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NSWC TECHNICAL REPORT

6

WHITE OAK LABORATORY

DETONATING CORDS LOADED WITH HEXANITROSTILBENE (HNS) RECRYSTALLIZED FROM ACID OR ORGANIC SOLVENTS

By
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2 September 1975

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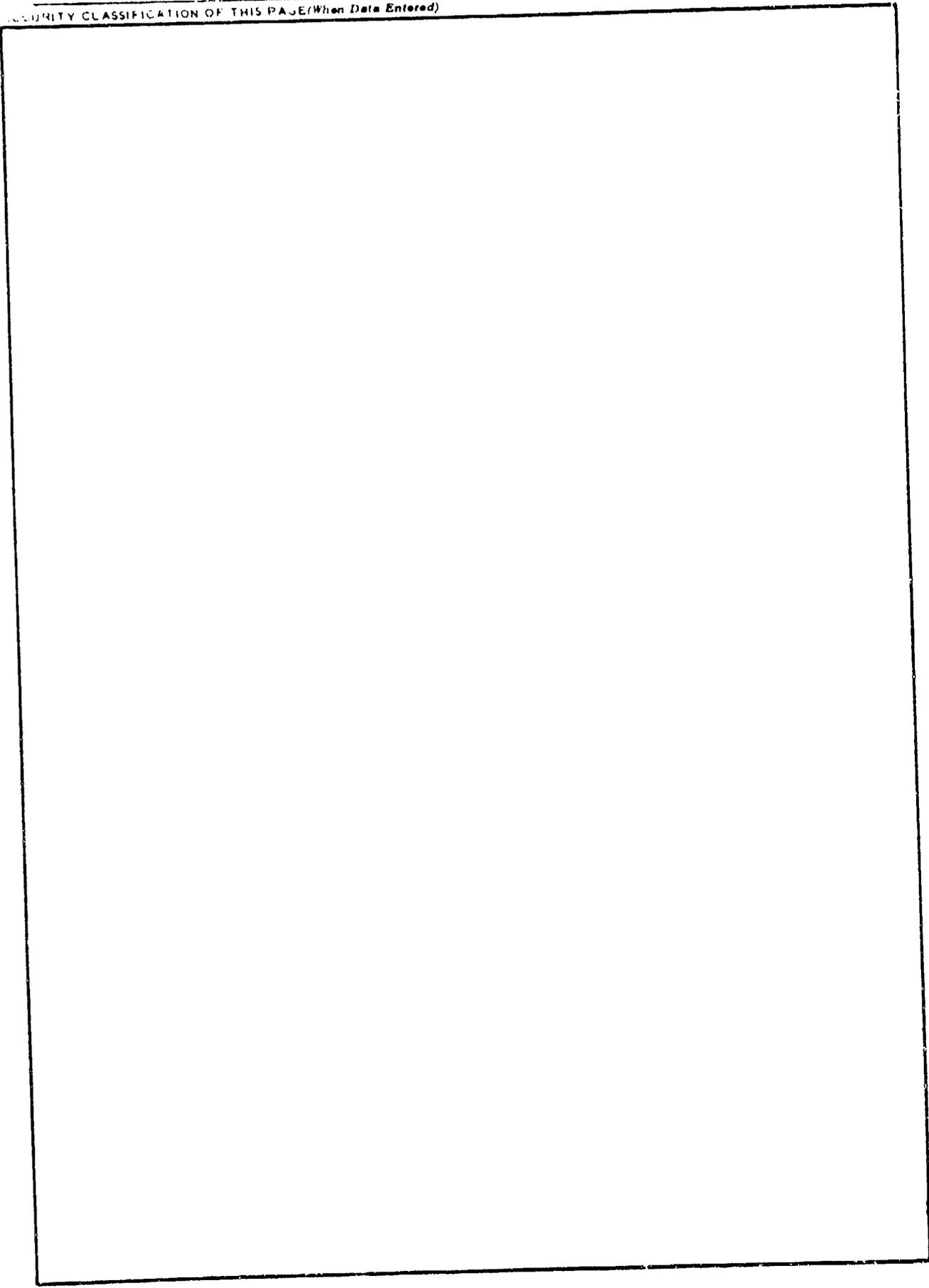
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1.0 Introduction/History

1.1 Detonating cords are flexible metal, plastic, or fabric tubes loaded with explosives. They have long been used in the blasting industry. For many years these cords were loaded with PETN and RDX. Historically, the first detonating cord was called cordeau détonant. Cordeau was fabricated by filling a lead tube with TNT and then drawing the tube down to wire size using wire drawing equipment. PETN was suggested for detonating cords about 1913. Primacord loaded with PETN has a detonation velocity of approximately 6400 m/sec. When the cords have an outside diameter below 1/2 inch and are flexible they are known as detonating fuse.

Mild detonating fuse (MDF- also called mild detonating cord or low energy detonating cord) was a development of the Ensign-Bickford Co. It is used primarily to conduct detonation from one point to another with little, if any, disruption of the surrounding medium.

Originally, MDF was loaded with PETN or RDX. However, for many military applications the explosives have been changed to meet military safety requirements and to meet special environmental requirements, particularly those of high temperatures and low pressures. The sheathing materials have also been altered; either to meet these same requirements or to be more compatible with the explosive at high temperature.

Nonetheless, in some military applications RDX has been used in a lead sheath. Several years ago failures to propagate detonation in RDX-loaded MDF was discovered(1). The ensuing investigation revealed the problem as insensitivity because of crystal growth in the RDX at a critical section of the cord.

1.2 At about the same time, the popularity of a relatively new explosive Hexanitrostilbene (HNS)(2) was increasing. This explosive meets the military requirements for safety and has superior thermal stability. It had already been loaded into MDF and accepted for use

(1) Kilmer, E., "ZAP Rocket Motor Ignition System-II," NOLTR 72-7, 16 May 1972

(2) Shipp, K. G., "Reactions of - Substituted Polynitrotoluenes, I. Synthesis of 2,2',4,4',6,6' - Hexanitrostilbene," J. Org. Chem. 29, 2620 (1964).

in spacecraft, supersonic aircraft, and weapons⁽³⁾. Two basic forms of HNS had been synthesized by NAVSURFWPNCEN (formerly NOL): HNS-I, prepared by the method of ref (2) and HNS-II, a recrystallized HNS-I (Ref. 4). Both of these materials were covered by specification WS 5003. HNS-I is a very small particle size material suitable for use in the end boosters of detonating cords (Ref. 5). HNS-II is much larger, less fluffy, and is more suitable for bulk loading such as used for the core of MDF. In addition to many White Oak Laboratory reports on the applications of these materials, a summary report on work done by Schwarz of Sandia Laboratories⁽⁶⁾ has also been written.

1.3 The original HNS preparations were made at the White Oak Laboratory. Later, Dr. Fred Hudson of the American Cyanamid Company synthesized some of the first commercially made material. Tables 1 and 2 give a history of industrial preparations. The first production quantities of HNS-I came from the Amcel Corp (1964) and Northrop-Carolina (1965) using a modified Shipp process. The largest quantities are now being made by Chemtronics. The process for the preparation of HNS-II was changed in 1973 from the Hudson Process to the "Nitric Acid" Process where acid solvents were substituted for the organic solvents in the preparation of the explosive.

1.4 Except for Northrop-Carolina, there were no other companies preparing HNS-I or II until 1969 when Del Mar Engineering began to produce the material under Mr. Leroy Syrop, who deviated from the Shipp process by recrystallizing HNS-II in the presence of Xylene⁽⁷⁾ instead of toluene. This explosive was used in the NASA APOLLO Program as well as in many military applications. Much of this material is still being used in the F-14 and B-1 airplanes. However, Del Mar Engineering is no longer in the business of synthesizing HNS. The Del Mar process was taken over by Teledyne/McCormick Selph who now furnishes HNS by either an organic solvent process or by a proprietary "Acid Process". In 1970 the Ensign-Bickford Company started to synthesize HNS-II by the Osterling-Taylor method.

1.5 To summarize: it appears that HNS-I and HNS-II were made by the Shipp Process with some deviation in organic solvent up until 1973. Since that time HNS-II has been recrystallized from both organic and inorganic solvents. More specific information on the history of the

(3) Kilmer, E., "Development of a High Temperature Resistant Mild Detonating Fuse-V," NOLTR 65-170, 10 Jun 1966.

(4) Taylor, Francis Jr., and Osterling, R. E., "Heat Resistant Explosives XX Production of Grade II HNS," NOLTR 65-142, 26 Aug 1965.

(5) Kilmer, E., "End Booster for Heat Resistant Mild Detonating Fuse," NOLTR 65-98, 6 Apr 1966.

(6) Schwarz, A. C., "Application of Hexanitrostilbene in Explosive Components," SC-RR-71 0673, May 1972, Sandia Laboratories, Albuquerque, N.M.

(7) Syrop, L. J., "Process for Recrystallizing Hexanitrostilbene," U.S. Patent No. 3,699,176, 17 Oct 1972.

TABLE 1
HISTORY OF INDUSTRIAL PREPARATION OF HNS-I

COMPANY	YEARS OF MANUFACTURE	PROCESS	USED ON
AMERICAN CYANAMID, AMCEL, NORTHROP, CHEMTRONICS	1964 TO PRESENT	HUDSON PROCESS (INDUSTRIAL PROPRIETARY)	F-111, F-14, F-15, B-1, APOLLO SYSTEMS ETC.
DEL MAR ENGINEERING	1969 TO 1972	SHIPP PROCESS (REF 2)	F-111, F-14, F-15, B-1, APOLLO SYSTEMS
TELEDYNE/McCORMICK SELPH	1973 TO PRESENT	SHIPP PROCESS (REF 2)	NOT KNOWN

TABLE 2
HISTORY OF INDUSTRIAL PREPARATION OF HNS-II

COMPANY	YEARS OF MANUFACTURE	PROCESS	USED ON
AMERICAN CYANAMID AMCEL, NORTHROP, CHEMTRONICS	1964 TO PRESENT	HUDSON PROCESS (INDUSTRIAL PROPRIETARY)	F-111, F-14, F-15, B-1, APOLLO SYSTEMS ETC.
DEL MAR ENGINEERING	1969 TO 1972	SYROP PROCESS (REF 7)	F-111, F-14, F-15, B-1, APOLLO SYSTEMS
TELEDYNE/McCORMICK SELPH	1972 TO PRESENT	SYROP PROCESS (REF 7) AND ACID PROCESS (INDUSTRIAL PROPRIETARY)	NOT KNOWN
ENSIGN BICKFORD	1970 TO PRESENT	OESTERLING-TAYLOR PROCESS (REF 4)	NOT KNOWN

explosive preparations can be found in Appendices, A, B, and C.

2.0 Scope of Problem Study

2.1 During 1973, detonation failures began to occur in mild detonating cords fabricated by Space Ordnance Systems for the Grumman F-14 airplane. McCormick Selph "acid process" HNS-II was used in these cords. Prior to the 1973 failure, HNS and another high temperature resistant detonating cord material, DIPAM, had shown a very high degree of reliability. See Tables 3 and 4. Because of this high demonstrated reliability, HNS was being considered for detonating cords in the TRIDENT C-4. However, it was believed worthwhile, before its adoption in this important strategic system, that more should be learned about the cause of the problem in the F-14 hardware. Therefore, the study reported here was undertaken. The purposes of the study were to determine:

- (a) If the particular lot of explosive was causing poor performance in the detonating cords.
- (b) If the method of fabrication of the cord was causing the poor performance.
- (c) If the Grumman specification for thermal cycling was at fault.
- (d) How particle size and geometry has varied since the first Hexanitrostilbene was synthesized, and whether or not the variation is related to the performance problem.

3.0 Identification of Explosives/Detonating Cords

3.1 To assist in easy identification of the cords to be discussed herein, Table 5 has been prepared. The SOS lot of cords (NSWC ID 1847*) which exhibited the "in-line" propagation failures was loaded with McCormick Selph HNS Lot #8203-2. A sample of this detonating cord was procured from SOS by NAVSURFWPNCEN. This was a silver sheathed material (core load about 2-1/2 gr/ft) with an outside diameter of 0.070 (SOS stock no. 30863).

3.2 A sample of McCormick Selph HNS Lot #8203-2 was also sent to Explosive Technology (ET) for loading and fabrication into 2-1/2 gr/ft (0.075 OD) silver sheathed detonating cord. This cord is identified as ID 1850. In addition, ET was asked to fabricate, using the same lot of explosive, a silver sheathed cord having a lower density and detonation velocity (6000 m/sec) than is normal (7200 m/sec) but having the same 2-1/2 gr/ft core load and the same 0.075 OD. This cord was designated ID 1995.

* For purposes of identification, an internal laboratory number is assigned to all cords and bulk explosive.

** "In-line" propagation failure is defined as a detonation stoppage in a properly initiated detonating cord.

3.3 Explosive synthesized from organic solvents (Northrop-Carolina Lot 8995-49) was fabricated into silver sheathed 3-1/2 gr/ft (0"075 OD) detonating cord by Explosive Technology and was identified as ID 841. Since McCormick Selph was not the only source of "acid precipitated" material, a sample of Chemtronics Lot 66-16, HNS-II was sent to Explosive Technology for fabrication into 2-1/2 gr/ft (0"082 OD) aluminum sheathed cord. It is identified as ID 1845.

3.4 The Chemtronics Lot 66-16 material was also investigated at a higher density by isostatically pressing ID 1845 cords to 30 Kpsi. The pressed cords were identified as ID 1968 (0"082 OD). The organic solvent precipitated explosive, Northrop-Carolina Lot 11138-3, was fabricated into an aluminum sheathed cord by Explosive Technology in 1966. This 2 gr/ft cord, diameter 0"042, is identified as ID 892. A lower velocity, 2-1/2 gr/ft aluminum sheathed, 0"075 OD detonating cord was fabricated by Explosive Technology in 1969 with Northrop-Carolina Lot 43-13 Organic Solvent Precip. HNS. This cord has the identification of ID 1018.

4.0 Environmental Temperature Cycling of Detonating Cords

4.1 "In-line" failures are uncommon in detonating cords. To determine the effect of temperature on failure probability, the White Oak Laboratory performed elevated temperature experiments using the Grumman F-14 Specification⁽⁸⁾ as a guide for the temperature cycling tests. The specification in part includes:

Temperature Cycling Test (8)

"The cords and manifolds shall be stabilized at -65°F and maintained for a period of one hour. The temperature shall then be raised to +350°F and maintained for a period of 55 minutes, the temperature shall then be increased to +425°F and maintained for a period of 5 minutes. The cords and manifolds shall then be stabilized at -65°F for the start of the next cycle. The maximum transition time between temperatures shall be 5 minutes. The change in temperature from -65°F to +350°F to +425°F shall constitute one cycle of a two-hour duration. The cords and manifolds shall be subjected to a minimum of 100 cycles."

4.2 It was expected that a repeat of the cycling tests mentioned earlier, with the sealed copper tubing simulating the SMDC enclosure (see Figure 1), would show the same failure as was found by SOS. For the first 100 cycle test, detonating cords fabricated by Space Ordnance Systems (ID 1847) and Explosive Technology (ID 1850) from the same lot of explosive (McCormick-Selph Lot 8203-2) were cut into 4-inch lengths and sealed.

(8) Specification No. A51DCVBA020, Grumman Aircraft Engineering Corporation, "Expanding Shielded Mild Detonating Cord Assembly Design Control Specification for F-14A Weapon System," 11 Dec 1970.

TABLE 3
PROGRAMS USING SMDC MANUFACTURED BY EXPLOSIVE TECHNOLOGY.
(EXPLOSIVE TECHNOLOGY)

PROGRAM	QUANTITY (a) MANUFACTURED	QUANTITY (b) FUNCTIONED	X-CORD CONFIG.			TIP CONFIG.
			MDC CORE LOAD 2.5 GR/FT	ME SHEATH MAT'L		
F-111	280,850	17,710	DIPAM	SILVER		STANDARD
F-14	53,260	4,900	HNS	SILVER		STANDARD
F-15	2,760	220	DIPAM	SILVER		STANDARD
S-3A	6,610	300	HNS	SILVER		STANDARD
EA-6B	3,500	400	HNS	SILVER		STANDARD
PROJECT 227	3,010	220	DIPAM	SILVER		STANDARD
ZUNI	—	—	HNS	ALUM.		DET. CORD LEAD
UPSTAGE	600	100	HNS	ALUM.		STANDARD
LIMPET	—	—	HNS	ALUM.		STANDARD
FAE	—	—	HNS	ALUM.		10GR/FT STD.
ZAP	2,200	200	HNS	ALUM.		STANDARD
APAM	—	—	HNS	LEAD		DET. CORD LEAD
LM CUTTER	2,300	1,050	HNS	SILVER		STANDARD
SPACE SHUTTLE TRANSFER	—	—	—	—		—
STANDARD ARM	4,500	120	DIPAM	SILVER		STANDARD
SPRINT	1,000	100	RDX	LEAD		STANDARD
CENTAUR	200	50	HNS	SILVER		STANDARD
TOTALS	360,790	25,370				

PROGRAM DATE OF FAILURE
F-111 1966
F-111 1968
PROJ 227 1973
F-14 1974

(a) ROUNDED TO NEAREST TWO SIGNIFICANT NUMBERS. NO TEST TIPS INCLUDED, ONLY DELIVERABLE ASSEMBLIES.

(b) ROUNDED TO NEAREST TWO SIGNIFICANT NUMBERS. TESTS INCLUDED ARE LAT, QUALIFICATION, DESIGN VERIFICATION AND STRUCTURAL INTEGRITY. NO DEVELOPMENT TESTS ARE INCLUDED. TESTS ARE THOSE CONDUCTED BY EXPLOSIVE TECHNOLOGY ONLY; CUSTOMER CONFIDENCE TESTS AND IN-SERVICE USAGE, IF INCLUDED, WOULD MORE THAN DOUBLE THE NUMBER OF ASSEMBLIES FUNCTIONED.

BASED ON THE ABOVE NUMBER OF TESTS WITH FOUR FAILURES, THE ACHIEVED RELIABILITY OF ET'S SMDC ASSEMBLY IS 0.999842, WITH A LOWER 90% CONFIDENCE LIMIT OF 0.999685.

