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PCDE PROPELLANT STUDIES (Unclassified Title)

Aerojet Solid Propulsion Company
Propellant Development Department
Sacramento, California

Anthony J. Di Milo, Leonard J. Rosen, Richard L. Lou

October 1973

Semiannual Report 1 March to 31 August 1973

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FOREWORD

This technical report, "PCDE Studies" was prepared under Contract No. D04611-72-C-0046 as partial fulfillment of the requirements of the Air Force Rocket Propulsion Laboratory, Research and Technology Division, Air Force Systems Command, Edwards, California. The work reported was done in the Propellant Development Department, Advanced Propellants Section of the Aerojet Solid Propulsion Company, Sacramento, California. This report, designated Aerojet Report 1938-76SA-3, records the results of work done during the interval 1 March to 31 August 1973. The program was monitored by Captain A. Crelrier.

Acknowledgement is made to the following persons who have contributed materially to the work performed during this period. Mr. J. Newey, for propellant formulation; Mr. I. Hazelton, for mechanical testing; Mr. T. Hickmon, for burning rate studies; Messrs. A. A. Almada and O. Dizney, for supporting efforts; and Mr. F. O'Dell, for safety testing.

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20. ABSTRACT (Continue on reverse side if necessary and identify by block number) (C) A PCDE-TMETN propellant has been developed and made in six 1-gal and three 5-gal batches. The formulation which consists of 40.5 wt% HMX, 20.5 wt% AP, and 18 wt% Al, and a binder based on equal parts of PCDE and TMETN is calculated to have a theoretical specific impulse of 271.2 of which 256.5 will be delivered in the Aerojet 10KS2500 motor. The propellant density is 0.06842 lb/in ³ . At 125°F, the processing temperature, the propellant is Aerojet Type 3 and does not require remote handling.		

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20. (Cont.)

(U) One of the 5-gal batches, made to provide aging samples for the Lockheed Propulsion Company, showed colored inclusions surrounded by larger areas of poorly cured propellant. Analysis of these indicated larger than normal concentrations of Zn, FeAA, and DNDPA. The conclusion was that these materials arose from poor dispersal of a mixture of ZnO, FeAA, sulfur, and DNDPA which is added to the prepolymer just previous to mixing. More efficient dispersal should alleviate the problem.

(C) The burning rate of the propellant is 0.466 in./sec at 1000 psia with a pressure exponent of 0.60. The π_k is 0.2%/°F between -65° and 150°F. In R&H 2C1.5-4 motors fired at AFRPL the burning rate was 0.49 in./sec at 1000 psia with a pressure exponent of 0.53.

(U) The propellant ages poorly at 150°F, but maintains adequate mechanical, safety, and ballistic properties at 110° and 77°F for more than eight weeks.

(C) Currently being developed is a PCDE-BDNPA/F propellant. The candidate formulation consists of 51 wt% AP, 22 wt% Al, and a binder based on PCDE and BDNPA/F at a ratio of 1.1 to 1. The calculated theoretical specific impulse of this Class 2 propellant is 263.4 of which 250.8 is delivered in a large motor. Propellant density is 0.0680 lb/in³. The chief problem is to achieve a burning rate of 1.3 in./sec at 1000 psia. Although this can be done by the use of iron oxide or by use of 1μ or 3μ AP, some difficulties of reproducing earlier values with propellants containing 0.5μ AP have been experienced. No major technical difficulty to achieving the burning rate is expected. Processing studies of this propellant have begun, and early work indicates a potlife in excess of four hours.

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GLOSSARY

AFRPL	Air Force Rocket Propulsion Laboratory
AP	Ammonium perchlorate
BATES	Ballistic Test and Evaluation System
BDNPA/F	Nitroplasticizer, bis-(2,2-dinitropropyl)-acetal and -formal; 1 to 1 wt ratio
C*	Thermodynamic parameter, characteristic exhaust velocity
C _D	Thermodynamic parameter, coefficient of thrust
DBR	Stabilizer, di-t-butylresorcinol
DBDL	Cure catalyst, dibutyltin dilaurate
DC200	Silicone surfactant
DEA	Bonding agent, diethanolamine
DNDPA	Stabilizer, 2,4-dinitrodiphenylamine
DOT	Department of Transportation
E ₀	Mechanical properties parameter, initial modulus
e and $\dot{e}$	Mechanical properties parameters, elongation and rate of elongation
FC-189	Surfactant
FeAA	Cure catalyst, ferric acetylacetonate
GPC	Chemical technique, gel permeation chromatography
H-5, -15, -60, and -95	Aluminum type, spherical, 5, 15, 60 and 95μ average particle sizes
HAA	Cure catalyst modifier, acetylacetone (used with FeAA)
HDI	Curing agent, hexamethylene diisocyanate
HMX	Oxidizer, cyclotetramethylene tetranitramine
HT	Crosslinker, 1,2,6-hexanetriol
ICC	Interstate Commerce Commission
IPDI	Curing agent, isophorone diisocyanate
JANNAF	Joint Army-Navy-NASA-Air Force
k	Thermodynamic parameter, exponent in the equation $\Omega = \left(\frac{I}{\rho}\right)^k$
MDX-65	Aluminum, 5μ, tear-drop
Neozone D	Stabilizer, N-phenyl-2-naphthylamine
NOL	Naval Ordnance Laboratory

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GLOSSARY (Cont.)

Ω	Performance parameter based on final boost velocity
P711	Plastinox 711, stabilizer, di(tridecyl) thiodipropionate
π_k	Temperature sensitivity of operating pressure at constant area ratio
(C) PCDE	Prepolymer, poly(1-cyano-1-difluoraminoethylene oxide)
R&H	Rohm & Haas
RRD	Rounded rotary-dried (in reference to AP)
S-8	Stabilizer, N-ethyl toluenesulfonamide
σ	Mechanical property parameter, tensile strength (if not specified, refers to maximum tensile strength)
TDI	Curing agent, tolylene 2,4-diisocyanate
TMETN	Energetic plasticizer, trimethylolethane trinitrate
UFAP	Oxidizer, ultrafine ammonium perchlorate, <5 μ

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I. INTRODUCTION (U)

(U) This is the third semiannual Technical Report submitted in partial fulfillment of the requirements of contract F04611-72-C-0046. The technical portion of this report covers the period 1 March 1973 to 31 August 1973. Dr. L. J. Rosen is Technical Director of the Program and Dr. A. J. Di Milo is the Principal Investigator.

II. OBJECTIVE (U)

(C) The initial objective of this technical effort was the demonstration of the potential of the energetic prepolymer PCDE in highly aluminized solid propellants containing high-energy nitrate- and nitro-plasticizers, ammonium perchlorate and/or HMX. The demonstration required formulation and scale-up of two propellants, one with Class 7 and one with Class 2 hazard characteristics. Scale-up was to culminate in preparation of 5-gallon batches and 15-lb BATES grains of each formulation. Characterization of mechanical properties, aging capability, and sensitivity was required in the course of achieving overall objectives.

(U) An add-on program increased the scope of work involving the Class 2 propellant without changing the objectives of the program concerned with the development of the Class 7 propellant.

(C) The overall objective of the add-on program is the development of a high-performance solid propellant for air-launched missiles based on PCDE prepolymer plasticized with a combination of BDNPA and BDNPF, and demonstration of the performance of this propellant by large-scale motor firings.

(C) The propellant property goals are:

- A minimum delivered specific impulse of 250 lbf-sec/lbm.
- A minimum density of 0.068 lb/cu in.
- Propellant detonability no greater than that of a Class 2 explosive.

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- (C)
- A burning rate of 1.6 with a range from 1.2 to 2.1 in./sec at 1000 psia, a pressure exponent at or below 0.5 and $\pi_k < 0.15\%/^{\circ}\text{F}$.
 - Better than the minimum target uniaxial mechanical properties ($\sigma_m = 100$ psi, $\epsilon_m = 30\%$ and $E_o = 500$ psi).
 - Adequate aging stability.
 - Adequate processing and cure properties.
 - Safe manufacturing, handling and use characteristics.
 - Adequate liner-bond properties.
 - Adequate combustion-stability characteristics.
 - High reproducibility.

III. SCOPE (U)

(U) The augmented program adds three phases to the two phases of the original program.

(U) In Phase II, motor performance tradeoffs necessary to attain ballistic objectives will be completed. The selected formulation will be evaluated and determined to be Class 2. The Phase II candidate propellant will be scaled up to 5-gal mixes and a total of six 15-lb BATES motors will be prepared and delivered to AFRPL. Three of these six motors will constitute an increase in the scope of this phase.

(C) Work on the add-on program was initiated in Phase III in January 1973. In this phase, the burning rate of PCDE-BDNPA/F shall be tailored to provide a target burning rate of 1.6 in./sec at 1000 psia and formulations with burning rates of from 1.2 to 2.1 in./sec at 1000 psia will be identified. Additional goals include a pressure exponent of less than 0.5 and a π_k of 0.15%/°F.

(U) Variables such as mix viscosity, pot life, viscosity versus time and shear rate or stress, bonding agents, curing agent, and cure catalyst will be assessed for their effect on processability, castability, and associated hazards.

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(U) Also in Phase III will be a systematic program of mechanical property tailoring and testing and propellant aging. Target properties will exceed σ_m 100 psi, ϵ_m 30%, and E_o 500 psi. Properties under multi-axial stress conditions will also be investigated, as will effects of temperatures of -65° to 180°F on mechanical properties. Aging studies of the propellant will also be initiated, and use of stabilizers and combinations of stabilizers to improve aging will be investigated. A compatible liner system will be developed.

(U) Based on burning rate tailoring, processing and mechanical properties studies, selected formulation(s) will be scaled up to 5- and 30-gal mixes for further characterization of ballistic and mechanical properties and aging stability. Ballistic test motors of 10- and 70-lb charge will be fired over a pressure range of 500 to 3000 psia. Propellant from these batches will be placed in storage for periodic testing to the end of the program. Additional 70-lb BATES grains and other samples will be delivered to AFRPL.

(U) ICC (DOT) explosive classification will be determined for the candidate propellant. An analysis of the hazards involved and their application to large-size grains will be undertaken. As part of the hazards evaluation, tests of critical diameter will be performed.

(U) In Phase IV a selected formulation will be scaled up to the 300-gal size. Three Genie motors with 300-lb grains and two instrumented analog motors will be prepared and shipped to AFRPL for testing. Samples of propellant will be placed in storage for testing in Phase V.

(U) Phase V of the proposed program is a propellant shelf life study program. Ambient and accelerated aging of propellant and motor grains prepared during Phases III and IV will be undertaken. Visual examination for swelling and cracking, and periodic measurement of mechanical and bond properties and burning rate will be made on aging samples. Motor grains will be fired after aging. Preliminary system safety analysis will be conducted.

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IV. SUMMARY (U)

(U) The following is a summary of the technical progress made during the period from 1 March 1973 to 31 August 1973.

(U) A. A total of 98 lb of PCDE has been received from Hercules, Inc. during the past six months. This material consisted of 13 lots. In addition, drums of Shell Lot 44 were also received to fulfill specific contract requirements. All the earlier lots are found to contain acetone by infrared analysis; the more recent lots have not yet been examined. The lots have averaged less than 10 lb per lot, and have thus required considerable labor to blend them into convenient-sized lots.

(U) B. PCDE Lots 6 and 8 were passed through molecular sieves and blended to create Lot 6+8. Because Shell Lot 44 was used up, experimental work was continued with Lot 6+8.

(U) C. The viscosity of BDNPA/F varies considerably with change of temperature, decreasing from 730 poise at 64°F to less than 20 at 140°F. These data are pertinent to processing studies which have begun.

(U) D. Both PCDE and its propellants evolve gases when aged. The gases are mainly HCN and CO₂, except in the case of PCDE-TMETN propellant which also evolves oxygen, nitrogen, and water.

(U) E. The antioxidant Neozone D interfered with the cure of PCDE-TMETN propellants. DNDPA + S was a satisfactory replacement. The cure reaction was speeded up by altering the FeAA:HAA:ZnO catalyst ratio from 0.03:0.09:0.10 to 0.02:0.02:0.20.

(U) F. At higher temperatures the friction sensitivity of PCDE-TMETN propellants decreases while the impact sensitivity increases. At the processing temperature of 125°F, the propellant is safe enough to allow direct manipulation. The cured propellant is Military Class 7 and DOT Class A.

(U) G. Early 1-gal batches of PCDE-TMETN propellant contained unacceptable amounts of porosity, caused principally by impurities in the PCDE;

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(U) moisture in the HMX and AP had an additional minor effect. The PCDE impurities could be removed by passing the prepolymer through molecular sieves. By the use of this method, three 1-gal batches were made from which were cast 14 R&H 2C1.5-4 grains and samples for aging and hazards studies.

(U) H. Three 5-gal batches of the propellant were prepared. These allowed preparation of five 15-lb BATES grains and aging samples for the Lockheed Propulsion Company. Although the grains were satisfactory, the aging samples had numerous colored inclusions which were the nuclei of larger areas of poorly cured propellant. Emission and infrared analysis of these inclusions indicated the presence of higher than normal amounts of Zn, FeAA, and DNDPA. It was concluded that these materials were not properly dispersed in the batch in question and that the Zn in the form of ZnO affected the cure. Future batches of this propellant will require attention to the need for properly dispersing these additives.

(C) I. The strand burning rate of the PCDE-TMETN propellant was 0.466 in./sec at 1000 psia with a pressure exponent of 0.60. Twelve R&H 2C1.5-4 grains fired at AFRPL gave a burning rate of 0.49 in./sec with a pressure exponent of 0.53. Three of the 15-lb BATES grains have been successfully fired at AFRPL.

(U) J. PCDE-TMETN propellant aged at 150°F fissured seriously after 1 week. At 110°F with exposure to the ambient atmosphere or sealed in cans, the propellant essentially retained its mechanical capability after 8 weeks. Sealed in cans at 110°F, the propellant exhibited increased friction sensitivity after eight weeks, and the same was true for propellant stored at 150°F for six weeks. Burning rate decreased very slightly for propellants aged at all conditions, but the change may be too small to be significant.

(C) K. Iron oxide, copper chromite, and both 1μ and 3μ AP have been used to increase the burning rate of PCDE-BDNPA/F propellants. Copper chromite interfered with the cure of the propellant and no useful data

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(C) were obtained. The use of iron oxide will allow the target burning rate, 1.3 in./sec at 1000 psia, to be achieved with a pressure exponent of 0.6. The target burning rate could be obtained also with use of 1 μ and 3 μ AP. Recent work has uncovered some problem areas in that earlier burn rates could not be duplicated. The effect is believed to be the result of either variable UFAP quality or inefficient dispersal of the oxidizer in the recent experiment. Studies have been planned to provide clarification.

(U) L. DEI has been used to cure PCDE-BDNPA/F propellants. Good mechanical properties were achieved along with increased potlife provided by the slower-curing IPDI.

(U) M. Initial stabilizer studies are inconclusive; none of the stabilized propellants exhibits much difference in weight loss at 150°F compared to the unstabilized propellant. Di-t-butylresorcinol interfered with propellant curing. Santicizer 8 has the advantage of being liquid.

(U) N. Although earlier work with the PCDE-BDNPA/F system relied on a coated AP, experiments showed that adequate mechanical properties are obtained with uncoated oxidizer and DEA as bonding agent. Current work is being done with uncoated AP and DEA.

(U) O. Because Shell PCDE Lot 44 was used up, Hercules PCDE Lot 6+8 is now being used. NCO and crosslinker requirements have been determined. The observation has been made that MDX-65 aluminum cures better than H-60 in these formulations. The effect is not due to interaction between the aluminums and the curing agent, but the cause is still unknown. The potlife of the propellants exceed four hours, the longest measurement made.

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V. TECHNICAL DISCUSSION (U)

A. PCDE ACQUISITION (U)

(U) A total of 38.06 lb of PCDE in 9 lots was received from Hercules, Inc. in March 1973. Pertinent information for these materials is shown in Table 1. Lot 15 was received from Hercules in July 1973. The lot consists of 20.13 lb of PCDE. Properties reported by the vendor are listed below.

PROPERTIES OF PCDE LOT 15* (U)

Nitrogen, wt%	20.7
Fluorine, wt%	26.9
Molecular Weight	3040
Equivalent Weight	1750
Functionality	1.74
Thermal Stability at 140°C	208
Thermal Stability at 100°C**	10

* Vendor's Data

** Volume of gas (ml) evolved for 0.25-g sample at 110°C after 200 hr in 10-cc syringe

(U) A further shipment of 39.9 lb of PCDE was received from Hercules Incorporated in August 1973. The shipment consisted of 0.99 lb Lot 16A, 19.3 lb Lot 17B and 19.6 lb Lot 18. No analytical data are available.

(U) The PCDE (Lot 44) necessary for the 5-gal batches of PCDE-TMETN propellant was obtained from RPL. The PCDE was passed through molecular sieves until IR inspection indicated the absence of acetone. An additional amount of PCDE received was also treated as above. This material was used to prepare another 5-gal batch from which were cast three 15-lb BATES grains. Two grains made from earlier batch were topped. All five motors were delivered to AFRPL.

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TABLE 1

PERTINENT DATA CONCERNING HERCULES PCDE^a

Lot No.	No. of Drums	PCDE		Molecular Weight	Equivalent Weight	Thermal Stability ^b	Functionality
		Conc., %	Wt, lb				
1A1	2	10.5	1.42	2960	1223	13 ^c	2.40
1A2	5	8.4	4.04	2500	1466	12.3 ^c	1.71 ^d
1A2A	2	6.4	1.13	2020	1466		1.76
1A3	3	20.1	6.0	2230 ^e	1139	18 ^f	1.96 ^d
3A	8	4.4	3.5	2610	1493		1.74
3B	9	4.95	4.4	2610	1493	7.2	1.74
4	11	4.9	5.4	2840	1547	9.6	1.84
6	21	4.7	9.6	2840	1556	11.7	1.82
8	9	3.0	2.57	2750	1344	10.5	2.04

a - Data supplied by Hercules, except as noted

b - Volume of gas evolved from 0.25-g sample heated at 110°C for 200 hours in a 10-cc syringe

c - Evolved gas exceeded syringe volume; test limited to 188 hours

d - Calculated from molecular and equivalent weights

e - GPC analysis by Shell indicated 99% of molecular weight 2900

f - Evolved gas exceeded syringe volume; test limited to 141 hours

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(U) The following comments concerning these shipments are of considerable importance to the program. First, in view of the time required to qualify a lot of PCDE for use in current formulations (3 weeks), the number of lots corresponding to the total amount of PCDE received is a disadvantage. This handicap can be reduced, but not completely eliminated, by blending to provide larger lots. It should be understood, however, that blending involves the danger that considerable amounts of material might be irrevocably lost if one or more of the lots used for blending are exceptionally bad. It is recommended that the blending operations be performed by the supplier and that data for the lots blended be furnished.

(U) The lots received are in very dilute solutions, some as low as 3%. This entails shipping and handling of excessive amounts of containers and solvent. It is recommended that attempts be made to maintain the PCDE concentration at 20% or higher to avoid these problems.

B. INGREDIENTS STUDIES (U)

1. PCDE Lot 6+8 (U)

(U) Qualification of PCDE received from Hercules, Inc. was initiated. Figure 1 is an infrared scan of as-received Lot 6. The characteristic band for acetone is clearly visible at 1720 cm^{-1} . This band was also present in the scan for Lot 8. Lots 6 (9.6 lb) and 8 (2.57 lb) were reduced in volume after passing through 13X molecular sieves and combined. Data for the lots and the derived averages for the combination are given in the Table below.

