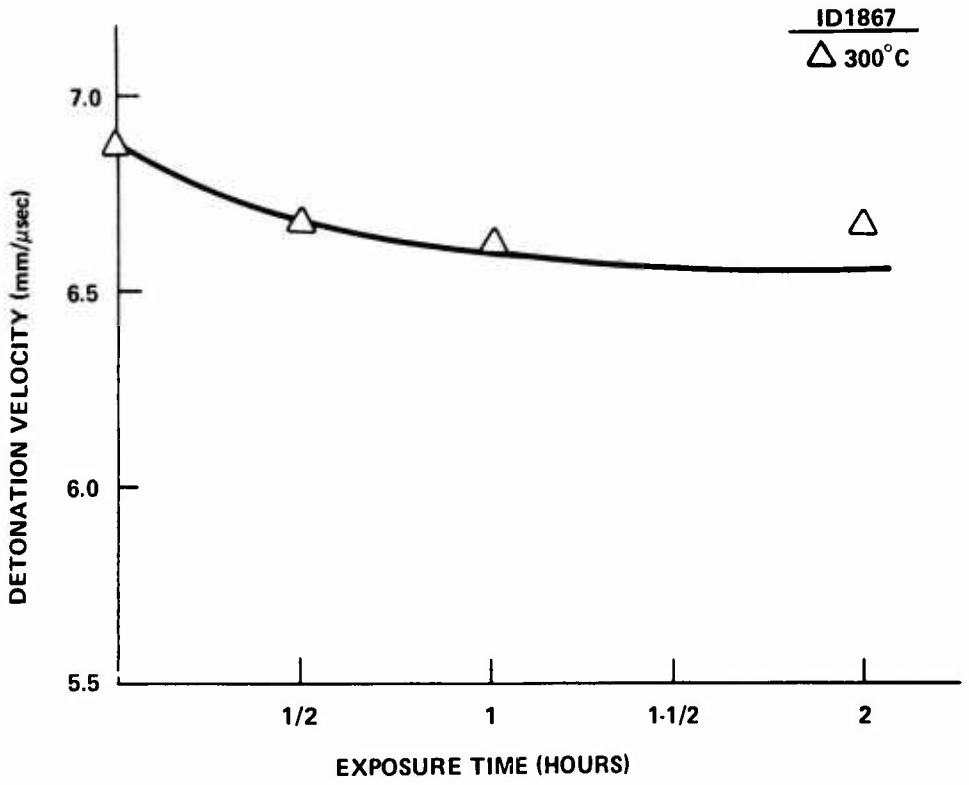


FIG. 11 THE DETONATION VELOCITY OF ONT IN ALUMINUM MDC (10gr/ft) AFTER EXPOSURE TO ELEVATED TEMPERATURE

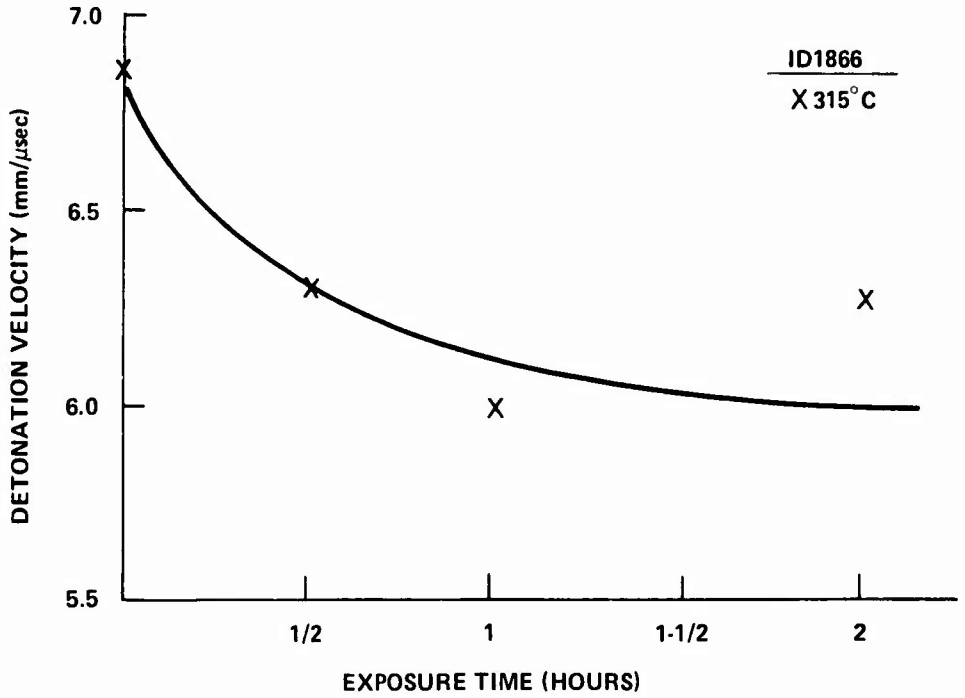


FIG. 12 THE DETONATION VELOCITY OF ONT IN ALUMINUM MDC 2-1/2gr/ft) AFTER EXPOSURE TO ELEVATED TEMPERATURE

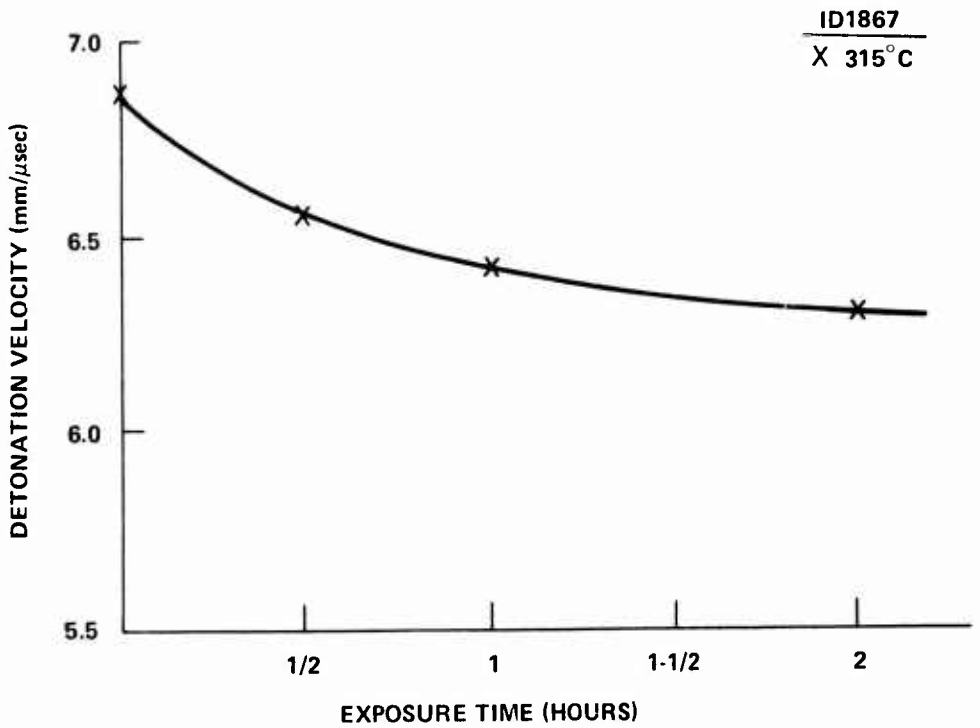


FIG. 13 THE DETONATION VELOCITY OF ONT IN ALUMINUM MDC (10gr/ft) AFTER EXPOSURE TO ELEVATED TEMPERATURE

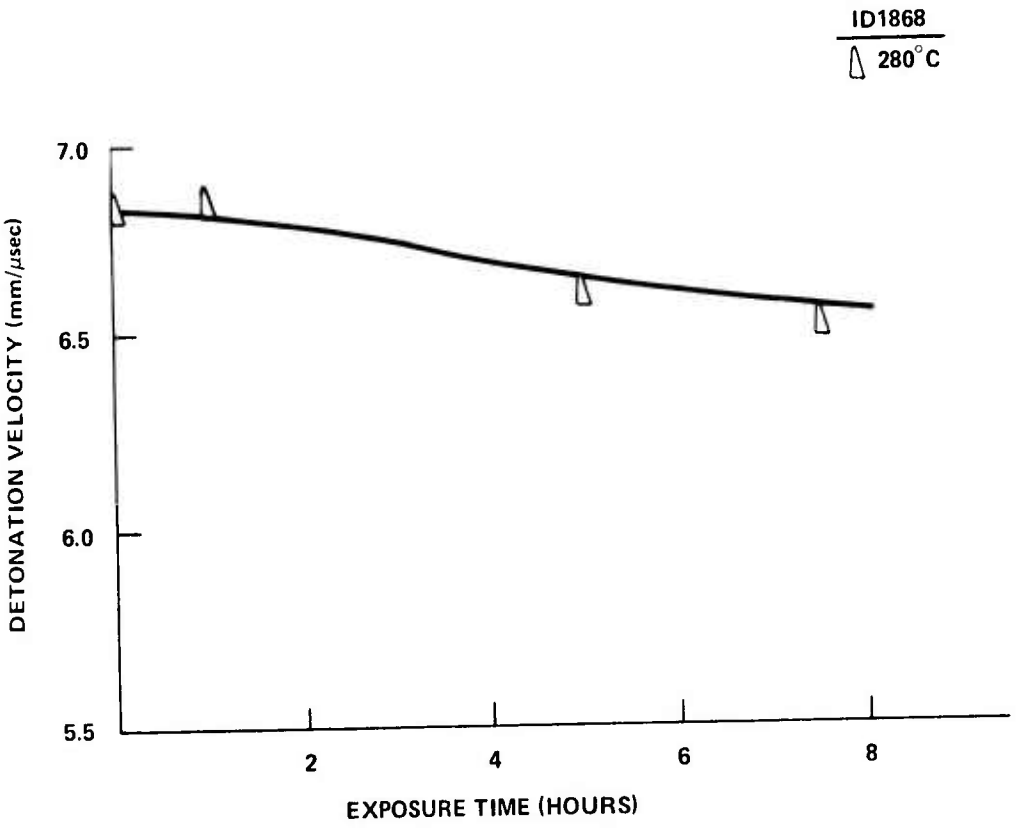


FIG. 14 THE DETONATION VELOCITY OF ONT IN SILVER MDC (2-1/2gr/ft) AFTER EXPOSURE TO ELEVATED TEMPERATURE

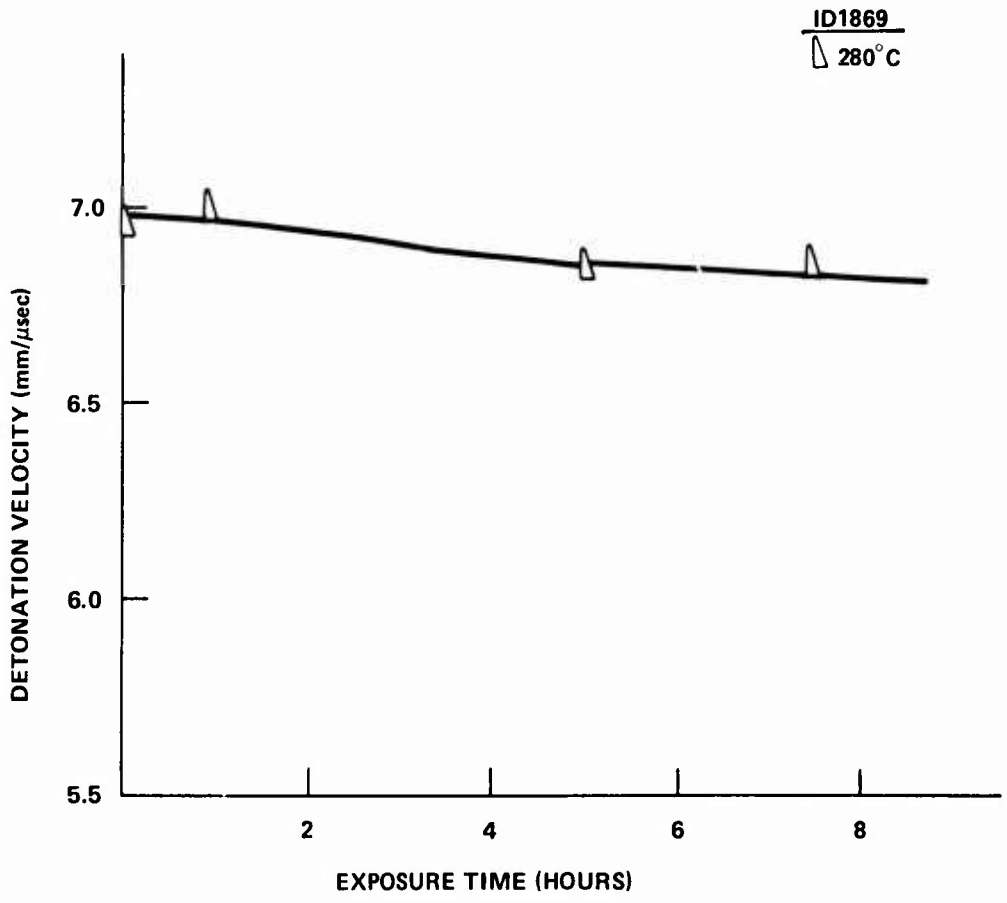


FIG. 15 THE DETONATION VELOCITY OF ONT IN SILVER MDC (10gr/ft) AFTER EXPOSURE TO ELEVATED TEMPERATURE

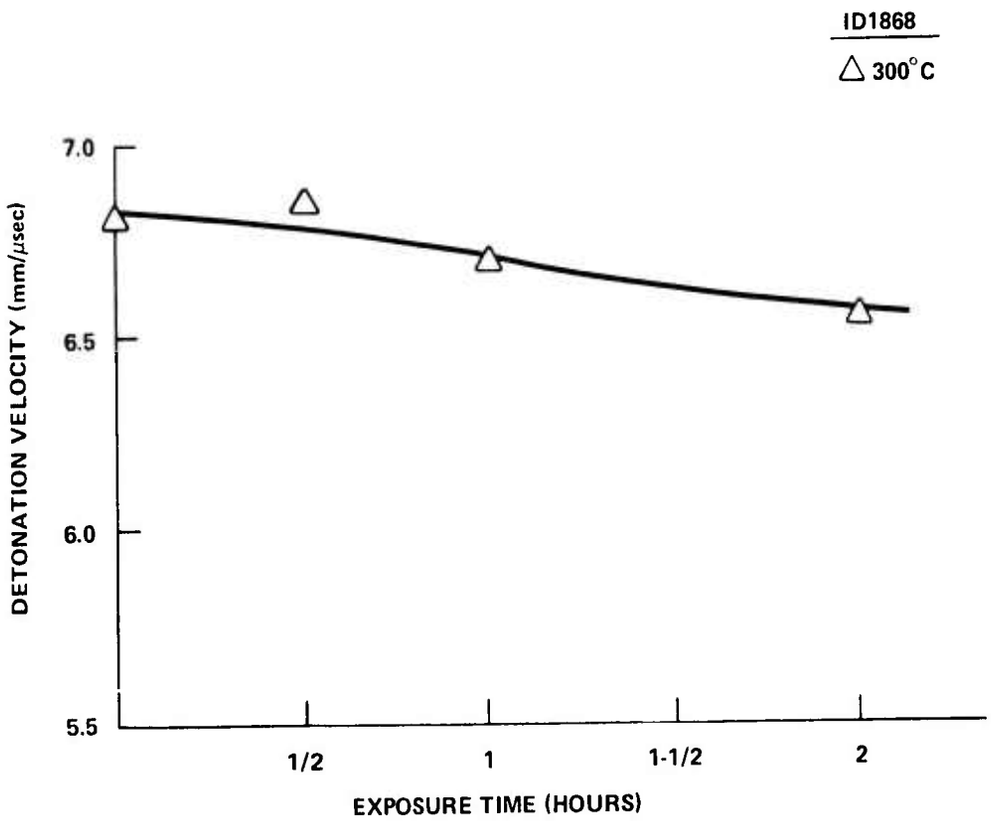


FIG. 16 THE DETONATION VELOCITY OF ONT IN SILVER MDC (2-1/2gr/ft) AFTER EXPOSURE TO ELEVATED TEMPERATURE

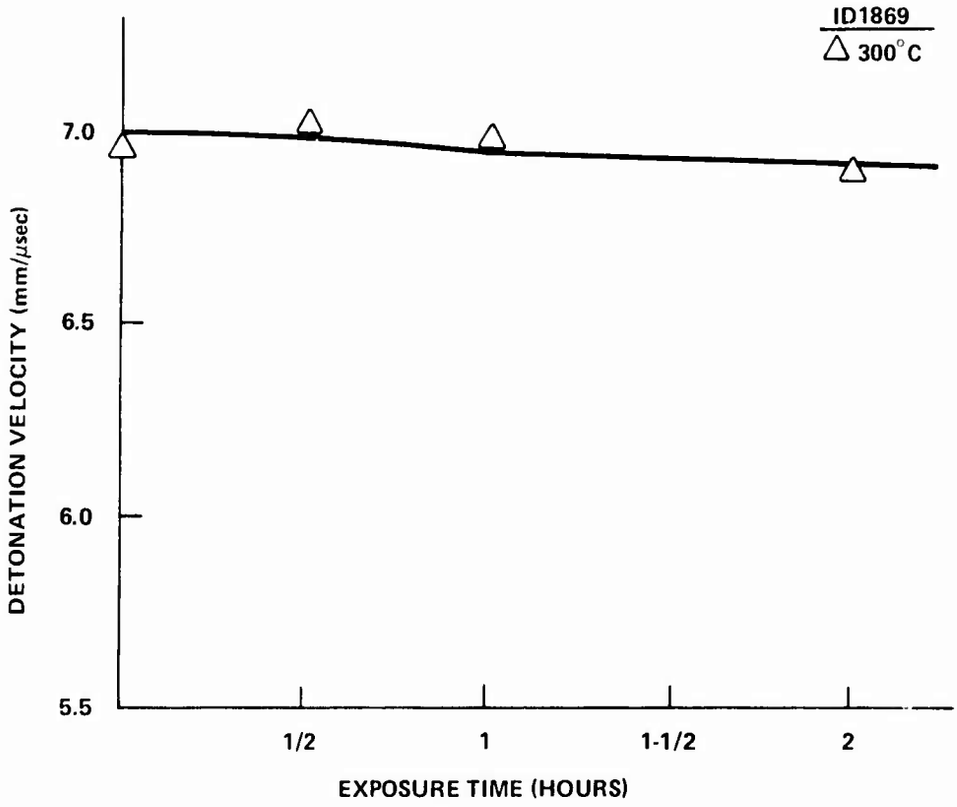


FIG. 17 THE DETONATION VELOCITY OF ONT IN SILVER MDC (10gr/ft) AFTER EXPOSURE TO ELEVATED TEMPERATURE

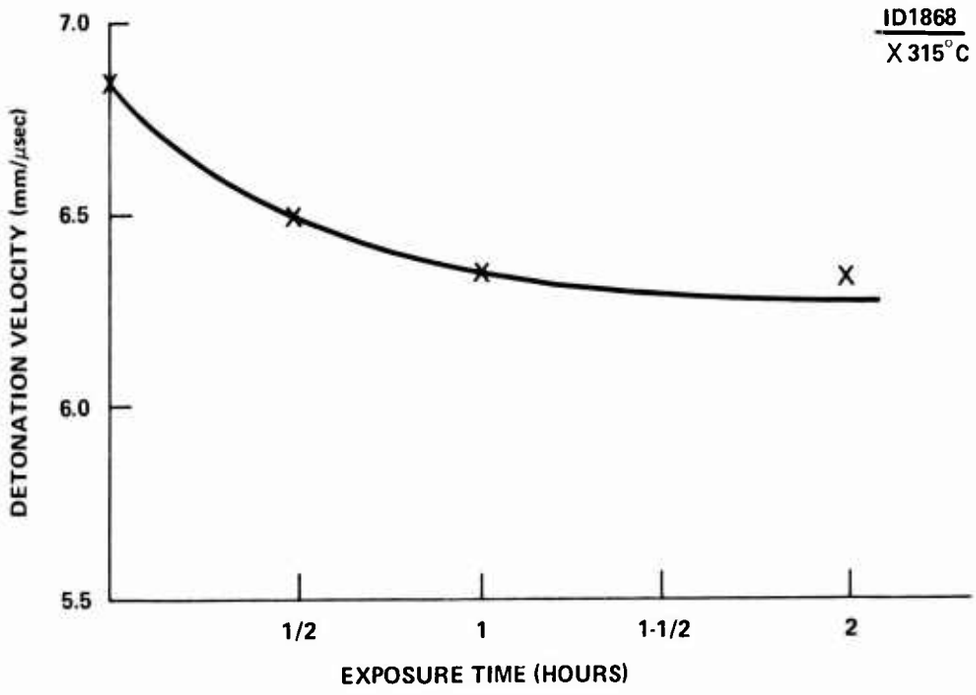


FIG. 18 THE DETONATION VELOCITY OF ONT IN SILVER MDC (2gr/ft) AFTER EXPOSURE TO ELEVATED TEMPERATURE

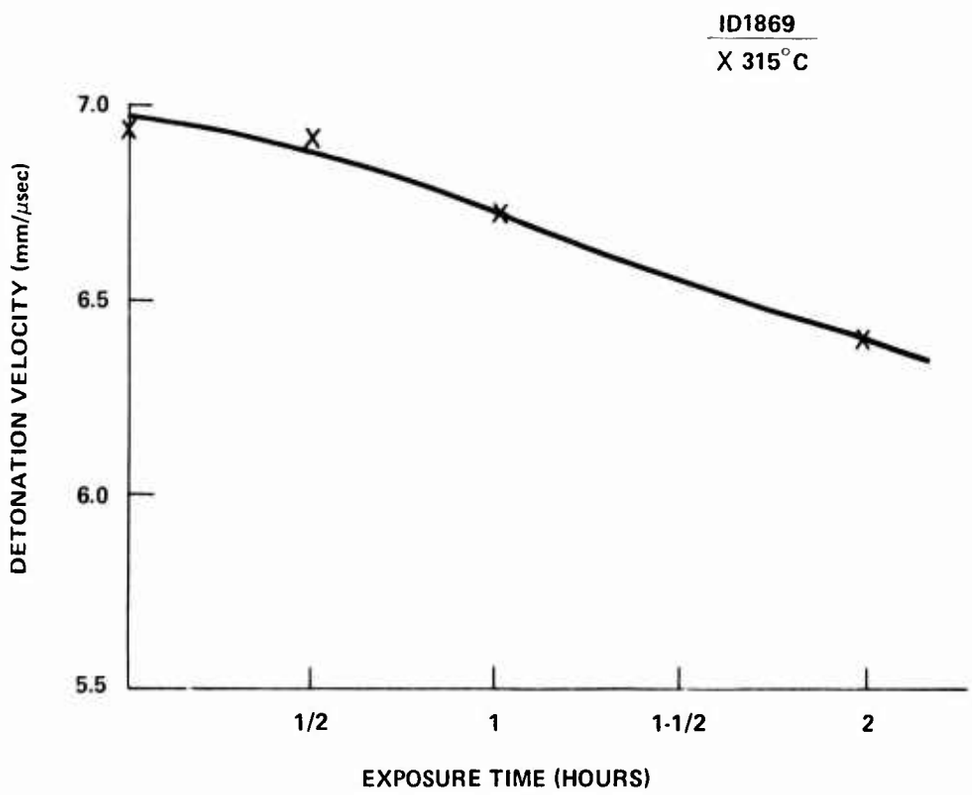


FIG. 19 THE DETONATION VELOCITY ON ONT IN SILVER MDC (10gr/ft) AFTER EXPOSURE TO ELEVATED TEMPERATURE