TABLE 4

PROGRAMS USING SMDC MANUFACTURED BY TELEDYNE McCORMICK-SELPH
(TELEDYNE McCORMICK-SELPH)

PROGRAM	SMDC TYPE CORD/END BOOSTER	CUSTOMER SPECIFICATION	QUANTITY MANUFACTURED TMC/S
F-111	SILVER-DIPAM MDC HNS END BOOSTERS	McDONNELL DOUGLAS ZK04151 AF KIT A PN 7027296	163,500
F-15	SILVER-DIPAM MDC HNS END BOOSTERS	McDONNELL DOUGLAS PS 68-710072	6,500
AH-1G, AH-1J	SILVER HNS MDC HNS END BOOSTERS	857AS100 NAVY BHC 100009 BELL	450 12,000
B-1	ALUM. HNS MDC HNS END BOOSTERS	ROCKWELL L325C2002	15,000
HARPOON	SILVER-DIPAM MDC HNS END BOOSTERS	McDONNELL DOUGLAS ZK04151	600
SKYNET	SILVER HNS MDC HNS END BOOSTERS	PHILCO-FORD SP-DB 1953	100
RSRA	SILVER HNS MDC HNS END BOOSTERS	SIKORSKY/NASA SES 721002	1,500
RECEPTOR/ DETONATOR (B-1)	SILVER-DIPAM MDC HNS END BOOSTERS	NORTHROP-NORWOOD 67352	180
MODEL - 301	SILVER HNS MDC HNS END BOOSTERS	ROCKWELL-FULSA	200
APOGEE BOOST MOTOR	SILVER HNS MDC HNS END BOOSTERS	THIOLCO CORP. SE-257 A	300
AIRCRAFT EMERGENCY SURVIVAL EQUIPMENT EGRESS	SILVER HNS MDC HNS END BOOSTER	WRIGHT PATTERSON AFB NR412A-07878-55003	150
UPSTAGE	SILVER-DIPAM MDC HNS END BOOSTER	DOUGLAS-SANTA MONICA 1B-91785	25

TABLE 5
 DETONATING CORD IDENTIFICATION

NSWC ID NO.	CORD MFG	HNS MFG	MFG'S CORD LOT NO.	MFG'S HNS LOT NO.	PREP METHOD OF HNS	CORE LOAD	SHEATH MATERIAL	REMARKS
841	ET	NORTHROP CAROLINA	141364-966-1	8995-49	ORGANIC SOLVENTS	3.5 gr/ft	SILVER	6200 m/sec
892	ET	NORTHROP CAROLINA	63330100	11138-3	ORGANIC SOLVENTS	2.1 gr/ft	ALUM	6800 m/sec
1018	ET	NORTHROP CAROLINA	64970100	43-13	ORGANIC SOLVENTS	2.5 gr/ft	ALUM	5700 m/sec
1845	ET	CHEMTRONICS	80928100	66-16	ACID SOLVENTS	3.5 gr/ft	ALUM	6200 m/sec
1847	SOS	T/Mc-S	30863	8203-2	ACID SOLVENTS	2.5 gr/ft	SILVER	7200 m/sec
1850	ET	T/Mc-S	81008100	8203-2	ACID SOLVENTS	2.5 gr/ft	SILVER	7200 m/sec
1968	ET	CHEMTRONICS	80928100	66-16	ACID SOLVENTS	3.5 gr/ft	ALUM	ISOSTATICALLY PRESSED LOT ID 1845 6500 m/sec
1995	ET	T/Mc-S	81568200	8203-2	ACID SOLVENT	5 gr/ft	SILVER	6000 m/sec (LOW DENSITY)

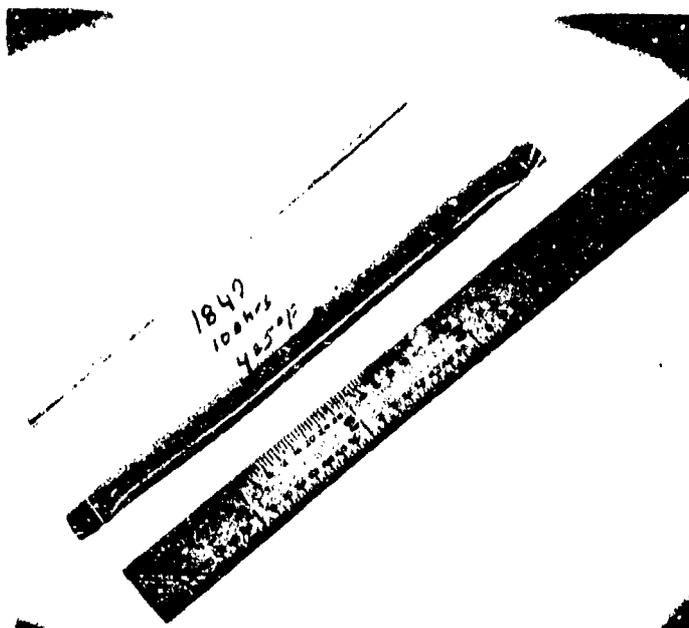


FIG. 1 DETONATING CORD ENCLOSURE FOR THERMAL CYCLING TESTS

4.3 Samples were withdrawn after the 100 cycle test. The individual copper tubes were cut open and each sample removed. Observation of the ends of the cords revealed some change in the explosive core.

4.4 The most significant finding was a change at the ends of the cords from the normal yellow to either dark brown or black of the explosive around the inside diameter of the silver sheath at the ends of the SOS (ID 1847) cords. The changed color seemed to be from a material which flowed from between the explosive column and the silver tube. Figure 2 shows the results along with the "as-received" material. It should be pointed out that the material at the ends of the detonating cord will be most susceptible to attack from gases present in the sealed copper tubes or other materials formed during the test. All cords and the copper tube had however been carefully cleaned prior to the test to remove any foreign material that might contaminate the unit. The end of the cord affected was removed and the explosive 1/4" farther down the cord observed. No color change occurred in the explosive column at this section.

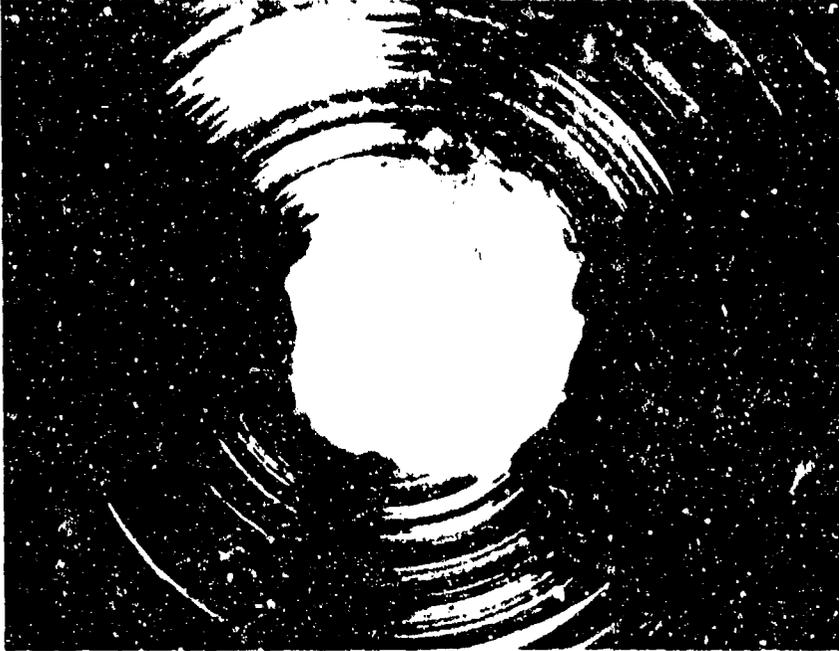
4.5 The same observations were made on ID 1850 (Explosive Technology). In contrast to the SOS material, no significant color change was observed. These cords were also cut back 1/4" to observe the explosive column. No change in color was observed. The 1/4" section was set aside for chemical analysis, and the remainder of the cord was held to determine detonation performance. In view of the color change and not knowing where in the 100 cycles the change began to take place, the cycling test was repeated with sample removals at 48 and 70 cycles using only ID 1847. The color change was observed after the 48 cycles test which means it probably occurred prior to 48 cycles.

5.0 Chemical Analysis

5.1 In view of the color change at the HNS/silver interface, samples of ID 1847 before and after 48 and 100 cycles were analyzed for possible chemical changes. Samples of HNS were removed from the cords by hand drilling using a #78 drill bit (0.016 diameter). Prior to the sampling operation, about 1/16 inch was cut off the end of the cord to expose a fresh surface. The diameter of the drill bit was less than the diameter of the HNS in the cord (approximately 0.020). A mass spectrum of each HNS sample was run using the solid probe mode of sample introduction (the sample is placed directly into the ion source and vaporized by controlled heating). No significant differences between samples could be discerned from their mass spectra. Thin layer chromatography⁽⁹⁾ (TLC) on each of the samples was run. The sample was put in acetone and the TLC plate developed in benzene. No differences between the samples were observed. No additional spots other than that due to HNS was observed.

5.2 A second set of HNS cords was subjected to thermal test. This time the sampling was done only after 100 cycles, the ends of the cord

(9) Stahl, Egon, "Thin Layer Chromatography," Spranger-Verlag, New York, 1969.



ID 1847
AS RECEIVED



ID 1847
AFTER
100 CYCLES
ENVIRONMENTAL

FIG. 2 DETONATING CORD SPACE ORDNANCE SYSTEMS ID 1847 BEFORE AND AFTER THERMAL CYCLING
(TELEDYNE MCCORMICK/SELPH LOT 8203-2 HNS-II)

were not removed following tests, and the drilling procedure was changed. All of the specimens showed a tan coloring at the air/silver/HNS interface. A sample of HNS from the specimen which had the darkest color was obtained by hand drilling using a larger diameter bit than before (#61, 0.039) to insure that the material at the HNS/silver interface would be sampled. The cord was not cut to expose a fresh, inner surface. A TLC plate was run as in the first set. No spots other than that due to HNS were observed. Care was taken to try to sample only the tan colored material at the HNS/silver interface. The mass spectrum obtained did not show any significant difference from that obtained from a reference sample of HNS.

5.3 It is estimated from the results that there were less than 0.1% of nitroaromatic type impurities in the HNS specimens examined. The results of these chemical analyses were unexpected, since the strong color change was thought to be indicative of some chemical decomposition. It was decided to continue with performance testing, where it was hoped that the suspicion of thermal decomposition of the explosive could be confirmed.

6.0 Measurement of the Performance of Various Detonating Cords

6.1 One measure of the performance of detonating cords is the velocity at which the detonation moves through the explosive core. A number of techniques can be used to obtain an accurate detonation velocity. Two methods were used in this work: the ionization probe and the streak camera. The ionization probe method gives an average velocity over some finite distance; the streak camera writes continuously on photographic film, from which velocity can be determined at any position of the trace.

6.2 In the ionization method, the passage of the detonation wave is sensed by the change it produces in the conductance and/or capacitance across the gap between the probes. This causes an abrupt change in the potential difference across the gap. Note the sketch of the ionization probes in Figure 3. The start-stop probe circuit is shown in Figure 4. A high resolution Hewlett Packard Model 5275A Time Interval Counter with a model 101A One Megacycle Oscillator as the clock source is used. In testing, the cords are initiated by the standard No. 6 blasting cap taped to the start probe end of the cord. The results of the performance tests using the probe method are shown in Table 6. It should be pointed out that the manufacturing data was collected by the vendor either from an ionization probe, some type of break circuit, or some method of measuring resistance change in a manganin wire. The standard detonating cord length for the probe work was 3-1/2 - 4 inches. Results using the smear camera are also given in Table 6 and the test method is described below.

The smear camera was used to provide a continuous observation of the detonation of the cords. Smear camera methods are not new as

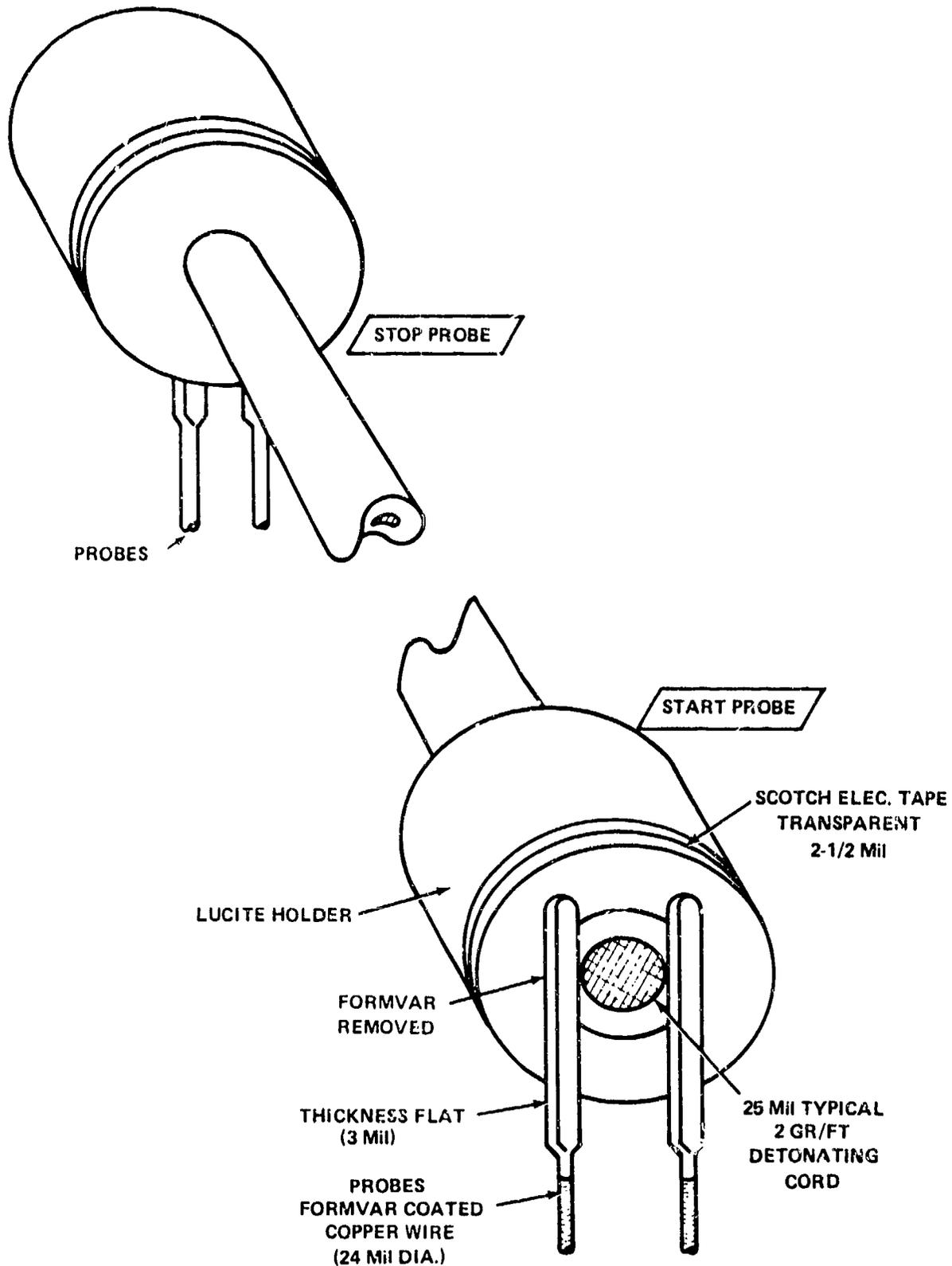


FIG. 3 IONIZATION PROBE/DETONATING CORD ARRANGEMENT

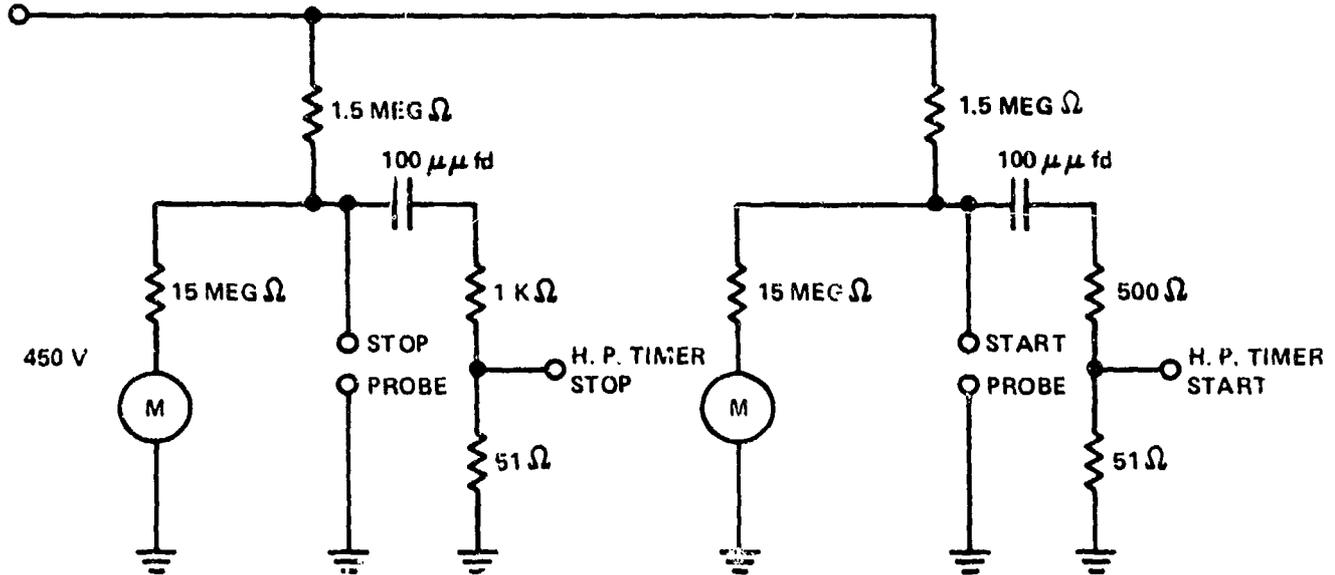


FIG. 4 START-STOP IONIZATION PROBE CIRCUIT

TABLE 6
SUMMARY OF DETONATION VELOCITY MEASUREMENTS AFTER
ENVIRONMENTAL TEMPERATURE CYCLING

MDF IDENTIFICATION	DETONATION VELOCITY (mm/ μ SEC)			
	AS RECEIVED	48 THERMAL CYCLES	70 THERMAL CYCLES	100 THERMAL CYCLES
ID 1847	7.1	—	—	6.9
(HNS-SILVER) ION	7.1	—	—	—
8203-2 SOS PROBE	7.1	—	—	—
2-1/2" SMEAR	7.0	—	—	—
LENGTH CAMERA				
4-1/2" SMEAR	7.1	—	—	—
LENGTH CAMERA	7.3	—	—	7.1
	MFG.	—	—	—
	DATA	NONE		
ID 1850				
(HNS-SILVER) ION	7.1	7.2	7.0	7.0
8203-2 ET PROBE	7.0	7.1	7.2	7.0
2" SMEAR	—	—	—	7.2
CAMERA				
2-1/4" SMEAR	7.0	—	—	7.1
CAMERA				
4" SMEAR	7.1	—	—	—
CAMERA				
	MFG.	—	—	—
	DATA			
ID 1845				
(HNS-ALUM) ION	6.2	—	6.2	6.1
66-16 ET PROBE	6.2	—	6.2	—
SMEAR	6.1	—	—	6.0
CAMERA				
	MFG.	—	—	—
	DATA			
ID 741				
(HNS-SILVER) ION	6.1	6.0	6.0	—
1966 PROBE				
ID 892				
(HNS-ALUM) ION	6.7	6.6	6.6	—
1962 PROBE				
SMEAR	6.8	—	—	6.7
CAMERA				
	MFG.	—	—	—
	DATA			

is pointed out by Liddiard and Drimmer⁽¹⁰⁾. There are a number of well-known optical and shock techniques for making the photographs: a light reflection system was chosen for this work because it offered no interference to the cord wall motion and it was accurate.

6.3 This study was intended to develop a camera technique and use it to detect differences in detonation velocity produced by the thermal environment. To understand how the measuring technique works requires an understanding of how the cord works. The detonation wave propagates down the center of the cord at a rate dependent on the confinement and the type and condition of the explosive core. An illustration of a detonation wave moving down the explosive core with attached shocks in the casing is shown in Figure 5. The reflected shocks and rarefactions in the casing behind the initial attached shocks are not shown. The boundary conditions require the attached shock to propagate down the casing with a velocity component along the length of the cord equal to the detonation rate. This is true when the sound velocity in the casing is low compared to the detonation rate. Therefore, the recording of the initial wall motion down the length of the cord measures the propagation rate of the detonation wave in the center.

