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FINAL REPORT

DEVELOPMENT OF ULTRA-FINE AMMONIUM PERCHLORATE TECHNOLOGY

Contract No. FO4611-72-C-0061

September 1973

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Prepared by:

Thiokol / HUNTSVILLE DIVISION
Huntsville, Alabama
A DIVISION OF THIOKOL CHEMICAL CORPORATION

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Air Force Rocket Propulsion Laboratory
Director of Science and Technology
Air Force Systems Command
Edwards Air Force Base, California 93523

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FOREWORD

This program was conducted by Thiokol Chemical Corporation under Contract F04611-72-C-0061 for the Air Force Systems Command, Air Force Rocket Propulsion Laboratory, Edwards Air Force Base, California 93523.

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Program Structure No.	023148
Project Officer	Norman J. Vander Hyde AFRPL/MKPA
Prime Contractor	Thiokol Chemical Corporation Huntsville Division Huntsville, Alabama

Thiokol internal report number 26-73, Control No. U-73-26, has been assigned to this report. The report contains no classified information.

Mr. G. F. Mangum served as Project Director, Dr. M. Miller as Program Manager and Dr. D. A. Flanigan as Principal Investigator. Dr. W. D. Stephens and Mr. S. L. Vance also assisted in the program and in the preparation of this report.

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20 ABSTRACT (Continue on reverse side if necessary and identify by block number)

Ultrafine ammonium perchlorate (UFAP) handling, storage, and shipping properties were investigated with respect to product coatings, solid propellant formulation processing and rocket ballistic parameters. Approximately 40 compounds were evaluated and six materials were identified that protect UFAP from particle growth during storage and handling. It was demonstrated that coated UFAP could be stored for several months under low (30%)R.H. humidity and temperatures to 60°C, but not under high humidity and temperature. Hazard and shipping tests showed UFAP could be shipped as DOT Class B if it contained 30% of "Sorbeads"

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as a "diluent/modifier". Ballistic and processing evaluations showed wide variations in properties with different types and blends of UFAP and different coating materials.

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SUMMARY

Approximately forty (40) compounds were evaluated to identify materials which will protect UFAP from particle growth during storage and handling. Six compounds (n-phenethylaziridine, HX-868, HX-752, Eastman 910, Silanox 101 and Johnson's Wax) were selected in screening tests for a more extensive evaluation. It was demonstrated, as a result of tests on product sensitivity, thermal stability, handling and processing characteristics and capability to serve multi-functional purposes (e.g., coating agent and grinding aid), that the aziridynl compounds were the most effective and versatile and HX-868 was recommended for (and used in) subsequent evaluations. These coatings or their interaction products did not interfere with the Karl Fischer water analysis.

It was demonstrated that coated UFAP could be stored for several months with insignificant particle growth under low humidity (less than 30% RH) conditions and at temperature up to 60°C. Unacceptable high growth was encountered with uncoated UFAP and when samples, either coated or uncoated, were exposed to high humidity - high temperature storage conditions.

Hazards and shipping tests data showed that the coated UFAP would not detonate when packaged with the recommended (30%) desiccant. The data have been submitted for DOT review and shipping classification assignment. It is anticipated that a DOT Class B or Military Class 2 classification will be applicable.

Attempts at determining that physical characteristic or "distribution describing parameter" of UFAP which best correlates the burn rate of propellants containing UFAP were not entirely successful in that at high concentrations of ultra-fine particles, the burn rate correlations failed. The lack of success has been attributed to a lack of dispersibility of the UFAP in the propellant. Correlative trends are more apparent in catalyzed (1% Fe2O3) propellants, but the indication of lack of dispersion is still apparent.

A dramatic effect of humidity upon the ballistic character of propellants containing UFAP was illustrated by manufacturing identical propellants from freshly prepared UFAP, UFAP stored at 10% relative humidity for one week, and UFAP stored at 50% relative humidity for one week. Burn rate decreased with increasing storage humidity and a large increase in viscosity accompanied the higher humidity storage.

The processing characteristics of UFAP prepared by several techniques were evaluated through utilization of viscosity surface response plots. Aziridine coated UFAP prepared from the SWECO Vibro-energy Mill was shown to be more readily processible than equal concentrations of freeze-dried or fluid energy milled UFAP. In several trimodal mixtures containing HX-868 coated UFAP, the regime of minimum viscosity was shown to be formulations containing high concentrations of the UFAP. Physical techniques designed for improved dispersion of UFAP were evaluated with little success.

INTRODUCTION

I. PROGRAM APPROACH

A. OBJECTIVE AND BACKGROUND

The goal of this program was to upgrade the state-of-the-art of UFAP technology to the point that UFAP could be treated, stored, shipped, and utilized according to the best available technique.

Although techniques had been developed for the preparation of ultra-fine ammonium perchlorate (UFAP) and extensive data accrued on the characteristics of the oxidizer and propellant containing this oxidizer, a better definition of the inter-relationships which existed in certain areas was needed. Major areas which needed technical clarification include: Whether a coating (type, how best applied and at what concentration) is needed to achieve UFAP particle size stability over a range of environmental conditions; definition of storage conditions that minimize particle growth; definition of hazards associated with the production, handling and shipping of UFAP, including mode of shipment; influence of UFAP preparation method and particle size on propellant processing, ballistic and physical properties.

B. PROGRAM ORGANIZATION

The program was implemented in the following tasks:

I. Coatings and Coating Techniques

II. Particle Size Growth

III. Water Analysis

IV. Hazards

V. Shipping

VI. Ballistics

VII. Processing

DISCUSSION

TASK I. COATINGS AND COATING TECHNIQUES

Introduction

Task I, coatings and coating techniques, was divided in six subtasks designed to cover the major facets of technology applicable to coatings. Subtask 1 involved a set of screening experiments in which a large number of coating materials were evaluated in survey type tests. These tests were run on three different sizes of fine oxidizer, and the crucial experiments involved particle growth assessment in a humid atmosphere.

Subtask 2 was designed to establish the optimum coating agent level for the best coating materials selected as a result of Subtask 1. In Subtask 3, a number of different methods of applying coating agents to fine oxidizer particles were investigated. The stability of the coating was examined in Subtask 4.

Since coating agents can serve a wide variety of subsidiary purposes in addition to particle growth inhibition, Subtask 5 was designed to evaluate use of the coating to adjust propellant physical properties, processing, sensitivity, combustion characteristics, cure rate, high and low temperature stability, ignition properties and moisture resistance.

Subtask 6, the last subtask in Task I, was essentially a data analysis task, where the results of the preceding subtasks were assembled and analyzed.

Results

Subtask 1 - Initial Screening

Subtask 1 involved a set of screening experiments in which approximately 40 different coating materials were evaluated. The coating materials represented a wide variety of classes of compounds. Ammonium perchlorate samples were solution coated with 2% by weight of these materials (except for Silanox 101 which was coated at 0.2%).

The coated samples were exposed to 45% relative humidity and the particle size measured after 24 hours and after two weeks of exposure. The results of these tests for the various coatings are shown in Tables 1, 2, and 3.

Data analyses consisted of eliminating coating agents which caused caking of the oxidizer or caused other adverse effects. The remaining coatings were ranked according to particle growth at 45% relative humidity. On the basis of this data six of the coating materials were selected for further evaluation. The six materials selected were: Eastman 910, HX-868, HX-752, CW4-019 (an adduct of equal equivalents of MAPO and 12-hydroxy stearic acid), DOW XD 1040, and Silanox 101.

TABLE I

UFAP COATING AND PARTICLE GROWTH STUDY

2% Coating, Exposure 45% RH, 75°F

Coating Material	EFEM-5 (1.75 Å m) 24 hrs	EFEM-5 (1.75 Å m) 14 days	ULM-68 (0.37 Å m) 24 hrs	ULM-68 (0.37 Å m) 14 days	SVM-266 (0.78 Å m) 24 hrs	SVM-266 (0.78 Å m) 14 days
Eastman 910	1.81	1.73	0.42	0.41	0.77	0.79
Johnson Paste Wax	1.89	1.92	0.38	0.53	0.76	0.83
HX-760	1.88	1.73	0.39	0.39	0.90	0.80
HX-868	1.89	1.63	0.39	0.42	0.81	0.82
HX-752	1.75	1.72	0.43	0.43	0.79	0.82
"3-M", L-2483	8.30	----	----	----	----	----
"3-M", L-2650	2.13	2.28	0.50	0.48	0.80	1.06
Chemigum, Nitrile Latex	3.02	----	----	----	----	----
CW4-019	2.00	1.86	0.48	0.44	0.83	0.80
Monozoline O	2.45	2.37	0.68	----	----	----
"3-M", L-1606	2.28	3.25	----	----	----	----
Dow Corning 550 Fluid	2.35	2.42	0.45	0.49	0.78	0.88
Dow Corning 1200 Primer	3.5	Could not measure.		----	----	----
GE SF-99 Silicone Fluid	2.38	3.38	----	----	----	----
Gulfwax, Paraffin Wax	2.34	3.38	----	----	----	----
Goodyear, Pliobond HT-30	4.15	----	----	----	----	----
Chemlok 231	Could not Determine particle size			----	----	----

TABLE 1. (Continued)

UFAP COATING AND PARTICLE GROWTH STUDY

2% Coating, Exposure 45% RH, 75°F

Coating Material	EFEM-5 (1.75 μm) 24 hrs	EFEM-5 (1.75 μm) 14 days	ULM-68 (0.37 μm) 24 hrs	ULM-68 (0.37 μm) 14 days	SVM-266 (0.78 μm) 24 hrs	SVM-266 (0.78 μm) 14 days
Chemlok 205	4.22	----	----	----	----	----
Solvent Cement for PVC Pipe	6.86	----	----	----	----	----
N-Lauryl Methacrylate	2.10	2.50	0.47	0.53	0.97	1.08
Halocarbon 25-5S Grease	2.52	2.87	0.53	----	----	----
Strodex PK-90	2.05	2.21	0.39	0.50	0.75	0.98
Polyox FRA	(Rubbery Lumps-No Test Conducted)			----	----	----
EMCOL 4200	2.87	2.82	1.15	----	----	----
Chemlok 607	2.40	2.38	0.87	----	----	----
Chemlok 218	Could not measure		----	----	----	----
Methyl Methacrylate	2.50	3.57	----	----	----	----
Ethyl Acrylate	2.70	3.36	----	----	----	----
VYLF	6.10	----	----	----	----	----
Silanox 101	Could not measure		----	----	----	----
DOW XD-1040	1.80	1.56	0.43	0.42	0.70	0.86
EMCOL-G MOP	Could not measure		----	----	----	----
EMCOL-511	10.6	----	----	----	----	----
EMCOL-5120	10.6	----	----	----	----	----

TABLE I. (Continued)

UFAP COATING AND PARTICLE GROWTH STUDY

2% Coating, Exposure 45% RH, 75°F

Coating Material	EFEM-5 (1.75 λm) 24 hrs	EFEM-5 (1.75 λm) 14 days	ULM-68 (0.37 λm) 24 hrs	ULM-68 (0.37 λm) 14 days	SVM-266 (0.78 λm) 24 hrs	SVM-266 (0.78 λm) 14 days
SPAN 85	1.93	2.06	0.47	0.60	0.85	0.97
SPAN 60	7.2	----	----	----	----	----
SPAN 40	7.0	----	----	----	----	----
Octadecylamine	7	----	----	----	----	----
Monozoline O Stearate	9	----	----	----	----	----
HX-874	2.73	2.67	0.48	0.58	Could not measure	
CAB-O-SIL	2.35	2.59	0.45	0.52	0.92	0.89
Standard, No Coating (Mixed with Benzene, Heated and Dried)	2.12	----	0.46	0.58	1.04	----
			Coated 0.2% By Weight			
Silanox 101			0.50	0.48	0.87	0.89
	Unexposed Samples		Unexposed Samples		Unexposed Samples	
Standard, No Coating (Mixed with Benzene, Heated and Dried)	1.75		0.37		0.78	
NH_4ClO_4 As Received	1.86		0.39		0.70	

Subtask 2. Coating Agent Level

The coating materials were evaluated at three different levels to determine the preferred level and are presented in Table 2. The data is represented graphically in Figure 1. The differences are small but there does seem to be a trend toward less growth with heavier coatings. It cannot be said positively that this trend is not within experimental error.

The sensitivity data for AP coated with the six materials also appears in Table 2. It can be concluded that no extreme sensitivity is incurred by the coatings.

Subtask 3. Application Methods

Subtask 3 was designed to investigate methods of applying coating agents to the AP. The base-line method was to suspend the AP in a solvent containing the coating material, then slowly evaporate the solvent. In this subtask other methods were evaluated.

A second method evaluated involved dissolving the coating agent in an organic solvent, adding the oxidizer and stirring or warming and allowing the solution to stand while the reactive coating diffuses to the oxidizer surface and attaches itself through chemical reaction with the oxidizer or homopolymerizes on the surface of the oxidizer under catalysis by the ammonium perchlorate. Afterwards the oxidizer is filtered off and dried. The particle size data obtained using this method appears in Table 3. It must be noted that the filtering process appears to allow the small AP particles to pass through and only collect the larger particles.

A third technique investigated was the vapor deposition of the coating material onto the oxidizer surface. This technique is limited to the use of coating agents which have high enough vapor pressure and which will not decompose or polymerize under vaporization conditions. The only coating agent which could be applied by this technique was carnauba wax, the major high boiling constituent of Johnson's paste wax. The presence of the coating on the AP was verified by working the AP with an organic solvent and examination of the solvent containing the extracted material by infrared spectrometry. It can be seen from Table 3 that this is an efficient method of coating oxidizer. The major advantage with a techniques of this type is the ability to coat AP without involving a solvent or lengthy drying period.

A fourth method of coating was eliminated because of experimental difficulties. This involves spraying an organic solution of the coating material on the oxidizer which is stirred or agitated. In attempting to spray fine AP, much of the oxidizer is blown away. Spraying in a closed system results in clogging of the apparatus. Because of these difficulties the method was abandoned.

TABLE 2

COATED UFAP DATA

Coating Material	Particle Size (microns) of coated ULM-68 after exposure to 45% RH and 75°F for 14 days[1] Coating (percent by Wt.) 2.0	1.0	0.3	Sensitivity data on ULM-68 coated 2% by weight Impact (kg-cm)	Friction (lbs)	Spark (joules)
Eastman 910	0.41	0.42	0.43	(-) 250	(-) 90	(-) 9 (+) 16
Johnson Paste Wax	0.53	0.49	0.52	(-) 130 (+) 140	(-) 90	(-) 9 (+) 16
HX 868	0.42	0.38	0.41	(-) 152 (+) 160	(-) 90	(-) 9 (+) 16
HX 752	0.43	0.47	0.52	(-) 150 (+) 160	(-) 90	(-) 9 (+) 16
CW4 - 019	0.44	0.45	0.50	(-) 150 (+) 175	(-) 90	(-) 9 (+) 16
DOW XD - 1040	0.42	0.49	0.48	(-) 152 (+) 160	(-) 90	(-) 9 (+) 16
	Coating (percent by Wt.) 0.2	0.1	0.05	Coated 0.2% by Wt.		
Silanox 101	0.48	0.55	0.50	(-) 140 (+) 150	(-) 90	(-) 9 (+) 16
NH_4ClO_4, as received (ULM - 68, See Note 1)				(-) 200 (+) 250	(-) 90	(-) 25
Standard, No coating (Mixed with benzene, heated and dried)			0.58[2]			

Notes:

1. Particle Size of ULM - 68

Date	Microns	Remarks
4/13/72	0.38	Start of 2% coatings
4/6/72	0.39	
5/8/72	0.43	Start of reduced level coatings

2. Uncoated material grew from 0.43 to 0.58 during the same period.

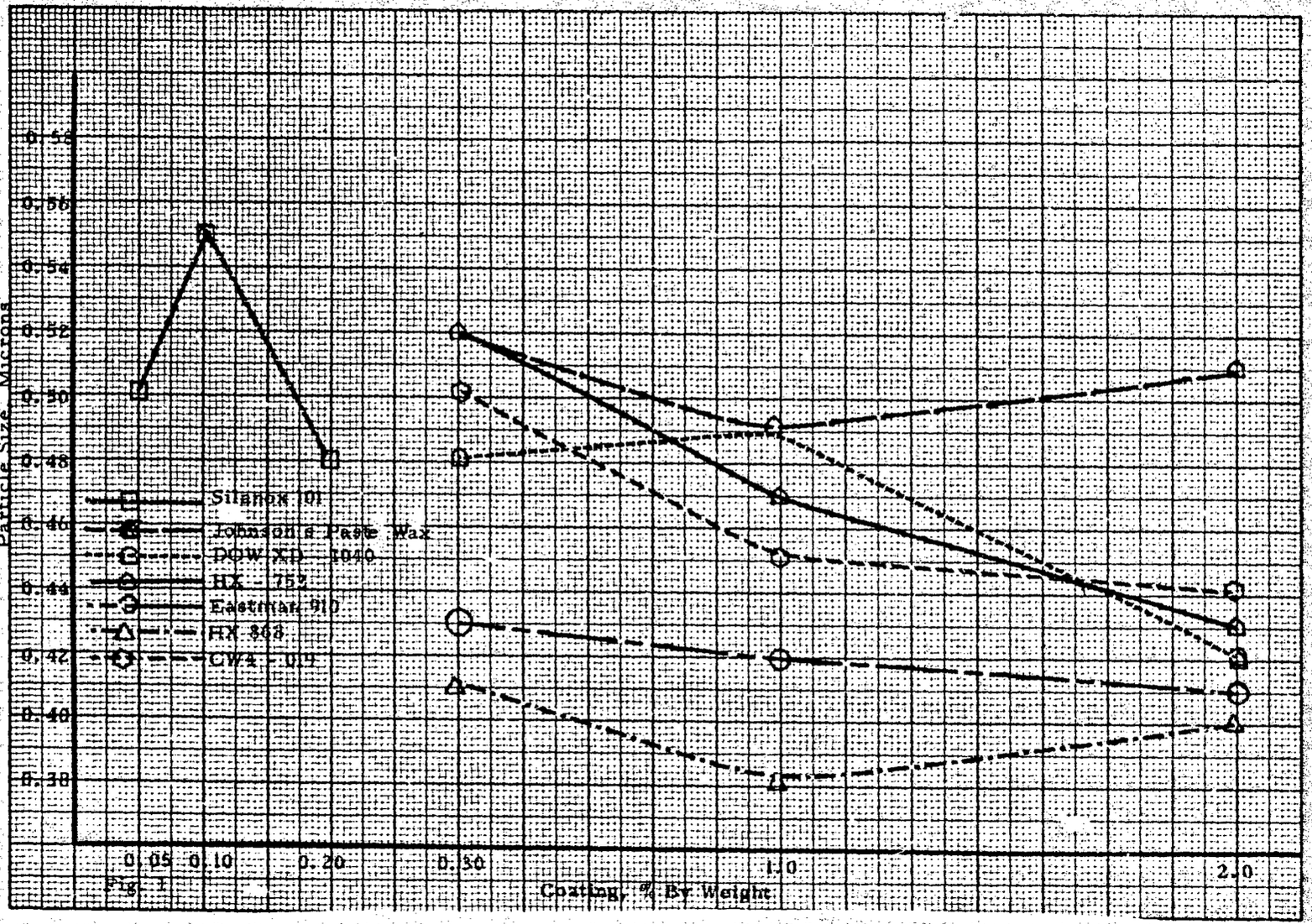


Fig. 1

Figure 1. Effect of Coating Agent Concentration on AP Particle Growth

TABLE 3

COATING METHOD STUDY

2% Coating, [1] 24 Hr. Exposure 45% RH, 75°F

COATING MATERIAL	COATING METHOD		
	Solution Deposition	Ball Milled	Vapor Deposition
Control	1.39		
HX 868	1.23		
HX 752	1.43		
CW 4-019	0.77		
Carnauba Wax	1.47		1.07
Silanox 101	1.85	1.31	

(1) AP samples were all taken from batch ULM-68 particle 0.47μ

The fifth method is applicable to solid antiagglomerates and involves ball milling with the oxidizer. This method was evaluated using Silanox 101. A sample of AP was ball milled with 0.2% of Silanox 101 and samples withdrawn at two, four, and six hours. Particle size data from aging samples for 24 hours at 45% RH are shown in Table 4.

Coated particle growth rate, at 45% RH, was used to assess the efficiency of the coating on the products prepared by the various techniques. Deposition from solution while evaporating the solvent was determined to be the most efficient method of coating oxidizer samples. It must be mentioned at this, however, that vapor deposition under a vacuum is still very advantageous if one had a coating material applicable to this method.

Subtask 4. Coating Stability

This subtask was designed to evaluate the stability of the coating on the oxidizer. This would determine if a coating can withstand the rigors of normal handling or operations.

In order to evaluate the effects of organic solvent vapor on the coated oxidizer, samples of coated oxidizer were exposed to acetone and benzene vapors in a closed vessel. After this the samples were put in storage at 45% RH. Measurements of particle size growth relative to coated oxidizer not exposed to organic vapors is interpreted as a measure of the stability of the coating to organic vapors. Particle size measurements resulting from this experiment are shown in Table 4. It can be concluded that polar vapors, represented by acetone, are much worse than the non-polar, represented by benzene.

Resistance of the coatings to organic liquids was determined by rapidly washing a sample of coated oxidizer on a Buchner Funnel and drying the oxidizer under a vacuum. After exposure to 45% RH the oxidizer particle size was measured. Again, the particle growth was interpreted as a measure of coating stability. The results are shown in Table 4. None of the coating will stand up to liquid solvent treatment.

Abrasive resistance of the coating was evaluated by ball milling the coated oxidizer with and without balls for one hour. These samples were then exposed to 45% RH for 24 hours at 25°C. Figure 2 shows the results of the abrasive test. It can be seen that Eastman 910 and CW4-019 have the best abrasive resistant properties. Temperature effects are shown in Figure 3. It is concluded that HX-868 and HX-752 are preferred as far as low temperature hardening of the coating and high temperature agglomeration.

TABLE 4

RESISTANCE TO ORGANIC LIQUIDS AND VAPORS

2% Coating[2], 24 Hour Exposure to 45% RH, 75°F

Coating Material	Benzene Wash	Acetone Wash	Benzene Vapors	Acetone Vapors
HX-868	1.12	2.78	0.39	2.85
HX-752	1.52	2.82	0.49	4.3
CW4-019	2.45	1.70	0.474	1.39
Johnson's Paste Wax	1.92	2.38	0.60	3.10
Silanox 101	4.0	4.0	0.575	3.20
DOW XD 1040	1.22	3.81	0.73	AGL[1]
Eastman 910	3.55	3.02	0.436	2.95

[1] Agglomerated into a few large, hard lumps.

[2] AP samples were all taken from batch ULM-68 particle 0.47μ.

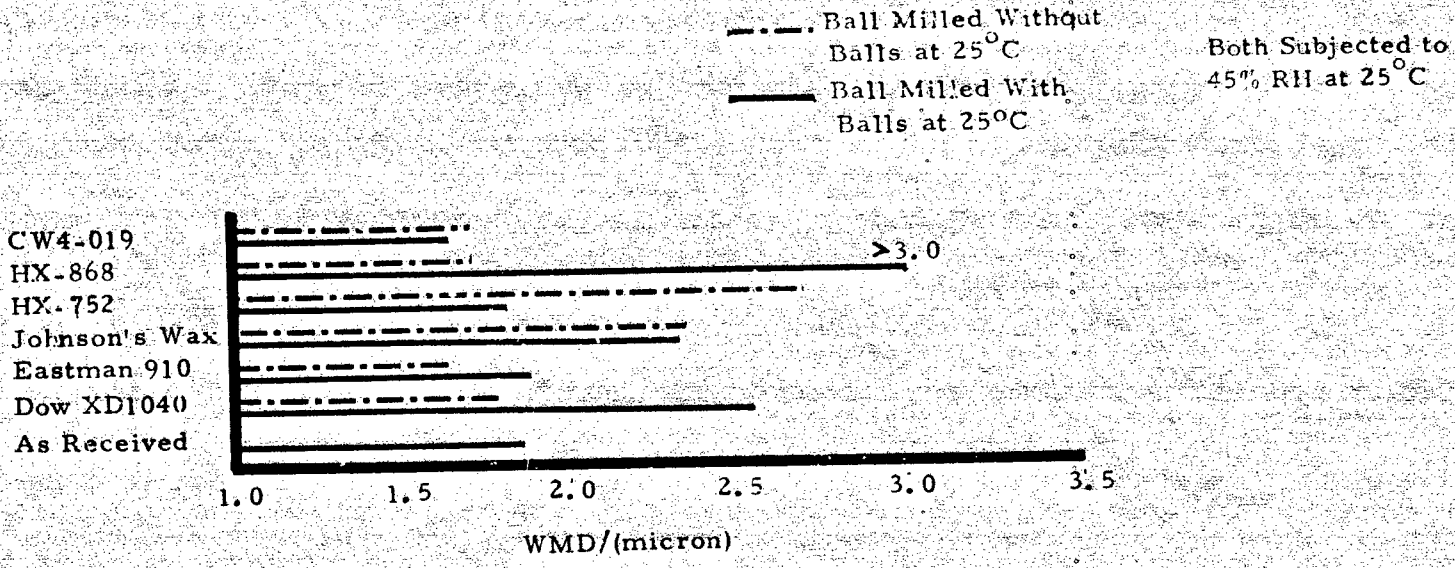


Figure 2. Effect of Abrasion on Coatings

Subtask 5. Multipurpose Coatings

1. Propellant Properties

The objective of this study was to determine the rheological properties of pseudo-propellant formulations containing precoated ultra-fine oxidizer. Past experience has indicated that solid-binder interaction plays a significant part in the rheological properties of uncured propellant formulations. Therefore, this test was designed to determine if the coatings on the ultra-fine oxidizer would detrimentally change the rheological properties of uncured pseudo-propellant formulations. The coated oxidizers were selected from the previous screening test series.

Fifteen gram pseudo-propellant mixes were prepared as described in Table 5. Before the experimentally coated oxidizers were formulated, two standardization mixes were prepared and tested.

All tests were determined on the Haake Rotovisko viscometer at +145°F. The mixes were tested at various shear rates and the resulting shear stress recorded. Rheograms were prepared by plotting shear rate versus shear stress and shear rate versus viscosity. (See Figures 4 through 18.) The individual mixes are plotted separately on Figures 4 through 14 and composite rheograms on Figures 15 through 18.

The coating materials are listed and identified in Table 6. The coating material was applied to the oxidizer at 1.0% by weight.

The materials specified in Table 5 were blended over a 1/2 hour period then heated to +145°F and stirred intermittently for approximately three hours. This three-hour period allowed for complete wetting and stabilizing of the mix at 145°F. This mix was then placed in the Haake viscometer and tested at +145°F. The mix was stored at +145°F for 24 hours, stirred thoroughly and again tested on the Haake viscometer. The results of the "3-hour" test and the "24-hour" test are presented on the same figure. It becomes immediately evident when reviewing the figures that the mixes change rheological properties between the 3-hour test and the 24-hour test. Generally, the mixes drop in viscosity over this time period and change from a pseudoplastic material with a yield value to an almost Newtonian material without a yield value or with a very low yield value.

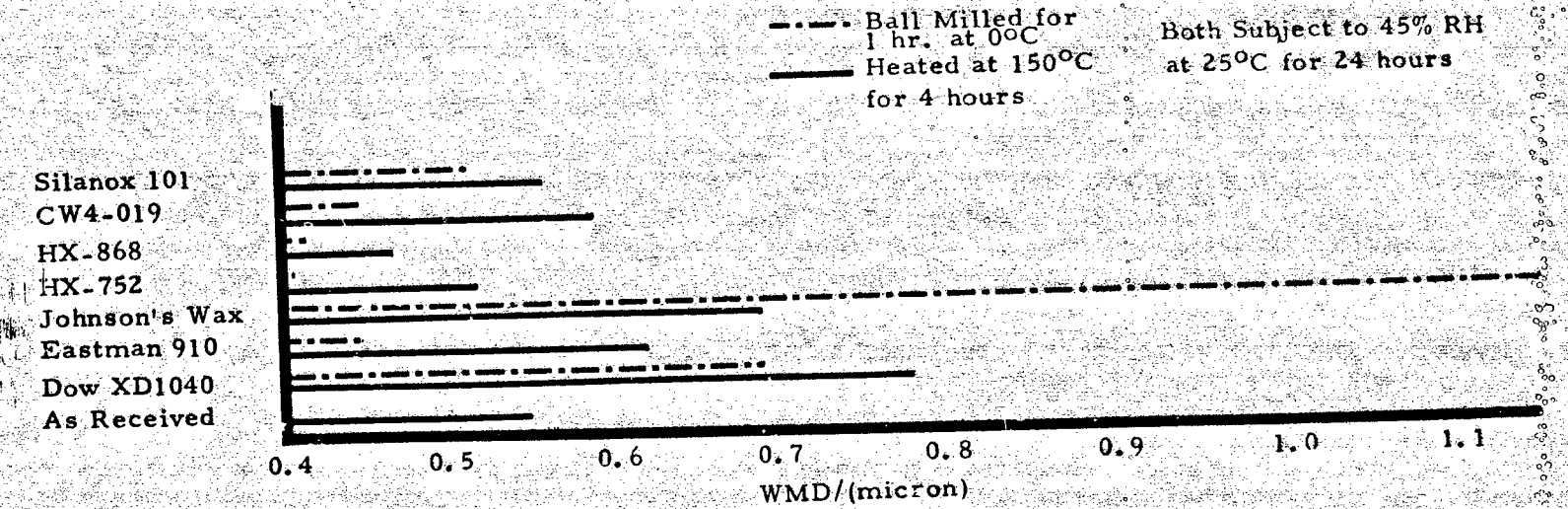


Figure3. Effect of Temperature on Coatings

TABLE 5

EXPERIMENTAL COATING, 15-GRAM MIXES

Add Order	Materials	Amounts
1	HTPB (R45M, lot AA80)	30.00%
2	Al (H-10, lot 3980)	10.00%
4	AP (50 μ, lot 4086)	26.67%
3	AP (0.43 μ, lot ULM-68)	33.33% coated*

*NOTES: 1 - ULM-68 is "pre-coated" with 0.4% HX-874 used as a grinding aid. This material is then "over-coated" for the immediate study.

2 - Oxidizer lot VMA-91 used in two of the mixes is "pre-coated" with 0.4% HX-868 used as a grinding aid.

HX-874: [Tris 2, 4, 6-(1 butylazirindyl) 1, 3, 5 trizine]

HX-868: Trimesoyl 1-(2-ethyl) aziridine

TABLE 6

COATING MATERIALS

Identification	
#1A	Eastman 910: Cyano acrylate
#2A	Johnson Paste Wax: major constituent is carnauba wax
#4A	HX-868: Trimesoyl 1(2-ethyl) aziridine
#5A	HX-752: Bis-isophthaloyl (1-2 methyl) aziridine
#9A	CW 4-019: Reaction product of MAPO and Hydroxy Stearic Acid
#31A	Dow XD-1040: N-phenethyl aziridine
#43A	Silanox 101: Hydrophobic Fumed Silica

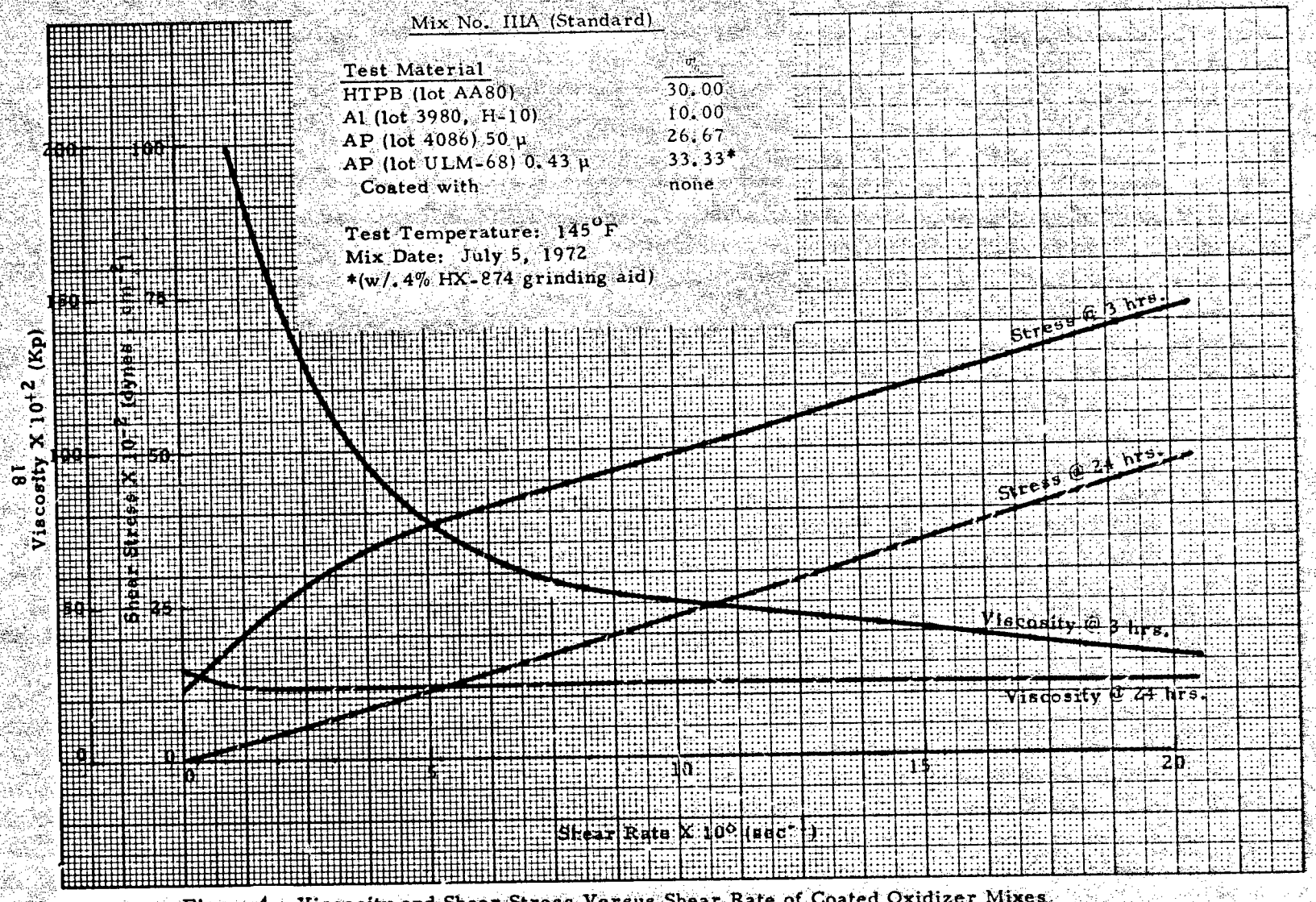


Figure 4. Viscosity and Shear Stress Versus Shear Rate of Coated Oxidizer Mixes

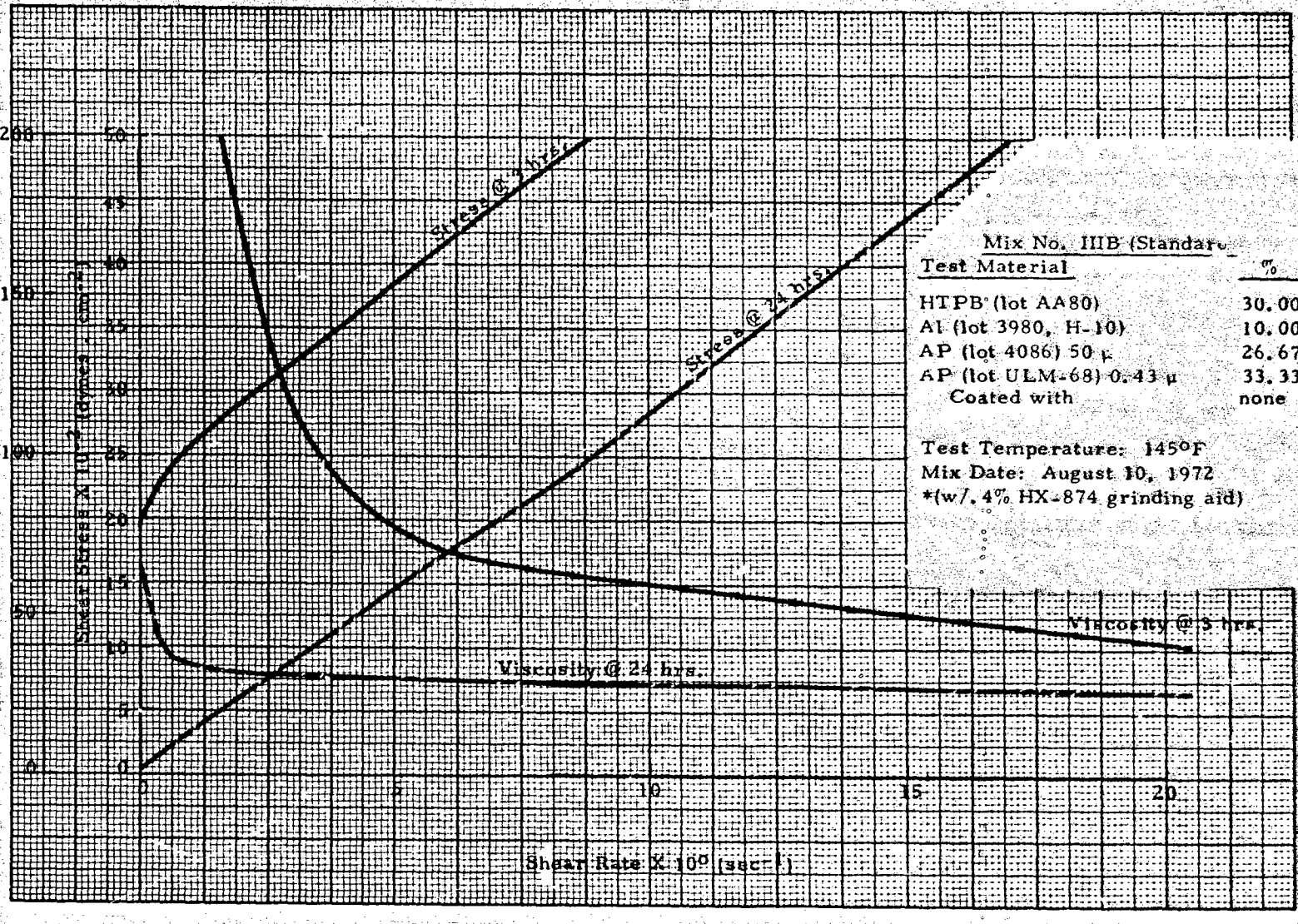


Figure 5. Viscosity and Shear Stress Versus Shear Rate of Coated Oxidizer Mixes

Mix No. 1A

Test Material	%
HTPB (lot AA80)	30.00
Al (lot 3980, H-10)	10.00
AP (lot 4086) 50 μ	26.67
AP (lot ULM-68) 0.43 μ	33.33 (w/. 4% HX-874 grinding aid)
Coated with	Eastman 910

Test Temperature: 145°F
Mix Date: July 31, 1972

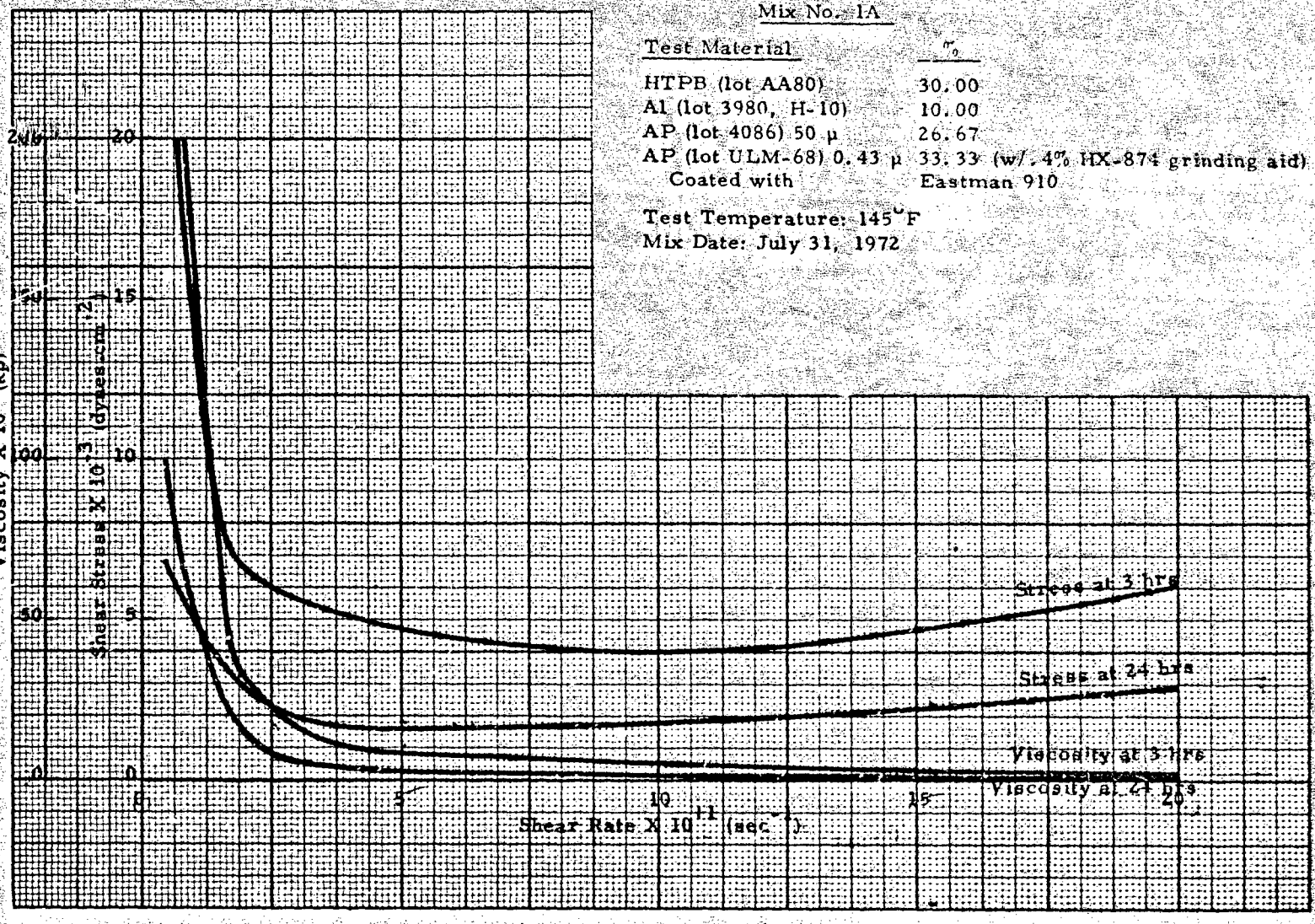


Figure 6. Viscosity and Shear Stress Versus Shear Rate of Coated Oxidizer Mixes.

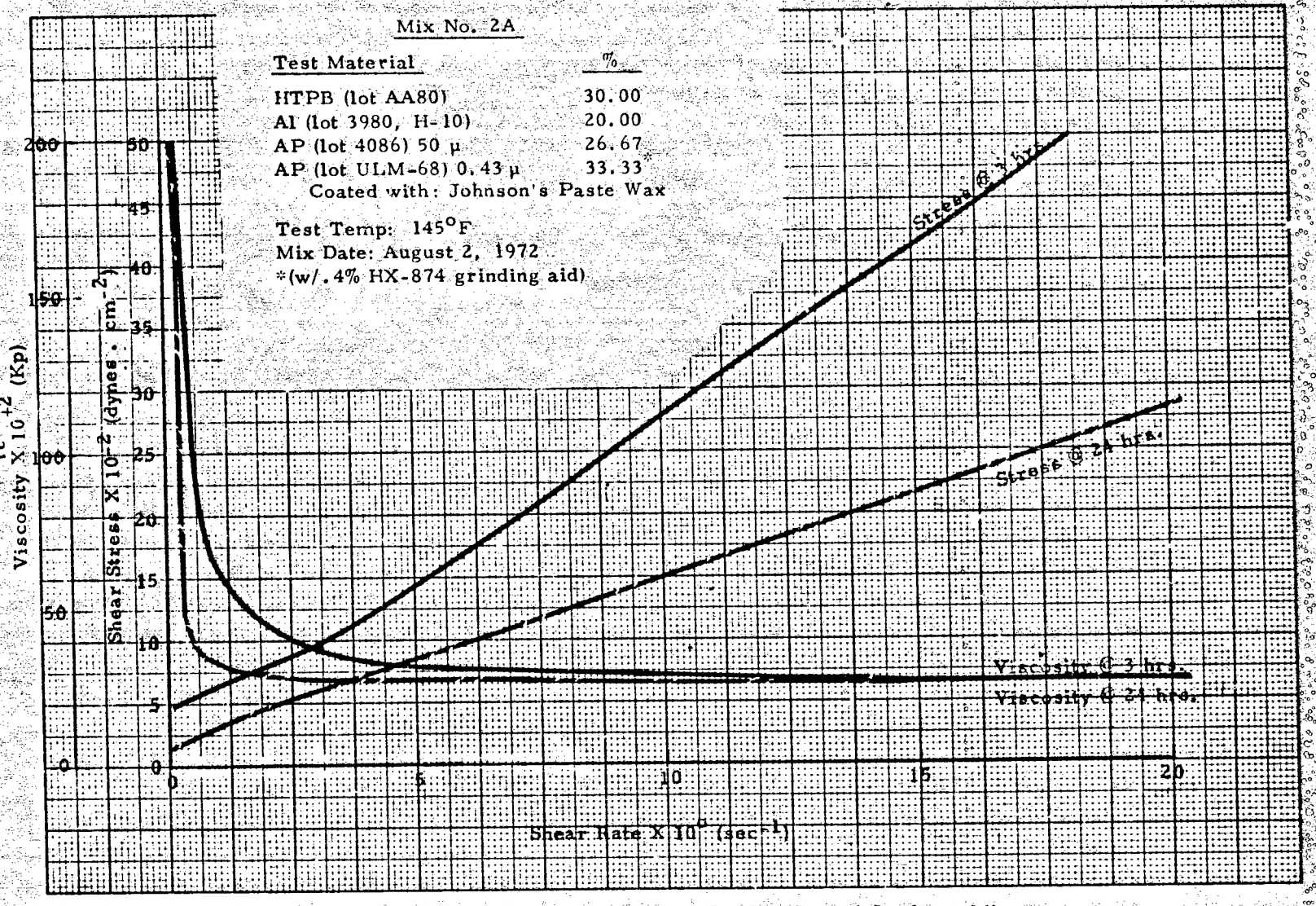


Figure 7. Viscosity and Shear Stress Versus Shear Rate of Coated Oxidizer Mixes

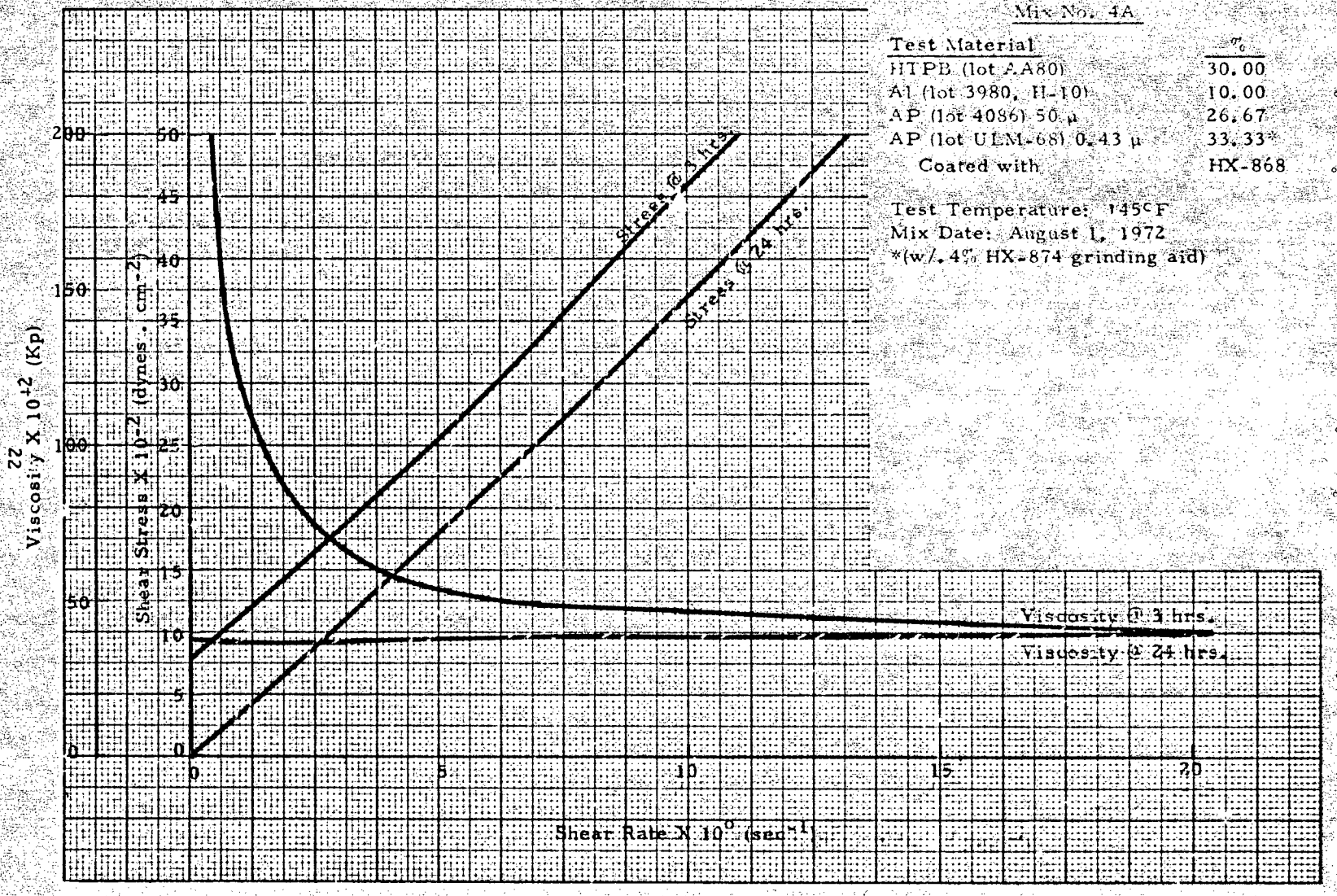


Figure 8. Viscosity and Shear Stress Versus Shear Rate of Coated Oxidizer Mixes

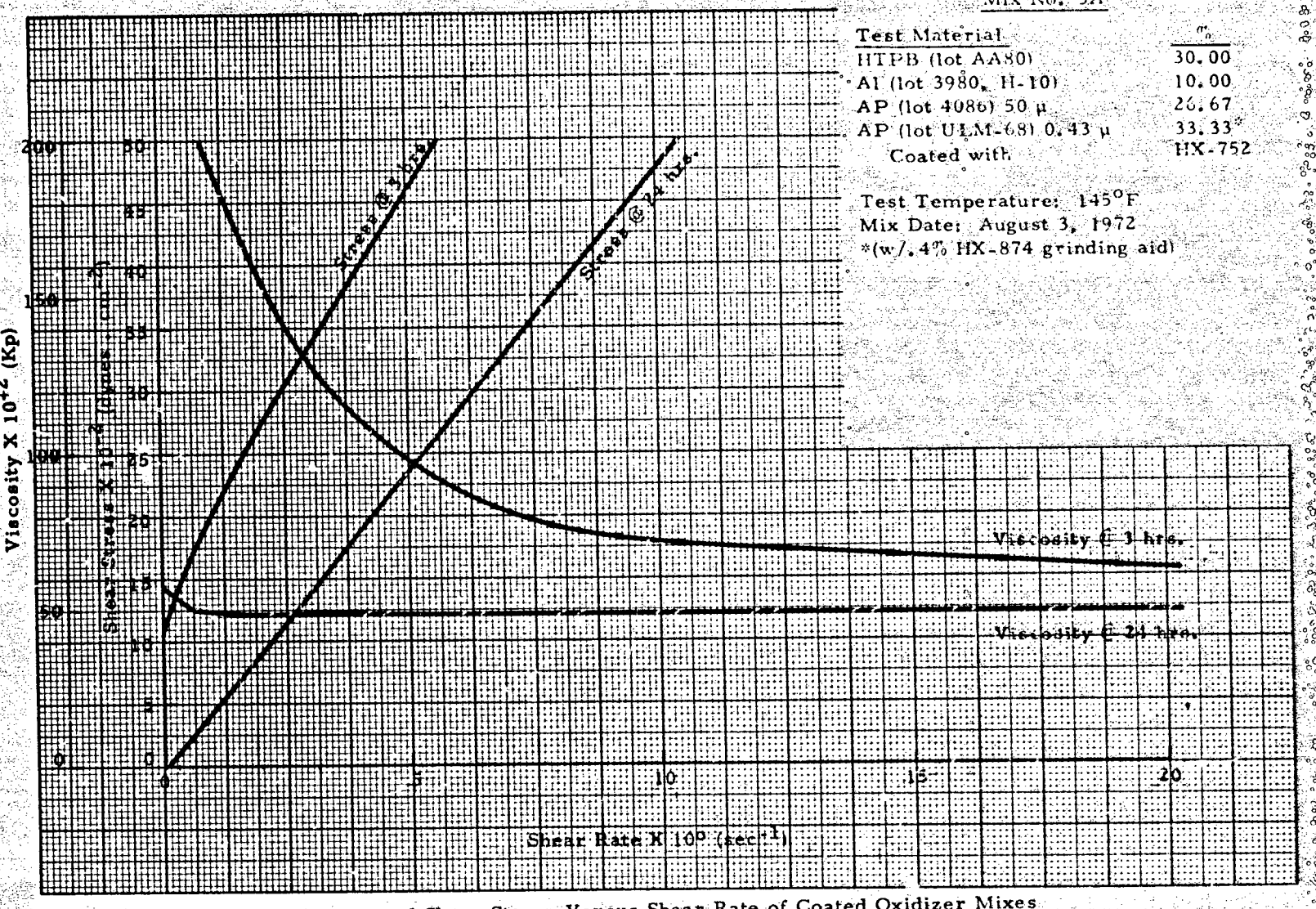


Figure 9. Viscosity and Shear Stress Versus Shear Rate of Coated Oxidizer Mixes

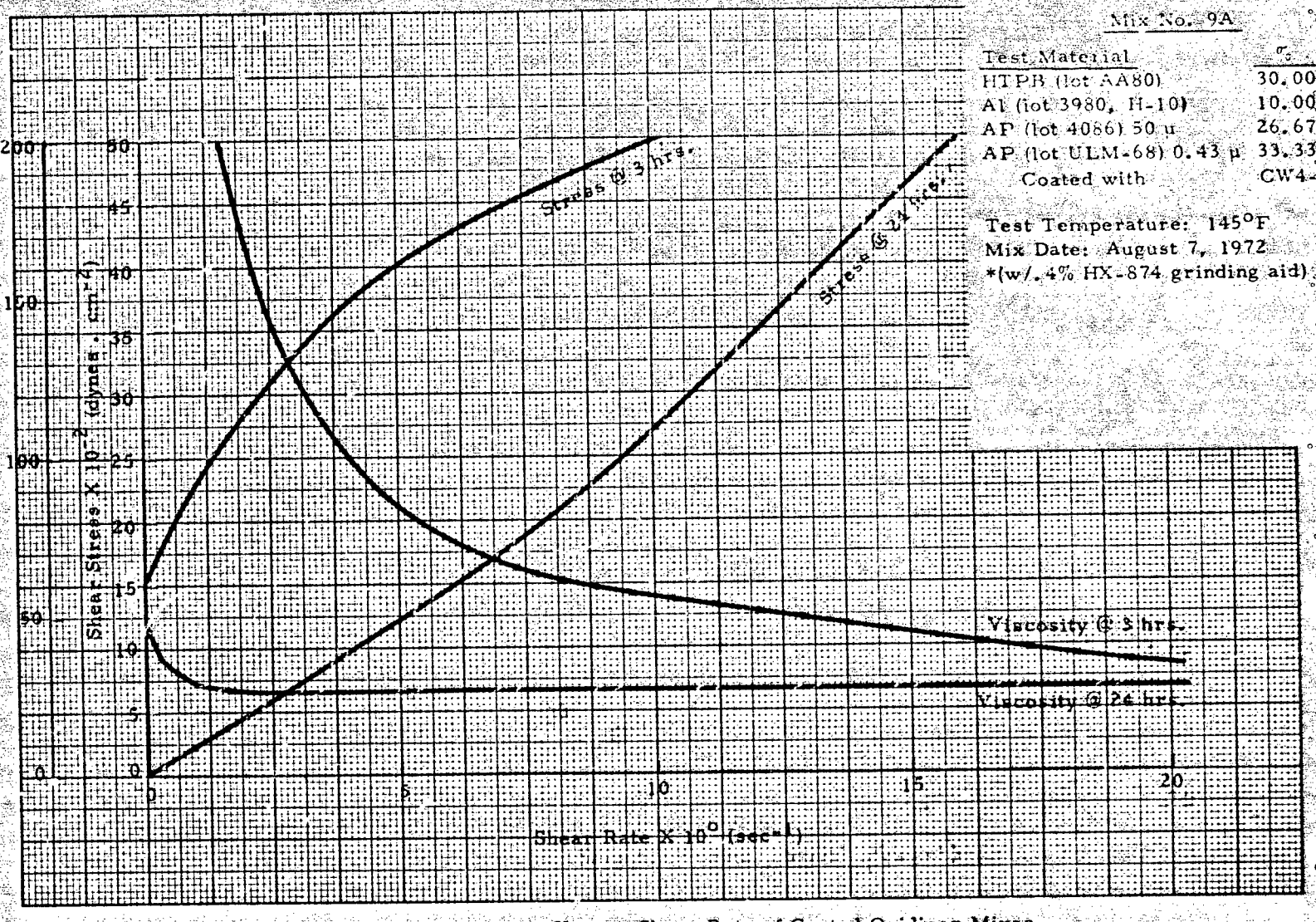


Figure 10. Viscosity and Shear Stress Versus Shear Rate of Coated Oxidizer Mixes

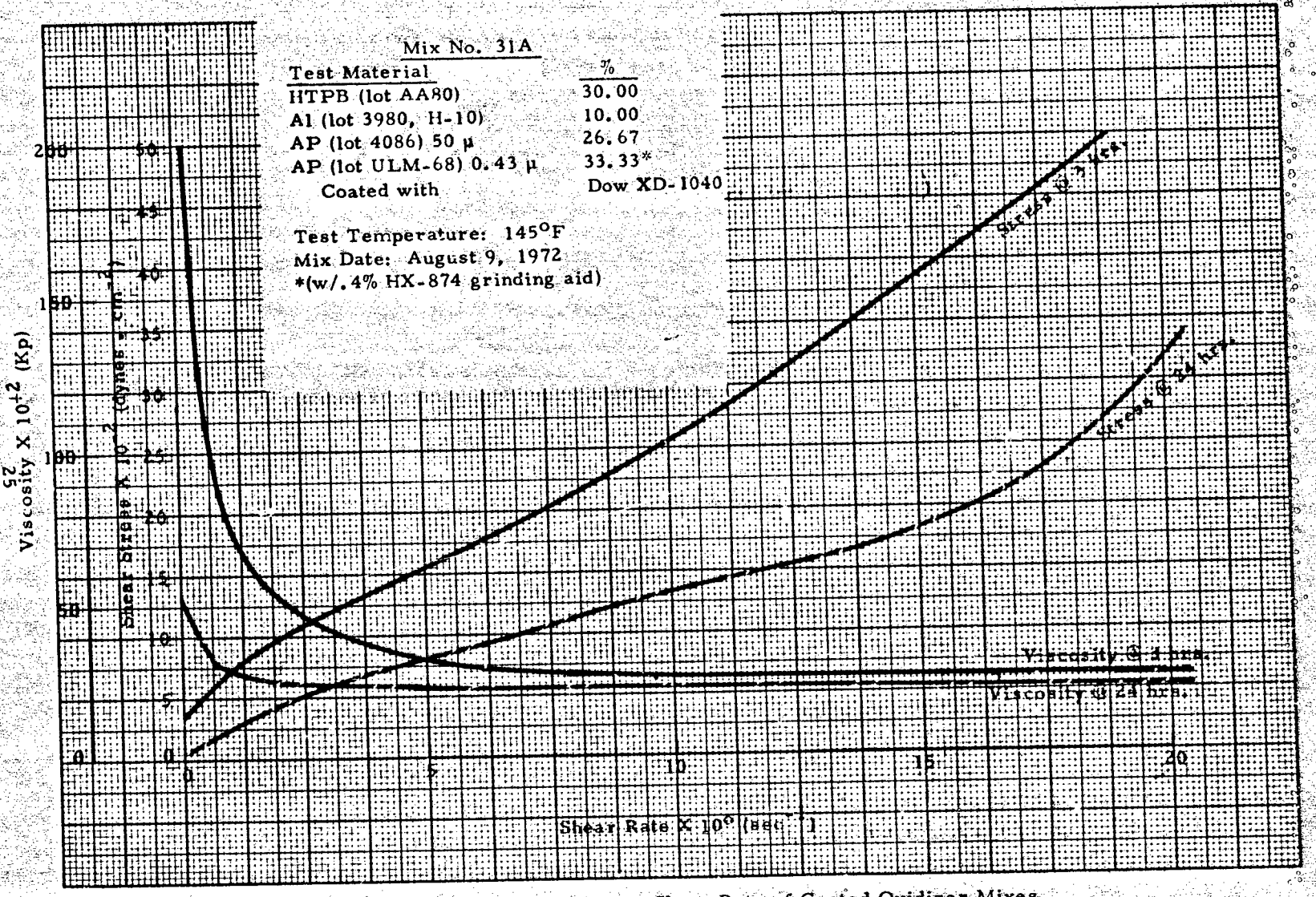


Figure 11. Viscosity and Shear Stress Versus Shear Rate of Coated Oxidizer Mixes

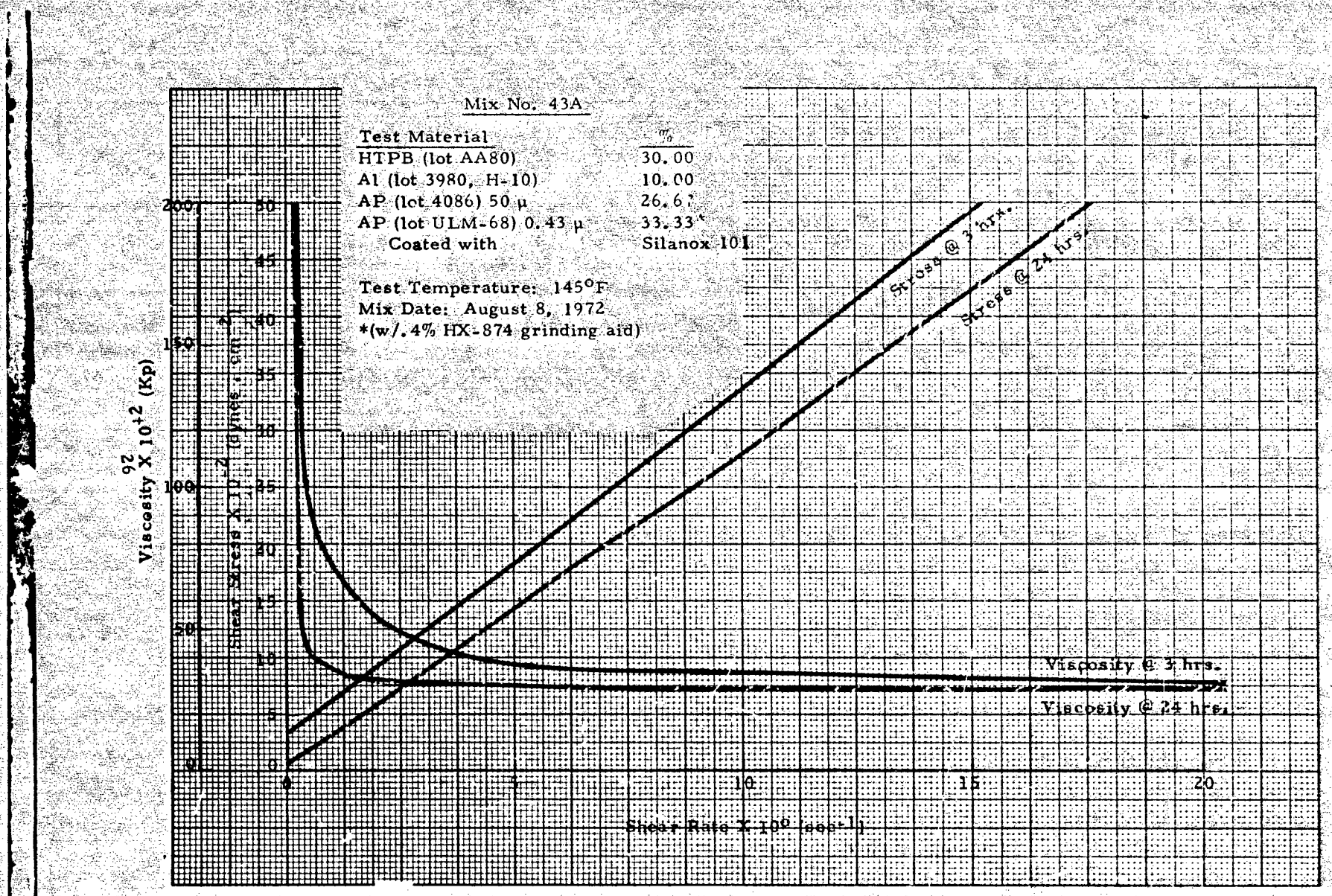


Figure 12. Viscosity and Shear Stress Versus Shear Rate of Coated Oxidizer Mixes

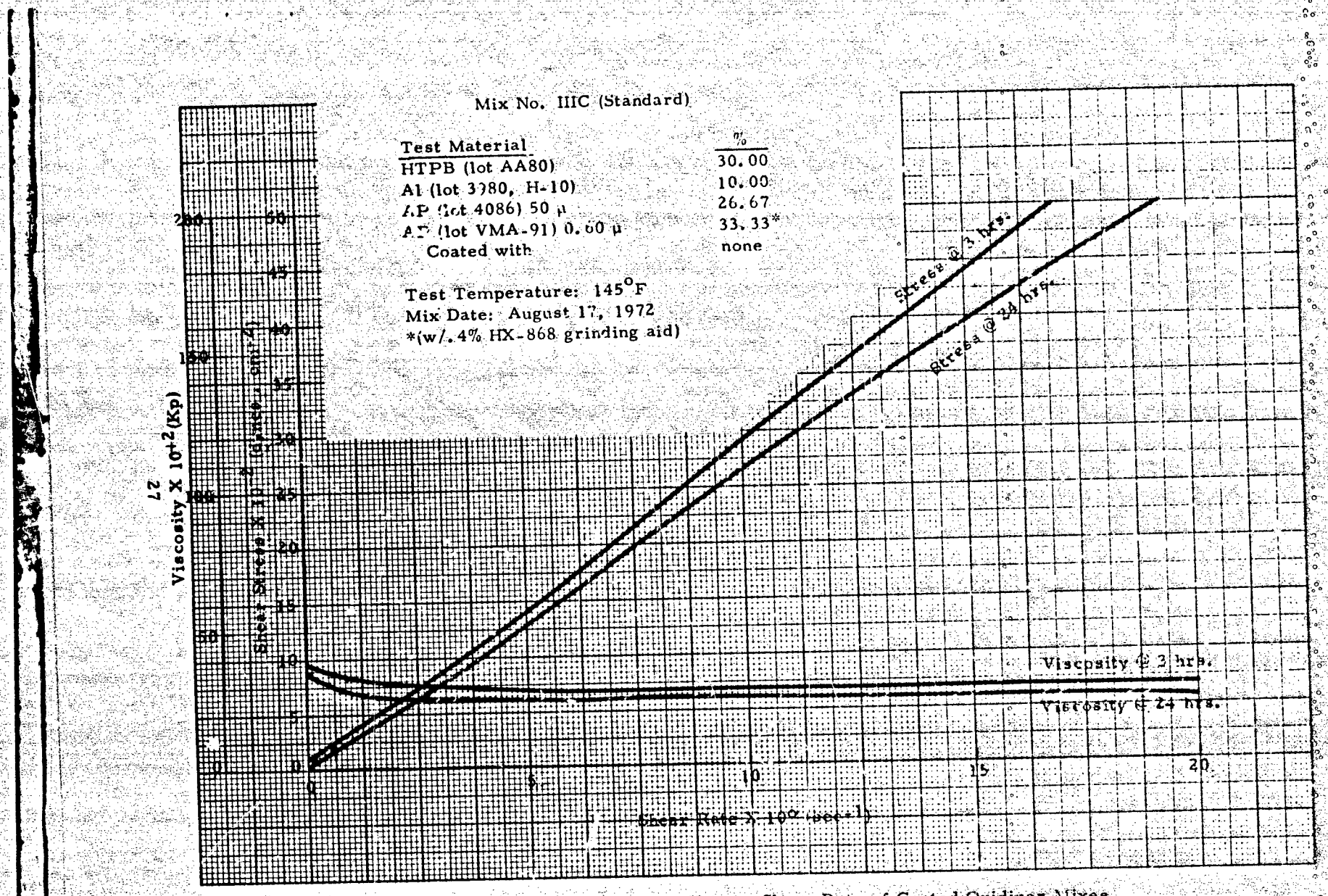


Figure 13. Viscosity and Shear Stress Versus Shear Rate of Coated Oxidizer Mixes

Mix No. 1A-R

Test Material	%
HTPB (lot AA80)	30.00
Al (lot 3980, H-10)	10.00
AP (lot 4086) 50 μ	26.67
AP (lot VMA-91) 0.60 μ	33.33 (w/.4% HX-868 grinding aid)
Coated with	Eastman 910