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INFRARED SCAN OF AS-RECEIVED PCDE - LOT 6

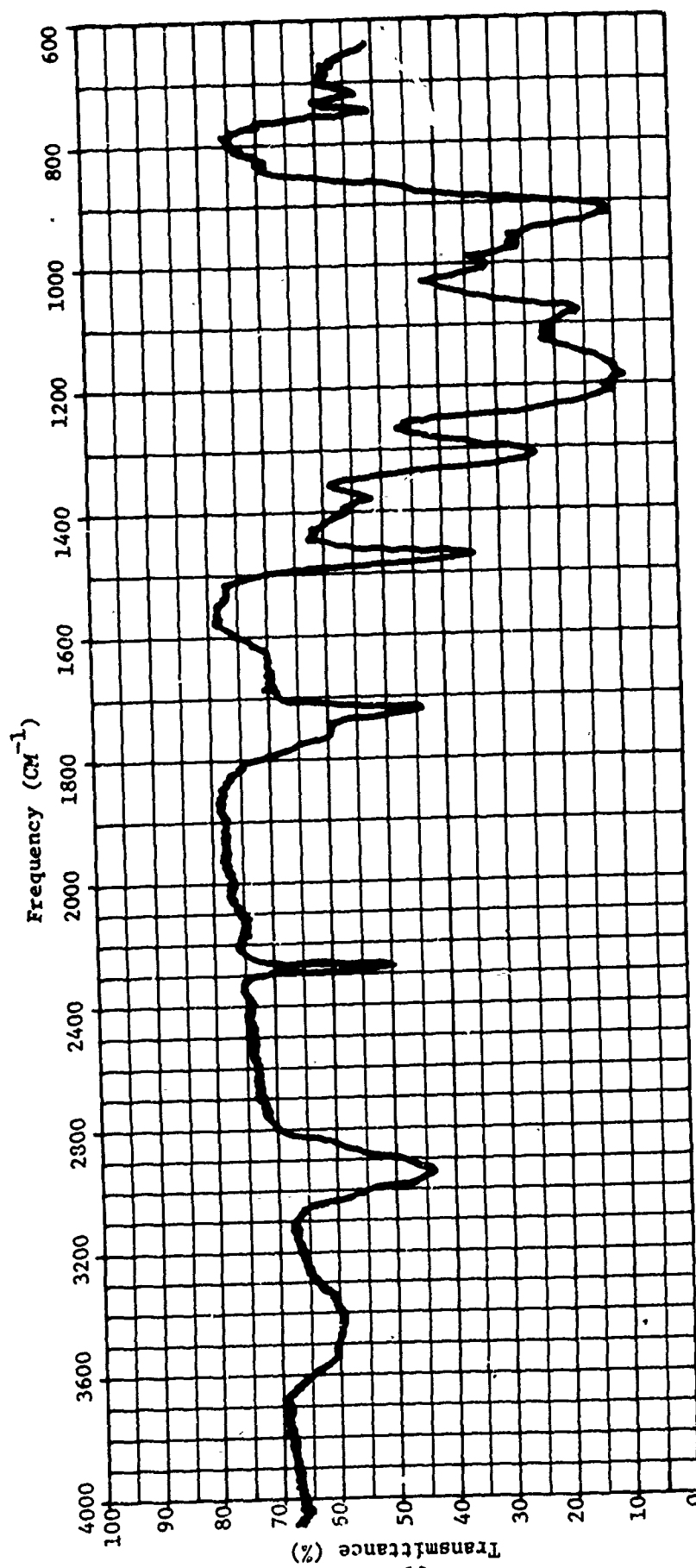


Figure 1

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PROPERTIES OF PCDE LOTS 6 AND 8 AND THEIR COMBINATION (U)

	Lot No.		
	6	8	6+8
Molecular Weight	2840	2750	2821
Equivalent Weight	1556	1344	1511
Functionality	1.82	2.04	1.87
Thermal Stability*	11.7	10.5	-
Weight, lb	9.6	2.57	12.17

* Volume of gas from 0.25 gm PCDE at 110°C for 200 hr in 10 cc syringe.

(U) The mixed lot was evaluated for NCO to OH ratio for cure and for PCDE to HT ratio for best mechanical properties in PCDE-BDNPA/F propellants and the results are described in Section V.E.7.

2. Viscosity of BDNPA/F (U)

(U) Because PCDE-BDNPA/F propellants are highly viscous, a study was made of the effect of temperature on viscosity of BDNPA/F. The data are shown graphically in Figure 2.

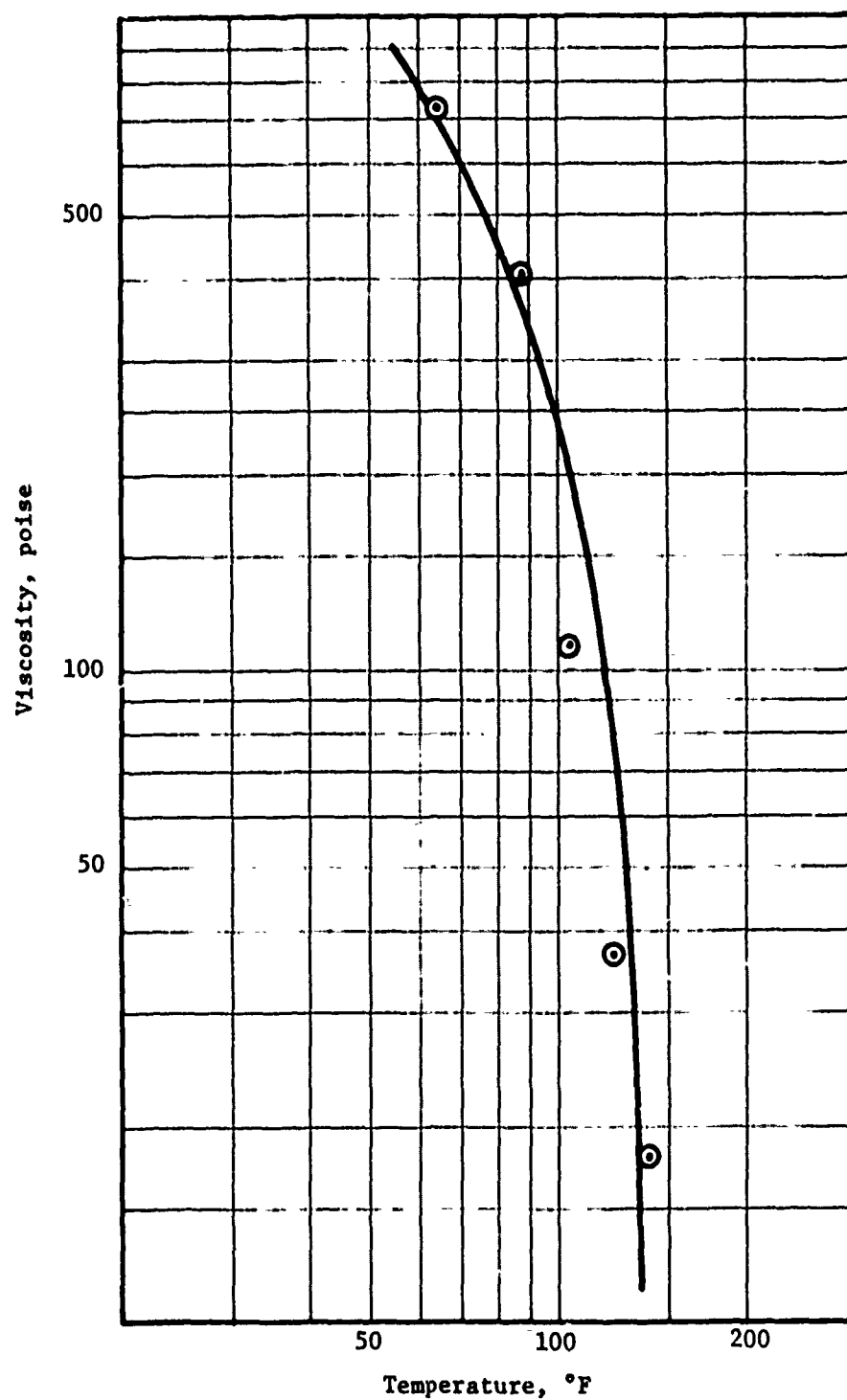
(U) There is a considerable advantage to using BDNPA/F at 140°F. The viscosity decreases from 730 poises at 64°F to less than 20 at 140°F. This information will be useful for process studies.

C. GAS ANALYSIS (U)

(U) Because the odor of HCN has been frequently detected and HCN qualitatively identified in gases over PCDE and its propellants, an analysis of gases evolved from PCDE and its propellants on aging was made. The results are shown in Table 2 in which is also shown the fluoride ion (HF) content of the materials. Fluoride content, however, is confusing. In PCDE, itself, there is indication of enough fluoride ion to convert about 0.015 wt% FeAA to FeF_3 , a serious interference. On the other hand, fluoride ion is also found in the propellants; either the analysis is in error or the fluoride ion does not react with FeAA.

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EFFECT OF TEMPERATURE ON THE BROOKFIELD VISCOSITY OF BDNPA/F



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Figure 2

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TABLE 2
FLUORIDE CONTENT AND GAS ANALYSIS OF PCDE AND ITS PROPELLANTS AGED 14 DAYS AT 150°F

Material	PCDE		PCDE Propellants	
	As Received	Through 13X Sieves	Heated to 180°F	BDNPA/F ^a TMEIN
Fluoride Ion, µg/g of sample ^b	222	200	156	218 212
Evolved gas, Vol., ml at 25°C and 760 mm ^c	4.04	- ^d	2.74	0.254 0.15
Gas composition, mol %				
Oxygen	-	-	-	- 1.8
HCN	14.3	10.0	5.9	4.6 2.7
Nitrogen	-	9.1	-	- 15.0
Carbon dioxide	15.8	10.5	-	48.3 24.6
Acetone	35.5	3.2	-	- -
Methylene chloride	34.5	67.0	40.7	- -
Chloroform	-	-	53.4	- -
Water	-	-	-	- 56.1

a - Gas evolved consisted of 47.1 mol % of unidentified gases probably a mixture of acetaldehyde, formaldehyde, and oxides of nitrogen

b - Measured before aging

c - Volume per gram of sample

d - Not measured

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(U) The amount of gas evolved from PCDE or its propellants is very moderate. If the presence of solvent molecules in the gas is ignored, HCN and carbon dioxide are the chief constituents except in the case of the PCDE-TMETN propellant. In the latter, oxygen, nitrogen, and water are also found. No HNF_2 was detected.

D. PCDE-TMETN PROPELLANT STUDIES (U)

1. Introduction (U)

(U) A comprehensive discussion of the thermodynamic rationale determining the choice of propellant compositions to be studied was presented earlier⁽¹⁾ and will not be repeated here. However, Table 3 is presented to make the reader aware of some of the possible formulations.

(C) On the basis of these considerations, the baseline composition selected for the Class 7 propellant studies and scale-up was fixed at 79 wt% solids, 18 wt% aluminum, an HMX to AP wt ratio of 2 and a plasticizer to crosslinked polymer ratio of 1. The reasons for these choices are that (1) other compositions which can be processed would provide only a marginal increase in specific impulse and (2) the volume fraction of the binder (0.27) is conducive to achieving adequate processability, mechanical properties and aging stability. Table 3 presents the background for establishing the baseline composition.

2. Effect of Neozone D on Cure (U)

(U) Cure failure in 1-gal batches of PCDE-TMETN propellant was attributed to Neozone D, an antioxidant. A number of 450-g batches were made to test this idea and to obtain viscosity data for new formulations under consideration. These batches are shown in Table 4. PCDE 1-78 and -79 containing Neozone D did not cure at 110° or 125°F although the corresponding batches, PCDE 1-82 and -83, containing DNDPA + S, cured without difficulty. PCDE 1-83 had only 0.01 wt% FeAA and was definitely softer than PCDE 1-82 with 0.02 wt% FeAA. The latter two propellants were also made in 100-g batches, PCDE 228 and 229, which also cured well.

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TABLE 3
PERFORMANCE POTENTIAL OF PCDE-TMETN PROPELLANTS (U)

Solids, ^a Wt%	Al Wt%	AP/HMX	Plast./ Binder	Density lb/in. ³	Specific Impulse, lbf-sec/lbm		
					Theor.	Expected ^b	Ω^c $\frac{k=0.3}{k=0.7}$
79	20	1/3	3	0.0685	273.6	260.7	$\frac{264.8}{270.2}$
79	18	1/3	2	0.0681	273.4	261.0	$\frac{264.6}{269.5}$
79	18	1/3	1	0.0682	272.6	260.6	$\frac{264.3}{269.3}$
79	18	1/2	2	0.0685	272.6	260.3	$\frac{264.3}{269.9}$
79	18	1/2	1	0.0685	272.2	260.1	$\frac{264.2}{269.7}$

- a - Formulations restricted to 0.26-0.29 vol. fraction binder to assure processability.
b - Expected in large motor; mass flow 400 lb/sec and exposed area 400 sq in.; 15° half-angle and 1000/14.7 psi.
c - Figure of merit which incorporates the effect of density. For tactical application $k = 0.7$ or higher and for upper-stage vehicles, $k = 0.3$ are considered applicable.

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TABLE 4

COMPOSITION AND PROCESSING PROPERTIES OF PCDE-TMETN PROPELLANTS CURED WITH FeAA-HAA-ZnO (U)

Component	PCDE No. 1-				
	78	79	80	81	82
AP, 180u, RRD, coated					
HMX, 5u			20.5		
HMX, 150u, coated			15.0		
Al, MDX-65			25.5		
			18.0		
Neozone D					
DNDPA	0.02	0.02	-0-	-0-	-0-
S	-0-	-0-	0.20	0.20	0.20
FeAA	-0-	-0-	0.10	0.10	0.10
HAA	0.02	0.01	0.03	0.03	0.02
ZnO	0.02	0.01	0.09	0.06	0.02
			0.1		
TMETN ^a	9.744	9.764	9.564	9.594	9.644
PCDE ^{a,b}					
HT ^b			9.541		
IPDI ^b			0.209		
			1.165		
Safety Properties, uncured					
Impact, cm/2kg	8.3	10.2	9.4	8.2	8.7
Friction, g/3000 rpm	440	440	.835	245	570
					6.8
					300
Viscosity, Kp at 5000 dynes/cm ²	- ^c	- ^c	31/6.0	35/6.3	41/6.0
and time (hr) after NCO addition					29/6.5
Estimated Potlife, hr	- ^c	- ^c	6.0	6.3	6.0
					6.5

a - Passed through molecular sieves

b - Equivalents ratio, PCDE/HT/IPDI = 55/45/100

c - Did not cure at 110°F or 125°F

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(U) As a result of these experiments, a mixture of DNDPA + S was used in place of Neozone D as stabilizer.

3. Propellant Processability (U)

(U) To obtain more definitive data on processability, Rotovisko measurements were made of propellant viscosity. Because these measurements require larger amounts of propellant, batch size was scaled up to 450g. The figure of merit for processability was taken as 50000 poise at a shear rate of 5000 dynes/cm² (2).

(U) Viscosity measurements were made on PCDE 1-80 through 1-83 (Table 3). The results are shown in Figures 3 through 6. All of the catalyst combinations used provide at least six hours of potlife. The catalyst system FeAA/HAA/ZnO⁽³⁾ at a ratio of 0.03:0.09:0.10 was used in the third 1-gal batch PCDE 10GP-9306, which cured very slowly. The slowness of the cure may have been due to the large amount of HAA used. Because it retards the cure reaction, the HAA must be removed before the FeAA can exert a catalytic effect. For subsequent 1-gal or larger batches, the FeAA/HAA/ZnO was limited to 0.02:0.02:0.10, as in PCDE 1-82.

4. Hazards Studies (U)

a. Introduction (U)

(U) PCDE imposes a hazards problem greater than that imposed by more conventional propellants. The chief problem is their sensitivity to friction. Mixtures of PCDE and TMETN are not sensitive to friction even when they contain aluminum, but the addition of oxidizer increases the sensitivity of the propellant to friction. The uncured propellant is generally more sensitive than the cured. This friction sensitivity has required that caution be exercised in scaling-up propellant production. More extensive discussion of hazards was reported earlier⁽¹⁾.

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PROPELLANT VISCOSITY AS A FUNCTION OF SHEAR STRESS AND TIME FROM CURING AGENT ADDITION

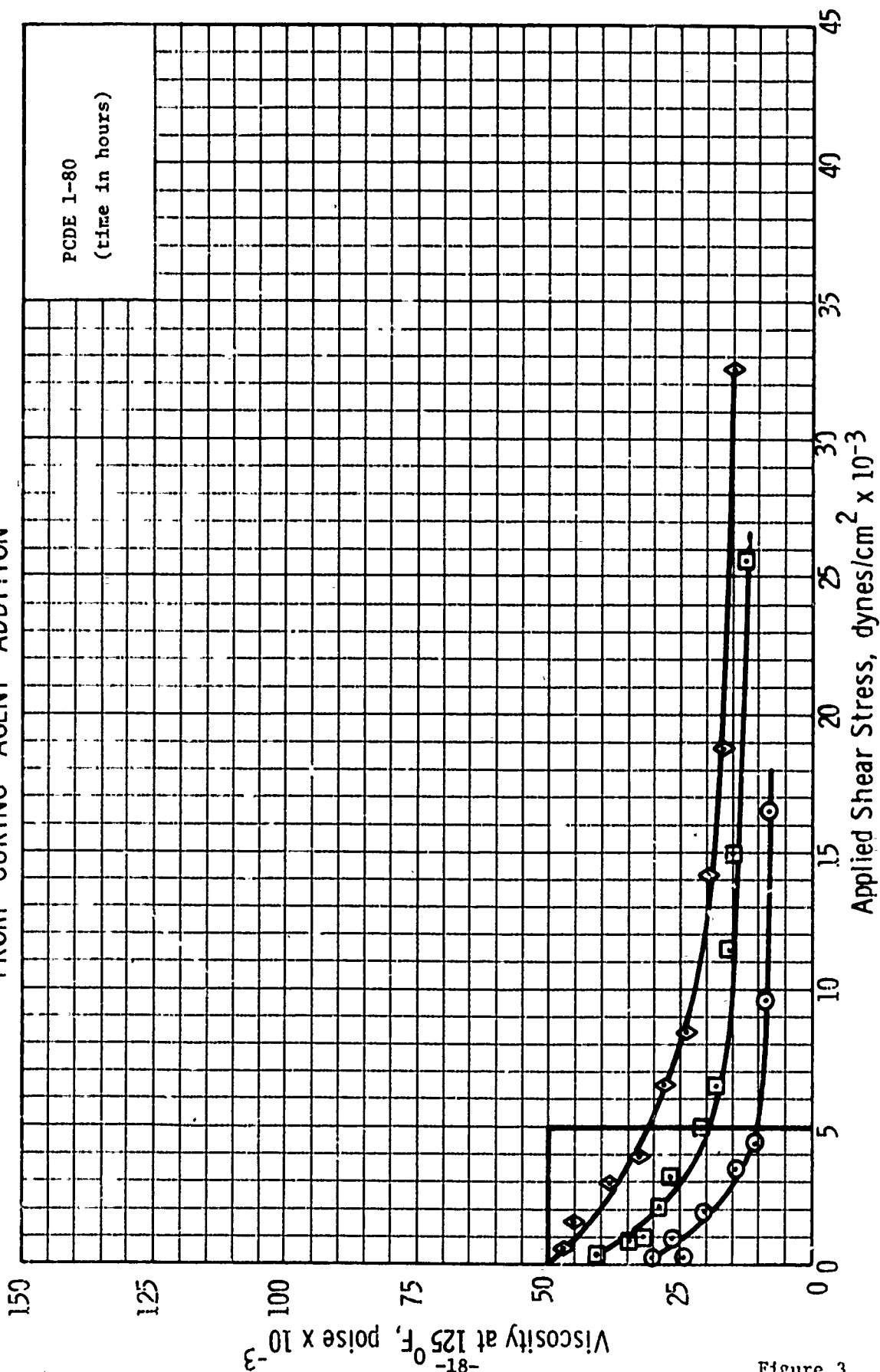


Figure 3

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PROPELLANT VISCOSITY AS A FUNCTION OF SHEAR STRESS AND TIME
FROM CURING AGENT ADDITION