6.4 In this technique the streak camera was used to record the cutoff of reflected light from the surface of the cord. A small reflective aluminized mylar foil was attached to the surface with an immeasurable amount of silicone grease making contact between the cord surface and the foil surface. This silicone grease insured that no air was trapped between these surfaces. When a shock wave reaches the foil, light immediately ceases to reflect at the same intensity as before. This change in intensity of light is recorded as a function of time by the smear camera. The motion of the foil after the shock arrival aids in changing the light intensity reaching the camera by changing the critical optical alignment.

The optics of the experimental setup are shown in Figure 6. The light from an exploding wire reflects off the cord and into the camera. The camera has a very fine slit which allows only light in the slit plane to reach the film. This plane of light (illustrated on the figure) is incident on the rotating mirror in the camera which exposes the high speed film as a function of light intensity and time.

6.5 An actual record is shown in Figure 7. The slope of the line defined by the sharp cutoff of light is a measure of the initial wall motion. The line's slope allows the rate to be determined by

$$D = \frac{u_c}{M_f} \cdot \frac{dy}{dx}$$

where D is the detonation rate, u_c is the camera writing rate, M_f is the film magnification and dy/dx is the slope of the line on the film.

(10) Liddiard, T. P. and Drimmer, B. E. "Smear Camera Techniques," J. SMPTE 70, 106 (1961)

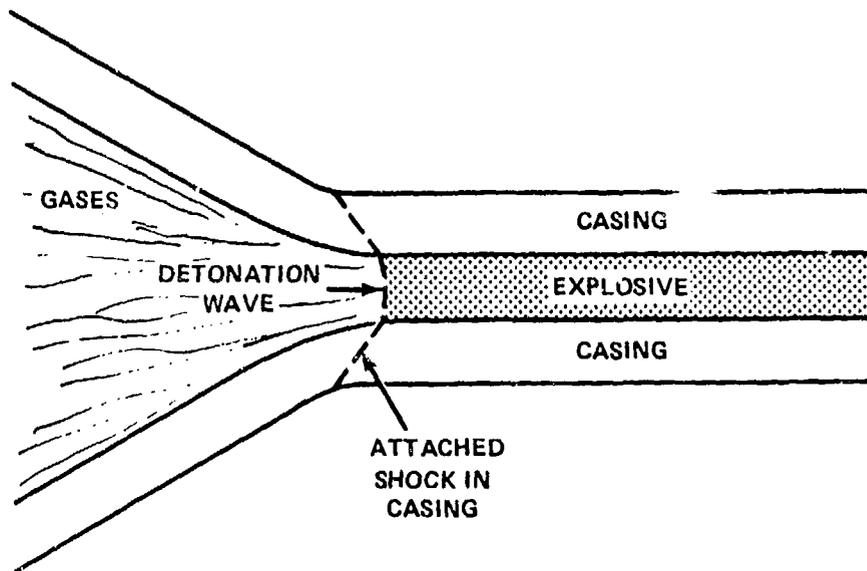


FIG. 5 CROSS-SECTION OF LENGTH OF REACTING DETONATING CORD.

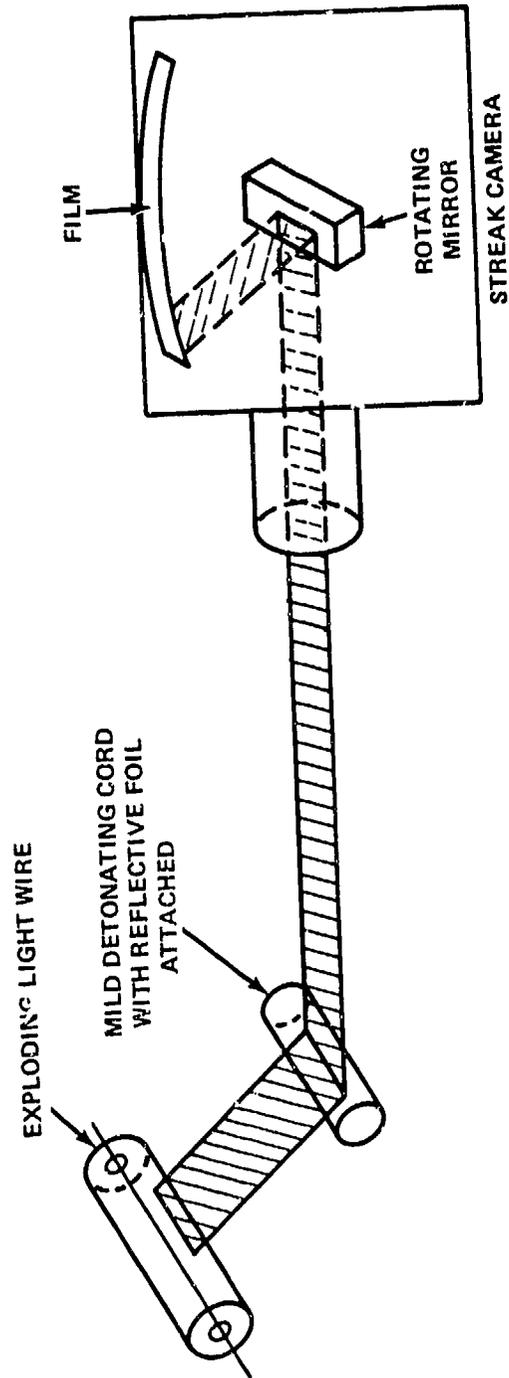


FIG. 6 OPTICS OF EXPERIMENT FOR PLANE OF LIGHT REACHING CAMERA.

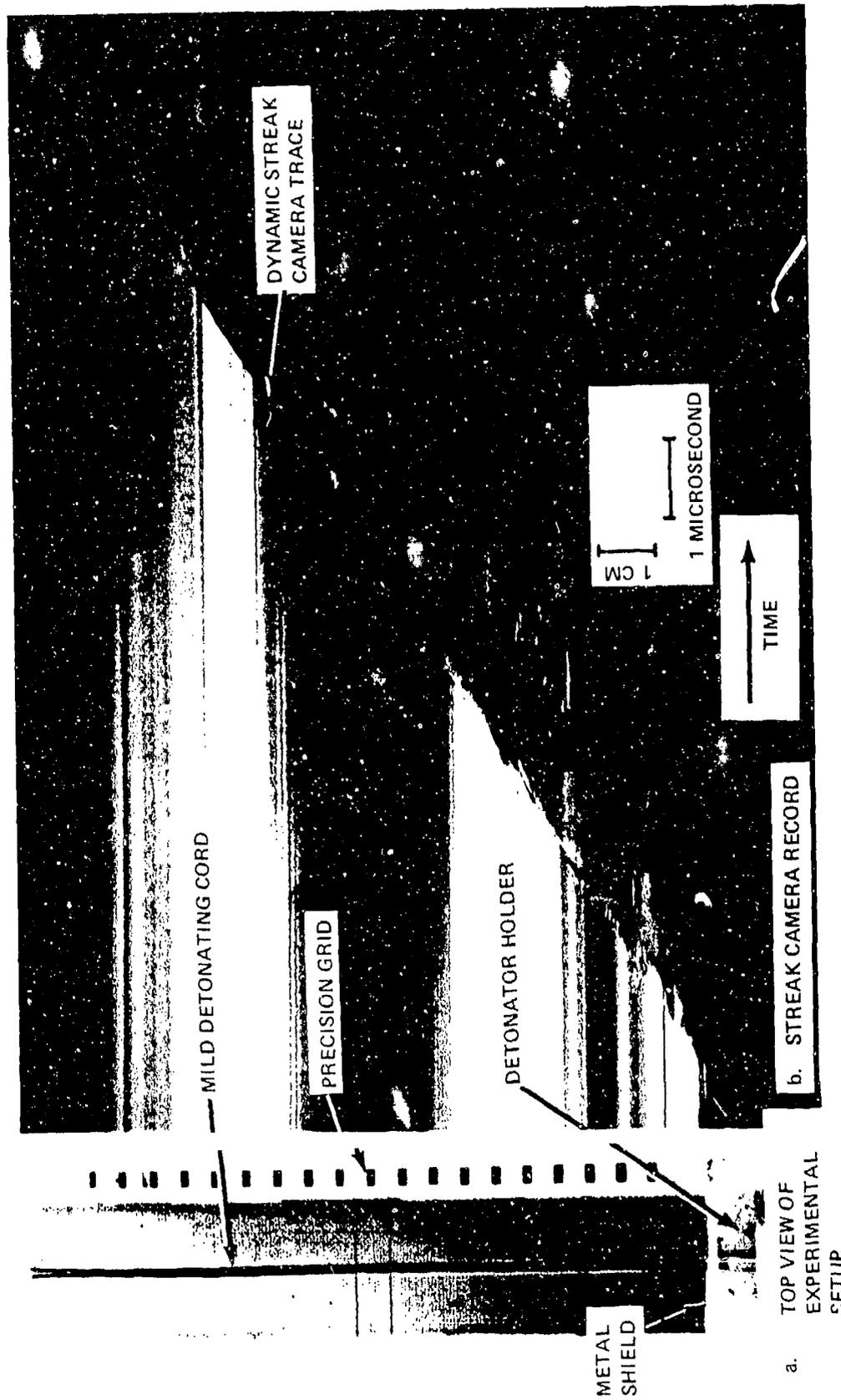


FIG. 7 TYPICAL STREAK CAMERA RECORD OF REACTING DETONATING CORD (SINGLE)

The detonation rate of the cords was determined near the midpoint of the cords. The midpoint was taken to avoid the possible error from overdriving the detonation by the detonator and to avoid other end effects. The rate near the midpoint appeared steady in all cases.

6.6 A major goal of this work was to compare two cords where, hopefully, the only difference was in the temperature cycling they had experienced. To do this, a dual experimental setup was made where both cords were set off nearly simultaneously by separate detonators. The detonators were of the exploding bridge wire type. They were connected in series electrically and set off by a 4000-volt pulse from a 4-microfarad capacitor. A typical record of such an experiment is shown in Figure 8. The metal shields were used to prevent reaction product gases from interfering while the measurements were being conducted.

The only information used from the records were the apparent steady rates measured near the midpoint of the cords. These steady rates seemed to be independent of size for the two lengths measured. An end effect was noted on all lengths and for all cords measured. This feature was not explored. A summary of results is given in Table 6. The results are within an experimental error of +5%. The heat cycled material had the same detonation rate as the "as-received" material.

6.7 As a part of the study, cords fabricated using Chemtronics "Acid precip" explosive ID 1845 and cords using "Organic Solvent Precip" material ID 841 and ID 892 were submitted to the environmental cycling test. This was done to obtain a better cross section of results for HNS-II and to observe any problem which might arise from incompatibilities with silver or aluminum at elevated temperature.

There were no color changes observed in these cords, and their performance indicated no greater change in velocity than was experienced with ID 1850. (See Table 6).

6.8 Scanning electron photomicrographs were taken of the two lots of HNS explosive loaded into cords: SOS/McCormick Selph Lot 8203-2 (cord ID 1847 and ID 1850) and ET/Chemtronics Lot 66-16 (cord ID 1845). Photographs of the HNS removed from cords in the "as-received" condition and following the 100 cycle temperature exposure are shown in Figures 9 and 10.

The surface of the explosives after the 100 cycles shows no indication of crystal growth or thermal decomposition. Either of these conditions would be expected to be reflected as a change in the detonation velocity. The higher density of the explosive in ID 1847 and ID 1850 compared to that of ID 1845 is reflected as a higher detonation velocity than for ID 1845, which is as expected.

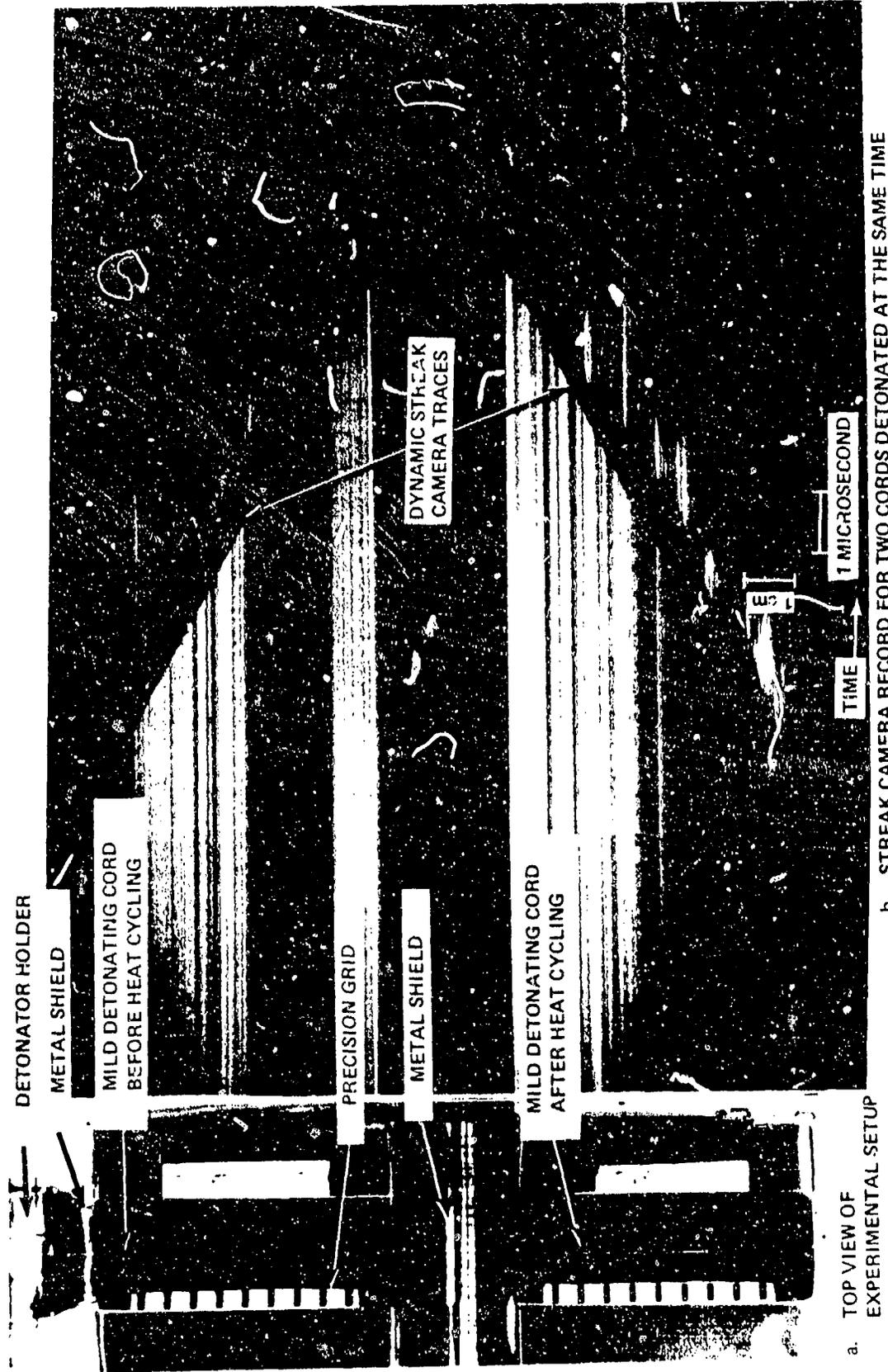
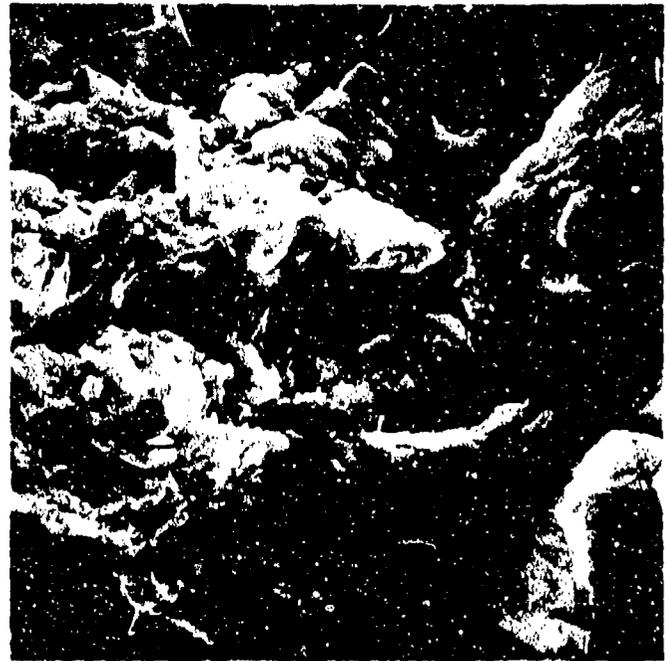


FIG. 8 TYPICAL STREAK CAMERA RECORD OF REACTING DETONATING CORD (DUAL ARRANGEMENT)



2500X

ID 1847
AS RECEIVED



ID 1850
AS RECEIVED



ID 1847
AFTER 100 CYCLES



ID 1850
AFTER 100 CYCLES

FIG. 9 ACID PRECIPITATED MATERIALS IN DETONATING CORDS (M. CORMICK/SELPH LOT 8203-2)



2500X

ID 1845
AS RECEIVED



ID 1845
AFTER 100 CYCLES



FIG. 10 ACID PRECIPITATED MATERIALS IN DETONATING CORDS (CHEMTRONICS LOT 66-16)

7.0 Penalization of the Grumman Design Control Specification

It was decided to extend the upper limit of exposure time at 425°F from the five minute time limit to about four days. This is not unreasonable since past experience with HNS (Organic Solvent Process) indicates it can be exposed to 400°F for over 100 days and still perform satisfactorily. This information is plotted in Figure 11.

7.1 Samples of cords ID 1847 and 1850 containing HNS lot 8203-2 were submitted to a temperature environment of 425°F. The cords were prepared and sealed in the same way as previously described (Section 4.2). Cord samples were removed after exposure to the elevated temperature at 4, 8, 32, and 56 hours. A short section was removed from the cords after the exposure for end viewing in the scanning electron microscope (SEM). Definite changes in the physical appearance occurred after the first four hours of exposure. There were crystal changes. These crystal changes could be indicative of changes to be expected in the performance of the detonating cords. Note photomicrographs in Figure 12. A section of ID 1850 is shown in Figure 13 at two different magnifications. The sample shows the same crystal habit as present in ID 1847 except the greater number of voids present after the thermal test is more detectable in ID 1850.

7.2 One significant finding in the vacuum thermal stability tests on the Teledyne McCormick Selph Lot 8203-2 which was used to fabricate detonating cords ID 1847, 1850 and 1995 is that it has an initial 20 minute gas surge at 260°C considerably higher than other reference samples of HNS (Appendix C, Table 3A) prepared from either the acid or organic solvent precipitation methods. This outgassing may have some effect on the porosity of the explosive core material.