Test Temperature: -145°F
Mix Date: August 17, 1972

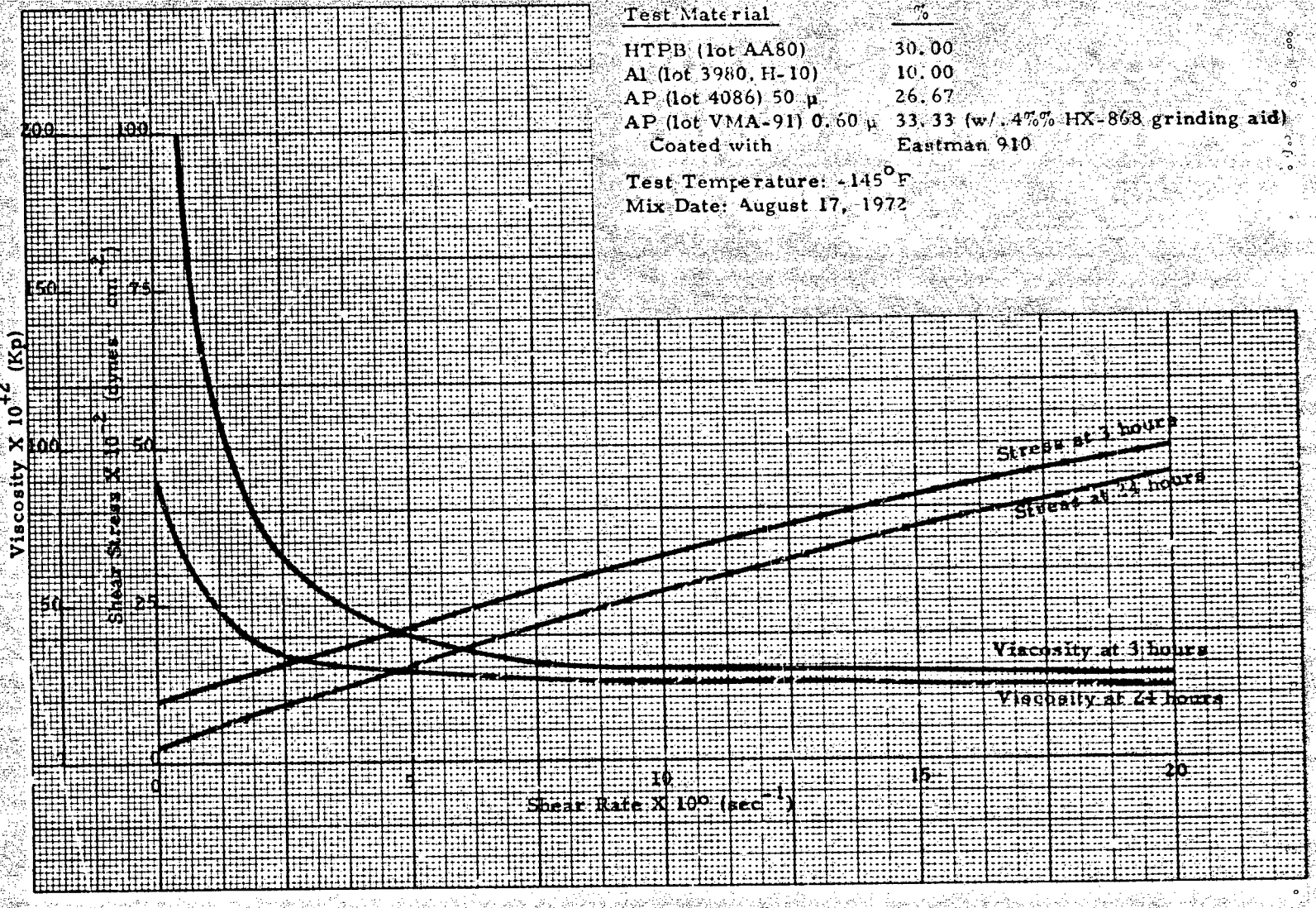


Figure 14. Viscosity and Shear Stress Versus Shear Rate of Coated Oxidizer Mixes

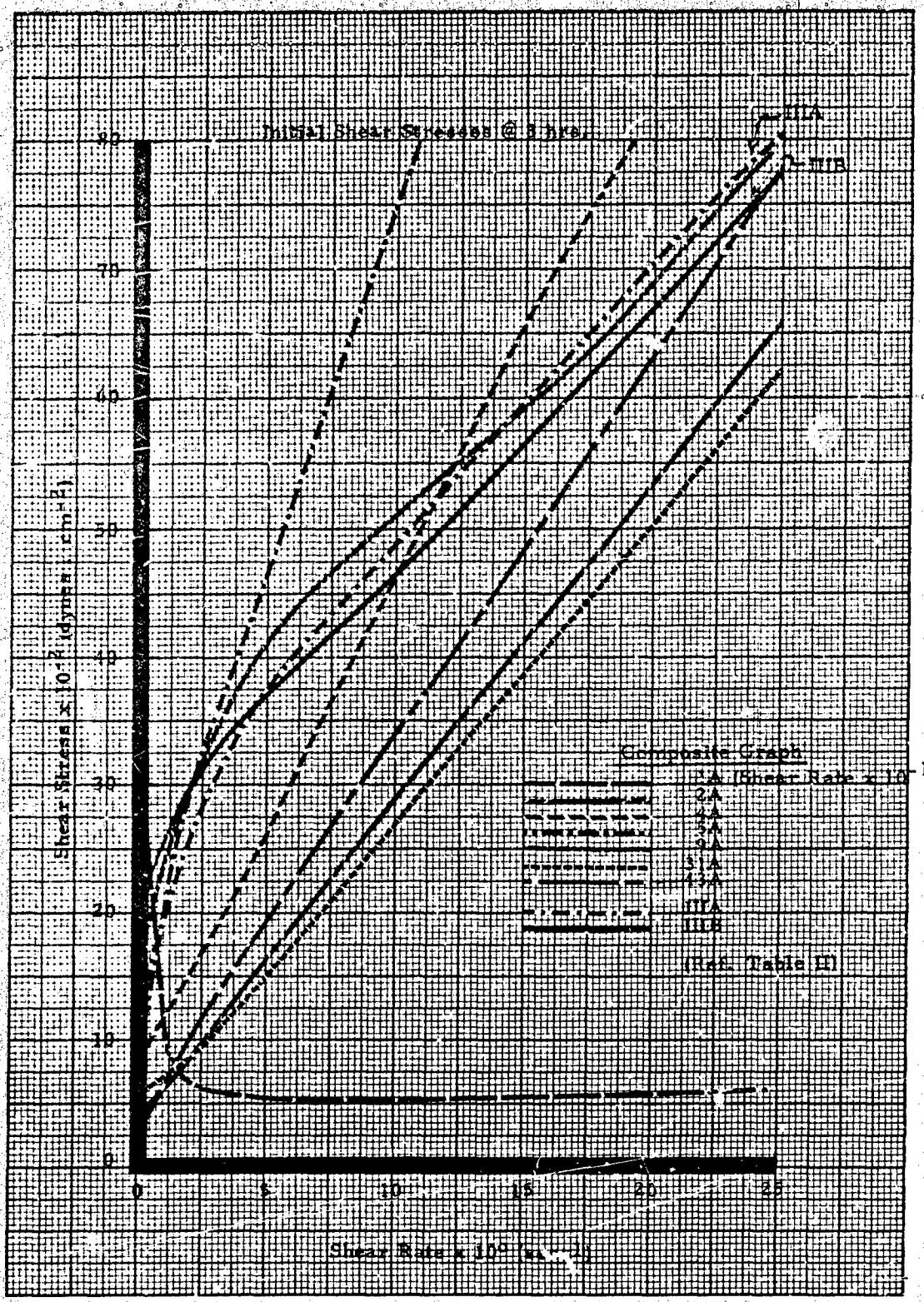


Figure 15. Shear Stress Versus Shear Rate of Coated Oxidizer Mixes

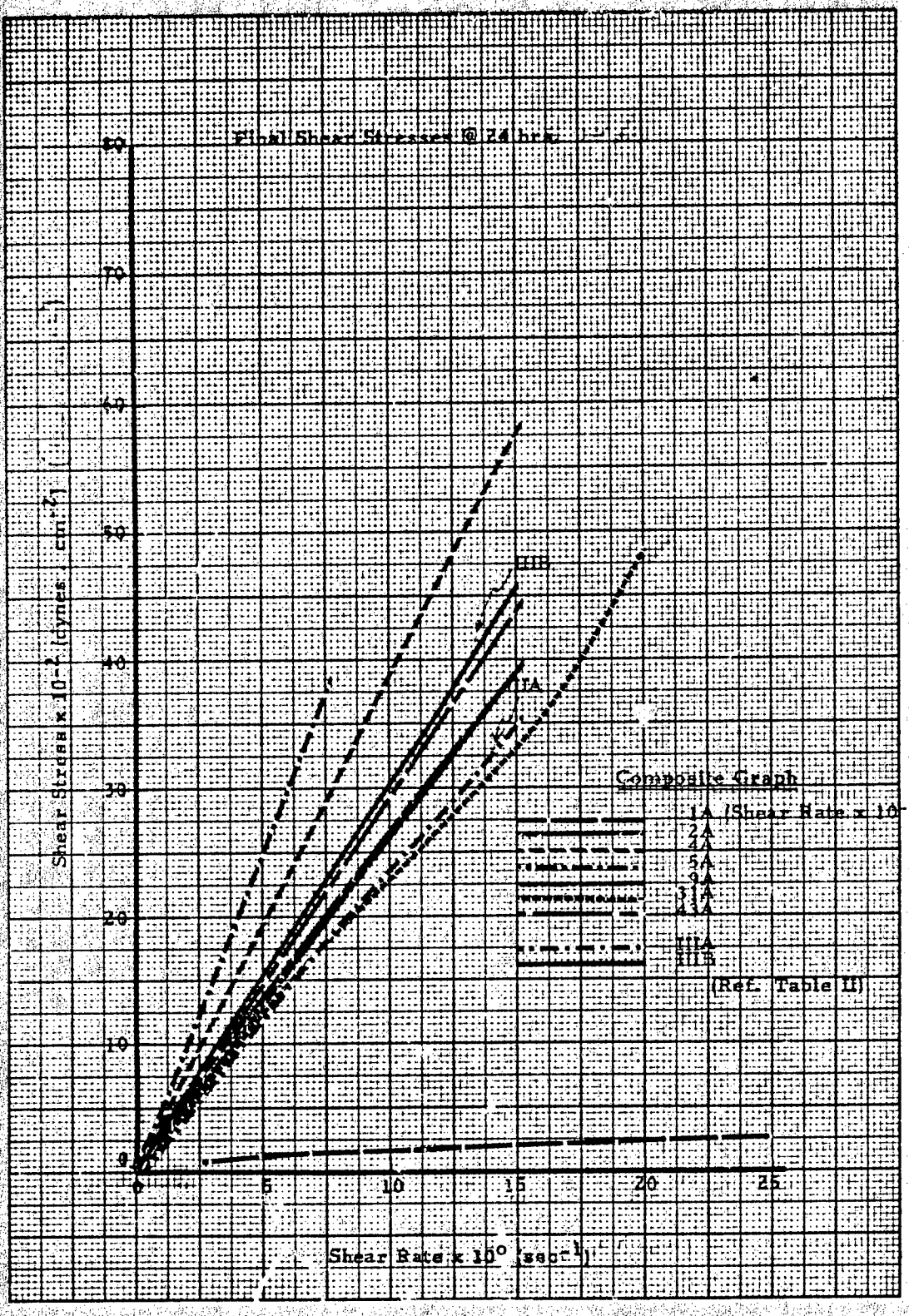


Figure 16. Shear Stress Versus Shear Rate of Coated Oxidizer Mixes

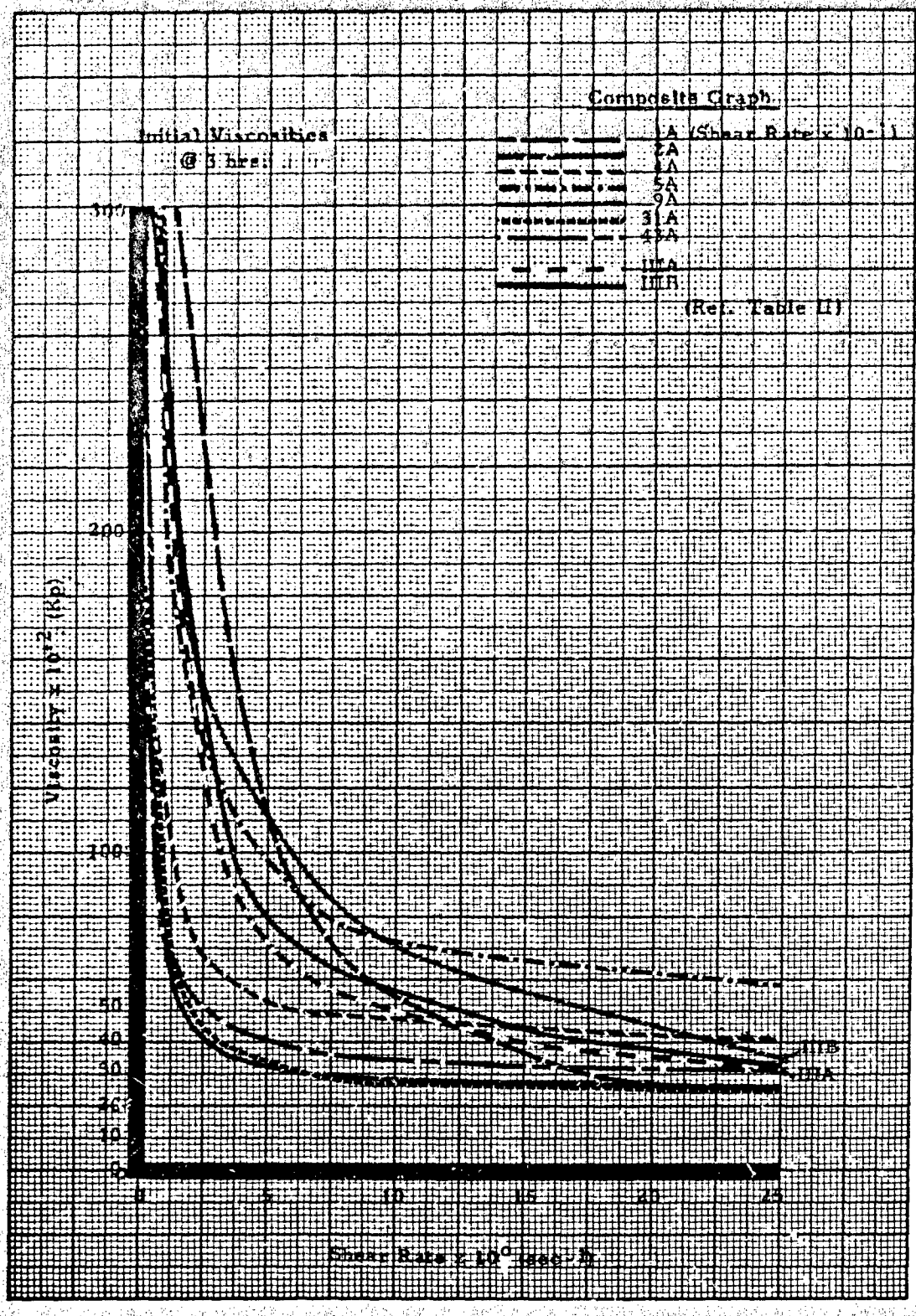


Figure 17. Viscosity Versus Shear Rate of Coated Oxidizer Mixes

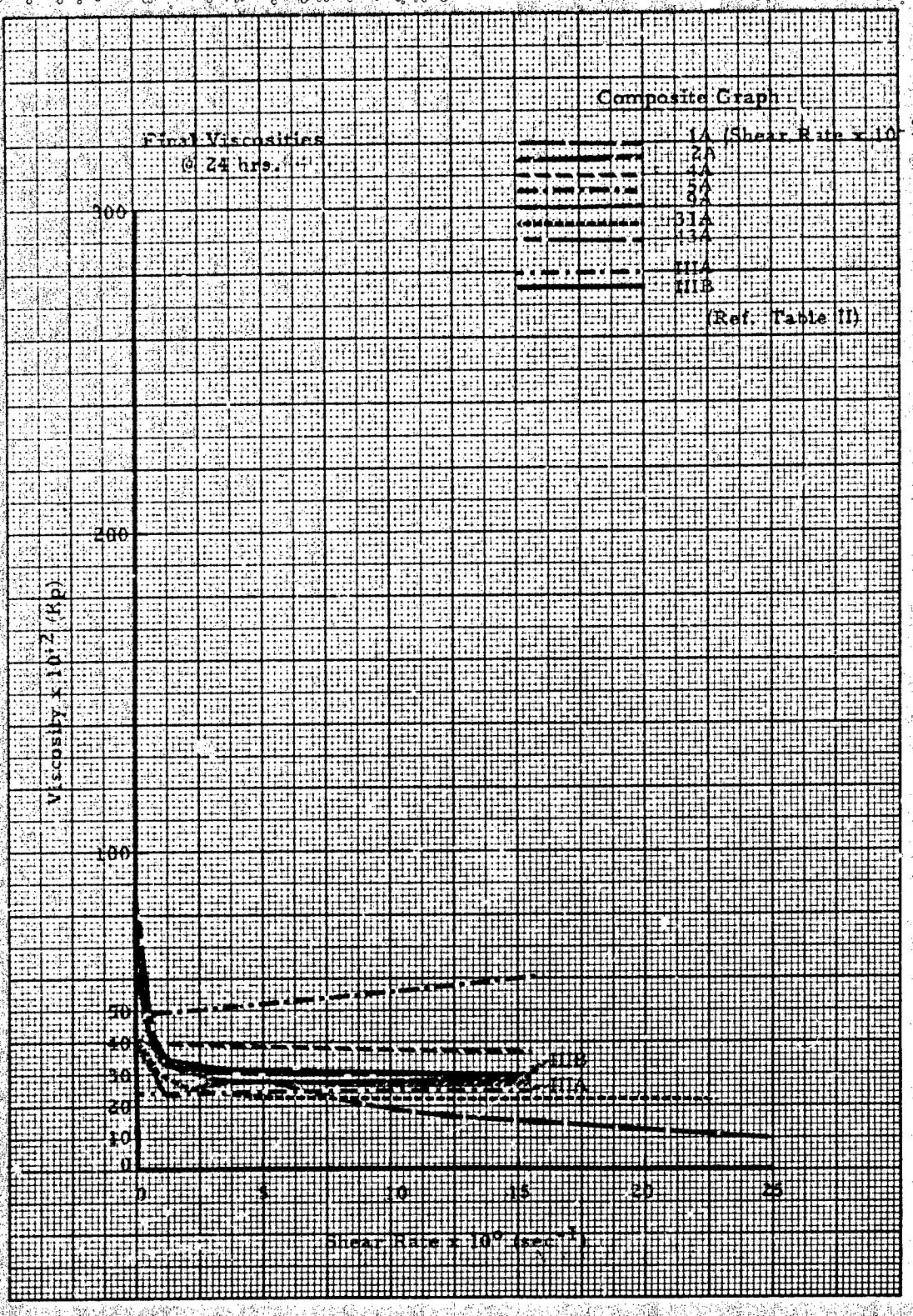


Figure 18. Viscosity Versus Shear Rate of Coated Oxidizer Mixes

The composite rheograms (see Figure 15 through 18) show that several of the mixes, 4A, 43A, 31A, 2A and 1A (Table 5) have lower values than the two standard tests. The one outstanding reduction in shear stress for both the 3 hour and 24 hour test was the mix containing the oxidizer coated with Eastman 910. It should be also noted that the shear rate for this mix had to be adjusted one decade in order to obtain measurable values because of the very low viscosity. The 24 hour test does not indicate any significant difference except with the use of Eastman 910 coated oxidizer. The mix was repeated in order to confirm this reduction in shear stress and viscosity with the use of Eastman 910 coated oxidize.. However, a different lot of ultra-fine oxidizer was used which contained a different grinding aid (HX-868) as opposed to the HX-874 used on the other oxidizer. The rheograms (Figures 13 and 14) for the standard and the experimental mix for this "repeated" test show that Eastman 910 has no effect on the rheological properties when HX-868 is used.

From the data generated the following conclusions were made:

1. All mixes have improved flow characteristics between evaluation at 3 hours after preparation to 24 hours after preparation at the test temperature of +145°F.

2. The ultra-fine oxidizer coated with Eastman 910 on HX-874 resulted in a mix with the lowest shear stress values compared to the other tests. The 3 hour test and the 24 hour test indicated similar results except for the initial portion of the 3 hour curve. This initial portion indicated a high yield value.

3. Dow XD-1040 (n-phenethylaziridine) coating improved the rheological properties consistently throughout the shear rate range while inclusion of HX-752 had an adverse effect.

4. Grinding aids also affect the rheological properties as can be observed.

5. The combination of Eastman 910 on HX-874 appears particularly attractive.

TABLE 7

PROPELLANT MIXES

14Q-179	14Q-177	14Q-176	14Q-173
XD-1040	HX-868	HX-752	Eastman 910
EOM Vis. 8 Kp/140°F	8 Kp/140°F	8/140	10/137
2 hr. 16 Kp/140°F	20 Kp/140°F	20/140	30/140
3 hr. 26 Kp/140°F	34 Kp/140°F	29/140	------
4 hr. ----------	----------	36/140	37/140
R_b @ 1000 1.12	1.15	1.15	1.14
"n" 0.63	0.72	0.64	0.61

Propellants (one-gallon size mixes) were made from oxidizer coated with several of the candidate materials and processing and strand burning properties were observed. The results of mixes made using XD-1040, HX-868, HX-752 and Eastman 910 coatings are shown in Table 7. From a processing standpoint, XD-1040 looks best, however, the Eastman 910 sample had agglomerated slightly and had to be screened, and may not be representative of the best results obtainable from this material. Silanox and Johnson's Wax were not processible in propellant. Burning rates of all the propellants were quite similar, around 1.1 inches per second at 1000 psi, and exponents were high - in the 0.6 - 0.7 range. Physical properties of all of the propellants were good.

In order to further investigate the relationship between Eastman 910 as an overcoating on HX-874 ground oxidizer by comparison with HX-868, a repeat of the Eastman 910 evaluation was done. This repeat showed a significant viscosity reduction by the use of Eastman 910 overcoated on HX-874. The significant gains obtained by the use of the Eastman 910 overcoating on the HX-874 grinding agent are illustrated in Table 8 and in Figure 19. It was thought that the agglomeration which appeared in the first duplicate test was avoided in this later experiment.

2. Grinding Aids

Six of the coatings were evaluated as grinding aids (Table 9) in the SWECO Vibro-energy mill using Lecithin as a standard. Johnson's Wax and CW4-019 were not effective, but HX-752, Silanox 101, XD-1040 and HX-868 were as effective as lecithin. This work indicates that these compounds can be used to grind AP in the SWECO mill, and will give good processing, physical properties, moisture protection and growth inhibition.

TASK I. SUMMARY

Approximately 40 coatings have been screened to find material which will protect UFAP from particle growth in a moist atmosphere. Six coatings have emerged as superior from this testing. Several of these have been shown to be effective in grinding UFAP, and in protecting UFAP from solvent vapor or abrasion. Methods of coating application have been assessed, and shown to be dependent upon the particular type of coating. A formulation guide summary is shown in Table 10. (Plus marks indicates good performance, and a zero indicates no change.)

TABLE 8

EASTMAN 910 EVALUATION FORMULATION

R-45M/IPDI/ AO-2246 COATING AGENT	13.7
HX-752	.3
Al (25μ)	18.0
AP (0.6μ)	30.0
(6μ)	38.0

UFAP COATING AGENT	HX-868	HX-874	HX-874 PLUS EASTMAN 910
COATING AGENT ON UFAP, %	.4	.4	.4/1
UFAP PARTICLE SIZE, μ	.6	.6	.6
EOM VISCOSITY, KP	23	60	10

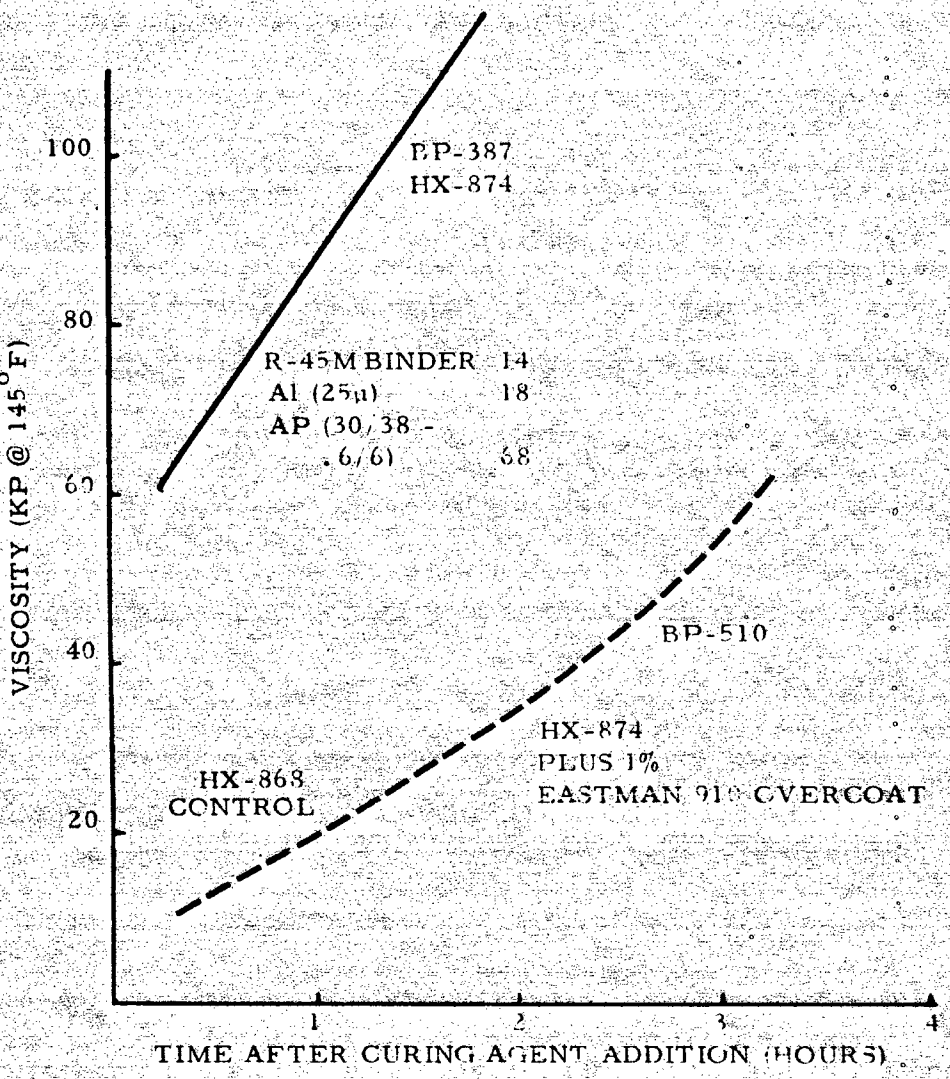


Figure 19. Demonstration of Unique UFAP Coating Combination

TABLE 9

EVALUATION OF CANDIDATE COMPOUNDS AS GRINDING* AIDS

Hours Ground	Lecithin	HX - 752	Silanox - 101	CW4- 019	HX - 868	XD - 1040	Johnson Wax
22-24	0.83	0.86	0.88	1.26	0.83	0.79	1.32
46-48	0.73	0.68	0.77	0.98	0.60	0.62	1.12
72	0.61	0.59	0.76	0.89	0.50	0.62	1.04

*SWECO Multi-Chamber Mill

TABLE 10

FORMULATION GUIDE

	DOW XD1040	Eastman 910	Silanox 101	HX-752	HX-868	Johnson's Paste Wax	CW4-019
Particle Growth for 0.4 μ AP	+	+	+	+	+	+	+
Particle Growth for 0.8 μ AP	+	+	+	+	+	+	+
Particle Growth for 1.8 μ AP	+	+	+	+	+	+	+
AP E_o	0	0	+	0	0	0	0
AP F_o	0	0	0	0	0	0	0
AP Spark	0	0	0	0	0	0	0
Resistance to Vapor	-	+	0	-	+	-	+
Resistance to Liquids	-	-	-	+	+	+	+
Resistance to Abrasion	+	+		-	+	-	+
Resistance to High Temperature	-	-	0	+	+	-	-
Resistance to Low Temperature	-	+	+	+	+	-	+
Physical Properties	0	0	0	0	0	0	0
Processing	+	+	-	+	+	+	0
Burning Rate	---------- all identical ----------						
Exponent	+	+		+	+		

TASK II - PARTICLE SIZE GROWTH

The objective of this task was to determine the conditions which will permit successful storage of large quantities of UFAP for up to one year, in any geographic location which might be encountered in a large scale production program. The goal for "successful storage" was a zero growth in UFAP particle size. Also, conditions were defined for the storage of UFAP over shorter periods (one-two months) so that the material would still meet the requirements for usability. The effect of intermittent exposure to ambient conditions was studied.

Sub-Tasks 1 and 3 - Particle Size Growth of UFAP (Coated and Uncoated)

This study was directed at obtaining baseline particle growth rate data on three standard UFAP materials; fluid energy milled, SWECO vibro-energy milled and emulsion freeze-dried UFAP.

The growth rate of uncoated fluid energy milled AP ($\sim 1.7\ \mu$) was determined by exposing samples of this material to a two temperature - three relative humidity test matrix. Particle size of each sample was measured as a function of time and the results are shown in Table 11. Since prohibitive particle growth occurred at all test conditions it was concluded that simple storage conditions will not prevent particle growth of uncoated UFAP.

A phenomenon that has been observed throughout the course of these studies is that the oxidizer particle will grow during the first 24 hours of storage and then tend to de-agglomerate with further exposure. Since these analyses were performed over a widely varying time period, it appears that this phenomenon is real and necessitates the re-assessment of particle size data that is obtained prior to at least 48 hours of exposure.

Two types of coated UFAP have been subjected to storage conditions of varying relative humidities and temperatures in order to determine the growth rate of UFAP prepared by different techniques. A series of constant humidity desiccators was prepared and temperature was controlled by enclosing these desiccators in a constant temperature oven. The first sample examined was one (prepared on the Vibro-energy mill) coated with 0.4% HX-874 during the grinding process. Fifty gram samples were stored at 10, 30, and 50% relative humidities and at three temperatures, 80, 60, and 25°C and particle sizes were measured after 24 hours, 48 hours and one week. These data were continuously collected up to a time period of ten weeks. The data resulting from these tests is shown on Table 12. Note that essentially no particle growth occurred when the samples were stored at 10% relative humidity. Note further that 30% relative humidity appears to be a satisfactory

TABLE II

BASELINE PARTICLE GROWTH STUDIES

Uncoated Fluid Energy Milled AP

$L_{68_{11}}$

Relative Humidity, % / Temperature	Size, micron 0	24 hrs.	48 hrs.	1 wk.
10 — 25°C	1.68	2.23	1.70	3.41
50 — 25°C	1.68	2.78	2.58	3.55
30 — 60°C	1.68	2.75	3.27	4.31

TABLE 12

PARTICLE GROWTH STUDIES

Vibro-Energy Milled AP (VMA-88A)

0.65_{u}

Coated with 0.4% HX-764

Relative Humidity, %	Temp., °C	Size, micron					
		0	24 hr.	48 hr.	1 wk.	6 wk.	10 wk.
10	80	0.65	0.71	0.58	0.62	0.67	0.64
30	80	0.65	0.84	0.71	1.15	1.92	1.62
50	80	0.65	>4.00	----	----	----	----
10	60	0.65	----	----	0.55	0.53	0.57
30	60	0.65	----	----	0.66	0.66	0.71
50	60	0.65	0.89	0.72	0.71	0.85	0.71
10	25	0.65	----	----	0.56	0.55	0.63
30	25	0.65	----	----	0.66	0.60	0.65
50	25	0.65	0.90	0.77	0.79	0.80	0.83

storage condition of the Vibro-energy mill prepared oxidizer if the temperature is 60°C or less. A relative humidity of 50% appears to be too harsh a storage condition even at ambient temperature.

An exact duplication of the preceding experiment was performed with UFAP prepared by the freeze drying process. The oxidizer prepared in this manner was coated with 1.4% Emcol-511 (emulsifier) and 0.6% Emcol-4200 (coating agent). The resultant particle size of the oxidizer prepared in this process was 0.48 micron. The results of storing this oxidizer at relative humidities of 10, 30, and 50% at temperatures of 80, 60, and 25°C are shown in Table 13. The trends noted in the data are similar to those found with the Vibro-energy milled UFAP, namely, ten percent RH was a satisfactory storage condition even at high temperatures and 50% relative humidity provided conditions conducive to particle growth. These studies were continued for a period of ten weeks.

Data (Table 14) showed that neither SWECO nor freeze-dried UFAP particle size was significantly affected by intermittent exposure to ambient temperature and humidity under conditions that simulated weigh-up and mixer addition procedures during propellant production. Consequently, the particle growth rate of the coated UFAP studied can be controlled under conditions which can be met in production situations.

Sub-Task 2 - Storage Conditions for Stabilization of UFAP Growth Rate

An evaluation of other storage conditions as a means of preventing particle growth was performed on the three previously described UFAP materials. The storage conditions were as follows:

1. A nitrogen filled desiccator at ambient temperature.
2. A desiccator stored at -35°C.
3. A desiccator containing P_2O_5 (ambient temperature).
4. An evacuated desiccator at ambient temperature.
5. A forced air oven maintained at 115°C.

Fifty gram samples of the fluid energy milled, SWECO Vibro-energy milled and emulsion freeze-dried UFAP were exposed to each of these conditions and particle size measured at several time intervals up to ten weeks.

As can be seen from Table 15, the uncoated fluid energy milled AP exhibited a prohibitive increase in size at all condition except the 115°C storage The data at -35°C is somewhat questionable in that much past data at TCC-Huntsville, as well as other laboratories, had indicated the -35°C storage condition a most effective technique to prevent particle growth. Failure to keep the sample closed to the atmosphere before reaching room temperature could possibly account for the growth noted in Table 15.

TABLE I

PARTICLE GROWTH TESTS

Freeze Dried UF/AP (F12-104)

0.48 U

1.4% E-511 — 0.6% E-1200

Relative Humidity, %	Temp., °C	Size, micron				
		0	24 hr.	1 wk.	6 wk.	10 wk.
10	80	0.48	0.48	0.48	0.55	0.57
30	80	0.48	0.93	3.50	4.93	4.00
50	80	0.48	6.23	6.00	10.00	10.00
10	60	0.48	0.51	0.50	0.53	0.53
30	60	0.48	0.56	0.98	3.90	3.60
50	60	0.48	3.10	4.50	10.00	----
10	25	0.48	0.49	0.46	0.49	0.55
30	25	0.48	0.45	0.55	0.53	0.55
50	25	0.48	0.48	0.60	3.40	1.95

TABLE 14

EFFECT OF INTERMITTENT EXPOSURE TO AMBIENT TEMPERATURE AND HUMIDITY UPON UFAP PARTICLE GROWTH

(15 Min. exposure each day)

	Initial Size (micron)	Exposures							
		1	2	3	4	5	6	7	8
VMA-88A 0.4% HX-874	.65	.83	--	--	.61	.68	.63	--	.62
FDS-104 1.4% E511 0.6% E4200	.48	.51	--	--	.48	.54	.50	.49	--

TABLE 15

EFFECT OF VARIOUS STORAGE CONDITIONS UPON THE GROWTH RATE OF FEM OXIDIZER

Uncoated - 1.68μ

Storage Condition	Time					
	0	24 hr.	48 hr.	1 wk.	6 wks.	10 wks.
Under N_2 -35°C	1.68	2.21	1.80	3.45	2.23	2.90
Over P_2O_5	1.68	3.68	3.27	4.50	4.50	4.90
Under Vacuum 115°C	1.68	1.65	2.16	3.30	2.07	2.44
	1.68	2.29	2.53	3.20	2.15	2.33
	1.68	1.48	1.33	3.37	1.66	1.54

The data obtained for vibro-energy milled AP at the same storage conditions is depicted in Table 16. This oxidizer, which is coated with 0.4% HX-874, exhibited excellent particle size stability at all test conditions up to ten weeks.

Similar conclusions can be drawn from identical tests on emulsion freeze-dried UFAP (Table 17).

In order to determine the effectiveness of storing UFAP as a paste in HTPB polymer, a sample of uncoated fluid energy milled oxidizer was mixed with HTPB polymer in two different ratios (60% UFAP/40% HTPB and 70% UFAP/30% HTPB) and the blends exposed to temperatures of -20, 77, and 140°F and relative humidities of 50% and 10%. Particle growth was followed with time (Table 18). Data showed that growth had only occurred at the high temperature, high humidity condition after six weeks of storage.

The stability of the polymer from which the paste is manufactured is of critical importance. The molecular weight, functionality and hydroxyl equivalents of the polymer were followed as a function of time. After six weeks of storage slight decreases in hydroxyl content are apparent especially at the high temperature, low humidity condition. This would indicate that a hydroxyl uptake reaction is occurring which is, as would be expected, driven by higher temperature, but which is tempered by the presence of moisture. In other words, the hydroxyl from water is more reactive than is the polymer hydroxyl.

The decrease in hydroxyl is accompanied by a significant increase in the weight average molecular weight. This indicates that the polymer molecular weight distribution is much more broad (more low molecular weight and more high molecular weight).

It is concluded that storage of UFAP in a paste form must be done at moderate humidity and low temperature and not for extended periods of time.

TABLE 16

EFFECT OF VARIOUS STORAGE CONDITIONS

UPON GROWTH RATE OF UFAP

Vibro Energy Milled AP
VMA-88A - 0.65_{μ}

	Size, micron					
	0	24 hr.	48 hr.	1 wk.	6 wks.	10 wks.
Under N_2	0.65	----	----	0.66	0.66	0.68
-35°C	0.65	0.65	----	0.81	0.63	0.68
Over P_2O_5	0.65	----	----	0.66	0.67	0.67
Under Vacuum	0.65	----	----	0.73	0.65	0.66
115°C	0.65	----	----	0.67	0.61	0.69

TABLE 17

EFFECT OF VARIOUS STORAGE CONDITIONS

UPON GROWTH RATE OF UFAP

Freeze Dried UFAP
FDS-104 - 0.48$_{\mu}$

	Size, micron					
	0	24 hrs.	48 hrs.	1 wk.	6 wks.	10 wks.
Under N_2	0.48	0.49	----	0.52	0.54	0.50
-35°C	0.54	0.51	----	0.52	0.55	0.53
Over P_2O_5	0.48	0.49	----	0.54	0.57	0.56
Under Vacuum	0.48	0.47	----	0.46	0.52	0.44
115°C	0.48	0.50	----	0.70	0.61	0.70

TABLE 18

EFFECT OF TEMPERATURE AND HUMIDITY UPON

STABILITY OF UFAP/HTPB PASTE

HTPB-40%, AP-60%

RH (%)	Temp. (°F)	"0" Time Particle Size(μ)	"0" Time OH	"0" Time $\overline{M}_n$	"0" Time $\overline{M}_w$	"0" Time M_n/M_w	1 Week Particle Size(μ)	1 Week OH	1 Week $\overline{M}_n$	1 Week $\overline{M}_w$	1 Week M_n/M_w	6 Weeks Particle Size(μ)	6 Weeks OH	6 Weeks $\overline{M}_n$	6 Weeks $\overline{M}_w$	6 Weeks M_n/M_w
10	-20	1.20	.0738	3466	7230	2.09	1.05					0.95	----	----	----	------
10	77	1.20	.0738	3466	7230	2.09	1.07					1.28	.0741	3117	8225	2.64
10	140	1.20	.0738	3466	7230	2.09	1.02		3289	7591	2.31	1.65	.0620	3254	9922	3.05
50	-20	1.20	.0738	3466	7230	2.09	1.06					1.06	----	----	----	------
50	77	1.20	.0738	3466	7230	2.09	1.22					1.28	.0752	3086	7622	2.47
50	140	1.20	.0738	3466	7230	2.09	1.55	.0737	3417	7476	2.19	2.88	.0728	3345	10,420	3.12

HTPB-30%, AP-70%

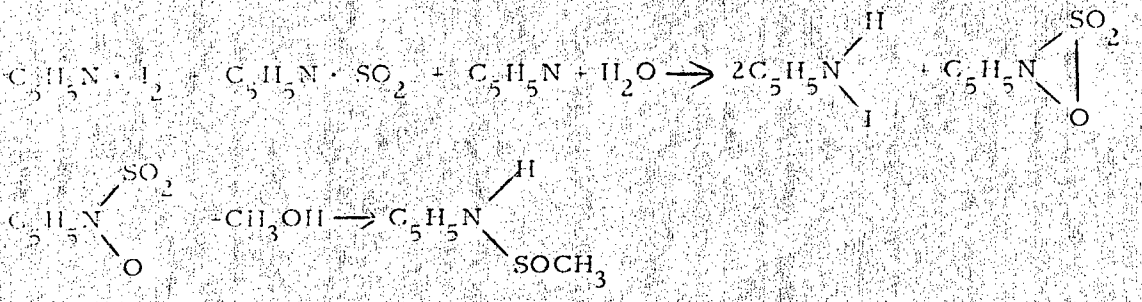
RH (%)	Temp. (°F)	"0" Time Particle Size(μ)	"0" Time OH	"0" Time $\overline{M}_n$	"0" Time $\overline{M}_w$	"0" Time M_n/M_w	1 Week Particle Size(μ)	1 Week OH	1 Week $\overline{M}_n$	1 Week $\overline{M}_w$	1 Week M_n/M_w	6 Weeks Particle Size(μ)	6 Weeks OH	6 Weeks $\overline{M}_n$	6 Weeks $\overline{M}_w$	6 Weeks M_n/M_w
10	-20	1.17	.0719	3477	7325	2.11	1.10					0.99	----	----	----	------
10	77	1.17	.0719	3477	7325	2.11	1.03					1.01	.0723	2954	7659	2.59
10	140	1.17	.0719	3477	7325	2.11	1.17		3419	7599	2.22	1.65	.0696	3337	9908	2.97
50	-20	1.17	.0719	3477	7325	2.11	1.06					1.08	----	----	----	------
50	77	1.17	.0719	3477	7325	2.11	1.03					1.22	.0743	3750	7682	2.05
50	140	1.17	.0719	3477	7325	2.11	1.79	.0726	3530	7458	2.11	3.45	.0721	3284	9138	2.78

TASK III - WATER ANALYSIS

The objective of this task was to develop a rapid water analysis technique for UFAP. The Karl Fischer technique was to be evaluated as to accuracy, precision and speed and, if found inapplicable, new techniques were to be evaluated.

The Karl Fischer technique is in widespread use and is the method of choice for the water analysis of ammonium perchlorate in the propulsion industry. Basically, the procedure involves the reaction of the Karl Fischer reagent with moisture and the end point is normally determined electrometrically. The Karl Fischer reagent, a mixture of pyridene-sulphur dioxide solution and iodine-methanol solution, reacts quantitatively with water.

Although the exact chemistry of the technique is not known, current thinking indicates the following:



The Karl Fischer reagent does not react chemically with most materials; however, there are some compounds and classes of compounds which react with one or more of the constituents of the reagent. Among those materials which do not react are metal oxides, ferric salts, substituted hydrazine salts and mercaptans. The technique is applicable to either soluble or insoluble solids and both direct and back titrations can readily be performed. The accuracy and precision of the Karl Fischer titration are unsurpassed. Standard deviations of 0.00045 and coefficients of variations of 2.81% are typical for water analysis in ammonium perchlorate. The maximum range between duplicates on a given sample is less than 0.0016.

The uncertainty concerning the effect of various UFAP coating agents upon the accuracy of the Karl Fischer titration led to a brief study into possible interferences. Five different selected coating agents were examined as to their effect upon the accuracy of the automated Karl Fischer titration. Two aziridines (HX-868 and XD-1040), two emulsifier-coating agents (Nopcogen 16-0 and Armeen SZ) and a hydrophobe (Silanox 101) were selected for study. A 5% solution or slurry of each in benzene/methanol was titrated with the Karl Fischer reagent and in each case, less than two drops of reagent were required to reach an endpoint. This not only indicated a dry material but also freedom from interference with the titration.

Studies were initiated to ascertain if interferences were encountered in those cases where the coating agent had reacted with the AP. Samples of UFAP containing the two aziridines and the two emulsifying agents were treated in methylene chloride with a reactive isocyanate (p-toluene sulfonyl isocyanate) to remove surface moisture. The samples were then stripped of excess methylene chloride, dissolved in benzene/methanol and titrated with Karl Fischer reagent. In each case, less than 10 drops (corresponds to limit of detection for water) of reagent were required to reach an end point. This data indicates no interference from the coating-AP product.

Five Karl Fischer titrations were performed on UFAP coated with the five selected coating agents. The precision of the results on each set of analyses was well within normally accepted limits.

These results do not indicate that the Karl Fischer titration is universal for coated UFAP, but is quite acceptable for those coated UFAP samples studied. Care should still be exercised in using the Karl Fischer titration on coated UFAP until a lack of interference from the coating agent can be ascertained.

TASK IV - HAZARDS ASSESSMENT

The safety aspects of preparing, handling, storing, and using various forms of UFAP products were studied and tested experimentally to assess the degree of hazards associated with each operation. Three UFAP preparation methods were selected as being representative of the present state-of-the-art - SWECO wet grinding, fluid energy milling, and freeze drying - for detailed study. Test methods used generally were based upon DSAR 8220.1, Explosives Hazard Classification Procedures (originally designated TB 700-2), with additional methods used where needed to supplement these tests. Such additional tests included friction sensitivity, electrostatic spark sensitivity, differential scanning calorimetry, and susceptibility to dust explosion. Specific operating procedures for most of the tests are given in Appendix A along with the tabulated experimental data and discussions of the test methods and their limitations.

Although UFAP can be handled, stored, and/or used in a number of physical forms (e.g., dry powder, paste, or slurried with a carrier fluid), the end products studied in this program involve dry powders, while the preparation steps pass through a wide variety of intermediate forms. Work was therefore directed toward characterization of intermediate, or in-process, forms or compositions, as well as toward the end-product. An important goal in the assessment study was determining whether UFAP in general, or at least in certain defined conditions, could qualify as a Class B explosive hazard (Military Class 2) for shipping purposes.

Dry Powder UFAP Product Forms

In general, dry powder UFAP materials do not exhibit a very high sensitivity to impact, although the sensitivity does increase as the coating agent content increases. Where tested, the dry powders were insensitive to electrostatic spark discharge, although the method of testing tends to disrupt the small particles of material in the testing apparatus before it can be initiated. The materials are reasonably thermally stable, although exposure to elevated temperature can cause physical changes, probably caused by equilibrium solubility processes involving residual moisture. When subjected to external flame, the materials burn smoothly and do not explode, when unconfined. When confined and initiated by a shock wave (explosive donor charge), all dry powder UFAP materials can be expected to develop a detonation, although the detonation velocity may be relatively low or may tend to drop off rapidly. All, however, exhibit destructive chemical decomposition reactions that yield explosive forces. Unconfined samples initiated with a blasting cap will generally explode,

particularly if the material contains a coating agent that acts as a fuel in the combustion reaction. The one sample that consistently did not explode in the lead column test, fluid energy milled UFAP without contaminants, may have been nonreactive either because of particle size (relatively large) or lack of combustible fuel contamination. Over the dust concentration range tested, only freeze-dried material, containing a relatively high concentration of coating agent, initiated and/or exploded in dust cloud form. None of the other materials would propagate throughout the cloud when ignited.

Contrary to initial expectations, reducing the bulk density of the UFAP charges in card gap tests did not increase the susceptibility of the material to detonation. Instead, in a number of instances, reducing the bulk density reduced the probability of punching the witness plate. However, severe plate damage resulted from every test, either in the form of plate shattering or cracking or deformation. The use of standoff separators between the test cylinder and the witness plate tended to reduce the plate breakage, but did not eliminate it.

It was concluded that dry powder UFAP materials should be treated as ICC (DOT) Class A explosive hazards (Military Class 7) unless desensitized in some manner. Although high velocity detonation waves may not develop in such materials if initiated, the explosive destruction results would be extremely severe. Summary Test Data Sheets, per Chapter 3 of DSAR 8220.1, for these materials are given as Tables 19, 20, 21, and 22. Chemical compositions and tabulated test data are given in Appendices B, C, and D.

Alternate UFAP Product Forms

Alternate product forms of UFAP, such as pastes made with HTPB polymer or inert carrier fluid slurries, were eliminated from consideration for various reasons. Pastes were dropped from consideration primarily because the successful development of storable, stable dry powder products virtually eliminated the need or utility of such materials. Characterization of slurries was minimized because of overlap with the SWECO wet grinding process which has been well characterized in earlier studies.

Intermediate or In-Process Materials

The SWECO process of grinding AP in a slurry has been comprehensively characterized in previous programs, so little work was deemed necessary on intermediate compositions. One set of card gap tests, run on a variation of the mill slurry composition, confirmed that such slurries are not detonable when the Freon 113 content is at least 75% by weight of the mixture. Processes where the slurry passes through the detonable

composition ranges, such as during Freon removal, are non-attendant, non-mechanical operations, so initiating mechanisms, as well as personnel exposure, are eliminated.

The fluid energy mill grinding process essentially has no intermediate compositions or forms, and the operation is performed remotely, so no additional testing was needed.

The freeze drying process for UFAP manufacture involves a number of process steps and compositions that must be done or handled by attendant personnel. As presently performed at Thiokol, these generally are preparation of the AP emulsion for freezing, and the freezing and subsequent handling of the frozen emulsion. Once in the sublimator, the material is presumed to be isolated from initiating forces or mechanisms, and when it is removed, it is in final product form which has already evaluated. Negative card gap tests results were obtained on a typical emulsion (consistency of mayonnaise). This was expected since the emulsion was nearly two thirds water. Card gap tests were also run on samples of the same emulsion after freezing with liquid nitrogen, with negative results. It was therefore assumed that the greatest hazards associated with that portion of the process prior to charging the sublimator were concerned with the liquid nitrogen (cryogenic temperatures and oxygen starvation) and the trichloroethylene or other solvent (toxicity or possibly flammability). Tabulated data from the tests are contained in the Appendix D.

TABLE 19

EXPLOSIVE HAZARDS CLASSIFICATION TEST SUMMARY
DATA SHEET (DSAR 8220.1, Chapter 3)

Date April 2, 1973

Sponsoring Agency Thiokol Chemical Corporation - Huntsville Division

Contract No. F04611-72-C-0061

Test Material Identity SWECO Ground UFAP Dry Powder

Composition Reference I-B & -C, App. B Batch No. VMA-93/-94

Date Manufactured 6/8-15/73

Detonation Test (Lead Column)

		Exploded Yes	Exploded No	Burned Yes	Burned No	Fragmented Yes	Fragmented No
No. 8 Blasting Cap	Test I	X					X
	Test II	X					X
	Test III						
	Test IV						
	Test V						

(Test Series No. 75)

Ignition & Unconfined Burning Test

	Exploded Yes	Exploded No	Average Burning Time Seconds
Single Charge		X	42
Single Charge		X	24
Four Charges		X	55

(Test Series No. 87)

Thermal Stability Test (48 hrs. @ 75°C)

Explosion Yes	Explosion No	Ignition Yes	Ignition No	Change In Configuration Yes	Change In Configuration No
	X		X		X

Card Gap Test--50% Value is 8-10 (No. of Cards)

(Test Series No. 72)

Impact Sensitivity Test

Olin Mathieson Drop Weight Tester
E_o greater than 250 Kgm. - cm.

Approved:

Test Supervisor G. L. Vance

Test Department Head W. S. Hunter

Assigned Classification	
DOT Forbidden	
DOT Restricted	
DOT Class A	
DOT Class B	

DOD Approval

Signature _____

Title _____

Organization _____

TABLE 20

EXPLOSIVE HAZARDS CLASSIFICATION TEST SUMMARY
DATA SHEET (DSAR 8220.1, Chapter 3)

Date April 2, 1973

Sponsoring Agency Thiokol Chemical Corporation - Huntsville Division

Contract No. FO4611-72-C-0061

Test Material Identity Freeze-Dried UFAP Dry Powder

Composition Reference III-B, Appendix B Batch No. FDI-10A

Date Manufactured 6/23/72

Detonation Test (Lead Column)

		Exploded Yes	Exploded No	Burned Yes	Burned No	Fragmented Yes	Fragmented No
No. 8 Blasting Cap	Test I	X					X
	Test II	X					X
	Test III						
	Test IV						
	Test V						

(Test Series No. 78)

Ignition & Unconfined Burning Test

	Exploded Yes	Exploded No	Average Burning Time Seconds
Single Charge		X	25
Single Charge		X	27
Four Charges		X	33

(Test Series No. 88)

Thermal Stability Test (48 hrs. @ 75°C)

Explosion Yes	Explosion No	Ignition Yes	Ignition No	Change In Configuration Yes	Change In Configuration No
	X		X	X (Shrunk)	

Card Gap Test--50% Value is Neg. @ 0 (No. of Cards)

(Test Series No. 74)

Impact Sensitivity Test

Olin Mathieson Drop Weight Tester
E_o greater than 250 Kgm. - cm.

Approved:

Test Supervisor S. L. Vaux

Test Department Head W. J. Hunter

Assigned Classification	
DOT Forbidden	
DOT Restricted	
DOT Class A	
DOT Class B	

DOD Approval

Signature

Title

Organization

TABLE 21

EXPLOSIVE HAZARDS CLASSIFICATION TEST SUMMARY
DATA SHEET (DSAR 8220.1, Chapter 3)

Date April 2, 1973

Sponsoring Agency Thiokol Chemical Corporation - Huntsville Division

Contract No. FO4611-72-C-0061

Test Material Identity Fluid Energy Milled UFAP Dry Powder

Composition Reference II-A, Appendix B Batch No. EFEM-33

Date Manufactured 5/19/72

Detonation Test (Lead Column)

		Exploded Yes	Exploded No	Burned Yes	Burned No	Fragmented Yes	Fragmented No
No. 8 Blasting Cap	Test I		X	X			X
	Test II		X	X			X
	Test III		X	X			X
	Test IV		X	X			X
	Test V		X	X			X

(Test Series No. 77)

Ignition & Unconfined Burning Test

	Exploded Yes	Exploded No	Average Burning Time Seconds
Single Charge		X	45
Single Charge		X	45
Four Charges		X	48

(Test Series No. 89)

Thermal Stability Test (48 hrs. @ 75°C)

Explosion Yes	Explosion No	Ignition Yes	Ignition No	Change In Configuration Yes	Change In Configuration No
	X		X	X (Shrunk)	

Card Gap Test--50% Value is 0-4 No. of Cards)

(Test Series No. 71)

Impact Sensitivity Test

Olin Mathieson Drop Weight Tester
E_o greater than 250 Kgm. - cm.

Approved:

Test Supervisor S. L. Vance

Test Department Head L. S. Hess

Assigned Classification	
DOT Forbidden	
DOT Restricted	
DOT Class A	
DOT Class B	

DOD Approval

Signature _____

Title _____

Organization _____

TABLE 22

EXPLOSIVE HAZARDS CLASSIFICATION TEST SUMMARY

DATA SHEET (DSAR 8220.1, Chapter 3)

Date April 2, 1973

Sponsoring Agency Thiokol Chemical Corporation - Huntsville Division

Contract No. F04611-72-C-0061

Test Material Identity EFEM-33 with 0.2% Silanox 101

Composition Reference II-A, Appendix B Batch No. EFEM-33

Date Manufactured 5/10/72

Detonation Test (Lead Column)

		Exploded Yes	Exploded No	Burned Yes	Burned No	Fragmented Yes	Fragmented No
No. 8 Blasting Cap	Test I		X	X			X
	Test II	X					X
	Test III	X					X
	Test IV						
	Test V						

(Test Series No. 76)

Ignition & Unconfined Burning Test

	Exploded Yes	Exploded No	Average Burning Time Seconds
Single Charge		X	45
Single Charge		X	53
Four Charges		X	69

(Test Series No. 90)

Thermal Stability Test (48 hrs. @ 75°C)

Explosion Yes	Explosion No	Ignition Yes	Ignition No	Change In Configuration Yes	Change In Configuration No
	X		X	X (Shrunk)	

Card Gap Test--50% Value is 8-10 (No. of Cards)

(Test Series No. 73)

Impact Sensitivity Test

Olin Mathieson Drop Weight Tester

E_o greater than 250 Kgm. - cm.

Approved:

Test Supervisor G. L. Vance

Test Department Head W. J. Hunter

Assigned Classification	
DOT Forbidden	
DOT Restricted	
DOT Class A	
DOT Class B	

DOD Approval

Signature

Title

Organization

TASK V - SHIPPING

The explosive hazards test results from Task IV were assessed, desensitization methods were tried, a UFAP shipping container and packaging method established, and shipping container qualification tests were run. The most desirable goal sought in this task was the development of sufficient data to allow obtaining from DOT and DOD a "blanket" approval of a Class B explosives hazard shipping classification for UFAP. In any event, criteria would be established, if possible, for dividing UFAP products, by such characteristics as composition (e.g., oxidizer to fuel ratio) or particle size, into Class A and Class B explosive hazard groupings. In the event that a Class B explosive hazard classification could not be obtained even for some portion of UFAP products, the test results would still allow shipment of UFAP in larger quantities than those currently specified for untested "Laboratory Samples."

As can be seen from the test Summary Data Sheets in Task IV, a number of conclusions could be drawn about the UFAP materials tested. Using the interpretation criteria of paragraph 3-13 of DSAR 8220.1, none of the four materials are DOT "Forbidden" because explosions, burning, or decomposition of the samples did not occur in the thermal stability tests. None of the materials are DOT "Restricted" because the ignition and unconfined burning tests resulted in simple burning only. Designation of the materials as DOT Class A or Class B explosives, however, becomes somewhat difficult, as there is no separation of the card gap test results from the lead column test results. None of the materials required 70 cards to prevent detonation, however, most exhibited positive lead column test results. A test material can be designated Class B only if it results in negative lead column shots and has a card gap sensitivity of less than seventy cards. Only the pure, fluid energy milled UFAP can be so defined. The other materials gave positive results in the lead column test, necessitating a Class A designation.

A series of tests was run in which varying quantities of Sorbeads, spherical shaped prills of fused silica gel, were blended with the UFAP products to effect a desensitization. These tests, tabulated in Appendices D & E, indicated that the addition of as little as 10% by weight of Sorbeads to the UFAP products would prevent detonation at zero cards in the card gap test. In the lead column test, the percentage of Sorbeads required to produce negative results varied with the percent of theoretical material density (% TMD), with between 25 and 35% required for hand-packed samples, and between 10 and 25% for loosely-filled samples. Although anomalous results were encountered since a calibration card gap test shot without any Sorbeads did not result in a positive test, a level of 30% sorbeads was selected to give a margin

of safety. These desensitization tests were run using the UFAP compositions showing the greatest sensitivity in Task IV work. Subsequent tests, discussed later in this text, indicated the Sorbead addition did not desensitize all types of UFAP materials.

The use of Sorbeads as a desensitizing diluent for UFAP provided one additional, very important benefit: the Sorbeads maintained a very low relative humidity (less than 5%) inside the sealed storage containers, thereby minimizing the tendency for the particle size to grow due to equilibrium solubility effects. Although other desiccant forms might provide such protection, such as the bagged charges of desiccant normally placed in AP drums by the material manufacturer, only the distribution or dispersement of the Sorbead type material throughout the UFAP mass would effect desensitization of the explosive characteristics. Although unfused desiccants could be blended in with UFAP, such material would be subject to particle breakage and dusting, resulting in contamination. The Sorbeads do not readily break and do not slough surface material as dust. The Sorbeads are also screened when received to remove any that will pass through a nominal 6 mesh screen sieve. They can then be readily removed from the UFAP with a 10 mesh screen (or smaller opening), which is normally used to screen all AP materials prior to incorporation into large mixer propellant batches.

An example of the excellent storage stability of otherwise unprotected UFAP can be seen in Figure 20, showing the particle size analysis versus storage time of a sample of fluid energy milled UFAP stored with 30% Sorbeads. Table 23 also gives storage stability data for freeze-dried UFAP materials stored with 30% Sorbeads.

A number of factors were considered during selection of a shipping container for packaging UFAP and for running the shipping container detonation tests. These included cost and availability of the container itself, the degree of protection afforded from the outside atmosphere and the adequacy of the seal that could be obtained, the degree of confinement the container would provide in the event of initiation, container construction specifications already approved for Class A or Class B explosives, and the ease of handling. A five gallon steel pail, made to DOT Specification 37A-60 for single trip containers (STC), was ultimately selected as the best compromise. This item is a standard shop pail with a crimp-type, gasket-sealed lid and lifting bail. This limits the quantity of UFAP that can be packaged per container (to about 10 lbs. maximum, depending on bulk density), however, it is still much better than the limits imposed on Laboratory Samples of Unclassified, Experimental Explosives.

Other facets which were considered in selection of the five gallon container rather than a larger one were: the larger the container, the more "head" of material there is above the bottom layer. This "head" tends to compact the material beneath it, particularly during motion or handling, and could not only increase the chance of particle size growth due to forced contact between adjacent particles, but could increase the susceptibility of the material to detonation; the cost and availability of an adequate supply of suitable UFAP test materials for the detonation tests became increasingly important. Although the UFAP material was packaged in five-gallon size containers, a number of such containers could be strapped and bundled or palletized to allow shipment of several hundreds of pounds of material at a time.

The packaging technique which has developed using the five gallon pails comprised the following steps:

1. Blend fresh Sorbead desiccant into the UFAP to a content of 30% by weight of the mixture.

2. Charge the UFAP/Sorbead mixture to a clean, dry conductive polyolefin bag and seal the neck with tape.

3. Insert the bagged material into a second conductive polyolefin bag and seal the neck with tape.

4. Insert the doubly bagged material into the 5-gallon steel pail. Pour 100-200 grams of Sorbead desiccant into the pail on top of the bagged UFAP. Place the lid on the pail and crimp the securement ears.

5. Place one lapped wrap of sealing tape over the lid/pail joint.

6. Label pail contents appropriately, on the lid and on the pail body.

This technique has been used successfully for all types of UFAP dry powder products made at this facility, and provides good storage stability of the materials in uncontrolled storage areas such as igloos or magazines. It is highly desirable, however, that the packaging be done in a low relative humidity area to minimize particle size growth problems and to ensure the Sorbead moisture capacity is not exceeded.

Since the recommended storage container is not one specifically approved for Class A or Class B explosives for shipment purposes, as noted in DOT regulations for the Transportation of Explosives and Other Dangerous Articles, CFR Title 49-Transportation, specific tests were performed per Chapter 4 of DSAR 8220.1. This chapter specifies three types of experimental tests to be performed on packaged explosive items (other than finished rocket motors, or other devices containing solid propellants), for assignment of shipping and storage classifications. The type "A" detonation test determines whether a single live container will detonate when initiated. The type "B" detonation test determines whether explosion or detonation in one live container will propagate to an adjacent live container and cause it to function. The type "C" External Heat Test determines whether the material will undergo a detonation when completely enveloped in a hot fire. The specific test requirements designated in Section 4, Minimum Test Criteria for Bulk Propellant, of Table 4, of Chapter 4 of DSAR 8220.1, were selected for evaluating UFAP in the designated shipping container packages.

Procedures used to prepare and complete the tests are given in Appendix A. As can be noted, detonation wave sensors consisting of lengths of detonating Primacord, one end buried in the UFAP and the other running to a lead witness plate, were installed in each of the live containers in the Type A and Type B tests. Six Type A tests, and one each Type B and Type C, were run.

It has been hoped that the fluid energy milled UFAP would be of the same degree of explosive sensitivity as the other types of products, and therefore usable as a low cost test material. However, this material was considerably less sensitive than the SWECO-ground or freeze-dried materials. Additional card gap tests were run on SWECO dry powder containing one percent coating agent (Test Series 108) to see if it were more sensitive than the usual material containing only 0.4% coating. The results indicated little or no difference. Card gap and lead column tests were run on samples of freeze-dried material of the same formulation used in the large sublimator batches, (approximately 96% AP and 4% emulsifier/coating agents). Card gap tests were all negative at zero cards until the test material was packed into the tubes to abnormally high % TMD levels (see Test Series 105, Appendix D). Lead column tests run at low (loose-filled) density without Sorbeads were positive. Positive results were also obtained when 30% Sorbeads (which had been sufficient to desensitize the SWECO ground composition) were used. Additional lead column tests were run in which the sample geometry, the Sorbead content, and the % TMD were varied, and all were positive, even when the Sorbead content was raised to 50%. Since this material was not desensitized by the addition of Sorbeads, preventing it from being classified a Class B explosive hazard per the Chapter 3, DSAR 8220.1, tests, it nevertheless was a suitable material for the shipping container tests. The test results are contained in Table 24, and photographs of the test sites before and after the tests as well as of

significant after-test recovered components, are given on Figures 21 through 52. It should be evident from the data and photographs that none of the samples detonated, although all, or nearly all, exhibited pressure-burst type explosions. With the exception of the special Type A test, using SWECO-ground UFAP (Test Series 113), all of the tests resulted in essentially complete consumption of the UFAP test material. Comparsion of the results obtained from the single Type A test made with SWECO ground UFAP with those made with the freeze-dried material visibly confirm that the freeze-dried material is more explosive of the two, as predicted by lead column tests. Consequently, conclusions drawn from the tests on freeze-dried UFAP can be applied to less sensitive material.

Since none of the Type A tests resulted in detonations, there was some question whether the Type B test was even needed; however, it was run to see if the combustion/explosion reaction would cause adjacent containers to function. Since the acceptor charge exploded several feet above ground, and at least a finite time after the donor charge was initiated, it is theorized that the acceptor container was thrown into the air, and probably partly opened up, when the donor was exploded. As it fell back to earth, UFAP spilling out caught fire and the flame propagated back up into the falling container, causing the remaining material to explode.

The container ruptures that occurred in the Type C external heat tests could possibly have occurred from any of several mechanisms. Since the lids from all the containers were bowed upwards internally, it is obvious that they were blown off by internal pressure inside the containers. Whether the pressure came from expansion of the air inside the containers or from decomposition of some of the UFAP from "cooking off" is unknown. Since the container bottoms and side walls were not significantly damaged by the explosions (as evidenced by lack of rupture and only slight bulging of the bottoms), one explanation is that air expansion and/or "cook-off" decomposition of some of the UFAP blew the lids off as well as expelling most of the remaining UFAP as a cloud. The cloud then ignited and exploded in the air, causing the mild reports heard by the observers. Regardless of the mechanism involved in the actual test, the most important result was that no detonation occurred.

Because of the anomalous results, difficulties are encountered in interpreting the results and applying classifications. For example, the shipping container tests indicate that UFAP, even the most sensitive material by other tests, when packaged by the prescribed procedure, should be assigned an DOT Class B Explosive hazard rating. Lead column tests, however, indicate the material should be expected to detonate, warranting a Class A designation. Card gap tests, using booster charges and no attenuation gap indicate the material is not detonable, unless highly compressed, but that it does undergo energetic chemical reaction, as evidenced by witness plate deformation. The following theory is offered to explain some of the anomalous results: the critical factor in the sensitivity of a UFAP product is the ratio of oxidizer to fuel. Pure AP, such as fluid energy milled material otherwise uncontaminated with combustible materials would therefore be the least sensitive. As the content of combustibles (coating agents for instance) is increased, the sensitivity increases due to the greater degree of complete combustion. When the fuel content gets above some level between one and four percent of the mixture, the sensitivity is great enough that solid diluents such as Sorbeads can no longer suppress the rate of combustion of the properly initiated material to prevent mushrooming of a lead witness column. The combustion reaction is not of sufficient velocity, however, even without any Sorbeads, to punch a card gap witness plate unless the sample density is comparatively high.

Assuming the above concept to be true, two possible conclusions can be reached: one, based on the shipping container tests alone, all the UFAP dry powder materials tested in this program should be designated Class B explosive hazards when packaged per the procedure; or, two, based on lead column tests and the shipping container tests, UFAP dry powder products containing some threshhold, or lower, should be designated Class B explosive hazards when packaged per the procedure, while all other materials should be assumed Class A explosive hazards unless proven by otherwise testing. Final hazards classification assignment must be made by DOT and/or the cognizant DOD agency.

TABLE 23

STORAGE STABILITY DATA FOR FREEZE-DRIED UFAP STORED WITH SORBEADS

SAMPLE AND DESCRIPTION	When[c] Made	Particle Size/Moisture of Samples[a] When Made	Days of Storage[b] 0[d]	14	35
FDI-9A, (0.1% J-wax, 1.0% Nopcogen 16-0)	6/20/72	0.96/0.068	1.29/0.045	1.33/ -	1.22/0.040
FDI-10B, (0.1% J-wax, 1.0% Nopcogen 16-0)	6/23/72	0.71/0.036	0.70/0.054	0.74/ -	0.73/0.055
FDI-14A, (0.65% J-wax, 10% Nopcogen 16-0 , 1.5% JB, 1.5% XB-23)	8/7/72	0.37/0.38	0.35/0.321	0.38/ -	0.36/0.298

Notes:
a) Particle size in microns WMD/percent water by weight.
b) Material removed from cold box storage on 11/21/72, allowed to thaw out at room temperature while still sealed, sampled for zero time analysis, then repackaged with 30% Sorbeads in quart fruit jars. Analyses run periodically on specimens removed from jars. All jars stored at ambient temperature (77°F).
c) When first removed from freeze-drying sublimator. Material immediately sealed in 5-gal. pails and stored in deep-freeze until 11/21/72.
d) Sample removed on 11/21/72 just before Sorbeads added.

TABLE 24

UFAP SHIPPING CONTAINER DETONATION TESTS

Test Series No.[b]	Date of Test	Type of Test	Test Operating Procedure No.	UFAP Source Designation	Composition Reference[c]	Atmospheric Conditions	Test Results[d]
109	2/14/73	Detonation Test A	PE 73-96	FDB-7	III-L.	Cool, overcast, cloudy day, just starting to rain when test initiated, temperature 50°F.	Test material exploded, but did not detonate. Confinement sand-filled containers blown all over areas, the furthest about 30 feet. Wood pallet destroyed, but gravelled site only slightly disturbed under test site. Primed container blown apart, but lid, cover, and side wall each recovered essentially intact. Primacord detonation sensor unaffected. Visible smoke cloud hung over area after test.
110	2/21/73	Detonation Test A	PE 73-106	FDB-7	III-L.	Cool, clear, sunny day; partly overcast. Temperature 45-50°F	Test material exploded with loud report, but did not detonate. Confinement containers scattered about the area. Pallet destroyed, but gravelled test site only slightly disturbed. Started several grass fires, and a portion of the plastic liner bag stuck on the primed container side wall was burning. Cover, bottom, and side walls recovered as individual, single pieces. Primacord severed at container wall, but recovered unaffected.
111	2/21/73	Detonation Test A	PE 73-106	FDB-7	III-L.	Bright, clear, sunny day. Temperature 60°F.	Test material exploded, but did not detonate. Same general results as for Tests 109 and 110, except that primed container side wall was torn into a number of pieces, not all of which were recovered. A number of grass fires were started. Confinement containers generally blown further than previous tests, but none were more than 30 feet from the test site.
112	2/22/73	Detonation Test A	PE 73-106	FDB-7	III-L.	Cool, clear, sunny day. Temperature about 60°F	Same result as tests 109-111. Container side walls ripped into a number of pieces.
113	2/22/73	Detonation Test A	PE 73-106	VMA-97	I-D.	Cool, sunny day. About 60°F.	Only part of the test material exploded. Top center confinement container shot out of stack like from a mortar. Cover of primed container blown off. Only two of the other confinement containers were damaged sufficiently to prevent reuse. Pallet was undamaged. Some 5.71 lbs. of UFAP and 2.56 lbs. of Sorbeads remained in the container. Primacord not even severed. Unreacted UFAP dust scattered over immediate area, and yellow reaction products smeared on outsides of remaining containers.

TABLE 24 (continued)

Test Series No.[b]	Date of Test	Type of Test	Test Operating Procedure No.	UFAP Source Designation	Composition Reference[c]	Atmospheric Conditions	Test Results[d]
114	2/23/73	Detonation Test A	PE 73-106	FDB-7	III-L	Cool, clear, sunny day. About 40°F.	Test set up using pallet and ten of the confinement pails from Test 113. Material exploded as in Tests 109-112, but did not detonate. Exactly the same as previous results.
115	2/27/73	Detonation Test B	PE 73-108	FDB-8	III-M	Cloudy, cold, overcast day. Temperature about 45°F.	Primed donor container exploded, blowing other containers over the area, and destroying the portion of the pallet beneath it. Cover, side walls, and bottom blown apart but recovered in single pieces. Less than a second after the donor report, the acceptor charge exploded in midair, about 3 or 4 feet above the ground. A large yellow-orange flash occurred with this report, and a large smoke cloud formed. Side wall of acceptor charge, with plastic liner and coating of unburned UFAP, found about 30 feet away. Cover of acceptor was found within a few feet of the test site. The bottom of the acceptor was blown about 110 feet away. Primacord from both containers severed at the can walls, but not initiated. Pallet not destroyed under acceptor.
116	2/28/73	External Heat Test C	PE 73-110	FDB-8/ FDI-52	III-M/ III-J	Cool, overcast, hazy day. Temperature about 50°F.	Diesel fuel and wood scraps burned with large flame and heavy black smoke when ignited. After a finite time, UFAP containers apparently burst open in groups of twos, blowing the covers off and into the air, carrying the necks of the plastic liners with them. Covers were blown 20-30 feet high, with mild reports. Fuel oil continued to burn for about 4 hours. Containers were physically intact, and four still contained considerable quantities of Sorbeads. Sorbeads were scattered over the area within a radius of about 25 feet. Furthest cover was about 32 feet from the test site. Several covers still had legible labels on them. All covers were bowed upwards, indicating they were deformed then blown upwards by internal pressure.

NOTES: a. Per Chapter 4, DSAR 8220.1, Explosives Hazard Classification Procedures, (formerly TB700-2).
b. Internal test operations logging number.
c. See tables in Appendix B for composition, particle size, and manufacture details.
d. Before and after photographs of each test are presented as Figures V-2 through V-33.