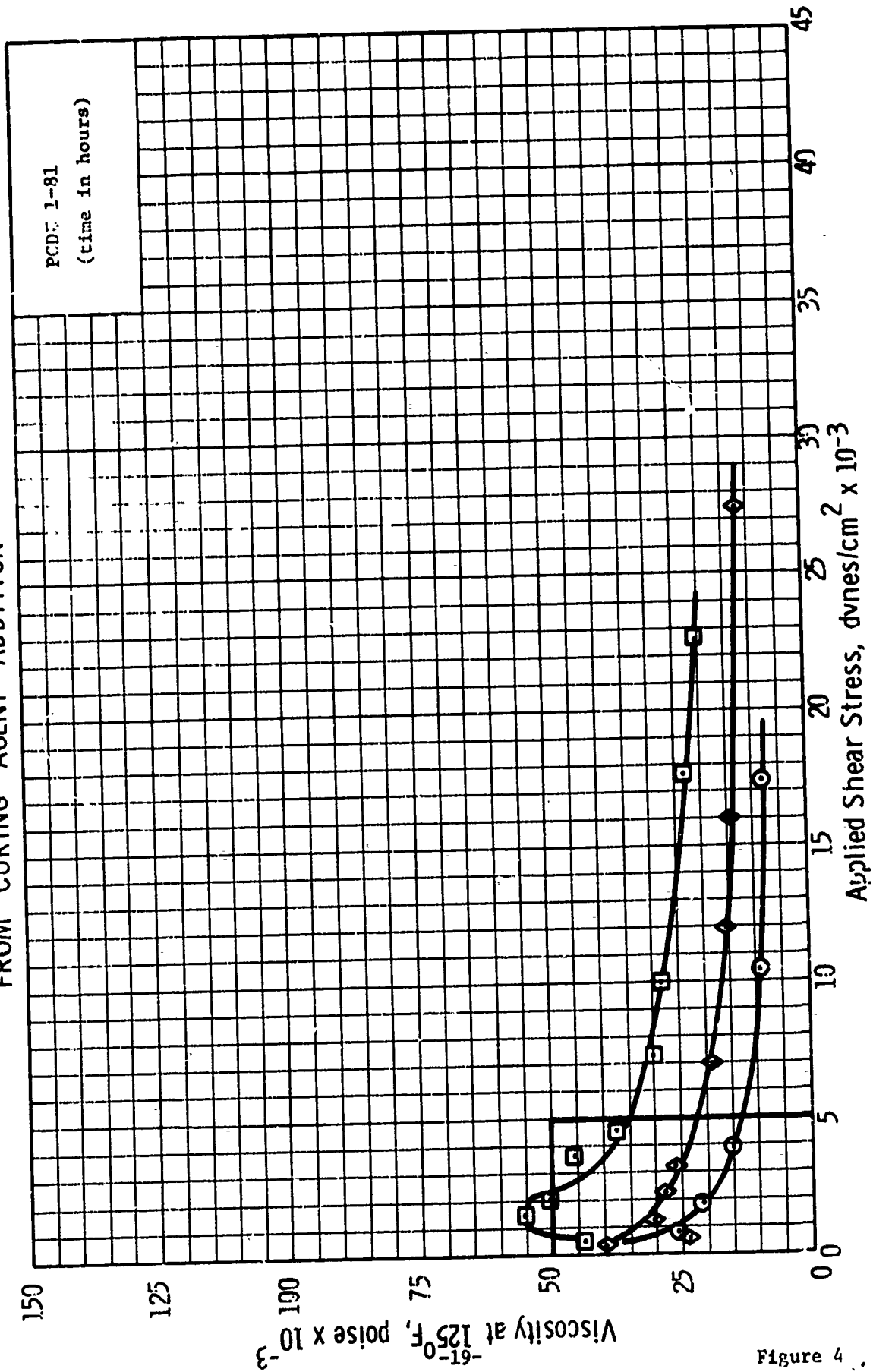
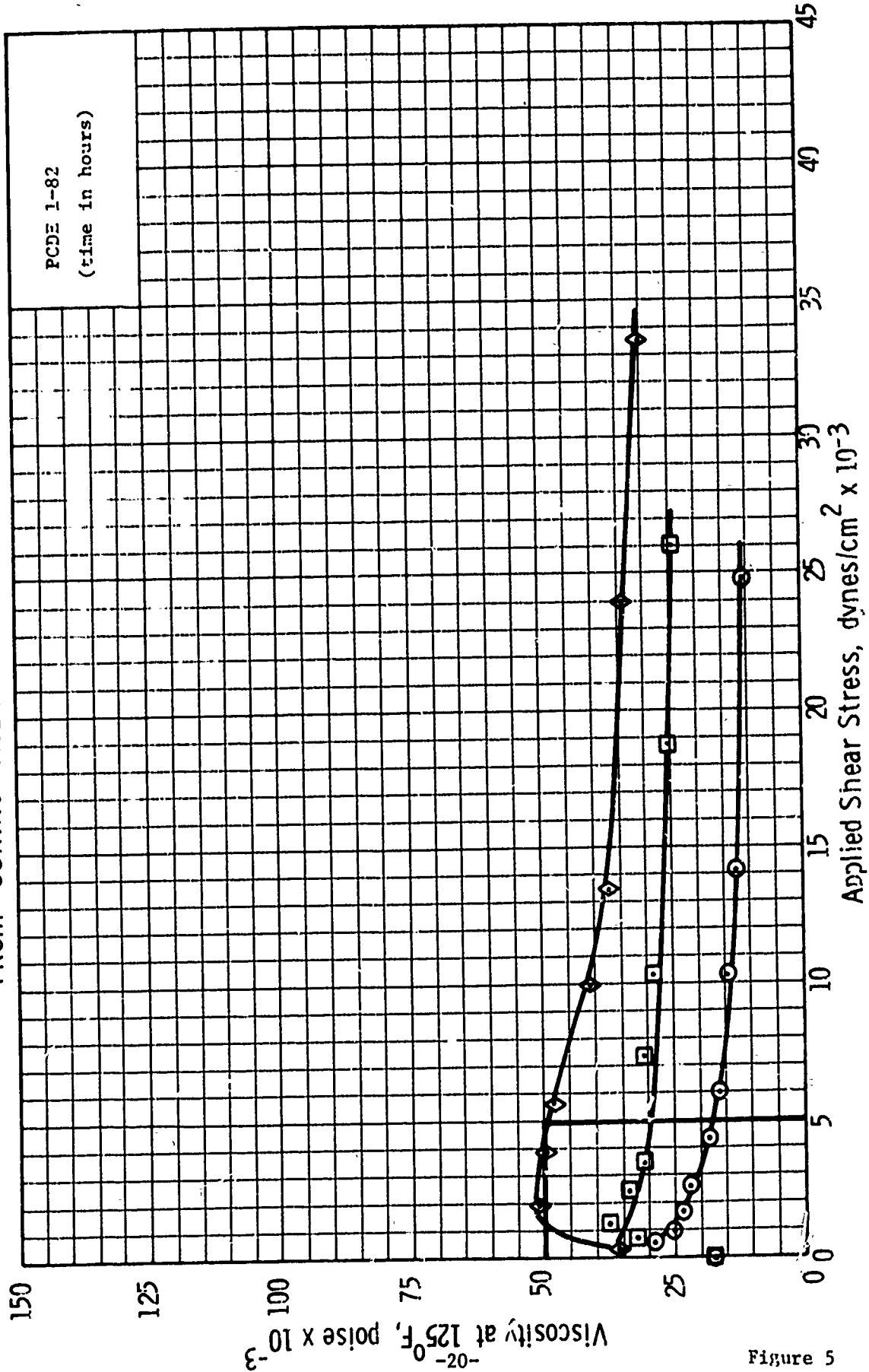


Figure 4

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PROPELLANT VISCOSITY AS A FUNCTION OF SHEAR STRESS AND TIME FROM CURING AGENT ADDITION



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PROPELLANT VISCOSITY AS A FUNCTION OF SHEAR STRESS AND TIME FROM CURING AGENT ADDITION

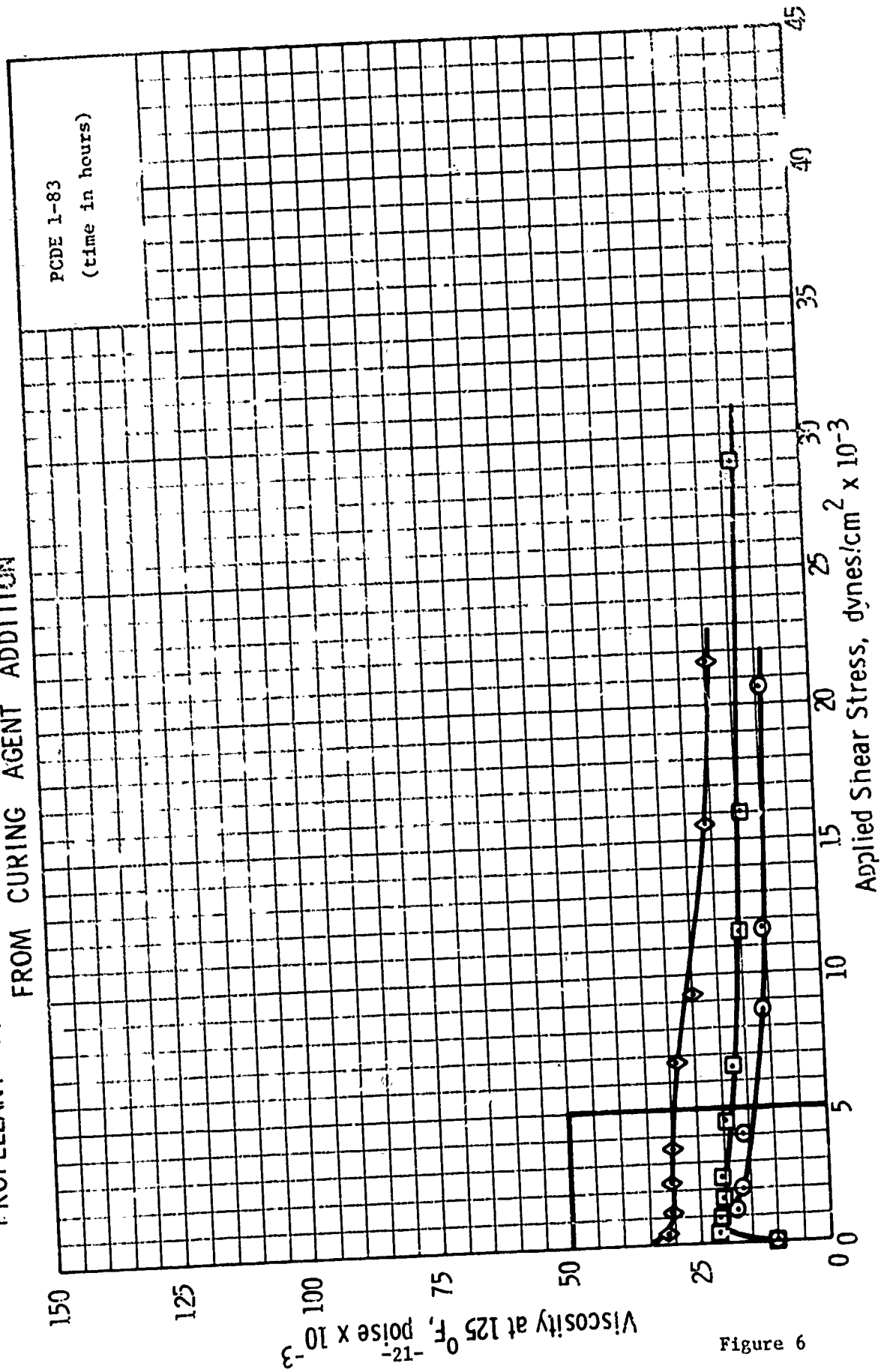


Figure 6

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b. Effect of Temperature (U)

(U) The PCDE-TMETN propellant was tested at the preparation temperature, 125°F. The first attempt indicated that the friction sensitivity was much less at this temperature. In fact, the propellant was negative even at 4000g/3000 rpm with one PCDE lot. On the other hand, the impact stability was decreased from 13 to about 4 cm/2 kg. Another test was made which generally confirmed the earlier tests. In the second test the impact sensitivity at 125°F was just above 6 cm/2 kg. At 125°F, the propellant is Aerojet Type 3D and remote casting is unnecessary. However, at lower temperatures, precautions are necessary.

(U) The data are reviewed in Table 5.

c. DOT Tests (U)

(U) PCDE-TMETN propellant, PCDE 10-4 (10GP9622), was subjected to hazards testing to establish its DOT classification. It is DOT Class A and Military Type 7. The tests and results are summarized in the table below.

PCDE-TMETN PROPELLANT HAZARDS TESTING (U)

(PCDE 10-4, Batch 10GP9622)

<u>Test</u>	<u>Results</u>
Impact, Bu of Mines, 50% fire point	7.5 cm/2kg
Rotary Friction, 50% fire point	765 g/3000 rpm
Differential Thermal Analysis, 9°F/min.	
Onset Temp., °F	283
Exothermic peaks, °F	339, 372, 409, 461, 498, 657
Autoignition by copper block, °F	352
Oven stability, 75°C, 48 hrs	Fissures throughout center of sample
Detonation Tests	
2 2-in. cubes with No. 8 blasting cap	Positive
2 NOL sleeves with 70 cards	Positive
Unconfined Burning	
5 2-in. cubes	Burned, 38 sec.

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TABLE 5

COMPOSITION OF PCDE-TMETN PROPELLANTS AND EFFECT OF TEMPERATURE
ON HAZARD PROPERTIES (U)

<u>Components</u> ^a	<u>PCDE No.</u>	
	<u>272</u>	<u>273</u>
AP, 180μ, RRD, coated	20.5	
HMX, 5μ	15.0	
HMX, 150μ, coated	25.5	
^, MDX-65	18.0	
DNDPA	0.20	
S	0.10	
FeAA	0.02	
HAA	0.02	
ZnO	0.10	
TMETN	9.552	
PCDE	9.552	
HT	0.227	
IPDI	1.211	
<u>Safety Properties</u> ^b		
Impact, cm/2 kg		
77°F	11.3	10.7
117°F	--	6.6
125°F	4.0	6.2
Friction, g/3000 rpm		
77°F	350	350
117°F	--	--
125°F	>4000	1450

^a PCDE and TMETN passed through molecular sieves. Equivalents ratio,
PCDE/HT/IPDI = 53/47/100 for both batches.

^b All samples uncured.

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5. 1-Gal Batches (U)

a. Batch 10GP9002 (PCDE 10-2) (U)

(U) Because the first 1-gal batch, 10GP8798 (10-1), had an unacceptably high porosity, a second batch (10GP9002, 10-2) was made with the formulation being modified in two ways. The catalyst system of FeAA-HAA-ZnO was substituted for DBTDL-benzilic acid-ZnO, and Neozone D was used in place of DNDPA + S. The composition of the batch is shown in Table 6. This batch did not cure.

b. Batch 10GP9306 (PCDE 10-3) (U)

(U) It became apparent on further examination of data for 450-g batches that these propellants do not cure as well with Neozone D as they do with DNDPA + S. Data to substantiate this were presented in Section V.D.2. Accordingly, a third batch, 10GP9306 (10-3), was formulated in which the Neozone D was removed and DNDPA + S was used in its place. The composition of this batch is also given in Table 6. This batch cured very slowly, taking almost 7 days at 110°F to reach measureable Shore "A" hardness. At 125°F, however, it cured more rapidly. Inspection of one container of propellant indicated that it contained a high degree of porosity.

c. Batches 10GP9622 and 9663 (PCDE 10-4 and 5) (U)

(1) FeAA and HAA Content (U)

(U) Batch 10-3 (10GP9306), a 1-gal batch prepared earlier in the program, cured very slowly, requiring over 7 days to achieve a measurable hardness. The slow cure was due to the large quantities of the catalyst suppressor, HAA, used. The batch contained 0.03 wt% FeAA and 0.09 wt% HAA. Experiments were made, therefore, to reduce both the FeAA and HAA to obtain adequate potlife and quick reliable cure. A system consisting of 0.02 wt% FeAA and 0.02 wt% HAA appeared to be promising; FeAA, 0.01 wt%, and HAA, 0.01 wt%, gave a much softer cure.

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TABLE 6

COMPOSITION OF PCDE-TMETN PROPELLANTS PREPARED IN 1-GAL BATCHES (U)

<u>Components</u>	<u>PCDE No.</u>	
	<u>10-2</u>	<u>10-3</u>
AP, 180 μ , RRD, coated	20.5	
HMX, 5 μ	15.0	
HMX, 150 μ , coated	25.5	
Al, MDX-65	18.0	
Neozone D	0.2	-0-
DNDPA	-0-	0.2
S	-0-	0.1
FeAA	0.03	
HAA	0.09	
ZnO	0.10	
TMETN ^a	9.664	9.564
PCDE ^{a,b}	9.541	
HT ^b	0.209	
IPDI ^b	1.165	
<u>Safety Properties (uncured)</u>		
Impact, cm/2kg	6.2	9.2
Friction, g/3000 rpm	180	550

a - Passed through molecular sieves

b - Equivalents ratio, PCDE/HT/IPDI = 55/45/100

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The former system provides a potlife of six hours (Section V.D.3., Table 4, PCDE 1-82 and 83) and was used in the 1-lb Batches 1-84 through -87 and 1-gal Batches 10-4 through -6. All cured in three to four days at 125°F. The composition and properties of these small batches are shown in Table 7.

(2) Causes of Propellant Porosity (U)

(U) Studies relating to porosity in 1-gal batches of PCDE-TMETN propellants were made at the 1-lb level. Four propellants, shown in Table 8, were made according to the variations indicated in the table. Considerable porosity was observed in PCDE 1-84 for which the PCDE has been passed only once through molecular sieves and the solids had received only normal drying. When PCDE was passed twice through the sieve column, as for PCDE 1-85, the porosity was reduced greatly even though the solids were only normally dried. Only negligible porosity was found in PCDE 1-86 and -87, both of which utilized PCDE that had passed through the sieves twice and solids that had undergone extra drying. The important effect of the special sieve treatment is apparent in considering the very low 24-hr hardness value of Batch 1-84. The batch actually never achieved the extent of cure of the others. It may not be necessary to pass PCDE through sieves twice, but rather to increase the ratio of sieves to PCDE for a given pass. It was noted by infrared analysis of effluents from the sieve column that although the material coming through first was free of the 1730 cm^{-1} band, the material coming through last showed the band, indicating acetone contamination and the need to investigate the effect of sieves to raw material ratio.

(U) The 1-lb batches were vacuum-cast to diminish the possibility of void formation resulting from that operation and to pin-point the actual cause.

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TABLE 7

COMPOSITION AND PROPERTIES OF PCDE-BDNPA/F PROPELLANTS
CURED WITH LOW FeAA-HAA CONTENT (U)

<u>Components</u>	<u>PCDE No.</u>	
	<u>228</u>	<u>229</u>
AP, 180 μ	20.5	
HMX, 150 μ , coated	25.5	
HMX, 5 μ	15.0	
Al, MDX-65	18.0	
DNDPA	0.20	
S	0.10	
FeAA	0.02	0.01
HA	0.02	0.01
ZnO	0.10	
TMETN ^a	9.644	9.664
PCDE ^{a,b}	9.541	
HT ^b	0.209	
IPDI ^b	1.165	
<u>Safety Properties</u>		
Impact, cm/2kg (uncured/cured)	13.0/5.6	13.0/5.6
Friction, g/3000 rpm (uncured/cured)	400/350	345/1200
Onset Temp., °F	251	300
<u>Mechanical Properties at 77°F</u>		
σ_m , psi	62	39
ϵ_m , %	24	24
E_o , psi	343	202
<u>Swelling Ratio in Acetone</u>	2.99	3.12

a - Passed through molecular sieves

b - Equivalents ratio, PCDE/HT/IPDI = 55/45/100 for both batches

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TABLE 8

COMPOSITION AND PROPERTIES OF PCDE-TMETN PROPELLANTS PREPARED
TO STUDY EFFECTS OF INGREDIENTS TREATMENT AND MIX CYCLE (U)

Components	PCDE No. 1-			
	84 ^a	85 ^b	86 ^c	87 ^d
AP, 180 μ , RRD, coated		20.5		
HMX, 5 μ		15.0		
HMX, 150 μ , coated		25.5		
Al, MDX-65		18.0		
DNDPA		0.20		
S		0.10		
FeAA		0.02		
HAA		0.02		
ZnO		0.10		
TMETN		9.644		
PCDE ^e		9.541		
HT ^e		0.209		
IPDI ^e		1.165		
<u>Safety Properties</u>				
Impact, cm/2kg (uncured/cured)	7.6/-	5.2/-	5.2/6.7	5.3/6.6
Friction, g/3000 rpm (uncured/cured)	235/-	180/-	240/550	220/450
Onset/Ignition Temp., °F (uncured)	309/-	300/-	292/459	295/455
<u>Mechanical Properties at 77°F</u>				
Shore A Hardness				
24 hours	24	34	42	45
Final	43	50	53	54
σ_m , psi	-	-	62	60
ϵ_m , %	-	-	23	21
E_o , psi	-	-	399	405
<u>Swelling Ratio in Acetone</u>	-	-	2.95	3.10

- a - AP and HMX dried normally; PCDE-TMETN passed once through molecular sieves
b - AP and HMX dried normally; PCDE-TMETN passed twice through molecular sieves
c - AP and HMX extra dry; PCDE-TMETN passed twice through molecular sieves
d - Ingredients as in (c); vacuum mix time after NCO addition doubled
e - Equivalents ratio, PCDE/HT/IPDI = 55/45/100

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(3) 1-Gal Mixes (U)

(U) In early scale-up batches, PCDE-TMETN propellant cured with unacceptable levels of porosity. In subsequent studies of the effects of PCDE pretreatment, extra drying of solids and mix cycle made with 1-lb batches, the porosity was found to be caused mainly by insufficient pretreatment of PCDE. Dryness of the solids was a less important factor. Details of these studies were given in the previous section.