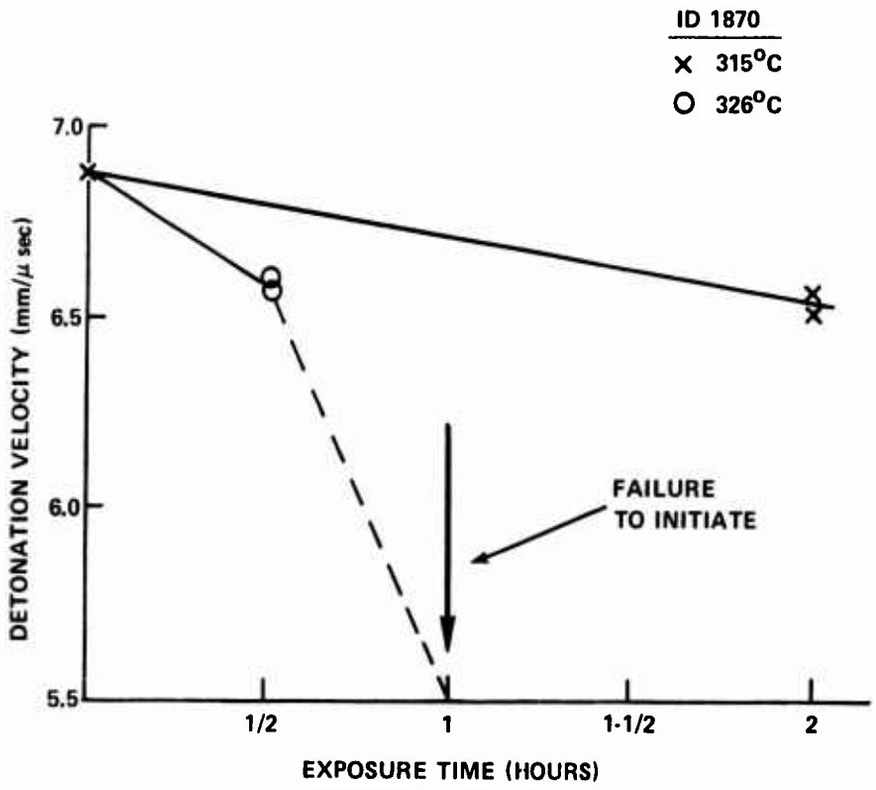


FIG. 20 THE DETONATION VELOCITY OF TPT IN ALUMINUM MDC (2-1/2gr/ft) AFTER EXPOSURE TO ELEVATED TEMPERATURES

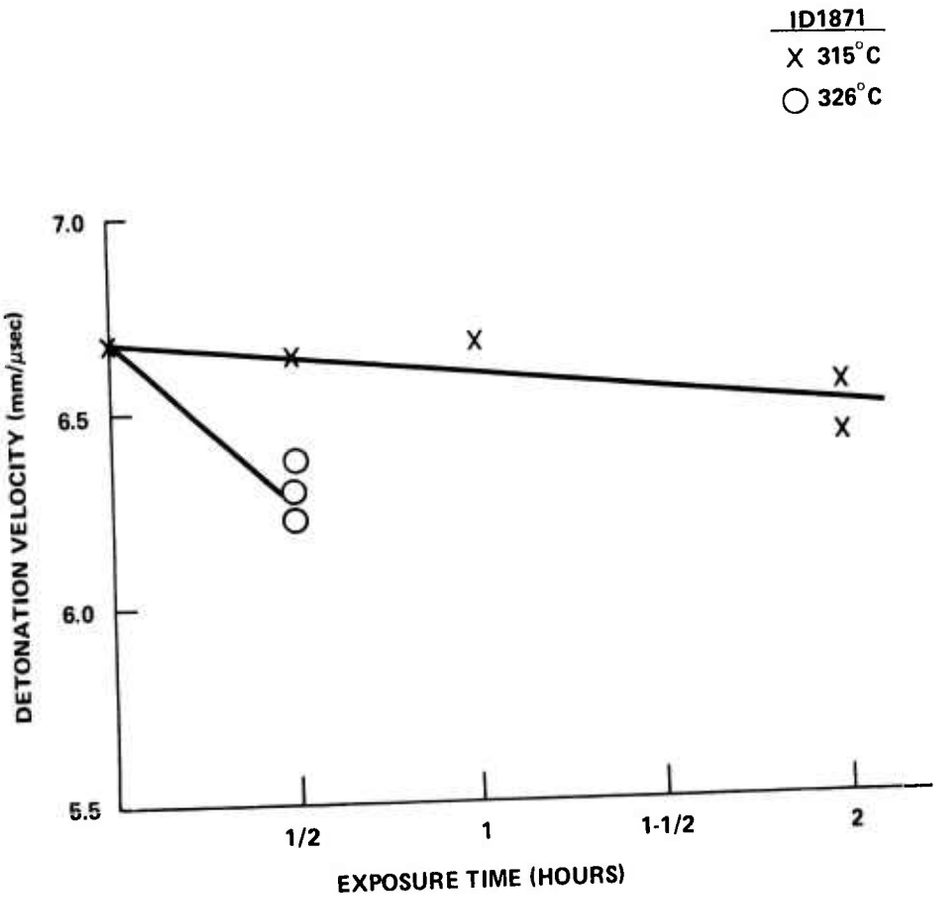


FIG. 21 THE DETONATION VELOCITY OF TPT IN ALUMINUM MDC (10gr/ft) AFTER EXPOSURE TO ELEVATED TEMPERATURES

100 X ID 203

250 X ID 203

500 X ID 203

1000 X ID 203

FIG. 22 SEM PHOTOMICROGRAPHS OF DIPAM (ID203)

250 X ID 1479

500 X ID 1479

1000 X ID 1479

FIG. 23 SEM PHOTOMICROGRAPH OF HNS-II (ID1479)

5.2 A better characterization of the temperature stability of TPT and PYX explosives can be made if the time of exposure is increased and the temperature decreased. These explosives show thermal capabilities well above HNS, the NAVY's best "in-service" heat resistant explosive, when exposed for periods longer than two hours.

5.3 A study of the performance of TPT and PYX in detonating cords was made for periods of time of up to 24 hours at temperatures from 260°C (500°F) to 300°C (572°F). The exposures were made in sealed tubes as described above. The results of the detonation velocity measurements are shown plotted in Figures 24 through 31.

5.4 TPT is shown to be more thermally stable than PYX over a 24-hour period of exposure at 260°C in the 10 gr/ft detonating cords. With the small number of samples tested the reverse appears to be the case for low core load, 2.5 gr/ft, detonating cords in silver. The performance of all cords after 280°C exposure (except the 2-1/2 gr/ft TPT in silver) appear to be about the same as after exposure to 260°C. The results are erratic for both materials after exposure to 300°C.

6. CONCLUSIONS/RECOMMENDATIONS

6.1 It has been shown that ONT has surpassed the design goal criterion (no significant degradation of the explosive after exposure to 315°C (600°F) for one hour) but it should be considered to yield only marginal performance at 315°C. ONT does not have as good thermal stability in mild detonating cords as PYX or TPT when exposed to very high temperatures for short periods of time.

6.2 PYX has been tested in detonating cords and has surpassed the design goal for performance after one hour exposure at 315°C (600°F). There is little degradation after two hours at this temperature in most of the cords tested.

6.3 TPT detonating cords tested under the same conditions of elevated temperatures as PYX detonating cords performed equally as well for short periods of time at 315°C.

6.4 It is recommended that a future study might be implemented by exploring the performance of PYX and TPT at <u>various</u> <u>loading</u> <u>densities</u> in the cords to determine if the performance could be improved at elevated temperatures.