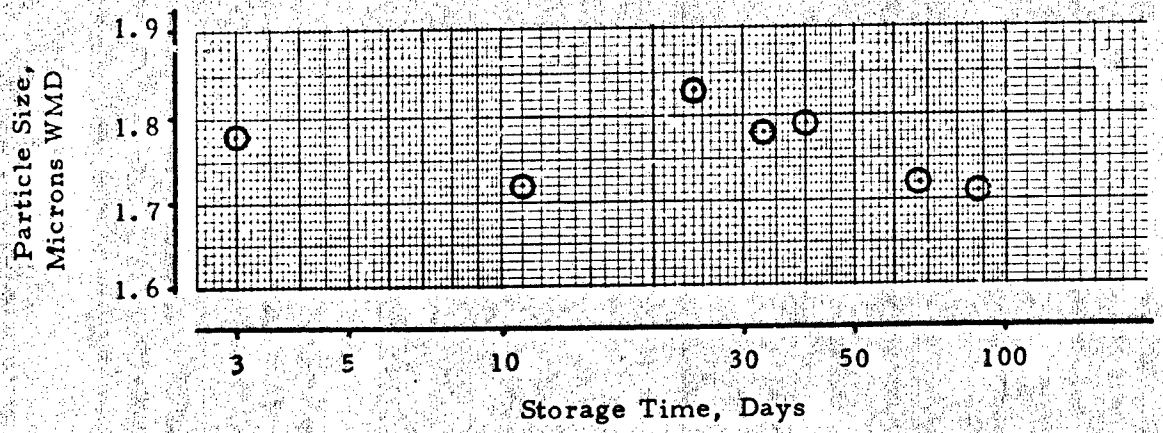


Figure 20. Nominal 1.7 Micron AP (Grind EFEM-49), Stored at Ambient Temperature in Sealed Half-Gallon Jars, with 30% Sorbeads

Figure 21. Palletized Test Assembly, First Type A Test

Figure 22. Conductive Plastic Weather Shield Over Palletized Test Assembly

Figure 23. Test Site, Looking North, After Initiation, First Type A Test

Figure 24. Test Site, Looking East, After Initiation, First Type A Test

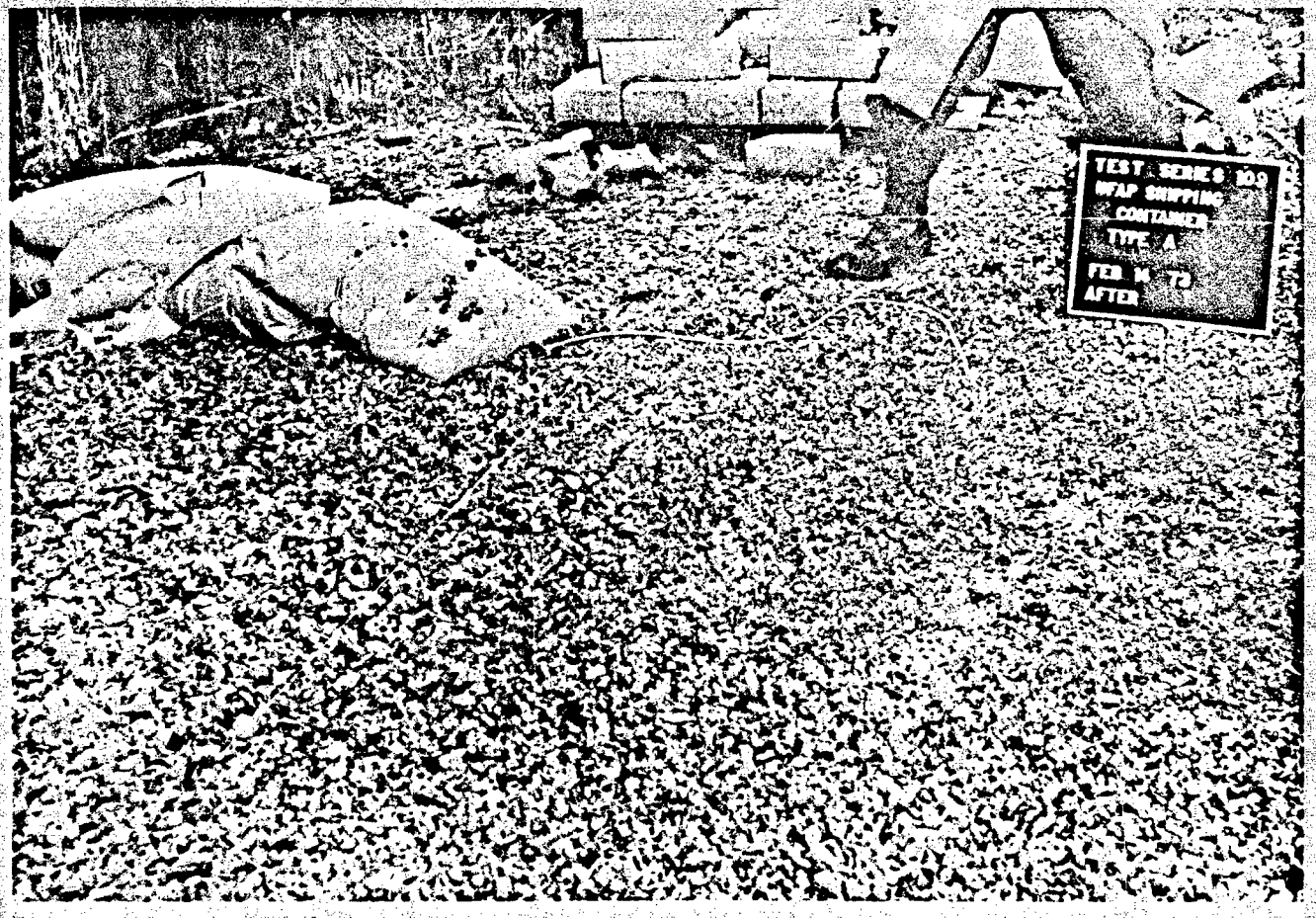


Figure 25. Immediate Area of Test Site After Initiation, Showing Unreacted Primacord, First Type A Test

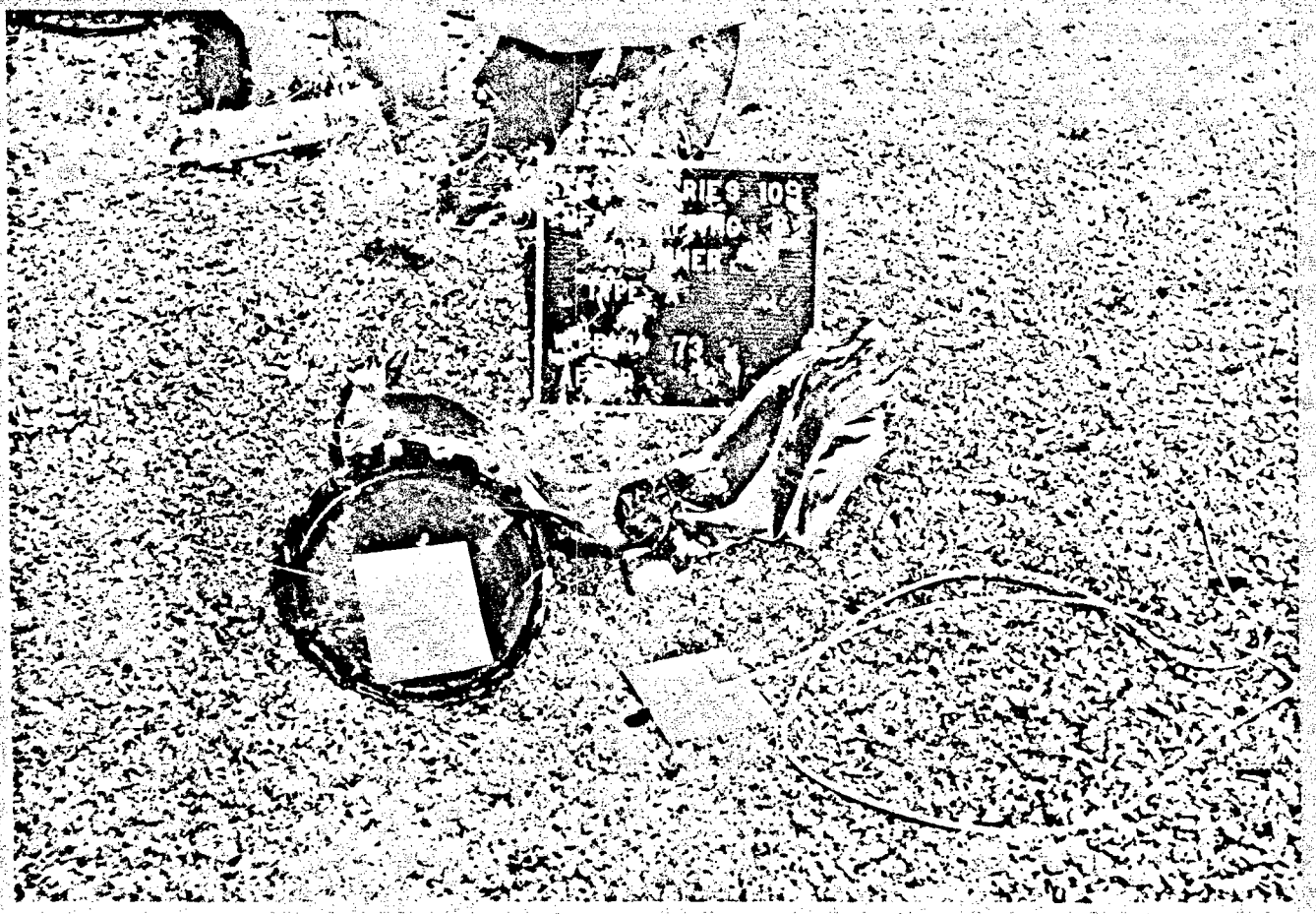


Figure 26. Parts of UFAP Container Found After Test and Primacord/Witness Plate, First Type A Test

Figure 27. Palletized Test Assembly, Second Type A Test

Figure 28. Test Site, Looking East, After Initiation, Second Type A Test

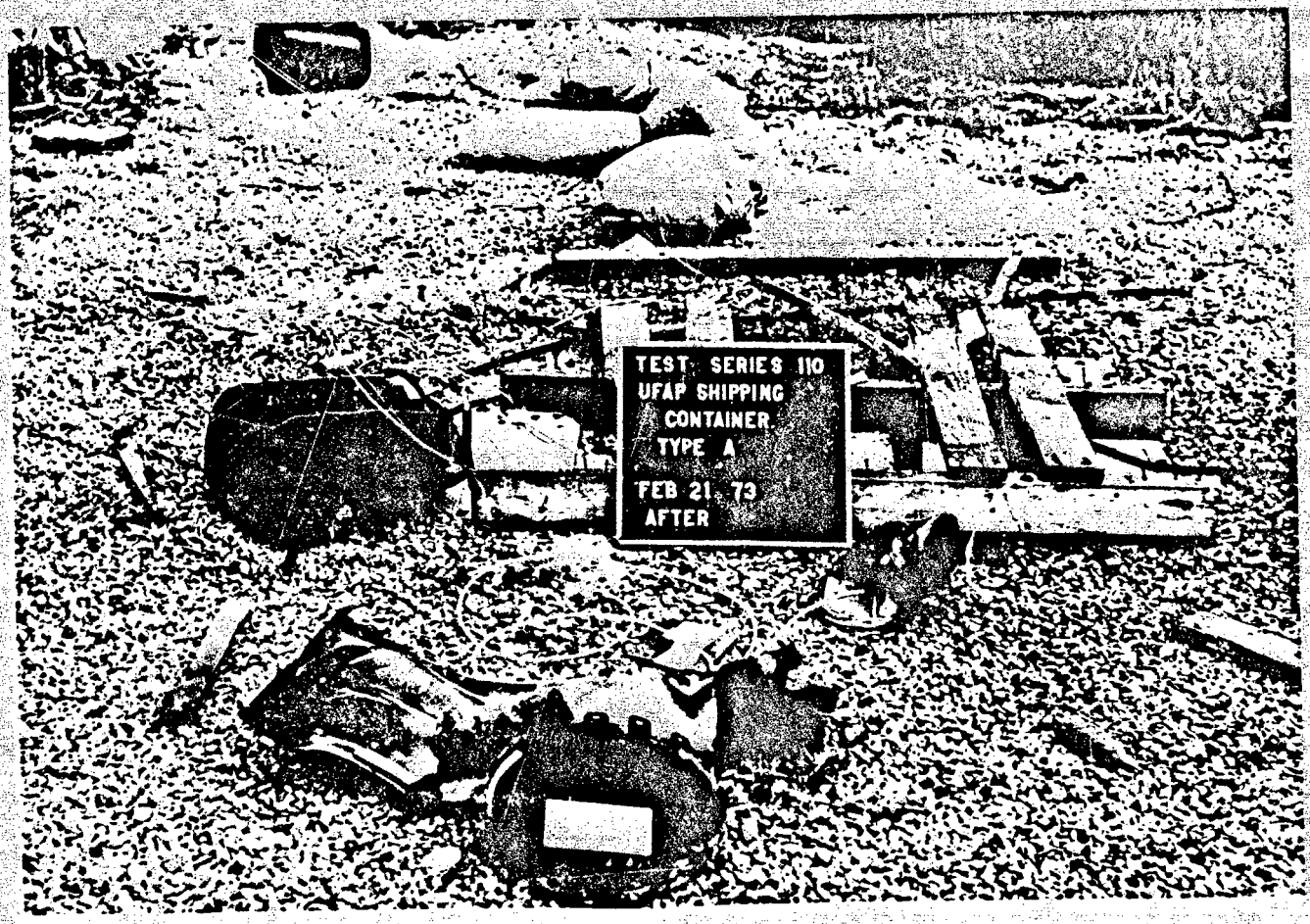


Figure 29. Parts of UFAP Container Found After Test, Primacord/ Witness Plate, and Other Debris, Second Type A Test

Figure 30. Palletized Test Assembly, Third Type A Test

Figure 31. Test Site, Looking East, After Initiation, Third Type A Test

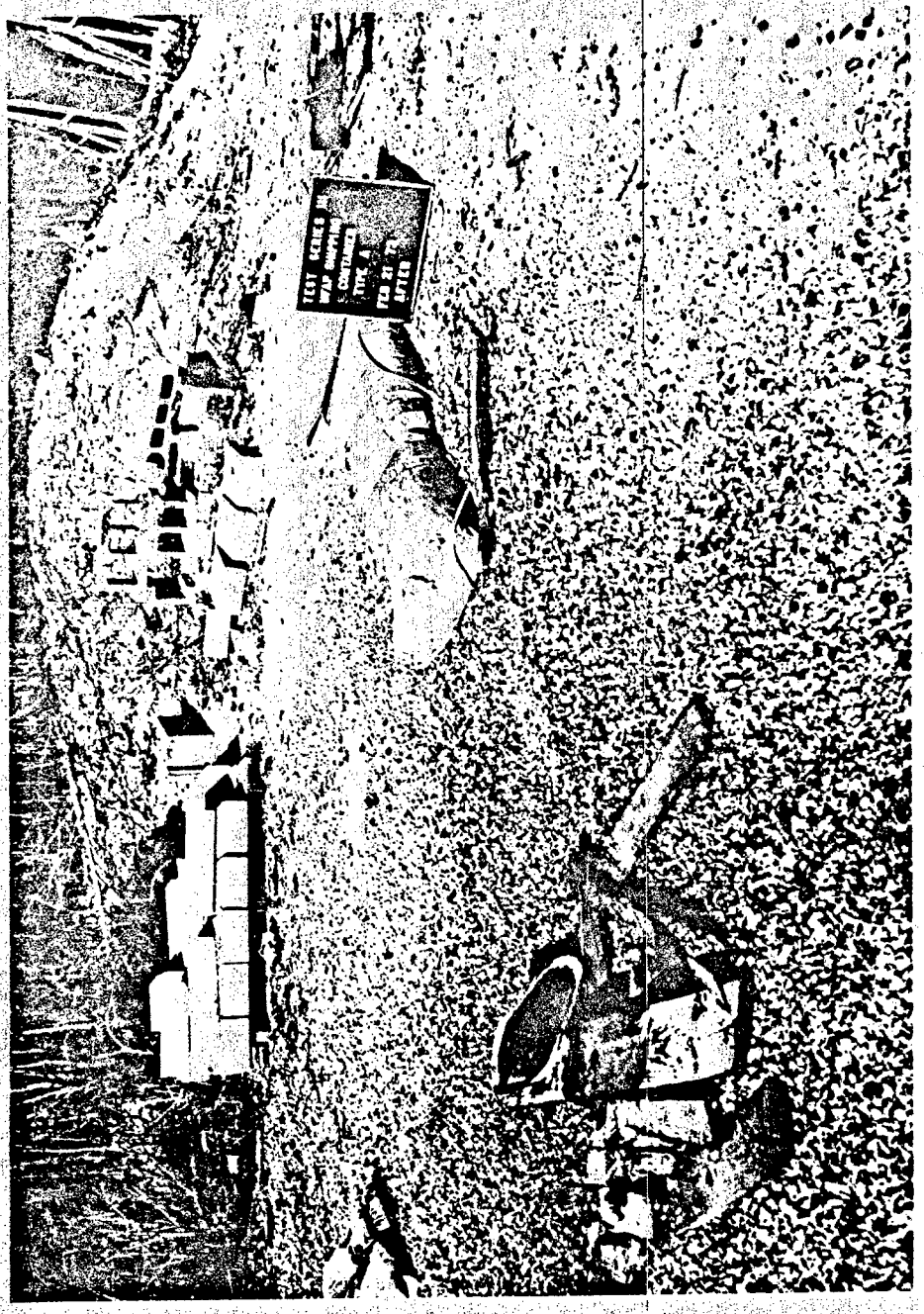


Figure 32. Immediate Area of Test Site After Initiation, Third Type A Test

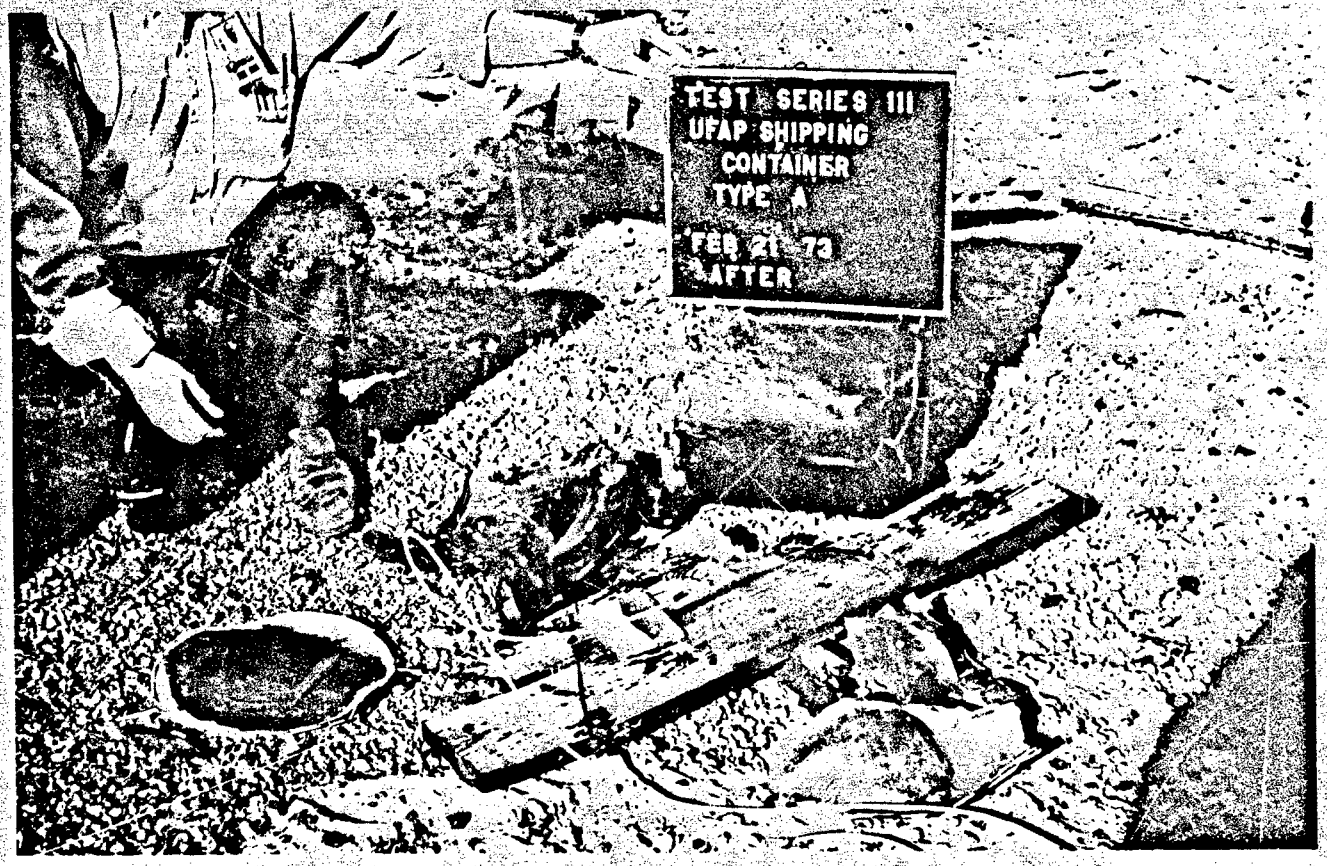


Figure 33. Parts of UFAP Container Four After Test, Primacord/ Witness Plate and Other Debris, Third Type A Test

Figure 34. Palletized Test Assembly and Sandbags Protecting Primacord and Witness Plate, Fourth Type A Test

Figure 35. Test Site, Looking East, After Initiation, Fourth Type A Test

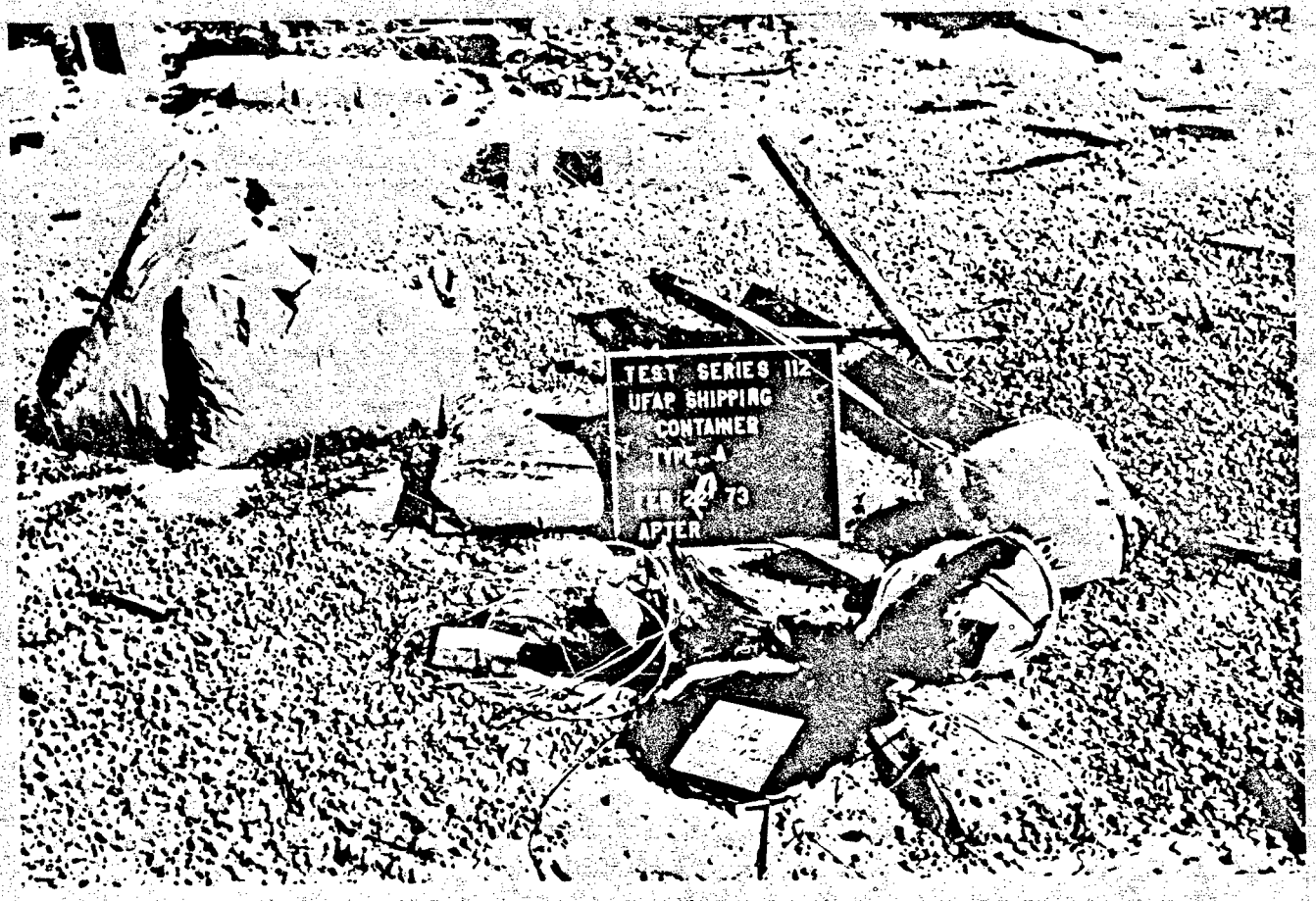


Figure 36. Parts of UFAP Container Found After Test, with Primacord/Witness Plate and Other Debris, Fourth Type A Test

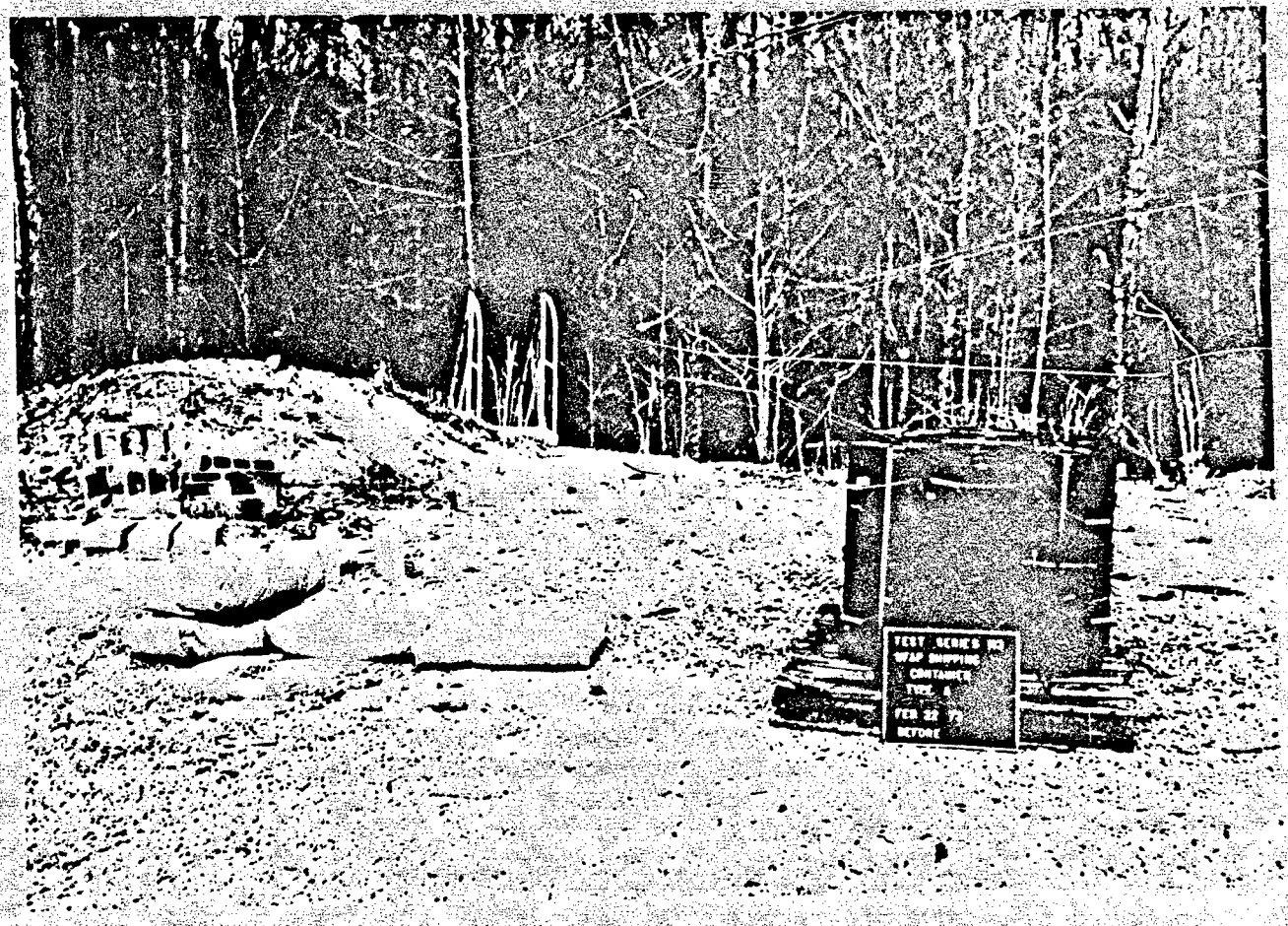


Figure 37. Palletized Test Assembly, Special Type A Test

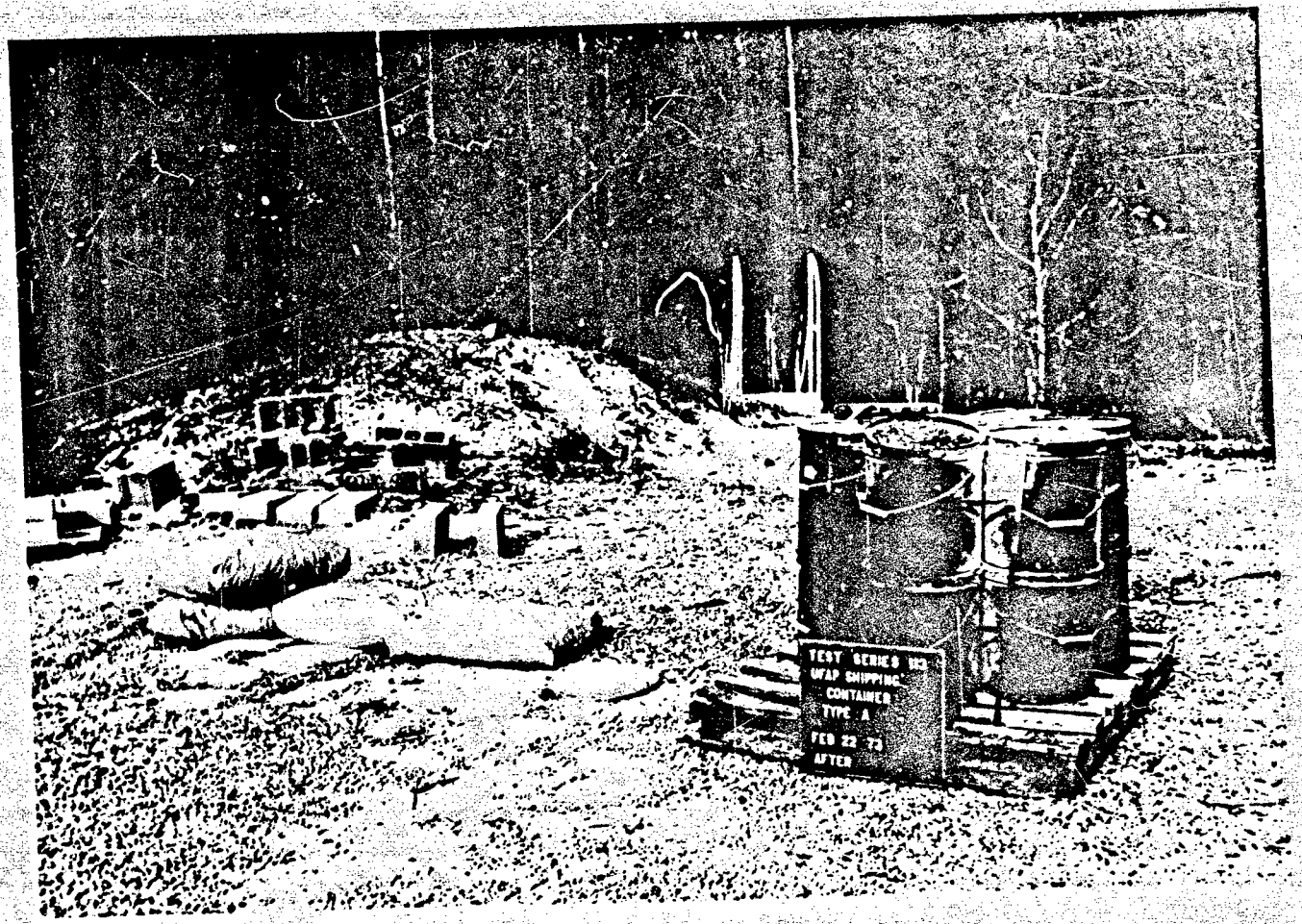


Figure 38. Test Assembly After Initiation, Special Type A Test

Figure 39. Undisturbed View of Top of Test Assembly After Initiation, Unconsumed UFAP and Sorbeads Covering Tops of Sand-Filled Containers, Special Type A Test

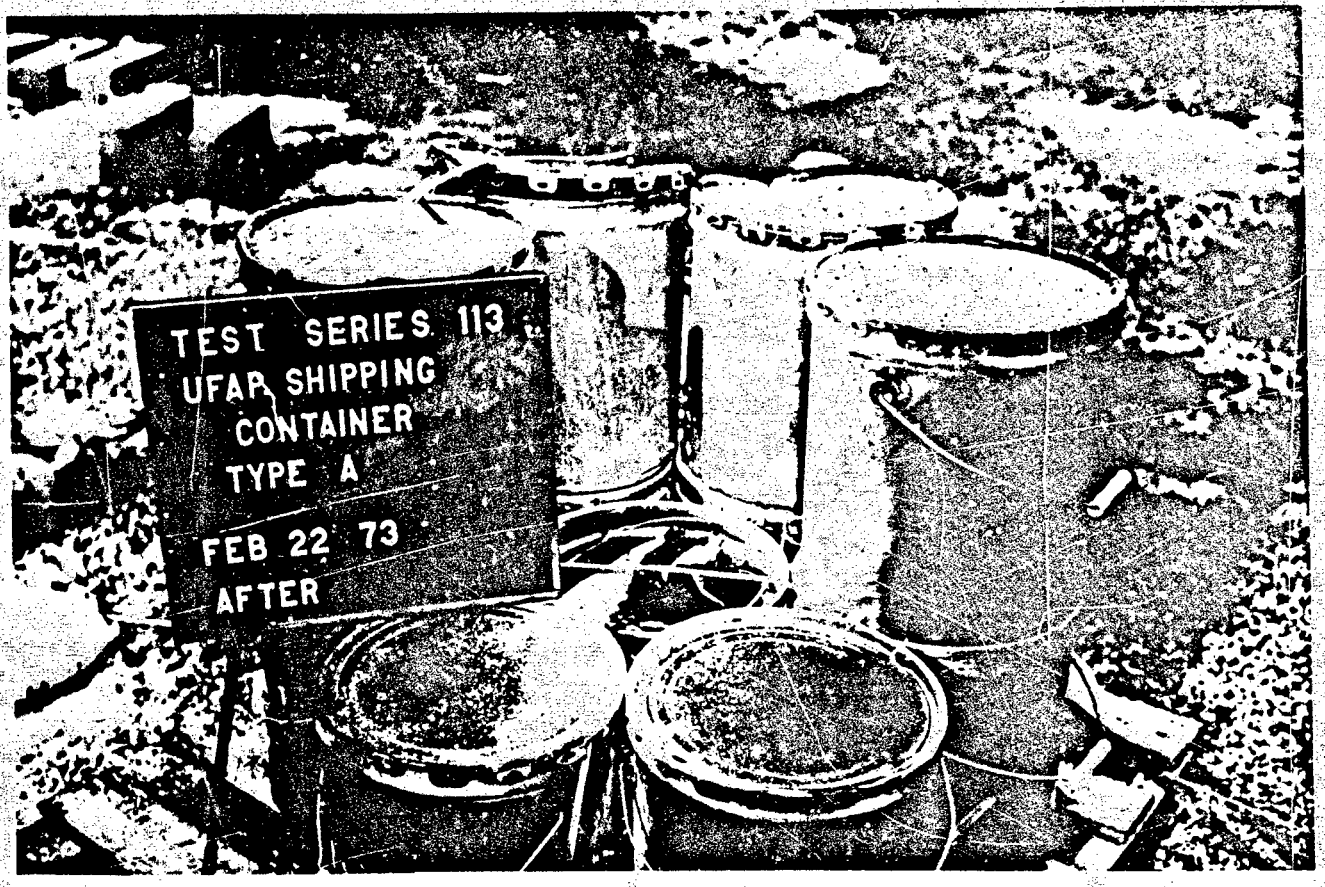


Figure 40. View of UFAP Container in Center of Bottom Layer of Containers (two sand-filled containers removed from upper layer), Special Type A Test

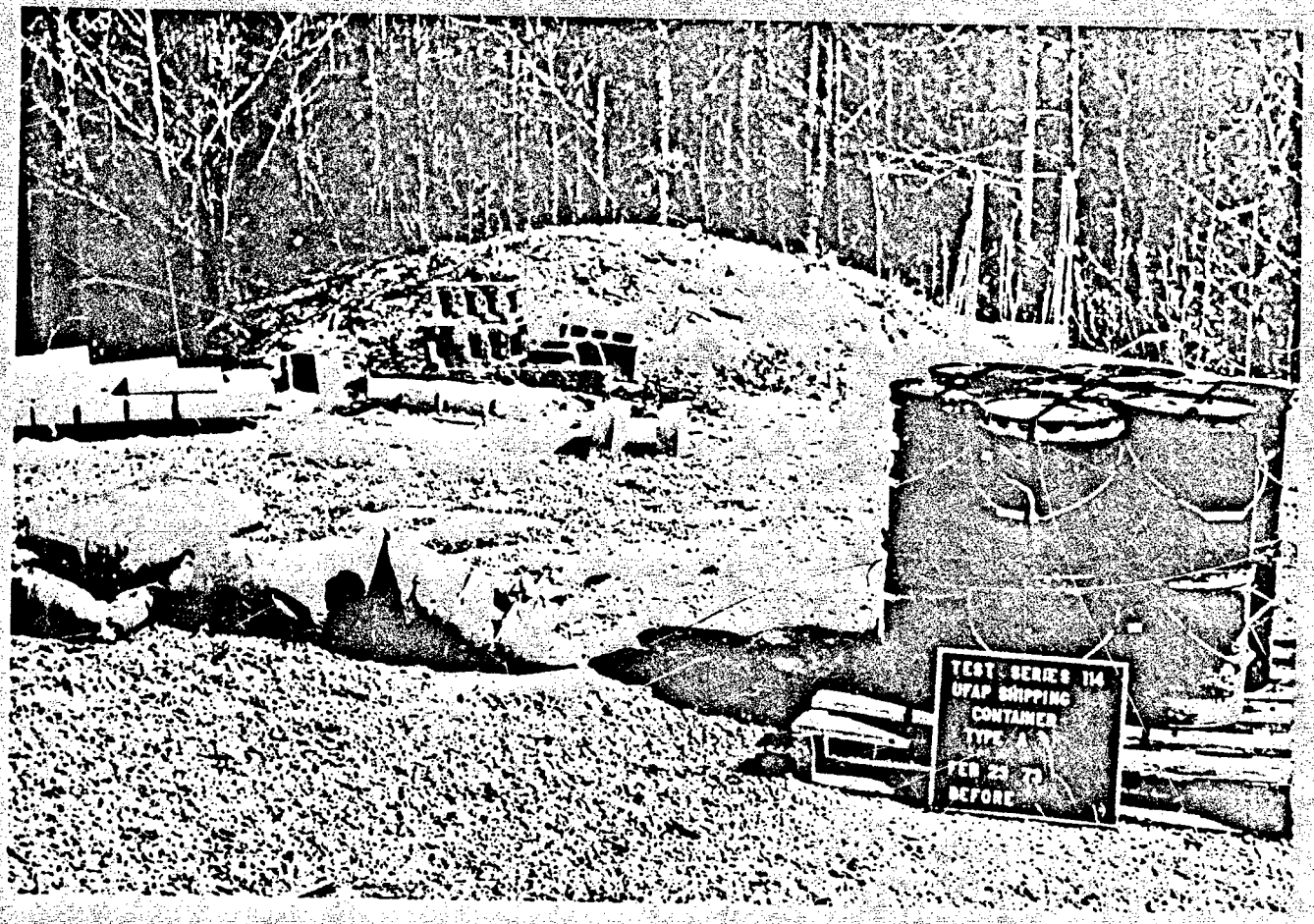


Figure 41. Palletized Test Assembly, Fifth Type A Test

Figure 42. Test Site, Looking East, After Initiation, Fifth Type A Test

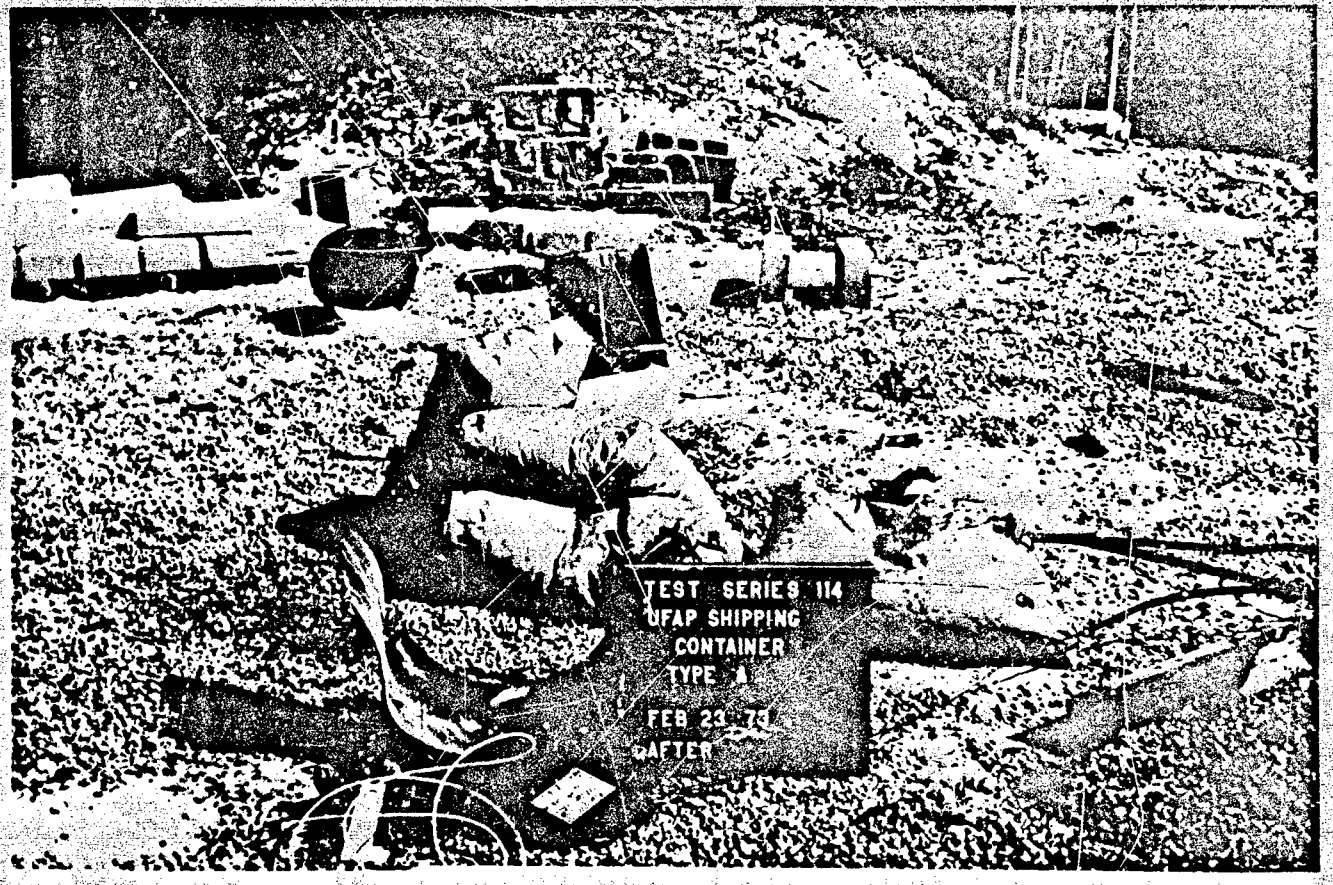


Figure 43. Parts of UFAP Container After Test, Primacord/Witness Plate and other Debris (note undamaged label on container lid), Fifth Type A Test

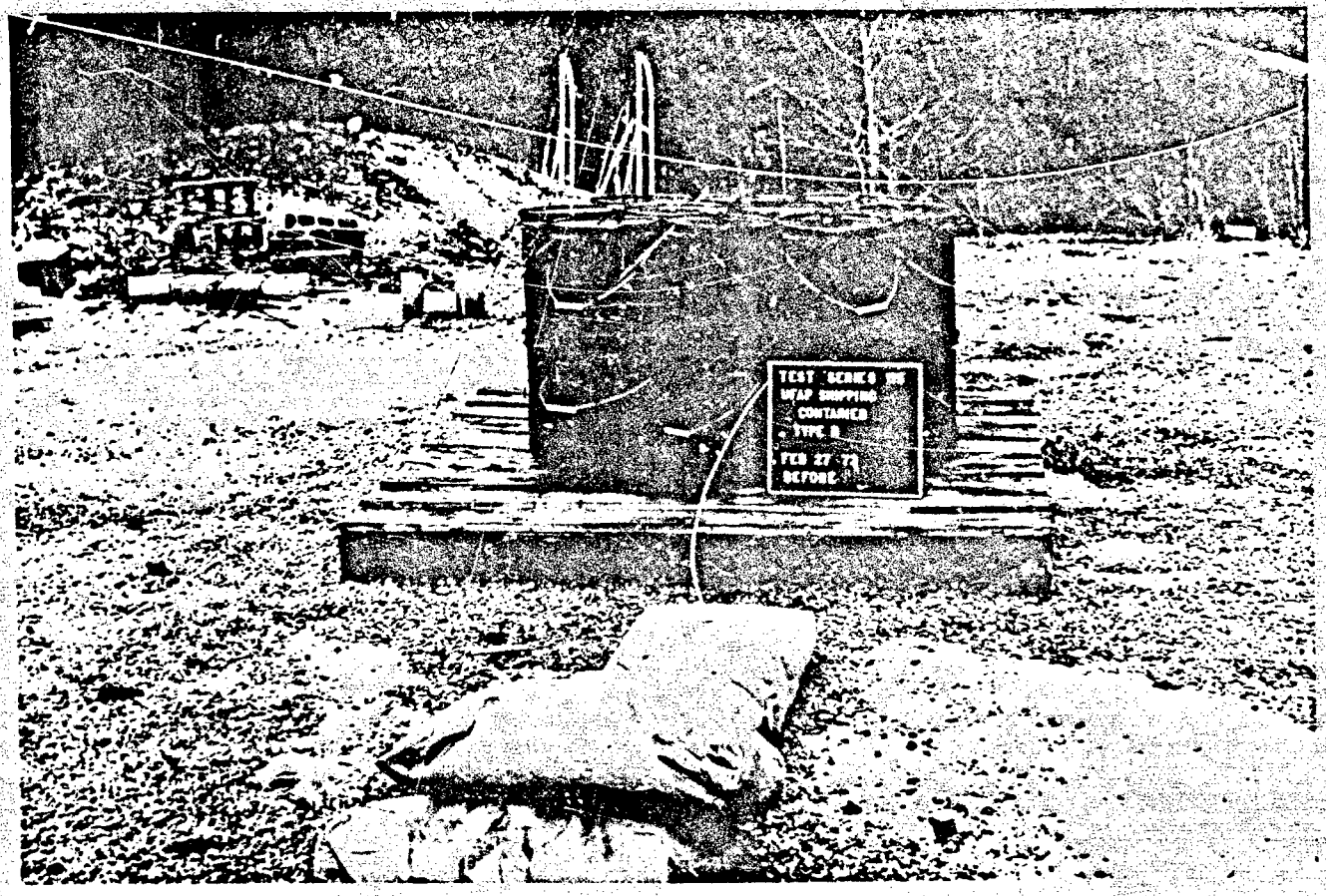


Figure 44. Palletized Test Assembly, Looking East, Primacord coming from donor (initiated) UFAP Container, Type B Test

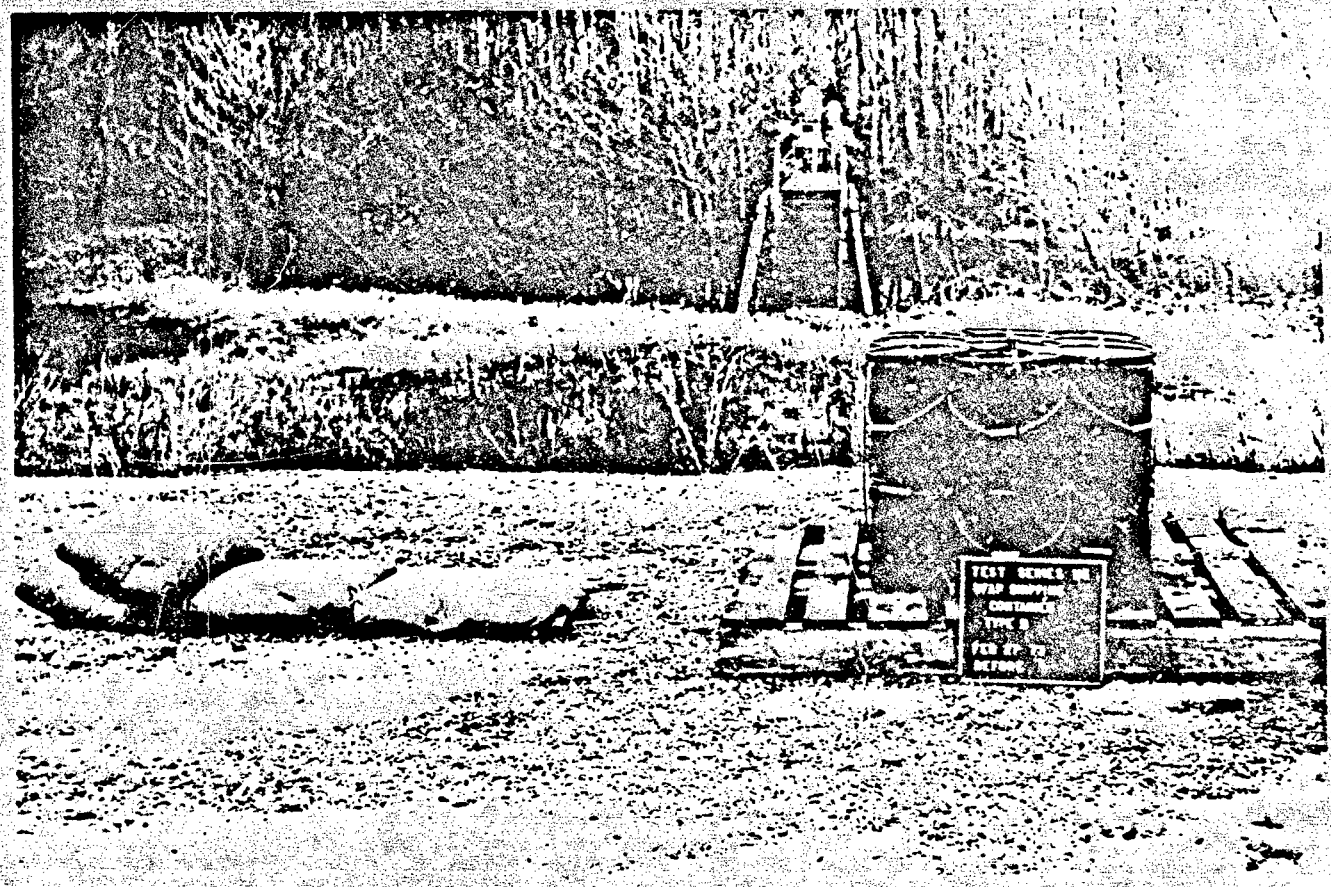


Figure 45. Palletized Test Assembly, Looking North, Primacord from Donor Container to Left and from Acceptor Container to Right, Type B Test

Figure 46. Test Site, Looking North-East, After Initiation, Type B Test

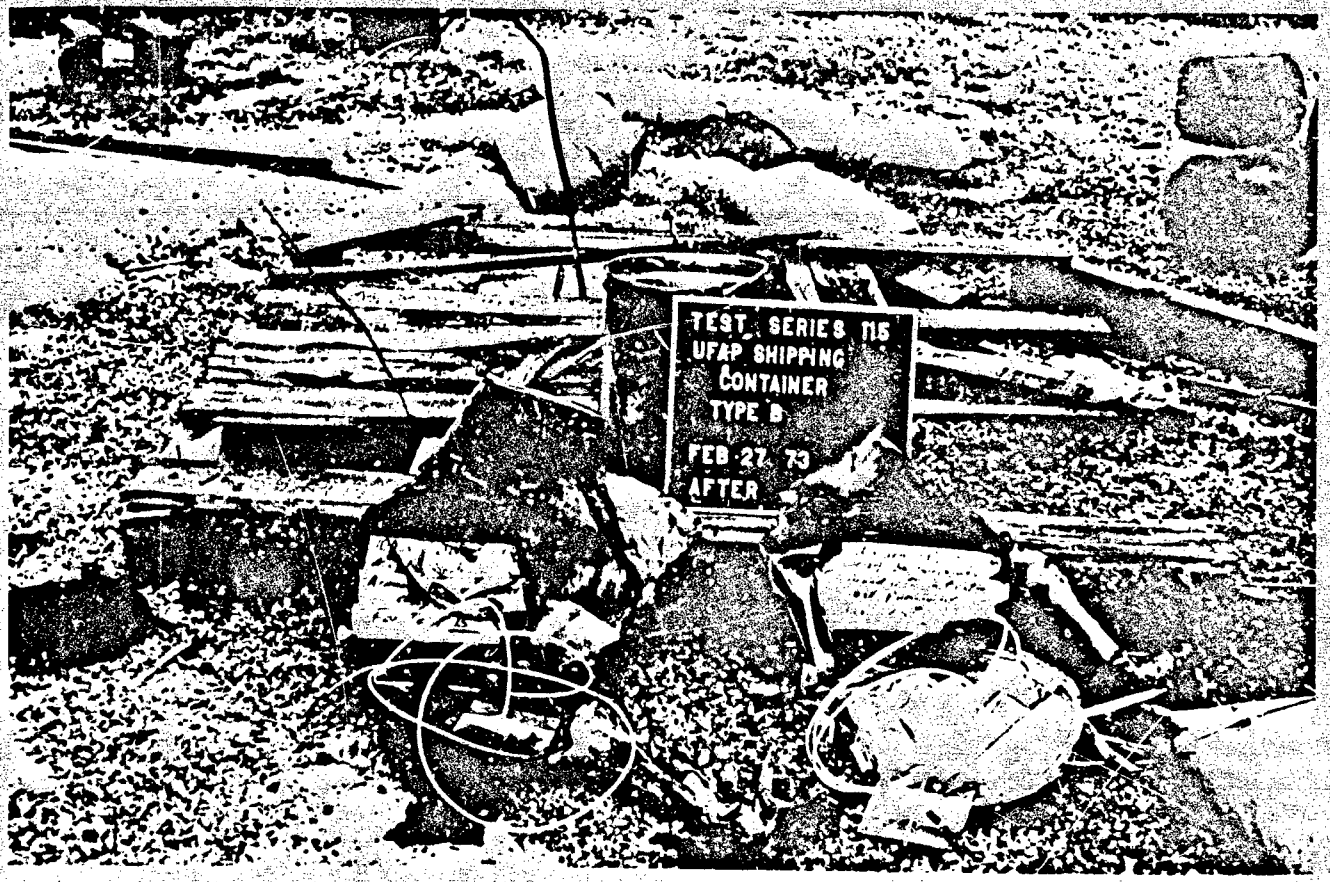


Figure 47. Parts of UFAP Containers After Test, Primacord/Witness Plates and other Debris, Acceptor Container on left, Donor on right, Type B Test

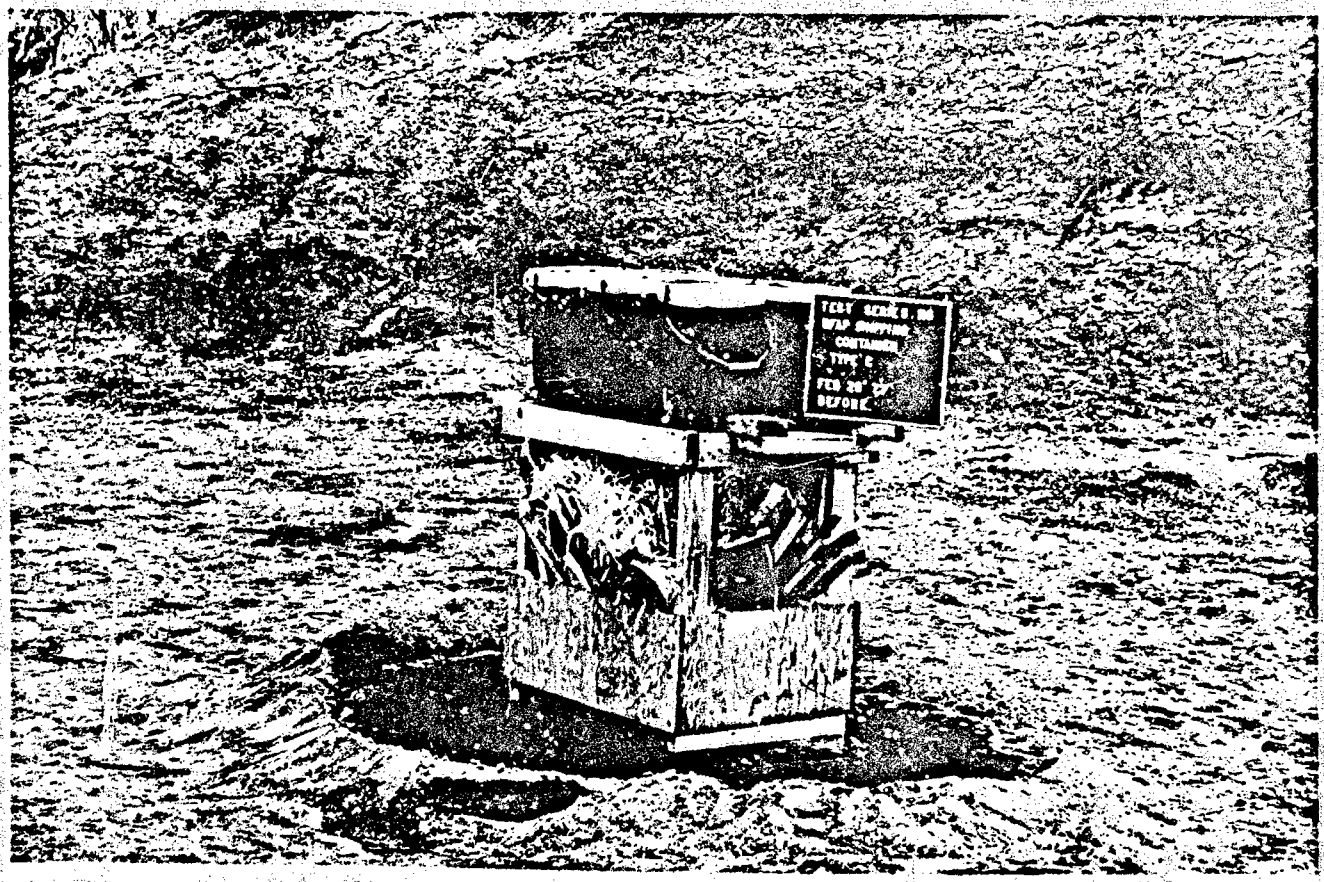


Figure 48. Test Assembly of Bundled Containers on Top of Crib Filled with Lumber and Sawdust, Crib Saturated with Diesel Fuel, Looking North-West, Type C Test

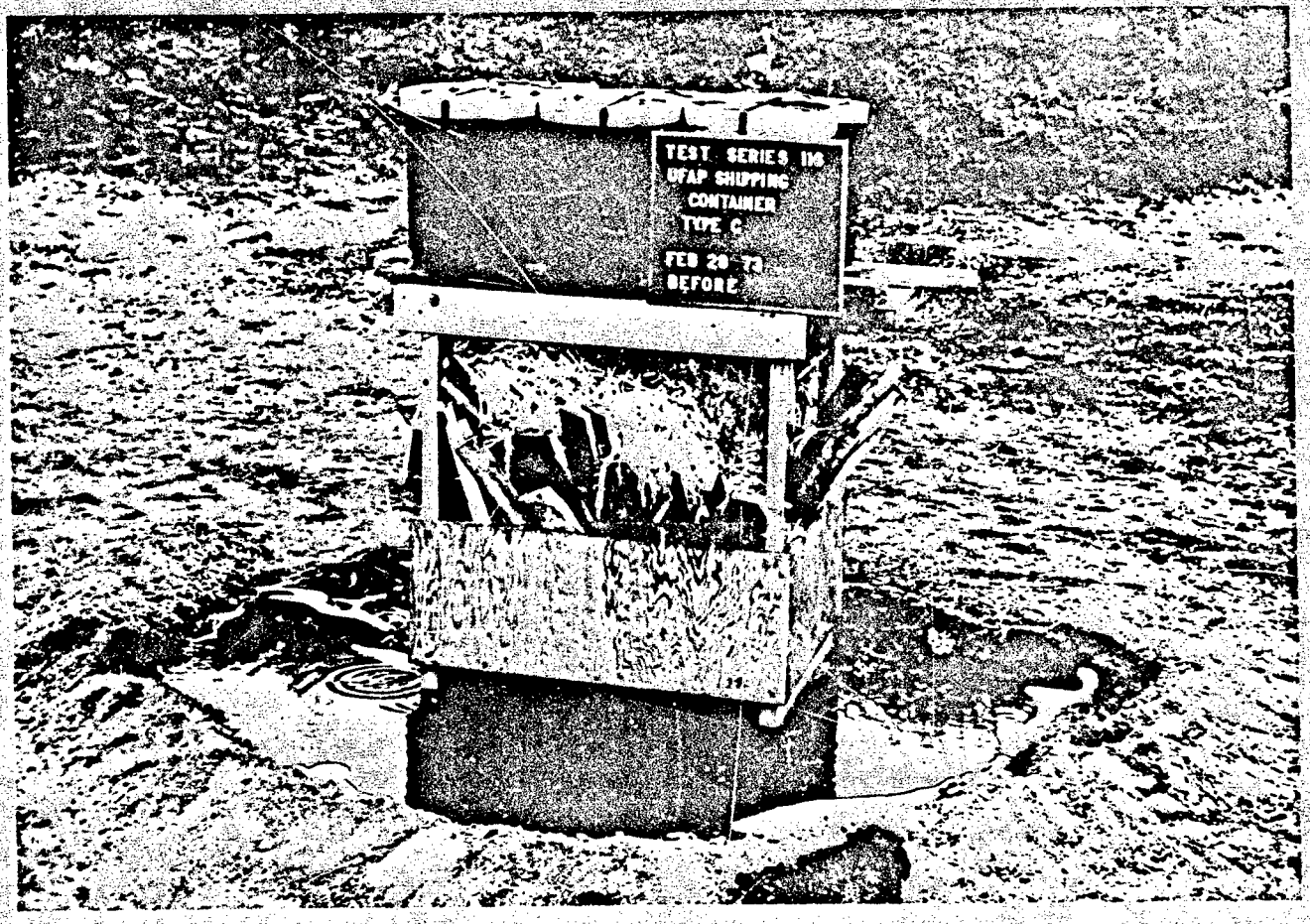


Figure 49. View of Test Assembly, Looking North, Type C Test

Figure 50. Test Site (after burning had ceased), Looking East, Lids and Black Plastic Liners Scattered over Area to Right, Type C Test

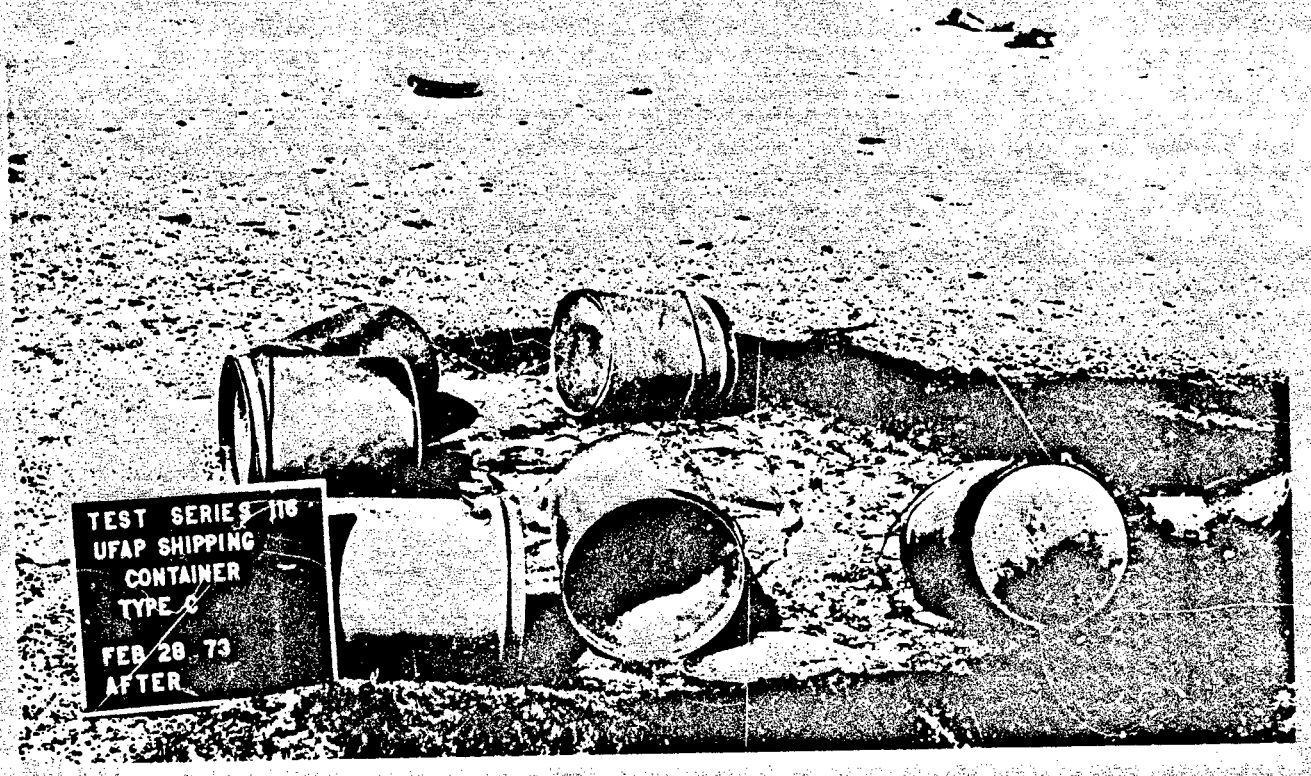


Figure 51. Immediate Area of Test Site (after burning ceased), Undisturbed Position of Containers and Lids (Note Sorbeads in container at center foreground), Type C Test

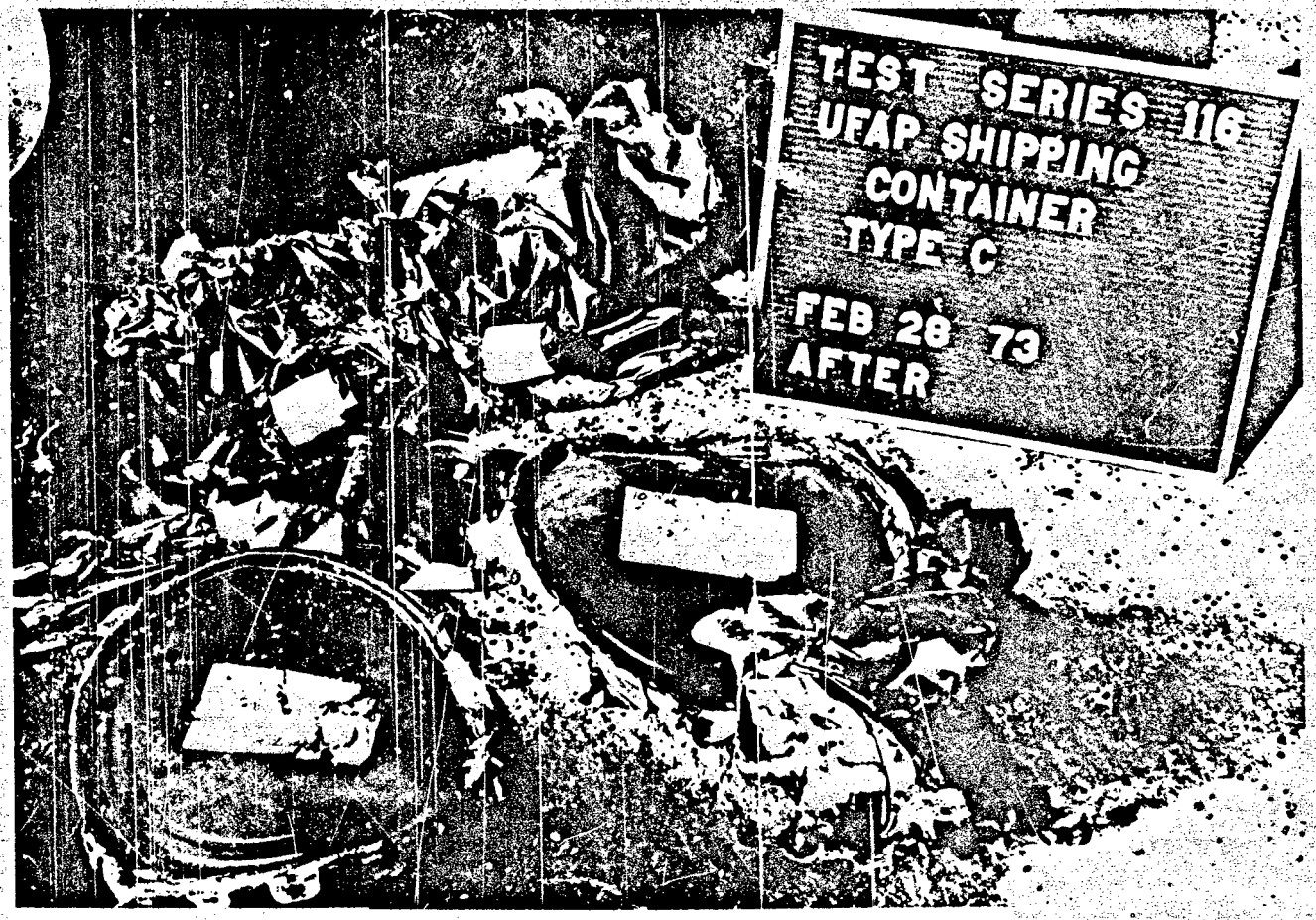


Figure 52. Two Container Lids, With Still Legible Labels and Tape-Sealed Necks of Plastic Bag Liners, After Test, Type C Test

TASK VI - UFAP BALLISTICS

The primary objective of this task was to determine that AP size distribution parameter which is best correlated with burn rate of propellants containing UFAP. In addition, assessment of the ballistic reproducibility from "lot to lot" and "within lots" for propellants containing UFAP will be made. Of further interest was a determination of the effect of UFAP age and storage conditions upon the ballistics of propellants containing UFAP.

In order to ascertain that AP size "distribution describing parameter" which best correlates with the burn rate of UFAP containing propellants, 13 blends were selected for characterization. These 13 blends were made from five different particle sized AP. These size fractions were 0.6 micron (SWECO ground), 1.0 micron (SWECO ground), 1.9 micron (fluid energy milled), 5.2 micron (fluid energy milled), and 16 micron. The blend compositions are shown in Table 25. Note that the first four bimodals all contain 50% of the 0.6 micron material in conjunction with 50% of each of the other sizes. Similarly the next four bimodal blends all contain 75% of these 0.6 micron materials. The trimodals contain equal amounts of each fraction. The 0.6 μ and 1.0 μfractions were coated with 0.4% HX-868.

The surface areas and particle sizes were measured on each of these 13 blends. The surface area was measured by Krypton absorption and particle size was measured by the MSA technique. From these two data points, a series of "reproducibility factors" were calculated on the computer for each of the 13 blends. Among these "reproducibility factors" are the average mean diameter, the mean surface diameter, the linear mean diameter, etc. The values for these "reproducibility factors" are shown in Table 26. In order to determine which of these "reproducibility factors" best correlates with burn rate, a series of 13 mixes was manufactured with each one of the aforementioned blends. The HTPB mix data is shown in Table 27. In addition, each of the mixes was manufactured with 1% iron oxide incorporated in order to determine if the presence of a catalyst changed the correlation. These data are shown in Table 27.

Following cure, the burn rates were determined for each of the 26 mixes (Table 28). The expected general trend of increasing burn rate with increased surface area or decreased size is apparent in uncatalyzed formulations, except in those formulations containing high concentrations of the smaller size fractions, (e.g., the lower than expected burn rates in formulations 1, 5, and 9). The catalyzed propellants exhibit the same general trend (formulations 5 and 9). The most plausible explanation for these observations is a lack of good dispersion of the UFAP in these

TABLE 25

OXIDIZER BLENDS FOR BALLISTIC CHARACTERIZATION

	MIX NUMBERS												
AP Size	1	2	3	4	5	6	7	8	9	10	11	12	13
0.6 micron	50	50	50	50	75	75	75	75	100	33.3	33.3	33.3	33.3
1.0 micron	50	---	---	---	25	---	---	---	---	33.3	---	---	33.3
1.9 micron	---	50	---	---	---	25	---	---	---	---	33.3	---	---
5.2 micron	---	---	50	---	---	---	25	---	---	33.3	33.3	33.3	
16 micron	---	---	---	50	---	---	---	25	---	---	---	33.3	33.3

TABLE 2

AP REPRODUCIBILITY FACTORS

Mix No.	Run	SSA	Weight Mean Diameter	Average Mean Diameter	Mean Surface Diameter	Linear Mean Diameter	HMD	MVD	SMD	MWD
1	2591	5.29203	1.02	0.286	0.327	0.374	0.2450	0.47	0.58	0.3961
2	2592	4.2	1.32	0.237	0.301	0.361	0.2450	0.48	0.72	0.3804
3	2593	3.61768	2.72	0.267	0.312	0.364	0.2351	0.55	0.84	0.4341
4	2594	3.64270	8.84	0.246	0.280	0.319	0.2180	0.52	0.84	0.4043
5	2595	5.89907	0.97	0.267	0.301	0.340	0.2340	0.42	0.52	0.3620
6	2596	5.54146	1.06	0.262	0.300	0.342	0.2295	0.44	0.56	0.3684
7	2597	5.10620	1.81	0.256	0.290	0.328	0.2274	0.44	0.60	0.3701
8	2598	4.96916	4.90	0.255	0.286	0.321	0.2272	0.45	0.62	0.3710
9	2599	6.47945	0.91	0.255	0.284	0.316	0.2275	0.39	0.47	0.3370
10	2600	3.84462	2.18	0.290	0.341	0.401	0.2462	0.57	0.80	0.4524
11	2601	2.87744	2.43	0.268	0.337	0.425	0.2247	0.65	1.00	0.4843
12	2602	2.82555	7.10	0.270	0.321	0.383	0.2318	0.64	1.07	0.4801
13	2603	2.60797	7.46	0.262	0.311	0.371	0.2290	0.66	1.16	0.4831

TABLE 25

HTPB MIX DATA

Mix No.	1	2	3	4	5	6	7	8	9	10	11	12	13
%													
0.6 micron	35	35	35	35	52.5	52.5	52.5	52.5	100	23.3	23.3	23.3	23.3
1.0 micron	35	--	--	--	17.5	--	--	--	--	23.3	--	--	23.3
1.9 micron	--	35	--	--	--	17.5	--	--	--	--	23.3	--	--
5.2 micron	--	--	35	--	--	--	17.5	--	--	23.3	23.3	23.3	--
16 micron	--	--	--	35	--	--	--	17.5	--	--	--	23.3	23.3
Aluminum	14	14	14	14	14	14	14	14	14	14	14	14	14
HTPB Binder	16	16	16	16	16	16	16	16	16	16	16	16	16
Viscosity (Kp)	8	5	18	2	4	2	3	8	6	4	4	4	2

Mix No.	14	15	16	17	18	19	20	21	22	23	24	25	26
0.6 micron	35	35	35	35	52.5	52.5	52.5	52.5	100	23.3	23.3	23.3	23.3
1.0 micron	35	--	--	--	17.5	--	--	--	--	23.3	--	--	23.3
1.9 micron	--	35	--	--	--	17.5	--	--	--	--	23.3	--	--
5.2 micron	--	--	35	--	--	--	17.5	--	--	23.3	23.3	23.3	--
16 micron	--	--	--	35	--	--	--	17.5	--	--	--	23.3	23.3
Aluminum	14	14	14	14	14	14	14	14	14	14	14	14	14
HTPB Binder	15	15	15	15	15	15	15	15	15	15	15	15	15
Fe_2O_3	1	1	1	1	1	1	1	1	1	1	1	1	1
Viscosity (Kp) (Kp)	20	10	6	4	44	8	4	4	180	4	18	8	3

TABLE 28

PROPELLANT BURN RATE STRANDS AT 1000 PSIA

	MIX NUMBERS												
Mix No.	1	2	3	4	5	6	7	8	9	10	11	12	13
Standard	1.58	1.65	1.15	0.93	1.50	1.50	1.30	1.09	1.36	1.61	1.36	1.02	1.07
1% Fe_2O_3	1.81	1.74	1.60	1.34	2.02	2.05	1.90	1.60	1.96	1.70	1.61	1.28	1.46

propellants. The degree of dispersion is improved if the mixes exhibit or have been exposed to high viscosity during mixing. At 84% total solids, the desired high viscosity was not achieved. The variance in burn rates probably results from non-reproducible dispersion of the UFAP. However, data are not available to indicate how completely the UFAP was dispersed. It is postulated that lack of complete dispersion of the UFAP is a major obstacle to effective utilization of UFAP.