(U) Based on these results, PCDE was passed twice through molecular sieves and solids were all dried for twenty four hours before use in 1-gal batches. One batch, PCDE 10-5 (10GP9663), differed from the other, PCDE 10-4 (10GP9622), in being mixed for twenty minutes in vacuum after NCO addition rather than the ten minutes used in the usual procedure. Although this change in mix time may have little or no bearing on the question of porosity, the longer mix time resulted in greater fluidity. The formulations and some of their properties are shown in Table 9.

d. Batch 10GP9710 (PCDE 10-6) (U)

(U) Because the tensile strength of PCDE 10-5 was somewhat low, the formulation was modified by the addition of more HT. The modified formulation, PCDE 10-6 (10GP9710), was prepared and cast into fourteen 1/4-lb motors. The propellant cured well, and the motors were shipped to RPL. The composition and properties of the propellant are shown in Table 9.

6. 5-Gal Batches (U)

a. Batch 73-05-130 (U)

(U) The PCDE-TMETN candidate propellant was prepared on a 5-gal scale. The composition of the batch, PCDE 60-1 (Batch No. 73-05-130), is shown in Table 10. The mixing of the batch presented no problems, but difficulties were experienced with special casting apparatus designed for

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TABLE 9

COMPOSITION AND PROPERTIES OF PCDE-TMETN
PROPELLANTS PREPARED IN 1-GAL BATCHES (U)

<u>Components</u>	<u>PCDE No. 10-</u>		
	<u>4</u>	<u>5</u>	<u>6</u>
AP, 180 μ , RRD, coated		20.5	
HMX, 5 μ		15.0	
HMX, 150 μ , coated		25.5	
Al, MDX-65		18.0	
DNDPA		0.20	
S		0.10	
FeAA		0.02	
HAA		0.02	
ZnO		0.10	
TMETN ^a	9.644		9.560
PCDE ^{a,b}	9.541		9.560
HT ^b	0.209		0.227
IPDI ^b	1.165		1.212
<u>Safety Properties</u>			
Impact, cm/2kg (uncured/cured)	9.0/7.5	6.7/5.5	10.9/-
Friction, g/3000 rpm (uncured/cured)	450/765	300/675	400/-
Onset Temp., °F ^c	310	292	289
<u>Mechanical Properties at 77°F^d</u>			
σ_m , psi	72	64	83
ϵ_m , %	24	24	24
E_o , psi	380	370	495
<u>Swelling Ratio in Acetone</u>	-	3.18	-

a - Passed through molecular sieves

b - Equivalents ratio, PCDE/HT/IPDI = 55/45/100 for 10-4 and -5 and 53/47/100 for 10-6

c - For uncured propellant.

d - For standard JANNAF specimen. Minibone values for 10-5 are $\sigma_m/\epsilon_m/E_o = 70/23/423$

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TABLE 10

COMPOSITION OF PCDE-TMETN PROPELLANTS FOR 5-GAL SCALE-UP (U)

<u>Components^a</u>	<u>PCDE No.</u>	
	<u>1-88</u>	<u>60-1^b</u>
AP, 180 μ , RRD, coated	20.5	
HMX, 5 μ	15.0	
HMX, 150 μ , coated	25.5	
Al, MDX-65	18.0	
DNDPA	0.20	
S	0.10	
FeAA	0.02	
HAA	0.02	
ZnO	0.10	
TMETN	9.560	9.552
PCDE	9.560	9.552
HT	0.227	0.227
IPDI	1.212	1.211
TDI	-0-	0.019

Safety Properties (uncured)

Impact, cm/2kg	8.6	12.9
Friction, g/3000 rpm	780	250
Onset/Ignition Temp., °F	-	-

Mechanical Properties at 77°F

Shore A Hardness	53	-
σ_m , psi	68	-
ϵ_m , %	22	-
E_o , psi	422	-
Burning Rate, in./sec at 1000 psia	-	0.484

a - PCDE and TMETN passed through molecular sieves. Equivalents ratio, PCDE/HT/IPDI/TDI = 53/47/100/0 and 53/47/100/2 for PCDE 1-88 and 60-1, respectively.

b - Batch No. 73-05-130

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remote operation. BATES motors cast by this method showed a large slump when the surrounding atmospheric pressure was increased from 2" to 15" of mercury. The slump indicates that propellant castability was poor and voids, which collapsed under atmospheric pressure, were trapped in the motors. On the other hand, the castability of the propellant has been measured and is considered adequate at 125°F. For these reasons, the poorer flow encountered in the scale-up batch was probably due to the failure to keep the propellant at 125°F during casting or to cooling of the propellant before release of the vacuum. Cooling of the propellant could make it rigid enough to successfully resist collapse around voids even at one atmosphere pressure.

(U) Subsequent x-ray photographs of the BATES grains indicated small voids in one of them; the others were free of voids and could be salvaged. During the casting which required six and a half hours, the propellant was constantly mixed.

(C) A carton of this propellant cured to a hardness of 24 Shore A, which is low for this propellant. The long mixing may have stripped the coating from the AP and HMX causing a lower state of cure. Two strands of the propellant were burned to determine if the burning rate had been affected. The burning rate, 0.484 in./sec at 1000 psia, compared favorably with that of 0.446 obtained from a 1-gal batch of propellant.

(U) Scale-up to 5-gal required the use of a new lot of coated AP. A preliminary 1-lb batch was made to test the effect of the new lot. The formulation, PCDE 1-88, is given in Table 10. It should be noted that this propellant is Type 3 in the Aerojet hazards classification. The propellant exhibited a Shore A hardness of 31 in four days, which was somewhat low for this propellant. In order to compensate, 2 eq.% TDI was added to the scale-up propellant formulation. TDI was added because the previous lot of AP had been TDI-coated, whereas the new lot was coated with HDI. (After seven days, PCDE 1-88 did attain a hardness of 53.)

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b. Batch 73-05-151 (PCDE 60-2) (U)

(U) Another 5-gal batch of propellant was made to provide aging samples for the Lockheed Propulsion Company. The batch mixed well and was cast manually because the propellant was shown to be Aerojet Type 3D at 125°F, see Section V.D.4.b. Fourteen elliptical cylinders and a 12 x 3-1/2 x 3-1/2" block were cast in vacuo, and a block was spatula cast from the propellant in the lines. All cured satisfactorily in one week at 125°F.

(U) The compositions and properties of the propellants are shown in Table 11. TDI, 2 eq.%, had been added to PCDE 60-1 because previous lots of AP used in the propellant had this isocyanate in the coating. The coated AP used in PCDE No. 60-1 had HDI instead of TDI in the coating. When it was discovered that the hardness of PCDE 60-1 was low, it was not tested mechanically, but instead the subsequent batch, PCDE No. 60-2, was made without the TDI and with more crosslinker to ensure a higher modulus.

(U) Colored inclusions which were the nuclei of larger uncured spots were observed in PCDE-TMETN propellant shipped to the Lockheed Propulsion Company. The inclusions, subjected to analysis by emission and infrared spectroscopy, contained high concentrations of Zn, FeAA and DNDPA. The Zn was very high and, in fact, none was found in the propellant proper. The FeAA concentration was only slightly higher in the inclusions. FeAA, ZnO and DNDPA are added together to the liquid fuel before propellant mixing and the ingredients dispersed by manual agitation. This procedure worked well in the 5-gal batches, 73-05-130 and -161. Henceforth, however, it would seem advisable to add the mixture of solids slowly with mechanical agitation to ensure dispersal of the ZnO. While the exact nature of the reactions occurring are not known, it is believed that the high concentration of basic ZnO could deplete the surrounding area of isocyanate and prevent adequate cure.

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TABLE 11

COMPOSITION AND PROPERTIES OF PCDE-TMETN PROPELLANTS PREPARED IN 5-GAL BATCHES (U)

<u>Components^a</u>	<u>PCDE No. 60-2</u>
AP, 180 μ , RRD, coated	20.5
HMX, 5 μ	15.0
HMX, 150 μ , coated	25.5
Al, MDX-65	18.0
DNDPA	0.20
S	0.10
FeAA	0.02
HAA	0.02
ZnO	0.10
TMETN	9.513
PCDE	9.513
HT	0.255
IPDI	1.278
<u>Safety Properties^b</u>	
Impact, cm/2kg	9.2
Friction, g/3000 rpm	375
Onset/Ignition Temp., °F	298/455
<u>Hardness, Shore A</u>	53
<u>Mechanical Properties at 77°F</u>	
σ_m , psi	61
ϵ_m , %	17
E_o , psi	448

a - PCDE and TMETN passed through molecular sieves. Equivalents ratio, PCDE/HT/IPDI = 50/50/100.

b - For uncured materials.

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c. Batch 73-05-161 (PCDE 60-3) (U)

(U) A third 5-gal batch of PCDE-TMETN propellant was made; its composition, shown in Table 12, is the same as the second one. Three 15-lb BATES grains were cast from the batch, and two BATES grains, which had been cast earlier, but slumped, were topped. The propellant cured to a hardness of 52 (Shore A) in seven days at 125°F.

(U) The BATES grains were x-rayed and sent to AFRPL, including the grains which had been topped.

(U) The table below shows the mechanical properties of the third 5-gal batch, PCDE No. 60-3 (73-05-161). The ambient tensile strength is about 70 psi instead of the target 80 psi. The low value is probably the result of a slight undercuring. It is observed in aging of this type of propellant that there is always an initial post cure.

MECHANICAL PROPERTIES OF PCDE-TMETN PROPELLANT, (60-3,73-05-161) (U)

	Temperature, °F		
	0	77	150
σ_m , psi	502	68	38
ϵ_m , %	14	24	21
E_o , psi	6177	407	220

7. Burning Rates and π_k (U)

(C) Burning rates at -65, 80, and 150°F were obtained from the Propellant Batch 10GP9710 (PCDE 10-6), cast into 1/4-lb motors. The data are summarized in Figure 7. At 80°F and 1000 psia, the burning rate is 0.466 in./sec with a pressure exponent of 0.60. The value of π_k between -65° and 150°F is 0.2%/°F. These data were utilized to fire two 1/4-lb motors at Aerojet to provide information for expediting more extensive testing at AFRPL.

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TABLE 12

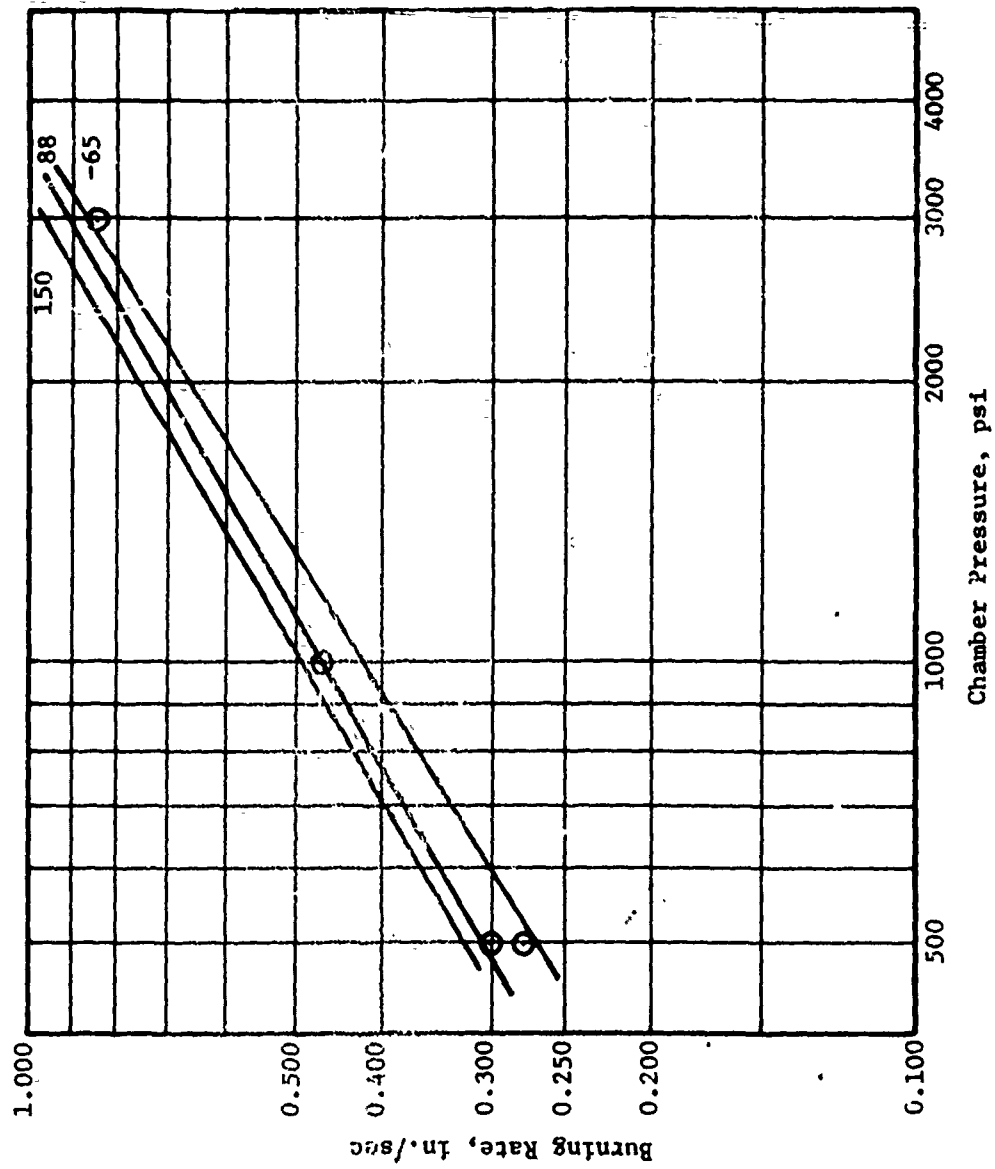
COMPOSITION AND PROPERTIES OF PCDE-TMETN PROPELLANT MADE ON A 5-GAL SCALE (U)

<u>Component</u> ^a	<u>60-3</u> ^b
AP, 180μ, RRD, coated	20.5
HMX, 5μ	15.0
HMX, 150μ, coated	25.5
Al, MDX-65	18.0
DNDPA	0.20
S	0.10
FeAA	0.02
AA	0.02
ZnO	0.10
TMETN	9.513
PCDE	9.513
HT	0.255
IPDI	1.278
<u>Safety Properties</u> ^c	
Impact, cm/2kg	9.0
Friction, g/3000 rpm	665
Onset/Ignition Temp., °F	283/457
<u>Mechanical Properties at 77°F</u> ^d	
σ_m , psi	68
ϵ_m , %	24
E_m , psi	407
<u>Burning Rates, in./sec</u>	
500 psia	0.293
1000 psia	0.456
n	0.61
<u>Hardness, Shore A</u> ^e	52

- a PCDE and TMETN passed through molecular sieves.
Equivalents ratio, PCDE/HT/IPDI = 50/50/100.
- b Also designated Batch 73-05-161
- c Cured propellant
- d Standard JANNAF instron specimen
- e After 7 days cure at 125°F

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BURNING RATE OF PCDE-TMETN PROPELLANT AT -65, 80, and 150°F (PCDE 10-6, 10GP-9710) (U)



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Figure 7

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(C) The solid strand burning rates of PCDE-TMETN Propellant 60-3 (Batch 73-05-161) were determined at ambient temperature from 500 to 3000 psia. The data are shown graphically in Figure 8. The burning rate, 0.456 in./sec at 1000 psia, and the pressure exponent, 0.61, are normal for this formulation.

8. Rohm & Haas 2C1.5-4 Propellant Grains (U)

(C) Table 13 gives weight data for 2C1.5-4 motors delivered to AFRPL. Two additional motors were fired at ASPC. The specific impulse efficiencies were 90.11% and 89.26% at 1000 and 800 psia, respectively. The data are given in Table 14.

(C) The burning rates obtained from these motor firings, 0.444 and 0.530 in./sec at 826 and 1063 psia, respectively, are higher than the rates expected from solid strand data (see previous section) and they give a higher pressure exponent, 0.70, than do the solid strands. The number of motors tested, however, is too small for firm conclusions.

(U) Firing traces for the two motor tests are shown in Figures 9 and 10.

(C) Figure 11 shows the burning rate vs pressure relationship reported by AFRPL on the basis of 12 firings of R & H 2C1.5-4 motors. They report the burning rate at 1000 psia to be 0.49 in./sec with a pressure exponent of 0.53.

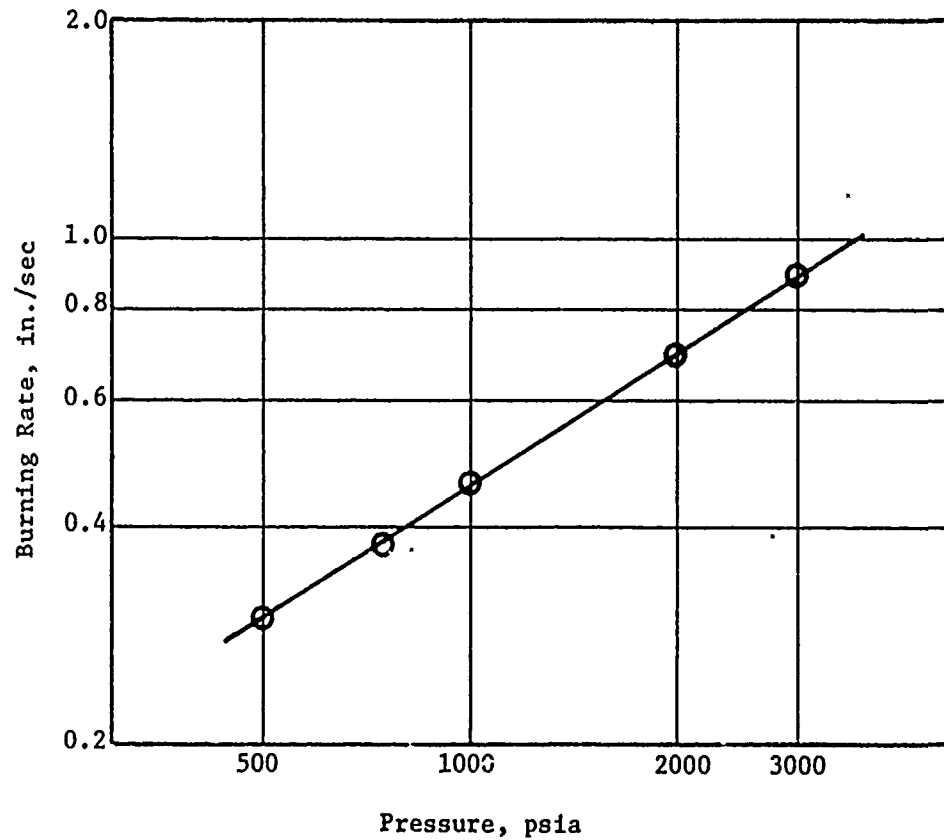
9. Aging Stability (U)

a. Introduction (U)

(U) A 1-gal batch of PCDE-TMETN propellant, PCDE 10-5 (10GP9663), was subjected to aging at 110°F, exposed to the atmosphere or sealed in a friction-top tin can, and 150°F. After two weeks at 150°F, the 2 in. propellant cubes had fissured seriously. No mechanical property data were obtained on these samples, but safety data were obtained. Other data for storage at 77° and 110°F are shown in Table 15.