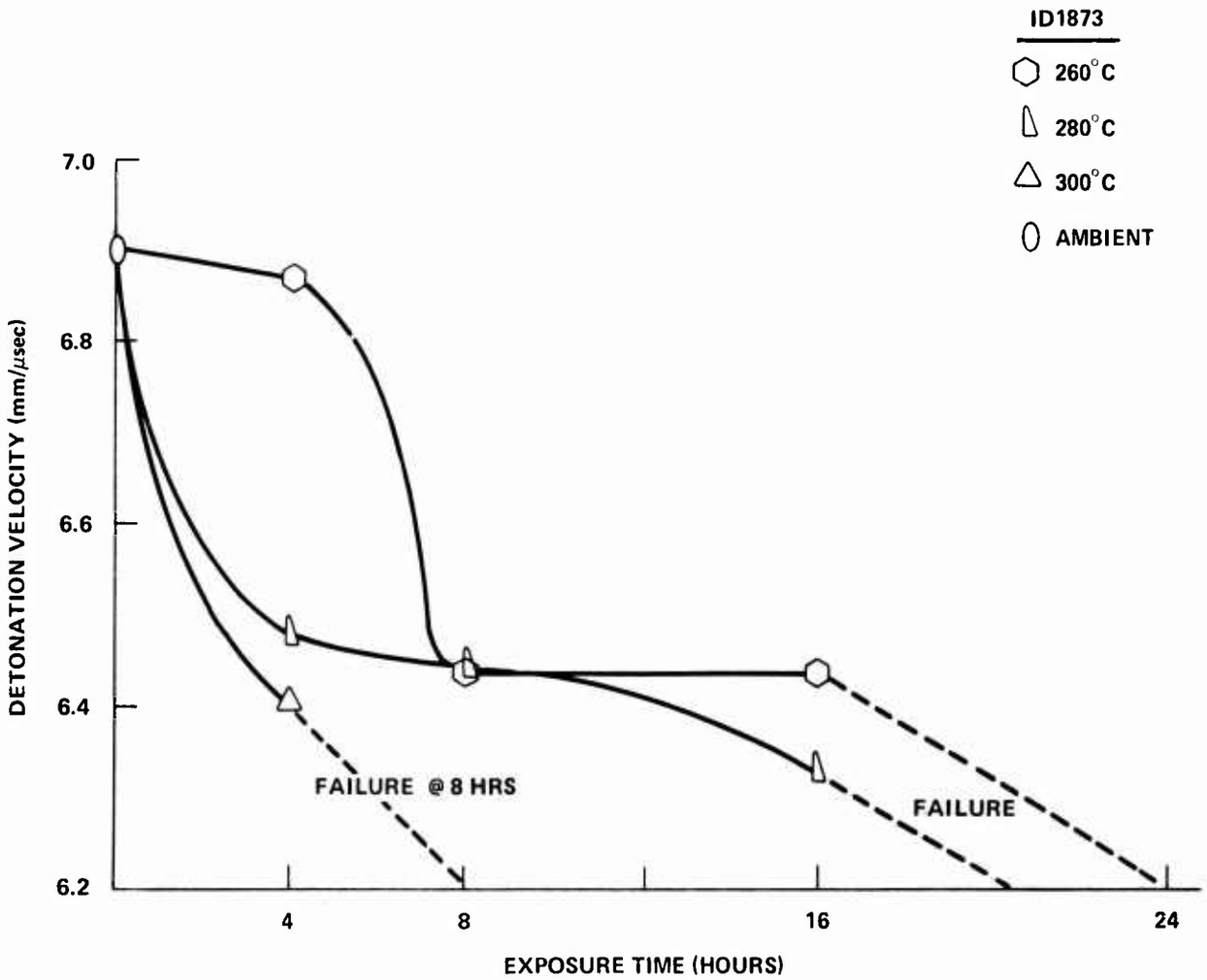


FIG. 24 THE DETONATION VELOCITY OF TPT IN SILVER MDC (2-1/2gr/ft) AFTER EXPOSURE TO ELEVATED TEMPERATURES

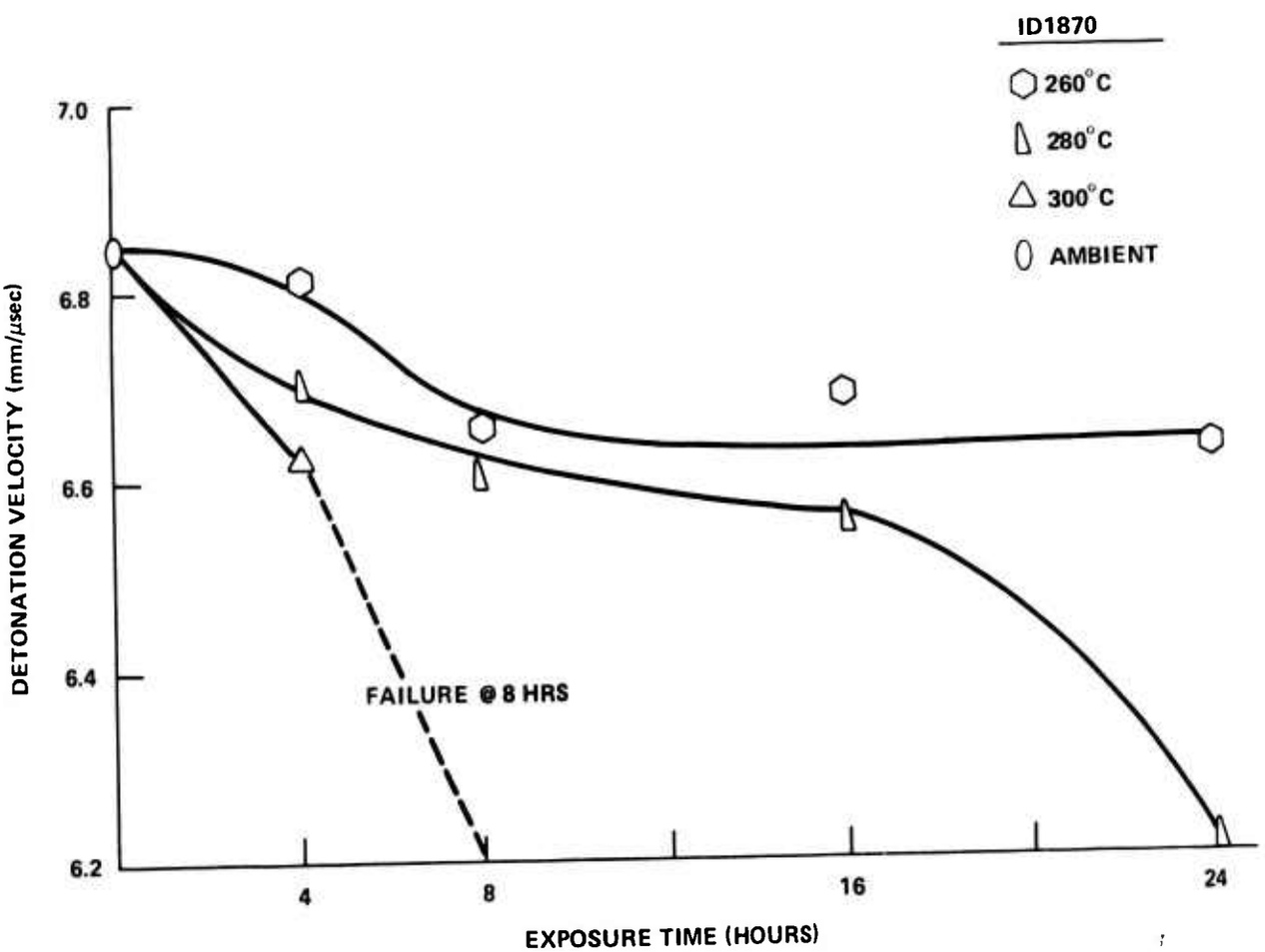


FIG. 25 THE DETONATION VELOCITY OF TPT IN ALUMINUM MDC (2-1/2gr/ft) AFTER EXPOSURE TO ELEVATED TEMPERATURES

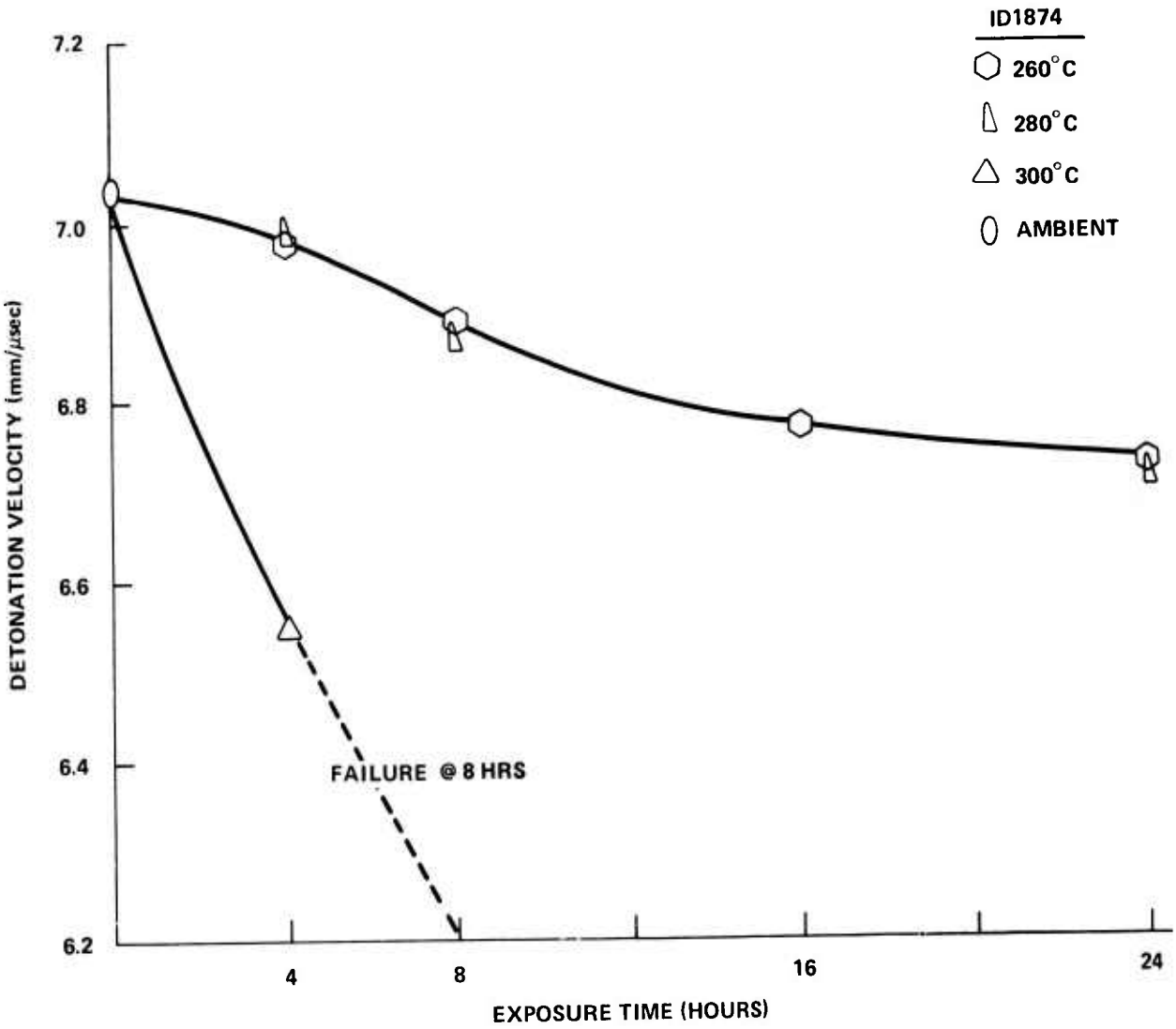


FIG. 26 THE DETONATION VELOCITY OF TPT IN SILVER MDC (10gr/ft) AFTER EXPOSURE TO ELEVATED TEMPERATURES

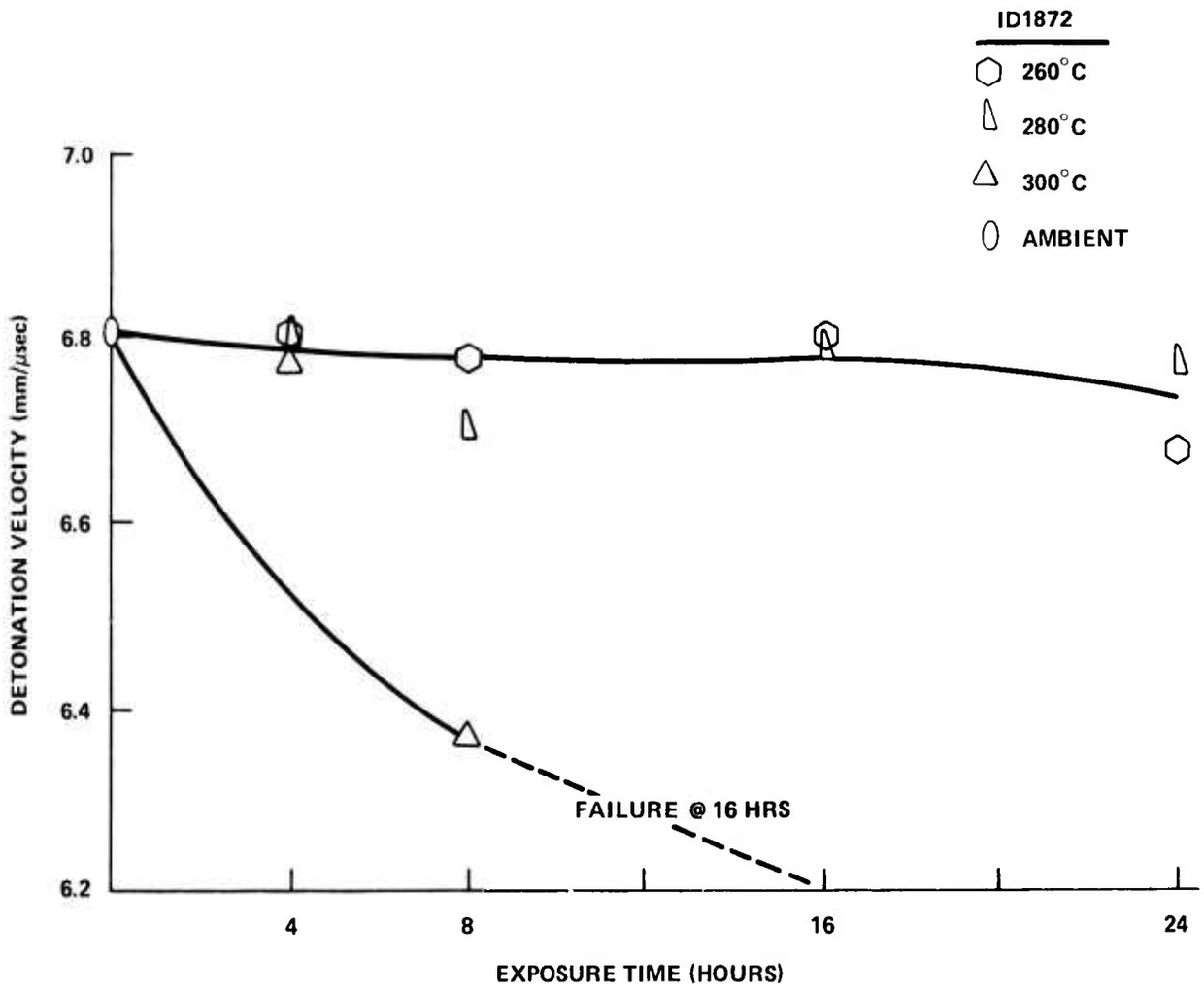


FIG. 27 THE DETONATION VELOCITY OF TPT IN ALUMINUM MDC (10gr/ft) AFTER EXPOSURE TO ELEVATED TEMPERATURES

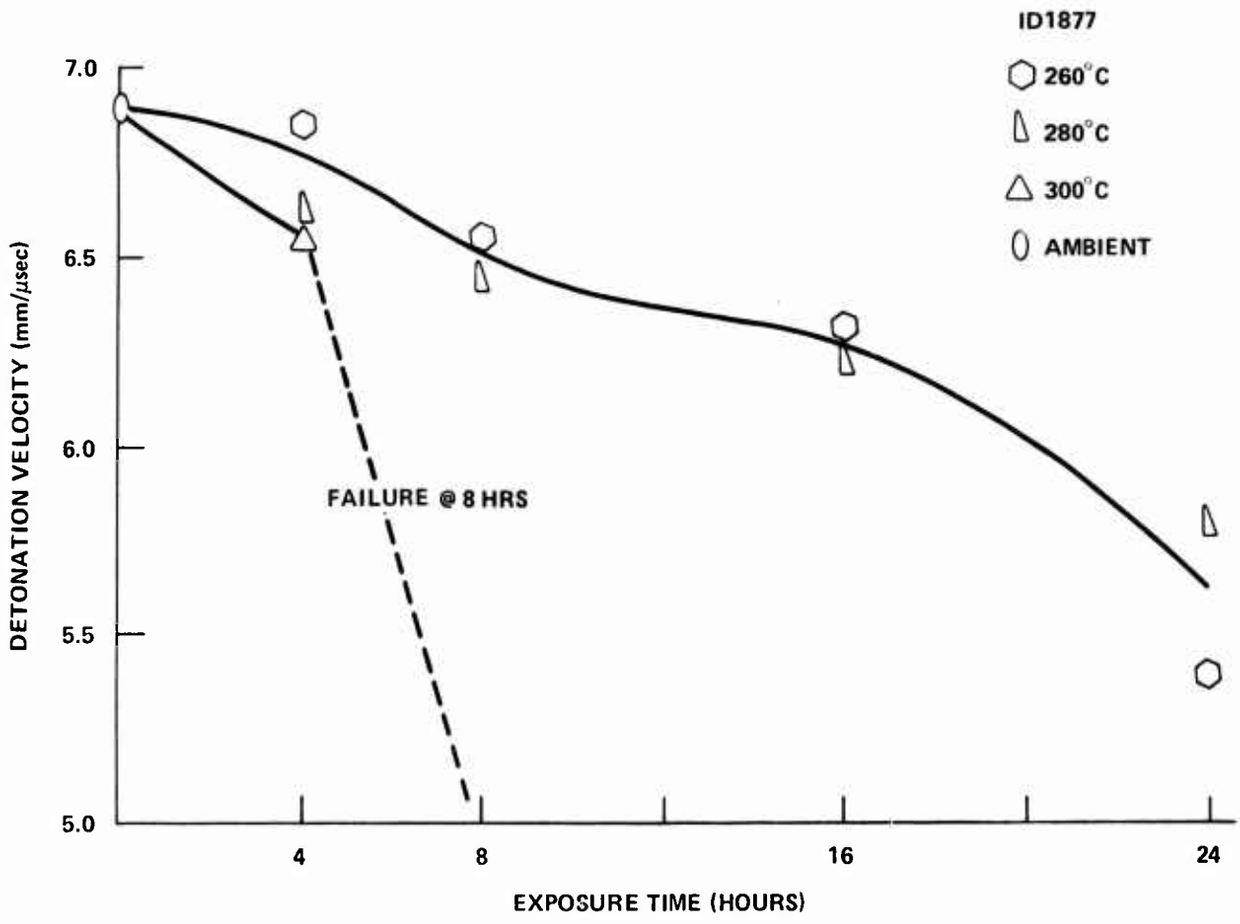


FIG. 28 THE DETONATION VELOCITY OF PYX IN SILVER MDC (2-1/2gr/ft) AFTER EXPOSURE TO ELEVATED TEMPERATURES

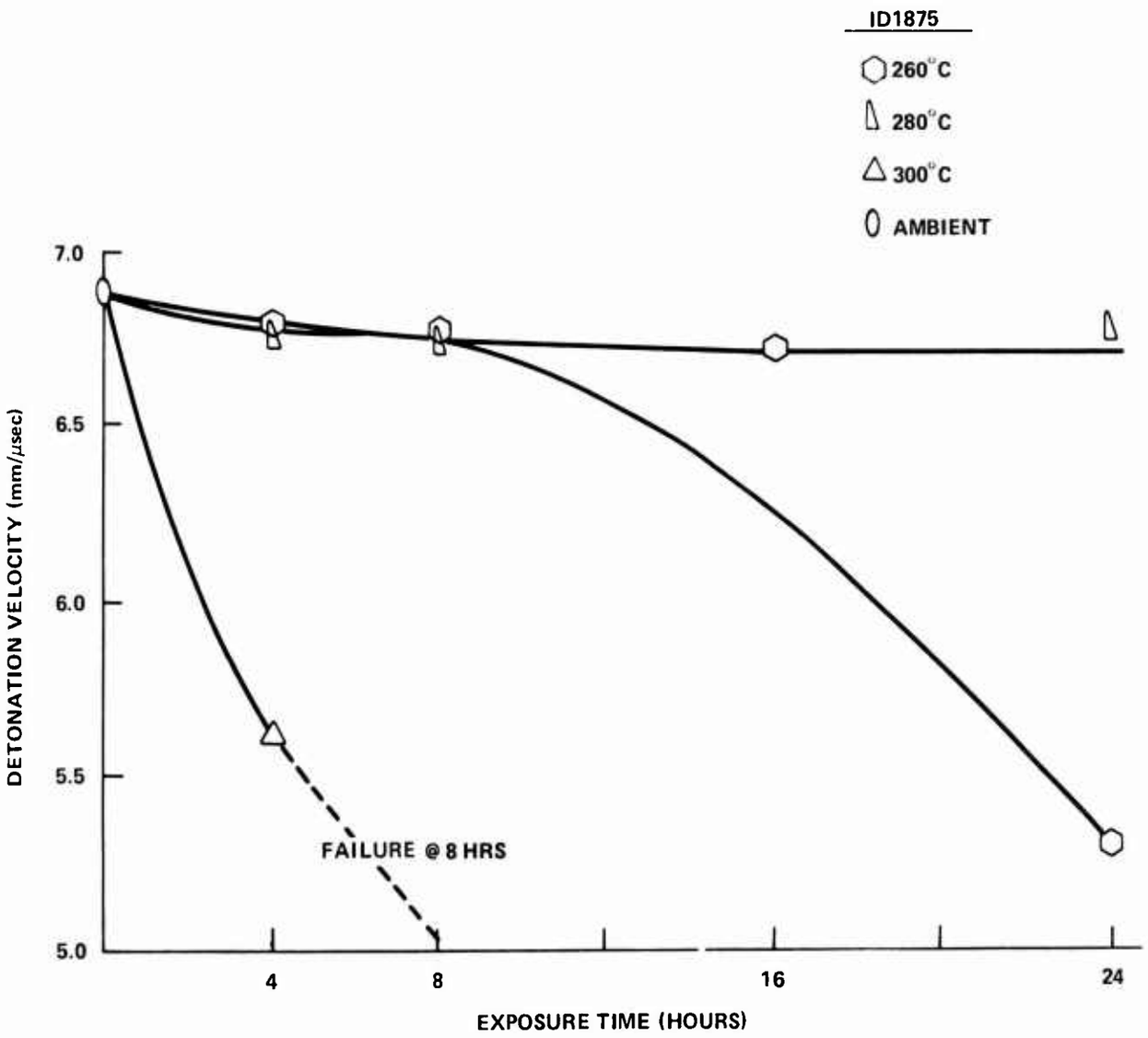


FIG. 29 THE DETONATION VELOCITY OF PYX IN ALUMINUM MDC (2-1/2gr/ft) AFTER EXPOSURE TO ELEVATED TEMPERATURES

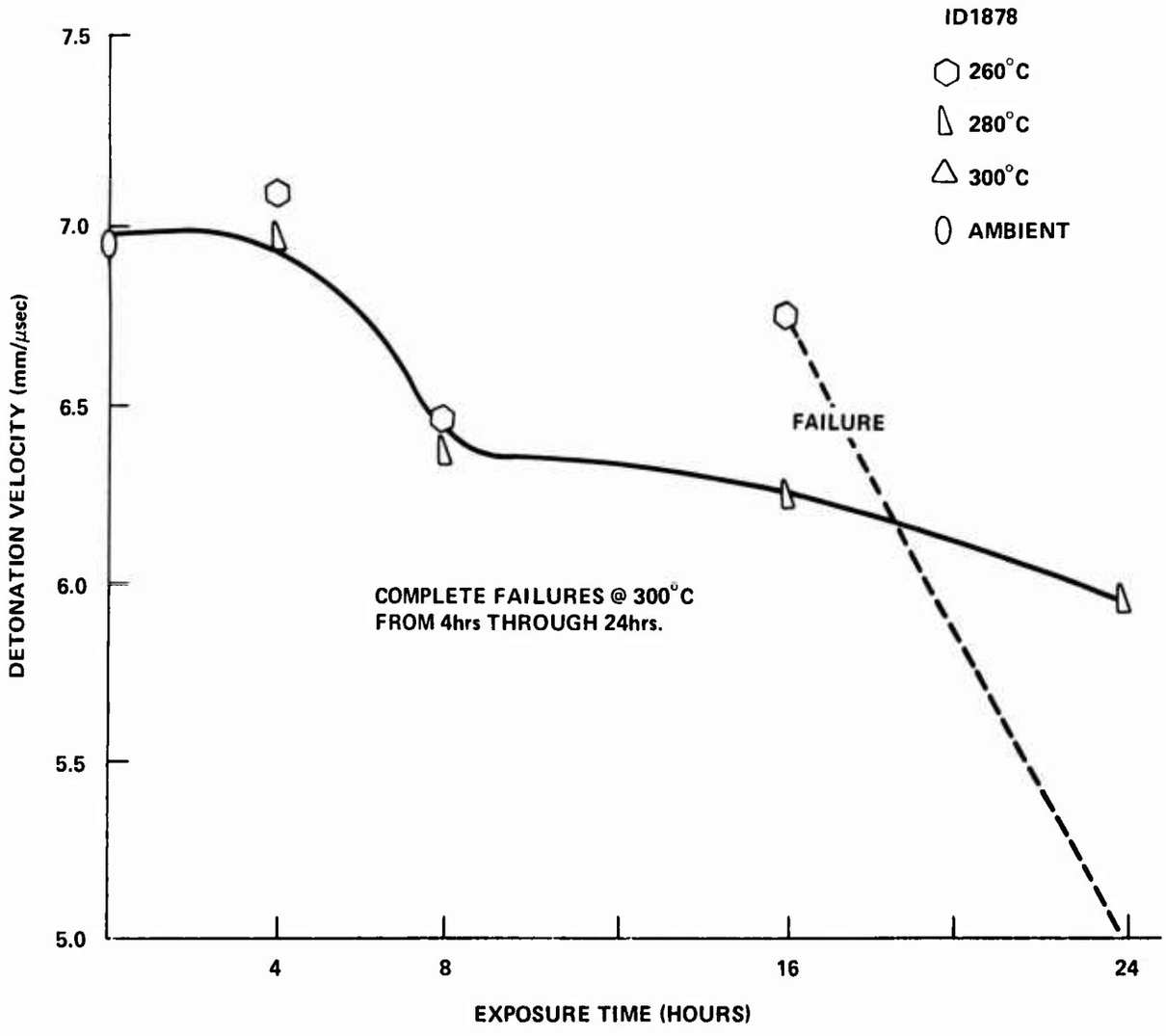


FIG. 30 THE DETONATION VELOCITY OF PYX IN SILVER MDC (10gr/ft) AFTER EXPOSURE TO ELEVATED TEMPERATURES

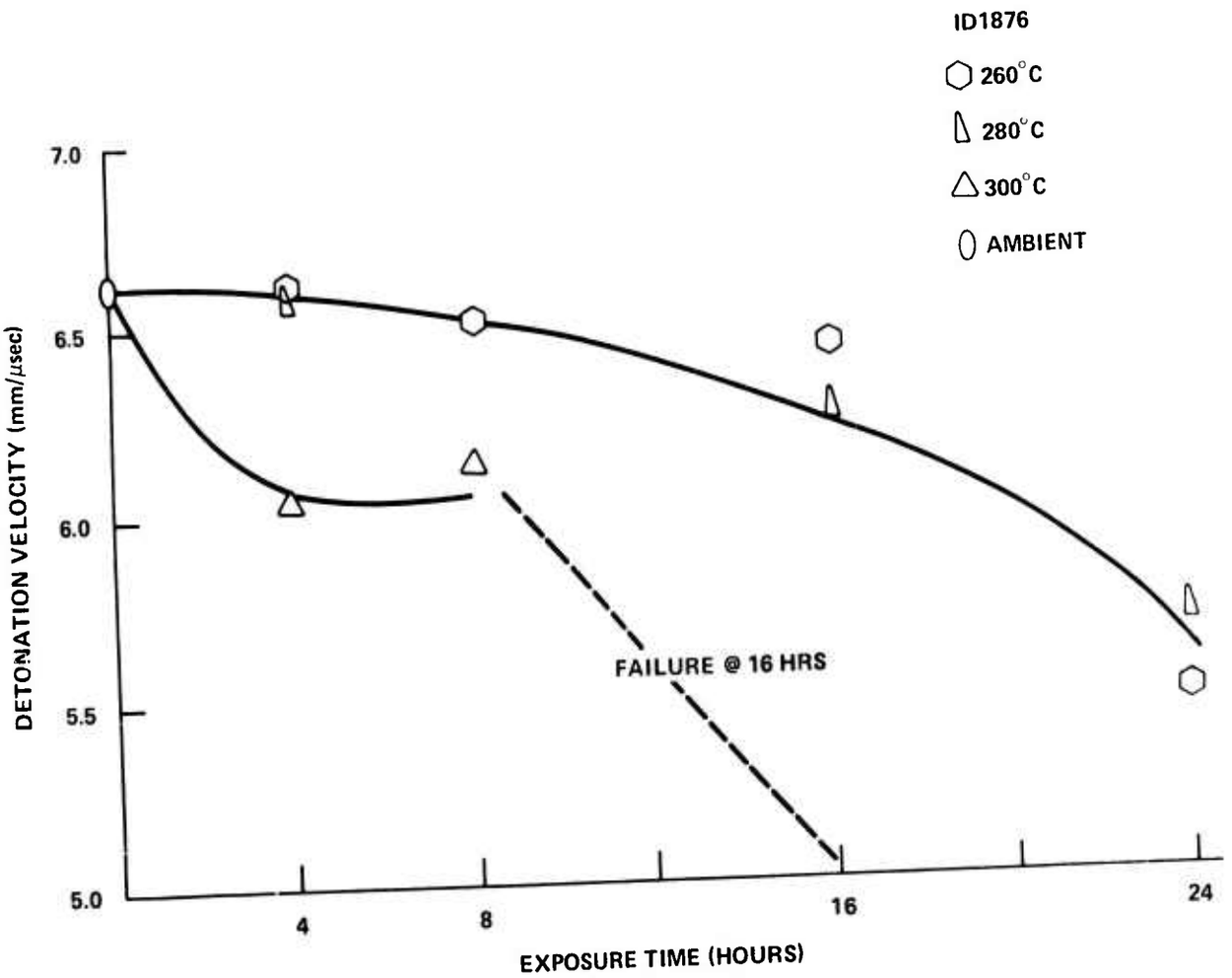


FIG. 31 THE DETONATION VELOCITY OF PYX IN ALUMINUM MDC (10gr/ft) AFTER EXPOSURE TO ELEVATED TEMPERATURES

APPENDIX A

A. Chemical Synthesis of 2,4,6-Tripicryl-s-Triazine, TPT

A-1 A study of the chemical synthesis[8] of the thermally stable explosive, tripicryl triazine (TPT), has been made at the NAVSURFWPNCEN by Dr. J. Dacons. The synthesis is documented in the above reference and also by a United States Patent[5]. The original material shown in Figure A.1 was prepared in small quantities by the Ullmann coupling reaction using cyanuric chloride in nitrobenzene, powdered copper, and the addition of picryl bromide. This reaction was scaled-up to yield batches of about 150g of finished product, which is three times the batch size of the original preparation. This larger batch size operation was used to prepare six pounds of TPT. Each batch was checked for chemical purity and melting point. This lot of explosive was used to set up the basic requirements for chemical purity and sensitivity for the procurement specification and for the fabrication of detonating cords.

A-2 The chemical synthesis of TPT is described below (reference 7):