Attempts at correlating this burn rate data with the reproducibility factor led to some anomalous results. The only factors with which the burn rate could be correlated were the weight mean diameter, the surface mean diameter, and the specific surface area. The correlation of burn rate with weight mean diameter in the uncatalyzed propellants is shown in Figure 53. A similar correlation for 1% iron oxide catalyst propellants is shown in Figure 54. The expected trend of increasing burn rate with decreasing diameter is shown.

A simple correlation of all the data was not possible and if the data were divided into families based upon content of the 0.6 micron fraction, then reasonable correlation could be obtained. The lack of parallelism in the family data for catalyzed propellant indicates poor correlation with WMD. The fact that the burn rate tends to decrease at small values of WMD is projected to result from a lack of dispersion of the small AP.

Figures 55 and 56 show the correlation of burn rate with surface mean diameter in both the catalyzed and uncatalyzed systems. The general shapes of the curves are similar but the direction in surface mean diameter one must take to obtain higher burn rates is not evident.

The correlation of surface area with burn rate is shown in Figures 57 and 58. Quite good correlation is obtained within the individual bimodal and trimodal families. A surprising result from Figure 57 is that as the surface area increases, the burn rate increases and then decreases. Such is not apparent in the catalyzed propellant (Figure 58). Either better dispersion has been obtained in the catalyzed propellant or else the presence of catalyst compensates for the lack of dispersion. A mechanism for the latter is unclear.

In order to determine the burn rate reproducibility of propellants containing UFAP, a series of 84% total solids (non-catalyzed) propellants were manufactured. The oxidizer blend (68%) was made up of 0.65 micron

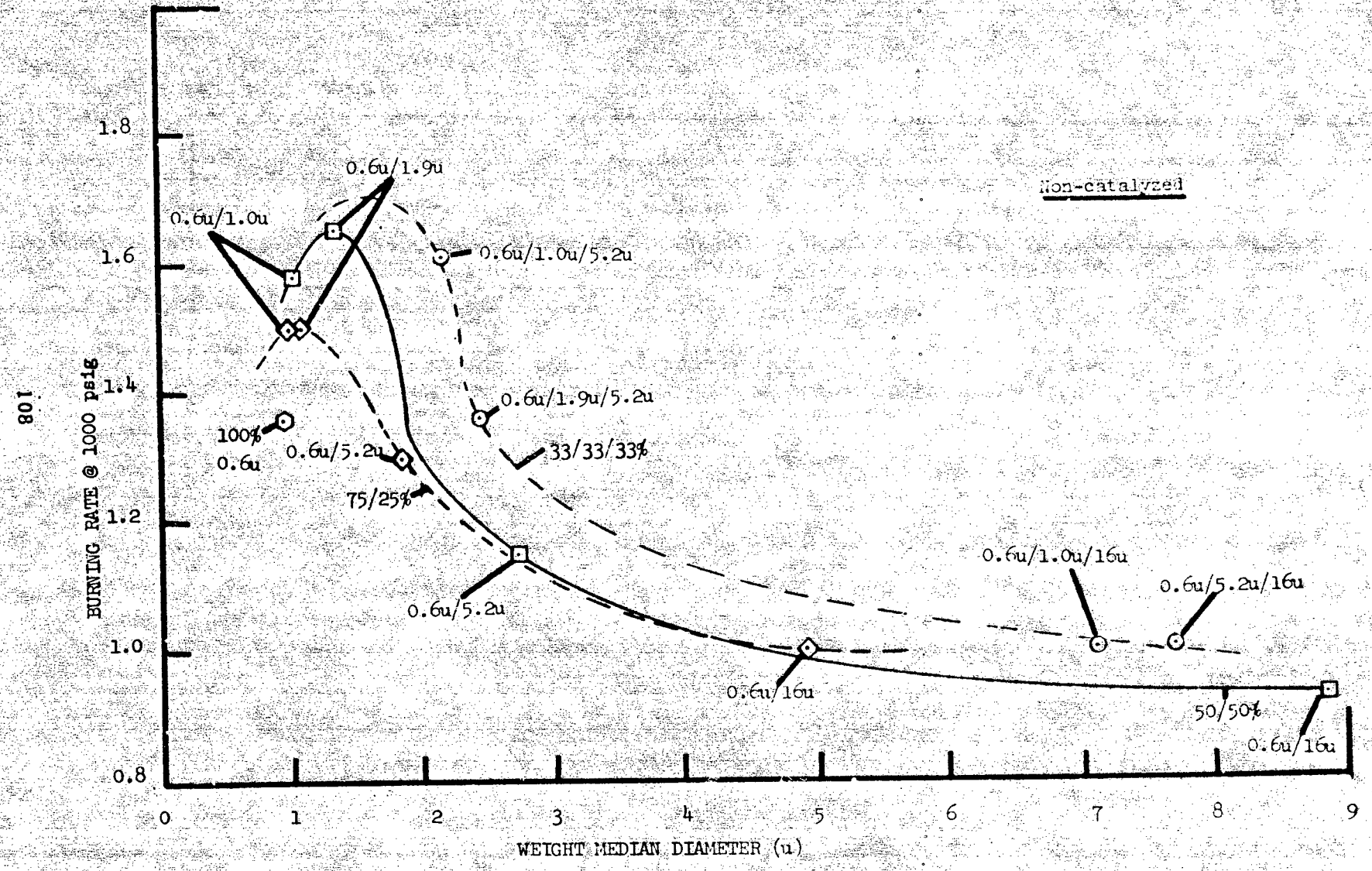


Figure 53. Correlation of Burn Rate with Weight Mean Diameter (Uncatalyzed)

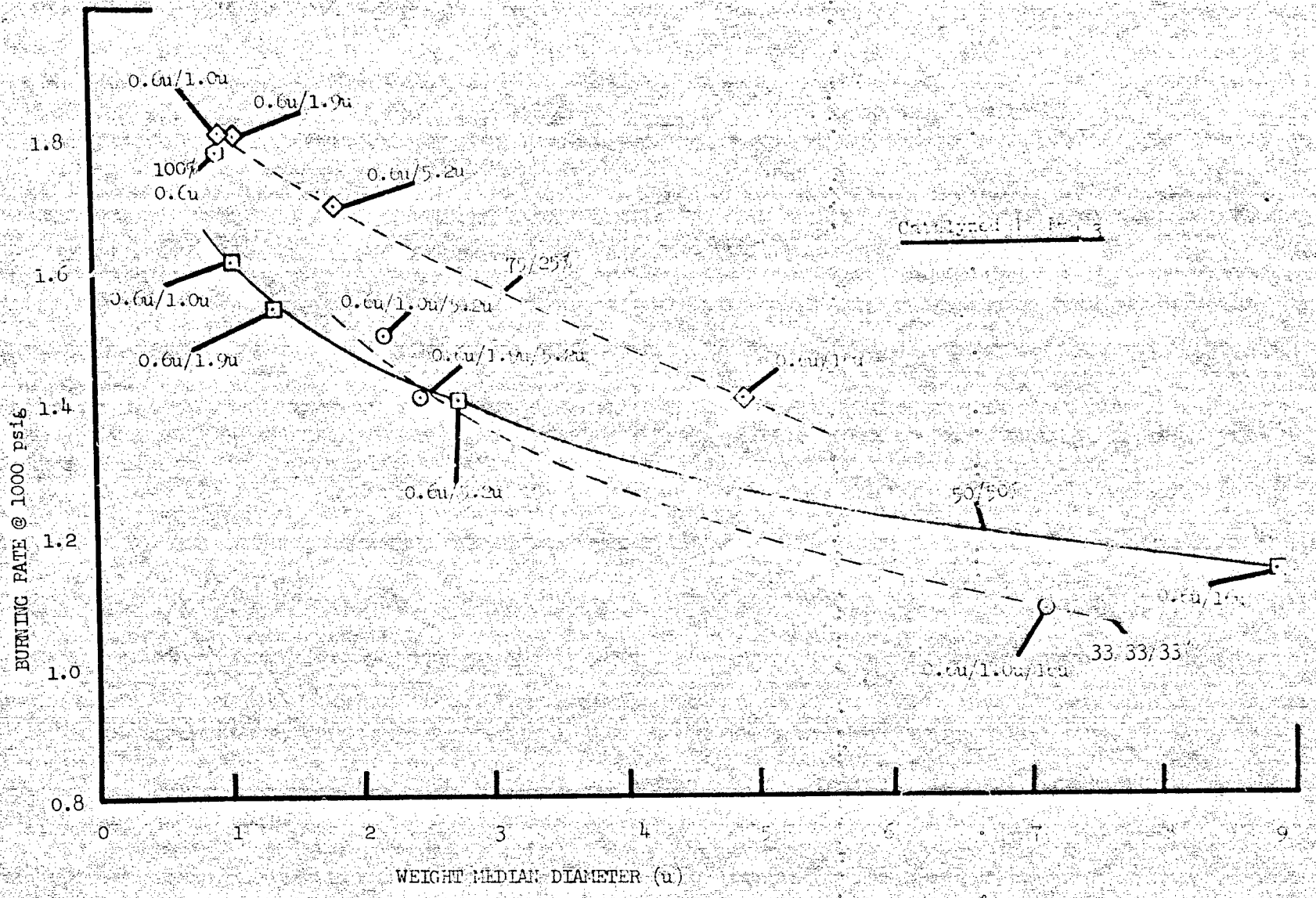


Figure 54. Correlation of Burn Rate with Weight Mean Diameter ($1\%\ Fe_2O_3$)

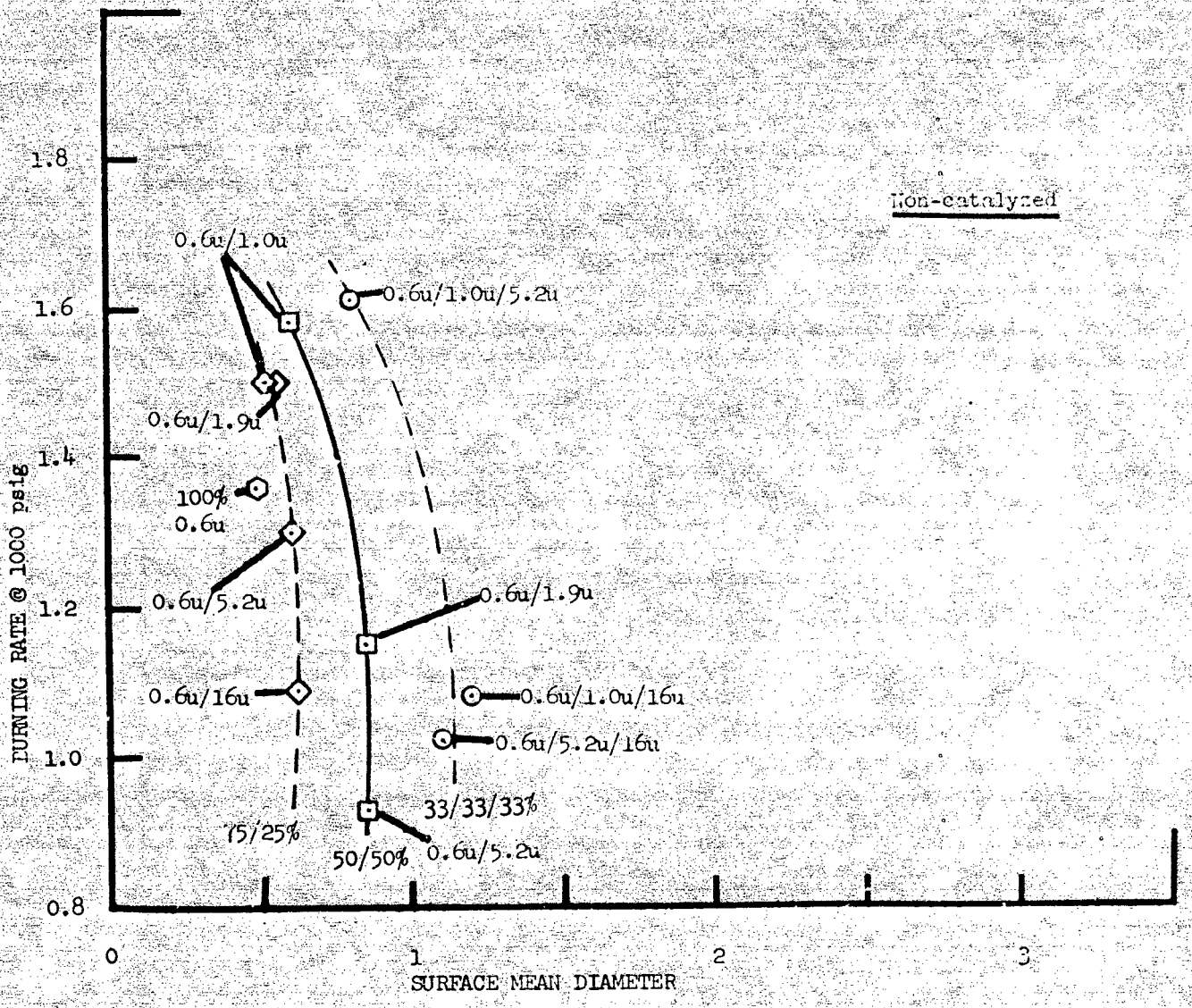


Figure 55. Correlation of Burn Rate with Surface Mean Diameter (Uncatalyzed)

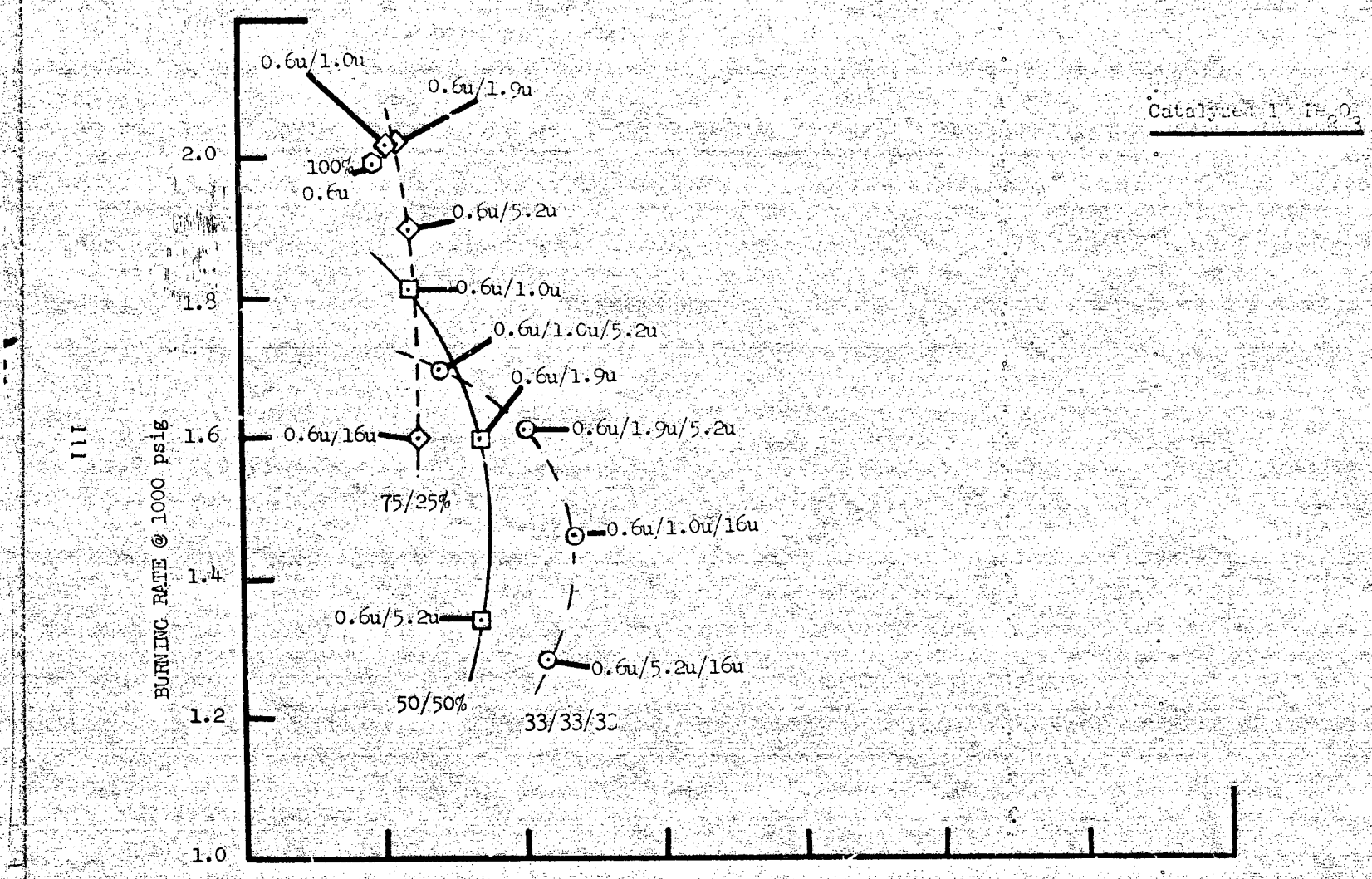


Figure 56. Correlation of Burn Rate with Surface Mean Diameter ($1\%\ Fe_2O_3$)

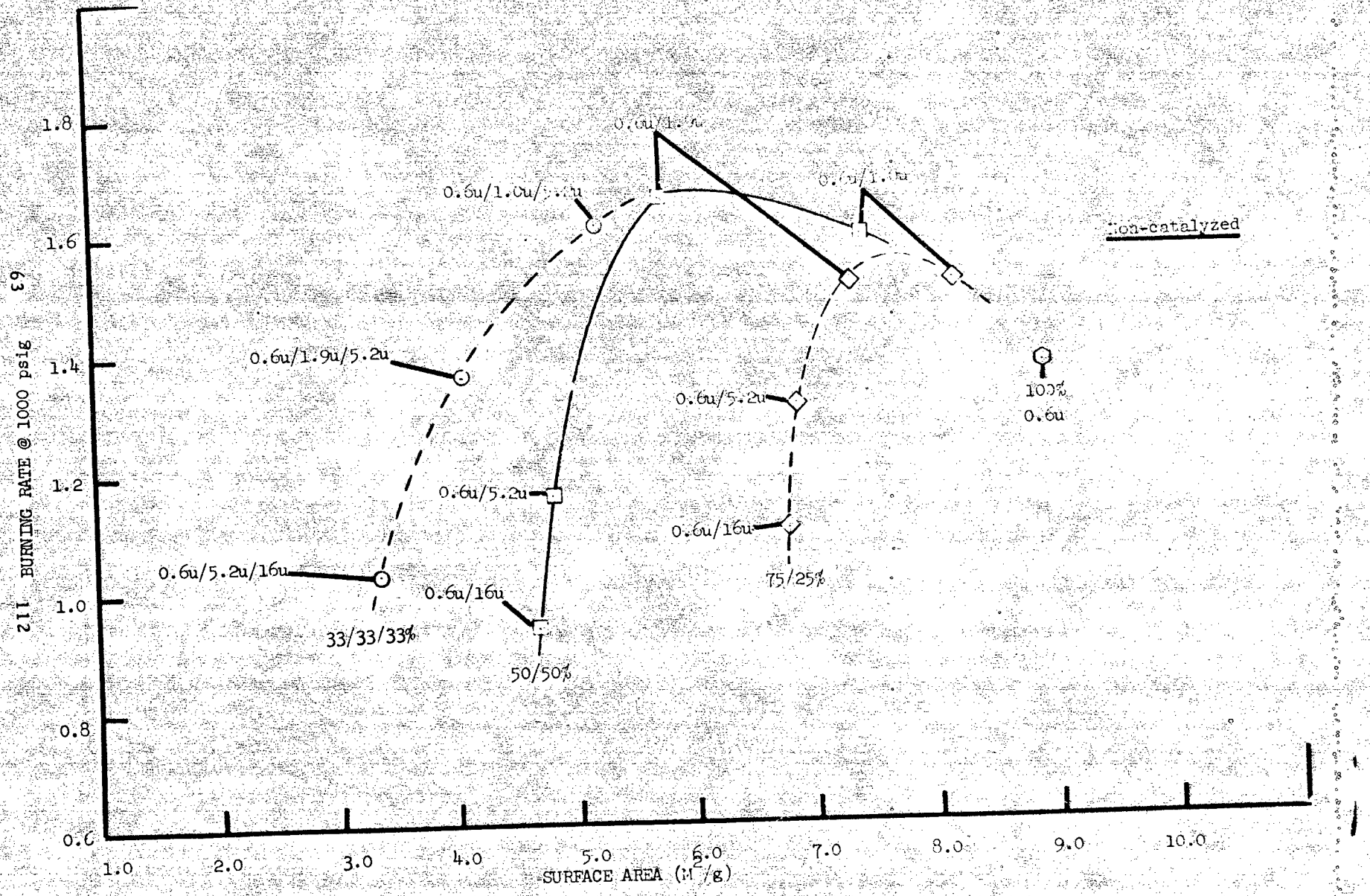


Figure 57. Correlation of Burn Rate with Surface Area
(Uncatalyzed)

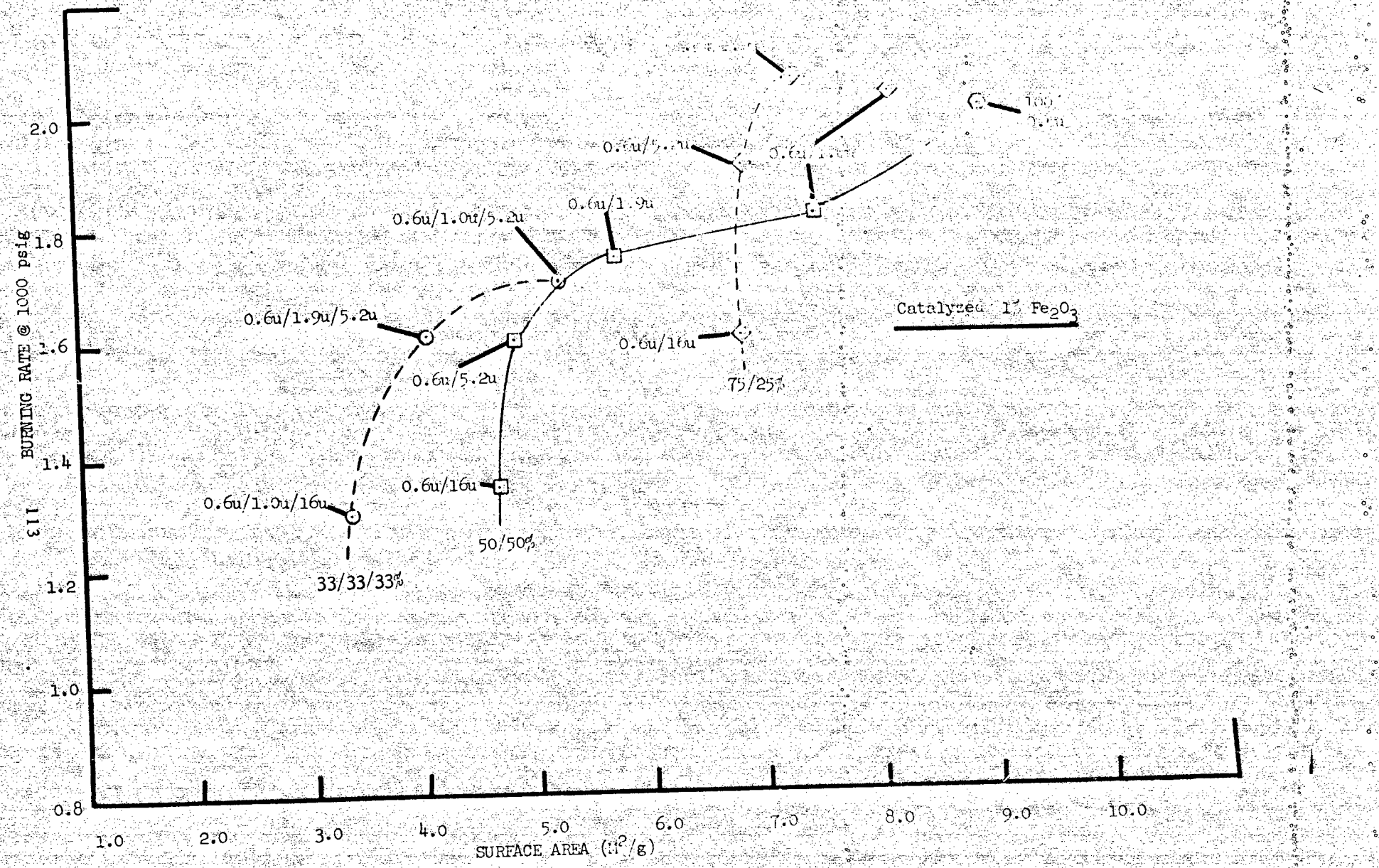


Figure 58. Correlation of Burn Rate with Surface Area ($1\%\ Fe_2O_3$)

SWECO milled oxidizer and a fluid energy milled fraction exhibiting a size of 2.1 micron. In order to test the "within lot" reproducibility, three mixes were manufactured with lot VMA-95 UFAP. These mixes were made one week apart and the strand burning rates shown in Table 29 illustrates good reproducibility.

In order to determine the reproducibility from one UFAP lot to another, two lots of SWECO oxidizer were utilized to manufacture the 84% total solids propellant. There was a slight variance (within experimental error) in the particle size for those two lots. The burn rates, as shown in Table 29, were quite reproducible.

Similar formulations were manufactured with SWECO milled oxidizer and emulsion freeze-dried oxidizer to determine if burn rate differences existed between two different types of UFAP. These mixes are shown in Table 30. Although the total solids in each mix are the same, the mixes made with SWECO oxidizer contained more AP due to the difficulty of processing (dispersing) the freeze-dried materials. Differences in end-of-mix viscosity and burn rates were demonstrated. The burn rate differences can probably be ascribed to the lower oxidizer concentration. The viscosity differences can probably be ascribed to difficulty in dispersing the freeze-dried UFAP or a difference in the ease of wetting of the freeze-dried AP by the polymer.

Studies were conducted to determine if the humidity under which the UFAP is stored has an effect on propellant processing and burn rate. Four blends (two bimodal and two trimodal) were selected for manufacture. Mixes were manufactured with each of these blends following oxidizer preparation, following exposure to 10% humidity for one week and following exposure to 50% RH for one week. In each case, slight decreases in burn rate were apparent with exposure to increasing relative humidity (Table 31). Of even greater significance is the increase in end-of-mix viscosity that occurs when the oxidizer is exposed to the relatively high humidity.

TABLE 29

BURN RATE REPRODUCIBILITY

SWECO Oxidizer

84% Total Solids (non-catalyzed)

AP (UFAP)	50
AP (FEM-2.1 micron)	18
Al (25 micron)	16

Within Lot Reproducibility

Lot VMA95 (0.65 micron)

	R_b @ 1000 psi (in/sec)
Mix 1	1.43
Mix 2	1.46
Mix 3	1.44

Lot to Lot Reproducibility

Lot VMA95 (0.65 micron)	Lot VMA96 (.63 micron)
R_b @ 1000 psi (in/sec)	
1.43	1.47

TABLE 30

COMPARISON FORMULATIONS

	SWECO	Freeze Dried
R-45M/AO-2246/IPDI	13.5	13.7
DOA	1.0	1.0
Lecithin	0.2	---
Fe_2O_3	1.0	1.0
Aluminum (25 micron)	16.0	18.0
AP (FAP)	50.0	50.0
AP (FEM) (1.78 micron)	18.0	16.0
FAP Coating Agent	HX-868	Nopcogen 16/DOA 0.7 2.1
Coating Agent Concentration	0.4	2.8
Particle Size	0.55	0.8
End of Mix Viscosity, Kp	5	32
Burn Rate at 1000 psi (in/sec)	1.83	1.67
n	.72	.63

TABLE 31

EFFECT OF HUMIDITY ON UFAP BALLISTICS

Mix No.	2595	2592	2601	2633
HTPB Binder	16	16	16	16
Aluminum	14	14	14	14
AP 0.6 micron	52.5	35	23.3	23.3
AP 1.1 micron	17.5	---	---	---
AP 1.9 micron	---	35	23.3	23.3
AP 5.2 micron	---	---	23.3	---
AP 4.8 micron Coated	---	---	---	23.3
Standard Burn Rate at 1000 psi/Viscosity*	1.50/4	1.65/6	1.35/4	1.40/6
10% RH - Burn Rate at 1000 psi/Viscosity *	1.36/10	1.46/80	1.34/42	1.38/20
50% RH - Burn Rate at 1000 psi/Viscosity *	1.36/14	1.36/22	1.33/200	1.28/120

* (in/sec)/Kp

TASK VII - UFAP PROCESSING

The objective of this effort is to define, through utilization of viscosity response plots, those formulation regimes (oxidizer sizes and distributions) which exhibit lowest viscosity at maximum burn rate. In order to encompass a wide range of oxidizer distributions, five different oxidizer sizes were selected for this study: 0.6, 1.0, 2.5-3.0, 6.0, and 16.0 microns. Only the 0.6 micron and the 1.0 micron SWECO-ground oxidizers contain coating agent (HX-868). From these five oxidizer sizes, two trimodal blends and three bimodal blends were selected for evaluation. These blends are as follows:

a. 0.6, 1.9, 16
b. 0.6, 1.9
c. 0.6, 4.8
d. 0.6, 1.0, 4.8
e. 1.0-1.9

Mixes were manufactured at seven different composition points in the standard viscosity response configuration and lines of constant viscosity were computed.[1] A low total solids propellant was selected since, for the purposes of preparing viscosity response plots, it is necessary that each of the seven mixes exhibit a moderate viscosity. This can only be accomplished with a low total solids propellant. In order to optimize particle size distribution of the UFAP fractions, viscosity response plots were prepared for five different oxidizer size blends. Three bimodal and two trimodal distributions were initially evaluated with ultrafine oxidizer and fluid energy milled oxidizer as normally prepared in the plant. Seven one-pint mixes were manufactured for each of the bimodal and trimodal blends. For the trimodal blends, compositions for mix manufacture were selected at the triangular matrix points prescribed for viscosity response plots. In bimodal blends, aluminum powder concentration would normally be the third variable. However, the aluminum concentration was maintained constant and, threfore, seven mixes were manufactured at composition points along a constant aluminum concentration line. The composition used for all mixes manufactured was 70% ammonium perchlorate, 10% aluminum powder (25 micron), and 20% HTPB binder. The oxidizer distributions for each of the 35 propellant mixes as well as the end-of-mix viscosities for each mix are shown in Table 32. The five oxidizer blends are listed as Blends A, B, C, D and E. A triangular representation of the composition points as well as the end-of-mix viscosities for each of these blends are shown on Figures 59 through 63. Blend A, a mixture of 0.6, 1.9, and 16 micron gives a variety of viscosities ranging from 1.5 to 160 Kp. Blends B and C are bimodal in nature and the viscosities were measured at composition points along a constant aluminum binder concentration line. Blend D, a

1. The surface response technique is described in Appendix E.

TABLE 32

COMPOSITION AND VISCOSITY OF BIMODAL AND TRIMODAL AP BLENDS

Oxidizer Lot No.	ULM-75	UMA 95	EFEM42	4102	3915	3941	R45M		30 min. EOM Visc.
Particle Size	1 μ	.6 μ	1.9 μ	4.8 μ	16 μ	Al 25 μ	Binder	Graph	Kp/° F
Mix No.									
2481	-	70	-	-	-	10	20		2
2482	-	-	70	-	-	10	20		160
2483	-	-	-	-	70	10	20	A	6
2484	-	35	35	-	-	10	20		2
2485	-	35	-	-	35	10	20		3
2486	-	23.3	23.3	-	23.3	10	20		1.5
2487	-	-	35	-	35	10	20		1
R2481	-	70	-	-	-	10	20		2
2489	-	58.3	11.7	-	-	10	20		4
2490	-	46.7	23.4	-	-	10	20	B	2
R2484	-	35	35	-	-	10	20		2
2492	-	23.3	46.7	-	-	10	20		4
2493	-	11.7	58.3	-	-	10	20		4
R2482	-	-	70.0	-	-	10	20		160
R2491	-	70	-	0	-	10	20		2
2495	-	58.3	-	11.7	-	10	20		2
2496	-	46.7	-	23.4	-	10	20	C	4
2497	-	35.0	-	35	-	10	20		4
2498	-	23.3	-	46.7	-	10	20		8
2499	-	11.7	-	58.3	-	10	20		26
2500	-	0	-	70	-	10	20		57
R2481	-	70	-	-	-	10	20		2
2501	70	-	-	-	-	10	20		6
R2500	-	-	-	70	-	10	20	D	57
2503	35	35	-	-	-	10	20		6
2504	35	-	-	35	-	10	20		4
R2497	-	35	-	35	-	10	20		4
2505	23.3	23.3	-	23.3	-	10	20		4
R2501	70	-	0	-	-	10	20		6
2506	58.3	-	11.7	-	-	10	20		6
2507	46.7	-	23.3	-	-	10	20	E	8
2510	35.0	-	35.0	-	-	10	20		10
2508	23.3	-	46.7	-	-	10	20		6
2509	11.7	-	58.3	-	-	10	20		12
R2482	0	-	70	-	-	10	20		160

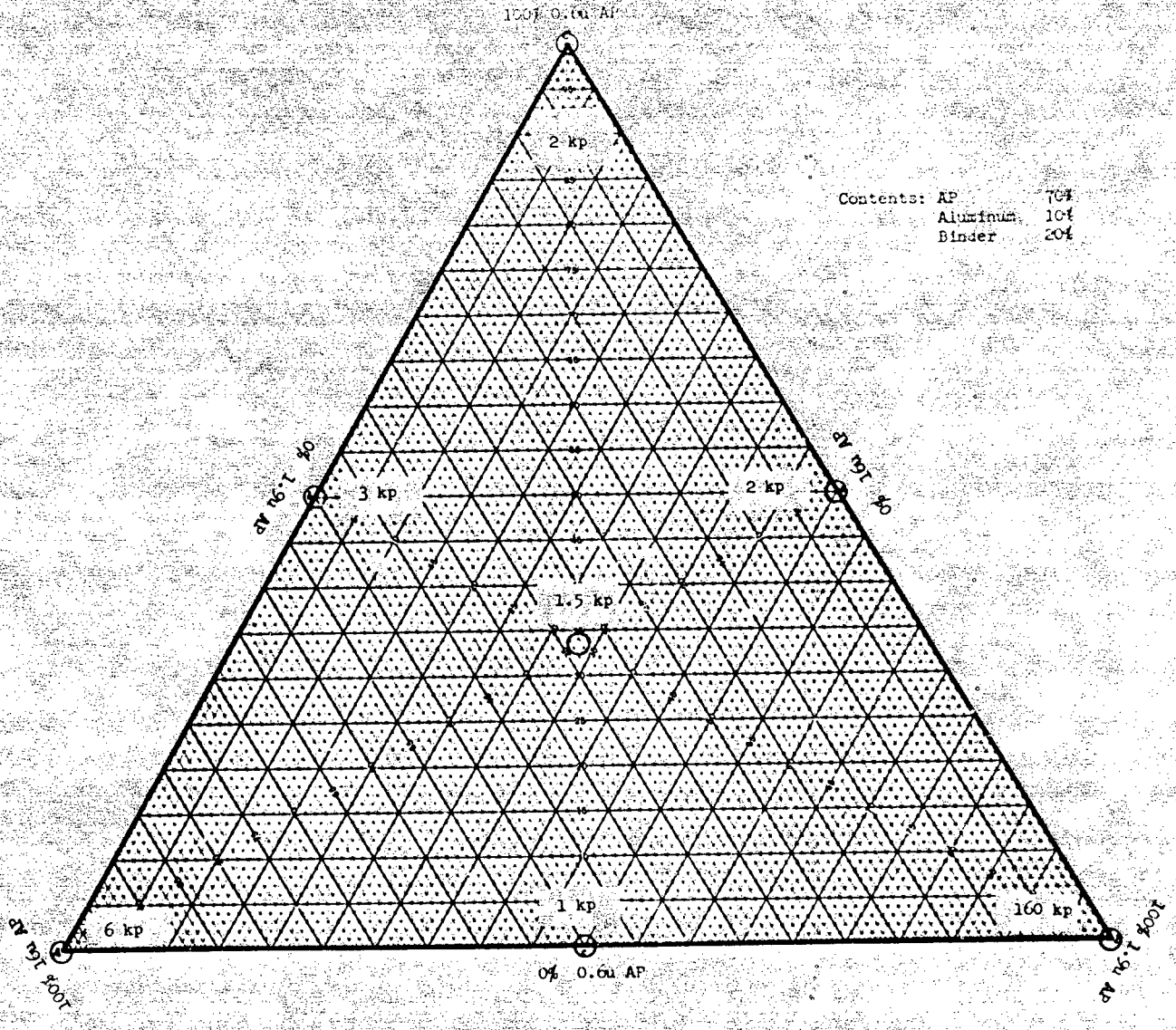


Figure 59. Viscosities of Blend "A" (0.6, 1.9, and 16 Micron) AP in 84% Total Solids Propellant

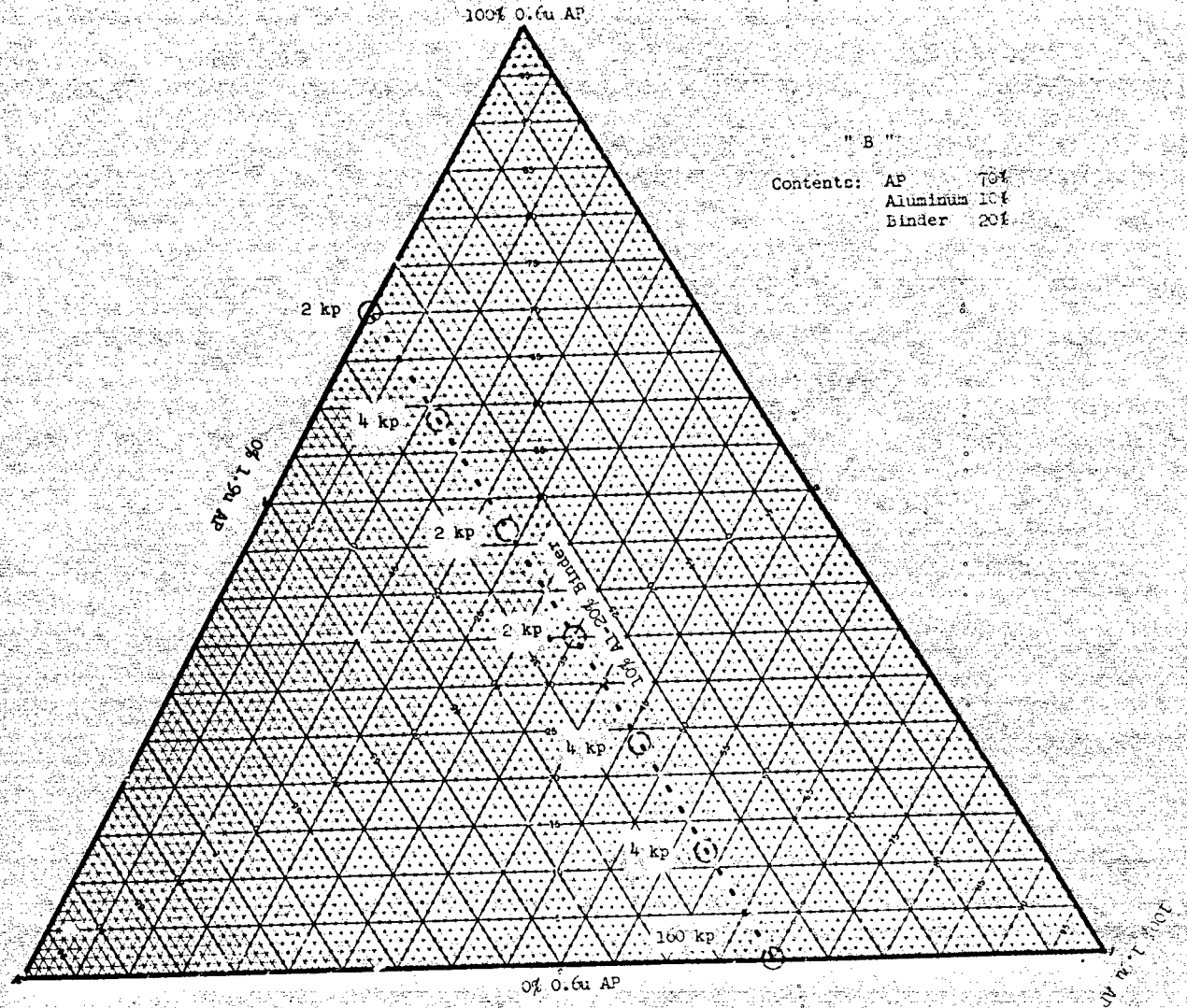


Figure 60. Viscosities of Blend "B" (0.6 and 0.9μ AP)

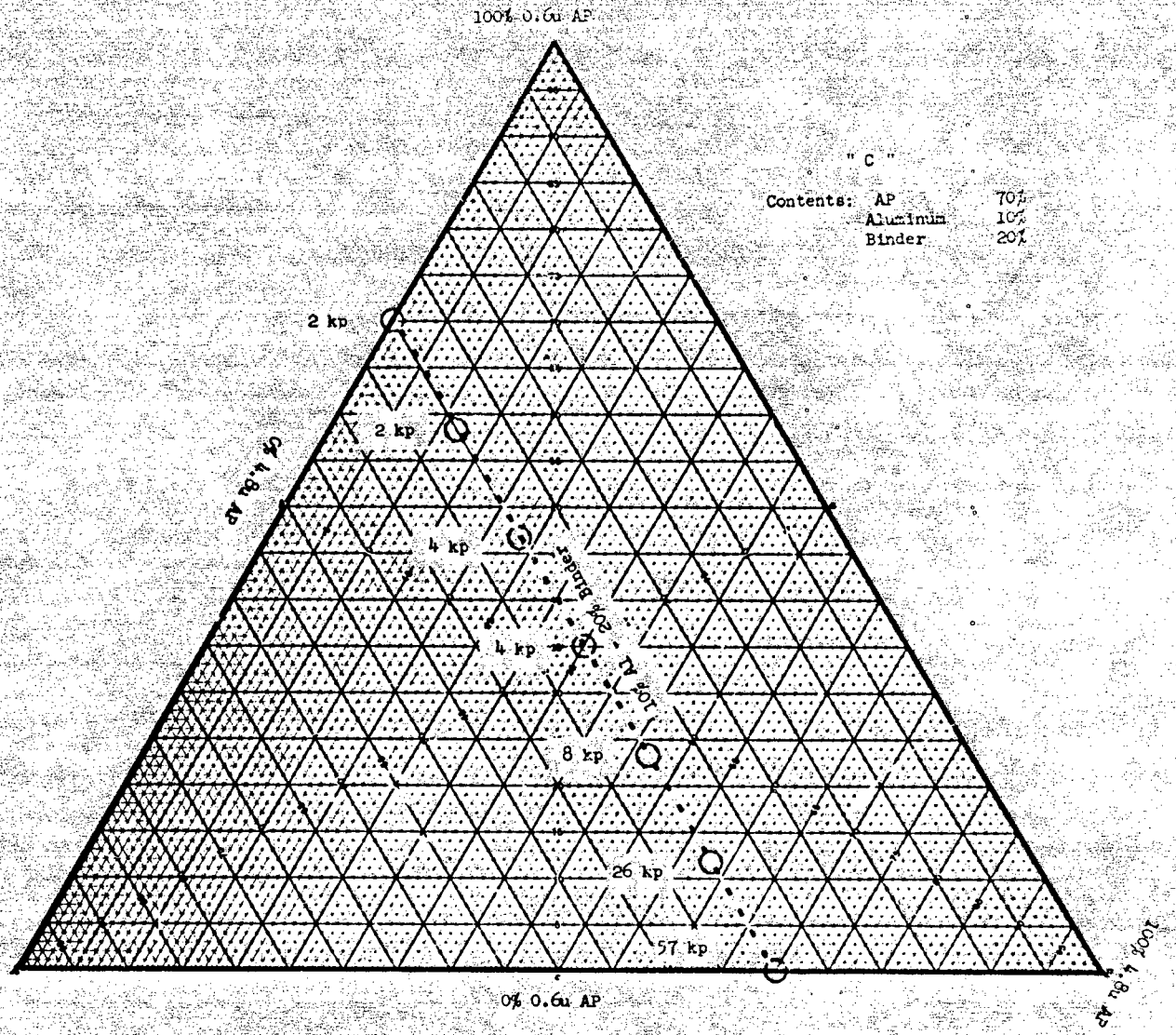


Figure 61. Viscosities of Blend "C" (0.6 and 4.8μ AP)

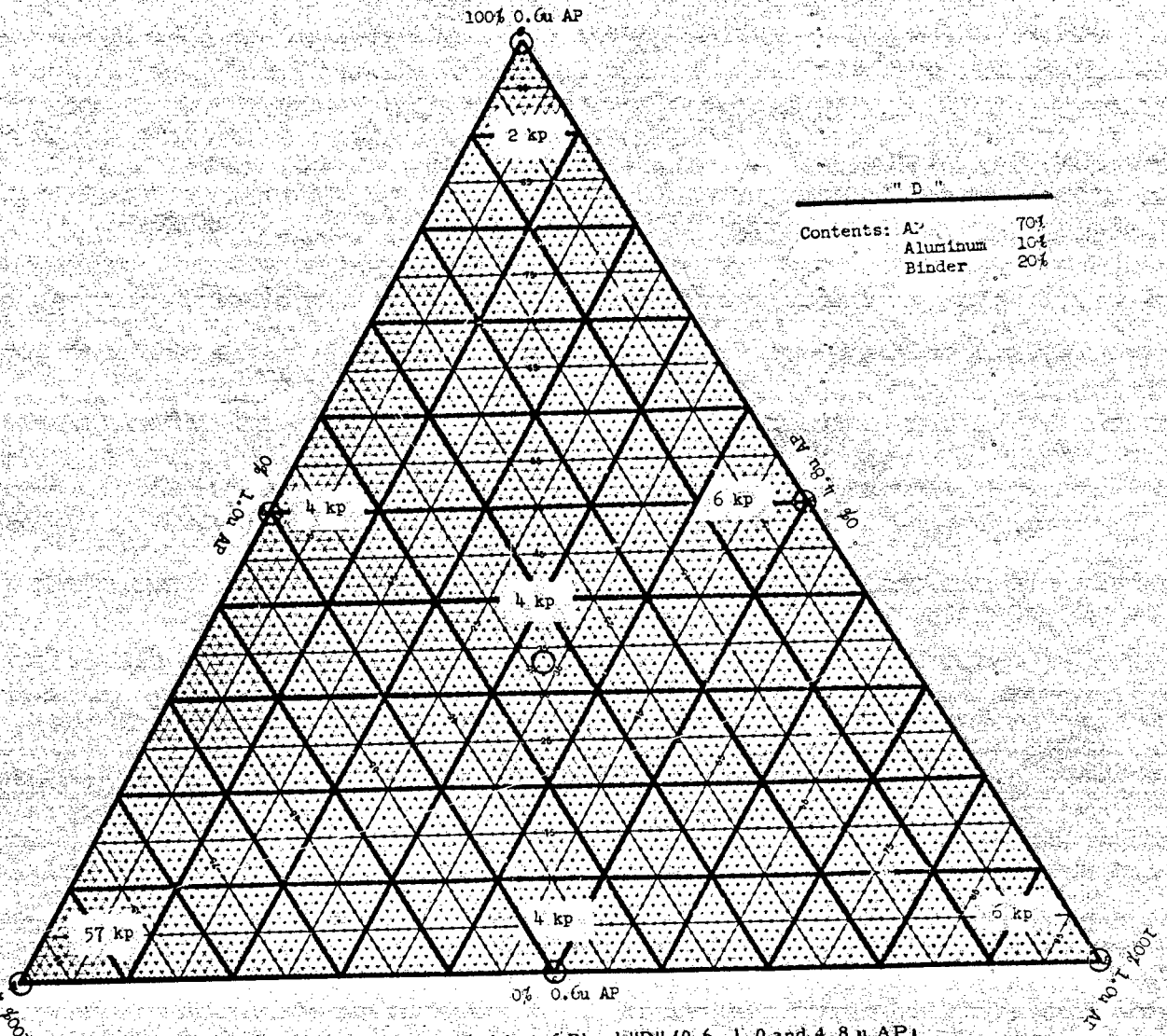


Figure 62. Viscosities of Blend "D" (0.6, 1.0 and 4.8 μ AP)

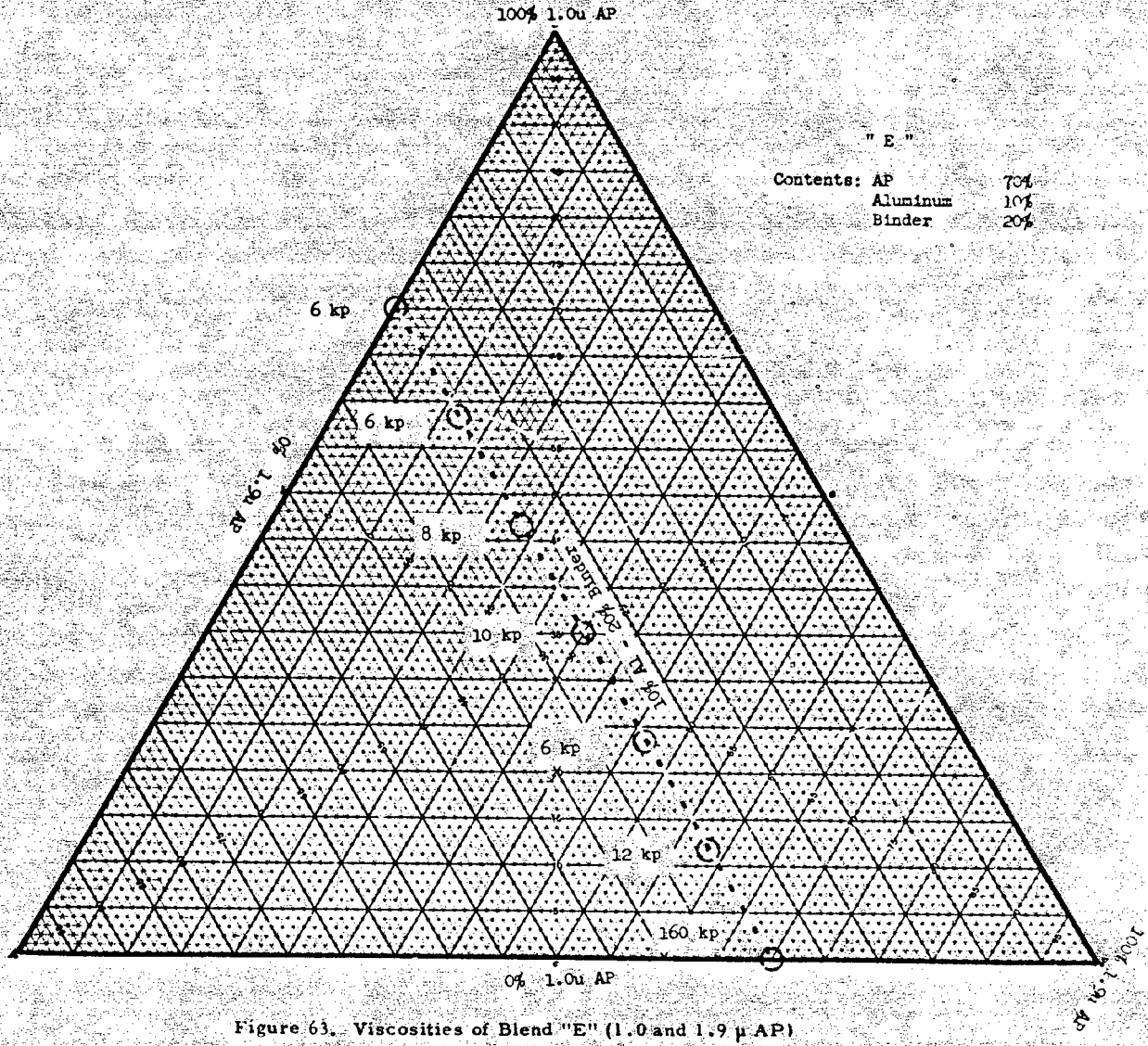


Figure 63. Viscosities of Blend "E" (1.0 and 1.9 μ AP)

trimodal of 0.6, 1.0 and 4.8 ammonium perchlorate had a moderately wide range of viscosities. Blend E is a bimodal of 1.0 and 1.9 micron. End-of-mix viscosities resulting from seven mixes manufactured along a constant aluminum-binder concentration line are shown on Figure 63.

Viscosity-response plots were prepared by the computer plotter for Blends A and D, the two trimodal blends. The viscosity response plot for Blend A is shown on Figure 64. These data show that a wide area of low viscosity formulations is available. Apparently, there is too little difference in the viscosities measured as the seven prescribed composition points to permit a more meaningful plot to be prepared. The viscosity response for Blend D, the trimodal of 0.6, 1.0, and 4.8 micron, is shown on Figure 65. These data show that the normally expected regime of formulation interest (approximately one-third of each size fraction) appears in this case to be a composition of moderately high viscosity and two areas of formulation interest which give very low viscosities do exist. These areas are compositions containing high concentrations of 0.6 micron ammonium perchlorate and compositions containing high concentrations of 1.0 micron ammonium perchlorate. Compositions containing high concentrations of the largest size fraction (4.8 micron) result in quite high propellant viscosities. It is speculated that this can be attributed to the fact that there is no coating agent on the 4.8 micron material whereas both the 0.6 micron and the 1.0 micron oxidizer contain an aziridine coating agent which, apparently, promotes wetting by the binder.

As described earlier, Blend A, a trimodal of 0.6 micron, 1.9 micron, and 16 micron AP, exhibited viscosities in an 80% total solids propellant that were too low for meaningful interpretation of the viscosity response plot. In order to better define the areas of minimum viscosity, the seven mixes required for a viscosity response plot were manufactured in an 84% total solids propellant which contained 74% AP and 10% aluminum in HTPB. Viscosities for this formulation are shown on Figure 66 and the viscosity response plot exhibited on Figure 67. The overall shape of the response curves is similar to that noted with the 80% total solids but better defined. The formulation areas for minimum viscosity are readily apparent.

By increasing the total solids to 86% (74% AP and 12% aluminum), a distinct difference in the viscosity response plots was obtained (Figure 68). The viscosities for the required seven mixes are also shown on Figure 68. The area of minimum viscosity would be a formulation containing high concentrations of the 0.6 micron (HX-868 coated) AP and low concentrations of the two uncoated oxidizer fractions. The difference in the shape of the curve can be attributed to changing the aluminum concentration but a more likely explanation is the fact that at high total solids, the uncoated fractions are difficult to disperse and the HX-868 coated SWECO ground fraction disperses extremely well.

RUN 41

VISCOSITY RESPONSE PLOT

X4=30 AL-B

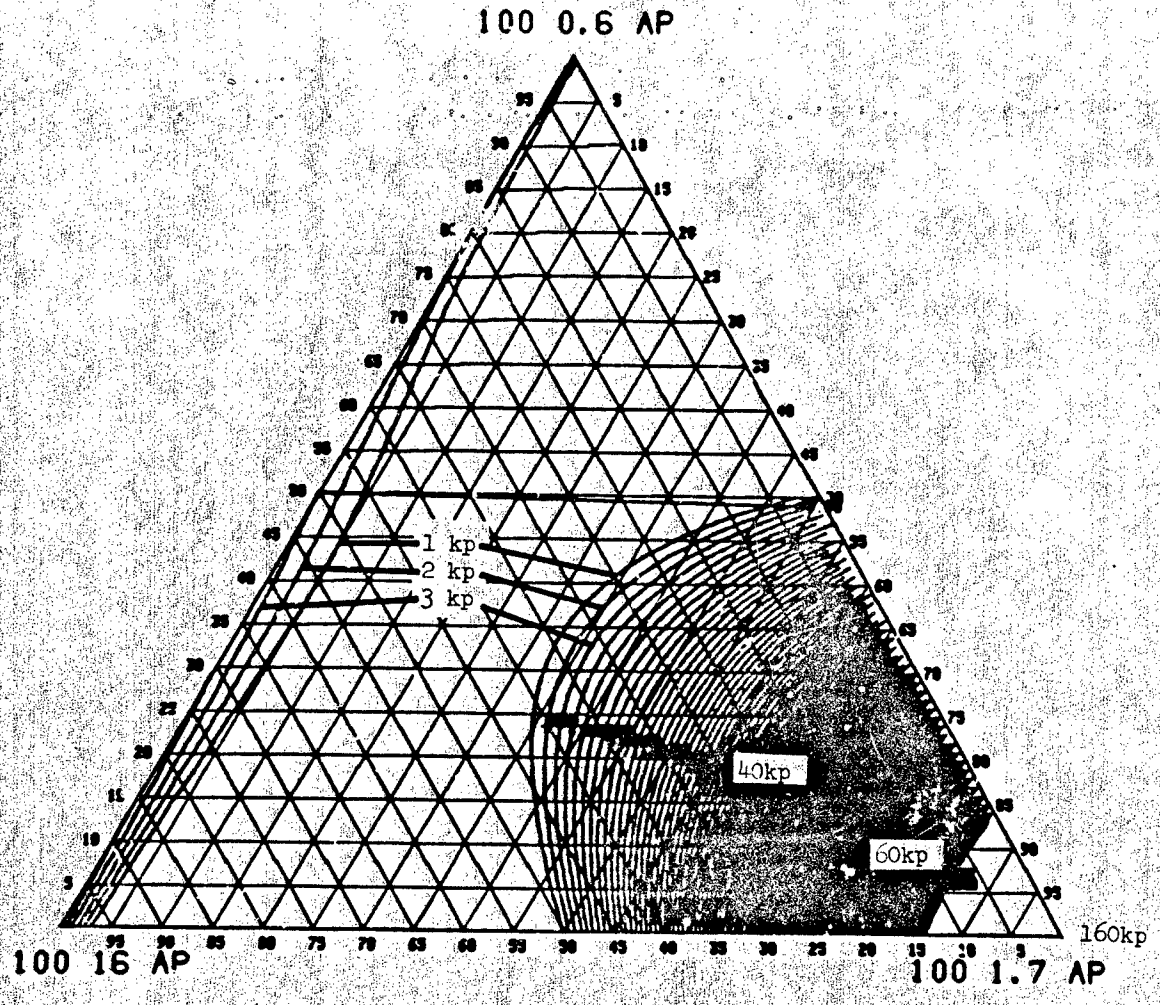


Figure 64. Viscosity Response Plot for Blend "A" (0.6, 1.7, and 16 μ AP)

RUN42 VISCOSITY RESPONSE PLOT

X4= 30 AL-B

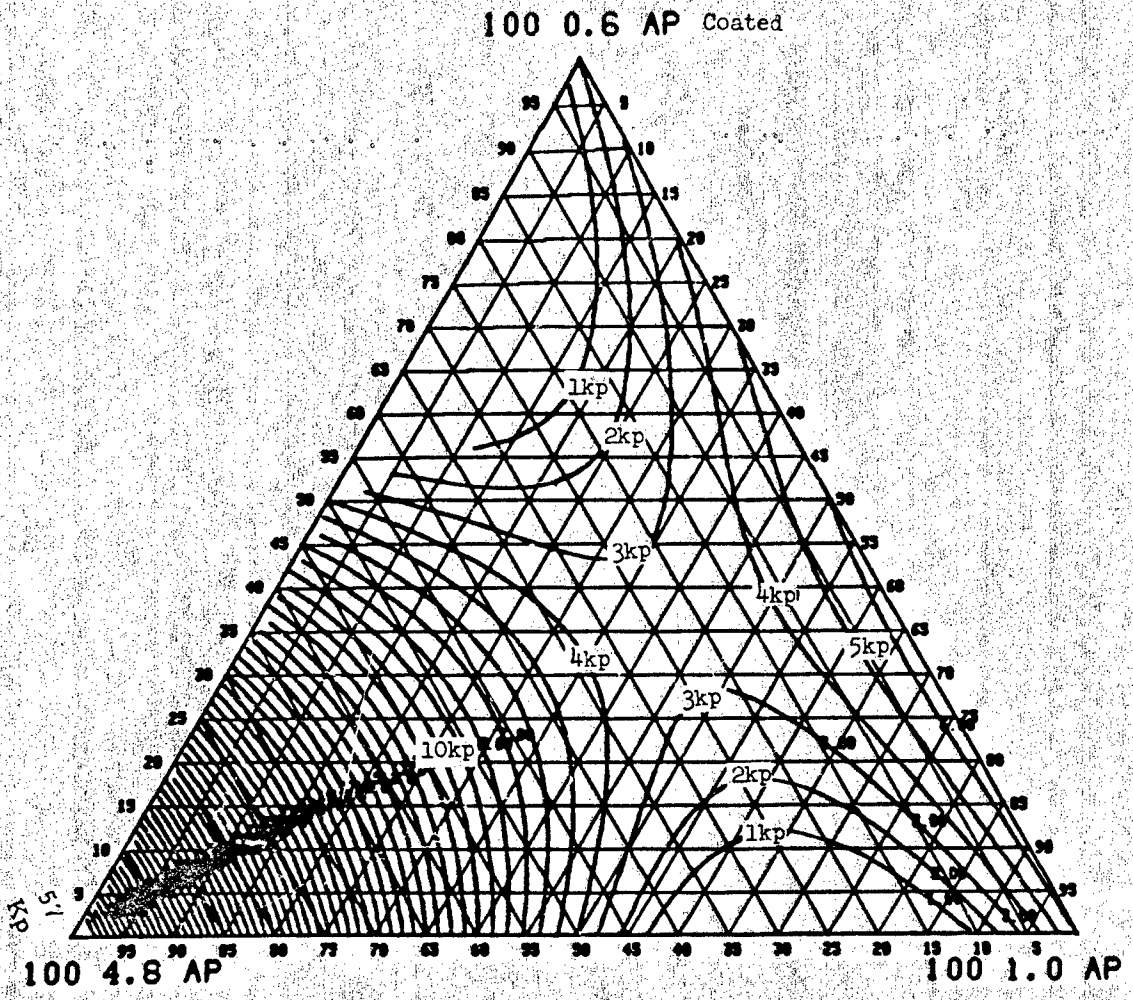


Figure 65. Viscosity Response Plot for Blend "D"
(0.6, 1.0, 4.8 μ AP)

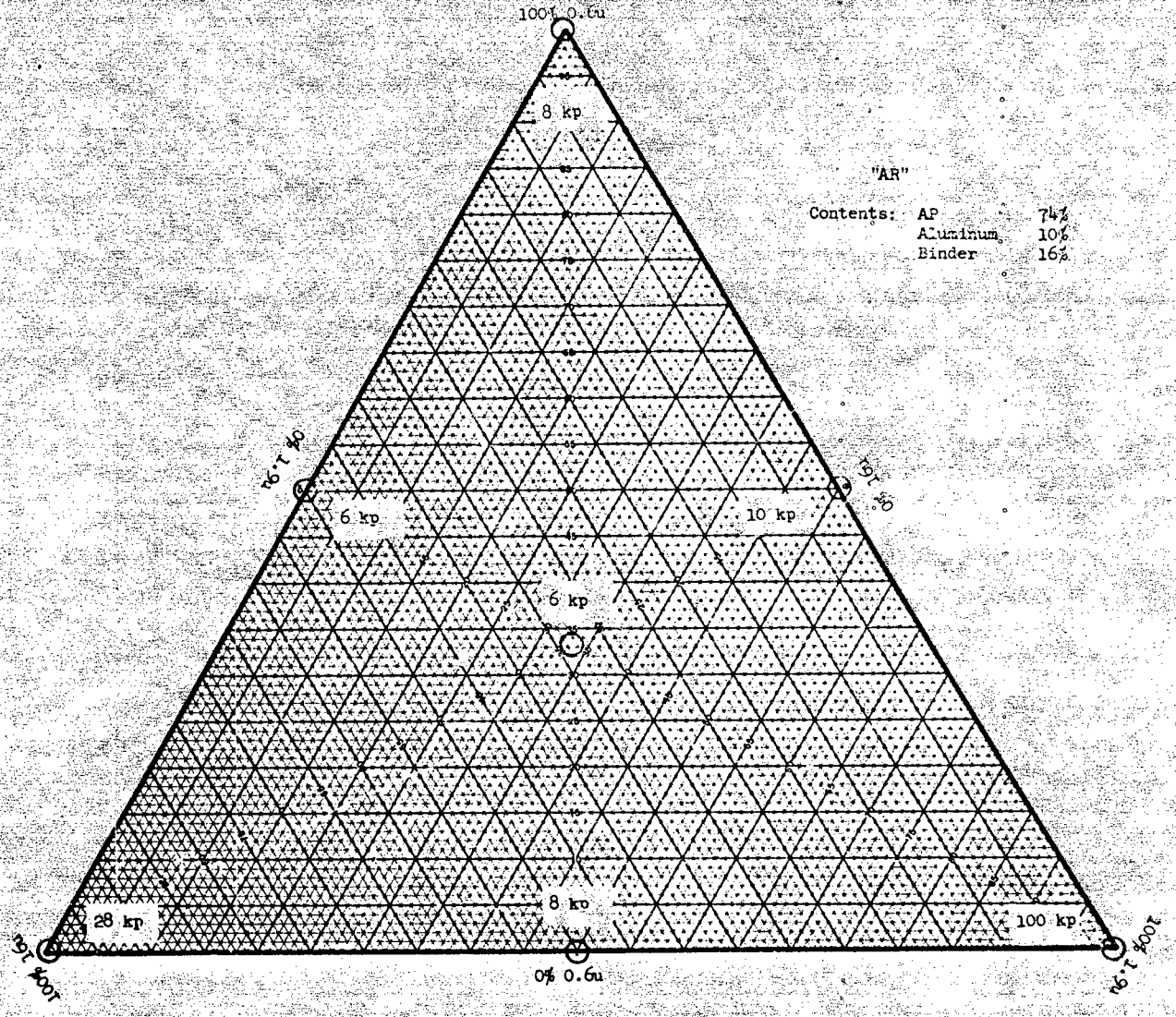


Figure 66. Viscosities of Blend "A" (0.6, 1.9 and 16 Micron) AP in 80% Total Solids Propellant.

VISCOSITY RESPONSE PLOT 74/10/16 AR

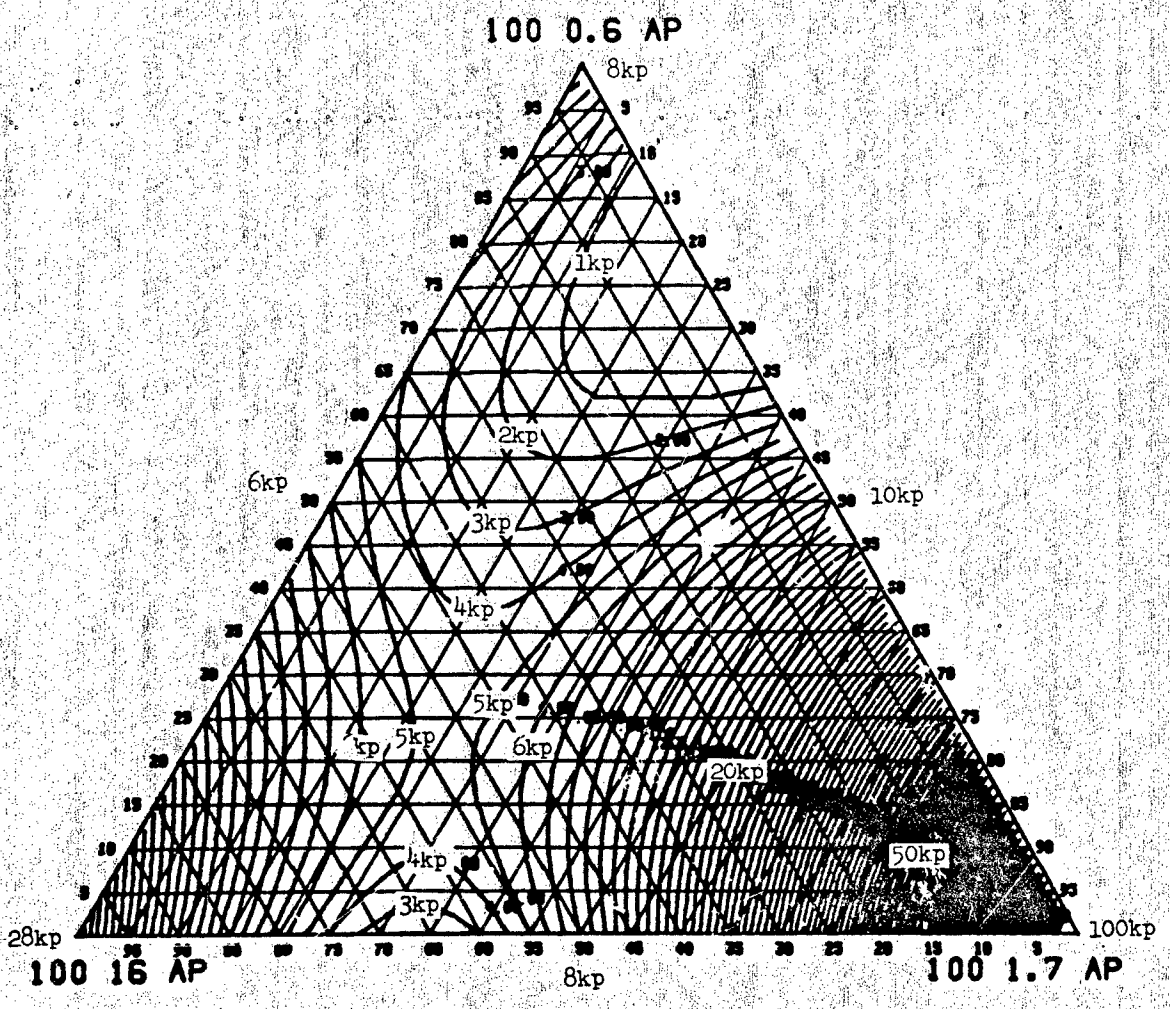


Figure 67. Viscosity Response Plot for 84% Total Solids Blend "A"

VISCOSITY RESPONSE PLOT 86% TOTAL SOLID

X4=26ALB

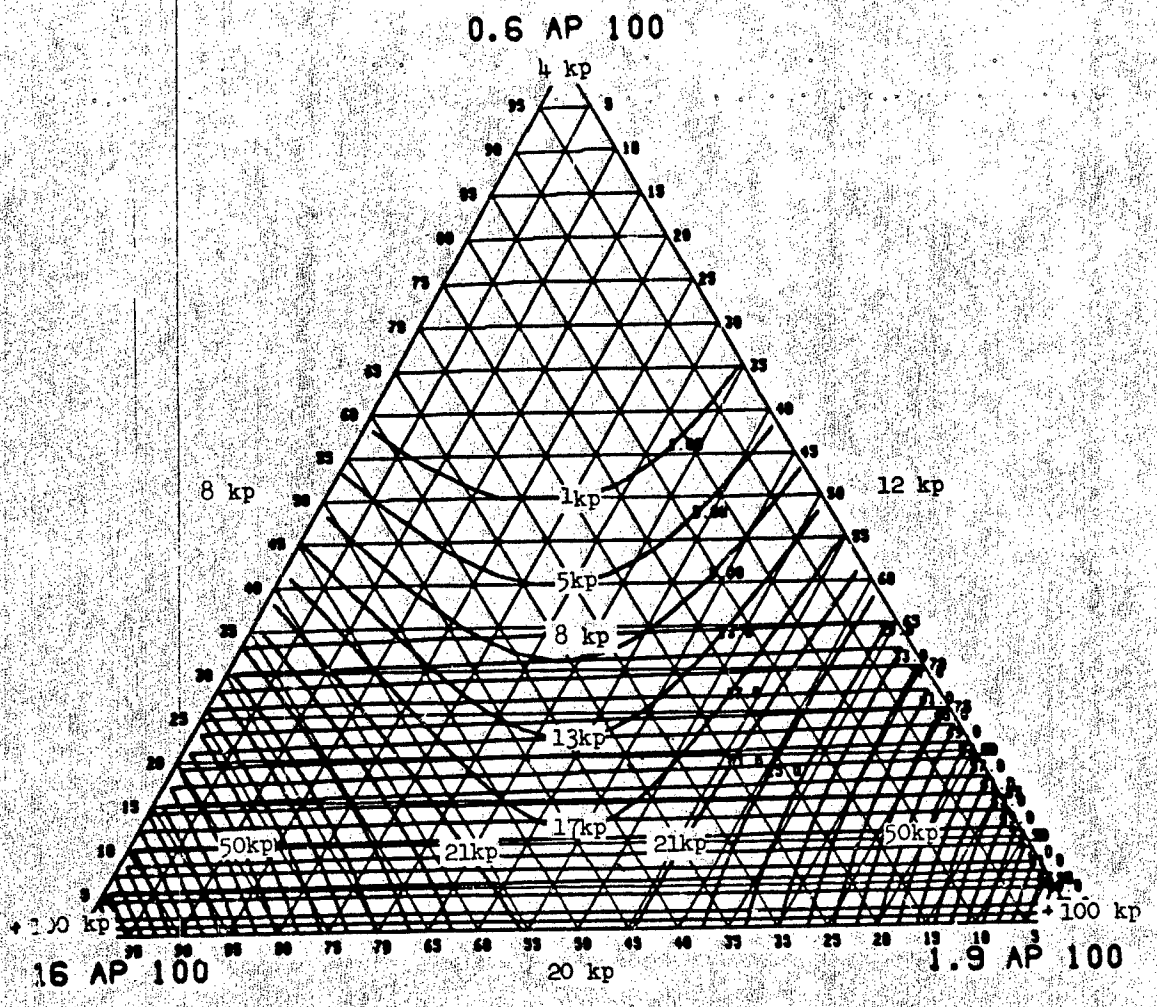


Figure 68. Viscosity Response Plot for 86% Total Solids Blend "A"

A similar test was performed on Blend "D" by increasing the total solids to 81%. The resulting viscosity response plot is shown in Figure 69. The plot now only indicates one area of low formulation viscosity and that point is high concentration of the 0.6 micron AP.