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SOLID STRAND BURNING RATES FOR PCDE-TMETN PROPELLANT (U)



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Figure 8

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TABLE 13

WEIGHTS OF PCDE-TMETN PROPELLANT GRAINS FOR 1/4-1b MOTORS

<u>Sleeve No.</u>	<u>Weight, g</u>		
	<u>Sleeve</u>	<u>Sleeve + Propellant</u>	<u>Propellant</u>
1	453.18	601.43	148.25
2	448.12	594.39	146.27
3	446.30	593.95	147.65
4 ^a	451.11	581.55	130.44
5	450.47	597.65	147.18
6	448.58	595.61	147.03
7	444.50	591.73	147.23
8	448.39	594.33	145.94
9	449.80	599.46	149.66
10	448.62	598.01	149.39
11	442.91	591.98	149.07
12	448.54	599.31	150.77

^aShort grain

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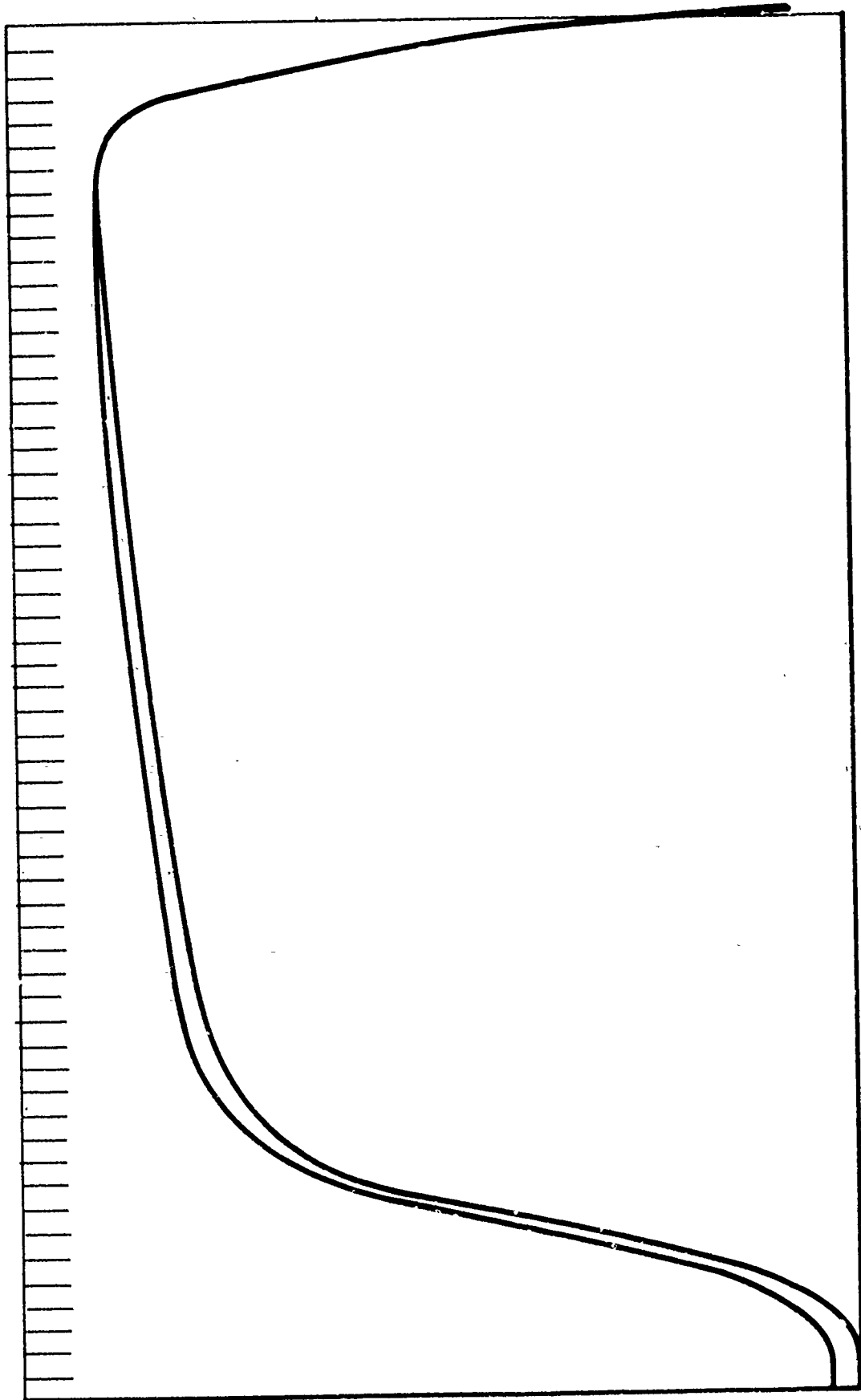
TABLE 14

BALLISTIC PROPERTIES FOR FIRINGS OF ROHM & HAAS
2C1.5-4 GRAINS OF PCDE-TMETN PROPELLANT (U)

<u>Property</u>	<u>Grain No.</u>	
	<u>1</u>	<u>2</u>
Propellant Weight, grams	149.1200	150.9200
Throat Diameter, in.		
Before Firing	.380	.360
After Firing	.375	.358
Average	.378	.359
Average Nozzle Expansion Ratio	9.040	9.050
Action Time, sec	.6090	.5465
Web Burning Time, sec	.5625	.4720
Average Pressure, psia		
Over Action Time	815	1006
Over Web Burning Time	826	1063
Web Time/Action Time	.924	.864
Web P Integral/Action P Integral	.937	.913
Web P Integral/Total P Integral	.937	.913
Action P Integral/Total P Integral	1.000	1.000
CD, lbf/lbf-sec		
Theoretical	.00603	.00602
Experimental	.00591	.00597
C*, ft/sec		
Theoretical	5331	5341
Experimental	5446	5392
C* Efficiency, percent	102.16	100.96
Mass Flow Rate, lbf/sec	.540	.609
Web Burning Rate, in./sec	.444	.530
Average Thrust, lbf	128	148
Theoretical Specific Impulse, lbf-sec/lbf		
With Solidification of Oxides	271.2	271.2
With Supercooling of Oxides	271.2	271.2
Experimental Specific Impulse, lbf-sec/lbf		
Motor Conditions, 15 deg. half-angle	237.4	244.5
Standard Conditions (CF Extrapolation)	242.1	244.4
Specific Impulse Efficiency, percent	89.26	90.11

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FIRING TRACE FOR PCDE-TMETN PROPELLANT -2Cl.5-4 ROHM & HAAS MOTOR #1 (SEE TABLE 14) (U)



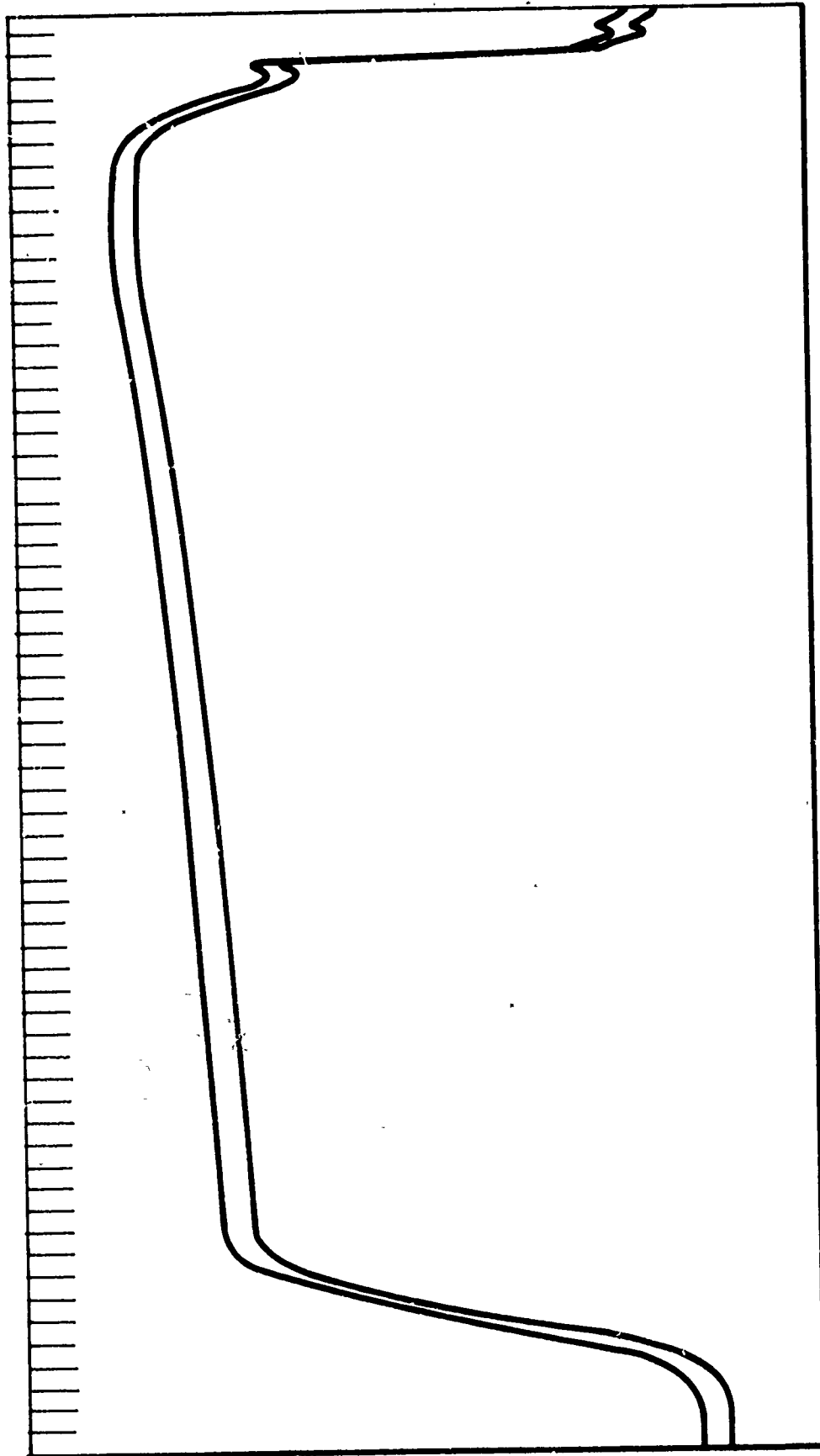
-42-

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Figure 9

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FIRING TRACE FOR PCDE-TMETN PROPELLANT - 2C1.5-4 ROHM & HAAS MOTOR #2 (SEE TABLE 14) (U)

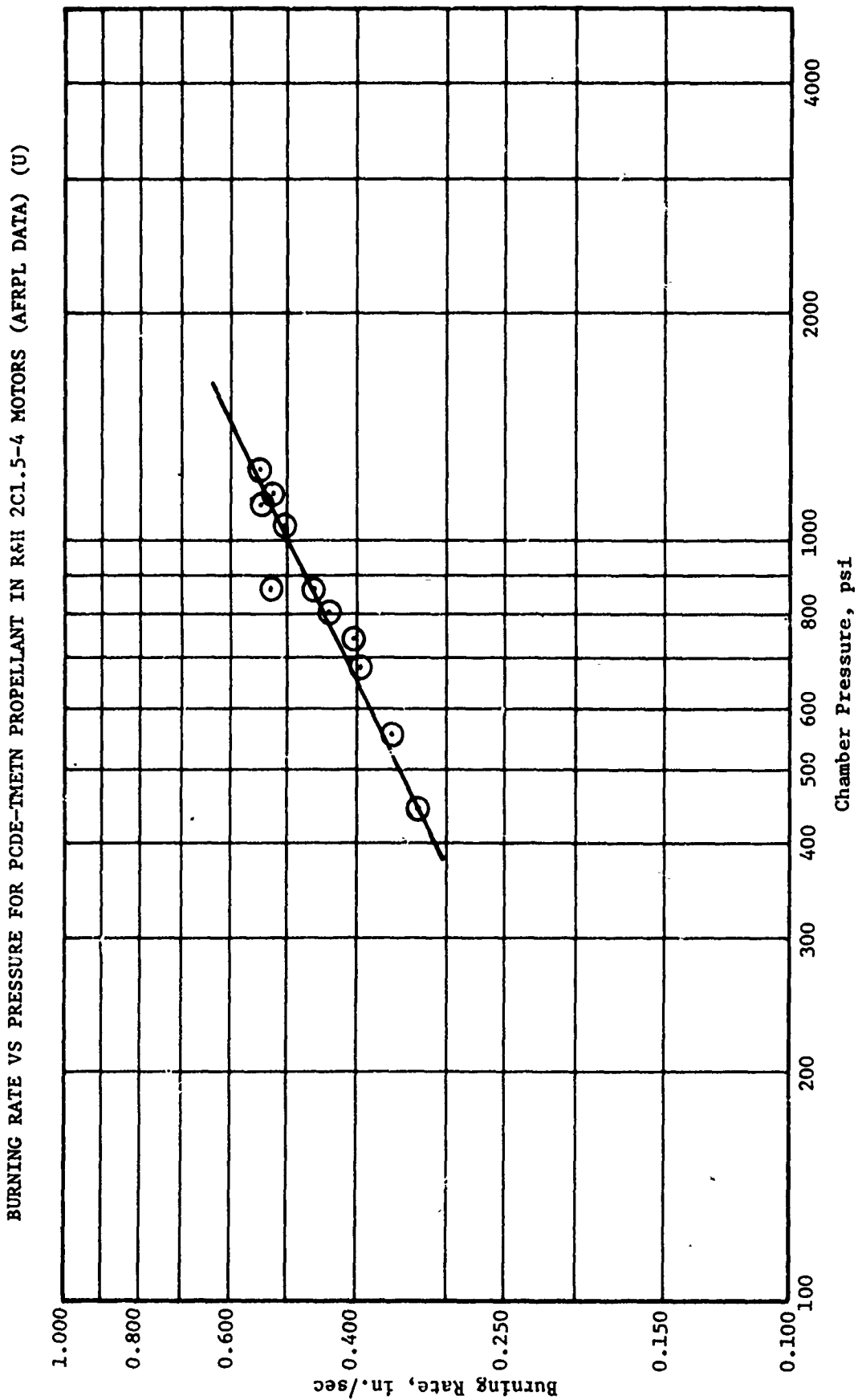


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Figure 10

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Figure 11

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TABLE 15

AGING OF PCDE-TMETN PROPELLANT^a (U)

Mechanical Properties at 77°F (σ_m , psi/ ϵ_m , %/ E_o , psi)

<u>Condition</u> ^b	<u>Time,</u> <u>Weeks</u>	<u>Properties</u> ^c
Initial	0	70/22/457
Exposed, 80°F	6	70/22/436
Exposed, 80°F	12	77/20/516
Exposed, 110°F	2	79/21/491
	4	77/18/549
	8	81/20/530
Sealed, 110°F	2	78/21/477
	4	75/18/579
	8	75/18/584

Safety Properties

<u>Condition</u> ^b	<u>Time,</u> <u>Weeks</u>	<u>Impact,</u> <u>cm/2kg</u>	<u>Friction,</u> <u>g/3000 rpm</u>	<u>Onset</u> <u>Temp., °F</u>
Initial	0	7.8	700	300
Exposed, 77°F	12	10.8	1300	305
Exposed, 110°F	8	7.4	900	297
Sealed, 110°F	8	7.5	337	291
Exposed, 150°F	6	7.6	191	281

Burning Rates

<u>Condition</u> ^b	<u>Time,</u> <u>Weeks</u>	<u>Burning Rate,</u> <u>in/sec at 1000 psia</u>
Initial	0	0.448
Exposed, 77°F	6	0.435
Exposed, 77°F	12	0.432
Exposed, 110°F	8	0.431
Sealed, 110°F	8	0.442

a PCDE No. 10-5 (Batch 10GP9663) aged as 2-in. cubes

b Sealed specimens in closed friction-top tin can

c Minibone specimen

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b. Mechanical Properties (U)

(U) The mechanical properties of propellant exposed at 80°F were essentially unchanged after 12 weeks. At 110°F, however, these properties improved, if anything, after two weeks and remained unchanged through the eighth week. In sealed-can aging at 110°F, improvement resulted after two weeks, but thereafter there was a slight hardening with time. After eight weeks, the propellant still has very useful properties.

c. Safety Properties (U)

(U) Open exposure does not change the safety properties of the propellant even after 8 weeks at 110°F or 12 weeks at 77°F. When sealed at 110°F for eight weeks, the propellant shows only a greater sensitivity to friction. However, only one datum is available and further testing would be required to establish a clear-cut trend. A similar effect is observed with open exposure at 150°F, but the change of friction sensitivity is more pronounced in this case.

d. Burning Rates (U)

(U) Apparently, there is a small decrease of the burning rate with time in those samples which remain exposed. This may indicate a small loss of plasticizer because the effect is not observed in specimens which were kept in sealed cans. More data would be required to establish the significance of the small change observed.

E. PCDE-BDNPA/F PROPELLANT STUDIES (U)

1. Introduction (U)

(C) Table 16 presents calculated performance potentials of all-AP propellants suitable for a Class 2 requirements. The data in Table 16, representing the more optimistic prediction of efficiency, indicate that an all-AP propellant could deliver 250.8 lbf/lbm/sec at a density of 0.068 lb/in.³. Such a propellant would contain 51 wt% AP and 22 wt% Al. The current baseline composition contains, therefore, 51 wt% AP, 22 wt% aluminum, and a 1/1 ratio of BDNPA/F plasticizer to the crosslinked PCDE polymer.

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TABLE 16
PERFORMANCE POTENTIAL OF ALL-AP PCDE-BDNPA/F (1 to 1) PROPELLANTS (U)

AP wt%	Al wt%	Binder Vol. Frac.	Density lb/in. ³	Specific Impulse		
				Theo.	Del. ^a	$\bar{n}$ (K=0.7) ^b
58	18	0.315	0.06795	262.1	250.1	257.9
54.5	20	0.335	0.06799	263.0	250.7	258.6
56	20	0.317	0.06832	262.5	250.1	258.9
51	22	0.354	0.06799	263.4	250.8	258.7
54	22	0.318	0.06871	262.7	249.9	259.7
47.5	24	0.374	0.06799	263.0	250.3	258.2
49	24	0.356	0.06835	262.9	250.1	258.9

^a Presence of fluorine included; expected for mass flow rate = 400 lb/sec with exposed area of 400 in.².

^b Figure of merit which includes effect of density

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2. Burning Rates (U)

a. Effect of Iron Oxide and UFAP (U)

(C) A burning rate of 1.6 in./sec was achieved earlier⁽¹⁾ by incorporating 15 wt% 0.5 μ AP into the PCDE-BDNPA/F baseline propellant, but only at the expense of increasing the pressure exponent to 0.75. In efforts to lower the exponent, Fe₂O₃ was used. This iron catalyst was selected because its catalytic effect is more pronounced at the lower than at the higher pressures. Also, because it accelerates burning, it might be possible to reduce the amount of 0.5 μ AP required to achieve the target burning rate and, thereby, reduce the pressure exponent. The results are indicated in Tables 17 through 19 and are summarized in Figure 12.

(C) In the absence of UFAP, 1.5 wt% iron oxide will give a burning rate of 1.26 in./sec, a rate just in the range of interest to AFRPL. For such propellants the pressure exponent is less than 0.5. The data seem to indicate that rates up to 1.3 in./sec with a pressure exponent of 0.6 may be obtained with 1.0 wt% iron oxide and 10 wt% 0.5 μ AP. With 15 wt% fine oxidizer the pressure exponents are of the order of 0.7.

(C) The burning rates achieved in this study are not as high as those obtained in an earlier study⁽¹⁾ with UFAP. This is, however, due to the fact that the present studies were based on formulations having less 6 μ AP than those of the earlier study. Thus, there is no conflict in the data. In fact, if one uses 10 wt% UFAP, as in the earlier formulations, with 1.0 wt% iron oxide, it may be possible to obtain a burning rate of 1.5 in./sec at a pressure exponent of about 0.65.

b. Effect of Copper Chromite and UFAP (U)

(C) With the goal of achieving a burning rate of 1.6 in./sec at 1000 psia with a minimum of UFAP, copper chromite, a burning rate catalyst, was added to PCDE-BDNPA/F propellant. The results were generally poor; copper chromite interfered with the cure and caused gassing. No valid burning rates were obtained from this study, but the compositions of the propellants are shown in Tables 20 through 23 to indicate the extent of the study.