7.3 HNS Lot 66-16 (Chemtronics Acid Prep) was submitted to elevated temperatures in ID 1968 and ID 1845. (Refer to Table 5 for cord identification). A review of photomicrographs in Figures 14 and 15 respectively, indicates crystal change and rounding of crystal forms in ID 1968 with the same appearance of voids as observed in the ID 1847 and ID 1850 high velocity cords. The lower velocity cord, ID 1845, does not appear to have been affected even after 264 hours at 425°F.

7.4 Another lot of HNS from Northrop-Carolina, Lot 11138-3, was exposed to high temperature as ID 892, which is a moderately high velocity cord. It is observed in Figure 16, that the explosive begins to become porous with an increase in voids at about 56 hours of exposure at 425°F. Massive voids are appearing in the column at 100 hours of exposure, with indications of possible fusion of the explosive crystals. The porosity was even greater at 264 hours (Figure 17).

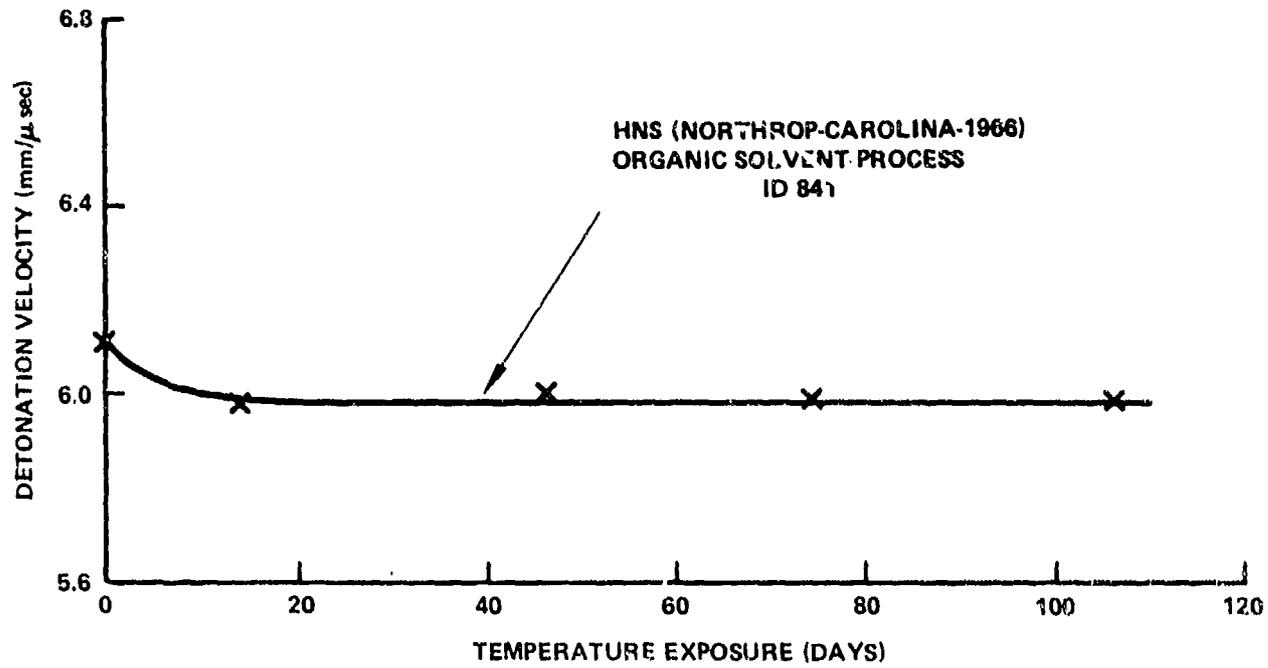


FIG. 11 ENVIRONMENTAL EXPOSURE OF DETONATING CORDS TO 204°C (399°F) IN SEALED TUBES



2500X

ID 1847 / 425° F
4 HRS.

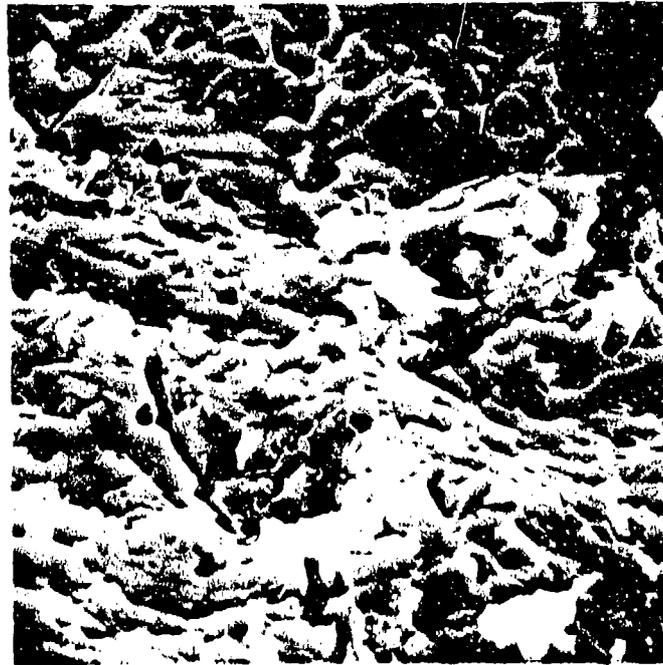


ID 1847 / 425° F
32 HRS.



ID 1847 / 425° F
56 HRS.

FIG. 12 CORD FABRICATED BY SPACE ORDNANCE SYSTEMS ID 1847
(McCORMICK/SELPH LOT 8203-2)



100X

ID 1850 / 425°F
56 HRS.



2500X

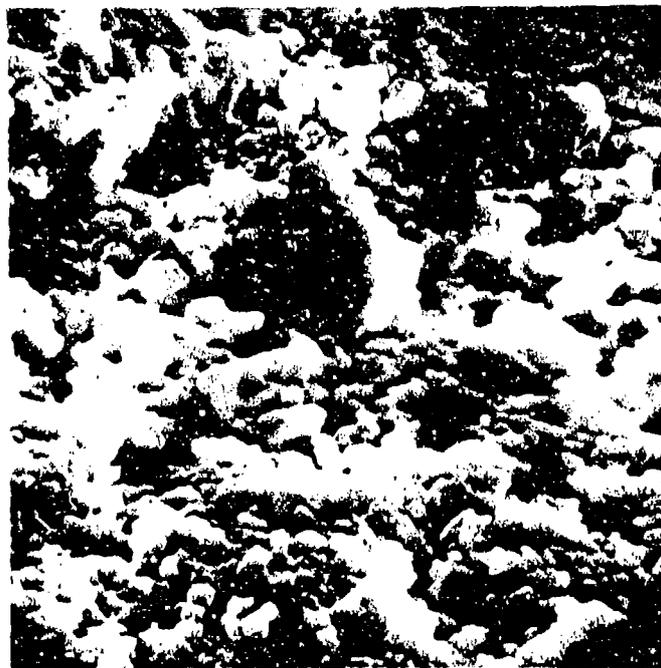
ID 1850 / 425°F
56 HRS.

FIG. 13 CORD FABRICATED BY EXPLOSIVE TECHNOLOGY ID 1850
(McCORMICK/SELPH LOT 8203-2)

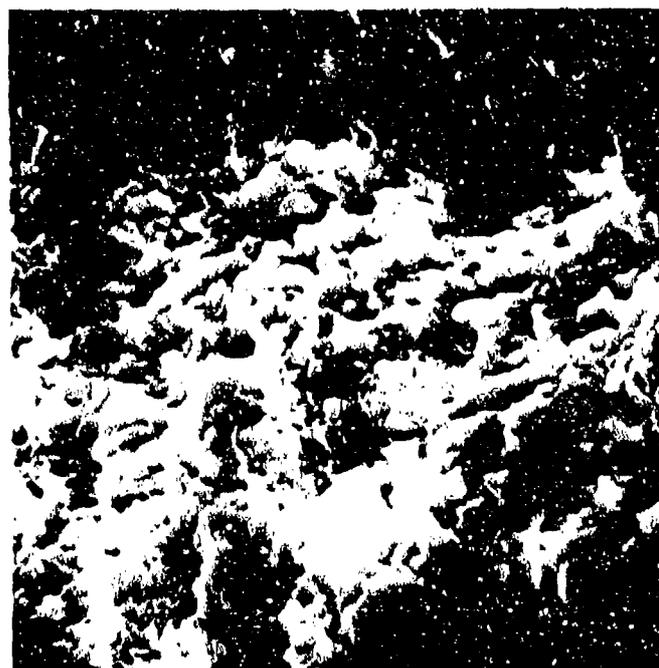


2500X

ID 1968/ORIG.



ID 1968 / 425°F
51 HRS.



ID 1968 / 425°F
99 HRS.

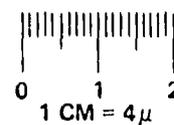
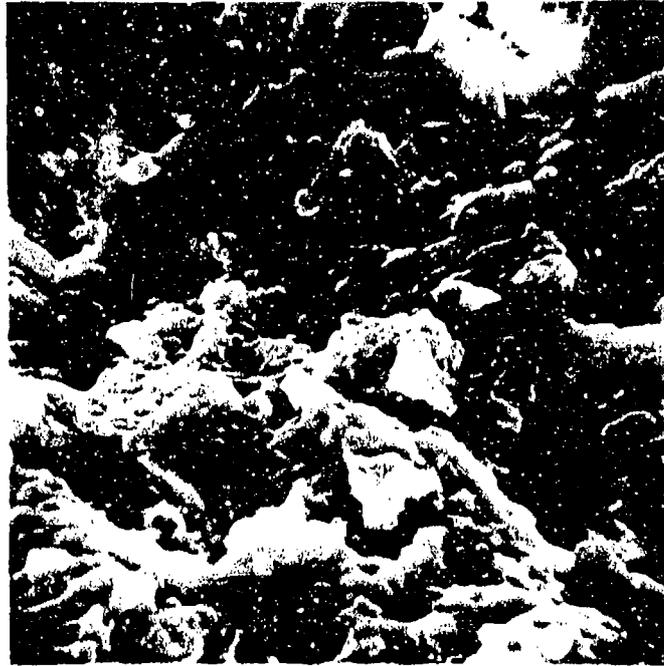


FIG. 14 CORD FABRICATED BY EXPLOSIVE TECHNOLOGY ID 1968
(CHEMTRONICS LO, 66-16)

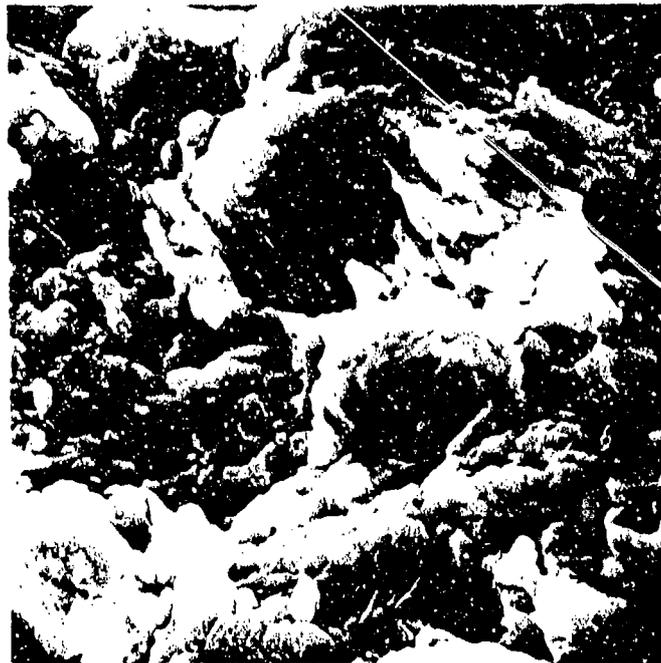


2500X

ID 1845/ORIG.

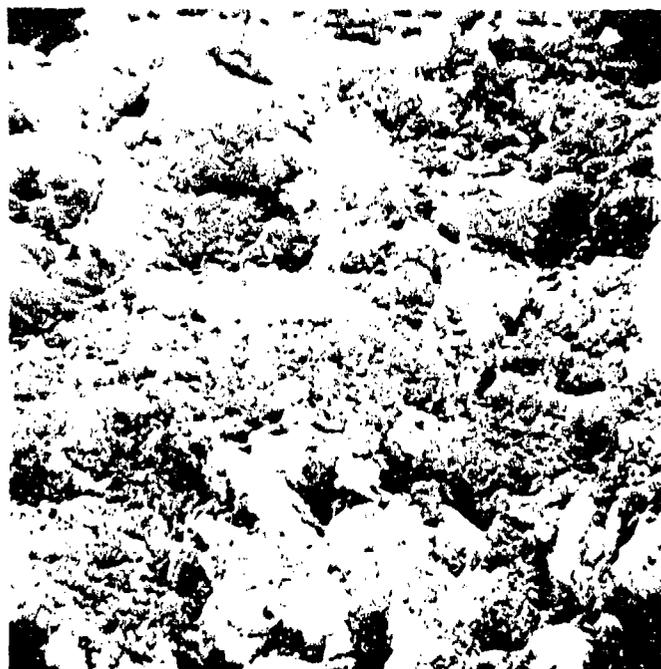


ID 1845 / 425° F
56 HRS.



ID 1845 / 425° F
264 HRS.

FIG. 15 CORD FABRICATED BY EXPLOSIVE TECHNOLOGY ID 1845
(CHEMTRONICS LOT 66-16)

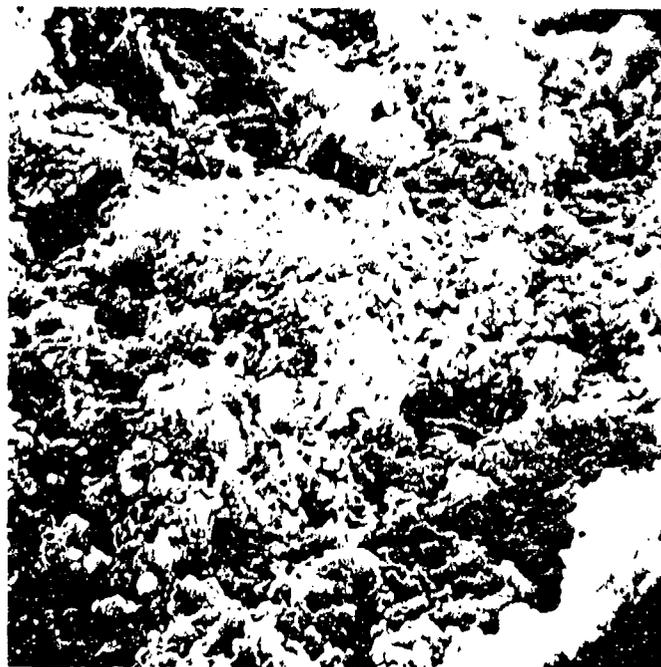


2500X

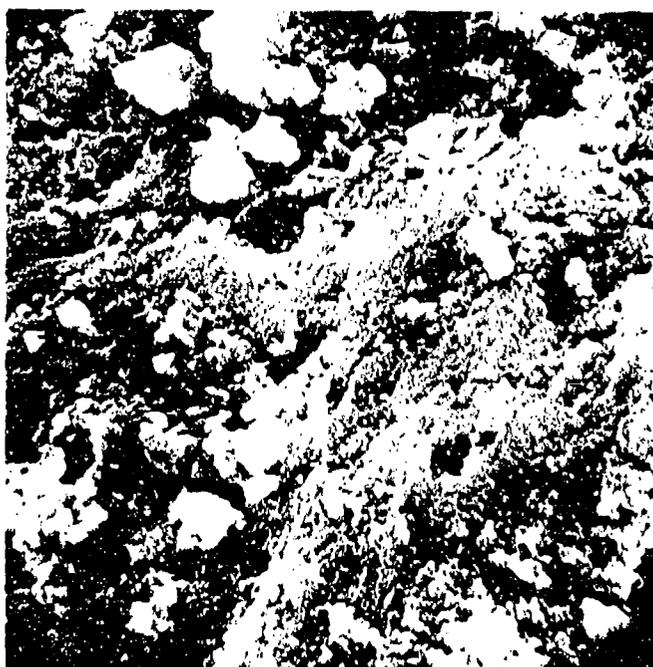
ID 892/ORIG.



ID 892 / 425°F
56 HRS.

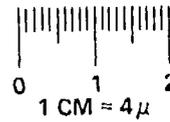


ID 892 / 425°F
75 HRS.



ID 892 / 425°F
100 HRS.

FIG. 16 CORD FABRICATED BY EXPLOSIVE TECHNOLOGY ID 892
(NORTHROP CAROLINA LOT 11138-3)



2500X

ID 892 / 425° F
264 HRS.



5000X

ID 892 / 425° F
264 HRS.

FIG. 17 CORD FABRICATED BY EXPLOSIVE TECHNOLOGY ID 892
(NORTHROP CAROLINA LOT 11138-3)

7.5 An additional lot of explosive, HNS Lot 43-13 (Northrop-Carolina) Organic Solv. Process, was exposed to elevated temperature as ID 1018. This cord is of considerably lower velocity than those of lot ID 892. It is obvious from Figure 18, that this lot of explosive material does not show the voids and massive crystal changes seen in cords from lot ID 892 because of its lower density. Photomicrographs of the above samples were taken from cross sections of clean, fractured, explosive columns about 1/4 inch from the ends of the cord. They were prepared by evaporating on the surface of the explosive material a thin film of gold (500-1000 Å thick) to insure proper conduction of electrons to the surface.

7.6 Chemical analysis of the explosive in the cords subjected to exposure at 425°F revealed different information than was found in the earlier cords that were exposed to an upper temperature of 400°F during the thermal cycling. According to Hoffsommer and Kubose of this Center, the results of thin layer chromatography show two spots on the plate which are indicative of nitroaromatics that are either polar or which exhibit a high molecular weight. It can be seen from the photograph of the TLC plate (Figure 19) that the additional materials are not present in the NSWC explosive standard (HNS-I, Sep 1965). The only materials detected are a spot each for HNS, hexanitrobenzyl, and some trace of TNT. The origin or the first spot on the plate of the exposed samples is loaded with decomposed materials which are extremely complicated to analyze. The important thing to note is that decomposition has taken place in the explosive. Both the "acid solvent" and the "organic solvent" material show decomposition, but the percentage of HNS remaining cannot be determined by TLC. More sophisticated analytical methods are available, but are not within the scope of this report.

8.0 A Measurement of Performance After Exposure to Elevated Temperature

8.1 After the 425°F exposure, the results of the thin layer chromatography indicate decomposition products are present in the cords. The SEM photos indicate physical changes also. Therefore the performance of the cords could have been affected. To determine whether the detonation velocity was affected by the high temperature exposure, and if so how, tests were run using the ion probe and smear camera methods previously described. Detonation velocities measured by the probe method were first conducted on cords ID 1847, ID 1850, and ID 1995. The results are plotted in Figure 20. The degradation in velocity with the increase in exposure time is obvious for cords ID 1847 and ID 1850. The lower velocity cord, ID 1995, containing the same HNS lot as ID 1847 and 1850 is seen to be unaffected. A smear camera trace of an "in-line" failure with a cord from lot ID 1847 (Figure 21) confirms the unreliability of these cords after exposure at 425°F. Note the velocity changes in Figure 21.



2500X

ID 1018 / 425° F
100 HRS.



5000X

ID 1018 / 425° F
100 HRS.