In order to obtain an assessment of the effect of a coating agent on processibility and dispersibility of UFAP the modified (84% total solids) Blend "A" matrix was reexamined by making a direct substitution of coated 1.7 micron AP (0.4% HX-868) for the uncoated fraction. The resulting viscosity response plot is shown in Figure 70. Note that the coating process (coated with HX-868 in methylene chloride) resulted in a slightly increased particle size. By comparing Figures 67 and 70, one observes no meaningful difference between the plots. Essentially the coating exhibited only a slight effect upon the viscosity. Several different techniques were used to coat the 1.7 micron AP but in each case, no apparent decrease in viscosity occurred. Even though the product in each case exhibited a low measured particle size, the physical appearance indicated large agglomerates which are probably not completely dispersed in the mixing process.

A more meaningful examination of the effect of coating agent was made by substituting SWECO vibro energy milled 4.8 micron AP for the uncoated 4.8 micron material in the 80% total solids blend "D". The sample was coated with 0.4% HX-868 in the grinding process. Examination of Figure 71 and comparing it with Figure 65 indicates that the only major change indicated is a slight lowering of the viscosity of those blends containing high concentrations of 4.8 micron (57 to 45 Kp). The formulation areas of low viscosity remain the same.

From these experiments it was concluded that size distribution probably contributes to good processing more than does the presence of a coating agent on the UFAP.

In order to obtain an assessment of the processibility of emulsion freeze dried AP a direct comparison was made between .6 micron SWECO ground material and several samples of freeze dried UFAP. A 50/50 mixture of uncoated 1.9 micron fluid energy milled AP and each of the samples to be compared were processed in an 84% total solid propellant (74% AP and 10% aluminum in HTPB). A listing of the oxidizer samples tested, the particles sizes and end of mix viscosities is shown in Table 33. The SWECO ground .6 micron AP processes significantly better than the freeze dried AP in this particular formulation. Wide variance was noted in the processibility of the various samples of freeze dried AP. No readily apparent trend can be detected which relates these viscosities to particle size or type and concentration of coating on the UFAP.

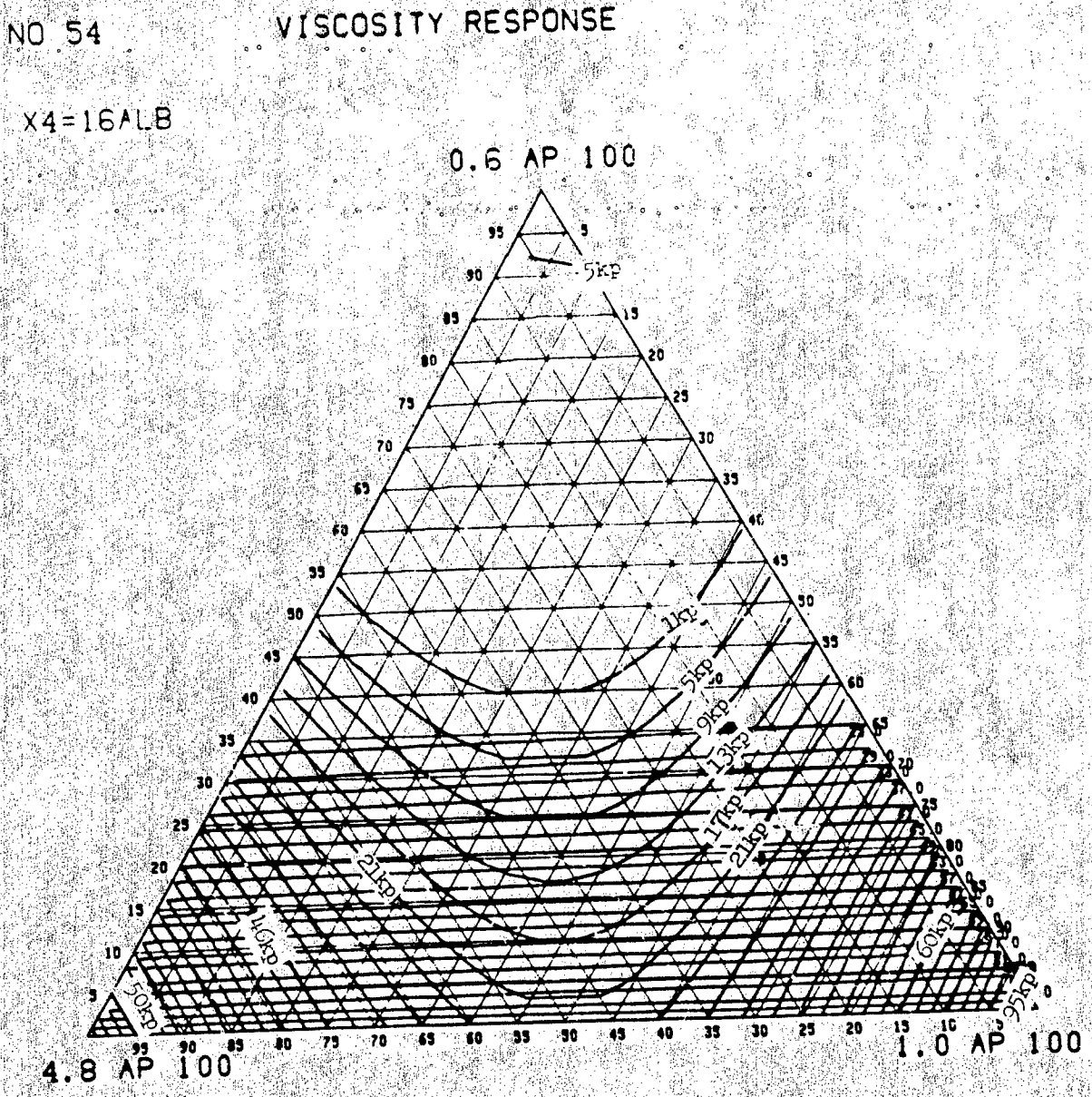


Figure 69. Viscosity Response Plot for 84% Total Solids Blend "D"

NO. 44 VISCOSITY RESPONSE PLOT HX-868

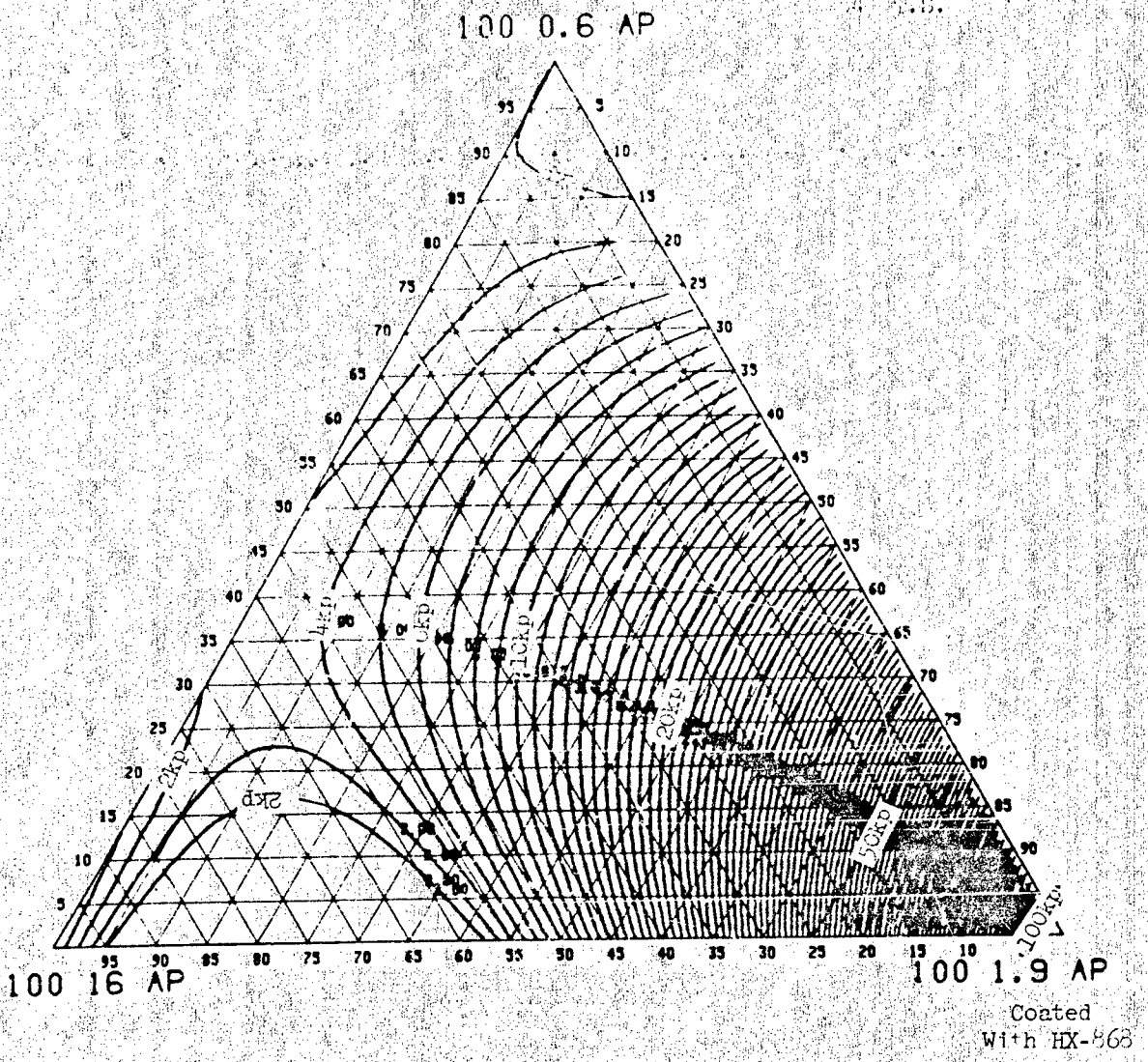


Figure 70. Viscosity Response Plot for 84% Total Solids Blend "D" with Coated 4.8 micron AP

VISCOSITY RESPONSE PLOT - BLEND D

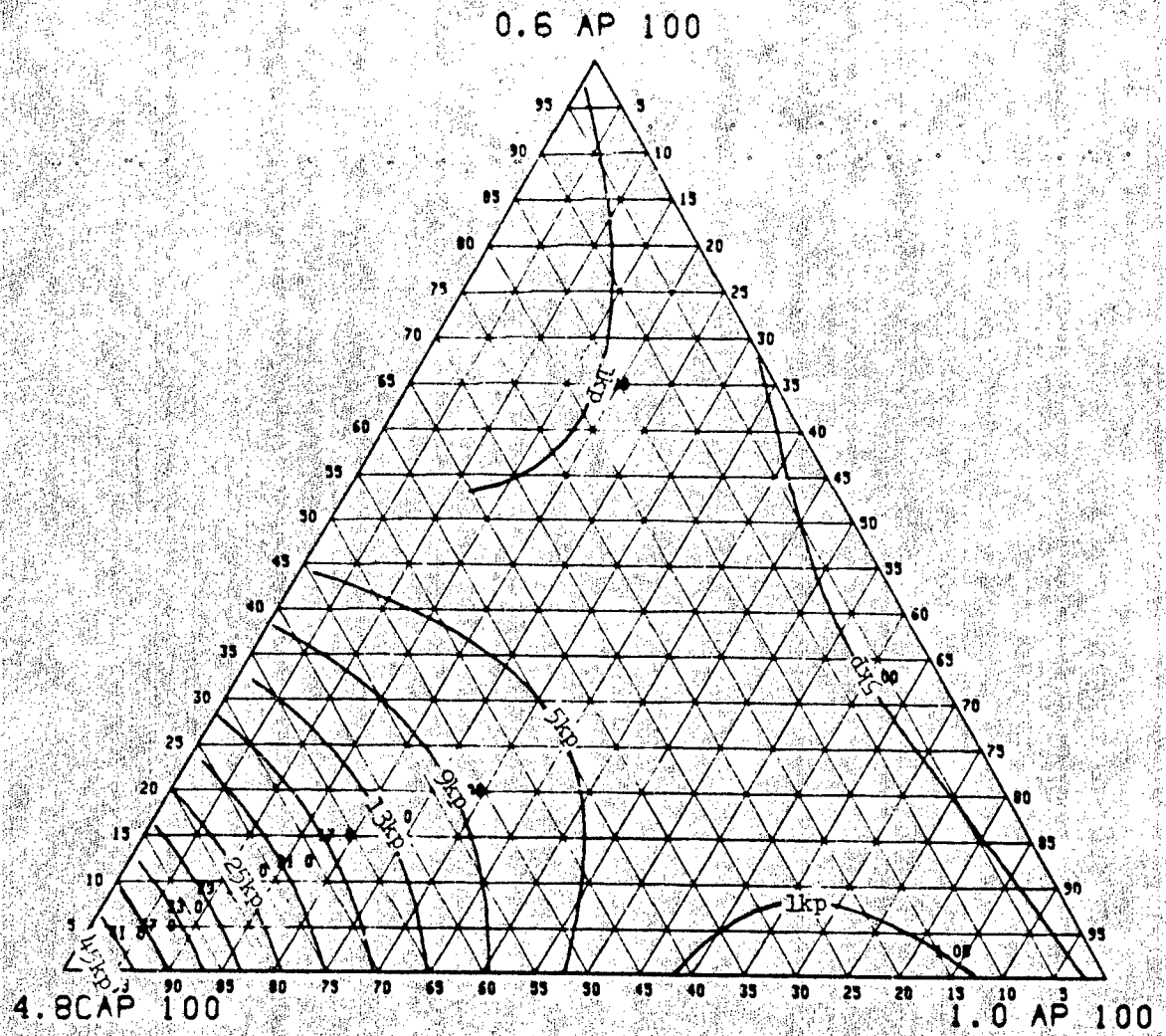


Figure 71. Viscosity Response Plot for Blend "D" with Coated 4.8 Micron AP

TABLE 33

PROCESSING OF FREEZE DRIED UFAP

AP - 70% Al - 10% HTPB - 20%

AP Blend is 50 parts 1.9 μ (uncoated) and 50 parts of each individual fraction listed in this table.

Sample No.	Coating	Particle Size	End-of-Mix Viscosity
FDI-47A	HX-868	0.6 μ	2 Kp
FDI-29D	1.0% NOPCOGEN 16-0 1.4% DOA	0.77 μ	52 Kp
FDI-29E	1.0% NOPCOGEN 16-0 0.5% Lecithin	0.76 μ	168 Kp
FDI-29F	1.0% NOPCOGEN 16-0 0.7% DOA 0.5% Lecithin	0.77 μ	36 Kp
FDI-30C	2.1% DOA, 0.7% NOP-COGEN 16-0 0.3% Twitchel	0.98 μ	12 Kp
FDI-41A	2.1% DOA, 1.0% NOPCOGEN 16-0	0.68 μ	30 Kp
FDB-L-43A)	0.5% Armeen SZ	0.95 μ	24 Kp
FDB-6A	2.1% DOA 0.7% NOPCOGEN 16-0	0.85 μ	12 Kp
FDB-6B	2.1% DOA 2.0% NOPCOGEN 16-0	0.56 μ	56 Kp

*SWECO Vibro-Energy mill UFAP

Figures 72-75 exhibit viscosity response plots for four samples of freeze dried AP which were substituted directly for the 0.6 micron SWECO ground UFAP in blend "A". The areas of minimum viscosity are all located in the lower left hand corner (low concentrations of FD-UFAP and 1.9 micron AP) of the triangular plot and the area of minimum viscosity increases with increasing particle size seemingly without regard for coating type and concentration. It is also apparent that the areas of minimum viscosity will also be formulation areas from which quite low burn rates will be obtained.

The width of the distribution for freeze dried AP approximates the distribution width for Vibro-energy milled UFAP; therefore, it is postulated that difficulty in processing freeze dried UFAP can be attributed to difficulty in dispersing the freeze dried material.

In view of the anomalies observed in the ballistics characteristics and the fact that equivalent sizes in distributions of UFAP process differently, a brief attempt was made to determine whether a technique could be developed which would result in significantly better dispersion of the various types of UFAP. It is obvious that if the UFAP can be dispersed then lower viscosities will result. This conclusion can be drawn from small packing fraction experiments which indicate high values of particle fraction for UFAP when completely dispersed.

Three techniques were evaluated for improving the dispersion of UFAP. The technique of keeping viscosity quite high during some portion of the mixing process should physically disperse AP if sample agglomeration is the problem. Secondly, application of ultrasonic energy to a low viscosity slurry of UFAP in a non-solvent (methylene chloride) should accomplish deagglomeration. Thirdly, the high speed mixing in the Waring blender of a low viscosity slurry should likewise result in better dispersion. All three of these techniques were examined as to their effect on propellant end-of-mix viscosity and essentially no change resulted. The best technique appeared to be the high shear mixing. This would indicate the dispersion problem is not one of physically agglomerated UFAP. The most likely suspect in preventing proper dispersion of the UFAP would be electrostatic attraction of the particles. This would be a particularly bad problem with freeze dried AP in that the drying process occurs under vacuum and a moderate high temperature. Large amounts of physical energy will not disperse particles agglomerated in this manner. Some technique must be developed to eliminate the electrostatic attraction before utilization of UFAP could be considered a viable technique for manufacturing reproducible high burning rate propellants.

VISCOSITY RESPONSE PLOT FDB6B

x=30ALB

FDB-6B - 2.1% IOA
2.0% Nopcogen 16-D

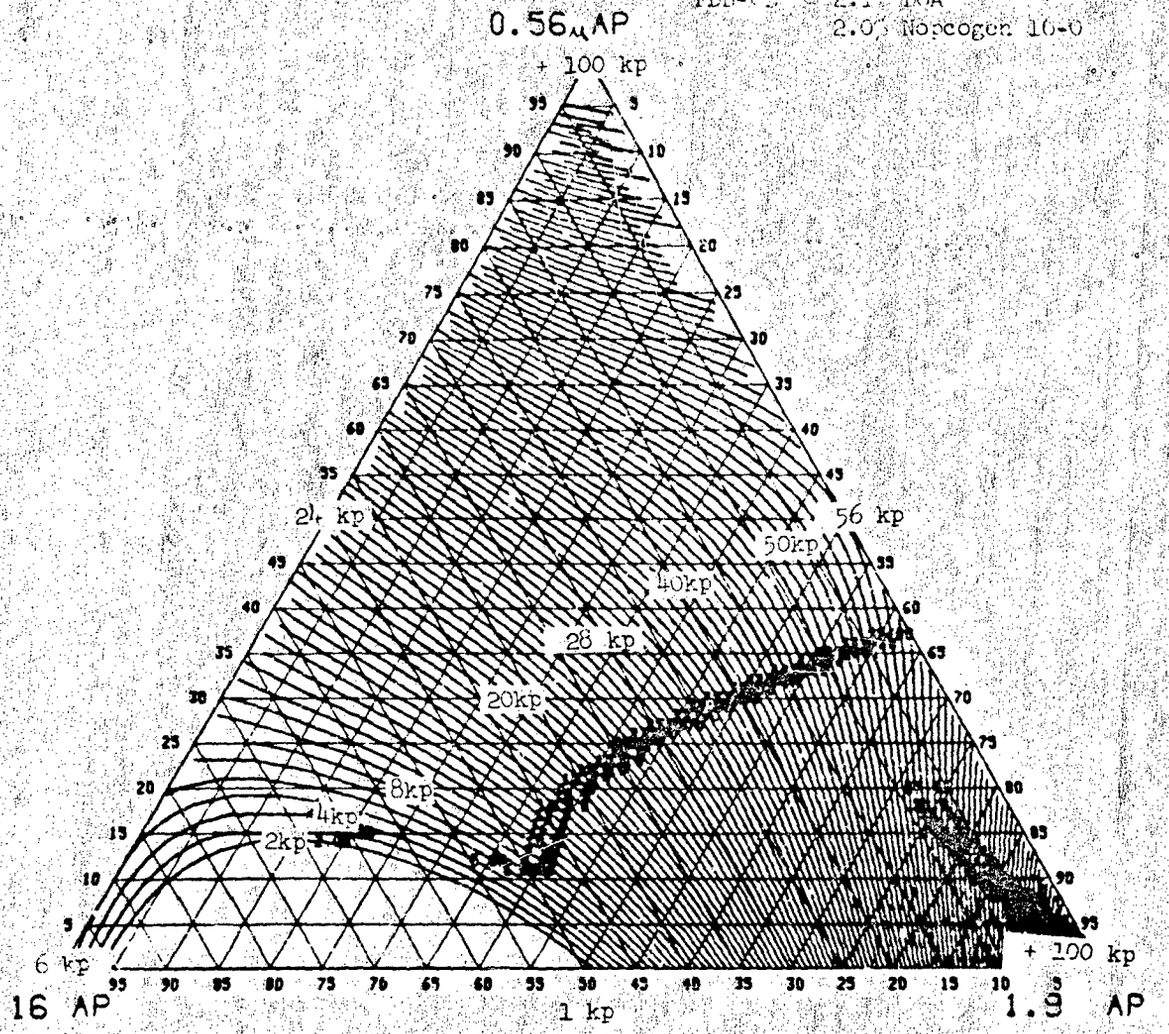


Figure 72. Viscosity Response Plot

VISCOSITY RESPONSE PLOT FDI-29F

x=30ALB

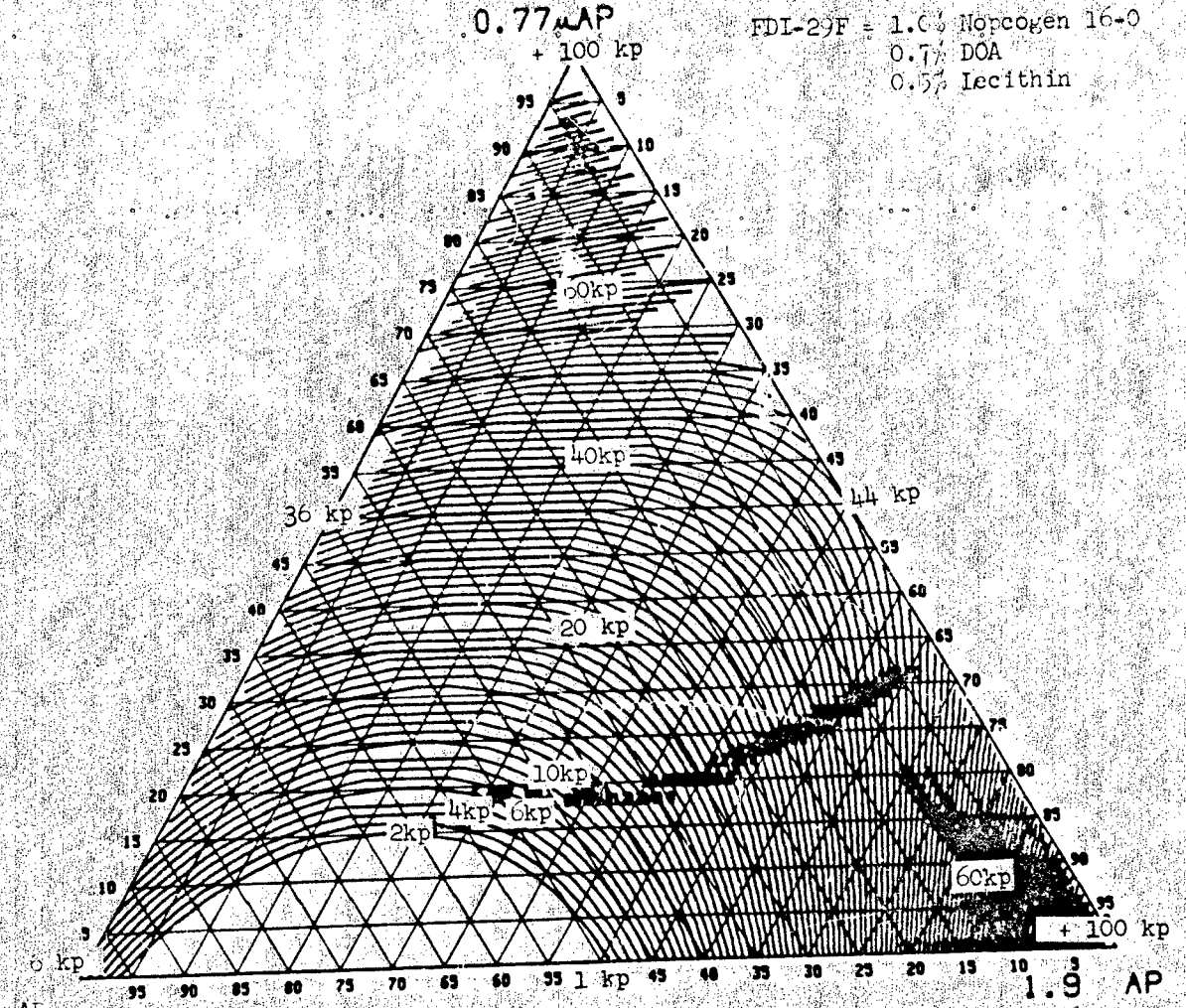


Figure 73. Viscosity Response Plot

VISCOSITY RESPONSE PLOT FDB-13A

x=30ALB

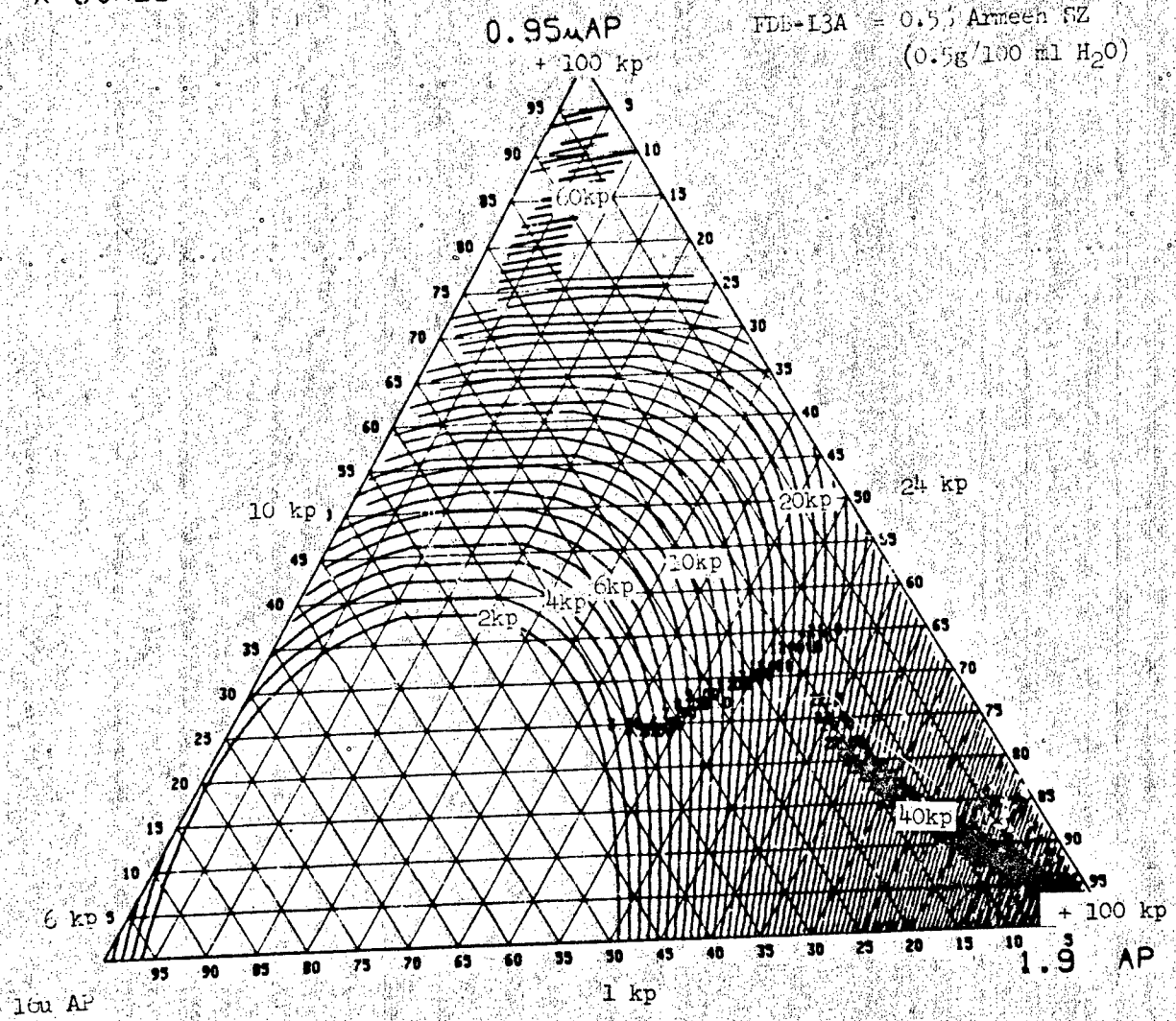


Figure 74. Viscosity Response Plot

VISCOSITY RESPONSE PLOT FDI-30C

X=30ALB

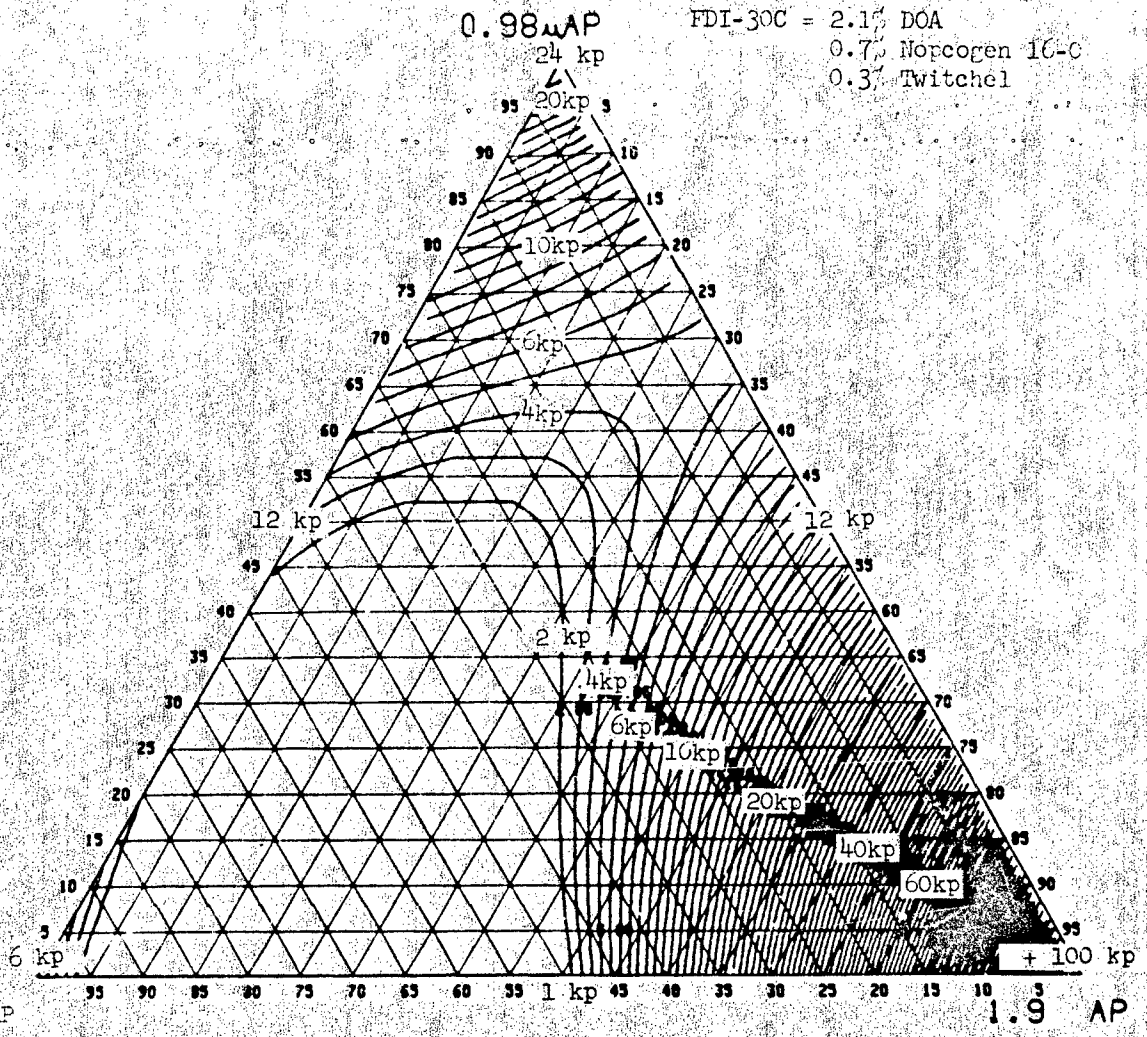


Figure 75. Viscosity Response Plot

APPENDIX A

OPERATING PROCEDURES FOR HAZARDS AND SENSITIVITY TESTS

CONTENTS:

Procedure No. 00000-1355-402 Card Gap Test

PE 72-251 Test Set-Up for Hot or Cold Card Gap Test Shots

Procedure No. 00000-1338-401 Lead Column Test

Procedure No. 00000-1337-401 Ignition and Unconfined Burning Tests

Extracts from RI 5624, Bureau of Mines (See pages 7, 8, 12, 13, 14, 15)

PE 73-96 Detonation Test on UFAP Shipping Container

PE 73-106 Detonation Tests on UFAP Shipping Containers

PE 73-108 Detonation Tests on UFAP Shipping Containers

PE 73-110 External Heat Test on UFAP Shipping Container

Chapters 3 & 4 of updated copy of DSAR 8220.1 (old TB 700-2)

ORIGINATOR	Thiokol CHEMICAL CORPORATION HUNTSVILLE DIVISION HUNTSVILLE, ALABAMA	PROCEDURE NO. 00000-1355-402	DATE 72 Apr 27
APPROVED		SUPERCEDES NO. 00000-1355-401	PAGE 1 of 6
APPROVED		REVISION / DATE / APPROVED	
APPROVED / QEC NO. 8767 / DATE	OPERATION PROCEDURE		

CARD GAP TEST (FOR DETONATION SUSCEPTIBILITY)

INTRODUCTION

This document will be used as a general operating procedure for conducting card gap tests. If the nature of the material warrants, an SPI will be written for approval referencing this procedure as to charge composition and/or test modification.

This test generally complies with paragraph 3.12 of TB-700-2 (Explosives Hazard Classification Procedure) but is modified to accommodate its use for determining the detonation sensitivity of cast, slurried or liquid explosives. These test materials which are charged into the pipe (sample holder) are referred to as the "acceptor" charge. The explosive charge which furnishes the necessary stimulus for the "acceptor" charge is called the donor and consists of two (2) pressed pentolite pellets 2 inches in diameter and one (1) inch high which will be initiated by a J-2 electric blasting cap. Maximum weight of charge (acceptor) will be 300 grams and will be contained in a steel pipe 1 7/8" O.D., 0.219 wall x 5.5." long. The general test set-up is shown in Figure 1. When liquids, slurries, or powders are to be tested, the sample holder pipe is to be prepared as shown in Figure 2 prior to set-up; for these materials, the standoffs can be omitted. For solids (e.g. cured propellant), the material can either be cast and cured in the pipe, or machined or trimmed from a bulk shape and inserted in the pipe. The test materials and set-up should be at a temperature of $25^{\circ}C \pm 5^{\circ}$ when initiated.

SAFETY REQUIREMENTS

1. Test will not be done on rainy days or during approach or progress of any electrical storm.
2. Radio transmitters will not be operated in the area when explosives and detonators are present.
3. Electric detonators will not be transported in the vehicle containing explosives. Inspect detonator lead wires to insure that the ends are electrically shorted.
4. One person will obtain one detonator and arm high explosive donor at the test site. Insure that firing cable is electrically shorted prior to Step 5 below.
5. The blasting cap will be installed after the test setup is completed and in place.
6. Personnel handling explosives will wear flame-proof lab coats or coveralls, conductive-soled shoes and approved eye protection.

7. Firing circuit will be completely insulated from the ground or other conductors such as bare wires, rails, pipes or other paths of stray currents.

8. Don't have electric wires or cables of any kind near electric blasting caps or other explosives except at the time of and for the purpose of firing the charge.

9. Electric blasting cap wires as well as the firing cable wires will be short circuited until ready to fire.

10. In the event of misfire, wait 15 minutes before approaching location of charge.

11. The test site should be a minimum of 400 yards from inhabited buildings.

12. Test observers shall take cover in the blockhouse during arming and testing.

PROCEDURE

The card gap tests will be conducted at the Burn-out pit area (Bldg. 7338) and setup as shown in Figure 1 at the base of the earth bank provided for this purpose. Two 80 gram Pentolite pellets, initiated by a J-2 Engineer's Special blasting cap will function as the donor explosive. These will be placed on top of the pipe and in a cardboard tube with the "gap" material (cellulose acetate cards or discs) between the donor and acceptor charge. At the other end of the acceptor will be placed a steel plate 6" x 6" x 3/8". This plate will be placed on the wooden stand and leveled. A round wooden plug with an axially drilled hole will be placed on the pentolite pellets and taped to the cardboard tube. One person will place the cap in the hole provided in the wooden plug.

Two 12-volt storage batteries connected in series are located in the test bunker (barricaded trailer) and will be used as the energy source to fire the cap. The firing will be accomplished in the following manner:

1. Verify that both ends of the firing cable (i.e. the test site end and the opposite end) are shorted.

2. While the blasting cap is being installed and connected at the test site, one man shall hold the end (opposite the test site) of the firing cable (shorted per Step 1) away from any power source by grasping the insulated portion rather than the naked wire.

3. After the test setup has been completed and the blasting cap put in place, the firing site will be evacuated. One man will then disconnect the shorted end of the cap wires and connect them to the firing cable by twisting each of the cap wires with one of the wires on the firing cable. Make certain that this connection is adequately protected against shorting out by allowing sufficient air space or taping. Wrap or tape cap wires on a wooden stake to insure that a tug on the firing cable will not dislodge the test specimen. This man will then return to the test bunker.

4. Sound warning horn.
5. Fire test specimen by touching bare ends of lead wires across 24 volt battery terminals.
6. Sound "all clear."

EVALUATION

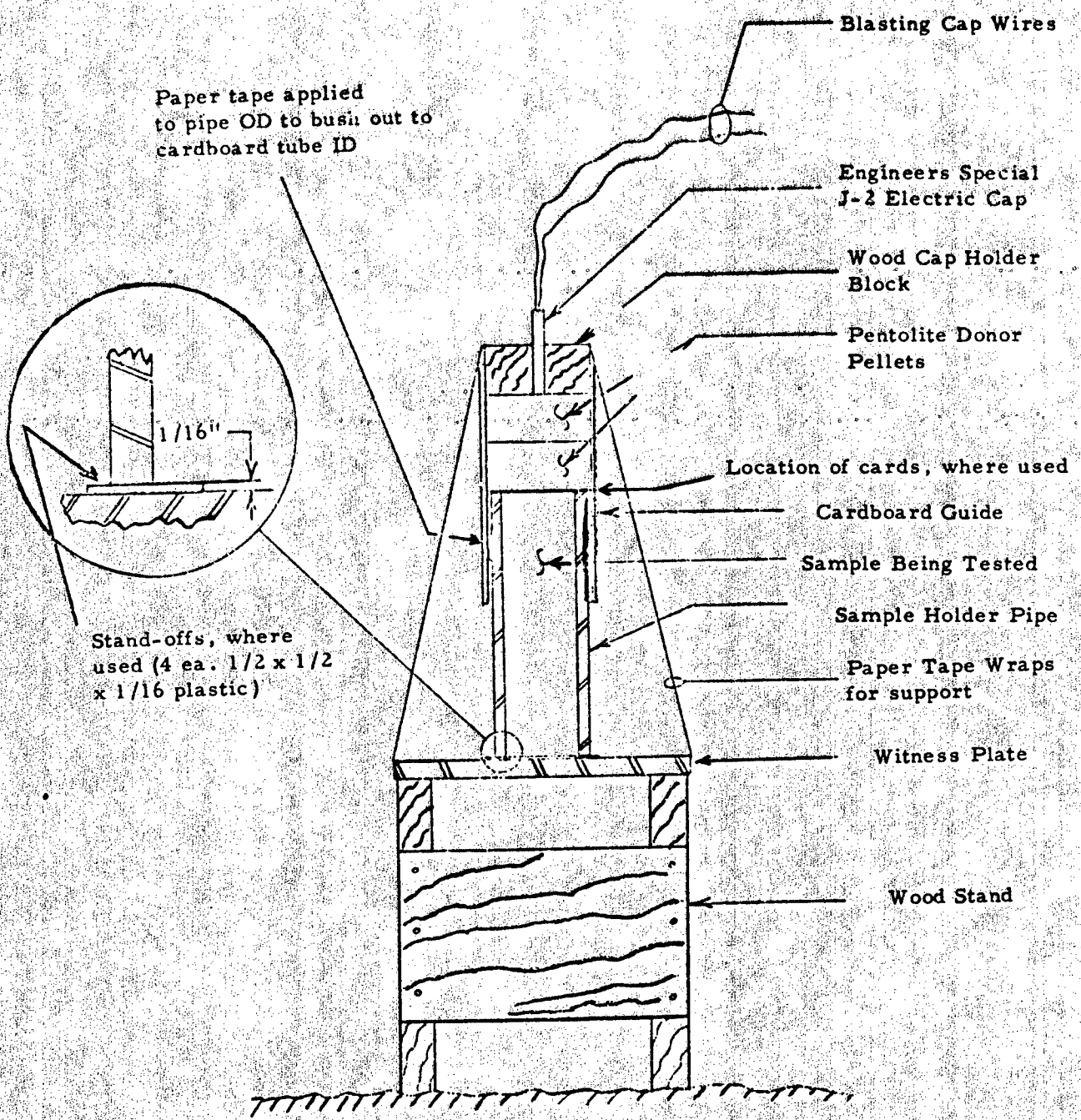
The card gap test is normally run a minimum of three times with zero gap (no cards) if no detonation occurs. Detonation of the sample material is indicated when a clean hole is cut in the witness plate. If a detonation occurs with any of the zero gap shots, additional tests are normally performed using cards between the pentolite donor and the test material in the pipe. The first such shot utilizes 8 cards. If a detonation occurs, the number of cards is doubled (i.e. 16 cards) for the second test. Doubling the number of cards for succeeding tests will be continued until no detonation occurs. When a number of cards is reached that prevents detonation, the next test will be with the number of cards reduced by half the preceding increment of increase. If detonation then occurs, the next shot will have the number of cards increased by half the preceding increment. This process will be followed until the break point between positive (detonation) and negative (no detonation) results is defined to the desired precision (i.e. 1-2 cards or possibly ± 5 cards). If the material being tested is known to be similar in sensitivity to a previously tested material, the first test can be made at the number of cards defined for the known material. Examples of test series are given in Table I.

If damage other than a clean puncture occurs to the witness plate (e.g. plate dented or broken into pieces), the extent of such damage should be noted in the test log book. Plate cracking or breaking up may indicate material defects, necessitating repeat tests.

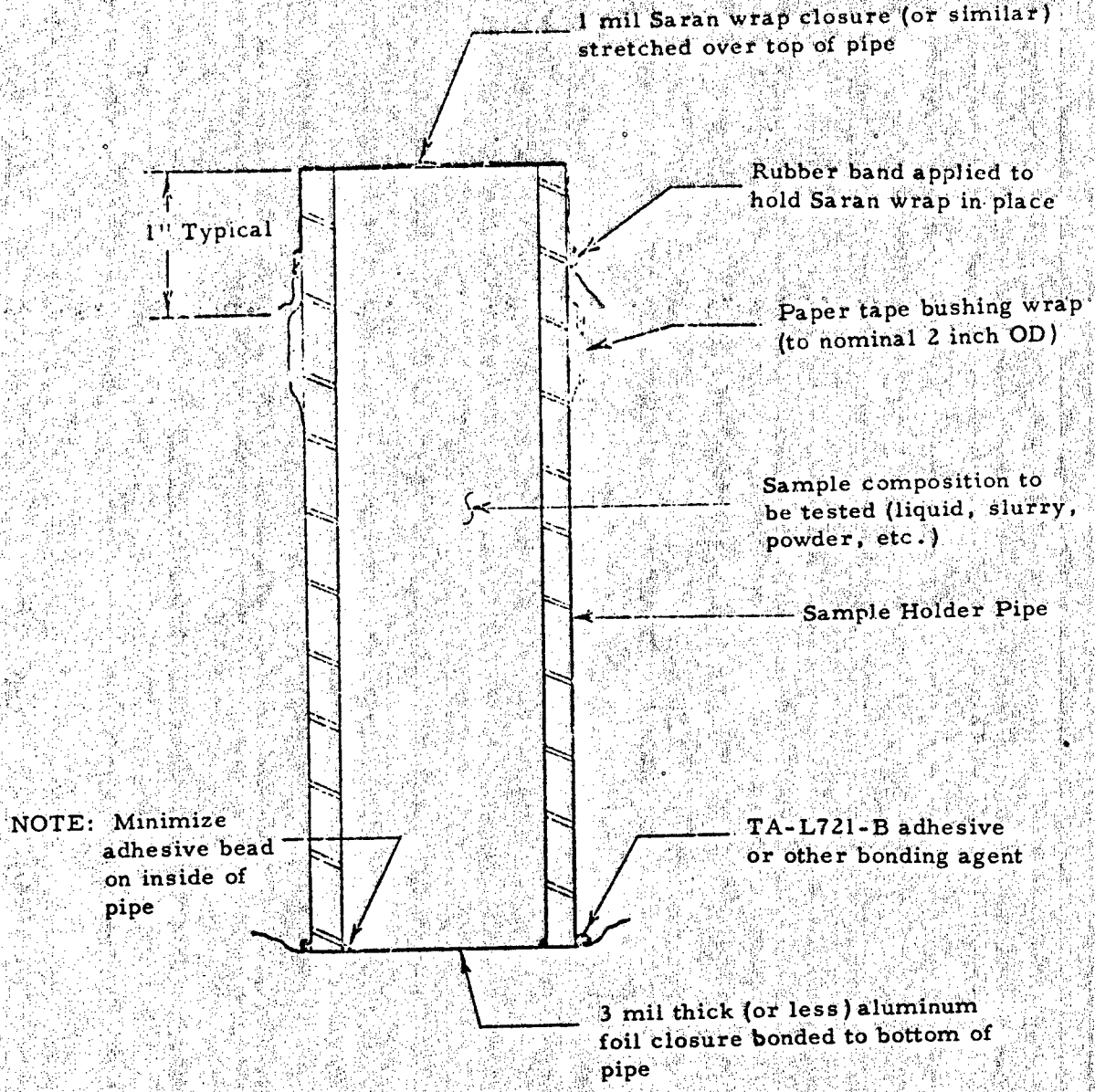
TABLE I

EXAMPLE CARD GAP TEST SERIES

Test Series	Shot No.	No. Cards	Positive	Negative	Cards for Next Shot
103	A	0		X	0
	B	0		X	0
	C	0		X	No further tests needed
Above material is negative at zero gap (or zero cards).					
118	A	0	X		8
	B	8	X		16
	C	16	X		32
	D	32		X	24
	E	24	X		28
	F	28		X	26
	G	26	X		27
	H	27		X	No further tests needed
Above material has a sensitivity of 26-27 cards.					
188	A	0		X	0
	B	0	X		8
	C	8		X	4
	D	4		X	2
	E	2	X		3
	F	3	X		No further tests needed
Above material has a nominal sensitivity of 3-4 cards, but apparently is not very reproducible.					



CARD GAP TEST SET-UP



SAMPLE HOLDER FOR LIQUIDS OR POWDERS

FIGURE 2

SPECIAL PROCESS INSTRUCTION PE 72 — 251

Thiokol / HUNTSVILLE DIVISION Huntsville, Alabama A DIVISION OF THIOKOL CHEMICAL CORPORATION	PROPELLANT: TP- N/A MIX	MOTOR: TX- N/A NO.	
	GTC NO. 8878 TASK RELEASE 460-84-01 CP CHANGE: YES ( ) NO (X)	INSPECTION YES ( ) NO (X)	DATE 6/22/72

ORIGINATOR	R&D GRP SUPERVISOR	R&D SECTION CHIEF	MFG. SECTION CHIEF
S. L. Vance	E Schreck	D. K. Murphy	N/A

TEST SET-UP FOR HOT OR COLD CARD GAP TEST SHOTS

The purpose of this SPI is to specify changes in the test set-up and procedure specified in Operating Procedure No. 00000-1355-402, CARD GAP TEST (FOR DETONATION SUSCEPTIBILITY), for hot or cold test shots. These changes are specifically required to allow testing on emulsion that must be held between 145° and 185°F, to keep the AP from precipitating out, and a frozen emulsion in liquid nitrogen. The same basic test set-up and procedure will be used whether hot or cold. In general, the material to be tested will be taken to the test area in a suitable insulated container such that it can be maintained at the required temperature until ready for use. The card gap pipes will likewise be preconditioned to approximately the needed temperature and transported in insulated containers. And, finally the heat sink fluid, liquid nitrogen or hot water, will be taken to the test area in Dewar type flasks. After positioning the witness plate on the wooden stand, the heat sink fluid reservoir, consisting of a quart ice cream cup with a special bottom (aluminum foil bonded with TA-L7218 adhesive), will be placed on the witness plate. The empty card gap pipe will then be placed inside the reservoir (see the attached figure), using gloves to prevent burns. Heat sink fluid will then be poured into the reservoir to fill the space around the pipe. The liquid level should be about 3/8 to 1/4 inch below the top of the pipe. The material to be tested will then be charged to the pipe, again using gloves to prevent burns. The cardboard support tube will then be placed down over the pipe, and the cards, if used, dropped into place. The pentolite booster pellets and cap holder and initiator cap will then be installed per procedure. Operations performed after the test material is charged should be rehearsed ahead of time so that the time involved is held to a minimum. Under no circumstances, however, will the safety precautions specified in the procedure be compromised.

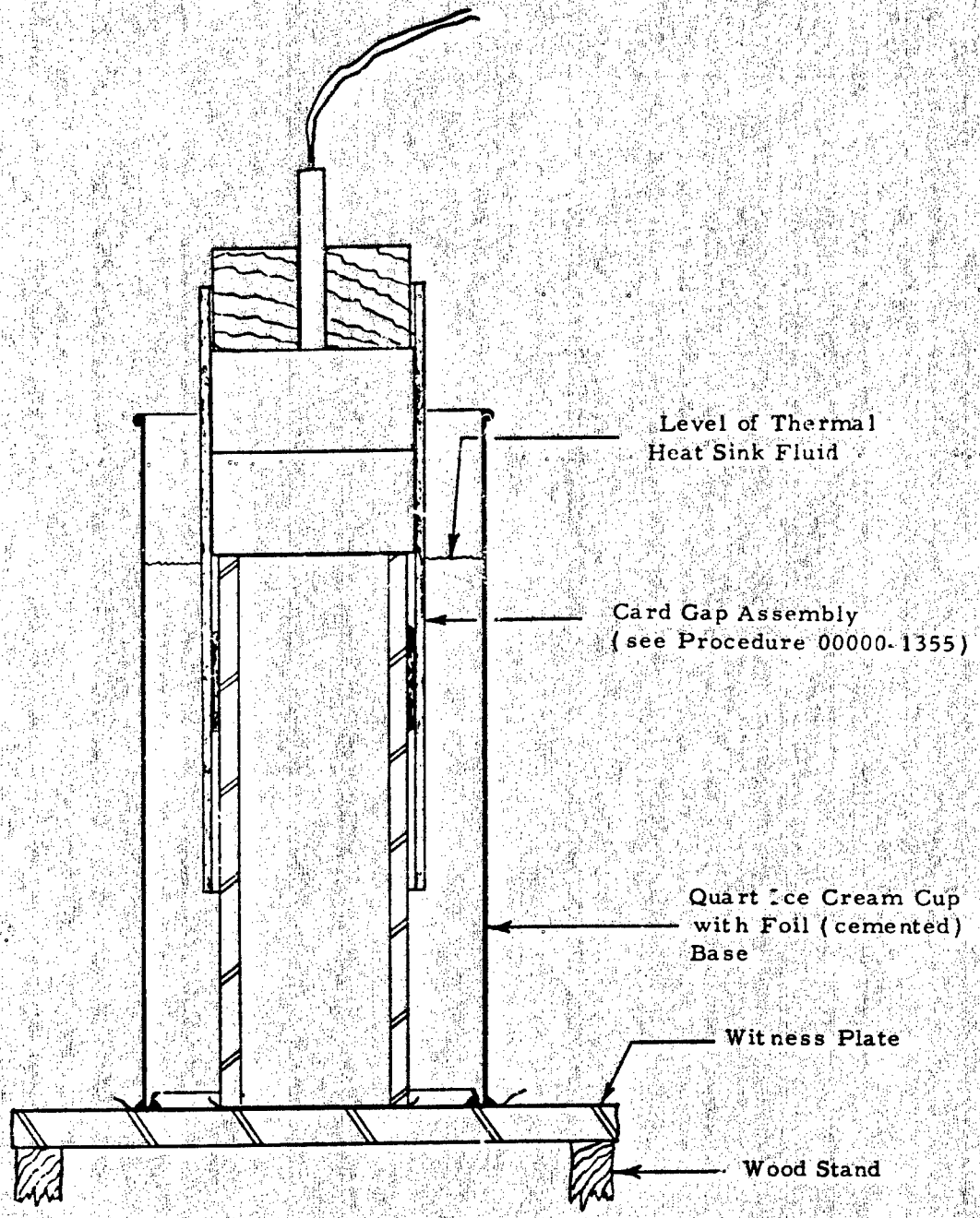


Figure IV-13. Card Gap Test Setup for Hot or Cold Test Shots

ORIGINATOR	Thiokol CHEMICAL CORPORATION HUNTSVILLE DIVISION HUNTSVILLE ALABAMA	PROCEDURE NO. 0000-1338-401	DATE 72 Mar 29
APPROVED		SUPERCEDES NO. NA	PAGE 1 of 5
APPROVED	OPERATION PROCEDURE	REVISION / DATE / APPROVED	
APPROVED			
QTC NO. 5720 / DATE 4/6/72			

LEAD COLUMN TEST (FOR UNCONFINED DETONATION)

INTRODUCTION

This document will be used as a general operating procedure for conducting lead column detonation tests on cured propellant samples, slurries, pastes, and powders. If the nature of the material warrants, an SPI will be written for approval referencing this procedure as to charge composition and/or test modification.

This test generally complies with paragraph 3-8 of TB 700-2 (Explosives Hazard Classification Procedures), but contains modifications to accommodate testing non-solids. For cured propellants or rigid solid bodies, the test sample will be a cube two inches (± 1/4 inch) on an edge. This sample will be placed on top of a lead "witness" column, which in turn is placed on a steel plate base. A No. 8 electric blasting cap is positioned against and on top of the solid sample and initiated. Deformation of the lead column (mushrooming) is considered evidence of sample detonation. For non-rigid solids (powders), slurries, or other liquids, the sample material will be contained in a suitable container which then is tested as above. For these materials, the test sample weight will be a minimum of 150 grams and a maximum of 300 grams, with the weight and volume of each sample recorded.

SAFETY REQUIREMENTS

1. Explosive Limits:

   All explosive components used in any one test will not exceed 2 1/2 lbs. of H.E. (UFAP in slurry, paste or powder considered as constituting part of the H.E.).

2. Personnel Limit:

   Three (3) - Active Test Personnel
   One (1) - Transient

3. Test will not be conducted on rainy days or during approach or progress of any electrical storm.

4. Radio transmitters will not be operated in the area when explosives and detonators are present.

5. Electric detonators will not be transported in the vehicle containing explosives. Inspect detonator lead wires to insure that the ends are electrically shorted.

6. One person will obtain one detonator and arm the test set-up at the test site. Insure that firing cable is electrically shorted prior to Step 7 below.

7. The blasting cap will be installed after the test setup is completed and in place and the firing cable checked for current flow with a blasting galvanometer or equivalent.

8. Personnel handling explosives will wear flame-proof lab coats or coveralls, conductive-soled shoes and approved eye protection.

9. Firing circuit will be completely insulated from the ground or other conductors such as bare wires, rails, pipes or other paths of stray currents.

10. Don't have electric wires or cables of any kind near electric blasting caps or other explosives except at the time of and for the purpose of firing the charge.

11. Electric blasting cap wires as well as the firing cable wires will be short circuited until ready to fire.

12. In the event of misfire, wait 15 minutes before approaching location of charge.

13. The test site should be a minimum of 400 yards from inhabited buildings.

14. Test observers shall take cover in the block house during arming and testing.

PROCEDURE

The test will be conducted at the burn-out pit area (Building 7338 or Snake Pit) with the test set-up shown in the attached sketches. The test set-up should be positioned in the testing trench dug in the end of the earth bank. A nominal 12 inch by 12 inch steel base plate (1/2 inch thick) is positioned on the ground or on a wooden stand and levelled. The lead column, 1 1/2 inches diameter by 4 inches long, with square ends, and cast of solid lead, is placed on end in the center of the plate. The test sample, or container filled with sample, is placed on top of the column. A wood or cork cap holder is placed on top of the sample and the setup secured with tape to prevent falling over. A No. 8 electric blasting cap is inserted in the hole in the cap holder until it contacts the sample. Two 12 volt batteries connected in series are located in the test bunker (barricaded trailer) and are used as the energy source to fire

the cap. The firing will be accomplished in the following manner:

1. One man will hold the end of the shorted firing cable in his hand and away from any power source.

2. After the firing site has been evacuated, one man will disconnect the shunted end of the electric blasting cap and connect it to the firing cable by twisting each end of the cap wires on the firing cable. Make certain that this connection is adequately protected against shorting out by allowing sufficient air space or taping. Wrap or tape cap wires on a wooden stake to insure that a tug on the firing cable will not dislodge the test specimen. This man will then return to the test bunker.

3. Make a continuity check as per "Safety Requirements."

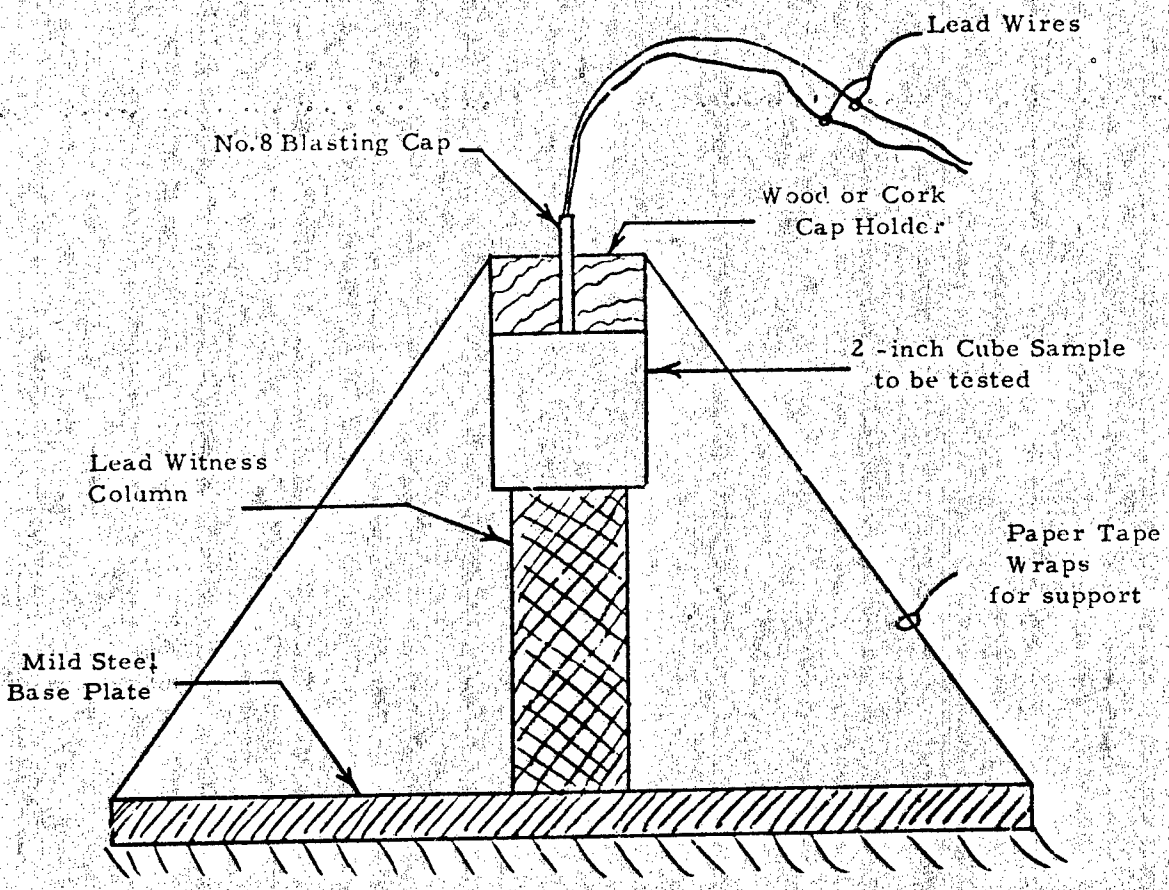
4. Sound warning horn.

5. Fire test specimen by touching battery clips on ends of lead wires across 24 volt battery terminals.

6. Sound horn for all clear.

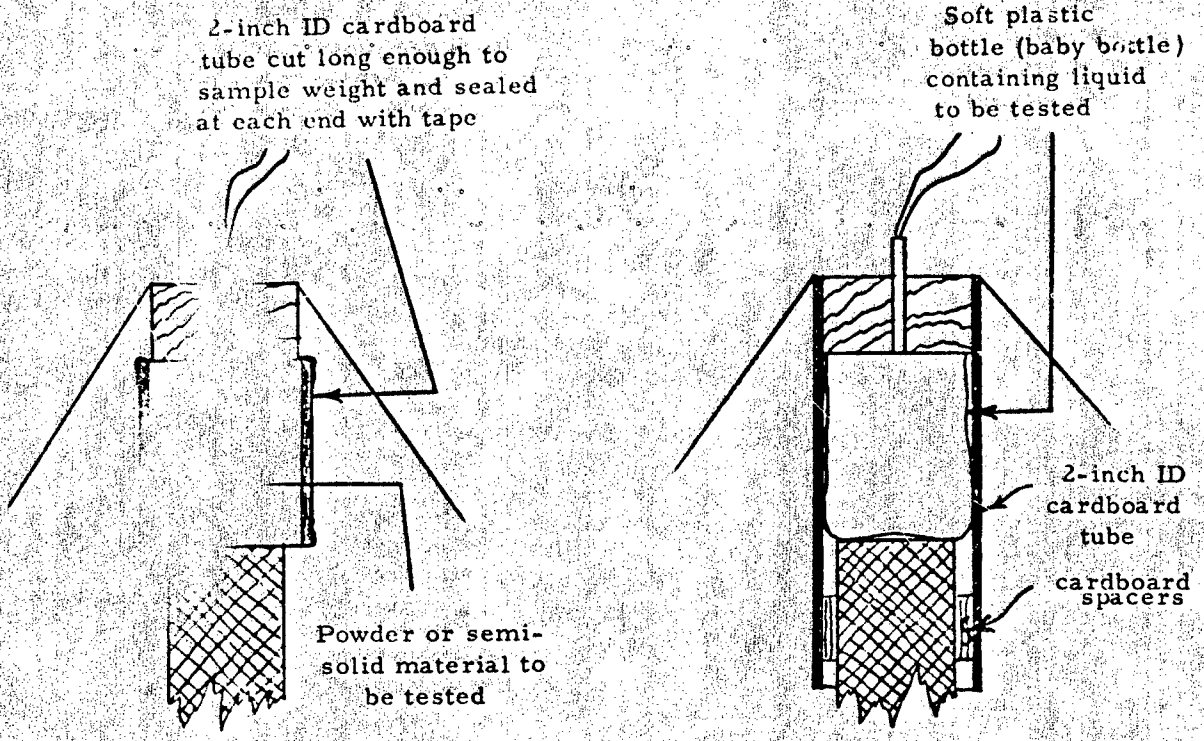
EVALUATION

Normally, this test will be conducted a minimum of five times, or until detonation occurs, whichever is the least number of tests. Where desirable for reporting purposes, photos can be taken of a typical "before" set-up, and either of the "after" condition or of the lead witness columns after the shots. For DOT Shipping Classification tests, a notation should be recorded for each shot as to whether the sample exploded, burned, or was simply fragmented.



Note: Base plate shown on ground, may be supported by wood stand

LEAD COLUMN TEST SET-UP



Note: Sample containers cut to length needed for sample weight; end of blasting cap to be in direct contact with sample material.

ALTERNATE SET-UPS FOR MATERIALS THAT ARE NOT RIGID SOLIDS

ORIGINATOR	Thiokol CHEMICAL CORPORATION HUNTSVILLE DIVISION	PROCEDURE NO. 00000-1337-401	DATE 72 Mar 29
S.L. Vance		SUPERSEDES NO. NA	PAGE 1 OF 4
APPROVED	OPERATION PROCEDURE	REVISION / DATE / APPROVED	
APPROVED			
APPROVED QTC NO 8721 DATE 4/6/72			

IGNITION AND UNCONFINED BURNING TESTS

INTRODUCTION

This document will be used as a general operating procedure for conducting ignition and unconfined burning tests on cured propellant samples, slurries, pastes, and powders.

This test generally complies with Paragraph 3-9 of TB-700-2 (Explosives Hazard Classification Procedures), but contains modifications to accommodate testing non-solids. For cured propellants or rigid solid bodies, the test sample will be a cube 2 inches (± 1/4 inch) on an edge. A cube is placed on a bed of kerosene-soaked sawdust (enough for one square foot 1/4 inch thick). The sawdust is then ignited with an electric match igniter and the results observed and recorded. This test (a single cube sample) is run two times. A third test is then run where four samples are placed end-to-end in a single row on sawdust as above. The sawdust is ignited at one end of the row. The purpose of the test is to determine if the sample will transit from deflagration (burning) to detonation when unconfined. For non-rigid solids (powders), slurries, or other liquids, the sample material will be contained in a suitable container which then is tested as above. For these materials, the test sample weight will be a minimum of 150 grams and a maximum of 300 grams, with the weight and volume of each sample recorded.

SAFETY REQUIREMENTS

1. Explosive Limits:

   All explosive components used in any one test will not exceed 2 1/2 lbs. of H.E. (UFAP in slurry, paste or powder considered as constituting part of the H.E.).

2. Personnel Limit:

   Three (3) - Active Test Personnel
   One (1) - Transient

3. Test will not be conducted on rainy days or during approach or progress of any electrical storm.

4. Radio transmitters will not be operated in the area when explosives and igniters are present.

5. Electrical igniters will not be transported in the vehicle containing explosives. Inspect igniter lead wires to insure that the ends are electrically shorted.

6. One person will obtain one igniter and arm test set-up at the test site. Insure that firing cable is electrically shorted prior to Step 7 below.

7. The igniter will be installed after the test setup is completed and in place and the firing cable checked for current flow with a blasting galvanometer or equivalent.

8. Personnel handling explosives will wear flame-proof lab coats or coveralls, conductive-soled shoes and approved eye protection.

9. Firing circuit will be completely insulated from the ground or other conductors such as bare wires, rails, pipes or other paths of stray currents.

10. Don't have electric wires or cables of any kind near electric igniters or other explosives except at the time of and for the purpose of firing the charge.

11. Electric igniter wires as well as the firing cable wires will be short circuited until ready to fire.

12. In the event of misfire, wait 15 minutes before approaching location of charge.

13. The test site should be a minimum of 400 yards from inhabited buildings.

14. Test observers shall take cover in the block house during arming and testing.

PROCEDURE

The tests will be conducted either at 7620 test pit or at the burn-out pit area (Bldg. 7338 or Snake-Pit), with the test set-up shown in the attached sketches. The test setup should be positioned where burning of the test sample can be seen (for timing purposes) either by remote CCTV or by direct sight from a remote location (such as the concrete blockhouse at the Snake-Pit). A bed of kerosene-soaked sawdust is spread out 1/4-inch thick by about one foot square in a tray made of aluminum foil. The sample (or samples) is placed in the center of the bed. An electric match igniter, with or without propellant booster (as needed), is placed in the bed at one side to cause ignition of the kerosene/sawdust. Two 12 volt batteries

[illegible] series, located in the bunker (barricaded trailer at 7338) are used as the energy source to fire the igniter. The firing will be accomplished in the following manner:

1. One man will hold the end of the shorted firing cable in his hand and away from any power source.

2. After the firing site has been evacuated, one man will disconnect the shorted end of the igniter and connect it to the firing cable by twisting each of the igniter wires with one of the wires on the firing cable. Make certain that this connection is adequately protected against shorting out by allowing sufficient air space or by taping. Wrap or tape firing cable to an anchor point to insure that a tug on the cable will not dislodge the igniter from the test set-up. This man will then return to the test bunker.

3. Make a continuity check as per Step 5 under Safety Requirements.

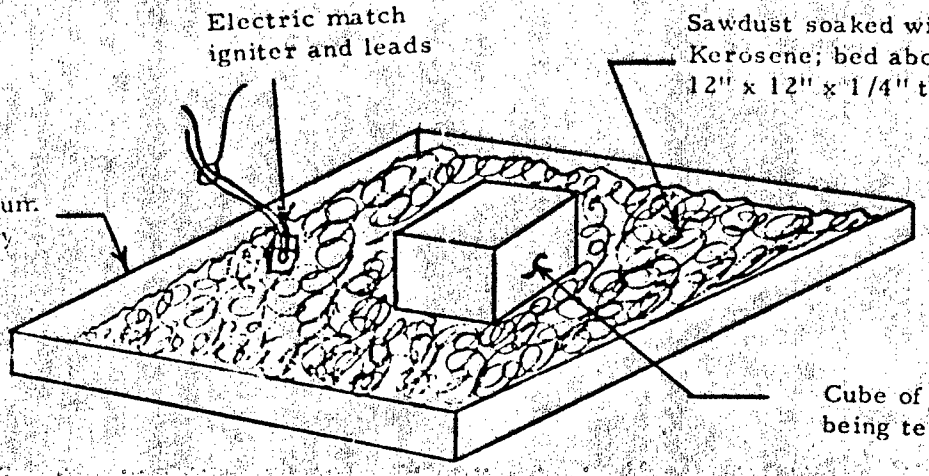
4. Sound warning horn.

5. Fire igniter by touching bare ends of firing cable lead wires across 24 volt battery terminals.

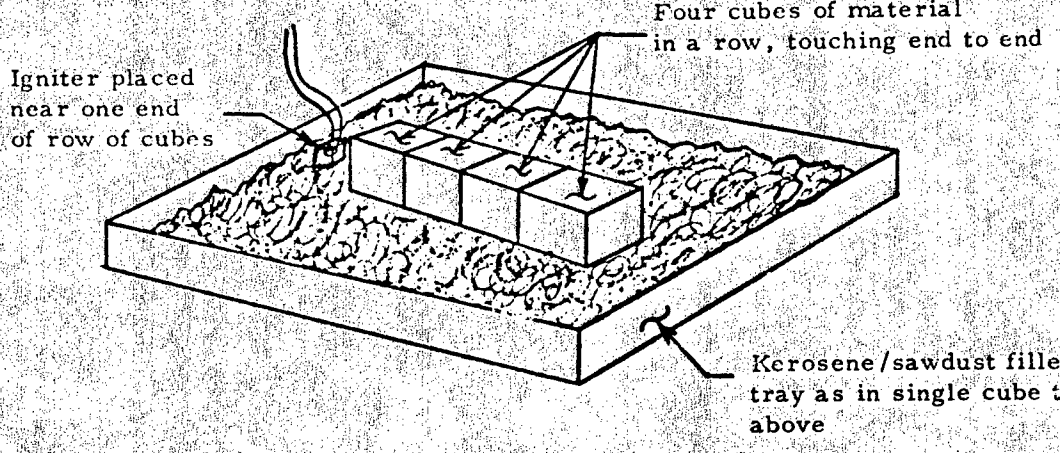
6. Sound "all clear" only after set-up has burned out.

EVALUATION

The test shot using one cube or sample will be run twice, and that using four cubes or samples in a row will be run one time. Where desirable for reporting purposes, photos can be taken of "before" and "after" firing set-ups. For DOT Shipping Classification tests, a notation should be recorded for each shot as to whether the sample exploded, and what the nominal burning time of the sample itself was.



SINGLE SAMPLE TEST SET-UP



FOUR SAMPLE TEST SET-UP

IGNITION AND UNCONFINED BURNING TESTS SET-UPS

Reproduced in part from Bureau of Mines Report of Investigation RI-5624, Laboratory Equipment and Test Procedures for Evaluating Explosibility of Dusts, by Henry G. Dorsett, Jr., Murray Jacobson, John Hagy, and Roger P. Williams, U. S. Dept. of the Interior, Bureau of Mines (1960).

5-minute period unless ignition occurs sooner. In the study of ignition of dust layers variations in diameter and depth of sample were investigated as well as the effects of air flow, duration of test, rate of heating and of preheating the dust.