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TABLE 17

EFFECT OF Fe_2O_3 ON THE BURNING RATES AND PROPERTIES
OF PCDE-BDNPA/F PROPELLANTS (U)

Components	PCDE No.			
	212	213	214	215
AP, 6 μ		30.0		
AP, 180 μ , RRD, coated		21.0		
Al, H-60		22.0		
Neozone D		0.10		
FeAA		0.05		
HAA		0.05		
Fe_2O_3	-0-	0.40	1.00	1.50
BDNPA/F ^a	13.400	12.900	12.400	11.900
PCDE ^{a,b}		11.877		
HT ^b		0.260		
TDI ^b		1.262		
<u>Safety Properties</u>				
Impact, cm/2 kg, (uncured/cured)	10.4/11.6	12.7/13.4	12.9/7.2	13.0/7.0
Friction, g/3000 rpm				
(uncured/cured)	230/480	270/520	200/450	340/580
Onset/Ignition Temp., °F	308/643	316/583	321/579	303/580
<u>Burning Rates, in./sec</u>				
1000 psia	0.978	1.111	1.130	1.257
500 psia	0.731	0.830	0.826	0.964
n	0.42	0.42	0.45	0.38
<u>Mechanical Properties at 77°F</u>				
σ_m , psi	62	114	119	135
ϵ_m , %	40	27	25	24
E_m , psi	201	510	550	660
<u>Swelling Ratio in Acetone</u>				
Initial	3.55	3.53	3.56	3.49
Aged 14 days, 150°F				
Unwrapped	3.32	3.23	3.42	3.35
Al-wrapped	3.39	3.41	3.28	3.31
<u>Weight Loss, aged 14 days, 150°F, %</u>				
Unwrapped	0.481	0.483	0.501	0.453
Al-wrapped	0.143	0.128	0.140	0.130

a - Passed through molecular sieves

b - Equivalents ratio, PCDE/HT/TDI = 55/45/112

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TABLE 18

EFFECT OF UFAP ON BURNING RATES AND PROPERTIES
OF PCDE-BDNPA/F PROPELLANTS CONTAINING Fe_2O_3 (U)

Components	PCDE No.			
	216	217	218	219
AP, 0.5 μ	-0-	5.0	10.0	15.0
AP, 6.0 μ	30.0	25.0	20.0	15.0
AP, 180 μ , RRD, coated			21.0	
Al, H-60			22.0	
Neozone D			0.10	
FeAA			0.05	
HAA			0.05	
Fe_2O_3			0.50	
BDNPA/F ^a			12.900	
PCDE ^{a,b}			11.877	
HT ^b			0.260	
TDI ^b			1.262	
<u>Safety Properties</u>				
Impact, cm/2 kg, (uncured/cured)	17.6/7.2	16.4/6.5	14.9/6.2	13.4/6.4
Friction, g/3000 rpm (uncured/cured)	165/720	265/940	165/1165	220/680
Onset/Ignition Temp., °F	308/580	300/581	290/582	300/576
<u>Burning Rate, in./sec</u>				
1000 psia	1.075	1.145	1.185	1.372
500 psia	0.782	0.777	0.765	0.834
n	0.46	0.56	0.63	0.72
<u>Mechanical Properties at 77°F</u>				
σ_m , psi	112	110	83	94
ϵ_m , %	28	29	31	31
E_m^o , psi	500	470	330	4000
<u>Swelling Ratio in Acetone</u>				
Initial	3.61	3.50	3.21	3.36
Aged 14 days, 150°F				
Unwrapped	3.25	3.29	3.16	3.16
Al-wrapped	3.30	3.16	2.80	3.09
<u>Weight Loss, aged 14 days, 150°F, %</u>				
Unwrapped	0.530	0.485	0.497	0.530
Al-wrapped	0.135	0.116	0.134	0.119

a - Passed through molecular sieves

b - Equivalents ratio, PCDE/HT/TDI = 55/45/112

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TABLE 19

EFFECT OF UFAP ON BURNING RATES AND PROPERTIES OF
PCDE-BDNPA/F PROPELLANTS CONTAINING Fe_2O_3 (U)

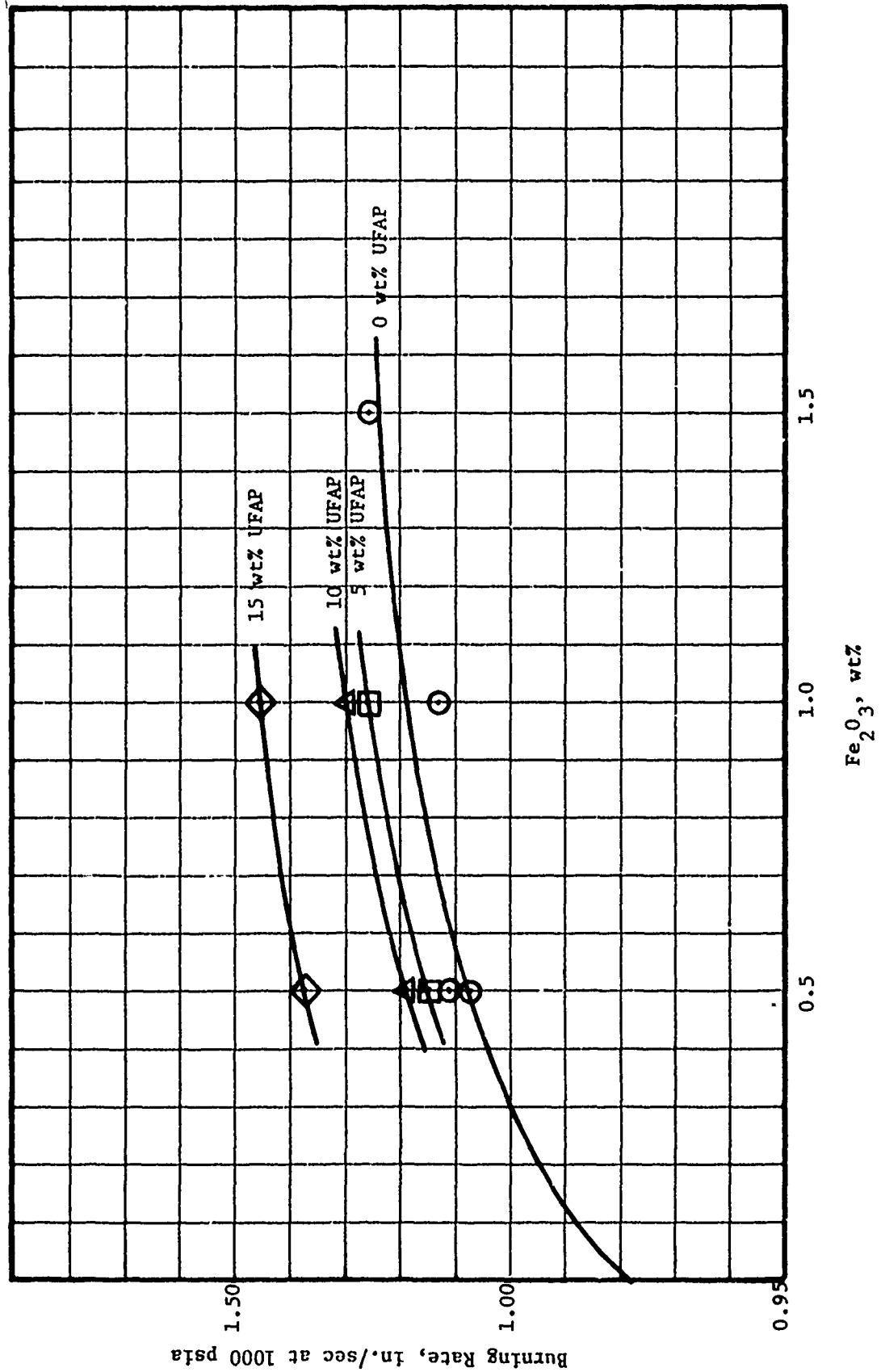
Components	PCDE No.			
	220	221	222	223
AP, 0.5 μ	-0-	5.0	10.0	15.0
AP, 6 μ	30.0	25.0	20.0	15.0
AP, 180 μ , RRD, coated		21.0		
Al, H-60		22.0		
Neozone D		0.10		
FeAA		0.05		
HAA		0.05		
Fe_2O_3		1.00		
BDNPA/F ^a		12.400		
PCDE ^{a,b}		11.877		
HT ^b		0.260		
TDI ^b		1.262		
<u>Safety Properties</u>				
Impact, cm/2kg (uncured/cured)	12.0/8.7	12.4/7.5	12.0/8.0	8.9/9.3
Friction, g/3000 rpm (uncured/cured)	235/435	280/570	265/365	285/470
Onset/Ignition Temp., °F	336/581	278/580	328/577	310/573
<u>Burning Rate, in./sec</u>				
1000 psia	1.123	1.256	1.295	1.448
500 psia	0.819	0.914	0.853	0.894
n	0.45	0.46	0.60	0.70
<u>Mechanical Properties at 77°F</u>				
σ_m , psi	93	90	90	106
ϵ_m , %	31	30	30	28
E_o , psi	380	370	370	470
<u>Swelling Ratio in Acetone</u>				
Initial	3.47	3.48	3.31	3.21
Aged 14 days, 150°F				
Unwrapped	3.18	3.10	3.10	2.68
Al-wrapped	3.41	3.15	3.02	2.49
<u>Weight Loss, Aged 14 days, 150°F, %</u>				
Unwrapped	0.471	0.442	0.427	0.439
Al-wrapped	0.135	0.113	0.121	0.107

a - Passed through molecular sieves

b - Equivalents ratio, PCDE/HT/TDI = 55/45/112

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EFFECT OF Fe_2O_3 ON THE BURNING RATES OF PCDE-BDPA/F PROPELLANTS CONTAINING 0.5% AP (U)



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Figure 12

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TABLE 20

COMPOSITION OF PCDE-BDNPA/F PROPELLANTS CONTAINING COPPER CHROMITE (U)

<u>Components</u>	<u>PCDE No.</u>			
	<u>236</u>	<u>237</u>	<u>238</u>	<u>239</u>
AP, 6μ		30.0		
AP, 180μ, RRD, coated		21.0		
Al, H-60		22.0		
Neozone D		0.10		
FeAA		0.05		
HAA		0.05		
Copper Chromite	-0-	0.50	1.0	1.5
BDNPA/Fa	13.40	12.90	12.40	11.90
PCDE ^{a,b}		11.877		
HT ^b		0.260		
TDI ^b		1.262		

Safety Properties^c

Impact, cm/2kg	-	-	-	7.5
Friction, g/3000 rpm	-	-	-	100

a - Passed through molecular sieves

b - Equivalents ratio, PCDE/HT/TDI = 55/45/112 for all batches

c - Uncured

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TABLE 21

COMPOSITION OF PCDE-BDNPA/F PROPELLANTS CONTAINING
5.0 WT% UFAP AND COPPER CHROMITE (U)

<u>Component</u> ^a	<u>PCDE No.</u>			
	<u>240</u>	<u>241</u>	<u>242</u>	<u>243</u>
AP, 0.5μ		5.0		
AP, 6μ		25.0		
AP, 180μ, RRD, coated		21.0		
Al, H-60		22.0		
Neozone D		0.10		
FeAA		0.05		
HAA		0.05		
Copper Chromite	-0-	0.50	1.00	1.50
BDNPA/F	13.400	12.900	12.400	11.900
PCDE		11.877		
HT		0.260		
TDI		1.262		

Safety Properties^b

Impact, cm/2kg	-	-	-	10.6
Friction, g/3000 rpm	-	-	-	340

a - PCDE and BDNPA/F passed through molecular sieves. Equivalents ratio,
PCDE/HT/TDI = 55/45/112 for all batches

b - Uncured

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TABLE 22

COMPOSITION OF PCDE-BDNPA/F PROPELLANTS CONTAINING
10.0 WT% UFAP AND COPPER CHROMITE (U)

<u>Components</u> ^a	<u>PCDE No.</u>			
	<u>244</u>	<u>245</u>	<u>246</u>	<u>247</u>
AP, 0.5μ		10.0		
AP, 6μ		20.0		
AP, 180μ, RRD, coated		21.0		
Al, H-60		22.0		
Neozone ⁿ		0.10		
FeAA		0.05		
HAA		0.05		
Copper Chromite	-0-	0.50	1.00	1.50
BDNPA/F	13.400	12.900	12.400	11.900
PCDE		11.877		
HT		0.260		
TDI		1.262		
<u>Safety Properties</u> ^b				
Impact, cm/2kg	-	-	-	9.4
Friction, g/3000 rpm	-	-	-	165

a - PCDE and BDNPA/F passed through molecular sieves. Equivalents ratio
PCDE/HT/TDI = 55/45/112 for all batches

b - Uncured

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TABLE 23

COMPOSITION OF PCDE-BDNPA/F PROPELLANTS CONTAINING
15.0 WT% V/FAP AND COPPER CHROMITE (U)

Components ^a	PCDE No.			
	248	249	250	251
AP, 0.5μ		15.0		
AP, 6 μ		15.0		
AP, 180μ, RRD, coated		21.0		
Al, H-60		22.0		
Neozone D		0.10		
FeAA		0.05		
HAA		0.05		
Copper Chromite	-0-	0.50	1.00	1.50
BDNPA/F	13,400	12,900	12,400	11,900
PCDE		11.877		
HT		0.260		
TDI		1.262		
Safety Properties ^b				
Impact, cm/2kg	-	-	-	11.1/7.0
Friction, g/3000 rpm.	-	-	-	250/400
Onset Temp., °F	-	-	-	270/252
Ignition Temp., °F	-	-	-	620/620

a - PCDE and BDNPA/F passed through molecular sieves. Equivalents ratio,
PCDE/HT/TDI - 55/45/112 for all batches.

b - Uncured/cured.

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c. Effect of 1 μ and 3 μ AP (U)

(C) The burning rates of PCDE-BDNPA/F propellants with 1 and 3 μ AP were determined and are reported in Tables 24 and 25 and Figures 13 through 16. With 3 μ AP replacing all the 6 μ AP and 15% of the 180 μ AP, the burning rate at 1000 psia was 1.65 in./sec. Even higher rates, 1.83 in./sec, were achieved with 1 μ AP in place of 6 μ oxidizer. The pressure exponent increased as the 6 μ oxidizer was replaced with the finer size.

d. Burning Rate Equation (U)

(C) The data on the burning rate of these propellants with 0.5, 1.0, and 3.0 μ AP allowed derivation of equations for approximating the burning rate and pressure exponent of any combination of these materials in the PCDE-BDNPA/F baseline propellant. The equations are

$$r_{1000} = 1.986x + 2.842y + 3.516w + 4.550v + 4.589z$$

$$n = 1.722y + 1.894w + 2.667v + 2.867z$$

where x, y, w, v and z are the fractions of the propellant of 180, 6, 3, 1 and 0.5 μ AP, respectively. The limited usefulness of these equations must be realized. They apply only to the baseline propellant, are based on a restricted sampling, have not been tested, and are intended only for guidance of formulation.

(C) Based on these equations, it was calculated that a formulation containing 28.33 wt% 180 μ and 22.67 wt% 0.5 μ AP would burn at 1.57 in./sec at a pressure exponent of 0.65. Propellants with various types of aluminum were made to test this prediction. The compositions are shown in Table 26. Solid strand data are shown in Table 26 and Figure 17. Although some of the rates were high, the pressure exponents were too high to be practical.

e. Problem Area (U)

(C) The required burning rate, 1.3 in./sec at 1000 psia, had been achieved early in the program by replacing both 6 μ and 180 μ

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TABLE 24

COMPOSITION AND PROPERTIES OF PCDE-BDNPA/F PROPELLANTS CONTAINING 3μ AP (U)

Components ^a	PCDE No.			
	252	253	254	255
AP, 3μ	-0-	15.0	30.0	45.0
AP, 6μ	30.0	15.0	-0-	-0-
AP, 180μ, RRD, coated	21.0	21.0	21.0	6.0
Al, H-60			22.0	
Neozone D			0.10	
FeAA			0.05	
HAA			0.05	
BDNPA/F			13.400	
PCDE			11.877	
HT			0.260	
TDI			1.262	
<u>Safety Properties</u>				
Impact, cm/2 kg (uncured/cured)	--	--	--	25/7.8
Friction, g/3000 rpm (uncured/cured)	--	--	--	380/390
Onset/Ignition Temp., °F ^b	-	-	-	302/632
<u>Burning Rates, in./sec</u>				
500 psia	0.744	--	0.804	1.039
1000 psia	1.076	--	1.295	1.651
n	0.53	--	0.69	0.67

^a PCDE and BDNPA/F passed through molecular sieves. Equivalents ratio, PCDE/HT/TDI = 55/45/112 for all batches.

^b For uncured specimens.

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TABLE 25

COMPOSITION AND PROPERTIES OF PCDE-BDNPA/F PROPELLANTS CONTAINING 1μ AF

Components ^a	PCDE No.			
	260	251	262	263
AP, 1μ	-0-	10.0	20.0	30.0
AP, 6μ	30.0	20.0	10.0	-0-
AP, 180μ, RRD, coated	21.0	21.0	21.0	21.0
Al, H-60		22.0		
Neozone D		0.10		
FeAA		0.05		
HAA		0.05		
BDNPA/F		13.400		
PCDE		11.877		
HT		0.260		
TDI		1.262		
<u>Safety Properties</u>				
Impact, cm/2 kg (uncured/cured)	--	--	--	13.6/-
Friction, g/3000 rpm (uncured/cured)	--	--	--	380/-
Onset/Ignition Temp., °F ^b	--	--	--	308/631
<u>Burning Rate, in./sec</u>				
500 psia	0.714	0.794	0.907	1.049
1000 psia	1.059	1.237	1.490	1.833
n	0.50	0.64	0.72	0.80

^a PCDE and BDNPA/F passed through molecular sieves; equivalents ratio,
PCDE/HT/TDI = 55/45/112 for all batches.

^b For uncured specimens

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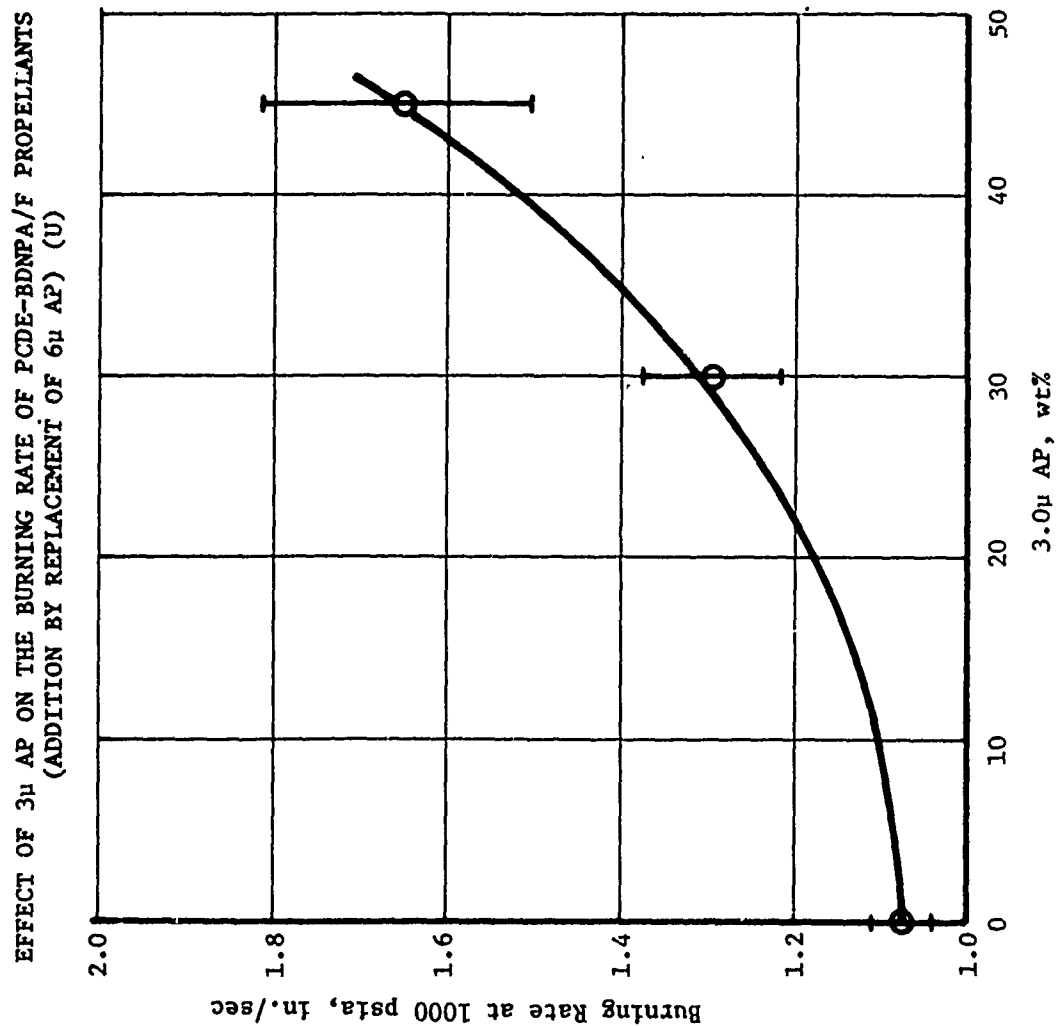
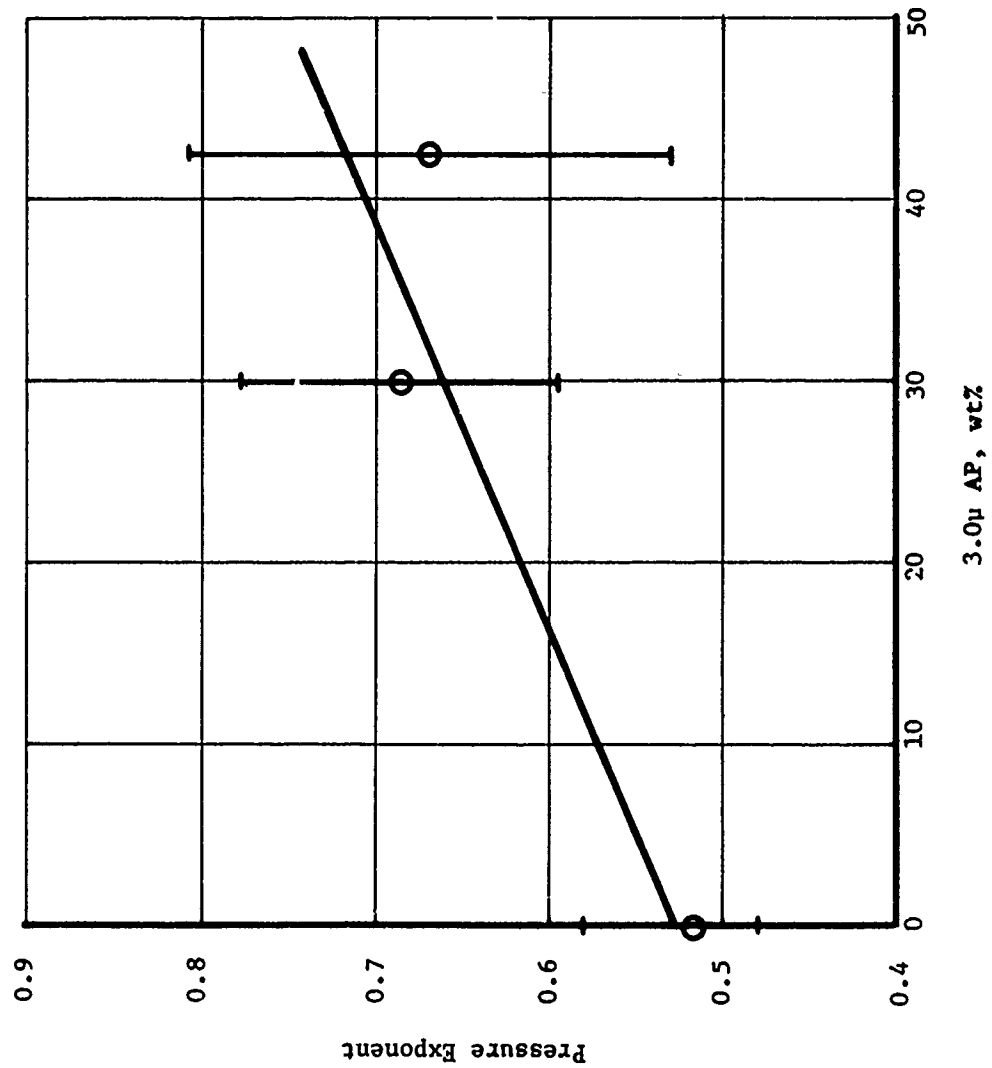