FIG. 18 CORD FABRICATED BY EXPLOSIVE TECHNOLOGY ID 1018
(NORTHROP CAROLINA LOT 43-13)

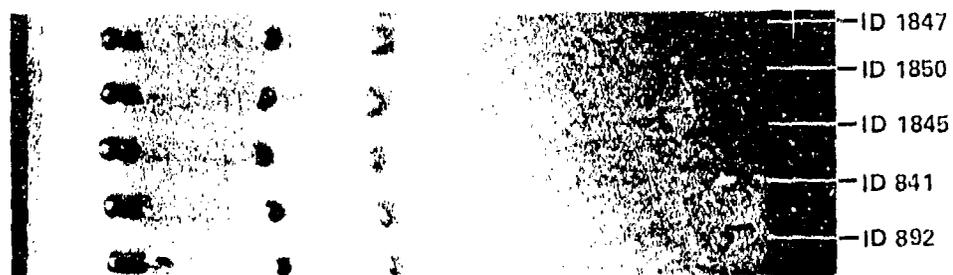


FIG. 19 THIN LAYER CHROMATOGRAPHY PLATE SHOWING SPOTS FROM SEVERAL DETONATING CORDS LOADED WITH HNS

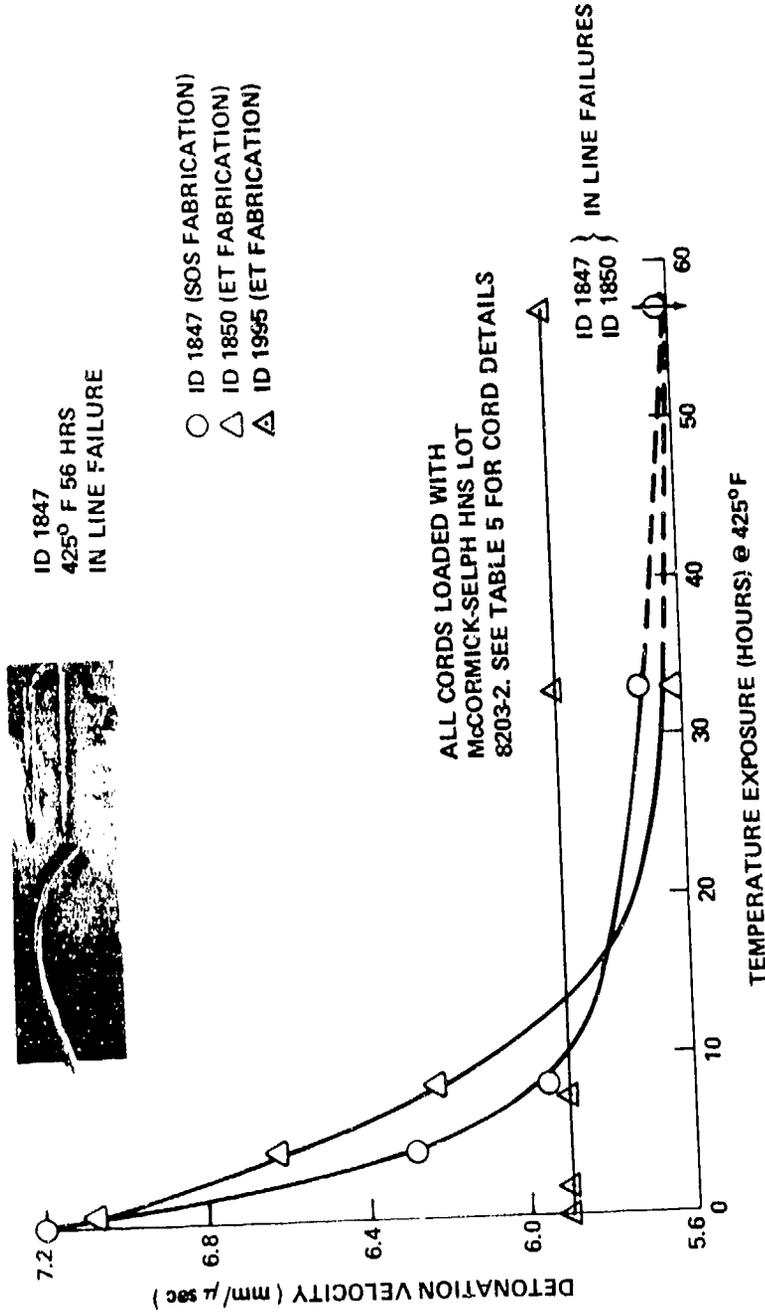
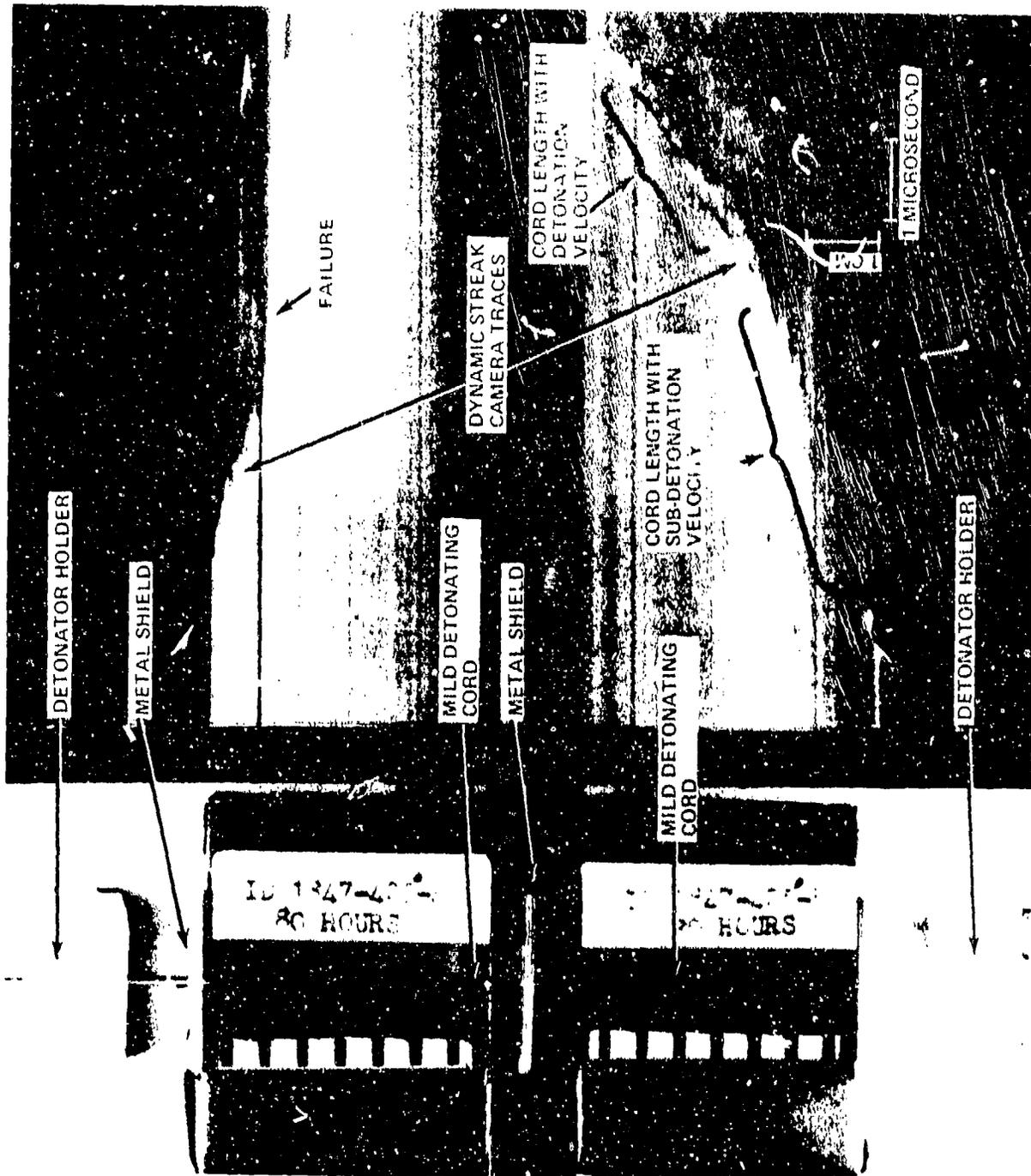


FIG. 20 THE DETONATION VELOCITY OF LOT 8203-2 HNS LOADED DETONATING CORDS AS A FUNCTION OF TIME OF TEMPERATURE EXPOSURE AT 425°F. (ID 1847, 1850, 1995)



a. TOP VIEW OF EXPERIMENTAL SETUP

b. STREAK CAMERA RECORD FOR TWO CORDS DETONATED AT THE SAME TIME

FIG. 21 DETONATING CORD FAILURE AND RAF. "BUILD-UP" TO DETONATION VELOCITY (DECOMPOSING EXPLOSIVE)

8.2 In the dual firing arrangement of Figure 21, one unit has failed to support detonation and the other, for all practical purposes, is unreliable. In the first case, the average velocity was found to be 4.5 mm/usec before failing completely. In the second case, the detonating cord was found to have two average velocities. The first measured about 3.24 mm/usec, and this changed very abruptly to 6.9 mm/usec.

8.3 The Chemtronics "Acid" material Lot 66-16 was tested in the same way. The results are shown in Figure 22. The moderately high velocity cord (ID 1968) did not show loss of detonation velocity as soon as the other "acid" material (Lot 8203-3 cords ID 1847 and ID 1850), but this may be due in part to the fact that the cords were not loaded to the same density. However, in either case, the high density cord shows a degradation of detonation velocity much sooner than the low density cord when subjected to the same conditions of time/temperature. It can be noted that the low velocity cord ID 1845, has a definite decrease in velocity at the beginning of the exposure period but remains relatively constant until about 100 hours where it then begins to degrade somewhere between the 100 hours and 264 hours period of time (graphed only to 100 hours exposure).

8.4 The ET/Northrop-Carolina (company name is now Chemtronics) "organic solvent precip" material was tested in cords ID 892, ID 1018 and ID 841. An SEM of the explosive from cord ID 841 is shown in Figure 23 and the detonation velocity in Figures 24 and 25. ID 892 exhibits the same properties as the high velocity acid material (ID 1968) except that it appears to be more thermally stable after 75 hours at 425°F. However, after 99 hours at 425°F it too exhibits "in-line" failures. The low velocity cords (ID 1018 and ID 841) exhibited stable detonation velocities through 264 hours (although only the first 100 hours are graphed).

8.5 ID 841 probably shows the best thermal stability of the cords exposed to 425°F. However, one should not draw the conclusion that this cord would give reliable performance at 425°F for longer periods of time.

8.6 The results of the performance tests show that ID 1850 (high velocity/acid material) is still showing a loss in performance at 375°F, as can be seen in Figure 26. The lower density "acid material" and the regular "organic solvent material" are relatively stable for long periods at 375°F and 400°F. These results are shown in Figures 26 and 27. The tests and test results reported herein are meant to be indications of the limits of high temperature performance not of reliability.

○ ID 1968 (ISOSTATIC PRESS - NSWC)

△ ID 1845

CORDS LOADED WITH
HNS LOT 66-16 (CHEMTRONICS-ACID PREP)
SEE TABLE 5

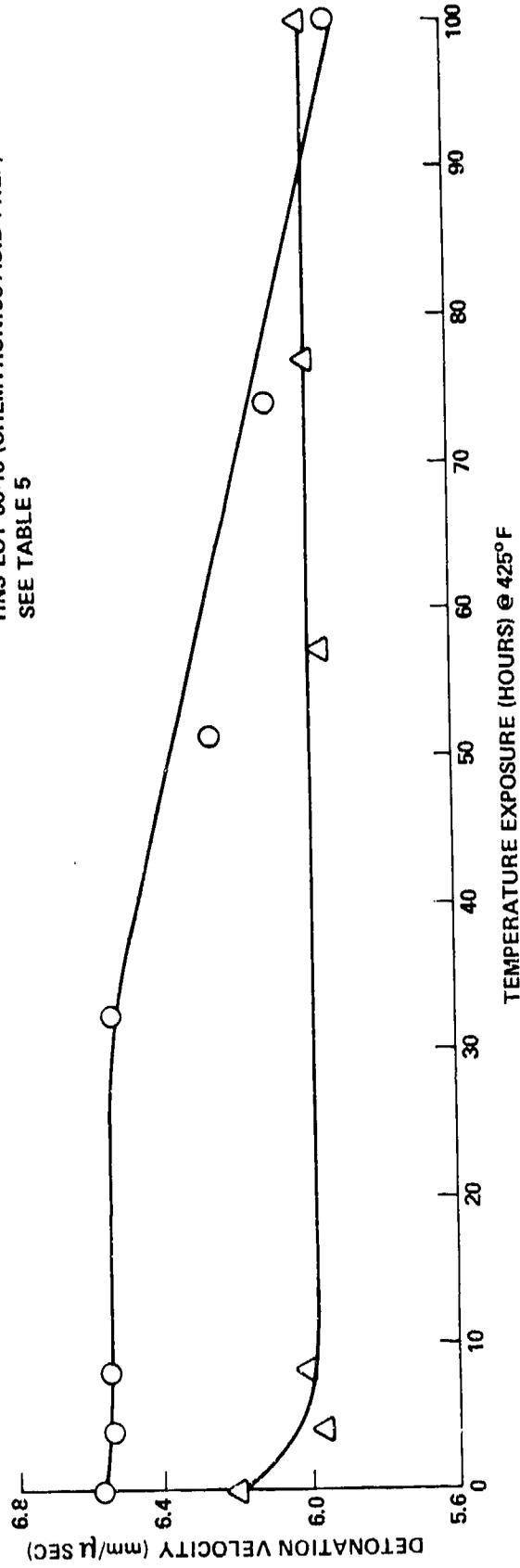


FIG. 22 THE DETONATION VELOCITY OF LOT 66-16 HNS LOADED DETONATING CORDS AS A FUNCTION OF TIME OF EXPOSURE AT 425°F. (ID 1968, 1845)

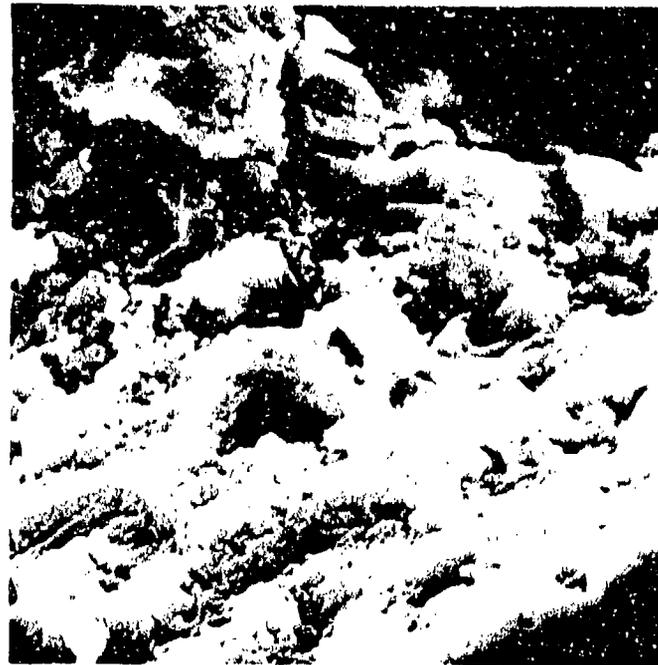


2500X

ID 841/ORIG.



ID 841 / 425° F
56 HRS.



ID 841 / 425° F
264 HRS.

FIG. 23 SEM OF HNS FROM CORD ID 841
FABRICATED BY EXPLOSIVE TECHNOLOGY

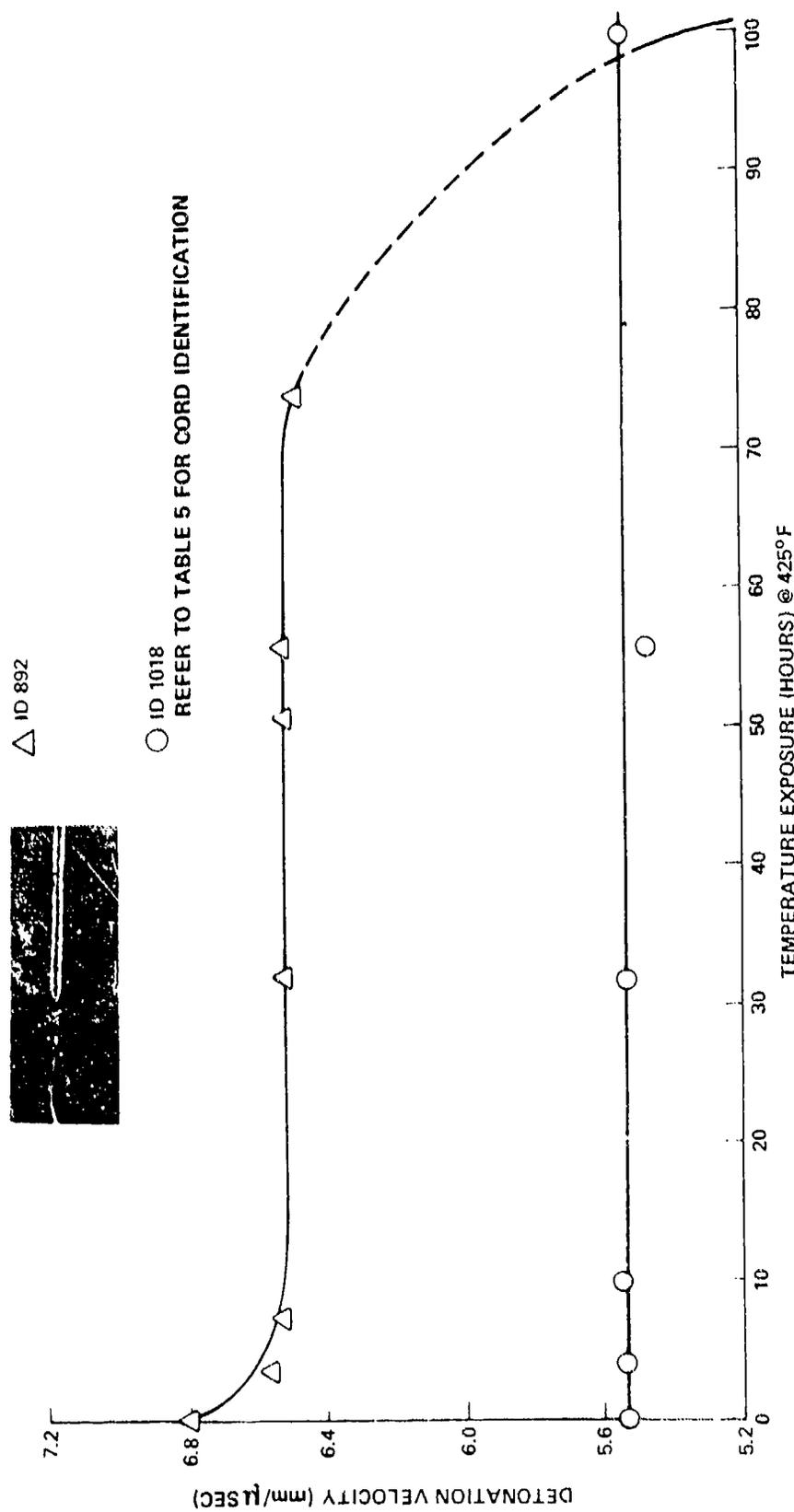


FIG. 24 THE DETONATION VELOCITY OF HNS DETONATING CORDS AS A FUNCTION OF TIME OF EXPOSURE AT 425°F. (ID 892, 1018)