Electrical Energy for Ignition of a Dust Cloud

The minimum electrical energy required to ignite a dust cloud is determined in the Hartmann apparatus. This consists of a vertically mounted, 2-3/4-inch diameter combustion tube 12 inches long and auxiliary equipment for producing the dust dispersion (fig. 3). The tube, made of lucite, is

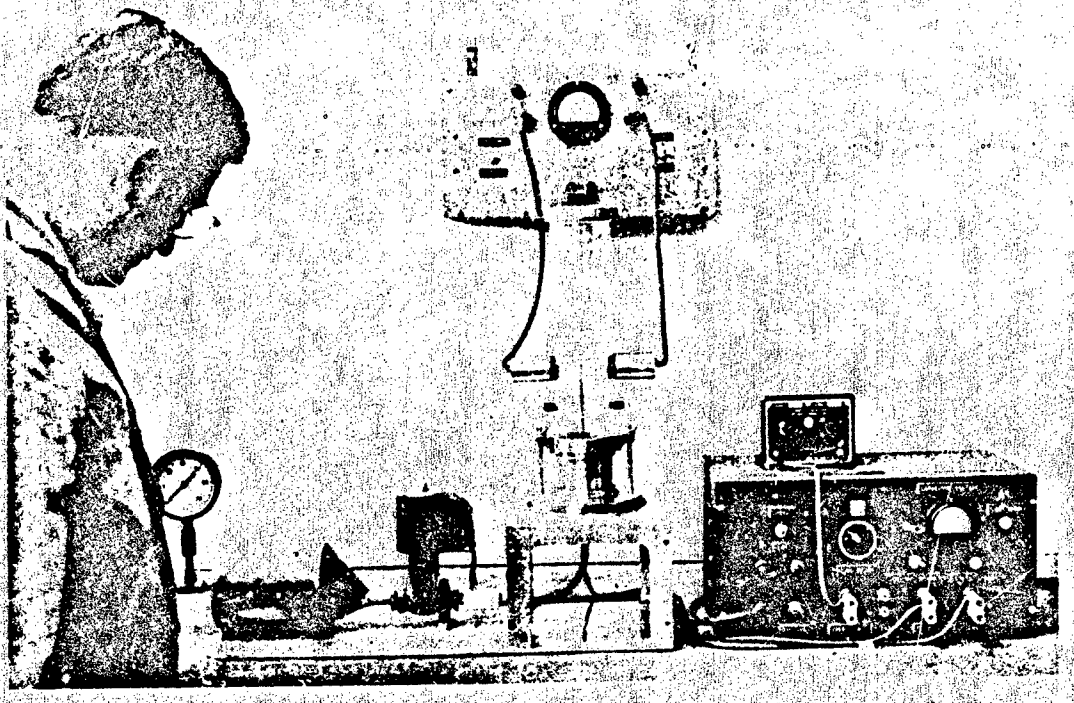


FIGURE 3. - Hartmann Explosibility Apparatus for Determining Electrical Energy for Ignition of a Dust Cloud.

attached to a cylindrical metal base or dispersion cup by hinged bolts. The top surface of the base is machined somewhat hemispherically in shape. The total volume of the chamber is 75 cubic inches (1.23 liters). Dispersion is accomplished by a single blast of air from an 80-cubic inch reservoir. The air flow, controlled by a 1/2-inch full-port solenoid valve, passes into the chamber to a mushroom-like deflector in the bottom of the dispersion cup. The optimum air pressure in the reservoir, determined by trial, ranges from 5 to 15 pounds per square inch gage (p.s.i.g.). The quantity of dust dispersed ranges from 5 to 10 times the minimum explosion concentration; lower dust

the value of minimum energy. The electrodes are 4 inches above the base of the tube. The ignition spark is obtained from the discharge of condensers at 100 volts (to increase the energy range the voltage is increased to 400 volts). Oil-impregnated, paper-dielectric condensers are used with a capacitance range from 2 to 100 microfarads. The condensers discharge through a luminous-tube transformer. An electronic timer, with adjustable delay, controls the spark discharge during dust dispersal; the optimum time is determined in preliminary trials. The energy of the spark (in joules) is calculated as $1/2\ CV^2$, where C is the capacitance of the condensers in farads, and V is the charging potential in volts. The minimum energy for ignition of the dust cloud is the least required to produce flame propagation 4 inches or longer in the tube. At low energy values (5 to 50 millijoules) the incremental capacitance is 1 microfarad; at higher energy values the increment is 4 microfarads. Four trials are made at each condenser setting; however, if the dust ignites in initial trials, lower energy is tried until a minimum is obtained. The value of the minimum ignition energy obtained is approximate as some electrical energy is dissipated in the transformer circuit and some remains stored in the condenser. For this reason, relative rather than absolute values of energy are obtained. In limited trials with direct condenser discharge at high voltages, comparable minimum ignition energies were obtained for several dusts.

Minimum Explosive Concentration

The minimum explosive concentration or the lower explosive limit of a dust is determined in the previously described Hartmann apparatus, except that an induction spark igniting source is used instead of the timed condenser discharge spark. This test was developed to provide data corresponding to those obtained in large-scale experiments in galleries and in the Experimental Coal Mine for Pittsburgh coal. A weighed amount of dust is spread in a thin layer in the dispersion cup. The top of the Hartmann tube (lucite) is covered with a filter-paper diaphragm held in place by a locking ring. A 1/16-inch hole is made in the center of the filter paper to prevent pressure build-up in the tube from the dispersing air and the tungsten electrodes are adjusted to a gap length of 3/16-inch. The electric spark is struck and the current adjusted to 23.5 milliamperes. The dust cloud is formed in the lucite tube by releasing air from the 80-cubic inch reservoir through the full-port solenoid valve; optimum air pressure ranges from 5 to 15 inches of mercury and is determined in preliminary trials. Following ignition of the dust sufficient pressure must develop to burst the filter-paper diaphragm; appearance of flame in the tube is not considered propagation. The pressure required to burst the paper diaphragm is about 2 to 3 p.s.i., depending on the rate of pressure rise. If propagation occurs for a given weight of dust, the weight is reduced by a 5-milligram increment and another trial made until a quantity is obtained which fails to propagate flame in any of four successive trials. The lowest weight at which flame propagates is used in calculating the minimum concentration. Tests are made with the electrodes 2 and 4 inches from the bottom of the tube. The average of the two weights is divided by the volume of the tube (75 cubic inches) for the value of the minimum concentration. For materials which tend to agglomerate, 3 to 5 percent fuller's earth may be admixed to facilitate dispersion

Explosion Pressure and Rates of Pressure Rise

Pressure and rates of pressure rise developed by a dust explosion are determined in a closed steel Hartmann tube (fig. 6). Dust dispersion is accomplished by releasing air from a 3-cubic inch (50 cc.) reservoir at 100 p.s.i.g., instead of from the 80-cubic inch reservoir at 14 p.s.i.g. previously described. The maximum pressure that can develop in the explosion tube from the dispersing air is 6.5 p.s.i.g.; however, because of rapid development of the dust explosion, the pressure from the dispersing air at the time of ignition is generally 2 to 3 p.s.i.g. Before 1950 the dust dispersion was accomplished by compressed air from the 80-cubic inch reservoir at a pressure of 28 inches (70 cm.) mercury. This dispersion system was less efficient than that currently in use. A full-port solenoid valve controls admission of the dispersing air and a check valve prevents the combustion gases from escaping back into the dispersion reservoir. Ignition of the dust cloud is normally produced by the 24-watt continuous spark source. For dusts which ignite with difficulty, the heated coil or guncotton source is tried.

The explosion pressure is measured by a Bureau of Mines manometer or by electronic transducers. The maximum pressure and the average and maximum rates of pressure rise developed in an explosion are determined from the pressure-time records, as shown on figure 7. The dispersion pressure (initial pressure in the tube at time of ignition) is subtracted from the peak explosion pressure to give a corrected maximum pressure. The average rate is obtained by dividing the maximum pressure by the time interval between ignition of the dust cloud and the occurrence of the maximum pressure. The maximum rate is the steepest slope of the pressure-time curve. Normally, explosion tests are made at dust concentrations of 0.10, 0.20, 0.50, 1.00, and 2.00 ounce per cubic foot.

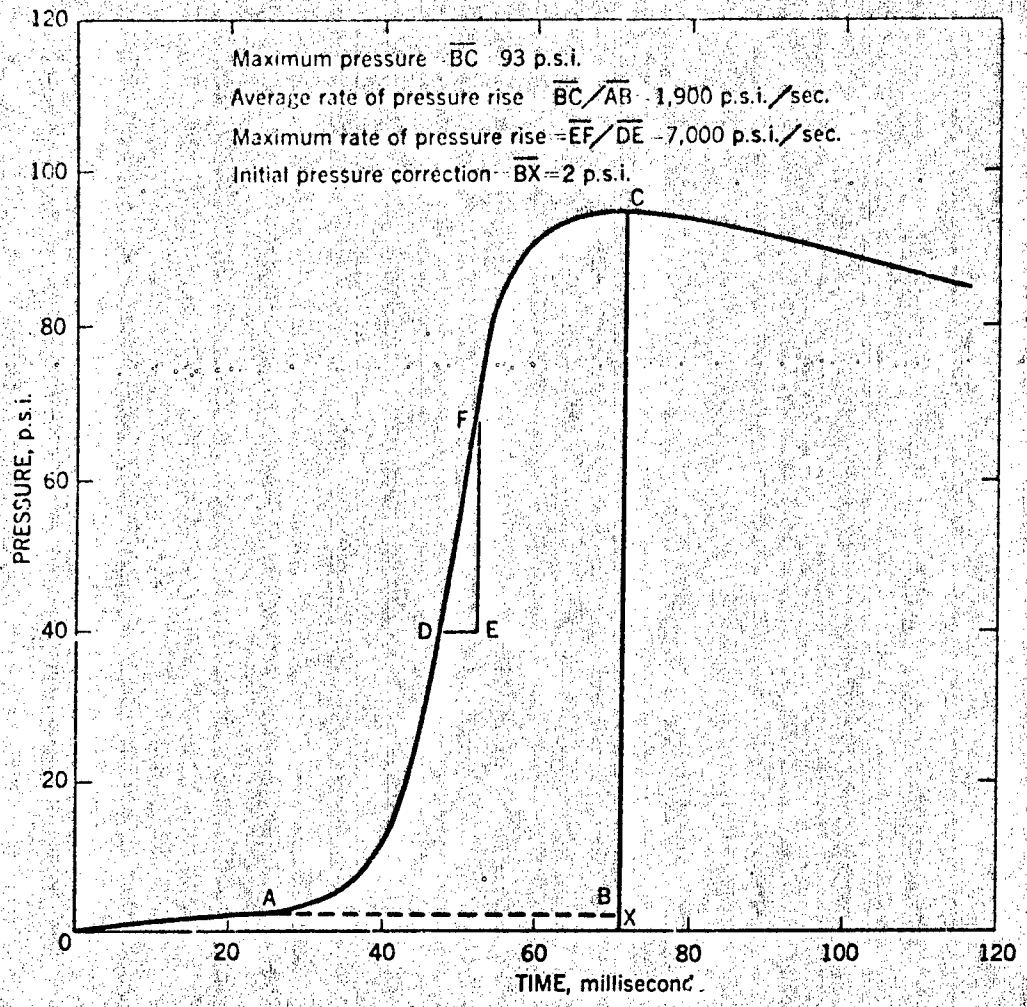


FIGURE 7. - Pressure-Time Record of a Cornstarch Explosion in the Hartmann Apparatus.

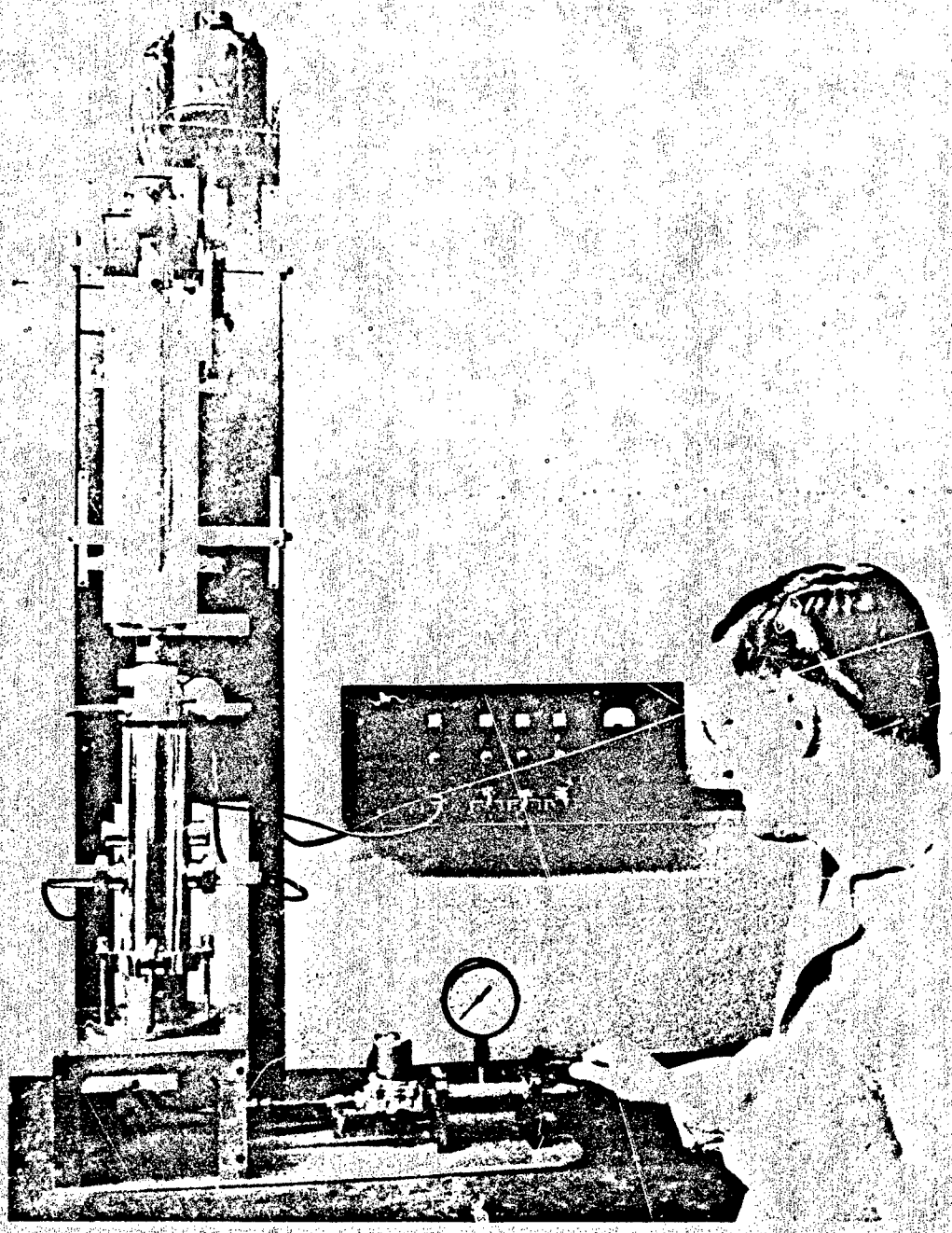


FIGURE 6. - Hartmann Apparatus for Determining Pressure and Rates of Pressure Rise in a Dust Explosion.

SPECIAL PROCESS INSTRUCTION PE 73 - 96

Thiokol / HUNTSVILLE DIVISION
Huntsville, Alabama
A DIVISION OF THIOKOL CHEMICAL CORPORATION

PROPELLANT:	MOTOR:
TP- N/A MIX	TX- N/A NO.
OTC NO. 9186 73 Feb 14	INSPECTION: DATE
TASK RELEASE 460-84-01	
OP CHANGE: YES ( ) NO (X)	YES ( ) NO (X) 73 Feb 13

ORIGINATOR	R&D OPP SUPERVISOR	R&D SECTION CHIEF	MFG. SECTION CHIEF
S. L. Vance	E. J. Schreck	J. L. Murphy, Jr.	

DETONATION TEST ON UFAP SHIPPING CONTAINER

INTRODUCTION

This document will be used as a general operating procedure for conducting at least one full-scale detonation test, Type A of Chapter 4, TB700-2, on a 5 gallon shipping container of UFAP.

This test, as spelled out in TB700-2, involves filling one shipping container with a live charge of material, and arming it with an initiator positioned about 4 inches above the bottom of the container. For this test, the initiator will be an electric squib and two ounces of black powder. The primed, live container will then be positioned on a wooden pallet and surrounded with six each containers loaded with sand. Steel packing strapping will be applied to bundle the containers together. This bundle will then be topped with seven more containers all filled with and and strapped together in a similar manner. Additional straps will be applied to hold the top layer in place. The palletized assembly will then be transported to the test area, positioned, photographed, then, after the area is cleared, initiated. Photographs taken after the test will be used to indicate the extent of damage that occurs.

As an additional means of determining whether the live test material undergoes a high order detonation, Primacord will be inserted in the UFAP inside the primed container, and run out to a remotely positioned lead witness plate. Undamaged Primacord, and no damage to the lead plate will be evidence that any reaction that occurs in the UFAP test material did not achieve a high order detonation, regardless of the damage that might occur from explosion or low-velocity-detonation of the material.

SAFETY REQUIREMENTS

1. Testing (initiation) will not be done on rainy days or during approach or progress of any electrical storm.
2. Radio transmitters will not be operated in the area when the assembled, palletized test charge is present.

3. Ensure initiator leads are shorted together prior to handling the initiator itself or any assembly containing the initiator.

4. Persons handling explosives shall wear flame-resistant lab coats or coveralls, conductive-soled shoes (or "legstats"), and approved eye protection.

5. One person will be responsible for arming the device (connecting it to the firing circuit) just prior to the test. This may be done only after all other persons have left the area.

6. The firing circuit will be completely insulated from the ground or other conductors such as bare wires, rails, pipes or other paths of stray currents.

7. The firing circuit must be shorted at the bunker at all times except when ready to initiate the test.

8. In the event of a misfire, wait 15 minutes before approching the location of the test assembly.

9. No person or vehicle shall be between the bunker and the test site during initiation of the test.

PROCEDURE

The test assembly is to be prepared in Building 7603 and transported from there to the test pad at the Burn-out Pit (Bldg. 7336-7338) for testing.

The arrangement of the primed shipping container, and the final assembly of the test package is as shown in Figure 1. Assembly will generally be as follows:

1. Drill a hole in the side of the five gallon pail just under the rolling ring, and install a rubber grommet in the hole. If necessary, pot the grommet in place with ambient cure adhesive.

2. Place two conductive plastic liners, one inside the other inside the pail.

3. Obtain a thoroughly blended mixture consisting of 10.00 lbs. FDB-7 freeze dried UFAP and 4.29 lbs. of Sorbeads.

4. Charge enough UFAP/Sorbead mixture to the inside bag to fill to a four inch depth.

5. Place the prepared initiator charge (electric squib a with 2 ounces of black powder) on the surface of the material in the center of the pail. Run the leads horizontally to the pail wall (where the grommeted hole is) then straight up.

6. Cover the initiator with UFAP/Sorbeads to hold it and the leads in place. Finish filling the container with the mixture.

7. Insert the initiator leads through the grommeted hole, from the inside, and pull through until only 5 or 6 inches of loose wire remains inside the pail.

8. Carefully insert the free end of the Primacord through the grommeted hole, from the outside, taking care not to move or otherwise disturb the initiator wires. Insert until the end is within one inch of the opposite side of the pail.

9. Dig a furrow in the UFAP/Sorbeads charge, at least one inch deep, and bury the Primacord under the material. Bend the Primacord just inside the grommeted hole as needed. See Figure 1.

10. Gather the neck of the inner conductive bag around the Primacord and initiator leads and apply a tape wrap to seal the bag opening around them. Repeaf with the outer bag.

11. Place about 100 gms. of additional Sorbeads inside the pail on top of the bags, then install the pail lid and crimp down the latching ears.

12. Place the primed container in the center of a wood pallet, and surround it with six containers each filled with 65 lbs. of dry sand (lids installed and crimpted). Position these outer containers so the Primacord and initiator leads come out between two of them. Insert small wood spacers between these two pails, one above the leads and Primacord, and one below.

13. Apply one wrap of 5/8 wide steel strap around the outside of the sand filled containers to tightly bundle them together. Recheck the Primacord and initiator leads just prior to this to assure they are still protected and properly positioned.

14. Place one sand-filled container (each containing 65 lbs. of dry sand and with lids installed and crimped) on top of each of the seven bottom containers. Apply a steel strap around the outside of them to tightly bundle them together.

15. Apply strapping, as shown in Figure 1, to hold the top bundle against the bottom one, and to hold the bundles against the pallet.

16. Tape the initiator leads, and the free end of the Primacord, with its lead witness plate (5" x 5" x 3/16"), to the outside of the assembly where they won't interfere with handling and transportation.

Testing of the assembly will generally be as follows:

17. Transport the test package to the test area (Snake-Pit) and position on the gravel pad just above the card gap test area.

18. Remove all transportation vehicles (truck and fork lift) from the area.

19. Unroll the Primacord and place the witness plate on the ground as far from the test package as possible. Place sand bags over the witness plate and Primacord to protect it and prevent it from being lost.

20. Unroll the initiator leads and extend out as far as possible toward the firing circuit clips.

21. Take photographs of the test package in its test position.

22. Clear the area except for the person to arm the device.

23. Verify that both ends of the firing circuit are shorted and the initiator leads are shorted.

24. Unshort the firing circuit leads at the test site and spread apart as far as possible. Unshort the initiator leads and connect to the firing circuit leads. Make certain the leads are adequately protected against shorting out by allowing sufficient air space or by taping.

25. Return to the bunker and ensure the test area is completely clear of personnel and vehicles.

26. Sound warning horn.

27. Initiate the test by unshorting the firing circuit (at the bunker end) and touching the bare leads across a 24 to 36 volt battery circuit.

28. Wait at least 5 minutes, then (if test initiated) sound "all clear".

29. Take photographs of the test site after the test, and make written description of condition. Locate and photograph condition of witness plate.

30. If crater exists, measure and record its dimensions.

31. Record any other pertinent information concerning explosive effects.

32. Unreacted Primacord should be gathered up and disposed of later by initiating with a blasting cap, following the general procedure for lead column or Card Gap Tests (Procedure No. 00000-1338-401 or 00000-1355-402, respectively).

STIPULATIONS:

1. Radio transmission silence in the Plant will be in effect during the transportation period.
2. Emergency vehicle will preceed the transport vehicle along the route.
3. Transportation will be performed during normal working hours.

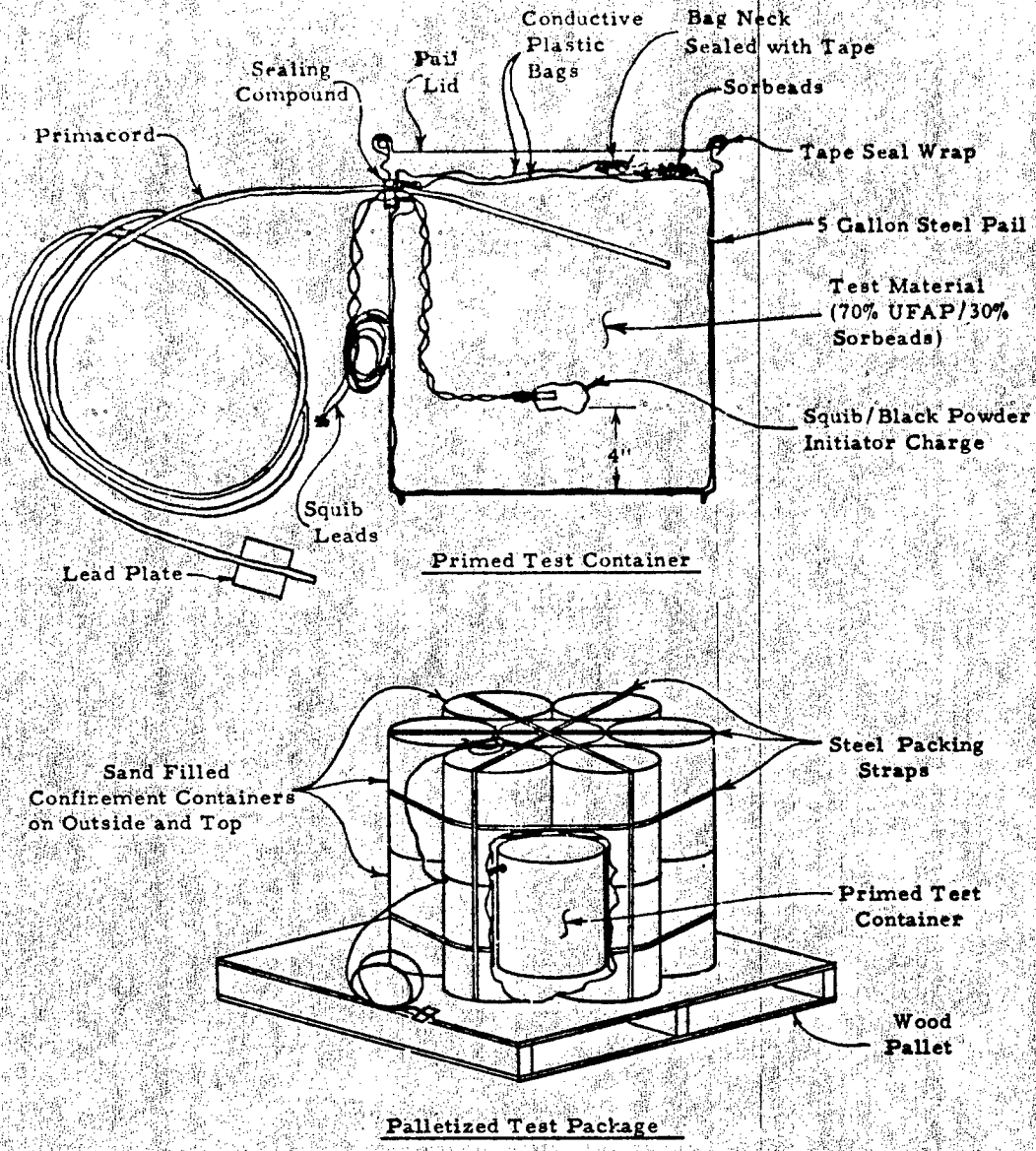


Figure 1 - Test Specimen Assembly for Shipping Container Detonation Tests

SPECIAL PROCESS INSTRUCTION PE 73 — 106

Thiokol / HUNTSVILLE DIVISION Huntsville, Alabama A DIVISION OF THIOKOL CHEMICAL CORPORATION	PROPELLANT: N/A TP- MIX	MOTOR: N/A TX- NO.
	OTC NO. 9197 2/14/73 TASK RELEASE 460-84-01 OP CHANGE: YES ( ) NO (x)	INSPECTION: YES ( ) NO (x)
		DATE: 73 Feb 16

ORIGINATOR	R&D GRP SUPERVISOR	R&D SECTION CHIEF	MFG. SECTION CHIEF
[illegible]	[illegible]	[illegible]	[illegible]

DETONATION TESTS ON UFAP SHIPPING CONTAINERS

Introduction

This document will be used as a general operating procedure for conducting full-scale detonation tests, Type A of Chapter 4, TB 700-2, on 5 gallon shipping containers of UFAP. Up to five tests will be made.

This test, as spelled out in TB 700-2, involves filling one shipping container with a live charge of material, and arming it with an initiator positioned about 4 inches above the bottom of the container For this test, the initiator will be an electric squib and two ounces of black powder. The primed, live container will then be positioned on a wooden pallet and surrounded with six each containers loaded with sand. Stee' packing strapping will be applied to bundle the containers together. This bundle will then be topped with seven more containers all filled with sand and strapped together in a similar manner. Additional straps will be applied to hold the top layer in place. The palletized assembly will then be transported to the test area, positioned, photographed, then, after the area is cleared, initiated. Photographs taken after the test will be used to indicate the extent of damage that occurs.

As an additional means of determining whether the live test material undergoes a high order detonation, Primacord will be inserted in the UFAP inside the primed container, and run out to a remotely positioned lead witness plate. Undamaged Primacord, and no damage to the lead plate will be evidence that any reaction that occurs in the UFAP test material did not achieve a high order detonation, regardless of the damage that might occur from explosion or low-velocity-detonation of the material.

Safety Requirements

1. Testing (initiation) will not be done on rainy days or during approach or progress of any electrical storm.
2. Radio transmitters will not be operated in the area when the assembled, palletized test charge is present.
3. Ensure initiator leads are shorted together prior to handling the initiator itself or any assembly containing the initiator.

4. Persons handling explosives shall wear flame-resistant lab coats or coveralls, conductive-soled shoes (or "legstats"), and approved eye protection.

5. One person will be responsible for arming the device (connecting it to the firing circuit) just prior to the test. This may be done only after all other persons have left the area.

6. The firing circuit will be completely insulated from the ground or other conductors such as bare wires, rails, pipes or other paths of stray currents.

7. The firing circuit must be shorted at the bunker at all times except when ready to initiate the test.

8. In the event of a misfire, wait 15 minutes before approaching the location of the test assembly.

9. No person or vehicle shall be between the bunker and the test site during initiation of the test.

Procedure

The test assembly may either be prepared in Building 7603 and transported from there to the test pad at the Burn-out Pit (Bldg. 7336-7338) for testing or assembled at the test pad. The UFAP containers must, however, be assembled at 7603 inside conditioned bay.

The arrangement of the primed shipping container, and the final assembly of the test package is as shown in Figure 1. Assembly will generally be as follows:

1. Drill a hole in the side of the five gallon pail just under the rolling ring, and install a rubber grommet in the hole. If necessary, pot the grommet in place with ambient cure adhesive.

2. Place two conductive plastic liners, one inside the other inside the pail.

3. Obtain a thoroughly blended mixture consisting of 10.00 lbs. UFAP and 4.29 lbs. of Sorbeads.

4. Charge enough UFAP/Sorbead mixture to the inside bag to fill to a four inch depth.

5. Place the prepared initiator charge (electric squib with 2 ounces of black powder) on the surface of the material in the center of the pail. Run the leads horizontally to the pail wall (where the grommeted hole is) then straight up. Shorted initiator leads should be grounded at all times per "Electrical Grounding."

6. Cover the initiator with UFAP/Sorbeads to hold it and the leads in place. Finish filling the container with the mixture.

7. Insert the initiator leads through the grommeted hole, from the inside, and pull through until only 5 or 6 inches of loose wire remains inside the pail.

8. Carefully insert the free end of the Primacord through the grommeted hole, from the outside, taking care not to move or otherwise disturb the initiator wires. Insert until the end is within one inch of the opposite side of the pail.

9. Dig a furrow in the UFAP/Sorbeads charge, at least one inch deep, and bury the Primacord under the material. Bend the Primacord just inside the grommeted hole as needed. See Figure 1.

10. Gather the neck of the inner conductive bag around the Primacord and initiator leads and apply a tape wrap to seal the bag opening around them. Repeat with the outer bag.

11. Place about 100 gms. of additional Sorbeads inside the pail on top of the bags, then install the pail lid and crimp down the latching ears.

12. Remove the paint from an area about 2" x 2" just below the grommet. Wrap the Primacord/initiator leads with aluminum foil to a point about 18" from the grommet, and ensuring the wrap is at least three layers thick, secure the wrap with tape. Tape a bonding strip at least three layers thick from the Primacord/lead wire wrap to the exposed pail wall. Secure with tape to provide a grounded connection from the wrapped leads to the metal can.

13. Coil the remaining Primacord and initiator leads and wrap in aluminum foil to completely enclose them. Fold all ends at least three times and tape. Wrap should be at least three layers thick, and should extend to and be taped into electrical contact with the wrap applied in Step 12, above.

14. The remaining steps may either be performed (up through Step 19) in 7603, or at the test site.

15. Place the primed container in the center of a wood pallet, and surround it with six containers each filled with 65 lbs. of dry sand (lids installed and crimped). Position these outer containers so the Primacord and initiator leads come out between two of them. Insert small wood spacers between these two pails, one above the leads and Primacord, and one below.

16. Apply one wrap of 5/8 wide steel strap around the outside of the sand filled containers to tightly bundle them together. Recheck the Primacord and initiator leads just prior to this to assure they are still protected and properly positioned.

17. Place one sand-filled container (each containing 65 lbs. of dry sand and with lids installed and crimped) on top of each of the seven bottom containers. Apply a steel strap around the outside of them to tightly bundle them together.

18. Apply strapping, as shown in Figure 1, to hold the top bundle against the bottom one, and to hold the bundles against the pallet.

19. Tape the initiator leads, and the free end of the Primacord, with its lead witness plate (5" x 5" x 3/16"), to the outside of the assembly where they won't interfere with handling and transportation.

Testing of the assembly will generally be as follows:

20. Transport the test package to the test area (Snake-Pit) and position on the gravel pad just above the card gap test area.

21. Remove all transportation vehicles (truck and fork lift) from the area.

22. Remove the aluminum foil wrap from the coiled Primacord and leads. Unroll the Primacord and place the witness plate on the ground as far from the test package as possible. Place sand bags over the witness plate and Primacord to protect it and prevent it from being lost.

23. Unroll the initiator leads and extend out as far as possible toward the firing circuit clips.

24. Take photographs of the test package in its test position.

25. Clear the area except for the person to arm the device.

26. Verify that both ends of the firing circuit are shorted and the initiator leads are shorted.

27. Unshort the firing circuit leads at the test site and spread apart as far as possible. Unshort the initiator leads and connect to the firing circuit leads. Make certain the leads are adequately protected against shorting out by allowing sufficient air space or by taping.

28. Return to the bunker and ensure the test area is completely clear of personnel and vehicles.

29. Sound warning horn.

30. Initiate the test by unshorting the firing circuit (at the bunker end) and touching the bare leads across a 24 to 36 volt battery circuit.

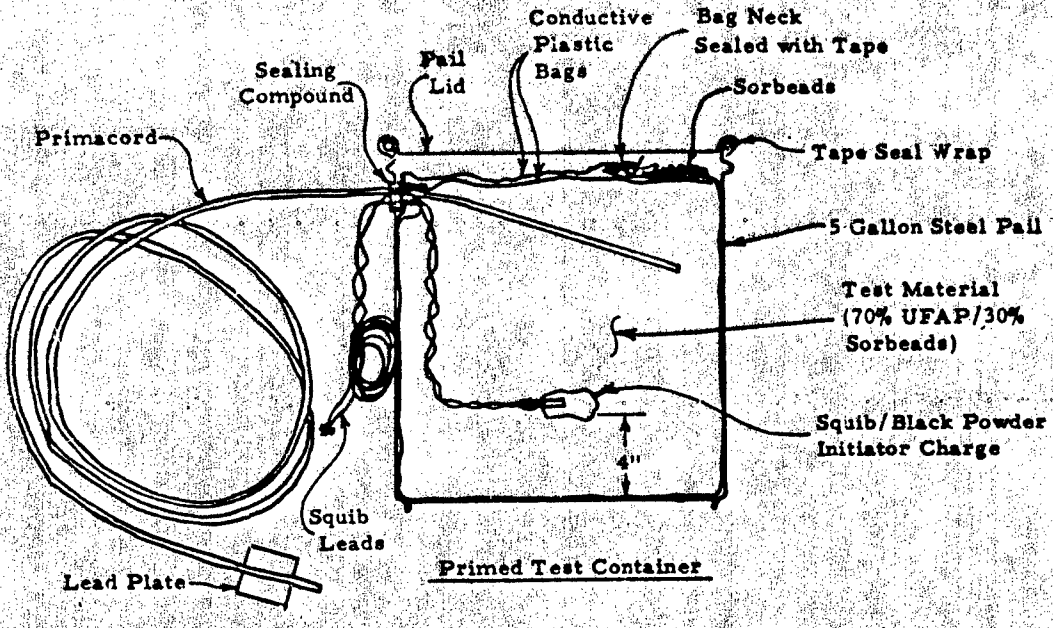
31. Wait at least 5 minutes, then (if test initiated) sound "all clear."

32. Take photographs of the test site after the test, and make written description of condition. Locate and photograph condition of witness plate.

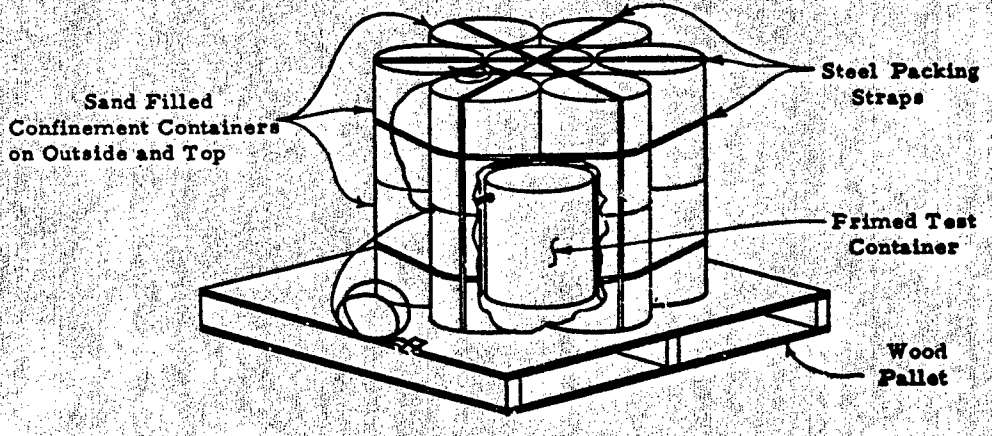
33. If crater exists, measure and record its dimensions.

34. Record any other pertinent information concerning explosive effects.

35. Unreacted Primacord should be gathered up and disposed of later by initiating with a blasting cap, following the general procedure for lead column or Card Gap Tests (Procedure No. 00000-1338-401 or 00000-1355-402, respectively).



Primed Test Container



Palletized Test Package

Test Specimen Assembly for Shipping Container Detonation Tests

SPECIAL PROCESS INSTRUCTION

PE 73 — 108

Thiokol / HUNTSVILLE DIVISION
Huntsville, Alabama
A DIVISION OF THIOKOL CHEMICAL CORPORATION

PROPELLANT: N/A TP- MIX	MOTOR: N/A TX- NO.	
OTC NO. 9201	INSPECTION	DATE
TASK RELEASE 460-84-01		
DP CHANGE: YES ( ) NO (X)	YES ( ) NO (X)	73 Feb 19

ORIGINATOR	R&D GRP SUPERVISOR	R&D SECTION CHIEF	MFG. SECTION CHIEF
[signature]	[signature]	[signature]	N/A

DETONATION TESTS ON UFAP SHIPPING CONTAINERS

Introduction

This document will be used as a general operating procedure for conducting one full-scale detonation test, Type B of Chapter 4, TB 700-2, on two 5 gallon shipping containers of UFAP.

This test, as spelled out in TB 700-2, involves filling one shipping container with a live charge of material, and arming it with an initiator positioned about 4 inches above the bottom of the container. For this test, the initiator will be an electric squib and two ounces of black powder. A second container will be charged with live material, but will not have an initiator. The primed, live container will then be positioned on a wooden pallet, and the unprimed container placed next to it. The two live containers will then be completely surrounded with inert containers, loaded with sand. Steel packing strapping will be applied to bundle the containers together. This bundle will then be topped with ten more containers all filled with sand and strapped together in a similar manner. Additional straps will be applied to hold the top layer in place. The palletized assembly will then be transported to the test area, positioned, photographed, then, after the area is cleared, initiated. Photographs taken after the test will be used to indicate the extent of damage that occurs.

As an additional means of determining whether the live test material undergoes a high order detonation, Primacord will be inserted in the UFAP inside the primed container, and run out to a remotely positioned lead witness plate. Undamaged Primacord, and no damage to the lead plate will be evidence that any reaction that occurs in the UFAP test material did not achieve a high order detonation, regardless of the damage that might occur from explosion or low-velocity-detonation of the material.

The primary purpose of this test is to determine if the primed (donor) container, when initiated, will cause the unprimed (or receptor) container to function (explode).

Safety Requirements

1. Testing (initiation) will not be done on rainy days or during approach or progress of any electrical storm.

2. Radio transmitters will not be operated in the area when the assembled, palletized test charge is present.

3. Ensure initiator leads are shorted together prior to handling the initiator itself or any assembly containing the initiator.

4. Persons handling explosives shall wear flame-resistant lab coats or coveralls, conductive-soled shoes (or "legstats"), and approved eye protection.

5. One person will be responsible for arming the device (connecting it to the firing circuit) just prior to the test. This may be done only after all other persons have left the area.

6. The firing circuit will be completely insulated from the ground or other conductors such as bare wires, rails, pipes or other paths of stray currents.

7. The firing circuit must be shorted at the bunker at all times except when ready to initiate the test.

8. In the event of a misfire, wait 15 minutes before approaching the location of the test assembly.

9. No person or vehicle shall be between the bunker and the test site during initiation of the test.

Procedure

The test assembly may either be prepared in Building 7603 and transported from there to the test pad at the Burn-out Pit (Bldg 7336-7338) for testing or assembled at the test pad. The UFAP containers must, however, be assembled at 7603 inside conditioned bay.

The arrangement of the primed shipping container, and the final assembly of the test package is as shown in Figure 1. Assembly will generally be as follows:

1. Drill a hole in the side of the five gallon pail just under the rolling ring, and install a rubber grommet in the hole. If necessary, pot the grommet in place with ambient cure adhesive.

2. Place two conductive plastic liners, one inside the other inside the pail.

3. Obtain a thoroughly blended mixture consisting of 10.00 lbs. UFAP and 4.29 lbs. of Sorbeads.

4. Charge enough UFAP/Sorbead mixture to the inside bag to fill to a four inch depth.

5. Place the prepared initiator charge (electric squib with 2 ounces of black powder) on the surface of the material in the center of the pail. Run the leads horizontally to the pail wall (where the grommeted hole is) then straight up. Shorted initiator leads should be grounded at all times per "Electrical Grounding."

6. Cover the initiator with UFAP/Sorbeads to hold it and the leads in place. Finish filling the container with the mixture.

7. Insert the initiator leads through the grommeted hole, from the inside, and pull through until only 5 or 6 inches of loose wire remains inside the pail.

8. Carefully insert the free end of the Primacord through the grommeted hole, from the outside, taking care not to move or otherwise disturb the initiator wires. Insert until the end is within one inch of the opposite side of the pail.

9. Dig a furrow in the UFAP/Sorbeads charge, at least one inch deep, and bury the Primacord under the material. Bend the Primacord just inside the grommeted hole as needed. See Figure 1.

10. Gather the neck of the inner conductive bag around the Primacord and initiator leads and apply a tape wrap to seal the bag opening around them. Repeat with the outer bag.

11. Place about 100 gms. of additional Sorbeads inside the pail on top of the bags, then install the pail lid and crimp down the latching ears.

12. Remove the paint from an area about 2" x 2" just below the grommet. Wrap the Primacord/initiator leads with aluminum foil to a point about 18" from the grommet, and ensuring the wrap is at least three layers thick, secure the wrap with tape. Tape a bonding strip at least three layers thick from the Primacord/lead wire wrap to the exposed pail wall. Secure with tape to provide a grounded connection from the wrapped leads to the metal can.

13. Coil the remaining Primacord and initiator leads and wrap in aluminum foil to completely enclose them. Fold all ends at least three times and tape. Wrap should be at least three layers thick, and should extend to and be taped into electrical contact with the wrap applied in Step 12, above.

14. The unprimed container will be prepared in the same manner, except that no initiator will be used, so Steps 5, 7, 12, and 13 may be eliminated. Coil the Primacord and tape up.

15. The remaining steps may either be performed (up through Step 19) in 7603, or at the test site.

16. Place the Primed (donor) container near the center of a wood shipping pallet, and the live receptor container next to it. Surround the containers with eight containers each filled with approximately 65 lbs. of dry sand (with the lids installed and crimped). Position these outer containers so the Primacord and initiator leads come out between them. Insert small wood spacers between these pails, one above the leads and Primacord, and one below.

17. Apply one wrap of 5/8 wide steel strap around the outside of the sand filled containers to tightly bundle them together. Recheck the Primacord and initiator leads just prior to this to assure they are still protected and properly positioned.

18. Place one sand-filled container (each containing 65 lbs. of dry sand and with lids installed and crimped) on top of each of the ten bottom containers. Apply a steel strap around the outside of them to tightly bundle them together.

19. Apply strapping, as shown in Figure 1, to hold the top bundle against the bottom one, and to hold the bundles against the pallet.

20. Tape the initiator leads, and the free end of the Primacord, with its lead witness plate (5" x 5" x 3/16"), to the outside of the assembly where they wont interfere with handling and transportation.

21. Testing of the assembly will generally be as follows:

21. Transport the test package to the test area (Snake-Pit) and position on the gravel pad just above the card gap test area.

22. Remove all transportation vehicles (truck and fork lift) from the area.

23. Remove the aluminum foil wrap from the coiled Primacord and leads. Unroll the Primacord and place the witness plate on the ground as far from the test package as possible. Place sand bags over the witness plate and Primacord to protect it and prevent it from being lost.

24. Unroll the initiator leads and extend out as far as possible toward the firing circuit clips.

25. Take photographs of the test package in its test position.

26. Clear the area except for the person to arm the device.

27. Verify that both ends of the firing circuit are shorted and the initiator leads are shorted.

28. Unshort the firing circuit leads at the test site and spread apart as far as possible. Unshort the initiator leads and connect to the firing circuit leads. Make certain the leads are adequately protected against shorting out by allowing sufficient air space or by taping.

29. Return to the bunker and ensure the test area is completely clear of personnel and vehicles.

30. Sound warning horn.

31. Initiate the test by unshorting the firing circuit (at the bunker end) and touching the bare leads across a 24 to 36 volt battery circuit.

32. Wait at least 5 minutes, then (if test initiated) sound "all clear."

33. Take photographs of the test site after the test, and make written description of condition. Locate and photograph condition of witness plate.

34. If crater exists, measure and record its dimensions.

35. Record any other pertinent information concerning explosive effects.

36. Unreacted Primacord should be gathered up and disposed of later by initiating with a blasting cap, following the general procedure for lead column or Card Cap Tests (Procedure No. 00000-1338-401 or 00000-1355-402, respectively).

NOTE: When transferring UFAP from one container to another, the actual UFAP containers must be grounded together or to a common ground per procedure "Electrical Grounding."

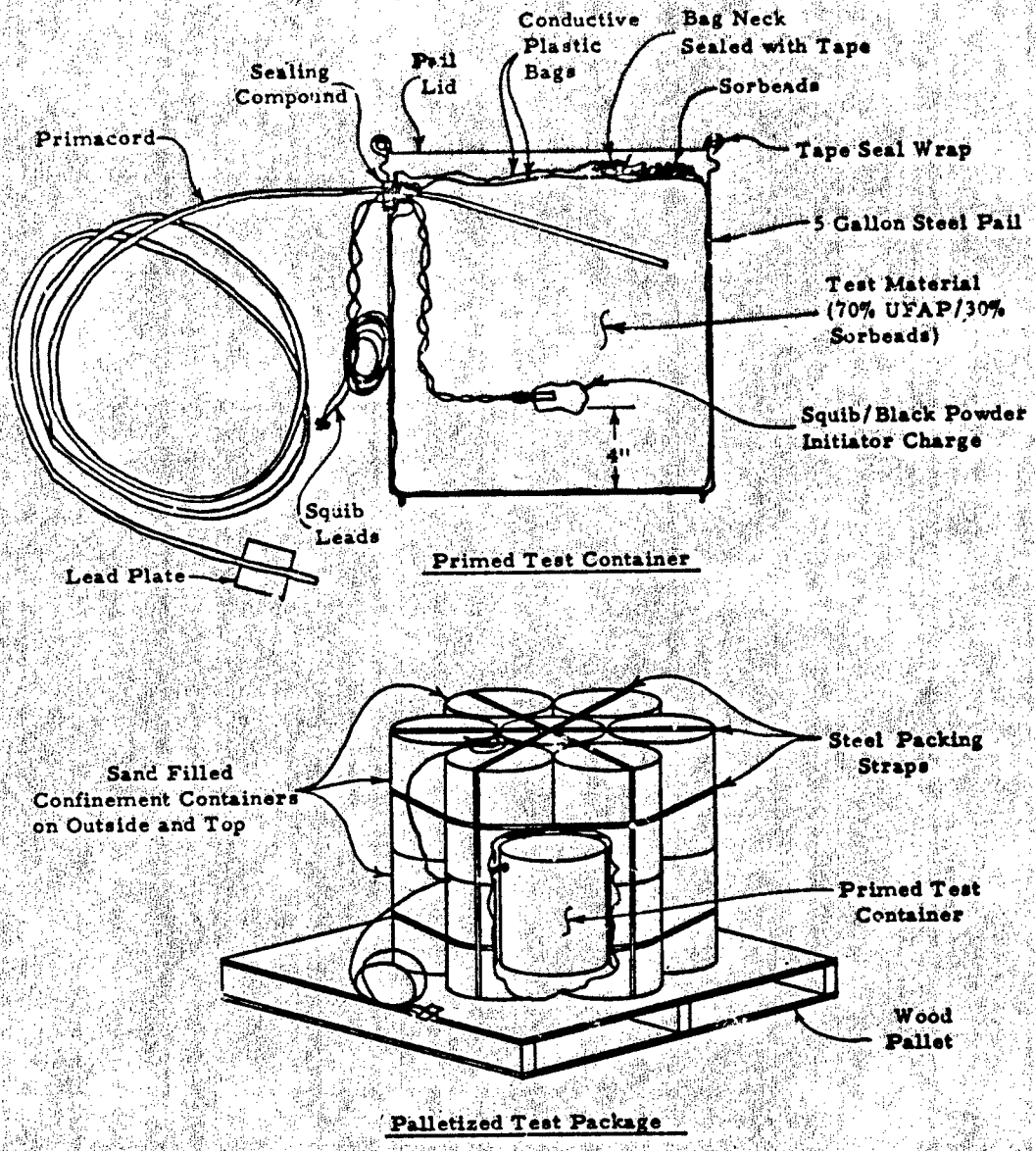


Figure 1 - Test Specimen Assembly for Shipping Container Detonation Tests

SPECIAL PROCESS INSTRUCTION PE 73 -110

Thiokol / HUNTSVILLE DIVISION
Huntsville, Alabama
A DIVISION OF THIOKOL CHEMICAL CORPORATION

PROPELLANT:	MOTOR:
TP- N/A MIX	TX- N/A NO.
OTC NO. 9202	INSPECTION: DATE:
TASK RELEASE 460-84-01	
OP CHANGE: YES ( ) NO ( )	YES ( ) NO ( ) 73 Feb 19

ORIGINATOR	R&D GRP SUPERVISOR	R&D SECTION CHIEF	MFG. SECTION CHIEF
[signature]	ESchreck	[signature]	N/A

EXTERNAL HEAT TEST ON UFAP SHIPPING CONTAINER

This test is to be performed to simulate a condition where shipping containers loaded with UFAP materials are completely enveloped in a hot fire. For this specific test, six fully loaded shipping containers will be banded together in a single layer, two by three on a side, with steel packing straps. The package will be placed on top of a prefabricated wooden crib such that the containers are approximately 30 inches above the ground. The interior of the crib will be filled with combustible material, such as scrap lumber and sawdust. This material will then be saturated with approximately 50 gallons of JP-4 or diesel fuel. Ignition of the combustible material will be accomplished from a remote location by way of two initiator packages, one each on opposite sides of the crib. The initiator packages will consist of an electric squib and two ounces of smokeless powder, suitably packaged for use, or other acceptable ignition means, as supplied by the Igniter Section. Still photographs will be taken before and after the test to show the results. Physical measurements of craters, missile locations, etc. will be made as needed.

The test conditions and requirements are as specified in Chapter 4 of DSAR 8220.1 (TB 700-2), Explosives Hazard Classification Procedures, and specifically Part 4 (minimum test criteria for bulk propellant) of Table 4, on page 15 of that document.

Preparation and initial assembly of the test components will be performed at Thiokol, per this procedure. Final assembly and testing will be accomplished at the Arsenal Demolition Area, under the direction and control of the range operating personnel of the Explosives Division, MICOM. Thiokol personnel working at the Demolition Area shall be at all times regulated by the safety regulations and limitations specified by the Explosives Division.

Preparation of Shipping Container Test Package

1. Prepare six each shipping containers of UFAP per the following:
   a. To a clean, dry 5 gallon steel pail, install two conductive plastic bags, one inside the other, as internal liners.

b. Charge a well blended mixture, consisting of 10.00 lbs. of freeze-dried UFAP of nominal 0.5 micron WMD and 4.29 lbs. Sorbeads (silica gel desiccant), to the inner plastic liner.

c. Seal the neck of the inner liner, taking care to exclude as much air as possible, with tape. Seal the outer liner in the same manner.

d. Put about 100 grams of Sorbeads inside the pail, loose on top of the plastic pail liners.

e. Install the pail lid and crimp down the lid ears with plier or other tool, to provide a tight seal.

f. Apply a single wrap of paper or plastic tape over the outside of the lid/pail joint.

g. Label the container appropriately.

2. Assemble the six loaded containers in a rectangular array, two by three, and band together with 5/8 inch wide steel packing straps. One strap should surround the array horizontally, while other straps should be applied vertically to hold the columns together.

3. Obtain a wooden crib, constructed of 2 x 4 lumber, of such dimensions to support the banded package approximately 30 inches above the floor.

4. Obtain a large quantity of scrap lumber - 2 x 4's, 2 x 2's, 2 x 1's, etc. and a cubic foot or more of oven-dried sawdust.

5. Obtain a solvent drum (55 gallon) filled with either JP-4 fuel or diesel fuel oil, or similar combustible fuel oil. The drum must be equipped with an approved dispensing valve and breather vent.

6. Arrange for two initiators to be prepared by the Igniter Section, each to be equivalent to an electric squib and two ounces of smokeless powder.

On the day scheduled for the test, perform the following operations:

7. Transport all the materials for the test assembly to the Arsenal Demolition Area.

NOTE: Three individual trucks or truck loads must be used, as the initiators may not be carried on the same vehicle or load with either the UFAP containers or the fuel oil. Likewise, the fuel oil may not be carried on the same vehicle or load as the UFAP containers. Vehicles entering the Demolition Area must stop

at Building 8404 and obtain permission to enter the area and to have the test site specified.

8. Assemble the test components, on a site designated by the range operating personnel. Figure 1 gives the proper assembly.

9. When all the wood and sawdust have been charged to the crib, obtain before-test photographs of the assembly.

10. Using five gallon buckets or pails, carefully transfer the fuel oil from the solvent drum and pour into the crib to saturate the scrap lumber and sawdust. It may be necessary to trench around the base of the crib to contain any excess oil.

NOTE: Ground the transfer buckets to the oil drum, per "Electrical Grounding", when discharging the oil to the buckets.

11. Position one initiator within the crib on one side, and weight it down with some of the lumber. Position the other in a similar way on the opposite side. Extend the lead wires out toward the firing circuit line, as directed by the range operating personnel.

NOTE: The initiator leads must remain shorted at all times up to the point of actual connection to the firing circuit.

12. Obtain one last before-test photograph, showing the location of the initiators and the fuel oil saturating the crib.

13. All Thiokol personnel must retire at this time to a suitable observation area, as designated by the range operating personnel, for the remainder of the test. Range operating personnel will perform the remaining functions, per their own standing operating procedures, to initiate the test.

14. When the range operating personnel sound the "All Clear" after the test, return to the test site and inspect the after-test conditions.

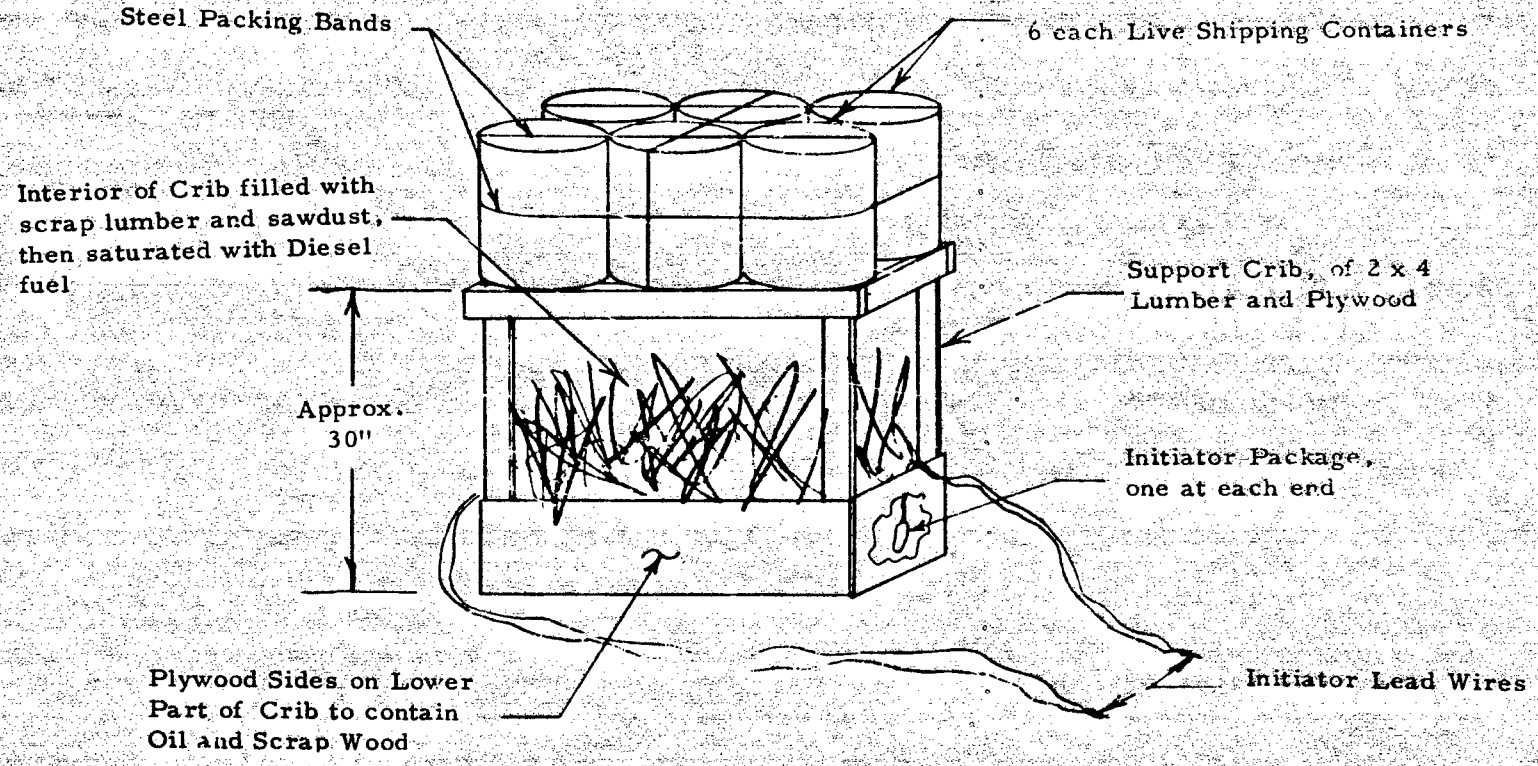
15. Obtain after-test photos to record the extent of damage that occurred, if any.

16. If any cratering occurred, determine the physical dimensions and prepare a sketch giving the details.

17. If any objects are thrown outside the area covered by photographs, prepare a missile map describing the distance from the test site, the weight of the object, and the description of the object itself.

18. Aid range operating personnel in cleaning up test site, as needed.

NOTE: When transferring UFAP from one container to another, the actual UFAP containers must be grounded together or to a common ground per procedure "Electrical Grounding."



ASSEMBLY OF COMPONENTS FOR EXTERNAL HEAT TEST (TEST "C")
Figure 1. PE 73-110

C 1, TB 700-2
NAVORDINST 8020.3 CH-1
TO 11A-1-47A
CH 1, DSAR 8220.1

DEPARTMENT OF THE ARMY TECHNICAL BULLETIN
DEPARTMENT OF THE NAVY PUBLICATION
DEPARTMENT OF THE AIR FORCE TECHNICAL ORDER
DEFENSE SUPPLY AGENCY REGULATION

EXPLOSIVES HAZARD CLASSIFICATION PROCEDURES

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Departments of the Army, the Navy, and the Air Force, and
Defense Supply Agency, Washington, D.C.
22 January 1968

TB 700-2/NAVORDINST 8020.3/TO 11A-1-47, DSAR 8220.1, 19 May 1967, is changed as follows:

1. All references to the Interstate Commerce Commission (ICC) should be changed to Department of Transportation (DOT).

2. *Page 11.* Paragraph 3-13*a*(2) is redesignated 3-13*a*(2)(*a*).

3. Paragraph 3-13*a*(2)(*b*) is added as follows: Ignition and unconfined burning test produces a detonation.

4. Paragraph 3-13*a*(3)(*c*) is superseded as follows: Ignition and unconfined burning test did not result in an explosion.

SMEA	1	[illegible]
SAL	1	SMME
SB	1	SMAM, Bailey
SD		SHPS
SO	2	MOT
SOA		SMAT
SEA	1	SS 2, Comm Off
SMC	1	SCB
SME	1	SSC 1
SMEB	1	SCK
SMEA	1	SSP
SMMA	3	CSS
SMMDA		SSSL
SMME		[illegible]

BM-RES-1 BM-HHS-1

TAGO 762A—Jan. [illegible]—68

*TB 700-2
*NAVORDINST 8020.3
*TO 11A-1-47
DSAR 4220.1

DEPARTMENT OF THE ARMY TECHNICAL BULLETIN
DEPARTMENT OF THE NAVY PUBLICATION
DEPARTMENT OF THE AIR FORCE TECHNICAL ORDER
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EXPLOSIVES HAZARD CLASSIFICATION PROCEDURES

Departments of the Army, the Navy, and the Air Force, and
Defense Supply Agency, Washington, D.C.
19 May 1967

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*This publication supersedes TB 700-2/ BUWEPINST 8020.3/TO 11A-1-47, 31 July 1962.

CHAPTER 3

MINIMUM TEST CRITERIA FOR BULK EXPLOSIVE COMPOSITIONS AND SOLID PROPELLANT COMPOSITIONS

3-1. Introduction. *a.* Tests in this chapter are intended to develop data on the stability and sensitivity of new compositions of bulk explosives and solid propellants. Such data is required in order to determine that these compositions are safe to handle, transport, and store.

b. These tests are conducted on laboratory samples of material. The sample weights or dimensions listed are the minimum upon which conclusions may be drawn; however, it is suggested that smaller samples be tested to give preliminary indications of the hazards to be encountered.

3-2. Scope. This chapter includes those tests required to assign hazard classifications for transportation of the bulk composition. These tests must be conducted prior to shipment in commerce of any explosive or propellant composition other than "Laboratory Samples" as specified by current ICC Regulations.

3-3. Classification of End Items. Procedures in chapters 4 and 5 must be followed in the assignment of the transportation and storage classifications to end items containing the explosive or propellant composition except when an analogy can be established with other items that are properly classified and identified.

3-4. Recording of Data. The results of tests performed under this chapter are to be recorded in a manner similar to that shown in figure 1.

3-5. Instrumentation. Due to the limited quantity of material involved in these tests, instrumentation to record peak pressure, impulse, and temperature as well as high speed photography may be eliminated.

3-6. Equipment. The following equipment is required for tests under this chapter:

a. One Bureau of Explosives impact apparatus. Drawings are available at the Bureau of Explosives, Association of American Railroads, 63 Vesey Street, New York, N. Y., 10007.

b. One ventilated explosionproof oven capable of maintaining a temperature of 75°C or above for a period of 48 hours. The oven will be equipped to continuously record the temperature. Dual devices for control of temperature should be provided.

c. Number 8 electric blasting cap, or caps of equivalent strength as required. A number 8 blasting cap is defined by ICC as one containing 2 grams of a mixture of 80 percent mercury fulminate and 20 percent potassium chlorate.

d. One blasting machine or equivalent for initiating electric blasting caps.

e. Kerosene-soaked sawdust sufficient for three beds, 1-foot square and ¼ inch thick.

f. Electric match-head igniters as required.

g. Solid lead cylinders 1½-inch diameter by 4 inches high as required.

h. One piece of mild steel plate SAE 1010 to 1030, ½ inch thick by 12 inches square.

i. Mild steel plates (SAE 1010 to 1030) 6 inch x 6 inch x ⅜ inch as required.

j. Tubing, steel, cold drawn seamless, mechanical, composition 1015, 1⅞ inch-OD, 0.219 inch-wall thickness variations ± 10 percent acceptable by 5½ inches long as required.

k. Cellulose acetate, or equivalent, cards, 2 inch diameter by 0.01 inch thick as required.

l. Wire (demolition cable) as required for connecting blasting machine to electrically initiated items.

m. Engineers special electric blasting caps (J-2) as required.

3-7. Test Samples. The following test samples are required for this chapter:

a. Twelve samples 2±¼-inch cubes.

Date _____

Sponsoring Agency _____

Contract No. _____

Propellant Identity (Type No.) _____

Propellant Spec. _____ Batch _____

Mfg. Date _____

Detonation Test

	Exploded Yes	Exploded No	Burned Yes	Burned No	Fragmented Yes	Fragmented No
No. 8 Blasting Cap Test I	___	___	___	___	___	___
Test II	___	___	___	___	___	___
Test III	___	___	___	___	___	___
Test IV	___	___	___	___	___	___
Test V	___	___	___	___	___	___

Samples: Five 2-inch cubes. Test: One blasting cap per sample.

Ignition & Unconfined Burning Test

	Exploded Yes	Exploded No	Average Burning Time Seconds
One 2-inch cube	___	___	_____
One 2-inch cube	___	___	_____
Four 2-inch cubes			_____

Samples: Six 2-inch cubes. Test: Ignite & burn unconfined.

Thermal Stability Test

	Explosion Yes	Explosion No	Ignition Yes	Ignition No	Change in Configuration Yes	Change in Configuration No
One 2-inch cube	___	___	___	___	___	___

Samples: One 2-inch cube Test: 48 hours at 75° C. in vented oven.

Card Gap Test 50% Value (No. of Cards) _____

Impact Sensitivity Test

Bureau of Explosives Impact Apparatus

Ten 3¼" (± 1/16") Drop Test 10 Trials			Ten 10" (± 1/16") Drop Test 10 Trials		
No. of Trials Exhibiting			No. of Trials Exhibiting		
Explosion Flame and Noise	Decomposition Smoke No Noise	No Reaction No Smoke No Noise	Explosion Flame and Noise	Decomposition Smoke No Noise	No Reaction No Smoke No Noise

Approved:

Test Director _____ Test Department Head _____

Assigned Classification	
ICC Forbidden	
ICC Restricted*	
ICC Class A	
ICC Class B	

DOD Approval

Signature _____

Title _____

Organization _____

*Shipping Instructions are to be requested from ICC (para 3-13a(2).

Figure 1. Sample summary data sheet.

AGO 7931A

b. Ten 10 mg (approx) samples suitable for use in the Bureau of Explosives impact apparatus (0.20 in. ± .02 in. diameter x 0.10 in. ± .02 in. long).

c. 2-inch diameter by 1-inch long pressed pentolite pellet, Federal stock No. 1375-991-8891 as required (para 5-1*c*).

d. Samples sufficient to fill 12 each of item 3-6*j* above (para 3-12*a*(2)).

3-8. Detonation Test. *a.* Place one lead cylinder (3-6*g*) upon the steel plate (3-6*h*). Place a No. 8 blasting cap (3-6*e*) perpendicular to and in contact with a flat surface of the 2-inch cube sample (3-7*a*) which is then placed on top of the lead cylinder. A 2-inch diameter wood block with a hole drilled in its center similar to that shown in figure 2 may be used for positioning the blasting cap. Deformation (mushrooming) of the lead cylinder will be considered as evidence of detonation. Conduct this test a minimum of five times, or until detonation occurs, whichever is the least number of tests.

b. Data from this test will be recorded under Detonation Test.

3-9. Ignition and Unconfined Burning Test. *a.* Place a 2-inch sample (3-7*a*) on a bed of kerosene-soaked sawdust (3-6*c*), and ignite the sawdust with an electric match-head igniter (3-6*f*). Perform this test twice.

b. Place four 2-inch (3-7*a*) samples end-to-end in a single row in contact with each other on a single bed of kerosene-soaked sawdust (3-6*c*) and ignite the sawdust with an electric match-head igniter (3-6*f*) at one end.

c. Record results under Ignition and Unconfined Burning Test.

3-10. Thermal Stability Test. *a.* Place one 2-inch sample (3-7*a*) in constant temperature explosionproof oven (3-6*b*). Raise the temperature of the oven to 75°C. and maintain the temperature at 75°C. for a period of 48 hours. These temperatures will be continuously recorded. Constant observation is not required.

b. Record results under Thermal Stability Test.

3-11. Impact Sensitivity Test. *a.* Conduct ten individual tests using one sample (3-7*b*) per test in the Bureau of Explosives impact apparatus (3-6*a*).

b. The sample (3-7*b*) is placed in the cup assembly, the weight is then dropped from the desired height (i.e., 3¾ or 10 in.) Observe results to supply data as required under Impact Sensitivity.

c. Use cleaning equipment as required to thoroughly clean and dry the anvil and cup assemblies of the impact apparatus prior to each test. Apparatus must be at ambient temperature (room temperature) 25°C. ± 5° prior to each test.

d. Check that the equipment is properly leveled and replace the tools when worn.

3-12. Card Gap Test. *a.* Materials required for each test are as follows:

(1) One each tubing (3-6*j*).
(2) Sample (3-7*d*) cast into or machined to fit into above tubing.
(3) Two pentolite pellets 2-inch diameter by 1 inch long (3-7*c*).
(4) One Engineers Special Blasting Cap J-2 (3-6*m*).
(5) One steel plate 6 inch x 6 inch x ⅜ inch (3-6*i*).
(6) Cellulose acetate, or equivalent, cards 2-inch diameter x 0.01 inch thick (3-6*k*).
(7) Four pieces of plastic material 1/16 inch x ½ inch x ½ inch.

b. Test configuration. The components of the test are arranged in the following manner. The witness plate is supported on two edges parallel to and approximately 6 inches above the ground surface. Four small pieces of material 1/16 inch x ½ inch x ½ inch are placed on the plate to support the pipe containing the test sample, and maintain the 1/16 inch air gap, which should not overlap onto the propellant or explosive. The air gap between the acceptor and witness plate should be free of solid material. The test sample is to be located approximately in the center of the witness plate. The pentolite booster is then placed on top of and in contact with the sample at the top of the pipe and the J-2 blasting cap attached. The arrangement of componets for this test is similar to that shown in figure 2 except the cellulose acetate cards and the cardboard tube are omitted in this test. Detonation is indicated when a clean hole is cut in the witness plate. The test sample and explosives booster are to be at a temperature of approximately 25°C. ± 5° at time of test. Should no detonation occur in the

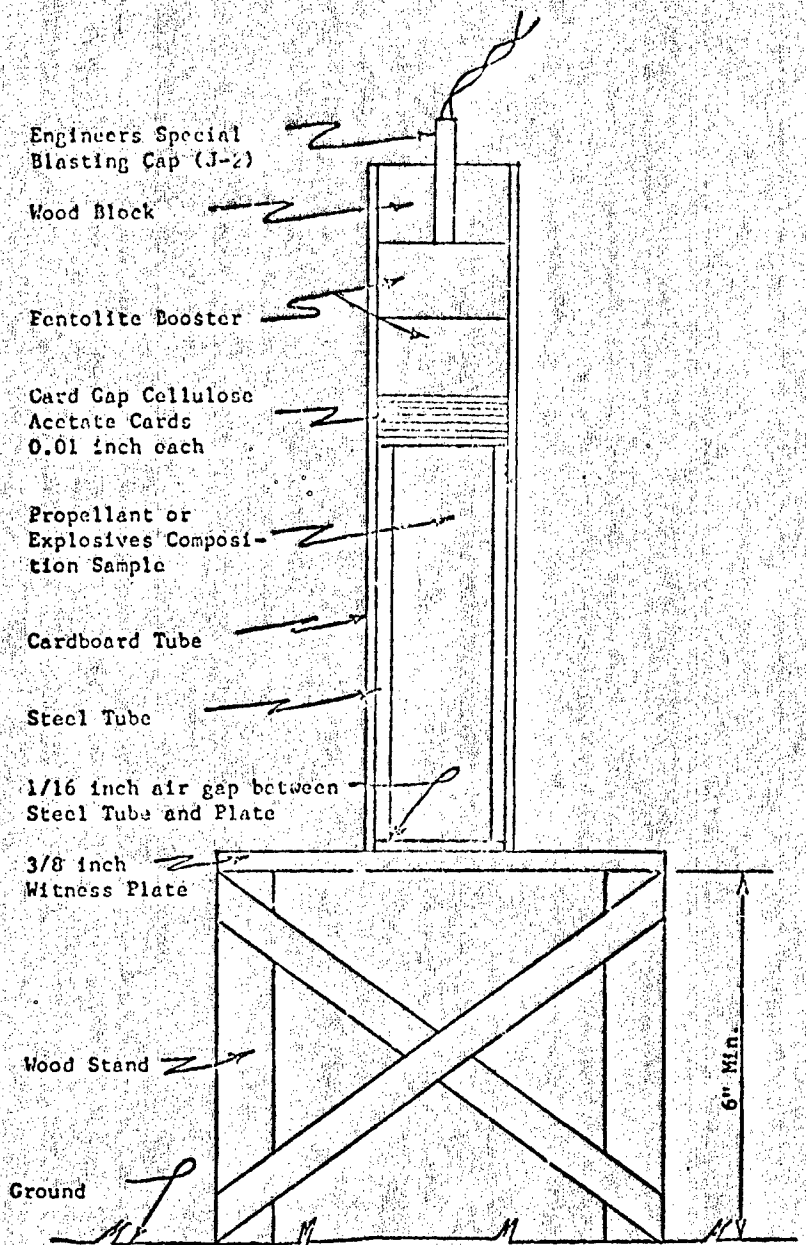

Figure 2. Setup for card gap test.