Figure 13

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EFFECT OF 3μ AP ON THE BURNING RATE PRESSURE EXPONENT OF
PCDE-BDNPA/F PROPELLANTS (ADDITION AT EXPENSE OF 6μ AP) (U)



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Figure 14

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EFFECT OF 1μ AP ON THE BURNING RATE OF PCDE-BDNPA/F PROPELLANTS
(ADDITION BY REPLACEMENT OF 6μ AP) (U)

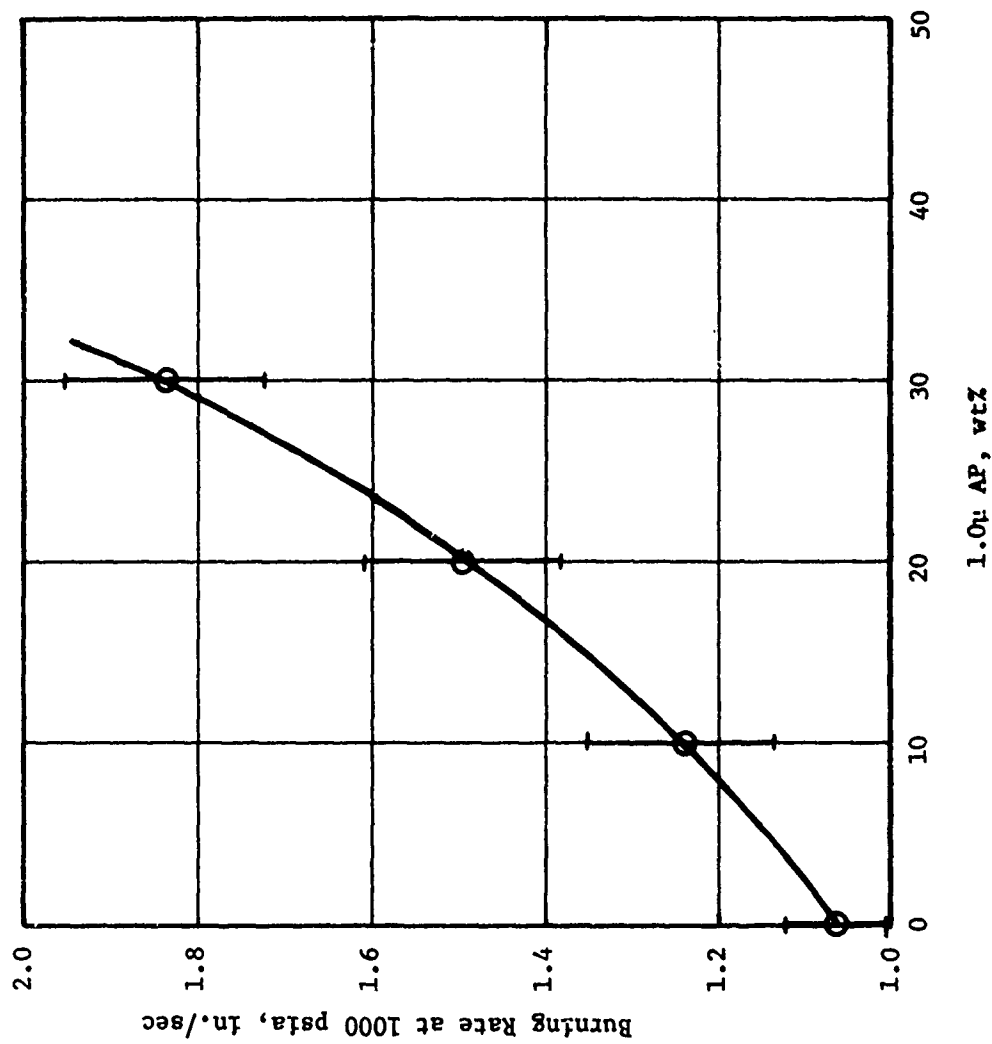
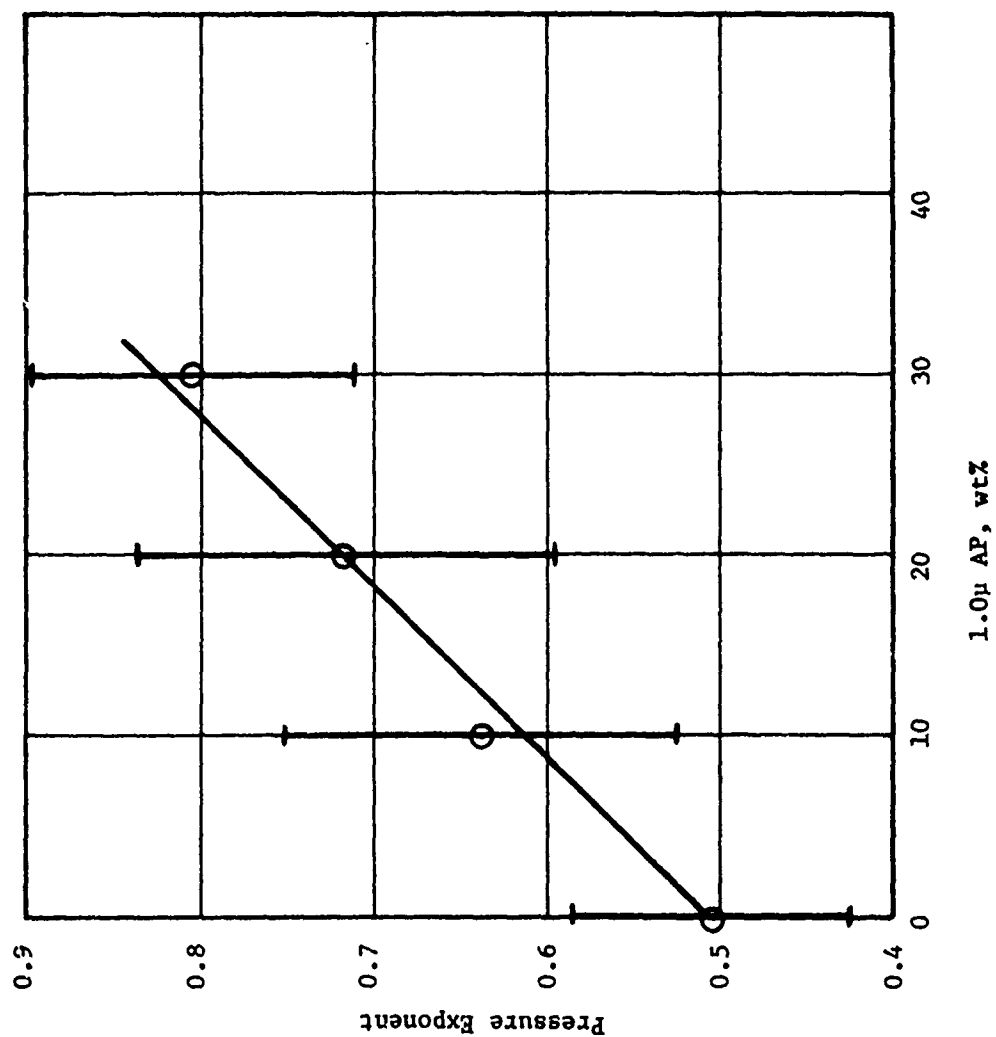


Figure 15

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EFFECT OF 1μ AP ON THE BURNING RATE PRESSURE EXPONENT OF
PCDE-BDPA/F PROPELLANTS (ADDITION BY REPLACEMENT OF 6μ AP) (U)



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Figure 16

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TABLE 26

COMPOSITION AND BURNING RATES OF PCDE-BDNPA/F PROPELLANTS (U)

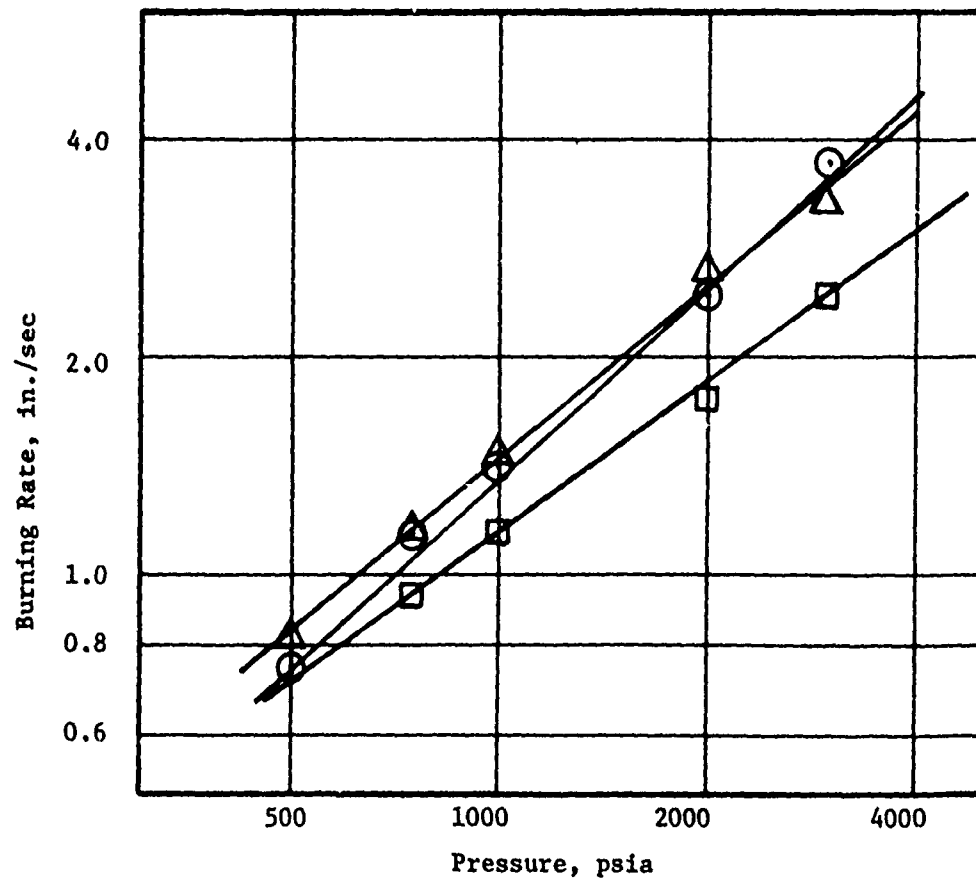
<u>Components^a</u>	<u>PCDE No.</u>		
	<u>274</u>	<u>275</u>	<u>276</u>
AP, 0.5μ		22.67	
AP, 180μ, RRD		28.33	
Al, H-60	22.00	0	0
Al, MDX-65	0	22.00	0
Al, H-95	0	0	22.00
FeAA		0.05	
HAA		0.05	
BDNPA/F		12.712	
PCDE		12.712	
HT		0.170	
DEA		0.067	
TDI		1.238	
<u>Safety Properties^b</u>			
Impact, cm/2kg	14.9	13.7	13.0
Friction, g/3000 rpm	139	132	175
Onset/Ignition Temp., °F	311/631	311/631	302/631
<u>Hardness, Shore A</u>	13	56	32
<u>Burning Rates, in./sec</u>			
500 psia	0.743	0.714	0.842
1000 psia	1.371	1.158	1.448
n	0.88	0.70	0.78

a PCDE and BDNPA/F passed through molecular sieves. Equivalents ratio, PCDE/HT/DEA/TDI = 60/30/10/112. Shell PCDE Lot 44.

b For uncured samples.

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BURNING RATES OF PCDE-BDNPA/F PROPELLANTS (U)



PCDE No.

○ 274

□ 275

△ 276

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Figure 17

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(C) AP with 5 wt% of 0.5 μ oxidizer.⁽¹⁾ Such a formulation is difficult to process owing to the poor balance of small and large particle sizes. The burning rate had been attained with H-60 aluminum. Propellant with this aluminum has exhibited poorer cures than that containing MDX-65, which gives lower burning rates (see Table 26). A series of propellants was made, therefore, to determine the burning rates obtainable with MDX-65, H-60 and H-95 aluminum. The results of the study were not conclusive.

(C) The composition and properties of the propellants are shown in Tables 27 through 29 and the burning rates are plotted in Figure 18. It is apparent that the burning rates are lower than were obtained earlier without the use of 0.5 μ AP; (see PCDE 212, Table 17) i.e. about 0.97 in./sec at 1000 psia. Except for the series containing the H-95, the burning rate changes very little with increasing UFAP. Moreover, even with 10% of 0.5 μ AP the pressure exponent is no higher than about 0.5. Contrary to reports by others, the highest burning rates were obtained with MDX-65.

(U) Clarification of these data are required and work is in progress to determine whether the problem is the result of UFAP which has agglomerated, a mix cycle which is too short to allow adequate dispersal of the solids, or a characteristic of PCDE Lot 6+8 which was used to prepare the test propellants.

3. IPDI-Cured Propellants (U)

(U) IPDI which was instrumental in extending the potlife of PCDE-TMETN propellants was investigated for this same purpose in PCDE-BDNPA/F propellants. The first propellants made (Table 30) did not cure. The Neozone D in these propellants was the cause; similar behavior was observed with the PCDE-TMETN system (Section V.D.2). A second series of

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TABLE 27

COMPOSITION AND PROPERTIES OF PCDE-BDNPA/F PROPELLANTS
CONTAINING MDX-65 AND VARYING CONTENTS OF 0.5μ AP (U)

Components ^a	PCDE No.			
	295	296	297	298
AP, 0.5μ	4.0	6.0	8.0	10.0
AP, 6μ	26.0	24.0	22.0	20.0
AP, 180μ, RRD		21.0		
Al, MDX-65		22.0		
FeAA		0.05		
HAA		0.05		
BDNPA/F		11.585		
PCDE		12.743		
HT		0.565		
IPDI		2.007		
<u>Safety Properties^b</u>				
Impact, cm/2kg	-	-	-	7.6
Friction, g/3000 rpm	-	-	-	360
Onset/Ignition Temp., °F	-	-	-	270/631
<u>Mechanical Properties at 77°F^c</u>				
σ_m , psi	169	156	126	52 ^d
ϵ_m , %	22	21	26	21 ^d
E_o^m , psi	923	846	586	291 ^d
<u>Burning Rate, in./sec</u>				
500 psia	0.742	0.712	0.715	0.700
1000 psia	0.972	0.966	0.985	0.993
n	0.39	0.44	0.46	0.50

a - PCDE Lot 6+8 and BDNPA/F run through molecular sieves; equivalents ratio,
PCDE/HT/IPDI = 40/60/85

b - Cured

c - Minibone specimens

d - One sample only

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TABLE 28

COMPOSITION AND PROPERTIES OF PCDE-BDNPA/F PROPELLANTS
CONTAINING H-60 AND VARYING AMOUNTS OF 0.5μ AP (U)

Components ^a	PCDE No.			
	306	307	308	309
AP, 0.5μ	4.0	6.0	8.0	10.0
AP, 6.0μ	26.0	24.0	22.0	20.0
AP, 180μ, RRD		21.0		
Al, H-60		22.0		
FeAA		0.05		
HAA		0.05		
BDNPA/F		11.484		
PCDE		12.632		
HT		0.560		
IPDI		2.224		
Safety Properties ^b				
Impact, cm/2kg	-	-	-	7.2
Friction, g/3000 rpm	-	-	-	365
Onset/Ignition Temp., °F	-	-	-	316/633
Burning Rates, in./sec				
500 psia	0.619	0.586	0.622	0.626
1000 psia	0.835	0.834	0.875	0.882
n	0.43	0.51	0.49	0.50

a - PCDE Lot. 6+8 and BDNPA/F passed through molecular sieves. Equivalents ratio,
PCDE/HT/IPDI = 40/60/95.

b - Cured specimens.

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TABLE 29

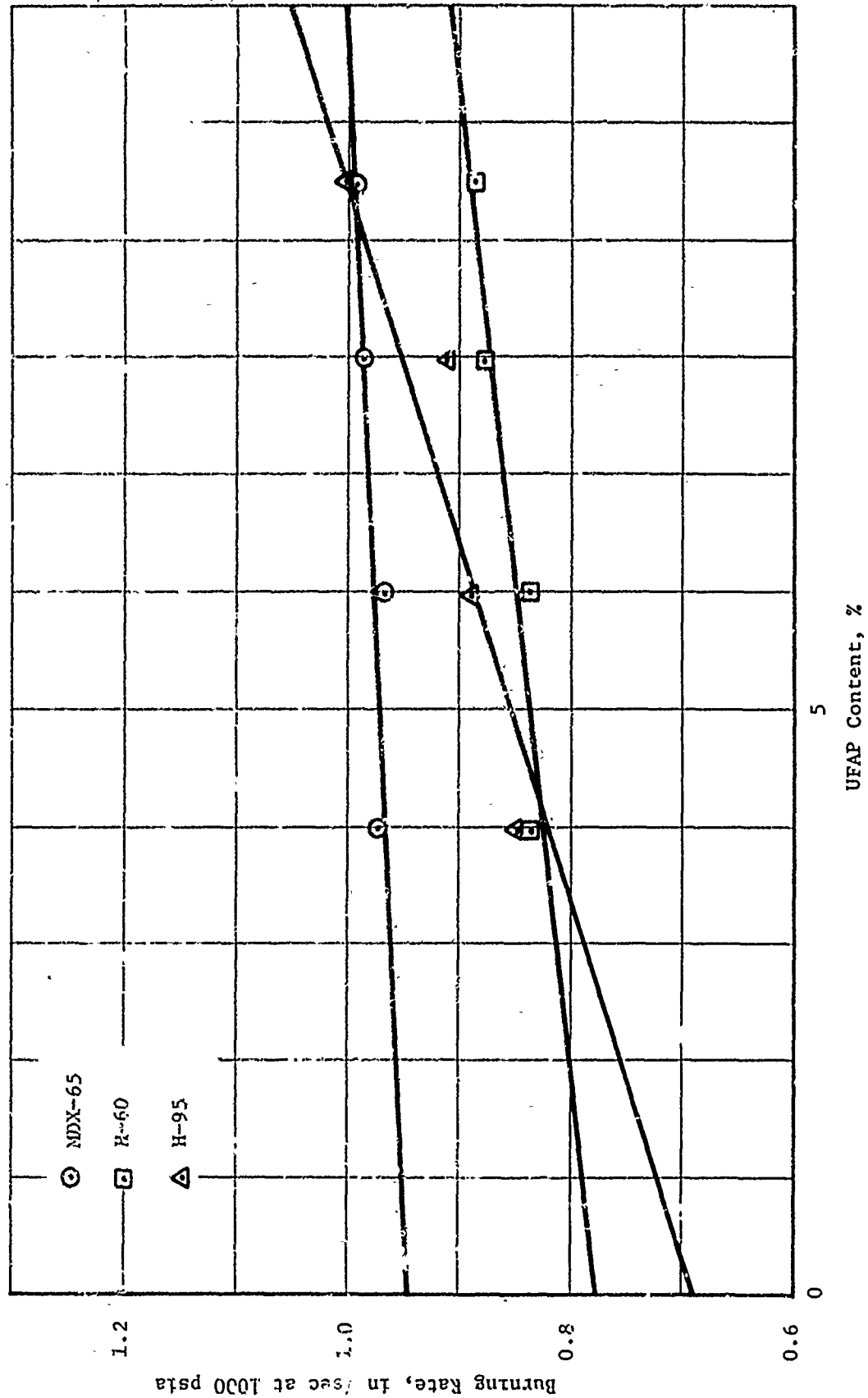
COMPOSITION AND PROPERTIES OF PCDE-BDNPA/F PROPELLANTS
CONTAINING H-95 AND VARYING AMOUNTS OF 0.5μ AP (U)

<u>Components^a</u>	<u>PCDE No.</u>			
	<u>310</u>	<u>311</u>	<u>312</u>	<u>313</u>
AP, 0.5μ	4.0	6.0	8.0	10.0
AP, 6.0μ	26.0	24.0	22.0	20.0
AP, 180μ, RRD			21.0	
Al, H-95			22.0	
S-8			0.2	
FeAA			0.05	
HAA			0.05	
BDNPA/F			11.399	
PCDE			12.538	
HT			0.556	
IPDI			2.207	
<u>Burning Rates, in./sec</u>				
500 psia	0.642	0.661	0.682	0.754
1000 psia	0.848	0.886	0.912	1.013
n	0.40	0.41	0.42	0.42

a - PCDE Lot 6+8 and BDNPA/F passed through molecular sieves. Equivalents ratio, PCDE/HT/IPDI = 40/60/95.