Preparation of Picryl Bromide. An amount of 355g (3.5 moles) of reagent grade potassium nitrate was dissolved in 800 ml of 30% oleum in a 2000 ml 3-neck round bottom flask fitted with a mechanical stirrer and a thermometer. During the addition of the potassium nitrate, the mixture was cooled and stirred on an ice bath, the temperature being kept below 60°C. When the addition was complete, the mixture was cooled to 30°C and 79g (0.5 moles) of bromobenzene was added at such a rate as to keep the temperature below 50°C. The ice bath was then replaced by an oil bath and the reaction mixture was heated to approximately 125°C for four hours. After cooling, the mixture was drowned in crushed ice, the product was collected by filtration (using a sintered glass funnel) and thoroughly washed with water. After drying in a warm oven, it was dissolved in 100 ml of acetone. 300 ml of methanol was added and the solution was cooled in the freeze compartment of the refrigerator. On filtration, 83.7g of pale yellow crystalline picryl bromide, mp 120-122°C (lit. 122-3°C) was recovered. The filtrate was concentrated to about 150 ml by boiling on the steam bath and again cooled in the freeze compartment. The second crop of precipitate weighed 28.0g and melted over the range 111-118°C. On recrystallization from acetone-ethanol, an additional 21.0g of crystalline product, mp 120-122°C was obtained to give a total yield melting at this temperature of 104.7g, 71.6%. Samples melting at 122-123°C were obtained by one additional recrystallization from acetone-methanol; however, the product melting at 120-122°C was of sufficient purity for use for synthesizing TPT.

(8) Dacons, J. C. and Sitzmann, M. E., "Heat Resistant Explosives XXVI. The Synthesis and Properties of 2,4,6-tripicryl-s-triazine, TPT," NOLTR 68-64, 29 May 1968

100 X ID 1103

250 X ID 1103

500 X ID 1103

400 X ID 1103

FIG. A-1 SEM PHOTOMICROGRAPHS OF TPT (ID 1103) NSWC ORIGINAL PREP.