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first test, it will be repeated two times for a total of three tests. If detonation occurs, proceed to paragraph 3–12c.

c. The following tests are to be performed when the test sample detonates in the above tests (3–12*b*). If no detonation of the test sample occurs in the above tests, this test will not be performed.

(1) The test samples, high explosive boosters and witness plates used in these tests are as given in paragraph 3–12*a* above. The attenuation cards used are 0.01-inch cellulose acetate sheet or equivalent (3–6*k*). These tests are to be conducted with the test sample and booster at a temperature of approximately 25°C. ± 5° throughout. The test setup is as shown in figure 2. The cellulose acetate cards should be held firmly but not pressed.

(2) The first test to be performed will utilize 8 cards; if a detonation occurs, the number of cards will be doubled (i.e., 16 cards) for the second test. If no detonation occurs reduce the number of cards by one-half (i.e., 4 cards). Doubling of the number of cards will be continued in succeeding tests until no detonation occurs. When a number of cards is reached that prevents detonation, the next test will be with the number of cards reduced by half the preceding increment of increase (i.e., if detonation occurs at 32 cards but not at 64 cards, the next test will be with 48 cards). If detonation occurs at the reduced number of cards (48 cards in the example cited above) the number of cards in the next test will be increased by one-half the preceding increment (i.e., from 48 to 56 cards), etc. This procedure will be followed until the point of 50 percent probability of detonation is obtained. If the card gap sensitivity of a similar propellant or explosive composition is known, testing should begin at approximately that number of cards.

(3) Detonation is indicated when a clean hole is cut in the witness plate. The measure of charge sensitivity is the length of attenuation (gap length) at which there is 50 percent probability of detonation according to the above criterion. The charge sensitivity will be expressed in terms of number of 0.01-inch cards necessary for the 50 percent value between detonation and no detonation. Normally, a maximum of 12 tests will be required to determine the 50 percent value. (See A–9, appendix A.)

3–13. Interpretation of Results. *a.* For ICC and military purposes, results of chapter 3 tests will be interpreted as follows:

(1) ICC "Forbidden" if the following occurs: Thermal stability test results in either an explosion, burning, or marked decomposition of the sample.

(2) ICC Restricted. Compositions with an explosive impact sensitivity of less than 4 inches of drop height (3–11) will not be shipped until shipping instructions have been requested and received from the Interstate Commerce Commission.

(3) ICC Class A–(Military Class 7) if one or more of the following occur:

(*a*) Detonation and card gap tests have determined a detonation sensitivity value of 70 or more cards.

(*b*) Impact sensitivity test produces an explosion above 4-inches of drop height.

(*c*) Ignition and unconfined burning test produces a detonation.

(4) ICC Class B–(Military Class 2) if all of the following occur:

(*a*) Ignition and unconfined burning tests did not result in an explosion.

(*b*) The Thermal Stability Test did not result in an explosion, burning, or marked decomposition..

(*c*) Detonation and card gap tests have indicated a detonation sensitivity value of less than 70 cards or no reaction at zero cards.

CHAPTER 4

MINIMUM TEST CRITERIA FOR AMMUNITION AND EXPLOSIVES ITEMS INCLUDING FUZES, IGNITERS, MAIN EXPLOSIVE CHARGES, GUN TYPE PROPELLANTS, ARTILLERY AMMUNITION, PYROTECHNICS, ROCKET MOTORS AND ROCKET AMMUNITION UP TO 8 INCHES DIAMETER

4-1. Introduction. The tests in this chapter are intended to develop data upon which storage and transportation classifications of ammunition items may be based. Rocket motors and devices, containing solid propellant, except as indicated, are to be tested in accordance with chapter 5. The following test criteria will be used in the development of test operational plans for indicated ammunition items.

4-2. Number of Tests. Tables 1 through 4 indicate the minimum tests to be conducted, however, additional tests should be performed when a greater level of confidence is required for specific applications.

4-3. Use of Additional Items in Tests. Many of the items to be tested under this chapter will be suitable for inclusion in storage hazard classes 3 through 6 which are based upon fragment dispersion. The number of containers of items specified for the external heat test in tables 1 through 4 are the minimum upon which the classification may be based. However, in order to improve the statistical value of these tests and to more accurately predict results of accidents under actual storage conditions, the number of containers used in this test should be increased whenever possible.

4-4. Tests in Storage and Shipping Containers. All tests under this chapter are to be conducted on items in standard storage and transportation containers. Should items be stored or shipped in more than one type of container, the test series must be conducted on each type of container (i.e., wood vs. metal). Classifications will then be assigned to the item in each type of container.

4-5. Description of Tests. In the performance of tests given in tables 1 through 4, the following will apply:

a. Detonation "Test A" (propagation within a container).

(1) This test is to be conducted on items which are packaged with more than one item in the standard storage and shipping container to determine if functioning of one item will cause other items in the container to function.

(2) The most centrally positioned item within the package will be primed as required in the applicable table.

(3) The primed item will be fired from a safe location. The results of the test will be documented as required by paragraphs 2-3*b*(1), 2-3*b*(2)(*b*), 2-3*b*(3)(*c*), and 2-3*b*(3)(*d*).

(4) Test A will be conducted the specified number of times, or, until communication to adjacent items occurs, whichever is less.

b. Detonation "Test B" (propagation between containers).

(1) This test is to be conducted to determine if the functioning of items in one container will cause functioning of items in adjacent containers.

(2) If the detonation "Test A," above resulted in no communication within the container or the outside container was not ruptured, these tests ("Test B") may be omitted.

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(3) The item in the donor container to be primed and boostered in accordance with tables 1 through 6 is that which is closest to explosives in the acceptor container. This should assure subjection of acceptor explosives to maximum blast effects from donor material.

(4) The acceptor container will be positioned in a manner which provides the minimum separation between the explosive components in the two containers (i.e., container of fuzes with boosters will be arranged so that the boosters in one box are immediately beneath those above, and the lower box will be inverted in order to have minimum separation distance between the boosters).

(5) The primed item will be fired from a safe location. The results of the test will be documented as required by paragraphs 2-3*b*(1), 2-3*b*(2) (*b*), 2-3*b*(3) (*c*) and 2-3*b*(3) (*d*).

c. External Heat "Test C" (open fire). This test is designed to simulate a condition where the containers of explosive items are completely enveloped in a hot fire. Tables 1 through 6 require that more than one outer shipping container be used in this test. They should be arranged in a compact stack, approximating a cube, if possible. This stack should then be secured with steel bands in two directions. These steel bands are intended to maintain stacking until initiation of one or more items occurs. They must be incapable of significantly affecting dispersal of fragments. The stack of containers will then be placed on a crib of sufficient dimensions to hold the stack of containers and approximately 30 inches high. The interior of the crib is to be filled with combustible material such as scrap lumber. The crib and the stack of items to be tested are then covered with additional combustible material, such as scrap lumber, sufficient to insure a sustained hot fire. The entire mass is then to be saturated with approximately 50 gallons of JP-4 or diesel fuel and ignited by such means as an electric squib and 2 ounces of smokeless powder. In order to reduce the effects of wind direction, it is advisable to ignite the pile in two places preferably on opposite sides. Still photographs will be taken before and after the test. Photos after the test will clearly show results of the test. Resulting fragments and missiles will be identified and their location with respect to the test position recorded.

d. Where sand filled containers are specified for confinement in tests B and C, they will be of the same material as the containers of the items being tested. The sides and top of the container to be tested will be completely inclosed by the sand filled containers in order to provide confinement similar to a central container in a large stack of containers.

Table 1. Minimum Test Criteria For Determining Hazard Classification of Fuzes

(Including projectile type, rocket, missile, hydrostatic, torpedo exploder mechanisms, safe and arming devices, and initiating devices of all types)

1. Packaging	*2. Type of Info To Be Determined by Test*	*3. Types of Initiation To Obtain Info Outlined in Item 2*
With Booster Assembled	Type of Propagation Within 1 Container	Detonation
Without Booster	Type of Propagation from 1 Container to Another	External Heat
Without Booster, but Booster Packed in Same Container	Determination of Fragment Hazard	

4. Minimum Test Criteria

Type test	*Number items per test*	*Number of tests*	*Priming*	*Booster*	*Confinement*
Test A. Detonation	1 Shipping Container	5	Engr Special Blasting Cap	None	Sand Filled Containers
Test B. Detonation	2 Shipping Containers	5	Engr Special Blasting Cap	None	Sand Filled Containers
Test C. External Heat	6 Shipping Containers	1	None	None	Steel banded

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Table 2. Minimum Test Criteria for Determining Hazard Classification of Igniters

(Including rocket and missile, gun type, etc.)

1. Type	2. Packaging	3. Type of Info To Be Determined by Test	4. Types of Initiation to Obtain Info Outlined in Item 3
Electric	1 Per container	Type of Propagation Within 1 Container	Simple Ignition, or Detonation, as Appropriate
Friction	More than 1 Per Container		
Percussion		Type of Propagation from 1 Container to Another	External Heat
Combination of Any of Above		Determination of Fragment Hazard	

5. *Minimum Test Criteria*

Type test	Number items per test	Number of tests	Priming	Booster	Confinement
Test A. Detonation	1 Shipping Container	5	Engr Special Blasting Cap	None	Sand Filled Containers
Test B. Detonation	2 Shipping Containers	5	Engr Special Blasting Cap	None	Sand Filled Containers
Test C. External Heat	6 Shipping Containers	1	None	None	Steel Banded

Table 3. Minimum Test Criteria for Determining Hazard Classification of Main Explosive Charge

(Including projectiles, mines, depth charges, rifle and hand grenades, bombs, and demolition explosives)

1. Type	2. Type of Info To Be Determined by Test	3. Types of Initiation To Obtain Info Outlined in Item 2
Warhead, Less Fuze	Propagation from 1 Container to Another	Explosive Priming
Warhead, Plus Fuze (1) Attached	Separation Distance from 1 Item or Stack to Another to Prevent Propagation	External Heat
(2) Unattached, but in Same Container	Determination of Fragment Hazard	
	Determination of Blast Hazard	

4. *Minimum Test Criteria*

Type test	Number items per test	Number of tests	Priming	Booster	Confinement
Test A. Detonation	2 Shipping Containers (Side-by-Side)*	5 — Or Until First Detonation of Both	Engr Special Blasting Cap	30 Gram Tetryl** or Equivalent (Fuze Booster–When Attached)	None
Test B. Detonation	2 Shipping Containers (Separated 50 inches)*	3 Det of Primed Container Only	Engr Special Blasting Cap	30 Gram Tetryl** or Equivalent (Fuze Booster–When Attached)	None
Test C. External Heat	2 Shipping Containers	1	None	None	Steel Banded

*Projectiles not normally boxed for storage or shipment will be tested with a minimum of 2 stacks, each containing not less than 12 projectiles. Stacks to be separated 50 inches nose-to-nose-base-to-base.

**Pressed pellet approximately 1 inch-diameter x 1¼ inch-long at density of 1.5.

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Table 4. Minimum Test Criteria for Determining Hazard Classification of Gun Type Propellants for Cannon, Gun, Tube Mortar, and Rocket Motors up to 8-Inch Diameter

1. Type	*2. Type of Info To Be Determined by Test*	*3. Types of Initiation To Obtain Info Outlined in Item 2*
Gun Type Propellants for Cannon, Gun Tube Mortar or other Cartridge Actuated or Launching Devices in Bulk, Bags and Cartridge Cases (1) Single Base (2) Double Base (3) Triple Base	Liability to Detonation in Approved Bulk Storage and Shipping Containers (1) Bulk Propellant in Storage and Shipping Containers (2) Loaded in Finished Items Type of Propagation Within 1 Container Type of Propagation from 1 Container or Item to Another Determination of Fragment Hazard of Finished Items Determination of Blast Hazard	Simple Ignition External Heat Explosive Priming

4. Minimum Test Criteria for Bulk Propellant

Type test	*Number items per test*	*Number of tests*	*Priming*	*Booster*	*Confinement*
Test A. Detonation	1 Shipping container	5	Electric Squib*	2 Oz Black Powder	Sand Filled Containers and Steel Straps
Test B. Detonation	2 Shipping Containers	1	Electric Squib*	2 Oz Black Powder	Sand Filled Containers and Steel Straps
Test C. External Heat	6 Shipping Containers	1	None	None	Steel Banded

5. Minimum Test Criteria—Finished Items

Type test	*Number items per test*	*Number of tests*	*Priming*	*Booster*	*Confinement*
Test A. Detonation	1 Shipping Container	Not Less than 3	Engr Special Blasting Cap	30 Gram Tetryl**	None
Test B. Detonation	2 Shipping Container	Not Less than 2***	Engr Special Blasting Cap	30 Grain Tetryl**	Sand Filled Containers
Test C. External Heat	6 Shipping Container or Item	1	None	None	Steel Banded

*Squib to be placed 4 inches from bottom of can and all cans to be standing vertical.
**Pressed pellet approximately 1 inch-diameter x 1½ inch-long at density of 1.5.
***Not to be conducted if no detonation occurs in Test A—then Test A to be conducted 5 times.

Table 5. Minimum Test Criteria for Determining Hazard Classification of Gun, Howitzer, Mortar, and Recoilless Rifle Ammunition and Rocket Ammunition Complete Rounds (Assembled or Unassembled) up to 8-Inch Diameter

1. Type	*2. Packaging*	*3. Type of Info To Be Determined by Test*	*4. Types of Initiation To Obtain Info Outlined in Item 3*
Complete Rounds, Fixed Semifixed Separated Loading In Same Package	1 Round Per Container More than 1 Round Per Container	Propagation within a Single Container Propagation from 1 Container to Another Determination of Contribution of Propellant to HE Charge in 1 Package Determination of Fragment Hazard Determination of Blast Hazard	Simple Ignition Detonation External Heat

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5. Minimum Test Criteria

Type test	Number items per test	Number of tests	Priming	Booster	Confinement
Test A. Detonation of Projectile or Head	1 Item	5 Complete Units	Engr Special Blasting Cap or Normal Fuze Train Armed	30 Gram Tetryl*	Sand Filled Containers
Test B. Detonation of Projectile or Head	2 Shipping Containers	5	Same as Above	30 Gram Tetryl*	Sand Filled Containers
Test B. Detonation	2 Shipping Containers or 2 Stacks with 12 Items Each Separated 50 Inches Nose-to-Nose, Base-to-Base, or as Stored	5	Same as Above	30 Gram Tetryl*	Same as Above
Test C. External Heat	6 Shipping Containers	1	None	None	Same as Above
Test C. External Heat	6 Shipping Containers	1	None	None	None

*Pressed pellet approximately 1 inch-diameter x 1½ inches long at density of 1.6.

Table 6. Minimum Test Criteria for Determining Hazard Classification of Pyrotechnics —All Types and Certain Small Items Containing Solid Propellants (Para 3-3d)

1. Type	2. Packaging, as Normally Store and Shipped	3. Type of Info To Be Determined by Test	4. Types of Initiation To Obtain Info Outlined in Item 3
Burning	Individual Item or Unit	Propagation Within a Single Container	Simple Ignition
Detonating	More Than 1 Item Per Unit	Propagation from 1 Container to Another	Detonation
		Determination of Fragment Hazard	External Heat
		Determination of Blast Hazard	
		Determination of Fire Dispersement Hazard	

5. Minimum Test Criteria

Type test	Number items per test	Number of tests	Priming	Booster	Confinement
Test A. Detonation	1 Container	5	Normal Means of Ignition or Engr Special Blasting Cap	None	None
Test B. Detonation	2 Containers	5	Same as Above	None	None
Test C. External Heat	1 to 6 Containers Depending on Size of Unit	1	None	None	Steel Banded

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APPENDIX B

MATERIALS USED IN HAZARDS ASSESSMENT AND SHIPPING CLASSIFICATION TESTS

CONTENTS:

I. SWECO Vibro-Energy Mill Products

II. Fluid Energy Mill Products

III. Freeze Dried Products and Intermediates

MATERIALS USED IN HAZARDS ASSESSMENT AND SHIPPING CLASSIFICATION TESTS

I. SWECO VIBRO-ENERGY MILL PRODUCTS

A. VMA-76

Ground as 85/15/0.15 weight ratio slurry of Freon 113/AP/XD-7604 for 160 hours. Particle size of slurry at end of grind 2.56 microns WMD on 11/8/71. Particle size of dry powder when packaged 1.72 microns WMD on 11/10/71. Re-analysis of dry powder using improved analysis technique 0.81 microns WMD on 11/17/72.

B. VMA-93

Ground as 85/15/0.03 weight ratio slurry of Freon 113/AP/HX-868 for 141 hours. Additional HX-868 added prior to end of grind to give final weight ratio in dry powder form of 100/0.4. AP/HX-868. Particle size of slurry at end of grind 0.56 microns WMD on 6/6/72. Particle size of dry powder when packaged 0.71 microns WMD on 6/8/73. Re-analysis of dry powder 0.60 microns WMD on 6/12/73.

C. VMA-94

Processed exactly identical to VMA-93 (see item B above). Particle size of slurry at end of grind 0.54 microns WMD on 6/13/73. Particle size of dry powder when packaged 0.57 microns WMD on 6/15/73.

D. VMA-97

Processed exactly identical to VMA-93 (see Item B above). Particle size of slurry at end of grind 0.52 microns WMD on 10/19/72. Particle size of dry powder when packaged 0.69 microns WMD on 10/23/72. Re-analysis of dry powder 0.62 microns WMD on 11/13/72.

II. FLUID ENERGY MILL PRODUCTS

A. EFEM-33

Ground on eight-inch micron Master Fluid Energy Mill using standard unground AP (contains 0.2% TCP) as feed. Product particle size 1.60 microns WMD on 5/19/72.

B. EFEM-49

Ground on eight-inch micron Master Fluid Energy Mill using standard unground AP (contains 0.2% TCP) as feed. Product particle size 2.2 microns WMD on 9/29/72. Re-analysis of product 1.78 microns WMD on 10/2/72.

C. EFEM-63

Ground on eight-inch micron Master Fluid Energy Mill using standard unground AP (contains 0.2% TCP) as feed. Product particle size 1.70 microns WMD on 1/17/73.

III. FREEZE-DRIED PRODUCTS

A. FDI-9A

Made using 0.1% residual Johnson's Wax and 1.0% Nopcogen 16-0 and dried in intermediate size sublimator. Particle size when packaged 0.96 microns WMD on 6/20/72. Residual moisture content at that time 0.068%. Stored in cold box until re-packaged 11/21/72 with Sorbeads; particle size 1.29 microns WMD at that time, with 0.045% residual moisture content.

B. FDI-10A

Made using 1% Nopcogen 16-0 and 0.1% residual Johnson's Wax and dried in intermediate size sublimator. Particle size when packaged 0.73 microns WMD on 6/23/72. Residual moisture content at that time 0.03% H_2O.

C. FDI-10B

Made identical to FDI-9A (see Item A, above). Particle size when packaged 0.71 microns WMD on 6/23/72. Residual moisture content at that time 0.036%. Stored in cold box until re-packaged 11/21/72 with Sorbeads; particle size 0.70 microns WMD at that time, with 0.054% residual moisture content.

D. Freeze Drying Emulsion

Made using 1% Nopcogen 16-0 and 0.5% Johnson's Wax (would have reduced to about 0.1% if processed through sublimator), based on AP content. Ratio of water to AP used was about 2 to 1 by weight and ratio of trichloroethylene to water was about 1 to 8 by volume. Material made up on 7/26/72, just prior to card gap test shots (Test Series 81).

E. Frozen Emulsion in Liquid Nitrogen

Made by freezing emulsion referenced as Item E, above, in liquid nitrogen, on 7/26/72, just prior to card gap test shots (Test Series 82).

F. FDI-14A

Made with 10% Nopcogen 16-0, 1.5% JB polymer, 1.5% XB-23 biopolymer, and 1.0% residual Johnson's Wax, and dried in intermediate

size sublimator. Particle size when packaged 0.37 microns WMD on 8/7/72. Residual moisture content at that time 0.38%. Stored in cold box until re-packaged 11/21/72 with Sorbeads; particle size 0.35 microns WMD at that time, with 0.321% residual moisture content.

G. FDI-33B

Made with 2% Nopcogen 16-0 and 2.1% residual DOA and dried in intermediate size sublimator. Particle size when packaged 0.48 microns WMD on 11/10/72. Residual moisture content at that time 0.06%.

H. FDI-33C

Made with 1% Nopcogen 16-0 and 2.1% residual DOA and dried in intermediate size sublimator. Particle size when packaged 0.74 microns WMD on 11/10/72. Residual moisture content at that time 0.04%.

I. FDI-43D

Made with 2.0% Nopcogen 16-0 and 2.1% residual DOA and dried in the intermediate size sublimator. Particle size when packaged 0.62 microns WMD on 12/22/72. Residual moisture content at that time 0.03%.

J. FDI-52

Made identical to FDI-43D (See Item J above). Particle size when packaged 0.47/0.54 microns WMD on 2/26/73. Residual moisture content at that time 0.053%.

K. FDB-6B

Made with 2.0% Nopcogen 16-0 and 2.1% (residual) DOA and dried in the large size sublimator. Particle size when packaged 0.56 microns WMD on 12/8/72. Residual moisture content at that time 0.07%.

L. FDB-7

Made identical to FDB-6B (See Item L, above). Particle size when packaged 0.40 to 0.54 microns WMD on 2/9/73. Residual moisture content at that time 0.03%.

M. FDB-8

Made identical to FDB-6B (See Item L, above). Particle size when packaged averaged 0.99 microns WMD on 2/20/73. Residual moisture content at that time 0.04%.

APPENDIX C

SMALL-SCALE AND SPECIAL SENSITIVITY TESTS ON UFAP MATERIALS

CONTENTS:

Small-Scale Sensitivity Tests

Thermal Stability Tests (48 Hours at 75°C)

Ignition and Unconfined Burning Tests

Dust Explosibility Tests

TABLE C-I

SMALL-SCALE SENSITIVITY TESTS ON UFAP MATERIALS

Material Type and Composition	Material Source	Particle Size, Microns	Impact Sensitivity	Friction Sensitivity	Spark Sensitivity	DSC Inflections, °C Endotherm	1st Exotherm	2nd Exotherm
Fluid energy mill product dry powder; 100% AP	EFEM-33	1.60	Eo >250 Kg-cm	---	---	247	---	443
Fluid energy mill product dry powder, with coating: 99.8% AP, 0.2% Silanox 101	EFEN-33	1.60	Eo >250 Kg-cm	---	---	247	---	443
SWECO mill ground dry powder; 99.6% AP, 0.4% HX-868	VMA-94	0.57	Eo >250 Kg-cm	---	---	248	324	449
Freeze-dried product dry powder; 98.95% AP, 0.99% Nopcogen, 0.06% J-Wax	FDI-10A	0.73	Eo >250 Kg-cm	---	---	247	385	473
Freeze-dried product dry powder; 96.06% AP, 1.92% Nopcogen, 2.02% DOA	FDI-33B	0.48	+ @ 125 Kg-cm - @ 100 Kg-cm	+ @ 90 lbs	- @ 25 joules			
Freeze-dried product dry powder; 96.99% AP 0.97% Nopcogen, 2.04% DOA	FDI-33C	0.74	+ @ 200 Kg-cm - @ 150 Kg-cm	+ @ 70 lbs - @ 60 lbs	- @ 25 joules			

Thermal Stability Tests

Thermal stability tests, run in a vented oven for 48 hours at 75°C per paragraph 3-10 of DSAR 8220.1, were run on the four primary dry powder UFAP products studied in Task IV. The samples were hand-packed into small polyethylene wide mouth jars (nominal 180 cc capacity):

VMA-94 SWECO ground material	115.0 gms net UFAP
EFEM-33 Fluid-energy-milled material	101.1 gms net UFAP
EFEM-33 with 0.2% Silanox 101	103.0 gms net UFAP
FDI-10A freeze-dried material	96.2 gms net UFAP

The samples were sealed and stored at the Igniter Lab until the tests could be run, some eight weeks later. When opened for placement in the oven, it was noted that the UFAP had shrunk in size, away from the walls of the polyethylene jars. The SWECO ground material, VMA-94, was just slightly loose in the jar, while the others had shrunk to a much greater extent. The results of the tests are reported in Table C-II . After the tests were completed, it was noticed that the freeze dried sample and the fluid energy milled samples had shrunk enough to fall out of the jars completely. See Figure C-1. It is assumed that the shrinkage occurred through surface tension and equilibrium solubility effects. The minimal shrinkage in the SWECO ground material can probably be attributed to the protection afforded by the chemically reacted aziridine coating.

THIOKOL CHEMICAL CORPORATION
Huntsville Division
Huntsville, Alabama

TO: S. L. Vance Date: 72 Oct 03

FROM: Igniter Lab

A. E. Graves

Experimental Data Report No. 81

Subject: Thermal stability test of UFAP dry powders at 75°C for 48 hours

Reference: Your memo dated 72 July 20
Subject: Thermal stability test on UFAP dry powders

Results: VMA-94

Very little shrinkage; no color change; slightly caked.

FDI-10A; EFEM-33; and EFEM-33 with 0.2% Silanox 101

These samples formed a solid cake and shrank to approximately 1/2 their original volume.

Conclusions: Samples showed no evidence of decomposition and had no significant weight change after being exposed for 48 hours to 75°C.

cc:

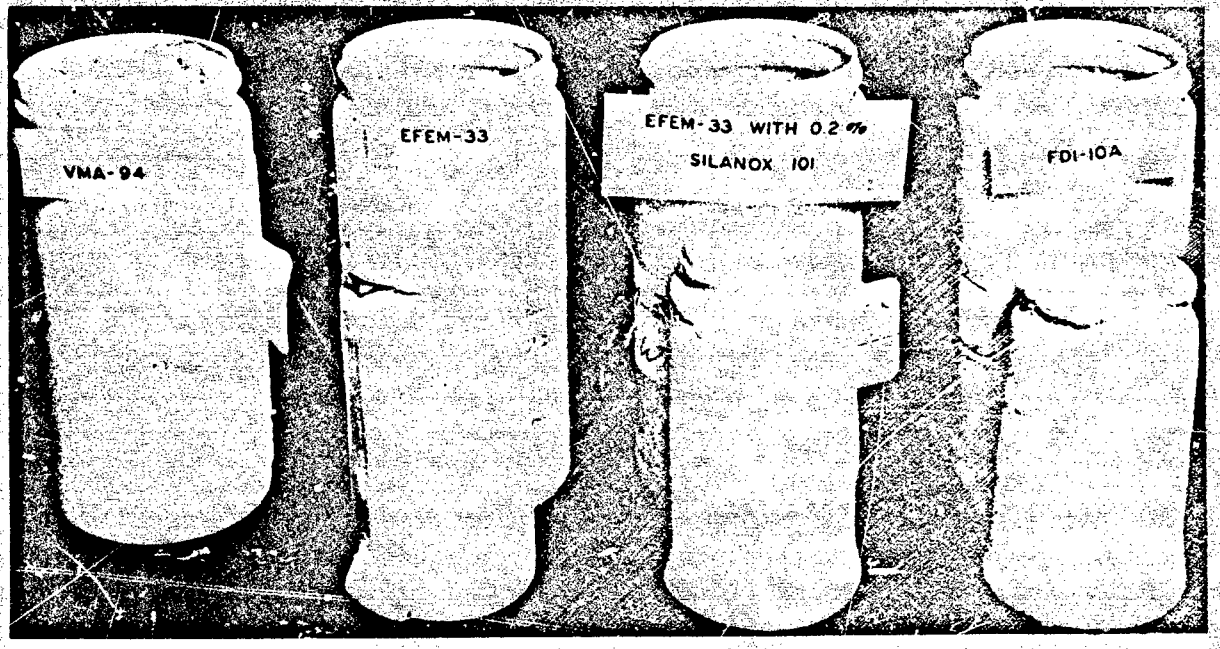


Figure C-1
Samples Used for Thermal Stability Tests

TABLE C-II

IGNITION AND UNCONFINED BURNING TESTS[a] ON UFAP MATERIALS

Date Tested	Test No.	Material Form	UFAP Source	Composition	% of T.M.D.[b]	Particle Size, Microns	Avg. Burning Time of Sample, secs.	Remarks[c]
8/3/72	87 A	Dry Powder, Loose-packed	VMA-93	99.6% AP, 0.4% HX-868	25	0.60	42	Single sample: smooth burning, with one puff at end.
	B				24		24	Single sample: smooth burning, with one puff at end.
	C				25		55	Four samples in row: smooth burning for entire reaction.
	88 A	Dry Powder, Loose-packed	FDI-10A	98.95% AP, 0.99% Nopcogen, 0.06% Johnsons Wax	25	0.73	25	Single sample: smooth burning.
	B				24		27	Single sample: smooth burning.
	C				24		33	Four samples in row: smooth burning.
	89 A	Dry Powder, Loose-packed	EFEM-33	100% AP	25	1.60	45	Single sample: jar burned off first, AP burned slowly and smoothly.
	B				25		45	Single sample: jar burned off first, AP burned slowly and smoothly.
	C				24		48	Four samples in row: jars burned off first, remaining AP burned slowly and smoothly.
	90 A	Dry Powder, Loose-packed	EFEM-33	99.8% AP, 0.2% Silanox 101	41	1.60	45	Single sample: jar burned off first, remaining AP burned slowly and smoothly.
	B				42		53	Single sample: jar burned off first, remaining AP burned slowly and smoothly.
	C				40		69	Four samples in row: jars burned off first, remaining AP burned slowly and smoothly.

NOTES: a. The sample materials were charged to a volume of about 150 cc. in small plastic jars (polyethylene, with 180 cc. total capacity). The filled jar is placed in a bed of Kerosine-soaked sawdust, which is then ignited remotely. The test is observed from a safe location to determine whether the sample explodes or burns, and the average time for the sample to burn up. This is run twice (using a single sample per test). A final test is then made in which four samples are placed in a line, contacting each other, and the sawdust/Kerosene is ignited at one end of the line.

b. Percent of theoretical material density (1.95 gms./cc. for AP). Denotes the degree of packing the material underwent during sample preparation.

c. When VMA-93 and FDI-10A materials tested, UFAP completely consumed as jar material ignited and burned. EFEM-33 samples did not react the same way. For these materials, the jar material burned off rapidly but only a thin film of UFAP was consumed with it. The remaining "stalks" of UFAP then burned very slowly, decomposing in the heat from the sawdust/Kerosene bed.

Dust Explosibility Tests

The standard method for electrostatic discharge sensitivity testing is generally not satisfactory for fine powdered materials because of their relative mobility. The energy released by the spark tends to blow the test material away from the electrodes before the spark induces initiation. As a result, the standard spark test was seldom run on UFAP dry powder. As an alternate, the Hartmann dust explosibility apparatus, developed by the Bureau of Mines, was evaluated. A general description of the equipment used and the operating techniques is given in Appendix A .

To conserve time and costs several changes were made during fabrication and assembly of the equipment, particularly in the power supply and controls, however, it was felt at the time that these changes would not interfere with the operation of the equipment. The Bureau of Mines provided a sample of Pittsburg coal dust, as a material of known explosive properties, to check out and calibrate our apparatus. Unfortunately, the sample was bad, and it wasn't until several months later that this fact was confirmed by the Bureau, based on tests of the same material on their apparatus.

According to Bureau of Mines Report RI-5624, the coal dust sample should have exhibited a minimum explosive concentration of 0.055 ounces per cubic foot, and a minimum energy for ignition of 60 millijoules. Attempts to duplicate these results in our apparatus failed. Although a very small, localized flame was achieved between the electrodes for most of the tests, only one shot exhibited satisfactory ignition and propagation throughout the cloud. This was at a dust concentration of 1.0 oz/ft^3 using the Plexiglas tube chamber. Attempts to repeat this with the steel chamber, to allow a pressure time trace to be recorded, were unsuccessful.

Some samples of Reynolds R-400 atomized aluminum powder and Alcoa Type 606 aluminum flake were located and tested in the steel tube chamber, yielding positive results in every case. These materials were used because they had been tested by the Bureau of Mines and the results reported in Report RI-6516. The Alcoa Type 606 aluminum flake was tested at concentrations of 0.1, 0.2, 0.5, and 1.0 oz/ft^3 and the Reynolds R-400 atomized powder at 0.2 and 0.5 oz/ft^3. Maximum pressures obtained were generally in the same order of magnitude as those reported by the Bureau of Mines, but equipment and instrumentation difficulties prevented any other parameters from being obtained from the pressure/time traces. The Bureau of Mines was then contacted for advice and for information about the problems we had encountered, and about the coal dust.

The Bureau was under considerable backlog of work at the time, and was not able to test the sample for more than a month. They confirmed that the sample was defective, requiring an abnormally high concentration to achieve repeatable ignitions. They were all out of their other usual test material, cornstarch, so promised to send a sample of cellulose acetate powder, and to run

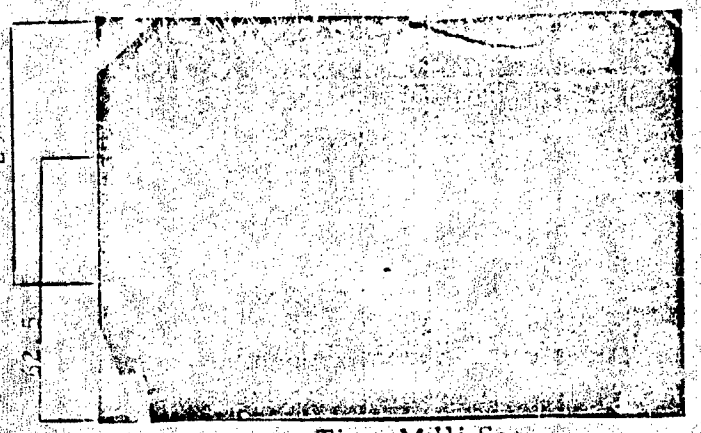
minimum energy for ignition and minimum explosive concentration tests on it prior to shipment.

As time was growing short, testing of samples of UFAP dry powder materials was initiated even though the apparatus had not been satisfactorily checked out with a known test material (except the aluminum powder and flake samples). These tests are summarized in Table C-III. It can be seen that the only material that gave any positive tests was the freeze dried UFAP containing nearly 4% coatings. Tests were even run at 10 oz/ft^3 concentrations without propagation even in the freeze dried material, although the samples for these tests had not been sieved through a 200 mesh screen prior to testing, as is normally done. The tests with freeze dried material that did propagate nevertheless did so at relatively slow rates, and, as can be noted, would not necessarily reproduce from one chamber to the other or even from one dispersing pressure chamber to another. Oscilloscope traces from three of the tests at 2.0 oz/ft^3 concentration are given as Figure C-2 through C-4.

Since some positive results were at least obtained from the freeze dried UFAP sample, an attempt was made to obtain minimum energy to ignition data using this material. The results are given in Table C-IV. Although the procedure recommends that the minimum energy to ignition test be run with concentrations from five to ten times the minimum explosive concentrations, the test personnel were unwilling to use this mesh material per test, so concentrations of 2.0, 3.0, and 4.0 oz/ft^3 were used. The lowest energy level resulting in ignition was six joules, at 3.0 oz/ft^3, at 3.0 oz/ft^3, which was not even repeatable. At 2.0 oz/ft^3, eight joules caused ignition, while six did not. (Using a nominal capacitance of 300 picofarads for the human body, charged to 5000 volts, yields only 3.75 millijoules of energy that can be electrostatically discharged by a person. Even increasing the voltage by a factor of ten brings the energy level only up to 0.38 joules). By comparison, Reynolds R-400 atomized aluminum powder ignites with only 30 millijoules, and the Alcoa 606 flake at 80 millijoules. Fine cake flour requires 60 millijoules, and powdered sugar only 30 millijoules.

The sample of cellulose acetate was received from the Bureau of Mines with a letter stating that the material had been tested there and gave a minimum energy required for ignition of 35 millijoules, and a minimum explosive concentration of 0.045 oz/ft^3. Tests run here are summarized in Tables C-V and C-VI. As can be seen, the minimum explosive concentration results agree fairly well, while the minimum energy to ignition tests ran three to four times the level reported by the Bureau of Mines. The samples marked "retest" were tested initially with negative results. The chamber was brushed out and the brushings put back in and shot a second time, yielding positive results. However, even these required at least twice the Bureau of Mines energy levels. It is assumed that our apparatus is not functioning properly in the minimum electrical energy to ignition type operation.

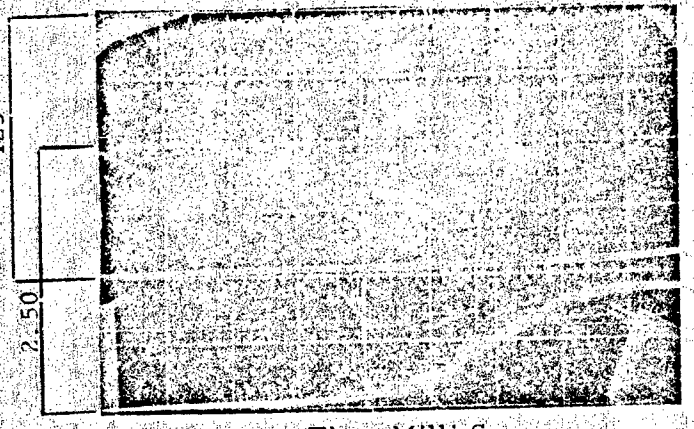
Although the Hartmann apparatus used may not have been operating properly, it is apparent that UFAP dry powder does not represent a very severe dust explosion hazard.



Time Milli Sec
Top - 20 milli Sec/cm
Bottom - 50 milli Sec/cm

March 1, 1973
FDB-6B
2.0 oz/ft^3
Steel Chamber 3 in^3
air chamber

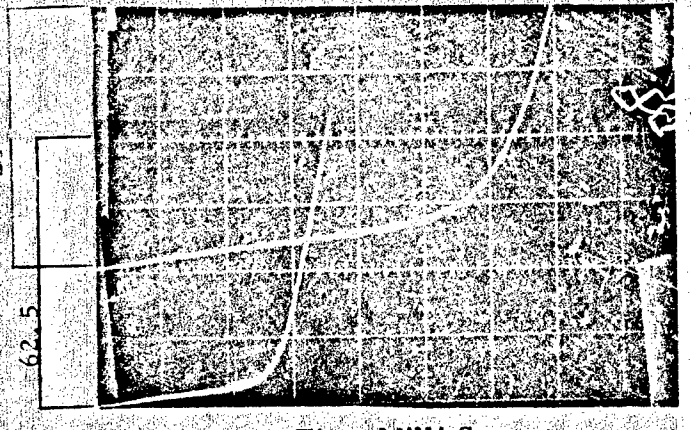
Figure C-2



Time Milli Sec
Top 20 milli Sec/cm
Bottom - 50 milli Sec/cm

March 1, 1973
FDB-6B
2.0 oz/ft^3
Steel Chamber - 80 in^3
air chamber

Figure C-3



Time Milli Sec
Top 20 milli Sec/cm
Bottom - 50 milli Sec/cm

March 1, 1973
FDB-6B
2.0 oz/ft^3
Steel chamber -80 in^3
air chamber

Figure C-4

TABLE C-III

DUST EXPLOSIBILITY TESTS[a] ON DRY POWDER UFAP MATERIALS

Type Material Tested (Source Designation)	Chamber Used / Air Reservoir Volume / Reservoir Pressure / Dust Concentration, oz/ft³	Plexiglas 80 in³ 15 psig 0.1	0.2	0.5	1.0	2.0	10[b]	Steel 3.0 in³ 150 psig 0.1	0.2	0.5	1.0	2.0	Steel 3.0 in³ 15 psig 0.2	Comments
Fluid Energy Milled AP (EFEM-63)		0	0	0	0	0						0		Did not disperse into dust cloud as readily as other materials
		0	0	0	0							0		
		0	0	0	0									
Fluid Energy Milled AP with 0.2% Silanox 101 added (EFEM-63)		0	0	0	0	0	0	0	0	0	0			Dispersed readily into good dust cloud
		0	0	0	0									
		0	0	0	0									
SWECO-Ground Material (VMA-97)		0	0	0	0	0	0					0		Dispersed readily into good dust cloud. Sample tried at 3.67 oz/ft³ but was negative
Freeze-dried UFAP (FDB-6B)		0	0	0[d]		0			0	0	0[e]			Dispersed readily into good dust cloud
		0	0[c]	0							0			
		0	0[c]								0			
										0				

NOTES:

a. Continuous arc discharge at 12 kilovolts used as ignition source on all tests. All samples exhibited localized ignition in immediate region of arc. Except as noted, all samples were sieved through a 200 mesh screen prior to testing. + denotes a positive test. 0 denotes a negative test. Absence of mark indicates no test performed.

b. Materials tested at this concentration were not sieved at all.

c. Some smoke evolved, but test negative.

d. Larger flame resulted than for other negative tests, but still negative.

e. Walls of chamber darkened by reaction products, but test still negative.

TABLE C-IV

MINIMUM ENERGY TO IGNITION TESTS[a] ON FREEZE-DRIED UFAP[b]

Dust Concentration Used, oz/ft^3	2.0				3.0				4.0			
Conditions and Results	Volts[c]	Microfarads[d]	Joules[e]	Result	Volts[c]	Microfarads[d]	Joules[e]	Results	Volts[c]	Microfarads[d]	Joules[e]	Result
	400	200	16	+	400	75	6	+	400	50	4	0
	400	200	16	+(?)	400	50	4	0				
	400	200	16	+	400	75	6	0				
	400	100	8	+	400	100	8	0				
	200	100	2	0								
	300	100	4.5	0								
	350	100	6	0								
	400	100	8	+								

NOTES:
a. Run in plexiglas chamber using pulsed discharge from charged capacitor bank. Positive tests, denoted by +, exhibited flame at least 4 inches long. Negative tests denoted by 0. 80 in^3 reservoir charged to 15 psig air pressure using as dust dispersant for all tests.
b. From FDB-6B
c. Voltage applied across capacitor bank.
d. Capacitance in microfarads
e. Calculated by 1/2 CV^2

TABLE C-V

MINIMUM EXPLOSIVE CONCENTRATION TESTS[a] ON CELLULOSE ACETATE[b]

Dust Concentration Tested, oz/in^3	Dispersing Air Pressure, psig	Result
0.10	15	+
0.050	15	0
0.075	15	+
0.050	10	+
0.045	10	0
0.045	10	0

NOTES: a. Run in Plexiglas tube chamber with continuous arc discharge ignition source. Reservoir used was 80 in^3 volume. Positive test, denoted by + in Result column, indicated filter paper diaphram at top of chamber was ruptured. Negative test denoted by 0.

b. Sample No. 2074, from Industrial Safety Group, Bureau of Mines, Pittsburgh, Pennsylvania

TABLE C-VI

MINIMUM ENERGY TO IGNITION TESTS[a] ON CELLULOSE ACETATE[b]

Sample Concentration, oz/ft²	Air Reservoir Volume, in³	Reservoir Pressure Used, psig	Pressure Switch Setting,[c] psig	Volts[d]	mf.[e]	Millijoules[f]	Result
0.4	80	15	13	100	10	50	0
0.25	80	15	13	100	10	50	0
0.25	3.0	80	13	100	40	200	+
0.25	3.0	80	13	100	30	150	0
0.25	3.0	60	13	100	40	200	+
0.25	3.0	60	13	100	30	150	0
0.25	3.0	60	(g)	100	40	200	0
0.25	3.0	60	13	100	30	150	+
0.25	3.0	60	13	100	20	100	+
0.25	3.0	60	13	100	15	75	0
0.25	3.0	60	13	100	20	100	+
0.25	3.0	60	40	100	20	100	0
0.25	3.0	80	40	100	20	100	0
0.25	3.0	100	9	100	20	100	0
0.25	3.0	50	9	100	20	100	0, +[h]
0.4	3.0	40	9	100	20	100	+
0.4	3.0	40	9	100	15	75	0, +[h]

NOTES:

a. Run in Plexiglas tube chamber using pulsed discharge from charged capacitor bank. Positive tests, denoted by + in Results column, exhibited a flame at least 4 inches long. Negative tests denoted by 0. Absence of mark indicates no test made.

b. Sample No. 2074 from Industrial Safety Group, Bureau of Mines, Pittsburgh, Pennsylvania.

c. Adjustment in ignition pulse triggering circuit. Used to control delay time between initial opening of dispersing air solenoid valve and actuation of pulse. The lower the setting, relative to set reservoir pressure, the longer the delay.

d. Voltage applied across capacitor bank.

e. Capacitance in microfarads.

f. Calculated by $1/2\ CV^2$.

g. Not used; pulse triggered manually.

h. Retest; see discussion.

APPENDIX D

CARD GAP TESTS, COMMENTS, DATA, PHOTOGRAPHS

TABLE D-I

CARD GAP TEST RESULTS ON UDAP MATERIALS

Date Tested	Test No.	Material Type	Material Source	Composition	% of T.M.D.	Particle Size, microns	No. Cards	Results	Remarks and Witness Plate Thickness
[illegible]	1 A	Dry Powder	EFSM-11	100% AP	72	1-40	0	neg	Dented 1 1/2" and cut surface
	B				70		0	neg	Dented 1 7/16 and cut surface
	C				71		0	pos	Broke plate and punched hole
	D				67		0	neg	Dented 1 1/2" and cut surface
	E				73		4	pos	Broke plate and punched hole
	F				73		4	neg	Dented 1 5/16" and cut surface
	G				71		4	neg	Dented 1 1/8" and cut surface
	H				71		4		Broke plate into but did not punch
	I				72	9-17	0	pos	Broke plate and punched hole
72	A	Dry Powder	UOP-98	99.4% AP 0.6% HTX-108	70		0	neg	Dented plate 1 3/8
	B				74		4	pos	Broke plate and punched hole
	D				69		4	pos	Broke plate but did not punch
	E				65		3	neg	Broke plate and punched hole
	F				64		4	pos	Broke plate and punched hole
	G				72		12	neg	Dented plate 1 3/8
	H				74		10	pos	Broke plate and may have punched hole
21	A	Dry Powder	EFSM-11	99.8% AP 0.2% Silanox 101	71	1-40	0	pos	Broke plate and punched hole
	B				72		0		Broke plate and punched hole part way
	C				74		11	neg	Dented 1 1/4" and cut surface
	D				72		12	neg	Dented 1 5/8" and cut surface
	E				73		10		Broke plate and cut surface but did not punch
	F				72		11	neg	Dented 1 1/4" and cut surface
	G				74		10	neg	Dented 1 1/2" and cut surface
	H				73		9	neg	Dented 1 3/8" and cut surface
	I				74		10	neg	Dented plate 1 3/8
14	A	Dry Powder	FDI-10A	98.5% AP 0.5% Nigrosyn 0.4% I-Wax	70	0-73	0	neg	Dented plate 1 5/8
	B				71		0	neg	Dented plate 1 7/8
	C				72		0	neg	Dented plate 1 7/8
1-2-64	14	Water for calibration purposes		100% H_2O	---	---	0	neg	Dented plate very slightly
	B				---	---	0	neg	Dented plate very slightly
	C				---	---	0	neg	Dented plate very slightly
61	A	Dry Powder	C-653	100% RDX, Type B	71	240	0	pos	Plate broke into 4 pieces clean hole
	B				64		8	pos	Plate broke in 3 pieces punched clean hole
	C				62		16	pos	Plate broke in 3 pieces punched clean hole
	D				63		32	pos	Plate broke into punched clean hole
	E				63		64	pos	Plate broke into punched clean hole
	F				63		128	pos	Punched clean hole
	G				64		256	pos	Punched clean hole
	H				66		512	neg	Plate undamaged, material not consumed
	I				65		384	neg	Plate undamaged, material not consumed
	J				66		320	neg	Plate undamaged, material not consumed
	K				63		256	pos	Punched clean hole
61	A	Use Water for calibration purposes		100% H_2O	---	---	0	neg	Dented plate 1/4
	B	Emulsion		38.11% AP, 61.14% H_2O Trimorethylene, 0.50% Nonylagen, 0.25% I-Wax	---	---	0	neg	Dented plate 3/16
	C				---	---	0	neg	Dented plate 3/16
	D				---	---	0	neg	Dented plate 3/16
62	A	Liquid N_2 for calibration purposes		100% liquid N_2	---	---	0	neg	Dented plate 3/16"
	B	Frozen emulsion in liquid N_2, emulsion composition as in test series 61, ratio of emulsion/N_2 unknown			---	---	0	neg	Dented plate 3/16"
	C				---	---	0	neg	Dented plate 3/8"
	D				---	---	0	neg	Dented plate 1/4"

NOTES: a. The sample material is contained in a steel stainless mechanical tubing container such that the sample is 1 7/16 inches diameter by 5 1/2 inches long. The loaded container is placed on end on a 3/8 inch thick mild steel witness plate. Cards, of 10 mil thick cellulose acetate are placed on top of the container to separate the sample material from two 50 gram pentolite donor pellets, each 2 inches in diameter by 1 inch thick. The pentolite is initiated with a 1-2 blasting cap. If a stable detonation wave is developed in the test material, a clean hole is punched in the witness plate. Deformation or breaking of the plate without a hole being punched indicates a deflagrative reaction occurred in the sample material, contributing substantially to the net explosive energy released, but not a reaction of detonation velocity.

b. Percent of theoretical material density (1.95 gms./cc. for AP, 1.82 gms./cc. for RDX). Denotes degree of packing the material underwent during loading.

c. The number of cards used to separate the donor pellets (pentolite) from the sample material. Zero indicates no cards or separation were used. If a negative result is obtained at zero cards, this condition is repeated twice to ensure the result was valid. If a positive result is obtained, cards are used for the next shot, and the number of cards increased for successive shots until the detonation is just suppressed (i.e., one less card results in a positive test).

d. Negative indicates a hole was not punched through the witness plate, so a stable detonation did not occur. Positive denotes a detonation occurred, as indicated by a clean hole punched through the witness plate. A question mark indicates the result was indeterminate due to loss of parts of the witness plate.

e. Dented means the witness plate was bulged by the explosion to the depth (in the remark) noted. A cut indicates a detonation reaction most probably reached the witness plate in certain places, causing localized cutting or gouging, but not in sufficient strength or area to cause punching. A break is when the plate material reached all the way through, usually resulting in two or more pieces. A punch is when a clean hole is rammed through the plate, with or without plate breakage.

TABLE D-II

CARD GAP[a] TESTS ON UFAP MATERIALS

Date Tested	Test No.	Material Form	UFAP Source	Composition	% of T.M.D.[b]	Particle Size, Microns	No. of Cards[c]	Results[d]	Remarks
8/3/72	83 A	Dry Powder	VMA-93	99.6% AP, 0.4% HX-868	37	0.60	5	positive	Punched hole and broke plate into 8 pieces.
	B				41		14	negative	Broke plate into at least 6 pieces, of which 4 recovered.
	C				37		12	?	Broke hole and broke plate into at least 8 pieces (7 recovered).
	D				41		10	negative	Bent plate up and broke hole through it, cuts in surface.
	84 A	Reconstituted Slurry	VMA-93	74.81% Free in; 24.94% AP, 0.25% HX-868	--	0.60	0	negative	Did not dent plate more than 1/16 inch.
	B				--		0	negative	Did not dent plate at all.
	C				--		0	negative	Did not dent plate more than 1/16 inch.
	85 A	Dry Powder, Loosely packed	VMA-93	99.6% AP, 0.4% HX-868	18	0.60	0	negative	Dented plate 1 1/2 inches.
	B				18		0	negative	Dented plate 1 1/2 inches.
	C				18		0	negative	Dented plate 1 1/2 inches and cracked through dent.
	86 A	Dry Powder, Lightly tamped	VMA-93	99.6% AP, 0.4% HX-868	27	0.60	0	negative	Dented plate 1 3/8 inches.
	B				26		0	negative	Dented plate 1 1/4 inches and cracked through dent.
	C				28		0	negative	Dented plate 1 3/8 inches.

NOTES:
a. The sample and test set-up are described in detail in the notes to Table IV-1 of the July, 1972, report.
b. Percent of theoretical material density (1.95 gms./cc. for AP). Denotes the degree of packing the material underwent during loading.
c. See notes to Table IV-1 of July, 1972, report.
d. Negative indicates a hole was not punched through the witness plate, i.e., a stable detonation did not develop. Positive denotes a detonation occurred, as indicated by a clean hole punched through the witness plate. A question mark indicates the result was indeterminate due to loss of parts of the witness plate.
e. Dented means the witness plate was bulged by the explosion to the depth (at the center) noted. A cut indicates a detonation reaction most probably reached the witness plate in certain places, causing localized cutting or gouging, but not in sufficient strength or area to cause punching. A break is when the plate material cracked all the way through, usually resulting in two or more pieces. A punch is when a clean hole is rammed through the plate, with or without plate breakage.

TABLE D-III

CARD GAP[a] TESTS ON [illegible]

Date Tested	Test No.	Material Form	UFAP Source	Composition	% of T.M.D.[b]	Particle Size, microns	Card Gap[c]	Results[d]	Remarks
8/8/72	92 A	Dry Powder	VMA-93	99.6% AP, 0.4% HX-868	32	6.60	0	negative	Dented plate 1 1/2 inches, [illegible] pieces
	B	Dry Powder with Sorbeads	VMA-93	25% Sorbeads, 75% as above	35			negative	Dented plate 1 [illegible] inches
	C			35% Sorbeads, 65% as above	34			negative	Dented plate [illegible] inches
	D			50% Sorbeads, 50% as above	39			negative	Dented plate 1 1/2 inches
	E			25% Sorbeads, 75% as above	33			negative	Dented plate 1 1/2 inches
	F			25% Sorbeads, 75% as above	33			negative	Dented plate 1 1/2 inches
	G			10% Sorbeads, 90% as above	32			negative	Dented plate 1 [illegible] inches
	H			10% Sorbeads, 90% as above	32			negative	Dented plate 1 [illegible] inches
	I			10% Sorbeads, 90% as above	33			negative	Dented plate 1 1/2 inches
	J			18.4% Sorbeads, 81.6% as above	34			negative	Dented plate 1 1/2 inches
	K			10% Sorbeads, 90% as above	31			negative	Dented plate 1 1/2 inches
	L			10% Sorbeads, 90% as above	30			negative	Dented plate 1 1/2 inches and [illegible]
	M			10% Sorbeads, 90% as above	29			negative	Dented plate 1 1/2 inches
	N			99.6% AP, 0.4% HX-868	33			negative	Dented plate 1 1/2 inches, cut [illegible]
	O			99.6% AP, 0.4% HX-868	33			negative	Dented plate 1 1/2 inches, cut [illegible]
	93 A	Dry Powder	EFEM-33	99.8% AP, 0.2% Silanox 101	34	1.10	0	negative	Dented plate 1 1/2 inches and broke [illegible]
	B				32			negative	Dented plate 1 1/2 inches
	C				36			negative	Dented plate 1 [illegible] inches and broke [illegible]
	D	Dry Powder with Sorbeads	EFEM-33	10% Sorbeads, 90% as above	28			negative	Dented plate 1 1/2 inches
	E			10% Sorbeads, 90% as above	29			negative	Dented plate 1 1/2 inches
	F			10% Sorbeads, 90% as above	28			negative	Dented plate 1 1/2 inches

NOTES: a. The sample and test set-up are described in detail in the notes to Table V-I of the July, 1972, report.

b. Percent of theoretical material density (1.95 gm/cc for AP). Denotes the degree of packing the material underwent during loading.

c. See notes to Table IV-I of July, 1972, report.

d. Negative indicates a hole was not punched through the witness plate, so a stable detonation did not develop. Positive denotes a detonation occurred, as indicated by a clean hole punched through the witness plate. A question mark indicates the results were indeterminate because parts of the witness plate.

e. Dented means the witness plate was bulged by the explosion to the depth at the center noted. A "cut" indicates a detonation reaction zone was established in the witness plate in certain places, causing localized cutting or gouging, but not in sufficient strength or areal coverage to punch a hole. "Broke" means the plate material cracked all the way through, usually resulting in two or more pieces. A piece is when a clean hole was punched but the plate broke into two pieces.

TABLE D-IV

CARD GAP[a] TESTS ON FREEZE-DRIED UFAP MATERIAL

Date Tested	Test No.	Material Form	UFAP Source	Composition	% of TMD[b]	Particle Size(microns)	No. of Cards[c]	Results[d]	Remarks
1/15/73	95A	Dry Powder Loosely Filled	FDI-43D	9.1% AP, 2.0% DOA, 1.0% Nopcogen 16-0	19.0	0.62	0	Negative	Dented plate 1-9/16 inches
	B				19.0		0	Negative	Dented plate 1-1/2 inches
	C				19.5		0	Negative	Dented plate 1-9/16 inches
	D	Dry Powder Hand-Packed			27.0		0	Negative	Dented plate 1-12/16 inches
	E				31.0		0	Negative	Dented plate 1-9/16 inches
	F				30.0		0	Negative	Dented plate 1-9/16 inches

NOTES:

a. The sample material is contained in a steel seamless mechanical tubing container such that the sample is 7/16 inches diameter by 5 1/2 inches long. The loaded container is placed on end on a 1/8 inch thick mild steel witness plate. Cards of 10 mil thick cellulose acetate are placed on the top of the container to separate the sample material from two 80 gram pentolite donor pellets, each 2 inches in diameter by 1 inch thick. The pentolite is initiated with a J-2 blasting cap. If a stable detonation wave is developed in the test material, a clean hole is punched in the witness plate. Deformation or breaking of the plate without a hole being punched indicates a definite reaction occurred in sample material, contributing substantially to the net explosive energy released, but not a reaction of detonation velocity.

b. Percent of theoretical material density (1.95 gms/cc for AP, 1.82 gms/cc of RDS). Denotes degree of packing the material underwent during loading.

c. The number of cards used to separate the donor pellets (pentolite) from the sample material. Zero indicates no cards or separation were used. If a negative result is obtained at zero cards, the condition is repeated twice to ensure the result was valid. If a positive result is obtained, cards are used for the next shot, and the number of cards increased for successive shots until the detonation is just suppressed (i.e., one less card results in a positive test).

d. Negative indicates a hole was not punched through the witness plate, so a stable detonation did not occur. Positive denotes a detonation occurred, as indicated by a clean hole punched through the witness plate. A question mark indicates the result was indeterminate due to loss of parts of the witness plate.

Date Tested	Test No.	Material (mix)	TMD[a]	Composition	Pressure Size (psi)	Density[b]	Cuts	Results	Remarks
1/15/73	103A	Dry powder, loosely filled		99.0% AP, 1.0% Sphaerocarb-70	0	0.62	0	Negative	Dented plate 1 3/4 inches
	B				0		0	Negative	Dented plate 1 3/4 inches
	C				0		0	Negative	Dented plate 1 3/4 inches
	A	Dry powder, hand packed			0		0	Negative	Dented plate 1 3/4 inches
	E				11.2		0	Negative	Dented plate 1 3/4 inches
	F				19.0		2	Negative	Dented plate 1 3/4 inches
2/4/73	103A	Dry powder, hand packed to high density	EDE-13D		47.2	0.62-0.56	0	Positive	Plate badly broken up
	B				45.0		2	Positive	Plate badly broken up
	C[c]				47.4		0	Positive	Still badly broken up
	D[c]				47.8		0	Positive	Still badly broken up
2/10/73	104A	Dry powder, loosely filled	EDE-13A	99.0% AP, 2.04% Silanox 101	16.0	1.17	0	Negative	Dented plate 1 7/16 inches
	B				19.7		0	Negative	Plate broken up
	C				17.4		0	Negative	Plate broken up
	D[c]				14.7		0	Negative	Plate dented 1 1/4 inch
	E[c]				15.0		0	Negative	Plate dented 1 7/32 inch
	F[c]				17.7		0	Negative	Plate dented 1 3/15 inch
	G[c]				20.5		0	Negative	Plate dented 1 3/16 inch
	H[c]	Dry powder, hand packed			26.8		0	Negative	Plate dented 1 1/8 inch
	I[c]				28.4		0	Negative	Plate dented 1 1/6 inch
	J[c]				29.6		0	Negative	Plate broken up
2/10/73	108A[c]	Dry powder, loosely filled	VMA-76	99.0% AP, 1.0% XD-7604	17.7	0.81	0	Negative	Plate broken up
	B[c]				17.3		8	Negative	Dented plate 1 1/4 inch
	C[c]				17.7		0	Negative	Dented plate 1 7/16 inch
	D[c]				16.1		0	Negative	Dented plate 1 3/4 inch
	E[c]	Dry powder, hand packed			28.3		0	Negative	Plate broken up
	F[c]				29.3		0	Negative	Plate broken up
	G[c]				28.1		0	Negative	Dented plate 1 5/32 inch

NOTES:
a. The sample and test set-up are described in detail in the notes to IV-I of the July, 1972, report.
b. Percent of theoretical material density (1.95 gms./cc. for AP). Denotes the degree of packing the material underwent during loading.
c. See notes to Table IV-I of July, 1972, report.
d. Negative indicates a hole was not punched through the witness plate, so a stable detonation did not develop. Positive denotes a detonation occurred, as indicated by a clean hole punched through the witness plate. A question mark indicates the result was indeterminate due to loss of parts of the witness plate.
e. Dented means the witness plate was bulged by the explosion to the depth at the center noted. A cut indicates a detonation reaction almost probably reached the witness plate in certain places, causing localized cutting or gouging, but not in sufficient strength or area to cause punching. A break is when the plate material cracked all the way through, usually resulting in two or more pieces. A punch is where a clean hole is punched through the plate, with or without plate breakage.
f. 1/16 inch thick stand-off spacers inserted between sample tube and witness plate.

A
0 CARDS

B
0 CARDS

C
0 CARDS

D
8 CARDS

TEST SERIES 71
CARD GAP

EFEM-33

Figure D-1. Card Gap Test Witness Plates

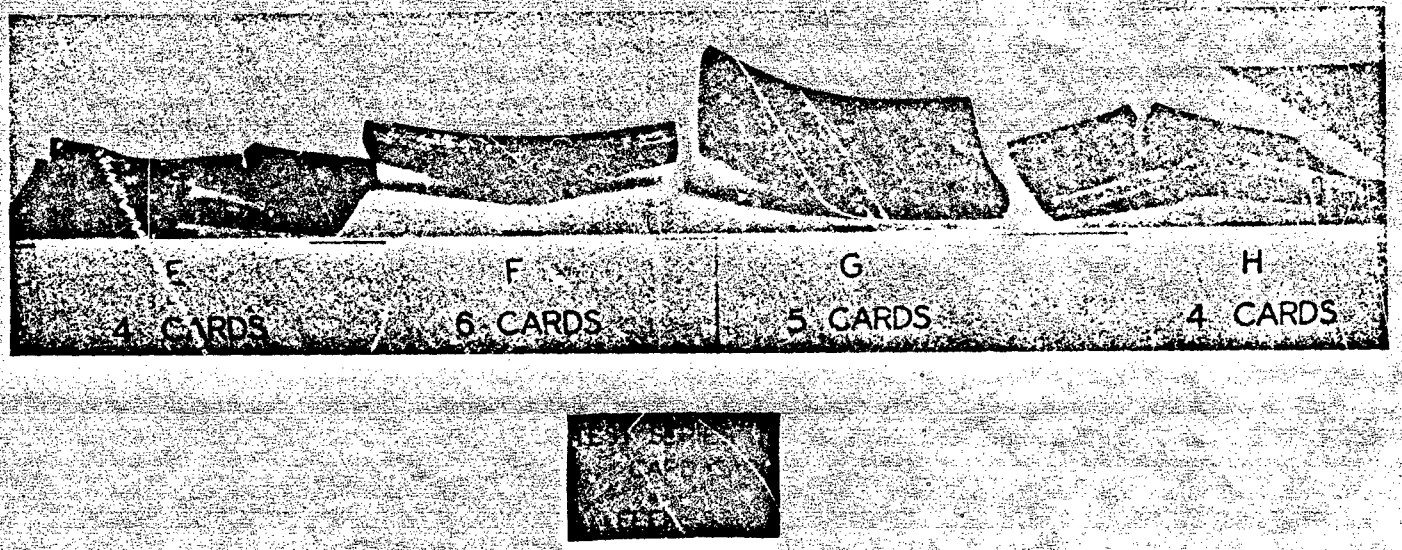


Figure D-2. Card Gap Test Witness Plates

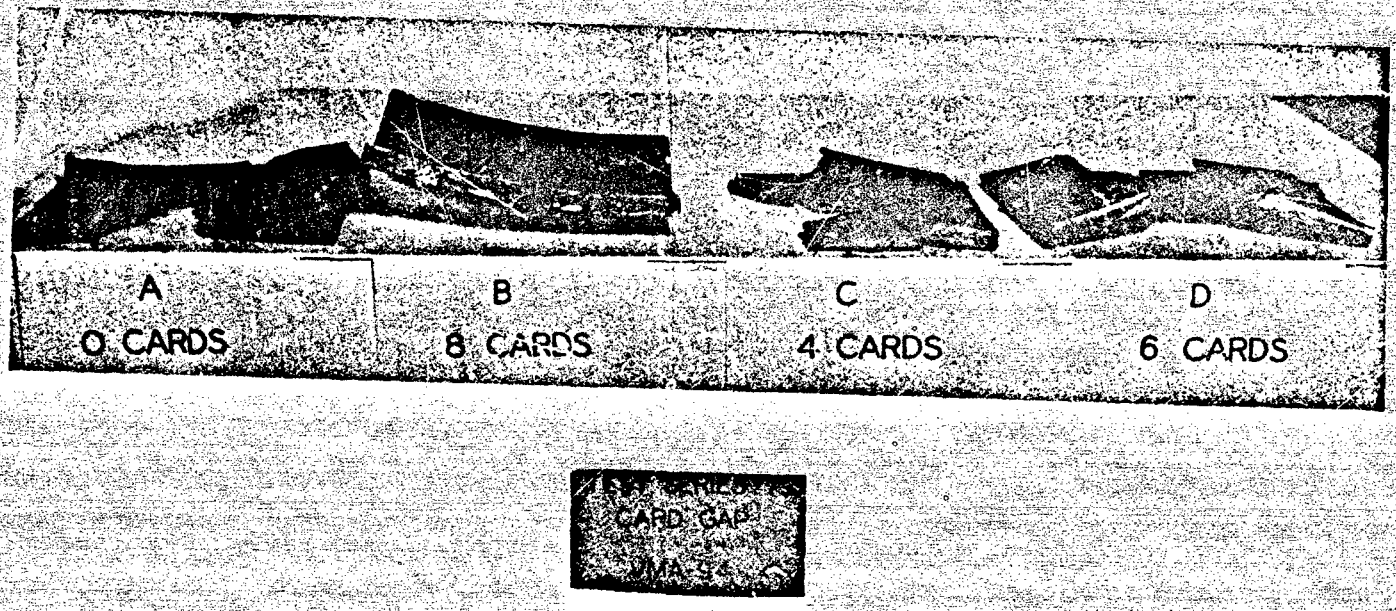


Figure D-3. Card Gap Test Witness Plates

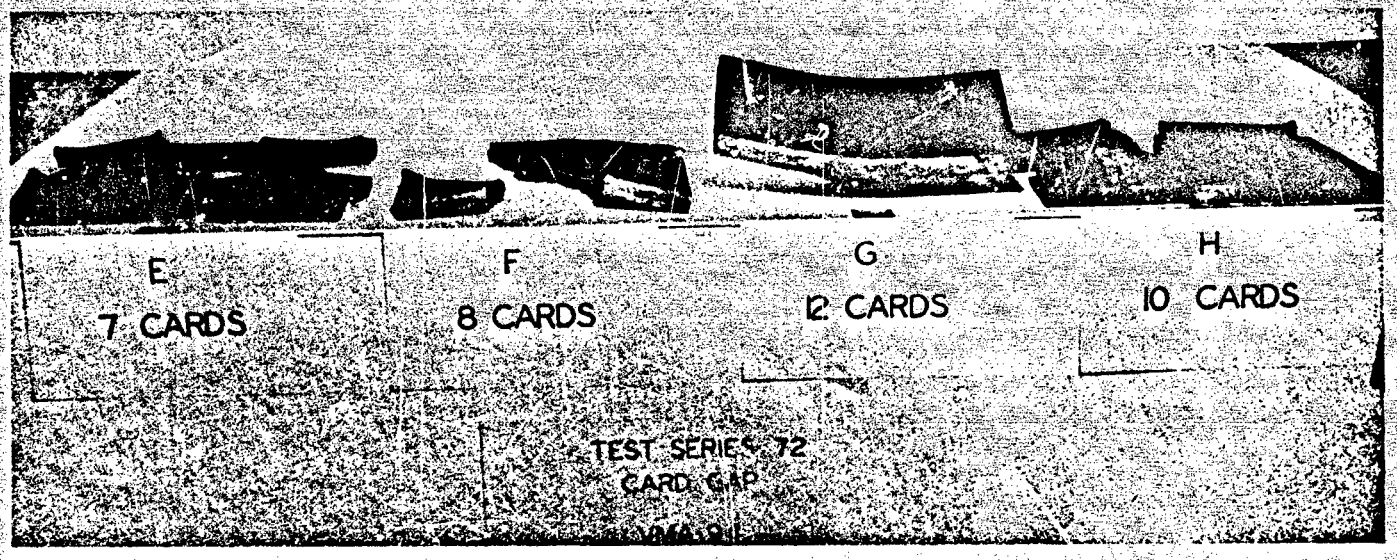
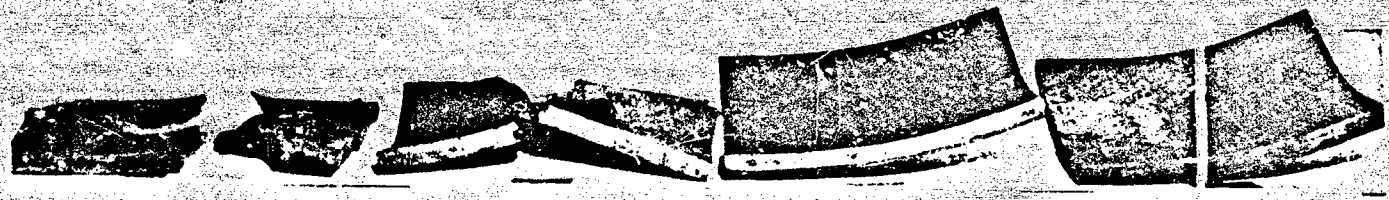