○ ID 841

REFER TO TABLE 5 FOR CORD IDENTIFICATION

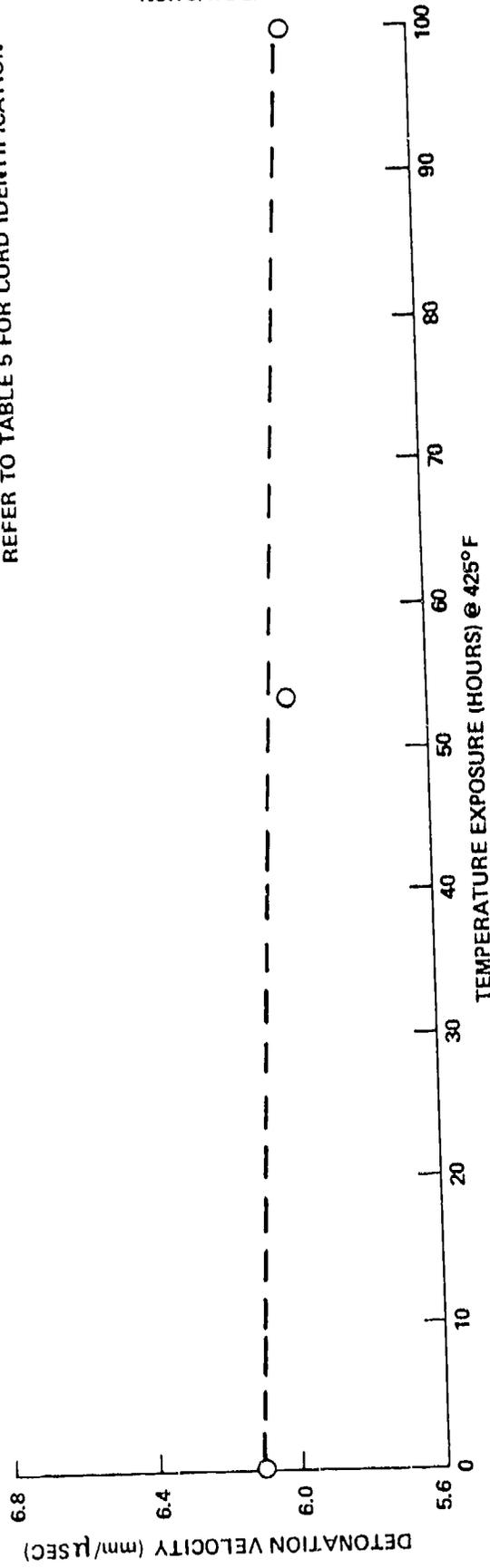


FIG. 25 THE DETONATION VELOCITY OF HNS DETONATING CORD AS A FUNCTION OF TIME OF EXPOSURE AT 425°F. (ID 841)

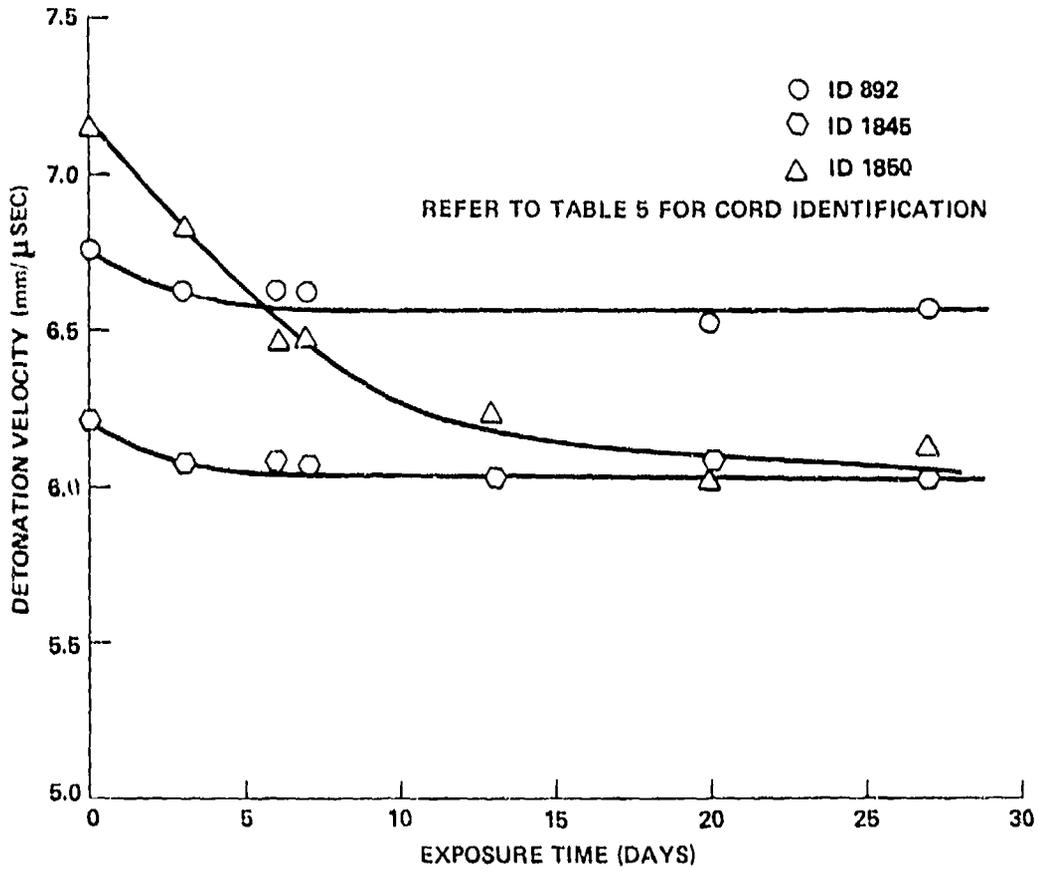


FIG. 26 THE DETONATION VELOCITY OF HNS DETONATING CORDS AS A FUNCTION OF TIME OF EXPOSURE AT 375°F. (ID 892, 1845, 1850)

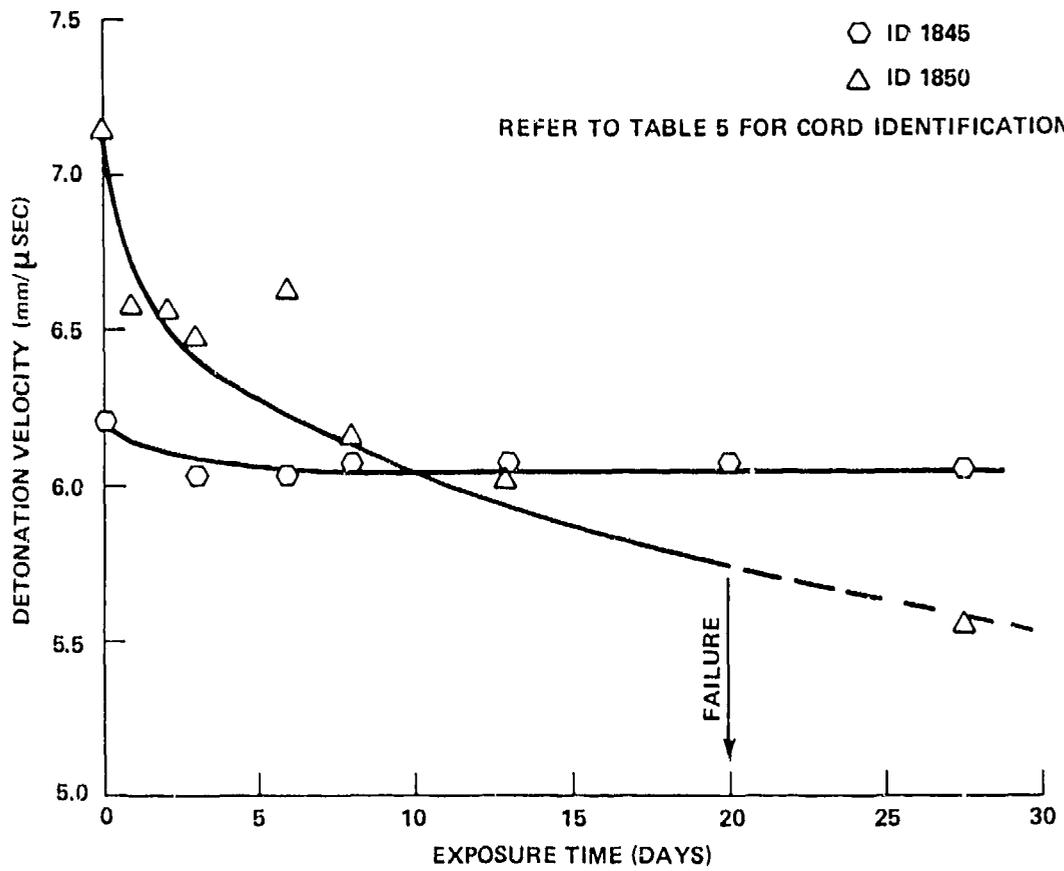


FIG. 27 THE DETONATION VELOCITY OF HNS DETONATING CORDS AS A FUNCTION OF TIME OF EXPOSURE AT 400° F. (ID 1845, 1850)

9.0 Conclusions and Recommendations

9.1 The combination of high temperature and high velocity i.e. high density explosive in HNS detonating cords has adverse effects on the cords performance. HNS-II processed from both "acid solvent recrystallization" and "organic solvent recrystallization" shows degradation of detonation velocity and chemical degradation following exposure to 425°F.

9.2 The high velocity (high density) "acid solvent recrystallized" HNS-II explosive in ID 1850 appears to be less stable than the lower density JD 1845 at 400°F. The lower density may account for the difference in performance after a long term exposure.

9.3 HNS from both processes will perform better at 425°F when it is present as a low density core load than as a high density load; however, exposure to 425°F is pushing the performance limits of the explosive in the cords.

9.4 Explosive cords with the low density loading of HNS will perform well at 375°F and 400°F for extended periods of time.

9.5 The results of the thin layer chromatography tests prove decomposition is taking place at elevated temperatures, but do not provide answers as to the nature of the decomposition products and the amount of decomposition. It is recommended that a more sophisticated analysis be used for a future study.

9.6 It is recommended that the ordnance designer consider carefully the expected upper limits of temperature in a realistic fashion when using explosive cords loaded with HNS. It is obvious from the results presented that a high velocity (high density), low core load material will not perform as well at elevated temperatures as a lower velocity (lower density) HNS tested under the same conditions. But if heat is not a problem, then there is no reason to expect that the high velocity (high density) material would not show good performance.

9.7 There is no evidence of sintering or crystal growth of HNS in either the "acid solvent" or "organic solvent" precipitated material at temperatures below 400°F.

9.8 It cannot be determined from the findings in this report how the particle size of both the HNS-I and HNS-II relates to the thermal stability of these explosives. The HNS-II in the detonating cords does not retain its crystal identity once it is processed into an explosive cord. In addition, the effect on sensitivity of the various particle sizes in detonating cords has not been determined.

10. Program Study Proposal

10.1 On the basis of the results of this study and the characterization study of various lots of HNS (in the appendix) the following program is proposed:

- (a) Determine the effect of HNS particle size on the stability and other properties of HNS-loaded detonating cords.
- (b) Determine, if necessary, how to control the particle size of HNS-I and HNS-II during the chemical preparation.
- (c) Re-evaluate the present specification, WS 5003, in terms of the need and methods for controlling the particle size range.
- (d) Study HNS crystal sizes in "most-used" hardware to evaluate the particle size ranges most suitable for each application.

APPENDIX A

Hexanitrostilbene-I

A-1 As a part of the effort to determine whether a problem existed with the thermal stability of HNS precipitated from an "acid solvent", it was necessary to characterize the explosive as to geometry, i.e., particle length, width, and thickness. The geometry of the crystal is determined by a number of variables e.g. temperature of the reaction, concentration of reactants, rate of addition of reactants, and polarity of solvent. The chemical reaction for the synthesis of HNS has been defined in the literature and is being used by industry to supply the material. The crystalline material synthesized by Dr. Shipp of the Naval Ordnance Laboratory (NOL) (now the Naval Surface Weapons Center (NSWC)) in 1962 was a needle shaped particle and is shown in Appendix A, Figure A-1 (ID 210). See Table A-1 for NSWC explosive procurements.

A-2 In 1963, the American Cyanamid Corporation (ACC) produced the same material for NOL in a 1850 gm. lot as shown in Figure A-2 (ID 363) Since these materials were found to be very difficult to handle and load into small explosive components, they were subsequently dropped. After discussions with Dr. Shipp about reducing the overall length of the particle and reducing its size, a new HNS was synthesized and is shown in Figure A-3 (ID 365). This material has found considerable application as HNS-I.

A-3 Since this material was produced in laboratory hardware, and since the quantity of reactants, rates of additions of reactants, and size of the continuous extractors have an effect on the particle size and geometry of the crystal, the effect of "scale-up" in production should be investigated.

A-4 In the early 1960's, the American Cyanamid Company was synthesizing material for NOL and for the Air Force/Navy F-111 Airplane program. These materials were synthesized in small glass laboratory hardware under the guidance of Dr. Fred Hudson of ACC. Some of the first material, sent to NOL about 1964 as HNS-I, was ID 1192. This sample is shown in Figure A-4. A year later ID 714 was procured. See Figure A-5. In 1966 ID 175 was procured, Figure A-6, and no more HNS-I was procured by NOL until 1969. In 1969, one lot of HNS-I was procured from Northrop-Carolina as ID 1071. This material was observed to be larger in particle size (Figure A-7) than previous lots, and failed to fall within the parallelogram of WS 5003 when tested for density and sensitivity. See parallelogram in Figure A-8.

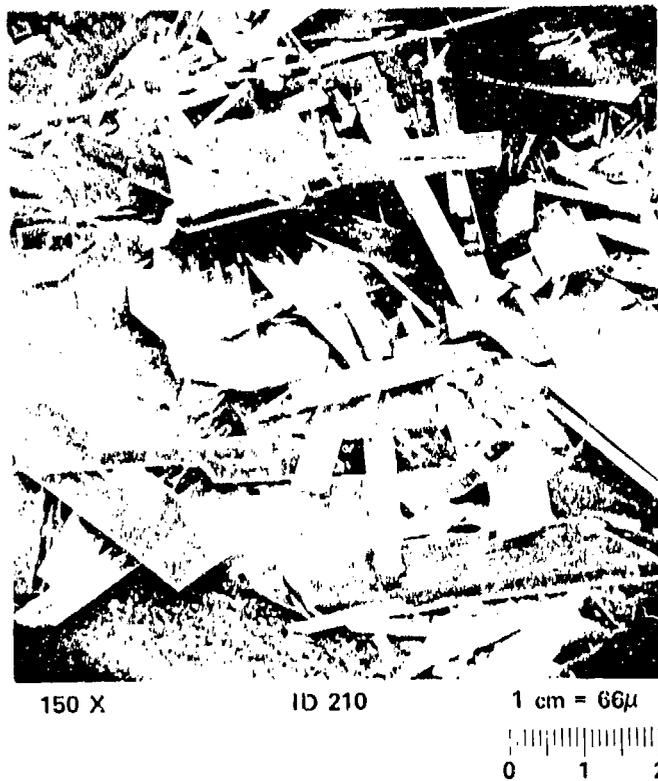


FIG. A-1 SEM PHOTOMICROGRAPH OF ID 210 HNS-R,
NSWC PREP.

TABLE A-1
EXPLOSIVE IDENTIFICATION (HEXANITROSTILBENE)

TYPE	NSWC ID NO.*	NSWC X NO.*	HNS MANUFACTURER	MANUFACTURER LOT. NO.	PREPARATION METHOD	DATE OF NSWC PROCUREMENT
HNS-R	210	401	NSWC	NOT KNOWN	SHIPP PROCESS	1962
HNS-R	363	420	AMER. CYN.	NOT KNOWN	SHIPP PROCESS	1963
HNS-I	365	477	NSWC	NOT KNOWN	ACETONE EXTRACTION	1964
HNS-I	1192	498	AMER. CYN.	333-544	HUDSON PROCESS	1964
HNS-I	714	537	AMER. CYN.	333-72	HUDSON PROCESS	1965
HNS-I	175	565	NORTH. CAR.	11138	HUDSON PROCESS	1966
HNS-I	1071	705	NORTH. CAR.	43-33	HUDSON PROCESS	1969
HNS-I	1090	716	DELMAR ENGR.	310-25	SHIPP PROCESS	1969
HNS-I	1920	NONE	CHEMTRONICS	66-48	HUDSON PROCESS	1974
HNS-I	1983	NONE	CHEMTRONICS	105-7	HUDSON PROCESS	1974
HNS-I	1984	NONE	CHEMTRONICS	66-39	HUDSON PROCESS	1974
HNS-I	1985	NONE	CHEMTRONICS	64-7	HUDSON PROCESS	1975
HNS-I	1987	NONE	CHEMTRONICS	105-6	HUDSON PROCESS	1975

*BULK EXPLOSIVE MATERIALS ARE IDENTIFIED BY EITHER OR BOTH THE ID NO. AND X NO.
AT THE NAVAL SURFACE WEAPONS CENTER (NSWC)



100 X

ID 363



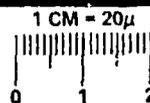
500 X

ID 363



500 X

ID 363

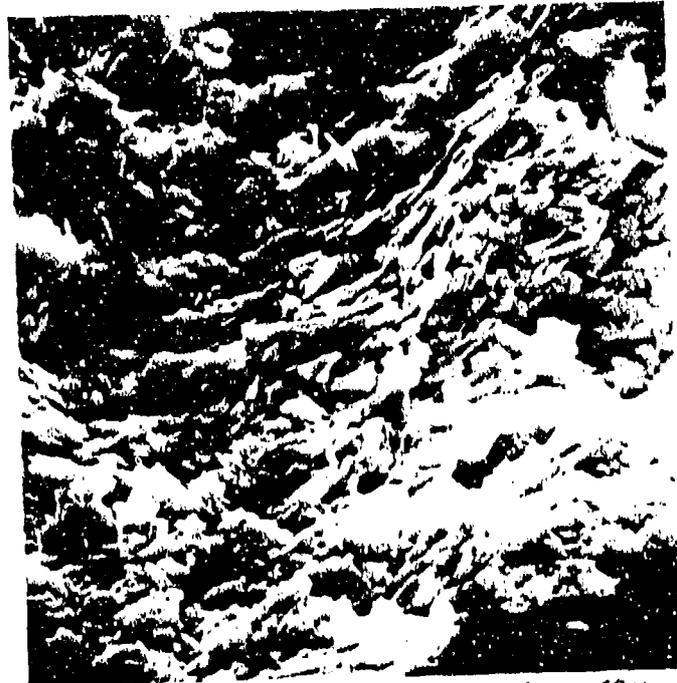


1000 X

ID 363



FIG. A-2 SEM PHOTOMICROGRAPHS OF ID 363 HNS-R,
AMERICAN CYNAMID PREP.