A-3 Preparation of 2,4,6-Tripicryl-s-triazine, TPT. A solution of 18.5g (0.1 moles) of cyanuric chloride in 100 ml of dry nitrobenzene was made in a 500 ml 3-neck round bottom flask fitted with a mechanical stirrer, an addition funnel, an air cooled condenser and a thermometer. To this solution was added 89.0g (1.4 moles) of copper powder* and, with stirring, the temperature was raised to 140°C on an oil bath held in place by a laboratory jack. A volume of 20 ml of a solution of 188g (0.4 moles) of picryl bromide in 150 ml of dry nitrobenzene was added rapidly. The temperature dropped to 134°C and there was an immediate reaction indicated by a change in the color of the copper and a rise in the temperature of the mixture to 146°C. Heating was reduced by lowering the oil bath and dropwise addition of the remainder of the picryl bromide solution was started. The rate of addition was adjusted so as to require approximately 40 minutes for its completion. A temperature range of 140-146°C was maintained during the addition and for 20 minutes thereafter. The reaction mixture was allowed to cool to ambient temperature and then poured into 700 ml of methanol and cooled to 5°C. It was then filtered with suction through a glass fiber filter and the residue was thoroughly washed with cold methanol. After drying in a warm oven, the residue containing the TPT along with cuprous halides and unused copper, was digested in 1000 ml of approximately 20% hydrochloric acid with stirring for about 20 minutes, then filtered through sintered glass. The residue, which was mainly TPT and copper, was washed first with dilute hydrochloric acid, then with water and finally with methanol until the washings were nearly colorless. After drying on the funnel for a few minutes, the filter cake was heated in three liters of acetone to dissolve the TPT, treated with 25 g of Darco G 60 and filtered. The amber filtrate was concentrated by rapid distillation with stirring until considerable crystallization had occurred and the volume was about 400 ml. Methanol was then added at about the same rate as the distillate was removed until the temperature of the distillate was 62°C. The mixture was then cooled to 5°C on an ice bath, the product was removed by filtration, washed with cold methanol and dried in a warm oven. The yield was 60.7 g of a fine, very pale yellow crystalline solid which melted at 350-1°C (d) after sintering at approximately 140°C.