Figure D-4. Card Gap Test Witness Plates



A
0 CARDS

B
8 CARDS

C
16 CARDS

D
12 CARDS

TEST SERIES 73
CARD GAP

EFEM-33 WITH
0.2 % SILANOX 101

Figure D-5. Card Gap Test Witness Plates

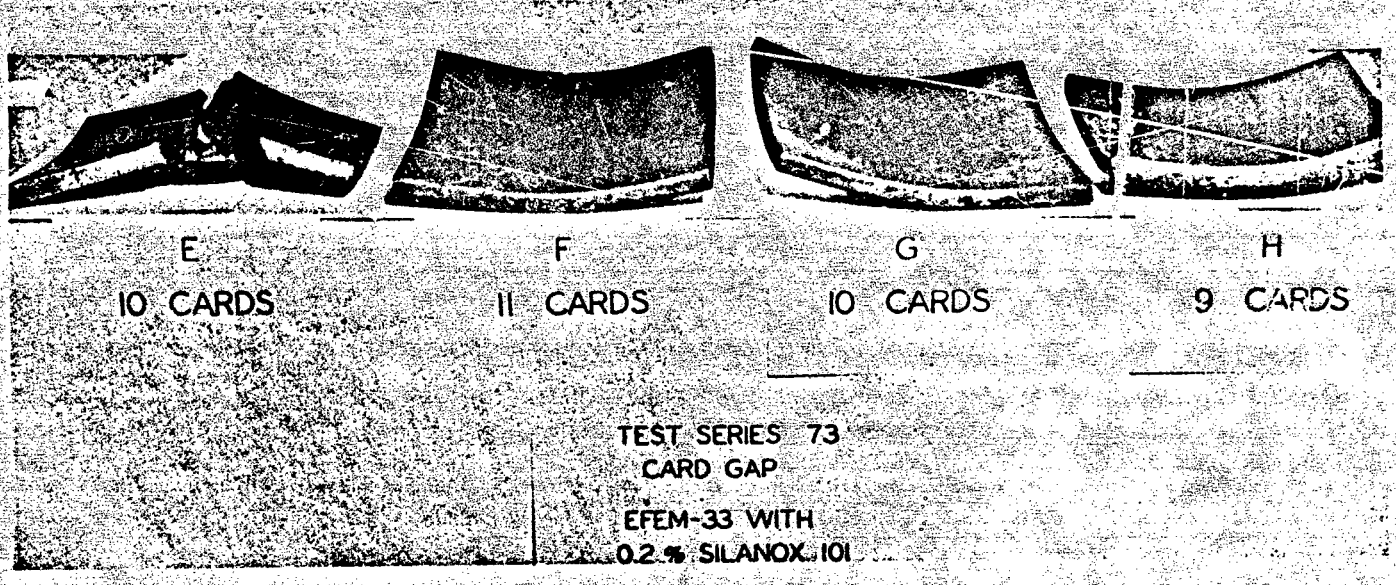


Figure D-6. Card Gap Test Witness Plates

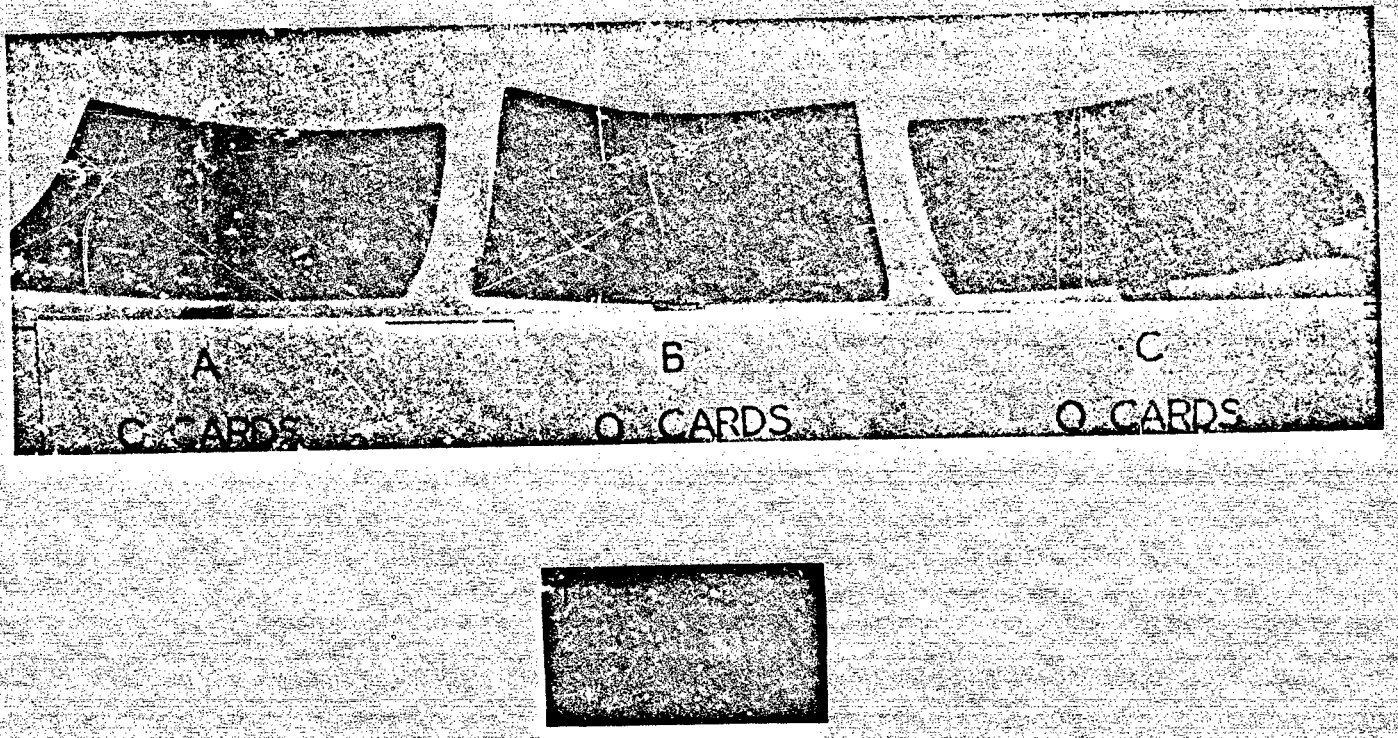


Figure D-7. Card Gap Test Witness Plates

Figure D-8

Card Gap Test Witness Plates

Figure D-9

Card Gap Test Witness Plates

Figure D-10

Card Gap Test Witness Plates

A
0 CARDS

B
0 CARDS

C
0 CARDS

TEST SERIES 86
CARD GAP
VMA-93 LIGHTLY TAMPED

Figure D-11

Card Gap Test Witness Plates

Figure D-12

Lead Column Test Witnesses

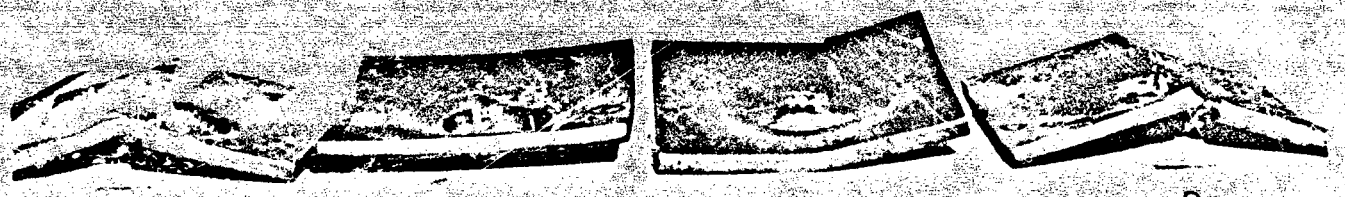
A
0 CARDS

B
0 CARDS

C
0 CARDS

TEST SERIES 79
CARD GAP
WATER

Figure D-13
Card Gap Test Witness Plates



D-19

A
0 CARDS

B
8 CARDS

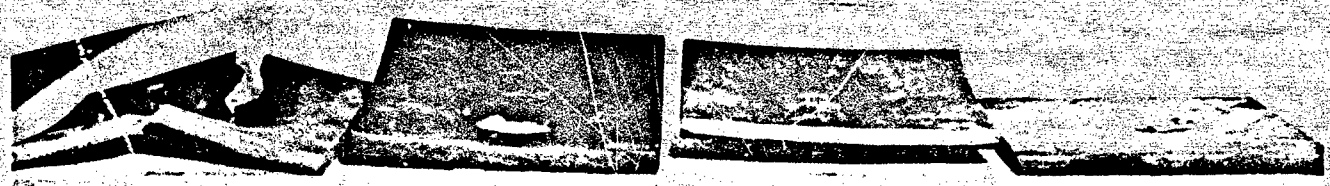
C
16 CARDS

D
32 CARDS

TEST SERIES 80
CARD GAP
25 micron RDX

Figure D-14

Card Gap Test Witness Plates



E
64 CARDS

F
128 CARDS

G
256 CARDS

H
512 CARDS

TEST SERIES 80
CARD GAP
25 micron RDX

Figure D-15
Card Gap Test Witness Plates

I
384 CARDS

J
320 CARDS

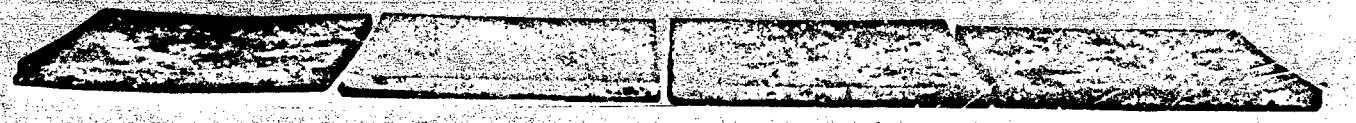
K
256 CARDS

TEST SERIES 80
CARD GAP
25 micron RDX

Figure D-16

Card Gap Test Witness Plates

Figure IV- D-17
Hole in Card Gap Witness Plate Punched
by Detonation



A
0 CARDS
WATER ONLY

B
0 CARDS

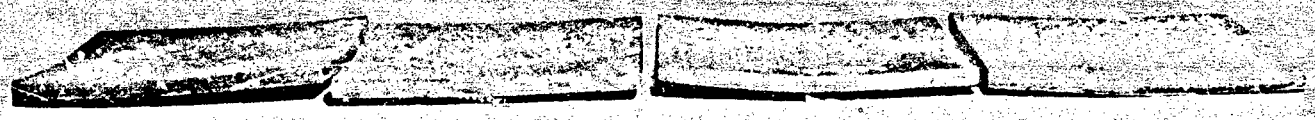
C
0 CARDS

D
0 CARDS

TEST SERIES 81
CARD GAP
EMULSION FOR FREEZE DRYING, SHOT WITH 165°F. WATER IN RESERVOIR

Figure D-18

Card Gap Test Witness Plates



A
0 CARDS
LN_2 ONLY

B
0 CARDS

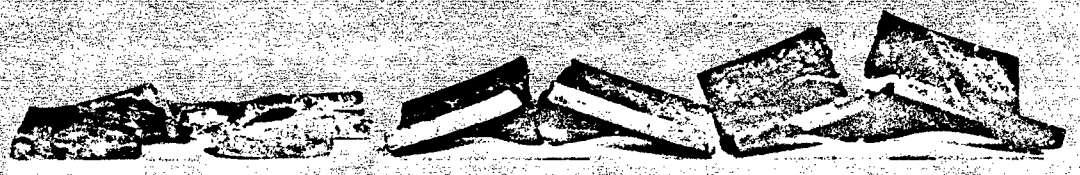
C
0 CARDS

D
0 CARDS

TEST SERIES 82
CARD GAP
FROZEN EMULSION IN
LIQUID NITROGEN

Figure D-19

Card Gap Test Witness Plates



A
0 %

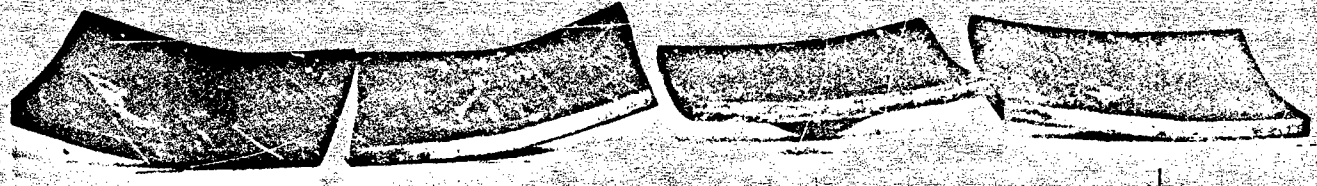
0 %

0
0%

TEST SERIES 92
CARD GAP
VMA-93 WITH VARYING
CONTENTS OF SORBEADS
AND AT VARYING SAMPLE
PACKED DENSITIES
ALL SHOTS AT ZERO CARDS

Figure D-20

Card Gap Test Witness Plates



TEST SERIES 92
CARD GAP
VMA-93 WITH VARYING
CONTENTS OF SORBEADS
AND AT VARYING SAMPLE
PACKED DENSITIES
ALL SHOTS AT ZERO CARDS

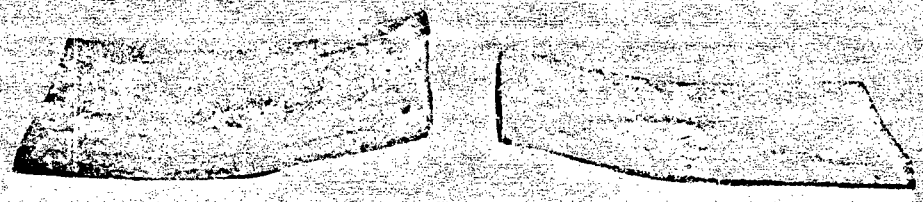
Figure D-21

Card Gap Test Witness Plates

TEST SERIES 92
CARD GAP
VMA-93 WITH VARYING
CONTENTS OF SORBEADS
AND AT VARYING SAMPLE
PACKED DENSITIES
ALL SHOTS AT ZERO CARDS

Figure D-22

Card Gap Test Witness Plates



C

35%

D

50%

TEST SERIES 92
CARD GAP
VMA-93 WITH VARYING
CONTENTS OF SORBEADS
AND AT VARYING SAMPLE
PACKED DENSITIES
ALL SHOTS AT ZERO CARDS

HAND-PACKED

Figure D-23

Card Gap Test Witness Plates

TEST SERIES 92
CARD GAP
VMA-93 WITH VARYING
CONTENTS OF SORBEADS
AND AT VARYING SAMPLE
PACKED DENSITIES
ALL SHOTS AT ZERO CARDS
LOOSELY FILLED

Figure D-24

Card Gap Test Witness Plates

A
0 %

B
0 %

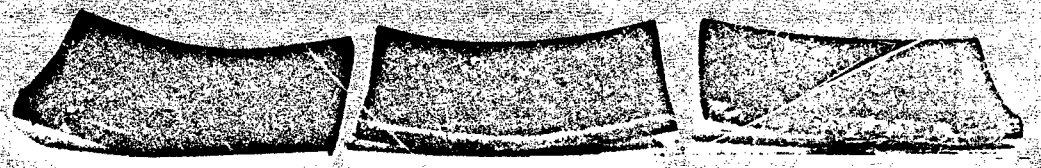
C
0 %

TEST SERIES 93
CARD GAP
EFEM-33 WITH 0.2 %
SILANOX 101
WITH VARYING CONTENTS
OF SORBEADS
ALL SHOTS AT ZERO CARDS

HAND-PACKED

Figure D-25

Card Gap Test Witness Plates



D
10 %

E
10 %

F
10 %

TEST SERIES 93
CARD GAP
EFEM-33 WITH 0.2 %
SILANOX 101
WITH VARYING CONTENTS
OF SORBEADS
ALL SHOTS AT ZERO CARDS

HAND-PACKED

Figure D-26

Card Gap Test Witness Plates

A B C

LOOSELY FILLED

TEST SERIES 95
CARD GAP
FDI 43 AT VARYING
DENSITIES
ALL SHOTS AT ZERO
CARDS

Figure D-27. Card Gap Test Witness Plates

D E F

HAND-PACKED

TEST SERIES 95

CARD GAP

FDI-43 AT VARYING

DENSITIES

ALL SHOTS AT ZERO

Figure D-28. Card Gap Test Witness Plates

A B

WITHOUT

TEST SERIES 105
CARD GAP
FDI-43D/FDB-6B WITHOUT
SORBEADS
ALL SHOTS AT ZERO CARDS
WITH AND WITHOUT
STANDOFFS, AS NOTED
HAND-PACKED TO HIGH
DENSITY

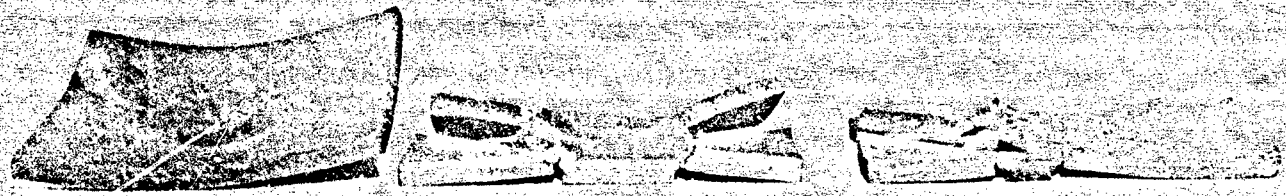
Figure D-29. Card Gap Test Witness Plates

C D

WITH

TEST SERIES 105
CARD GAP
FDI-43D/FDB-6B WITHOUT
SORBEADS
ALL SHOTS AT ZERO CARDS
WITH AND WITHOUT
STANDOFFS, AS NOTED
HAND-PACKED TO HIGH
DENSITY

Figure D-30. Card Gap Test Witness Plates



A B C

WITHOUT

TEST SERIES 106
CARD GAP
EFEM-63A WITH 0.4 %
SILANOX 101
ALL SHOTS AT ZERO CARDS
WITH AND WITHOUT
STANDOFFS, AS NOTED
LOOSE-FILLED

Figure D-31. Card Gap Test Witness Plates

Figure D-32. Card Gap Test Witness Plates

G

WITH

TEST SERIES 106
CARD GAP
EFEM-63A WITH 0.4 %
SILANOX 101
ALL SHOTS AT ZERO CARDS
WITH AND WITHOUT
STANDOFFS, AS NOTED
LOOSE-FILLED

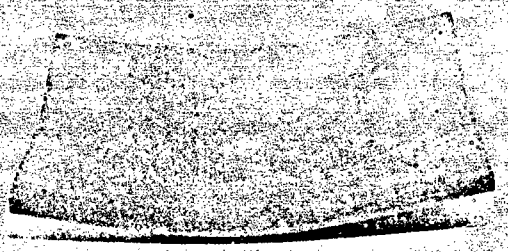
Figure D-33. Card Gap Test Witness Plates

Figure D-34. Card Gap Test Witness Plates

TEST SERIES 108-
CARD GAP
VMA-76 WITHOUT SORBEADS
ALL SHOTS AT ZERO CARDS
EXCEPT AS NOTED
ALL FIRED WITH STANDOFFS

LOOSE-FILLED

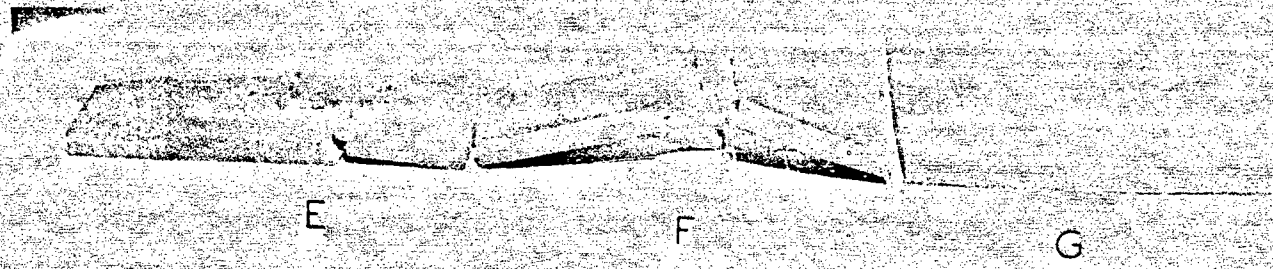
Figure D-35. Card Gap Test Witness Plates



D

TEST SERIES 108
CARD GAP
VMA-76 WITHOUT SORBEADS
ALL SHOTS AT ZERO CARDS
EXCEPT AS NOTED
ALL FIRED WITH STANDOFFS
LOOSE-FILLED

Figure D-36. Card Gap Test Witness Plates



TEST SERIES 108
CARD GAP
VMA-76 WITHOUT SORBEADS
ALL SHOTS AT ZERO CARDS
EXCEPT AS NOTED
ALL FIRED WITH STANDOFFS
HAND-PACKED

Figure D-37. Card Gap Test Witness Plates

APPENDIX E

LEAD COLUMN TESTS

COMMENTS, DATA, PHOTOGRAPHS

TABLE E-I

LEAD COLUMN TEST[a] RESULTS ON UFAP MATERIALS

Date Tested	Test No.	Material Type	Material Source	Composition	% of T. M. D.[b]	Particle Size, microns	Results[c]	Final Column[d] Height, inches
6/26/72	75 A	Dry Powder	VMA-94	99.6% AP, 0.4% HX-868	30	0.57	pos.	3 3/8
	B						pos.	3 7/16
	76 A	Dry Powder	EFEM-33	97.8% AP, 0.2% Silanox 101	30	1.60	neg.	4
	B				30		pos.	3 3/8
	C				30		pos.	3 1/2
	77 A	Dry Powder	EFEM-33	100% AP	30	1.60	neg.	4
	B				30		neg.	4
	C				30		neg.	4
	D				30		neg.	4
	E				30		neg.	4
	78 A	Dry Powder	FDI-10A	98.95% AP, 0.99% Nopcogen 0.06% Johnsons Wax	30	0.73	pos.	3 3/8
	B				30		pos.	3 7/16

NOTES:

a. A sample of material is charged to a cardboard tube such that the material is 2 1/32 inches long. The sample is placed on top of a lead column, then initiated with a No. 8 blasting cap positioned on top of the sample. If the sample detonates, the lead column is deformed to a mushroom shape on top, and the length is shortened in proportion to the explosive energy generated.

b. Percent of theoretical material density (1.95 gms/cc for AP). Denotes the degree of packing the material underwent.

c. Negative indicates no mushrooming or shortening of the lead column occurred. Positive indicates a detonation occurred in the material, causing mushrooming and shortening of the column.

d. The column originally is 4 inches long by 1 1/2 inches in diameter.

TABLE E-II

LEAD COLUMN TESTS[a] ON UFAP MATERIALS

Date Tested	Test No.	Material Form	UFAP Source	Composition	% of T.M.D.[b]	Particle Size, Microns	Results[c]	Final Column[d] Height, inches
8/3/72	91 A	Dry Powder, Loosely packed	VMA-93	99.6% AP, 0.4% HX-868	28	0.0	positive	3 7/16
	B				28		positive	3 1/2
	C				28		positive	3 3/8

NOTES:

a. The sample and test set-up are described in detail in the notes to Table IV-II of the July, 1972, report.

b. Percent of theoretical material density (1.95 gms./cc. for AP). Denotes the degree of packing the material underwent during loading.

c. Negative indicates no mushrooming or shortening of the lead column occurred. Positive indicates a detonation occurred in the material, causing mushrooming and shortening of the column.

d. The column originally is 4 inches long by 1 1/2 inches in diameter.

TABLE E-III

LEAD COLUMN TESTS[a] ON UFAP MATERIALS

Date Tested	Test No.	Material Form	UFAP Source	Composition	% of T.M.D.[b]	Particle Size, Microns	Results[c]	Final Column Height, inches[d]
8/8/72	94 A	Dry Powder, Hand-packed	VMA-93	99.6% AP, 0.4% HX-868	91	0.60	positive	3 7/16
	B			10% Sorbeads, 90% as above	91		positive	3 7/16
	C			25% Sorbeads, 75% as above	34		positive	3 1/2
	D			35% Sorbeads, 65% as above	33		negative	4
	E			50% Sorbeads, 50% as above	39		negative	4
	F	Dry Powder, Loosely filled		10% Sorbeads, 90% as above	21		positive	3 11/16
	G			25% Sorbeads, 75% as above	20		negative	4
	H			35% Sorbeads, 65% as above	25		negative	4
	I	Dry Powder, Lightly tapped to higher density		25% Sorbeads, 75% as above	27		negative	4

NOTES: a. The sample and test set-up are described in detail in the notes to Table IV-II of the J[illegible] 1972, report.

b. Percent of theoretical material density (1.95 gms./cc. for AP). Denotes the degree of packing the material underwent during loading.

c. Negative indicates no mushrooming or shortening of the lead column occurred. Positive indicates a detonation occurred in the material, causing mushrooming and shortening of the column.

d. The column originally is 4 inches long by 1 1/2 inches in diameter.

TABLE E-IV

LEAD COLUMN TESTS[a] ON FREEZE-DRIED UFAP MATERIALS

Date Tested	Test No.	Material Form	UF/I Source	Composition	% of TMD[b]	Particle Size, microns	Results[c]	Final Column[d] Height, inches
1/13/73	96A	Dry Powder Loosely Filled	FDI-43D	96.1% AP, 2.0% DOA, 1.9% Nopcogen 16-B	19.5	0.62	Positive	3-1/2
	B				19.0		Positive	3-1/2
1/15/73	97A	Dry Powder and Sorbeads Loosely Filled	FDI-43D	70% FDI-43D, 30% Sorbeads	20.5	0.62	Positive	3-9/16
	B				20.5		Positive	3-1/2
	C				21.0		Positive	3-1/2
	D				20.5		Positive	3-1/2
	E				20.5		Positive	3-7/16

NOTES:

a. A sample of material is charged to a cardboard tube such that the material is 2 1/32 inches long. The sample is placed on top of a lead column, then initiated with a No. 8 blasting cap positioned on top of the sample. If the sample detonates, the lead column is deformed to a mushroom shape on top, and the length is shortened in proportion to the explosive energy generated.

b. Percent of theoretical material density (1.95 gms/cc for AP). Denotes the degree of packing the material underwent.

c. Negative indicates no mushrooming or shortening of the lead column occurred. Positive indicates a detonation occurred in the material, causing mushrooming and shortening of the column.

d. The column originally is 4 inches long by 1 1/2 inches in diameter.

TABLE E-V

LEAD COLUMN TESTS[a] ON FREEZE-DRIED UFAP MATERIALS

Date Tested	Test No.	Material Form	UFAP Source	Composition	% of T.M.D.[b]	Particle Size Microns	Results[c]	Final Column[d] Height, Inches
1/15/73	96A	Dry Powder, loosely filled	FDL-43D	96.1% AP, 2.0% DOA, 1.9% Isoperon 1600	19.5	0.62	Positive	3 1/2
	B				19.0		Positive	3 1/2
1/15/73	97A	Dry Powder and sorbeads, loosely filled	FDL-43A	70% FDL-43D, 30% Sorbeads	20.5	0.62	Positive	3 9/16
	B				20.5		Positive	3 1/2
	C				20.0		Positive	3 1/2
	D				20.5		Positive	3 1/2
	E				20.5		Positive	3 7/16
1/17/73	99A[e]	Dry Powder and Sorbeads, loosely filled	FDL-43D/ FDB-6B	70% FDL-43D/FDL-6B, 30% Sorbeads	21.5	0.62/0.56	Positive	3 9/16
	B				20.4		Positive	3 1/2
	C				21.4		Positive	3 1/2
	D				21.5		Positive	3 5/8
	E				22.0		Positive	3 11/16
	F				16.3		Positive	3 1/4
1/18/73	91A	Dry Powder and Sorbeads, loosely filled	FDL-43D/ FDB-6B	65% UFAP, 35% Sorbeads	22.1	0.62/0.56	Positive	3 1/2
	B				22.3		Positive	3 1/2
1/19/73	100A	Dry Powder and Sorbeads, loosely filled	FDL-43D/ FDB-6	60% UFAP, 40% Sorbeads	21.6	0.62/0.56	Positive	3 1/2
	B				22.1		Positive	3 1/2
2/9/73	101A	Dry Powder and Sorbeads, loosely filled	FDL-43D/ FDB-6B	50% UFAP, 50% Sorbeads	27.2	0.62/0.56	Positive	3 11/16
	B				26.9		Positive	3 11/16
2/9/73	102A	Dry Powder and Sorbeads, hand packed	FDL-43D/ FDB-6B	70% UFAP, 30% Sorbeads	30.0	0.62/0.56	Positive	3 1/4
	B				31.1		Positive	3 5/16
2/9/73	103A	Dry powder and Sorbeads, hand packed	FDL-43D/ FDB-6B	65% UFAP, 35% Sorbeads	29.3	0.62/0.56	Positive	3 5/16
	B				31.4		Positive	3 1/2
2/9/73	104A	Dry Powder and Sorbeads, hand packed	FDL-43D/ FDB-6B	50% UFAP, 50% Sorbeads	32.7		Positive	3 5/8
	B				31.0		Positive	3 1/2
2/10/73	107A	Dry powder hand packed	FDB-6B		31.9	0.56	Positive	3 1/8
	E				31.8		Positive	3 3/16

NOTES:

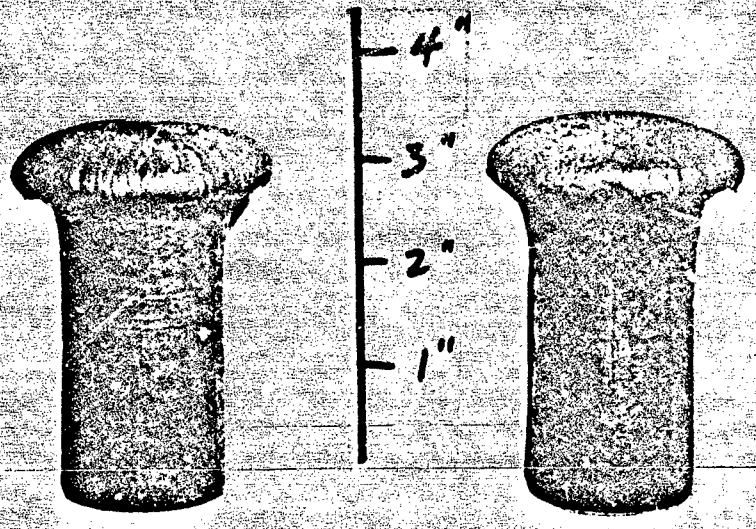
a. The sample and test set-up are described in detail in the notes to Table IV-II of the July, 1972, report.

b. Percent of theoretical material density (1.95 gms./cc. for AP). Denotes the degree of packing the material underwent during loading.

c. Negative indicates no mushrooming or shortening of the lead column occurred. Positive indicates a detonation occurred in the material, causing mushrooming and shortening of the column.

d. The column originally is 4 inches long by 1 1/2 inches in diameter.

e. This series consisted of a number of special sample material configurations, varying in length and diameter from the standard. The dimensions given below are those of the AP charge A: 2.031 x 5.25 (standard size); B: 2.031 x 7.25; C: 2.031 x 11.25; D: 1.50 x 5.25; E: 1.50 x 9.63; F: 3.50 x 5.25.



A B

TEST SERIES 75
LEAD COLUMN

Figure E-1 Lead Column Test Witnesses

TEST SERIES 76
LEAD COLUMN
EFEM-33 WITH
0.2% SILANOX 101

Figure E-2 Lead Column Test Witnesses

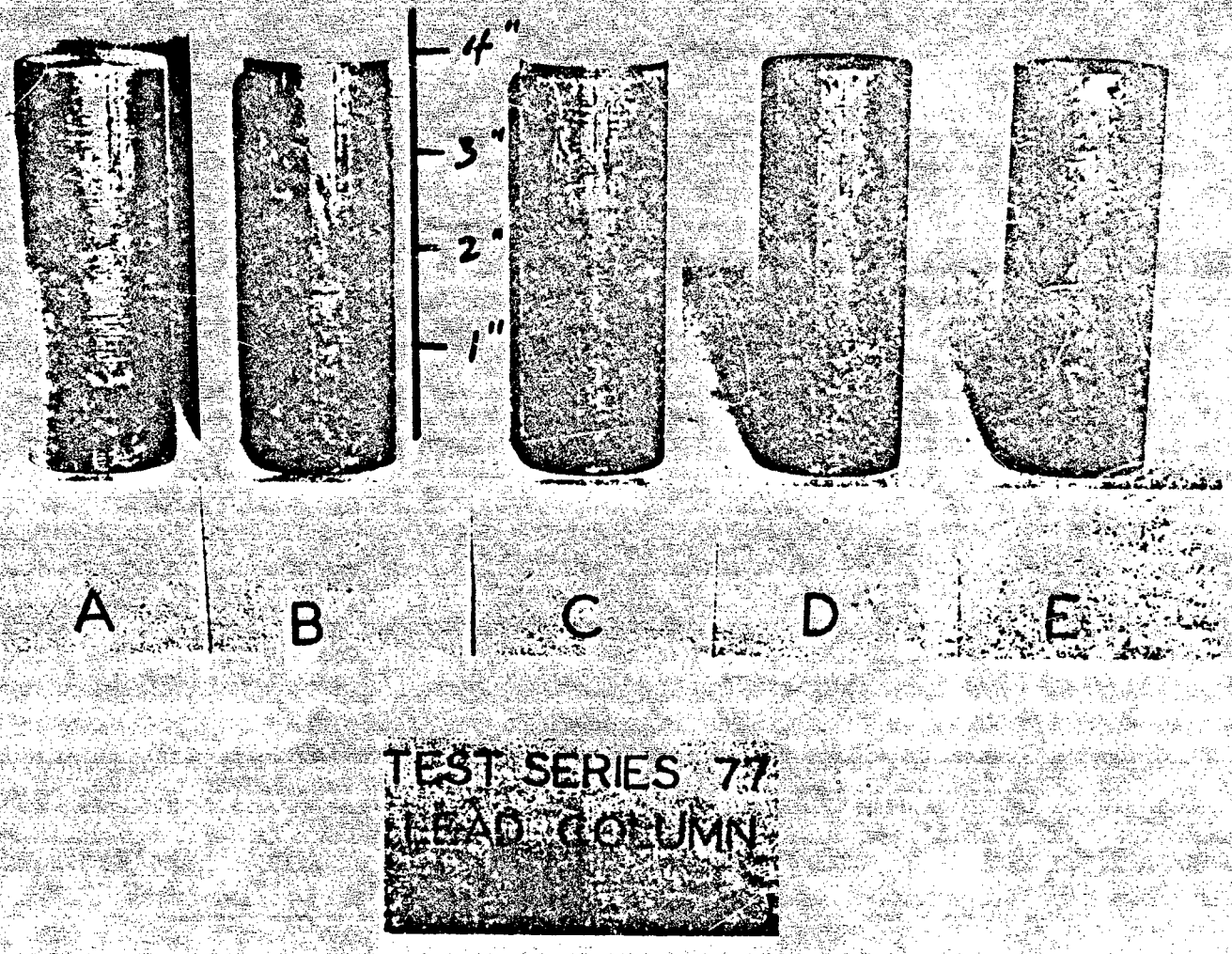
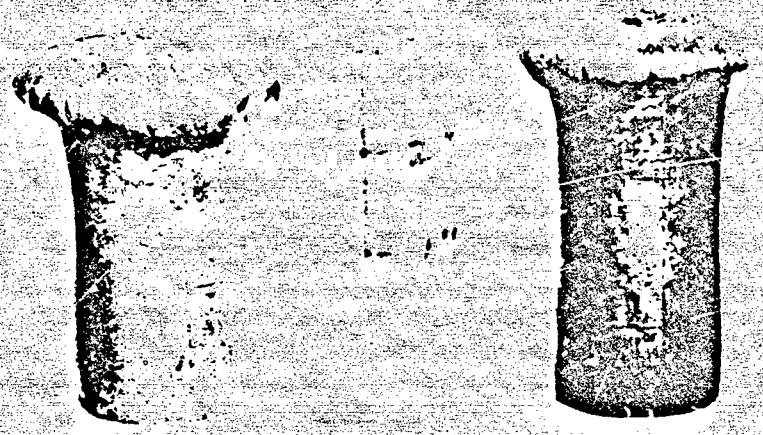


Figure E-3 Lead Column Test Witnesses



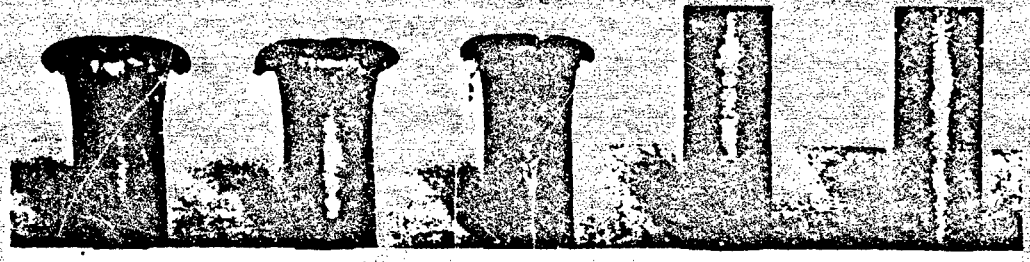
A B

TEST SERIES 78
LEAD COLUMN

FDI-10 A

Figure E-4 Lead Column Test Witnesses

Figure E-5. Lead Column Test Witnesses



A	B	C	D	E
0%	10%	25%	35%	50%

TEST SERIES 94
LEAD COLUMN
VMA-93 WITH VARYING
CONTENTS OF SORBEADS
AND AT VARYING SAMPLE
PACKED DENSITIES

HAND-PACKED

Figure E-6 Lead Column Test Witnesses

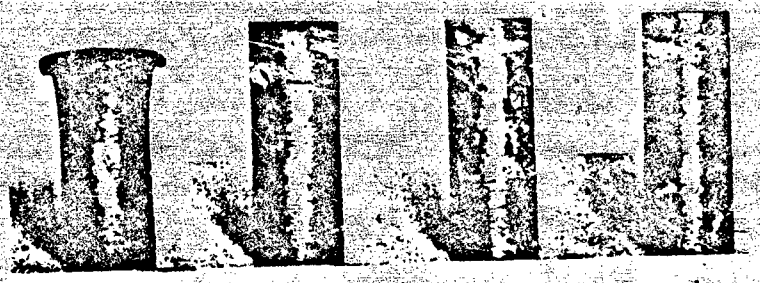
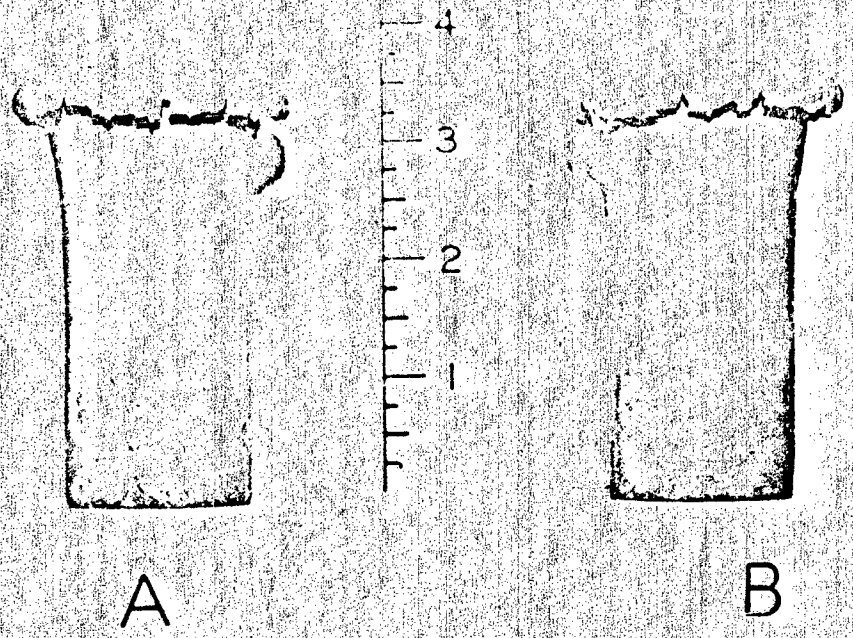
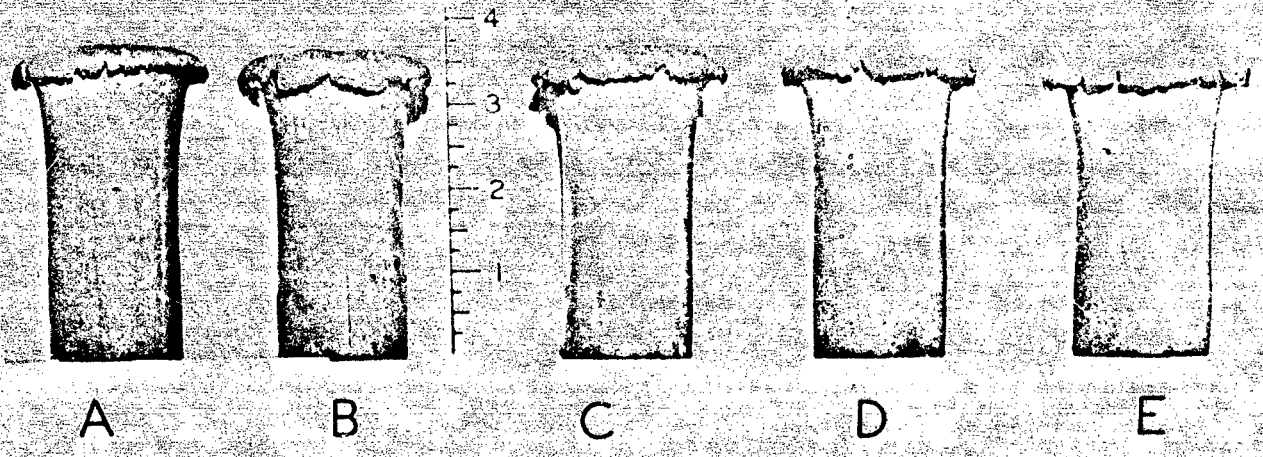


Figure E-7 Lead Column Test Witnesses



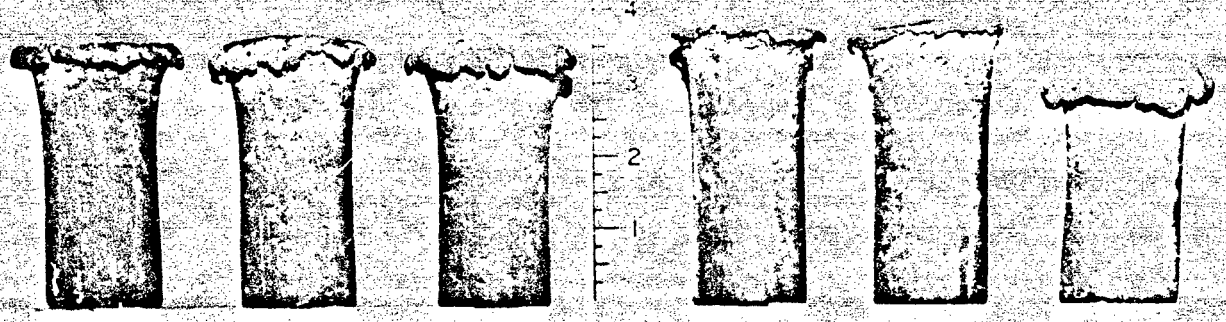
TEST SERIES 96
LEAD COLUMN
FDI-43 LOOSELY FILLED
WITHOUT SORBEADS

Figure E-8 Lead Column Test Witness



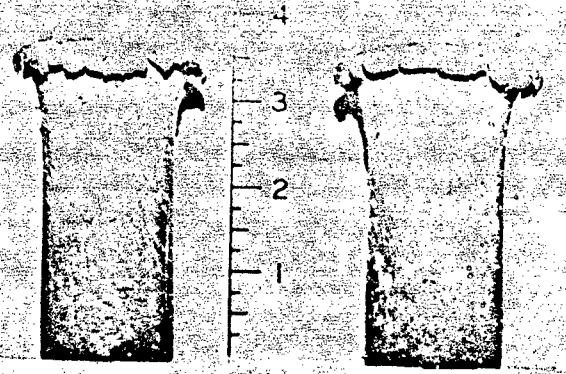
TEST SERIES 97
LEAD COLUMN
FDI-43 LOOSELY FILLED
WITH 30% SORBLADS

Figure E-9 Lead Colu[illegible]t Witnesses



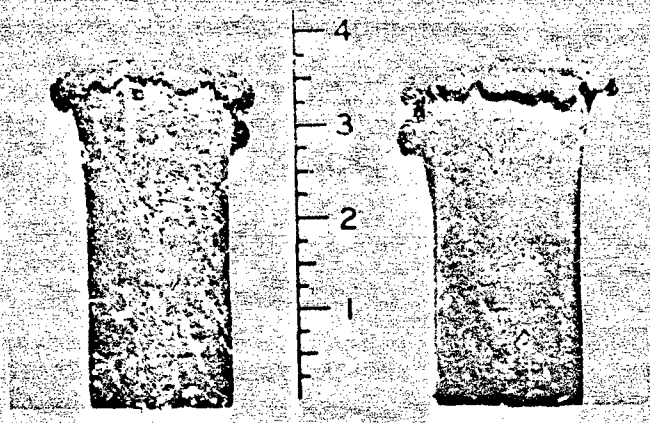
TEST SERIES 98
MODIFIED LEAD COLUMN
FDI-43D/FDB-6B WITH 30%
SORBEADS
LOOSE-FILLED INTO VARYING
SAMPLE SIZES

Figure E-10 Lead Column Test Witnesses



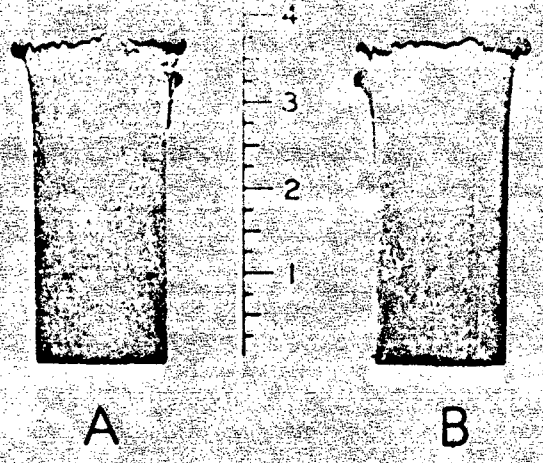
TEST SERIES 99
LEAD COLUMN
FDI-43D/FDB-6B WITH 35%
SORBEADS
LOOSE-FILLED

Figure E-11 Lead Column Test Witnesses



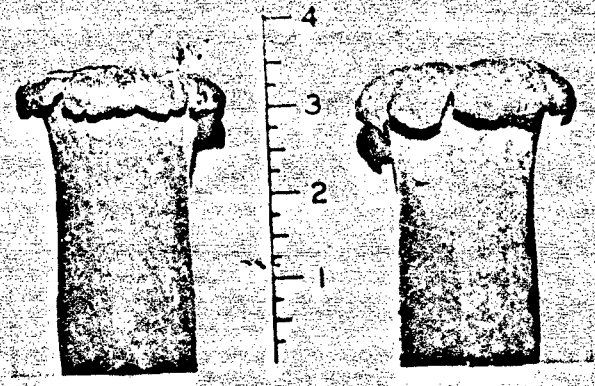
TEST SERIES 100
LEAD COLUMN
FDI-43D/FDB-6B WITH 40%
SORBEADS

Figure E-12 Lead Column Test Witnesses



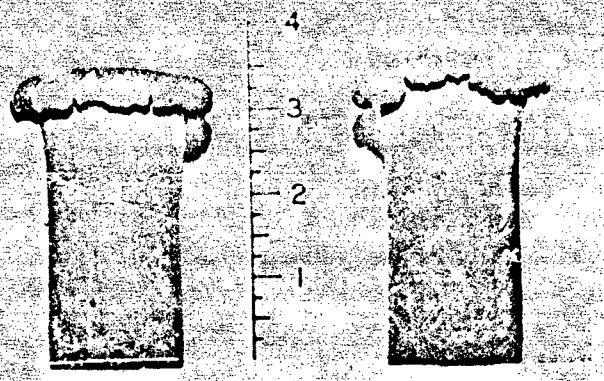
TEST SERIES 101
LEAD COLUMN
FDI-43D/FDB-6B WITH 50%
SORBEADS
LOOSE-FILLED

Figure E-13 Lead Column Test Witnesses



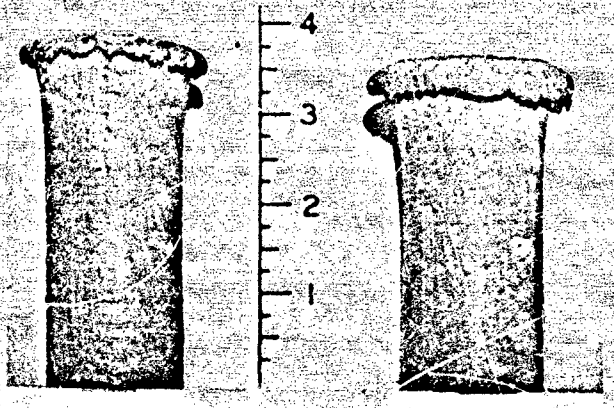
TEST SERIES 102
LEAD COLUMN
FDI-43D/FDB-6B WITH 30%
SORBEADS
HAND-PACKED

Figure E-14 Lead Column Test Witnesses



TEST SERIES 103
LEAD COLUMN
FDI-43D/FDB-6B WITH 35%
SORBEADS
HAND-PACKED

Figure E-15 Lead Column Test Witnesses



TEST SERIES 104
LEAD COLUMN
FDI-43D/FDB-6B WITH 50%
SORBEADS
HAND-PACKED

Figure E-16 Lead Column Test Witnesses

Figure E-17 Lead Column Test Witnesses

APPENDIX F

PACKING FRACTION RESPONSE SURFACES

Abstracted From

OPTIMIZING THE PACKING OF AMMONIUM PERCHLORATE AND ALUMINUM PARTICLE SIZE MIXTURES FOR SOLID PROPELLANTS (U)

Bernard J. Alley

Hiram W. H. Dykes

AMC RK-TR-67-2

January 1967

Packing Fraction Surface Response

The packing fraction of solids in a propellant has a pronounced effect on the rheological properties of the uncured slurry[1,2,3,4] and presumably on the equilibrium modules[5,6] and ultimate tensile properties of the cured propellant. The packing fraction is the volume fraction of the solids when they are packed to minimum volume, and therefore is independent of the actual solids loading in the propellant. The development of suitable high density-impulse propellants and other propellants having optimum properties require that the solids be efficiently packed. The solids mixture with the largest packing fraction gives propellant with potentially the highest solids loading capability and the minimum mix viscosity for a fixed solids loading.

To optimize a mixture of ammonium perchlorate and aluminum particle sizes from the standpoint of propellant rheological, physical, and ballistic properties, the packing fractions of all possible mixtures that might be considered must be known before hand. The determination of these packing fractions for multicomponent mixtures by strictly experimental procedures[4,7,8] is impractical. Based on a theoretical consideration of the packing probability of monodisperse spheres with respect to their diameters, Wiech and Moser[9] derived the following expression for estimating packing fractions:

$$\epsilon = 0.37 \left[1 + 0.707 \exp - \left(\frac{\beta s}{d} \right) \right].$$

Where s is a measure of the attractive force between particles, d is the particle diameter, and β is a constant. Alley and Dykes[10] demonstrated in an earlier paper that the packing fractions of multicomponent mixtures of ammonium perchlorate and aluminum particle sizes can be satisfactorily estimated using a special cubic statistical model suggested by Scheffé.[11] Regression equations, derived with a comparatively small amount of data, permit the estimations of packing fractions for any number and combination of sizes under consideration. The experimental data points are selected in accordance with a special cubic simplex lattice experimental design.[11,12]

It was previously demonstrated[10] that the packing fraction response surfaces for multicomponent mixtures of ammonium perchlorate and aluminum powders can be well represented by a special cubic response function suggested by Scheffé.[11] The canonical form of the special cubic model for a mixture of particle size components is

$$\epsilon = \sum \beta_i X_i + \sum \beta_{ij} X_i X_j + \sum \beta_{ijk} X_i X_j X_k + E \qquad (3)$$

$$1 \le i \le q \qquad 1 \le i < j \le q \qquad 1 \le i < j < k \le q$$

where ϵ is the measured packing fraction (response), β coefficients are constants, X's are weight fractions of the particle size components, and E is the random error associated with ϵ. The total composition of the mixture is restricted by the physical limitation

$$\sum_{i=1}^{q} X_i = 1$$

where $0 \le X_i \le 1$.

Scheffé introduced the concept of simplex lattice designs for q component mixtures when the experimental response depends only on the proportions of components in the mixtures. Simplex lattice designs are arrangements of experimental sample points on a regular-sided figure or simplex. The $[q, n]$ simplex lattice has n + 1 equally spaced points from zero to one for each component. The special cubic simplex lattice design for three components (augmented $[3, 2]$) is shown in Figure 1. The simplex for q = 3 is an equilateral triangle. The special cubic simplex lattice design for three components can be readily extended to include any number of components.

To estimate the β coefficients of Equation (3), packing fraction measurements are made at each lattice point. ϵ_i is the measured packing fraction for pure component i, ϵ_{ij} is the measured packing fraction for a 1:1 binary mixture of components i and j, and ϵ_{ijk} is the measured packing fraction for a 1:1:1 ternary mixture of components i, j, and k. The least squares estimators of the coefficients in Equation (3) are simple linear functions of these measured responses:

$$\hat{\beta}_i = \bar{\epsilon}_i \qquad (5)$$

$$\hat{\beta}_{ij} = 4\bar{\epsilon}_{ij} - 2\bar{\epsilon}_i - 2\bar{\epsilon}_j \qquad (6)$$

$$\hat{\beta}_{ijk} = 27\bar{\epsilon}_{ijk} - 12(\bar{\epsilon}_{ij} + \bar{\epsilon}_{ik} + \bar{\epsilon}_{jk}) + 3(\bar{\epsilon}_i + \bar{\epsilon}_j + \bar{\epsilon}_k) \qquad (7)$$

where $i < j < k$ and $\bar{\epsilon}_i$, $\bar{\epsilon}_{ij}$, and $\bar{\epsilon}_{ijk}$ are mean packing fraction measurements when the lattice points are replicated.

The regression equation for estimating the packing fractions of mixtures having any number or combination of components is

$$\hat{\epsilon} = \sum_{1 \le i \le q} \hat{\beta}_i X_i + \sum_{1 \le i < j \le q} \hat{\beta}_{ij} X_i X_j + \sum_{1 \le i < j < k \le q} \hat{\beta}_{ijk} X_i X_j X_k \qquad (8)$$

$\hat{\epsilon}$ is an estimate of the true mean packing fraction. The $\hat{\beta}$'s are the least squares estimators of the β's in Equation (3), and are determined by means of Equations (5), (6), and (7).

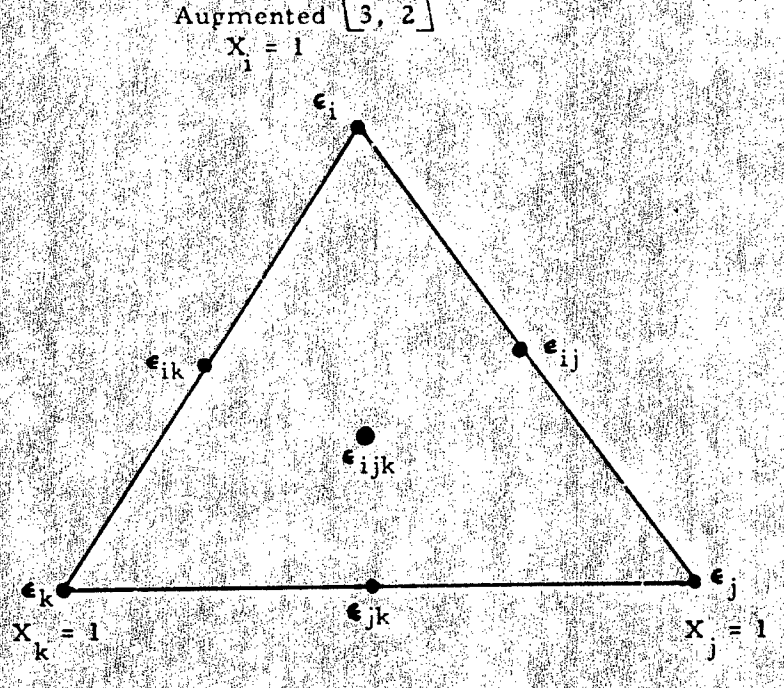


Figure 1. Graphical Representation of Special Cubic Simplex Lattice Design for Three Components

Packing fraction (wet) measurements are then made at all latice points of Figure 1. The β coefficients are calculated by means of packing fraction data at the lattice points and equations (5), (6) and (7).

The working equations, then for estimating the packing fractions of known particle size mixture compositions is

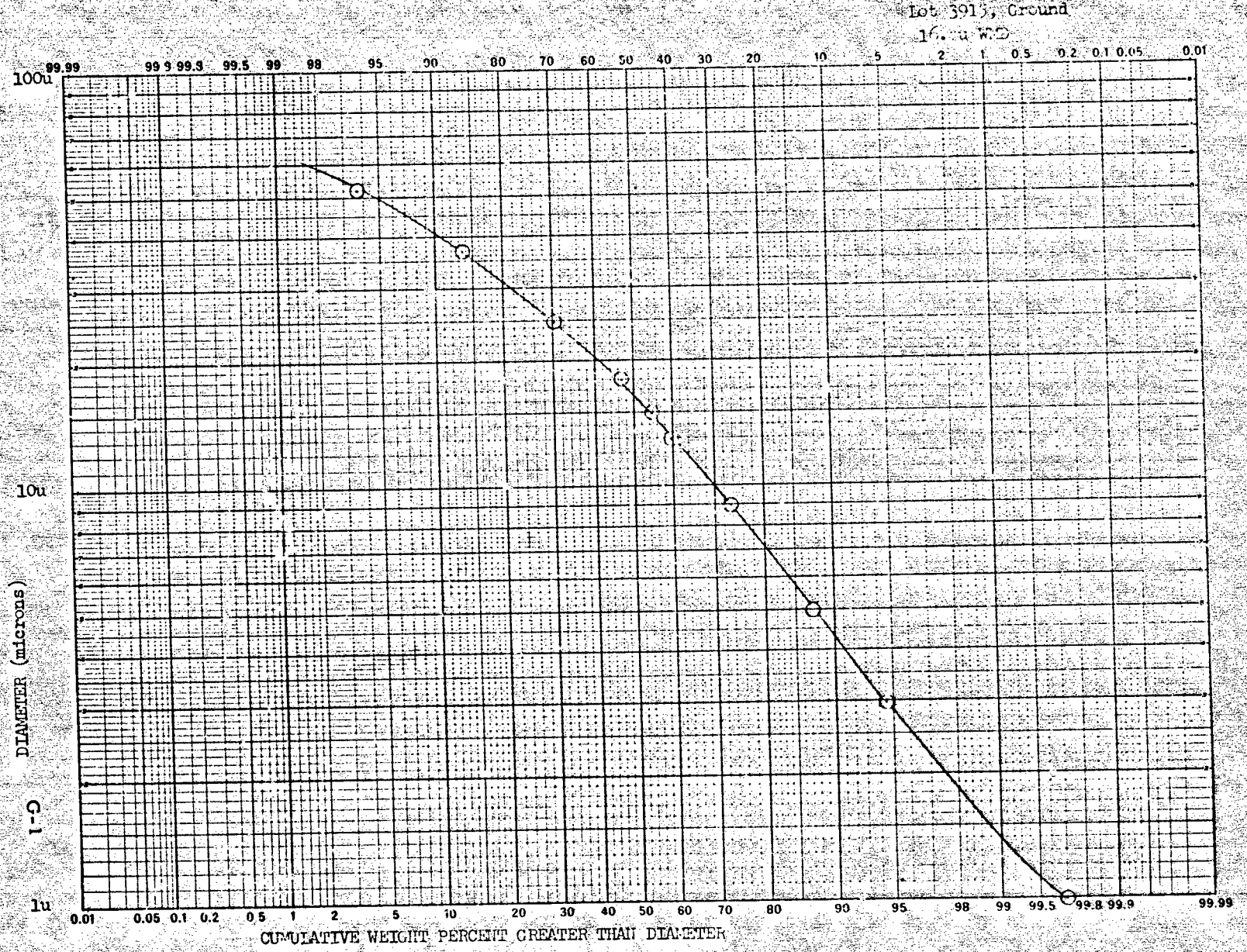
$$\hat{\zeta} = \hat{\beta}_1 X_1 + \ldots + \hat{\beta}_9 X_9 + \hat{\beta}_{12} X_1 X_2 + \ldots + \hat{\beta}_{59} X_5 X_9 + \hat{\beta}_{123} X_1 X_2 X_3 + \ldots + \hat{\beta}_{459} X_4 X_5 X_9. \quad (9)$$

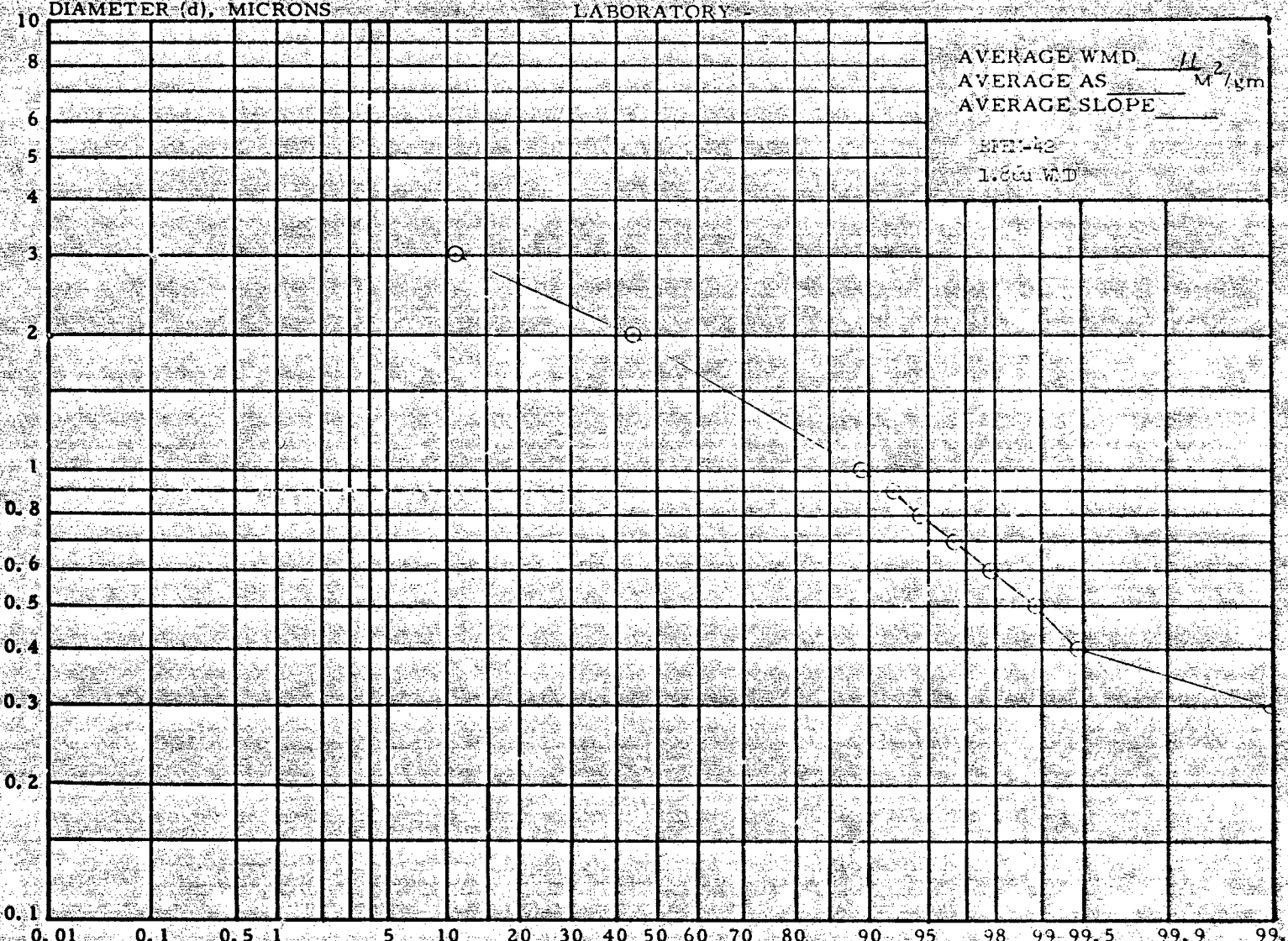
The value of a given $\hat{\beta}$ in Equation (9) is unaffected by the addition or deletion of others; therefore, only those terms are included which are needed to calculate $\hat{\zeta}$ for the particular mixture under consideration.

The estimated packing fraction response surfaces for three-component mixtures or three-component slices of q componentsmixtures can be represented by plotting contours of constant packing fraction on triangular-coordinate graphs. These graphs greatly facilitate the selection of optimum particle size mixtures during propellant development. Data fro the graphs are obtained by computer calculation using Equation (9).

APPENDIX G

UFAP PARTICLE SIZE DISTRIBUTION CURVES


Lot 3913, Ground
16. u W.D
99.99 99.9 99.8 99.5 99 98 95 90 80 70 60 50 40 30 20 10 5 2 1 0.5 0.2 0.1 0.05 0.01
100u
10u
1u
DIAMETER (microns)
0.01 0.05 0.1 0.2 0.5 1 2 5 10 20 30 40 50 60 70 80 90 95 98 99 99.5 99.8 99.9 99.99
CUMULATIVE WEIGHT PERCENT GREATER THAN DIAMETER



G-2

FIGURE____ CUMULATIVE WEIGHT PERCENT GREATER THAN DIAMETER

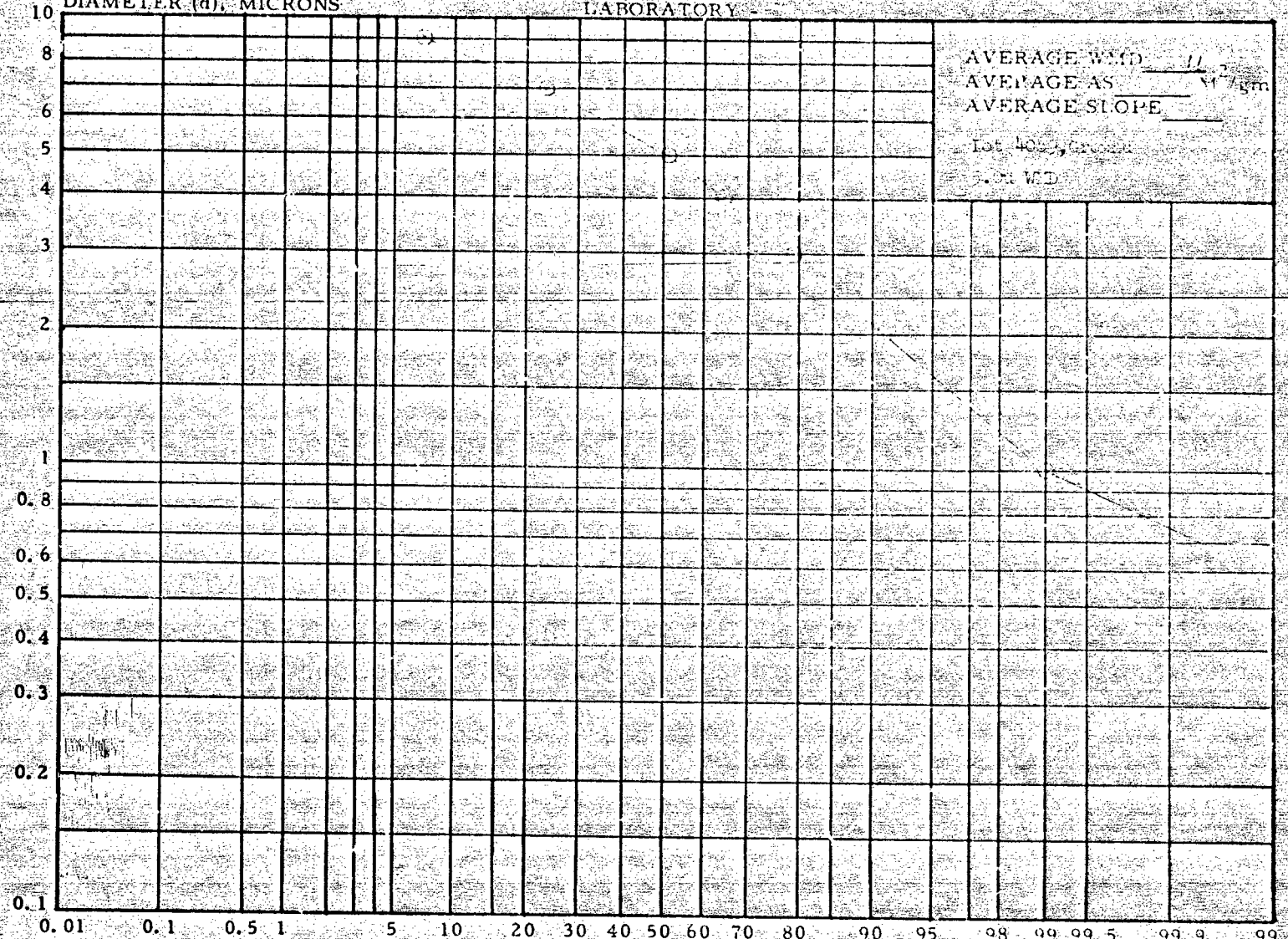

DIAMETER (d), MICRONS
LABORATORY
AVERAGE WSD
AVERAGE AS
AVERAGE SLOPE
10
8
6
5
4
3
2
1
0.8
0.6
0.5
0.4
0.3
0.2
0.1
0.01
0.1
0.5
1
5
10
20
30
40
50
60
70
80
90
95
98
99
99.5
99.9
99.99
CUMULATIVE WEIGHT PERCENT GREATER THAN DIAMETER

FIGURE C-3

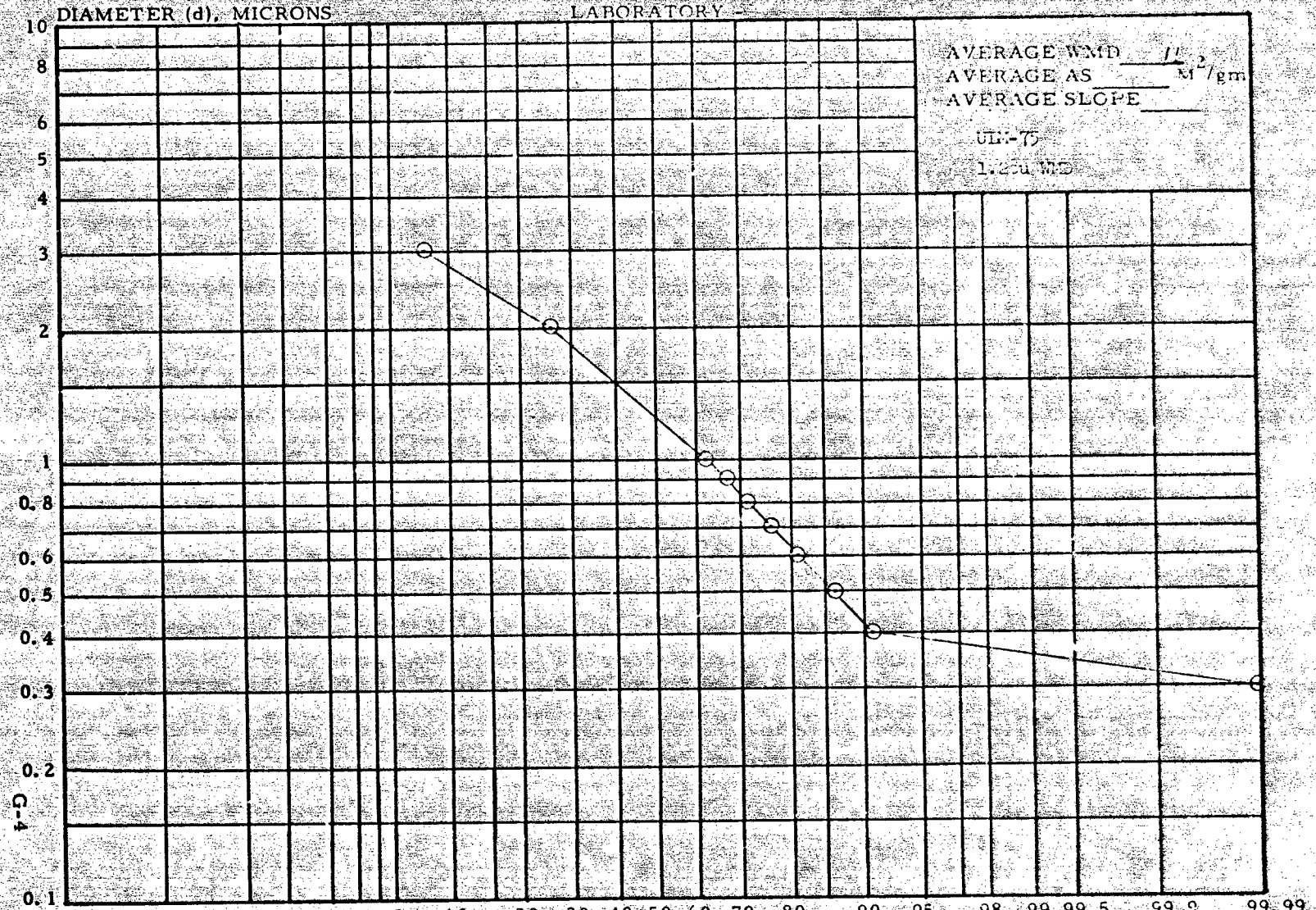


FIGURE____ CUMULATIVE WEIGHT PERCENT GREATER THAN DIAMETER

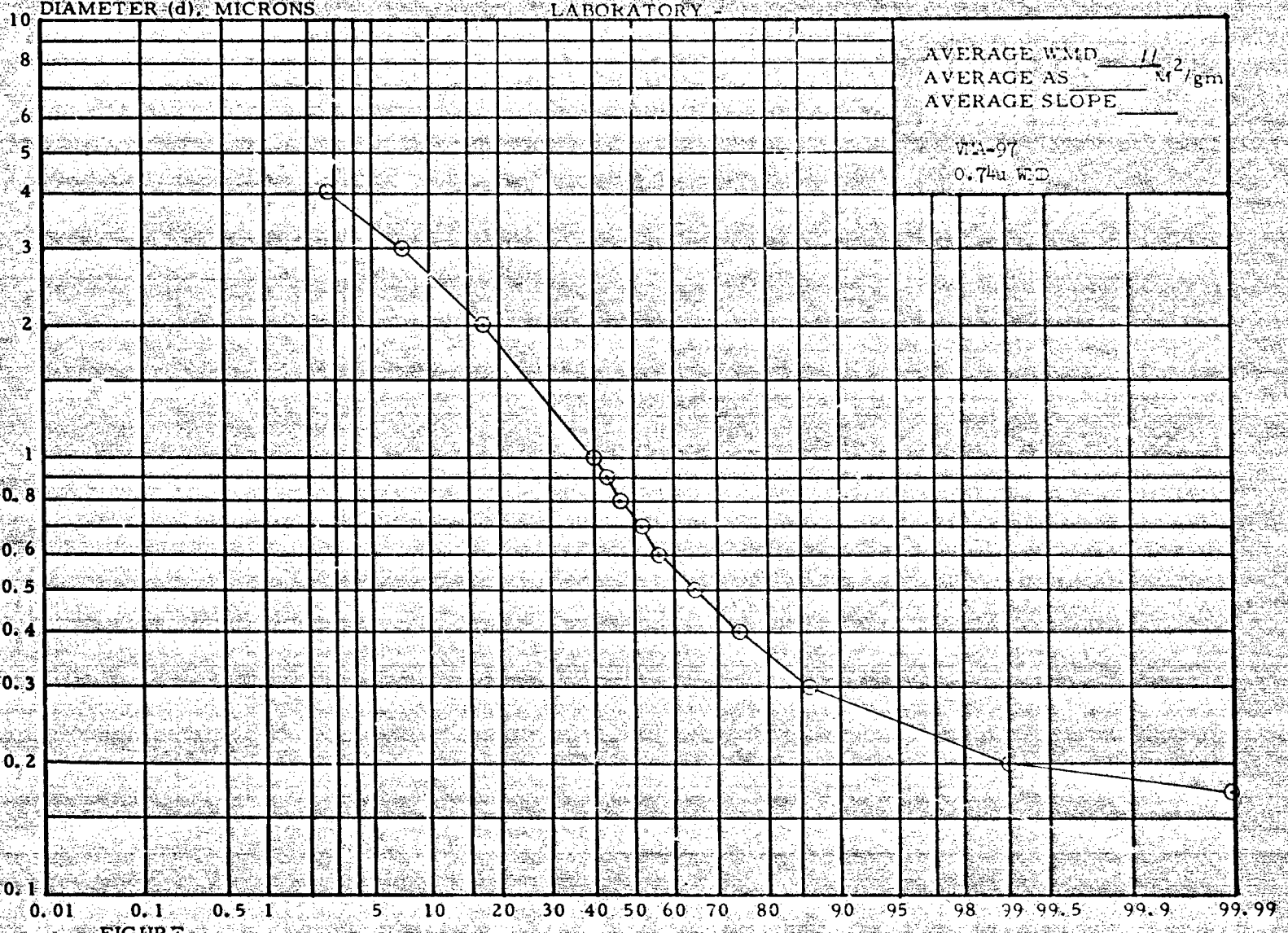


FIGURE ____ CUMULATIVE WEIGHT PERCENT GREATER THAN DIAMETER

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