1000 X

ID 365

1 cm = 10 μ



2500 X

ID 365

1 cm = 4 μ



FIG. A-3 SEM PHOTOMICROGRAPHS OF ID 365 HNS-I,
NSWC PREP (ACETONE EXTRACTION)

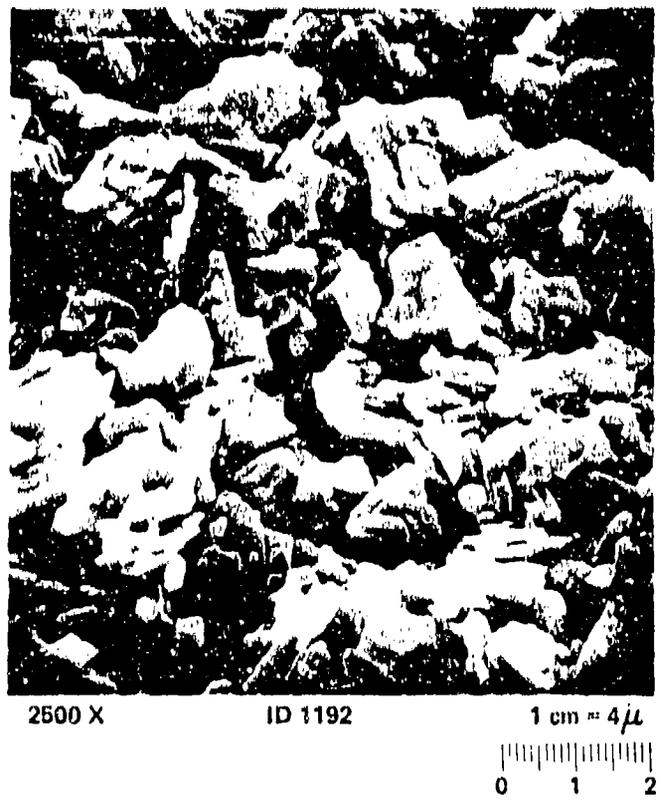
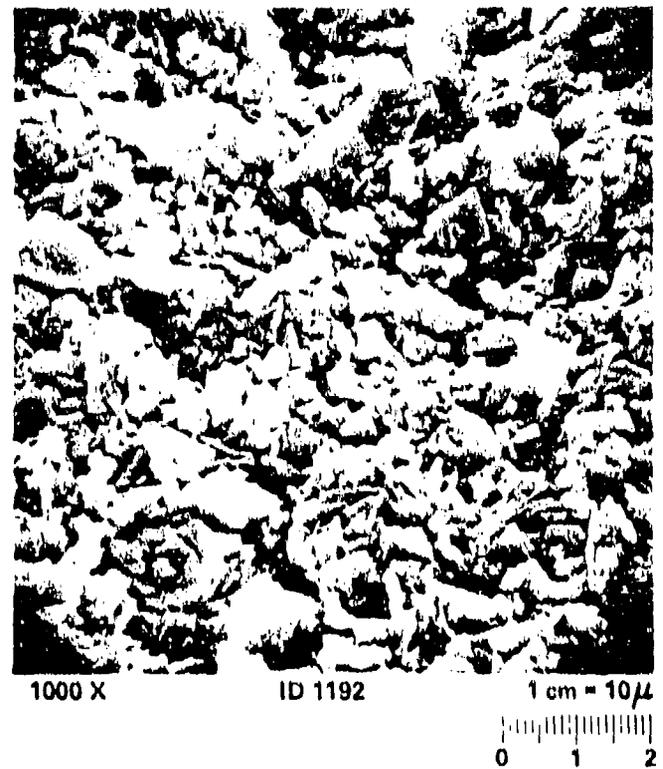


FIG. A-4 SEM PHOTOMICROGRAPHS OF ID 1192 HNS-I,
AMERICAN CYNAMID LOTS 333-544 TO -547



2500 X

ID 714

1 cm = 4 μ



2500 X

ID 714

1 cm = 4 μ



FIG. A-5 SEM PHOTOMICROGRAPHS OF ID 714 HNS-I,
AMERICAN CYNAMID LOT 333-72



1000 X

ID 175



2500 X

ID 175



5000 X

ID 175



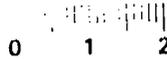
FIG. A-6 SEM PHOTOMICROGRAPHS OF ID 175 HNS-I,
NORTHROP CAROLINA LOT 11138



1000 X

ID 1071

1 cm = 10 μ



2500 X

ID 1071

1 cm = 4 μ

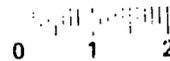


FIG. A-7 SEM PHOTOMICROGRAPHS OF ID 1071 HNS-1,
NORTHROP CAROLINA LOT 43-33

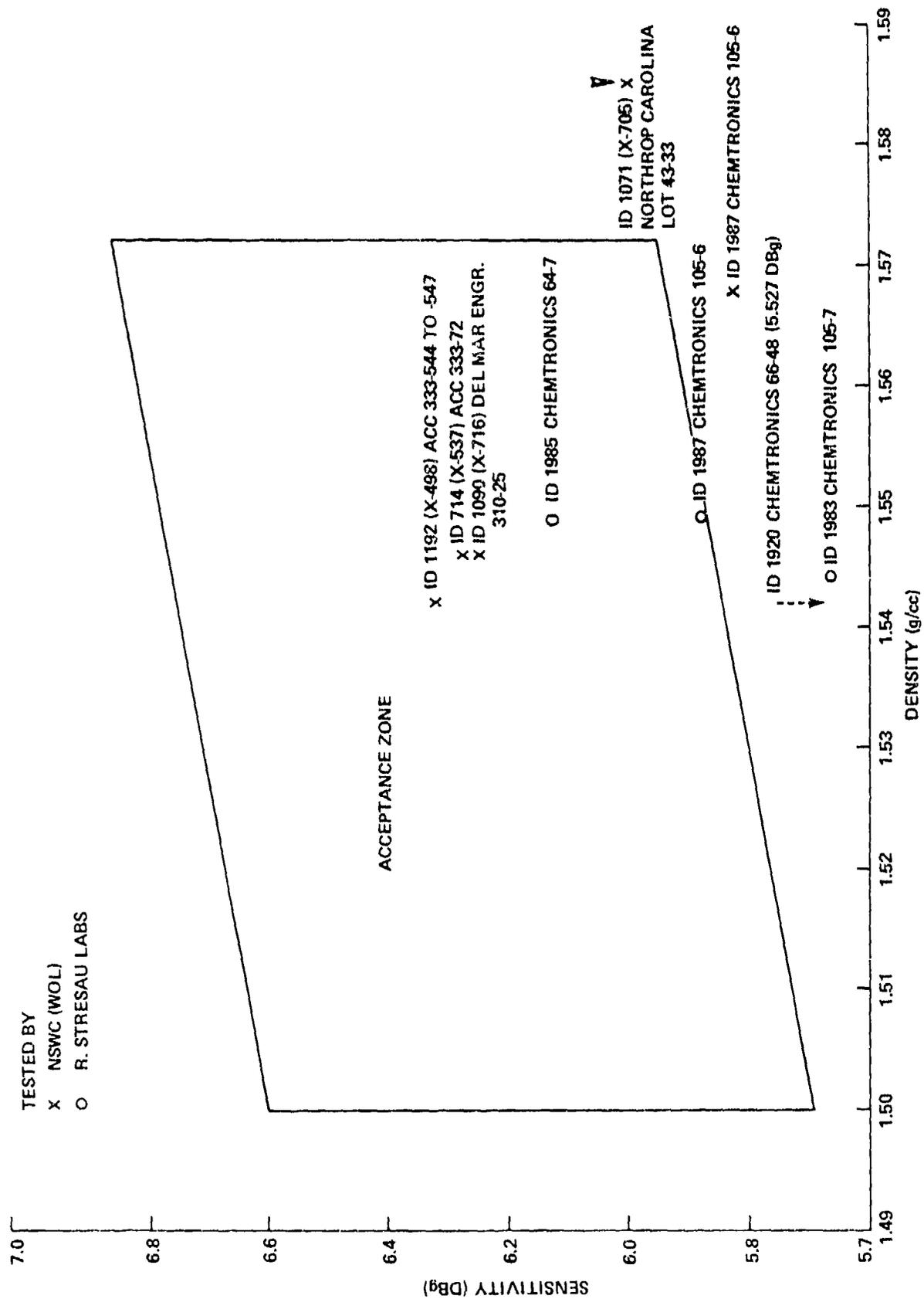


FIG. A-8 WS 5003 ACCEPTANCE CRITERIA FOR SENSITIVITY TEST OF HNS-I VENDOR QUALIFICATION.

During the same year Del Mar Engineering began to produce HNS, and NOL procured one lot from them identified as ID 1090. This is shown in Figure A-9. This material was of a particle size comparable to the earlier ACC material and included even smaller fines. The Del Mar Engineering material passed the density and sensitivity test in WS 5003. Prior to 1971 Northrop-Carolina used small scale equipment, but changed to large scale equipment during 1971. They produced with this equipment until February 1973, at which time larger holding tanks were installed.

A-5 In October 1974 Chemtronics (formerly Northrop-Carolina) sold a lot of HNS-I to the Naval Surface Weapons Center (NSWC), ID 1920. This material, shown in Figure A-10 was rejected because it could not meet WS 5003 density and sensitivity requirement. The particle length and thickness was greater than previous materials as is pointed out in Figure A-11 and Table A-2. Here again the "scale-up" in the process is suspect because of the appearance of the larger particles. A recent sample of HNS-I, ID 1983, was rejected because of density and sensitivity (see Figure A-12). This material also had a thick cross section (2-4 μ) as did some earlier rejections.

A-6 Other samples such as ID 1985 (Figure A-13) and ID 1987 (Figure A-14) also show differences in particle size and geometry. ID 1987 passed the sensitivity test conducted by Stresau and failed the test according to NSWC in March of 1975. The NSWC has not tested ID 1984 but the material did pass the sensitivity test according to Chemtronics. This material has a slightly different shape than other HNS-I's. The particle size is quite varied, with large quantities of both fine and large particles. The crystals appear to have a thin cross section. See Figure A-15.

A-7 In summary the following observations are made:

For HNS-I,

(a) In the synthesis of HNS-I, the early ACC material (1964) and the Del Mar Engineering explosive (1969) are most nearly alike. The Chemtronics (Northrop-Carolina) material from 1969 to 1975 changed in particle size and geometry.

(b) The particle length (range) affects the sensitivity--in addition the thickness of the particle has a significant effect on the sensitivity--it tends to become more sensitive with these increases.

(c) The indirect control on the synthesis of HNS-I by application of the WS 5003 specification is shown by the widely varied particle size range and shapes. The "scale-up" of the process, amongst other things, is probably the largest factor causing this shift in particle size.



1000 X

ID 1090

1 cm = 10 μ



2500 X

ID 1090

1 cm = 4 μ



FIG. A-9 SEM PHOTOMICROGRAPHS OF ID 1090 HNS-I,
DELMAR ENGINEERING LOT 310-25

NSWC/WOL/TR 75-142



1000 X

ID 1920

1 cm = 10 μ

0 1 2



2000 X

ID 1920

1 cm = 4 μ

0 1 2

FIG. A-10 SEM PHOTOMICROGRAPHS OF ID 1920 HNS-I,
CHEMTRONICS LOT 66-48

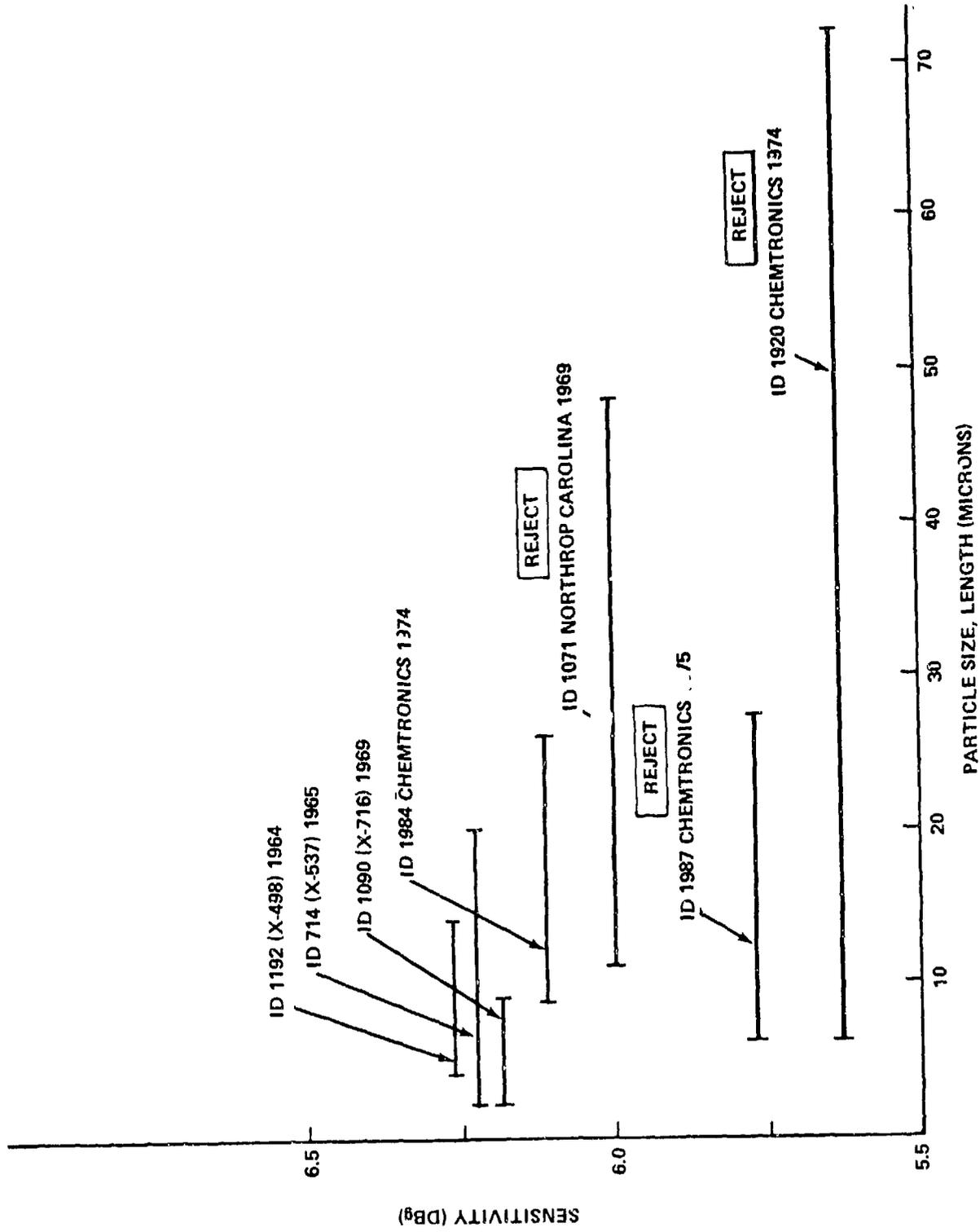


FIG. A-11 THE VARIATION OF PARTICLE SIZE LENGTH WITH SENSITIVITY OF HNS-1

TABLE A-2 THE IDENTIFICATION OF SEVERAL LOTS OF HNS-I AS TO SHOCK SENSITIVITY AND PARTICLE SIZE.

EXPLOSIVE ID#	SENSITIVITY SSGT(DBg)	PARTICLE SIZE RANGE	
		LENGTH μ	THICKNESS μ
1920 (REJECT - WS 5003)	5.527 @ 1.5418 g/cc	5-72	1-4
1987 (REJECT - WS 5003)	* 5.875 @ 1.549	5-28	2-5
1071 (REJECT - WS 5003)	6.057 @ 1.586	11-48	0.8
1192	6.322 @ 1.542	4-14	0.4-2 μ
1090	6.247 @ 1.546	2-8	1-2
714	6.263 @ 1.546	2-20	0.6

* TESTED BY R. STRESAU LABS



1000 X

ID 1983

1 cm = 10 μ



2500 X

ID 1983

1 cm = 4 μ

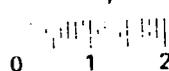


FIG. A-12 SEM PHOTOMICROGRAPHS OF ID 1983 HNS-I,
CHEMTRONICS LOT 105-7



500 X

ID 1985



1000 X

ID 1985



2500 X

ID 1985



5000 X

ID 1985



FIG. A-13 SEM PHOTOMICROGRAPHS OF ID 1985 HNS-1,
CHEMTRONICS LOT 64-7

NSWC/WOL/TR 75-142



1000 X

ID 1987

1 cm = 10 μ



2500 X

ID 1987

1 cm = 4 μ



2500 X

ID 1987

1 cm = 4 μ

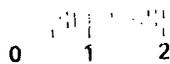


FIG. A-14 SEM PHOTOMICROGRAPHS OF ID 1987 HNS-I,
CHEMTRONICS LOT 105-6



1000 X

ID 1984

1 cm = 10 μ

0 1 2

FIG. A-15 SEM PHOTOMICROGRAPH OF ID 1984 HNS-I,
CHEMTRONICS LOT 66-39

APPENDIX B

Hexanitrostilbene-II

B-1 After the NOL conversion of HNS-I by the Taylor/Oesterling process to HNS-II in 1965, Appendix B (Figure B-1), the first commercial lot of HNS-II procured was from Northrop-Carolina in 1966. This material, ID 30, is shown in Figure B-2. In 1968 there were two lots procured; the first being ID 777 and the second ID 795. See Figure B-3, and note the difference in particle size. Table B-1 shows the explosive procurement at the NSWC.

B-2 By 1970 other companies were becoming interested in the synthesis of HNS-II. Ensign Bickford submitted a lot in 1970, ID 1279, Figure B-4, which had an extremely large particle size and was rejected on the basis of the density falling outside the parallelogram (Figure B-5). During the following year a lot was procured from Del Mar Engineering, ID 1479, Figure B-6, which had a different shape from previous HNS-II but the particle size range and sensitivity was very close to the NOL conversion material. The Del Mar synthesis used the Syrop patented method of continuous extraction with acetonitrile-xylene.

B-3 The detonating cord industry was making strong requests for reduced costs and large particle size HNS-II to facilitate their loading operations. So, during the early part of 1973, Chemtronics made a process change. Organic solvent recovery has always been expensive in the Taylor/Oesterling process, therefore, the new "acid solvent" method was introduced. Crystals from the acid process can be seen in Figure B-7 ID 1831. The crystals are very large and unlike those from the "organic solvent" process. Teledyne McCormick Selph also became active in this area about this time. Their material ID 1859, is also shown in Figure B-7. Interestingly enough, both materials passed the sensitivity test in WS 5003 (Figure B-5) but are toward the insensitive side of the parallelogram. Note the large particle lengths and the thicknesses in Figure B-8 and Table B-2 of Appendix B. This points out the trend toward decreasing sensitivity in HNS-II with an increase in particle thickness (Figure B-9). According to Stresau, the latest material, ID 1981 (Figure B-10), passed the sensitivity test in WS 5003 with 5.646 DBg @ 1.647 g/cc. This is the range expected for the particle size shown in Figure B-9. This material has rounded edges and is moderately thick in cross section.