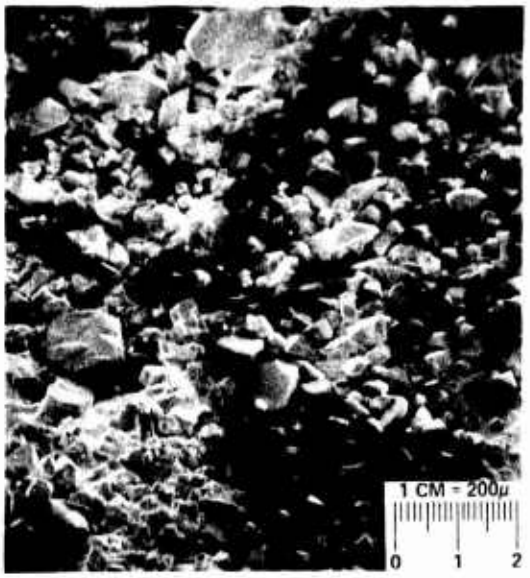
A-4 In order to obtain high purity TPT, the above material was dissolved in 2000 ml of acetone and recrystallized as described above. On drying in a warm oven overnight, the product was 55.5g of glistening pale yellow crystals, mp 352-3°C (d) after sintering at 140°C. As pointed out by Dacons[9], the physical combination of solute and solvent to form the solvate resulted in crystals "of a 1:1 with acetone." On heating to 140°C, the yellow crystals loose their solvent of crystallization, becoming opaque and white, but retain essentially the same shape and size.

*Purified Copper Powder from Matheson, Coleman and Bell and Venus Natural Copper Fine No. 44-F, U. S. Bronze Powders, Inc., Flemington, New Jersey have been found suitable for this reaction.

(9) Dacons, J., "2,4,6-Tripicryl-s-Triazine, TPT: It's Crystallization and Crystal Density Determination," NSWC/WOL/TR 76-16, 8 Mar 1976, Unclassified

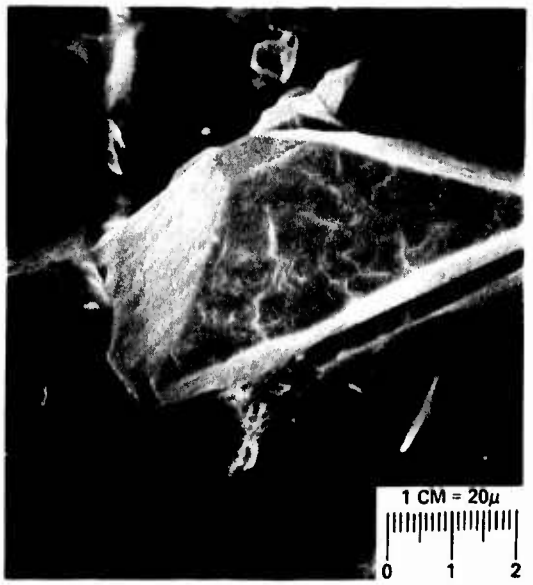
A-5 In order to determine the nature of the solvate, a twice recrystallized sample, which had been dried at 80°C was heated at 280°C for thirty minutes and the gas evolved was vacuum transferred to a gas chromatograph. The retention time identified the gas as acetone. Samples which had been dried overnight at 140°C showed no acetone content. By measuring the weight loss on heating at 140°C for extended periods, it was found that the solvate contained 7.5% acetone and 92.5% TPT corresponding to one mole of acetone per mole of TPT. (See Figure A.2).

A-6 A crystal density determination was made of the acetone solvate and was found[9] to be 1.64g/cc as shown in Figure A.3. This value is low because of the nature of the recrystallization. The material was recrystallized from dichlorobenzene to improve the crystal density to 1.72g/cc which is the best value for the unsolvated crystal (Figure A.4).



50 X ID 1888

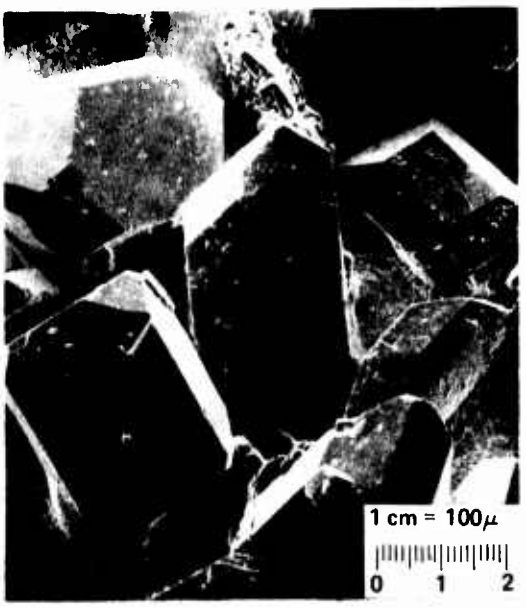
100 X ID 1888



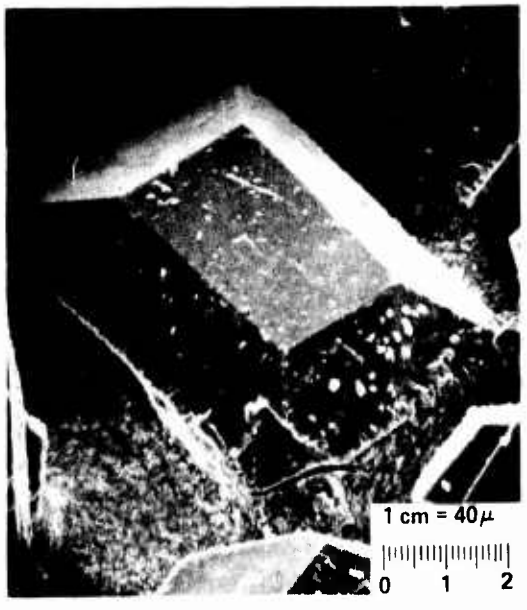
500 X ID 1888

FIG. A-2 SEM PHOTOMICROGRAPHS OF TPT (ID1888) PREPARED AT NSWC FOR SPECIFICATION TESTING

30 X ID 2063



100 X ID 2063

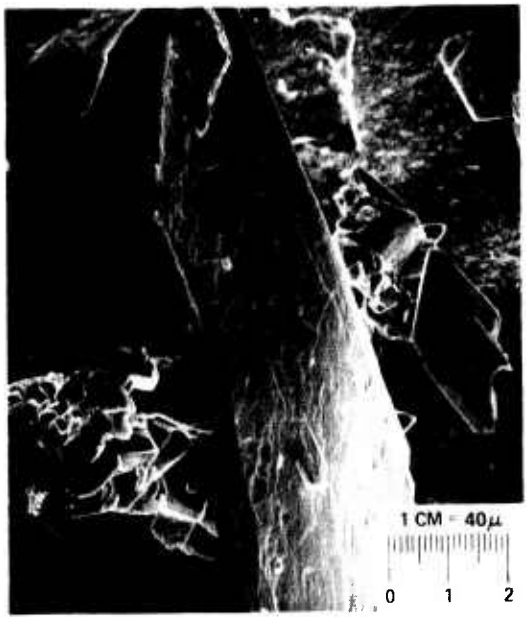


250 X ID 2063

FIG. A-3 SEM PHOTOMICROGRAPHS OF TPT (ID2063) SOLVATED CRYSTAL ρ=1.64g/cc

30 X ID 2065

100 X ID 2065



250 X ID 2065

FIG. A-4 SEM PHOTOMICROGRAPHS OF TPT (ID2065) UNSOLVATED CRYSTAL ρ=1.72g/cc

APPENDIX B

B. The Dynamic Compressibility of TPT

B-1 It is desirable in the characterization of any high explosive to measure accurately its shock sensitivity. The impact sensitivity test and the small scale gap test do not give refined values of shock pressure required to determine when the explosive will be initiated to deflagration or detonation. One measure of the sensitivity of an explosive to shock is the pressure needed to cause a chemical reaction. This information is available from measurements of the dynamic compressibility of the unreacted explosive. The pressure needed to cause reaction is important in designing shock initiated explosive components and in providing protection against accidental explosions.

B-2 The dynamic pressure-volume curve (shock Hugoniot) of TPT was determined from experiments which measured shock wave velocities in TPT samples and the shock properties of calibrated driver plate materials. Four pressure-volume points and two sound velocity points were measured in the TPT samples*.

B-3 The experimental arrangement used to determine the shock Hugoniot is shown in Figure B.1. A plane wave explosive lens shocks a driver plate on which the explosive samples are attached. The shock wave arrivals are recorded by a smear camera using a reflected light technique[10,11]. In this experimental technique, light is reflected continuously from the free surfaces into the camera. The shock wave arrival at any point along the reflective surfaces produces a sudden change in light reflected from that point. These changes in intensity are recorded on the camera film as a function of time. Measurements on the film give the time required for the shock wave to travel through the test samples. This data along with the thicknesses of the test samples allows the velocity of the shock wave to be calculated. The free-surface velocity of the driver plate material also is measured using the reflected-light technique.

*(A small amount (5%) of TEFLON was added to allow the TPT to be pelletized.)

(10) Coleburn, N. L., J. Chem. Phys., 40, 71, (1964)
(11) duVall, G. F. and Fowles, G. R., "Shock Waves," High Pressure Physics, Vol. 2, pp 209, Academic Press (1963)

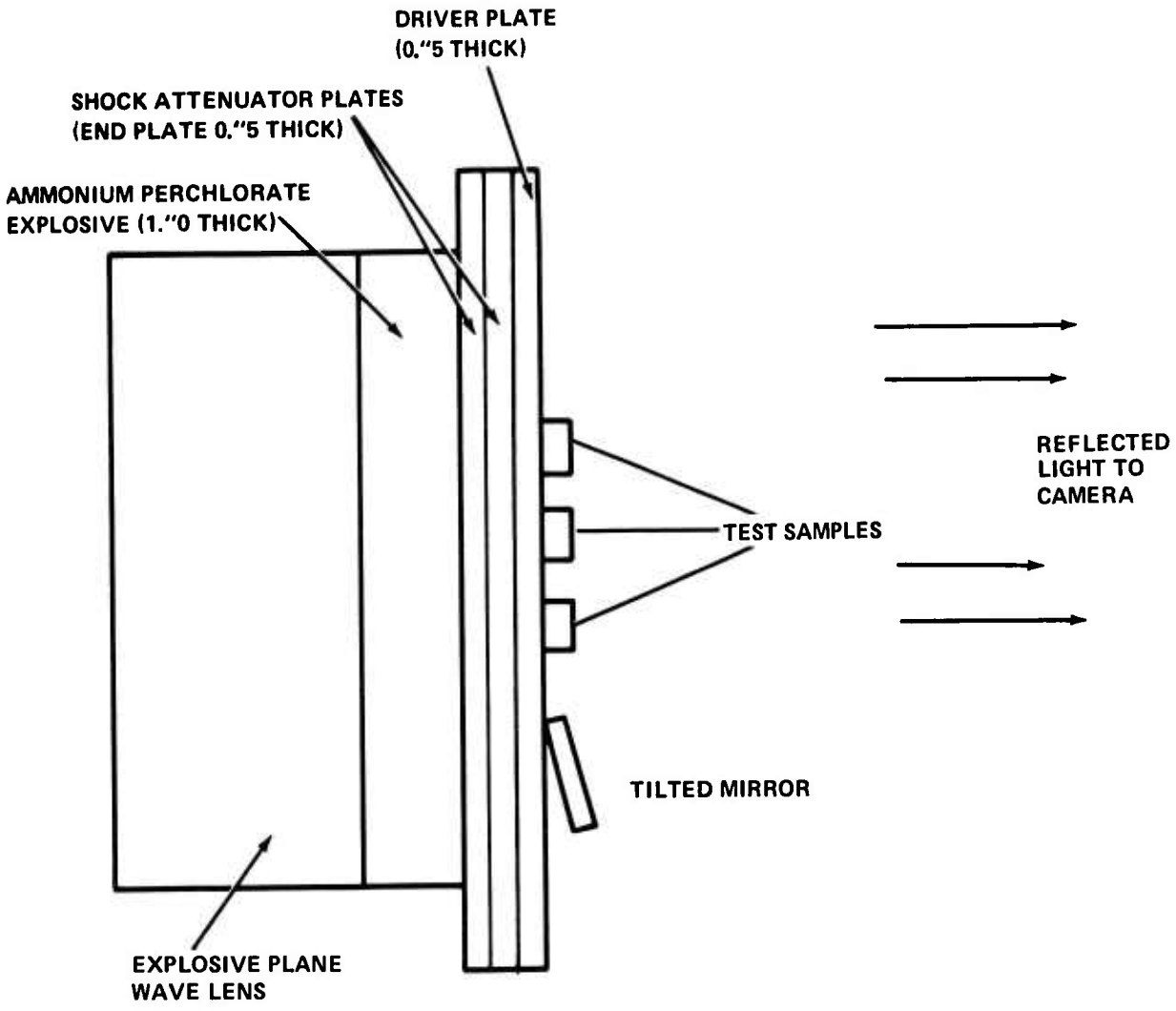


FIG. B-1 EXPERIMENTAL SETUP (SIDE VIEW)

B-4 The measured shock wave parameters were used in the conservation laws to obtain the pressure, volume, and particle velocity in the shocked materials. The conservation laws[11] are:

$$P = \rho_o U_s u_p \qquad (1)$$

$$\frac{V_1}{V_0} = \frac{U_s - u_p}{U_s} \qquad (2)$$

where P is the pressure behind the shock front, ρ_o is the initial density, U_s is the shock velocity, and u_p is the particle velocity behind the shock front (u_p = 1/2 the free-surface velocity). (In using these conservation laws, the assumptions of steady flow and no chemical reaction were made.)

B-5 It is obvious from equation (1) that the measurement of shock velocity and particle velocity are sufficient for determining the pressure in the driver plate. This pressure and the measured shock wave velocity in TPT was used in an impedance-matching data reduction technique[12] to determine the equation of state parameters (i.e., P, V, u_p).

B-6 The experimental results of pressure, shock velocity, and particle velocity obtained so far for TPT are summarized in Table B-1. The shock velocity-particle velocity data is represented by a straight line.

$$U_s = 1.35 \text{ mm/microsec} + 2.56\, u_p \text{ mm/microsec.} \qquad (3)$$

When chemical reaction occurs, a large deviation from linearity in a plot of the data will result. The data indicate that no significant reaction occurs at pressures up to 38 kilobars. Pressure necessary for reaction to begin will be established by additional experiments.

B-7 A bulk sound velocity of 1.34 mm/microsec was estimated in TPT using a weak shock technique[13,14] to measure the velocities of longitudinal elastic waves in TPT at pressures of about 100 bars. (The density of the test samples was 1.5 g/cm^3). This value of the bulk sound speed is in excellent agreement with the intercept (u_p = 0) of the U_s, u_p curve as it should be (in accordance with shock theory).

(12) Duvall, G. E., "Shock Waves in Condensed Media," Physics of High Energy Density, pp 41, Academic Press (1971)
(13) Coleburn, N. L., and Liddiard, T. P. Jr., J. Chem. Phys., 44, 1929, (1966)
(14) Coleburn, N. L., J. Acoust. Soc., 47, 269, (1970)

TABLE B-1
EXPERIMENTAL RESULTS OF SHOCK COMPRESSIBILITY TESTS

EXPERIMENT NUMBER	SHOCK ATTENUATOR PLATES*	DRIVER PLATE**	PRESSURE IN DRIVER PLATE (KBAR)	TPT			
				INITIAL DENSITY (GM/CM^3)	SHOCK VELOCITY (mm/μ sec)	PARTICLE VELOCITY (mm/μ sec)	PRESSURE (KBAR)
1	ALUMINUM/ PLEXIGLAS	ALUMINUM	83	1.57	3.21	0.75	38
2	BRASS/ POLYETHYLENE	ALUMINUM	73	1.52	3.15	0.68	32
3	BRASS/ POLYETHYLENE	BRASS	82	1.58	2.40	0.42	16
4	BRASS/ PLEXIGLAS	BRASS	80	1.61	2.30	0.38	14

*SHOCK SYSTEM WERE 6.25–INCH DIAMETER PLANE WAVE BOOSTERS WITH 1.0–INCH THICK AMMONIUM PERCHLORATE POD AND TWO 0.5–INCH THICK SHOCK ATTENUATOR PLATES.

**DRIVER PLATES WERE 0.5–INCHES THICK.

B-8 The dynamic pressure-volume curve for TPT is compared with the curve for TNT[14] in Figure B.2. Note that at any specific pressure TPT has a smaller volume ratio, V/V_o than TNT. Therefore, TPT is more compressible than TNT.

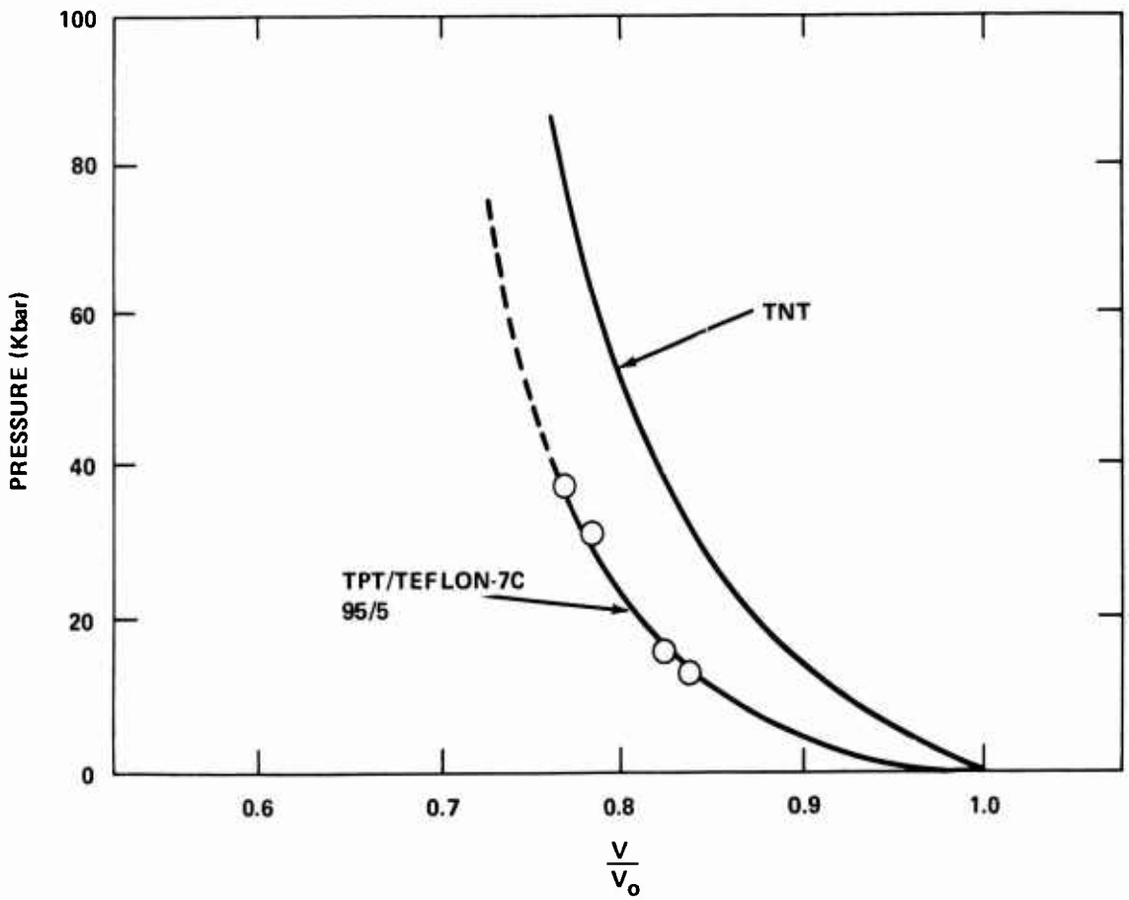


FIG. B-2 SHOCK COMPRESSIBILITY CURVE FOR UNREACTED TPT AND TNT

APPENDIX C

C. Determination of Heat of Formation of TPT

C-1 The heat of formation of TPT was determined by Baroody and Carpenter[15] of the Indian Head Facility of the Naval Surface Weapons Center. A mixture of the explosive and diethyl oxalate (DEO) was used for the combustion process. The results, along with the crystal density of TPT, can be used to obtain estimates of detonation velocity and pressure and heat of detonation.

C-2 The enthalpy of formation of 2,4,6-tripicryltriazine (TPT) was determined by combustion calorimetry, using a platinum-lined rotating-bomb calorimeter. A mixture of TPT and diethyloxalate (DEO) was used to desensitize and ensure complete combustion of TPT. The enthalpy of formation (298.15K) was determined from the following idealized equation at one atmosphere:

$$C_{21}H_6O_{18}N_{12}(c) + 13.5\ O_2(g) \rightarrow 21\ CO_2\ (g) + 3H_2O\ (l) + 6N_2\ (g)$$

C-3 Several trial runs were conducted on TPT with several solvents in a Parr stationary adiabatic-bomb calorimeter to determine the heat auxiliary material to use with TPT. Also, the proper weight ratios to approximate the heat released for initial temperature settings on the rotating bomb, and to test for ignitability and good combustion of TPT mixtures.

C-4 The enthalpy of formation of TPT was determined to be:

$$\Delta H_f^{\circ} = 7.56 + 0.99 \text{ k cal/mol}$$

(15) Baroody, E. E., J. Chem Eng. Data 21, (1976)

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