500 X

ID 207

1 cm = 20 μ



1000 X

ID 207

1 cm = 10 μ



FIG. B-1 SEM PHOTOMICROGRAPHS OF ID 207 HNS-II,
NSWC CONVERSION



100 X

ID 30



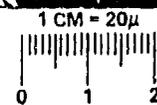
500 X

ID 30



500 X

ID 30



500 X

ID 30



FIG. B-2 SEM PHOTOMICROGRAPHS OF ID 30 HNS-II,
NORTHROP CAROLINA LOT 11138-8



250 X

ID 777

1 cm = 40 μ



250 X

ID 795

1 cm = 40 μ



FIG. B-3 SEM PHOTOMICROGRAPHS OF ID 777, ID 795 HNS-II,
NORTHROP CAROLINA LOT 11138-20 A&B AND LOT 11138-3

TABLE B-1
EXPLOSIVE IDENTIFICATION (HEXANITROSTILBENE)

TYPE	NSWC ID. NO.*	NSWC X NO.*	HNS MANUFACTURER	MANUFACTURER LOT NO.	PREPARATION METHOD	DATE OF NSWC PROCUREMENT
HNS-II	207	550	NSWC	NOT KNOWN	NSWC CONVERSION	1965
HNS-II	30	567	NORTH. CAR.	11138-8	HUDSON PROCESS	1966
HNS-II	777	580	NORTH. CAR.	11138-20	HUDSON PROCESS	1968
HNS-II	795	NONE	NORTH. CAR.	11138-3	HUDSON PROCESS	1968
HNS-II	1279	735	ENSIGN BICKFORD	ENB-1	OESTERLING-TAYLOR PROCESS	1970
HNS-II	1479	756	DELMAR ENGR.	250-7	SYROP PROCESS	1971
HNS-II	1859	NONE	T/McCORMICK SELPH	8203-2	(ACID) PROPRIETARY	1974
HNS-II	1831	NONE	CHEMTRONICS	66-16	(ACID) PROPRIETARY	1974
HNS-II	1981	NONE	CHEMTRONICS	105-5	(ACID) PROPRIETARY	1975

* BULK EXPLOSIVE MATERIALS ARE IDENTIFIED BY EITHER OR BOTH THE ID NO.
AND X NO. AT THE NAVAL SURFACE WEAPONS CENTER (NSWC)

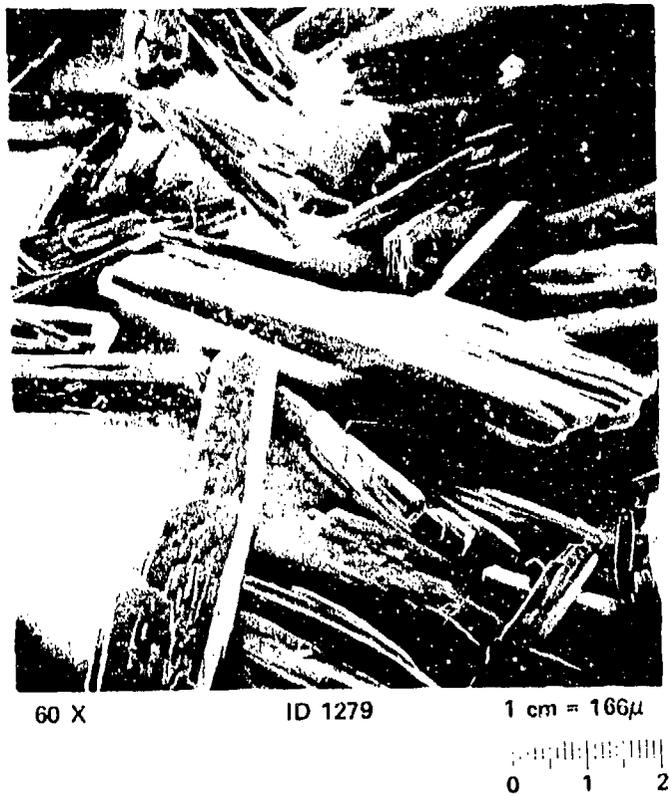


FIG. B-4 SEM PHOTOMICROGRAPH OF ID 1279 HNS-II,
ENSIGN BICKFORD LOT ENB-1

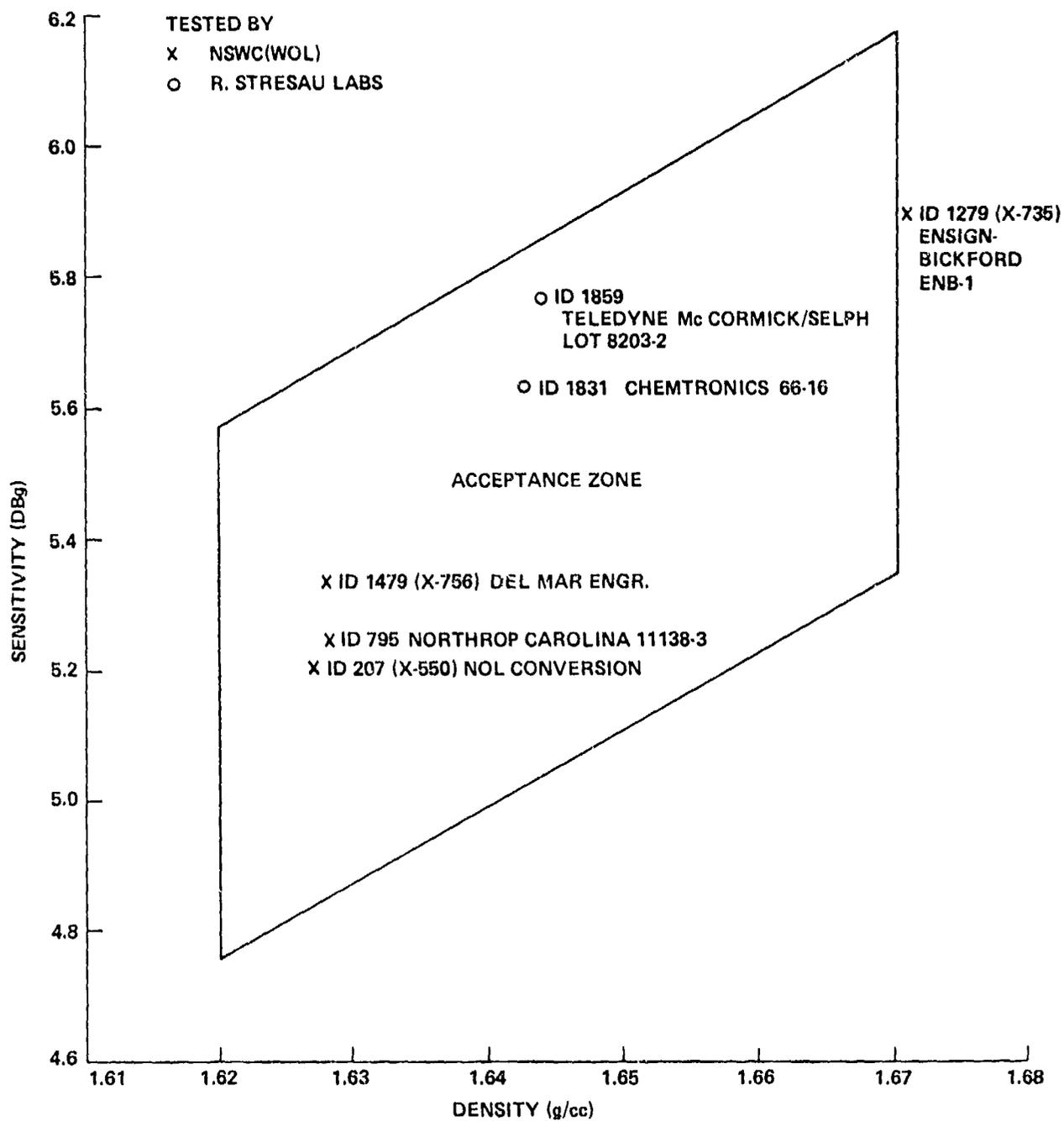


FIG. B-5 WS 5003 ACCEPTANCE CRITERIA FOR SENSITIVITY TEST OF HNS-II-VENDOR QUALIFICATION.



500 X

ID 1479

1 cm = 20 μ



500 X

ID 1479

1 cm = 20 μ



1000 X

ID 1479

1 cm = 10 μ



FIG. B-6 SEM PHOTOMICROGRAPHS OF ID 1479 HNS-II,
DELMAR ENGINEERING LOT 250-7



50 X

ID 1831

1 cm = 200 μ



100 X

ID 1859

1 cm = 100 μ

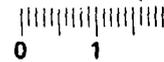


FIG. B-7 SEM PHOTOMICROGRAPHS OF ID 1831, ID 1859 HNS-II, CHEMTRONICS LOT 66-16, TELEDYNE McCORMICK SELPH LOT 8203-2

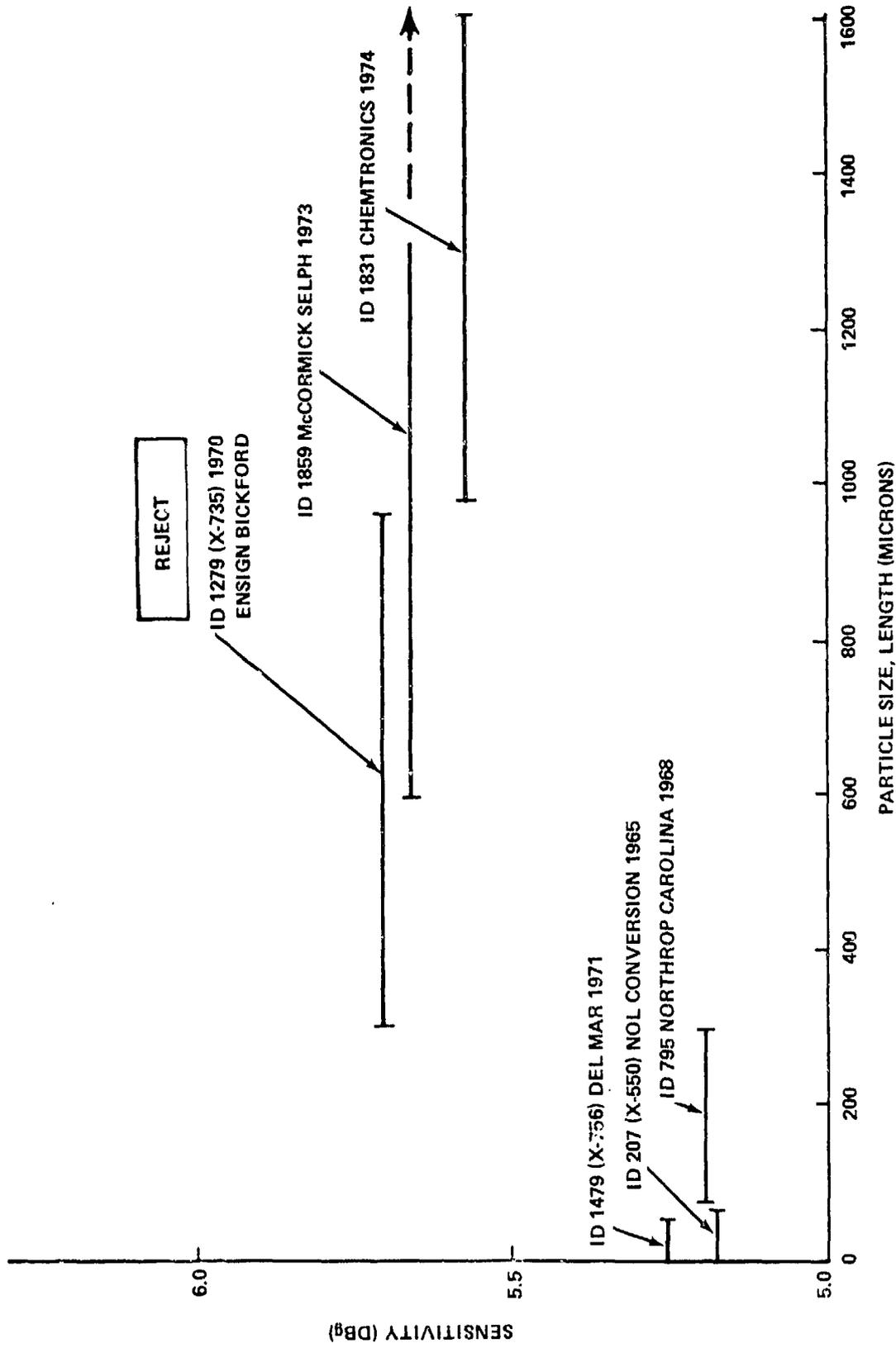


FIG. B-8 THE VARIATION OF PARTICLE SIZE LENGTH WITH SENSITIVITY OF HNS-II

TABLE B-2 THE IDENTIFICATION OF SEVERAL LOTS OF HNS-II AS TO SHOCK SENSITIVITY AND PARTICLE SIZE.

EXPLOSIVE ID #	SENSITIVITY SSGT(DBg)	PARTICLE SIZE RANGE	
		LENGTH μ	THICKNESS μ
1279 (REJECT - WS 5003)	5.878 @ 1.671 g/cc	300-962	75-150
1831	* 5.609 @ 1.6425	980-1607	112-118
1859	* 5.743 @ 1.644	600+	120
795	5.230 @ 1.628	81-300	30
1479	5.322 @ 1.628	50	10-13
207	5.192 @ 1.627	5-63	6

* TESTED BY R. STRESAU LABS

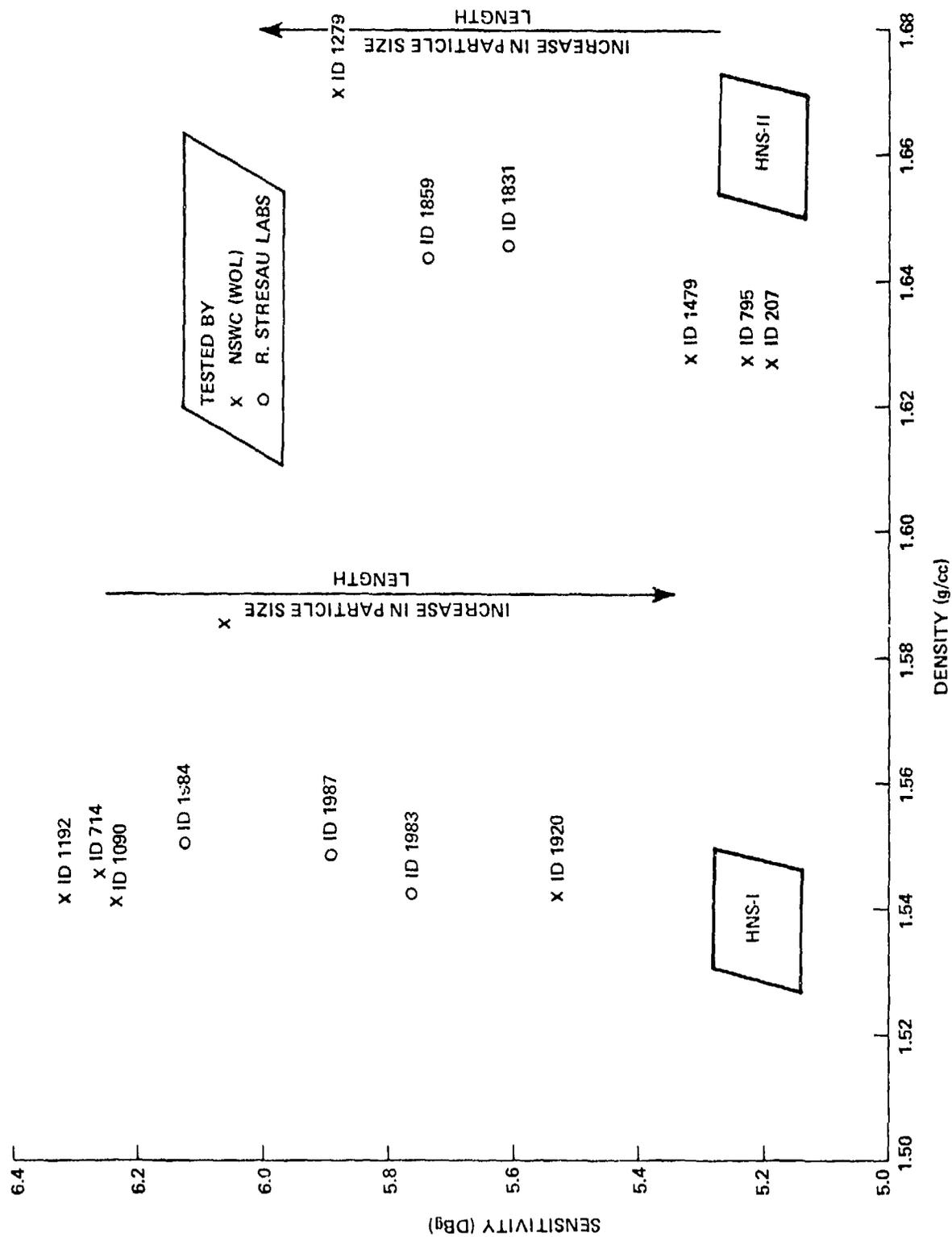


FIG. B-9 SENSITIVITY OF HNS-I AND HNS-II AS A FUNCTION OF DENSITY.

NSWC/WOL/TR 75-142



250 X

ID 1981

1 cm = 40 μ



800 X

ID 1981

1 cm = 13 μ



FIG. B-10 SEM PHOTOMICROGRAPHS OF ID 1981 HNS-II,
CHEMTRONICS LOT 105-5

B-4 In summary the following observations are made:

(a) The Northrop-Carolina (1968) material and the Del Mar Engineering (1971) material are the most nearly alike in properties but the geometries are different because different organic solvents were used in the extraction process.

(b) The particle length (range) affects the sensitivity, and the thickness of the particle also has a significant effect on the sensitivity--but differently from HNS-I: it tends to become more insensitive with these increases.

(c) Although it may not be the case with the "acid solvent material", it is a well known fact that the larger the particle the more the occluded solvent that will be entrapped during the precipitation process.

APPENDIX C

Vacuum Thermal Stabilities of Several HNS-II's

C-1 The vacuum thermal stability test is used to determine the thermal stability or compatibility of materials at various elevated temperatures. The temperature selected for the determination of the thermal stability of the "acid-solvent prep" HNS explosives was 260°C.

C-2 These materials seem to have a good thermal stability except that the Teledyne/McCormick Selph Lot 8203-2 had a relatively large initial gas surge, but it settled down later. Table C-1 gives the results of outgassing of several samples of HNS prepared by the "Organic-Solvent Method" and the "Acid-Solvent Method."

TABLE C-1 THE VACUUM THERMAL STABILITY OF SEVERAL SAMPLES OF HNS-II

<u>SAMPLE</u>	<u>TEMP.,C</u>	<u>PERIOD HRS</u>	<u>SURGE</u>	<u>CC/G</u>	<u>CC/G/HR</u>
(A) ID 1831 CHEMTRONICS LOT 66-16 (HNS-II, ACID PREP)	260	----	20 MIN	0.87	----
ID 1831 CHEMTRONICS LOT 66-16 (HNS-II, ACID PREP)	260	2	----	0.30 *	0.15
(B) ID 777 NORTHROP CAROLINA LOT 11138-20A HNS-II, ORGANIC SOLV. PREP	260	----	20 MIN	0.40	----
ID 777 NORTHROP CAROLINA LOT 11138-20A HNS-II, ORGANIC SOLV. PREP	260	2	----	0.50 *	0.25
(C) ID 1859 McCORMICK SELPH LOT 8203-2 (HNS-II, ACID PREP)	260	----	20 MIN	1.22	----
ID 1859 McCORMICK SELPH LOT 8203-2 (HNS-II, ACID PREP)	260	2	----	0.33 *	0.17
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