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EFFECT OF 0.5μ AP ON THE BURNING RATES OF PCDE-BDHPA/F PROPELLANTS WITH VARIOUS TYPES OF ALUMINUM (U)



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Figure 18

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TABLE 30

COMPOSITION AND PROPERTIES OF PCDE-BDNPA/F PROPELLANTS CURED WITH IPDI (U)

<u>Components</u>	<u>PCDE No.</u>			
	<u>224</u>	<u>225</u>	<u>226</u>	<u>227</u>
AP, 180 μ , RRD, coated			21.0	
AP, 6 μ			30.0	
Al, H-60			22.0	
Neozone D			0.10	
FeAA			0.05	
HAA			0.05	
BDNPA/F ^a			12.596	
PCDE ^{a,b}	12.376	12.232	12.092	11.956
HT ^b	0.332	0.328	0.324	0.320
IPDI ^b	1.497	1.644	1.787	1.928
<u>Safety Properties^c</u>				
Impact, cm/2kg	15.1	18.2	14.4	13.2
Friction, g/3000 rpm	600	630	485	190

a - Passed through molecular sieves

b - Equivalents ratio, PCDE/HT/IPDI = 50/50/90, 50/50/100, 50/50/110, and 50/50/120 for Batches 224 through 227, respectively.

c - Uncured

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propellants was made without antioxidant and these cured well. The data (Table 31) indicate that the correct NCO to OH ratio is 1:1, but that the crosslinker content is low. In order to increase the moduli of the IPDI-cured propellants, a third set was made with the PCDE to HT ratio fixed at 40 to 60 (Table 32). The results indicate that while the properties are good, some improvement might be made by lowering the HT content slightly. The third set did not show the maximum modulus at 100 equivalents percent NCO.

4. Effect of Stabilizers (U)

(U) A study of stabilizers for PCDE-BDNPA/F propellants was made at the 100-g batch size. Sulphur, dinitrodiphenylamine (DNDPA), di-t-butylresorcinol (DBR), Santicizer-8, and combinations of these were tested. The compositions and properties of the propellants are indicated in Table 33. Aging of these was in sealed metal containers at 150°F. All samples containing DBR had lower than normal moduli because DBR interfered with cure.

(U) The weight loss data indicate that except for DBR, which was worse, a combination of the other stabilizers are little different than the control with no added stabilizer. Because the propellant was viscous, it was decided to use the liquid Santicizer-8. Further studies will be made later to determine which stabilizer is best.

5. Use of Uncoated AP and DEA (U)

(U) It was noted earlier in the program that good mechanical properties could be achieved with uncoated AP if DEA was used as a bonding agent. Propellants were made to test this effect as there would be an advantage to using uncoated AP. The propellant compositions and properties are tabulated in Table 34. The experiment worked well. DEA not only gave good properties with uncoated AP, but also allowed a decrease in the total crosslinker content. In view of these results, the use of coated AP was discontinued.

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TABLE 31

COMPOSITION AND PROPERTIES OF PCDE-BDNPA/F PROPELLANTS CURED WITH IPDI (1)

<u>Components</u>	<u>PCDE No.</u>		
	<u>230</u>	<u>231</u>	<u>232</u>
AP, 180 μ , RRD, coated		21.0	
AP, 6 μ		30.0	
Al, H-60		22.0	
FeAA		0.05	
HAA		0.05	
BDNPA/F ^a		12.643	
PCDE ^{a,b}	12.349	12.278	12.270
HT ^b	0.331	0.329	0.327
IPDI ^b	1.576	1.650	1.722
<u>Safety Properties^c</u>			
Impact, cm/2kg	8.5	9.5	9.2
Friction, g/3000 rpm	440	280	375
Onset/Ignition Temp., °F	339/640	334/641	334/646
<u>Mechanical Properties at 77°F</u>			
σ_m , psi	57	68	42
ϵ_m , %	42	39	48
E_o , psi	184	232	114
<u>Swelling Ratio in Acetone</u>	4.15	4.15	4.58

a - Passed through molecular sieves

b - Equivalents ratio, PCDE/HT/IPDI = 50/50/95, 50/50/100, and 50/50/105, respectively, for PCDE 230 through 232.

c - Cured

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TABLE 32

COMPOSITION AND PROPERTIES OF PCDE-BDNPA/F PROPELLANTS CURED WITH IPDI (U)

<u>Components</u>	<u>PCDE No.</u>		
	<u>233</u>	<u>234</u>	<u>235</u>
AP, 180 μ , RRD, coated		21.0	
AP, 6 μ		30.0	
Al, H-60		22.0	
FeAA		0.05	
HAA		0.05	
BDNPA/F ^a		12.643	
PCDE ^{a,b}	11.883	11.800	11.719
HT ^b	0.478	0.475	0.471
IPDI ^b	1.836	1.982	2.067
<u>Safety Properties</u>			
Impact, cm/2kg	11.0	9.4	6.8
Friction, g/3000 rpm	360	380	385
Onset/Ignition Temp., °F	320/639	320/638	320/638
<u>Mechanical Properties at 77°F</u>			
σ_m , psi	116	107	103
ϵ_m , %	25	27	29
E_m , psi	567	505	458
E_o , psi			
<u>Swelling Ratio in Acetone</u>	3.31	3.28	3.35

a - Passed through molecular sieves

b - Equivalents ratio, PCDE/HT/IPDI = 40/60/95, 40/60/100, and 40/60/105, respectively, for PCDE 233, 234, and 235.

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TABLE 33

COMPOSITION AND PROPERTIES OF PCDE-BDNPA/F PROPELLANTS CONTAINING VARIOUS STABILIZERS (U)

Components ^a	PCDE No.									
	256	257	258	259	264	265	266	267		
AP, 6μ				30.0						
AP, iCON, RED, Coated				21.0						
Al, H-60				22.0						
DNDA	-0-	-0-	0.20	0.20	-0-	-0-	-0-	-0-		
DER	-0-	-0-	-0-	-0-	0.20	-0-	0.10	0.10		
S-8	-0-	-0-	-0-	-0-	-0-	0.20	0.10	-0-		
S	-0-	0.10	-0-	0.10	-0-	-0-	-0-	0.10		
FeAA				0.05						
HAA				0.05						
BDNPA/F	12.5	12.408	12.308	12.208	12.308	12.308	12.308	12.308		
PCDE				12.155						
HI				0.432						
IPDI				1.804						
Safety Properties										
Impact, cm/2 kg	8.5/-	8.5/-	9.3/-	8.6/-	11.7/7-7	16.9/8.5	15.9/7.5	8.6/7.5		
(Uncured/cured)										
Friction, g/3000 rpm	370/-	242/-	230/-	260/-	165/418	220/392	165/ 58	185/263		
(Uncured/cured)										
Onset/Ignition Temp., °F ^b	314/642	309/639	299/634	320/640	301/637	309/635	269/633	288/639		
Mechanical Properties at 77°F										
σ _m , psi	117	117	109	112	49	108	64	75		
ε _m , %	30	27	30	27	35	32	39	38		
E, psi	507	516	458	505	186	421	207	249		
Swelling Ratio in Acetone										
Initial	3.63	3.68	3.65	3.57	3.65	3.76	3.87	3.96		
Aged ^c , 2 weeks, 150°F	3.49	3.58	3.48	3.25	-	3.48	-	-		
Height Loss, %, Aged 2 wks, 150°F	0.095	0.095	0.102	0.093	0.148	0.099	0.126	0.114		

^aPCDE and BDNPA/F passed through molecular sieves. Equivalents ratio, PCDE/HT/IPDI = 43/57/95 for all batches.

^bFor uncured specimens

^cSealed in friction-top can.

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TABLE 34

COMPOSITION AND PROPERTIES OF PCDE-BDNPA/F PROPELLANTS CONTAINING DEA AND UNCOATED AP (U)

<u>Components^a</u>	<u>PCDE No.</u>			
	<u>268</u>	<u>269</u>	<u>270</u>	<u>271</u>
AP, 6 μ		30.0		
AP, 180 μ , RRD		21.0		
Al, H-60		22.0		
FeAA		0.05		
HAA		0.05		
BDNPA/F		12.778		
PCDE	12.518	12.504	12.654	12.646
HT	0.275	0.183	0.170	0.113
DEA	-0-	0.106	0.066	0.131
TDI	1.330	1.329	1.233	1.232
<u>Safety Properties</u>				
Impact, cm/2kg (uncured/cured)	-/8.2	-	-/7.0	9.2/-
Friction, g/3000 rpm (uncured/cured)	-/300	-	-/342	115/-
Onset/Ignition Temp., °F ^b	307/652	-	322/632	323/638
<u>Mechanical Properties at 77°F</u>				
σ_m , psi	152	Did	111	Did
ϵ_m , %	26	Not	32	Not
E_o , psi	744	Cure	437	Cure
<u>Swelling Ratio in Acetone</u>	3.50	-	3.92	-

a - PCDE and BDNPA/F passed through molecular sieves. Equivalents ratio, PCDE/HT/DEA/TDI = 55/45/0/112, 55/30/15/112, 60/30/10/112, and 60/20/20/112 for PCDE 268 through 271, respectively

b - Cured specimens for PCDE 268 and 270; uncured specimen for PCDE 271.

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6. PCDE to BDNPA/F Ratio (U)

(U) The addition of BDNPA/F to PCDE lowers the performance of propellants containing the latter. For this reason, it is advantageous to increase the PCDE at the expense of the plasticizer. Such a course is of only limited utility, however, because it will tend to make processing more difficult. As no determination had been made of the effect of increased PCDE on processing, a series of propellants was made at PCDE to BDNPA/F ratios in the range of 1.0 to 1.31. The compositions of these propellants are given in Table 35. The processing of propellants made at ratios of 1.0 and 1.1 was very similar, but that of those made at ratios of 1.2 and 1.31 was noticeably more difficult. Subsequent preparation of PCDE-BDNPA/F propellants was made at a PCDE to BDNPA/F ratio of 1.1.

7. PCDE Lot 6+8 (U)

a. Introduction (U)

(U) Heretofore, all PCDE work was done with Shell Lot 44. Because this lot of material was used up, a new lot of PCDE was investigated. The lot was created by combining Hercules Lots 6 and 8. Pertinent data concerning these lots and their mixture are given in Section V.B.

b. NCO Requirement (U)

(U) A series of propellants was formulated to determine the optimum NCO to OH ratio for curing Lot 6+8 with IPDI. The NCO to OH ratio was varied from 0.80 to 1.30 in equal increments of 0.1. In this series, PCDE Nos. 281 through 286, propellants with NCO to OH ratios above 1.10 did not cure. Another overlapping series, PCDE Nos. 291 through 294, was made at NCO to OH ratios between 0.60 and 0.90. Propellants with NCO to OH ratio less than 0.80 did not cure. The second series which contained MDX-65 in place of H-60 aluminum cured better. The compositions and properties of the two series are presented in Table 36.

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TABLE 35

COMPOSITION AND PROPERTIES OF PROPELLANTS WITH VARIOUS PCDE TO BDNPA/F RATIOS (U)

<u>Component</u> ^a	<u>PCDE No.</u>			
	<u>277</u>	<u>278</u>	<u>279</u>	<u>280</u>
AP, 6μ		30.0		
AP, 180μ, RRD		21.0		
Al, H-60		22.0		
FeAA		0.05		
HAA		0.05		
BDNPA/F	12.712	12.075	11.499	10.926
PCDE	12.712	13.283	13.799	14.313
HT	0.170	0.178	0.185	0.192
DEA	0.067	0.070	0.072	0.075
TDI	1.238	1.294	1.344	1.394
<u>Safety Properties</u> ^b				
Impact, cm/2kg	8.2	6.8	8.7	8.0
Friction, g/3000 rpm	750	530	900	880
Onset/Ignition Temp., °F	305/621	270/639	289/400	275/403
<u>Mechanical Properties at 77°F</u>				
σ_m , psi	142	145	122	160
ϵ_m , %	24	26	21	28
E_o , psi	698	690	640	698
<u>Swelling Ratio in Acetone</u>	3.25	3.29	3.44	3.42
<u>Castability</u> ^c	Fair	Fair	Poor	Poor

a - PCDE and BDNPA/F passed through molecular sieves. Equivalents ratio, PCDE/HT/DEA/TDI = 60/30/10/112.

b - For uncured samples of PCDE 280 values are 13.2, 240, 301/640, respectively.

c - Subjective visual evaluation.

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TABLE 36

COMPOSITION AND PROPERTIES OF PCDE-BDPA/F PROPELLANTS WITH H-60 OR MDX-65 ALUMINUM AND AT VARIOUS NCO TO OH RATIOS (U)

Components ^a	PCDE No.									
	281	282	283	284	285	286	291	292	293	294
AP, 5u			30.0					30.0		
AP, 180u, RRD			21.0					21.0		
Al			22.0					22.0		
FeAA			0.05					0.05		
HAA			0.05					0.05		
BDPA/F										
PCDE										
HT										
IPDI										
NCO/OH										
Safety Properties ^b										
Impact, cm/2kg										
Friction, g/3000 rpm										
Onset/Ignition Temp., °F										
Mechanical Properties at 77°F ^c										
σ _m , psi										
c _m , %										
E _c , psi										
Swelling Ratio in Acetone										
a	PCDE and BDPA/F passed through molecular sieve. PCDE Nos. 281-286 contain H-60 aluminum and 291-294 contain MDX-65 aluminum.									
b	In all cases PCDE to HT ratio maintained at 40 to 60.									
c	Cured specimens									
	Minibone specimens									

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(U) A plot of the propellant moduli vs the NCO to OH ratio for PCDE Nos. 281 through 286 (Figure 19) indicates the optimum ratio to be 0.95. The results of mechanical property testing of PCDE Nos. 291 through 294 are consistent with this ratio: Because the equivalent weight assumed for the formulation studies was 1511, the actual equivalent weight of the prepolymer (calculated from the optimum NCO/OH ratio) is 1586, which corresponds to a "true" optimum ratio of 1:1. For a molecular weight of 2821 the functionality of the material would be 1.78, if the true equivalent weight is 1586. This value is consistent with the results of crosslinker requirement studies.

c. Crosslinker Requirements (U)

(1) With HT (U)

(U) With the NCO to OH ratio held at 0.85, a series of propellants (PCDE Nos. 287 through 290) was made with the PCDE to HT equivalents ratio being varied from 35/65 to 50/50. The compositions and properties of these are presented in Table 37. The results indicate that 60 to 65 eq% HT will be required to cure propellants with Lot 6+8. This large crosslinker requirement indicates poor functionality for the PCDE.

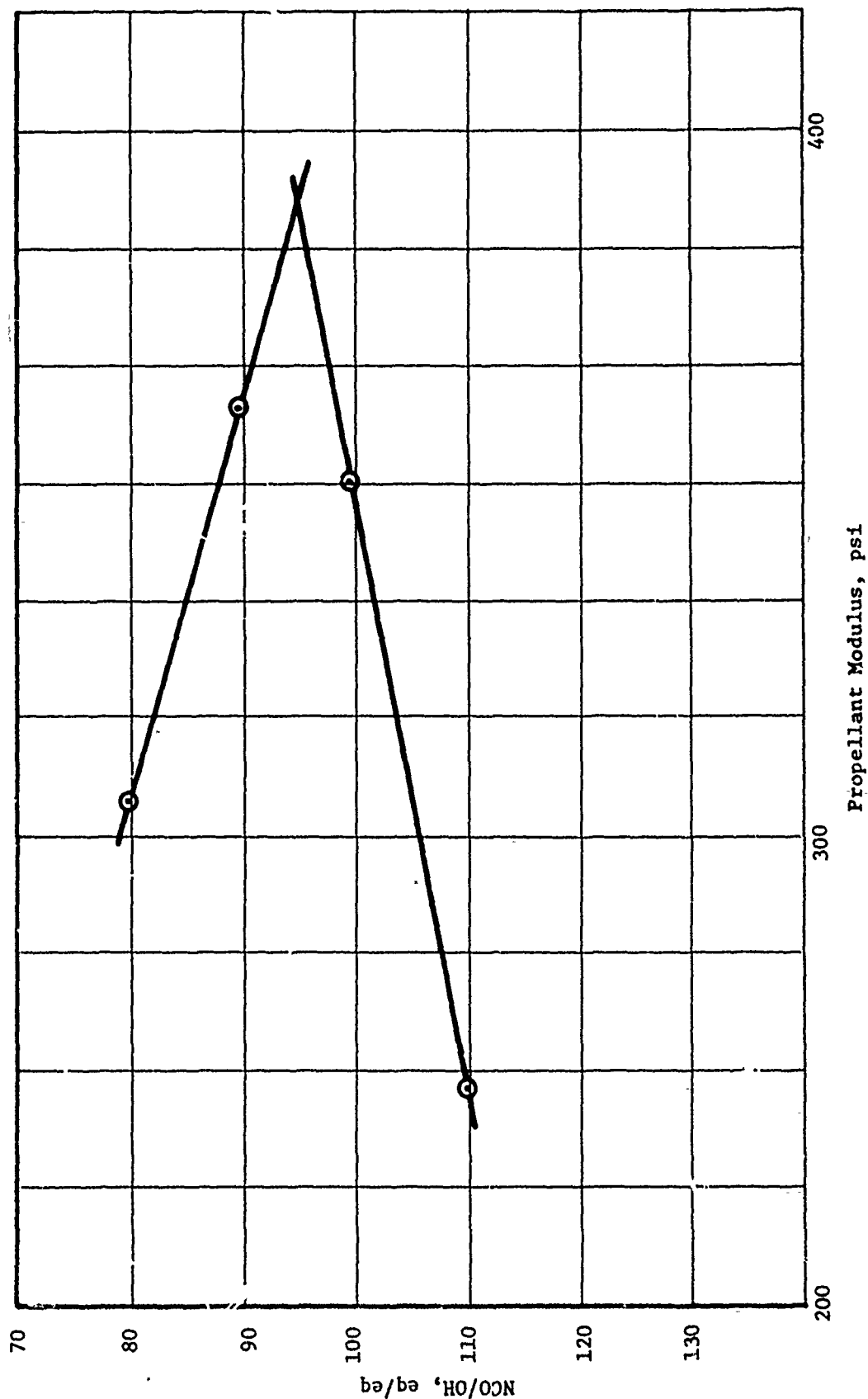
(2) Effect of Aluminum Type (U)

(U) It was observed that those propellants which were formulated with MDX-65 cured to higher moduli than those which contained H-60. This can readily be seen from the data in Table 36; compare PCDE 281 with 293 and PCDE 282 with 294.

It was postulated that the difference might be due to some effect of the metal on the isocyanate. To test this postulate, MDX-65 and H-60 were stirred overnight with IPDI at ambient temperature. After filtration and drying, the aluminums were used in propellant along with untreated ones. The composition of the propellants and their mechanical properties are shown in Table 38.

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VARIATION OF PROPELLANT MODULUS WITH NCO TO OH RATIO FOR IPDI-CURED PCDE-BDNPA/F PROPELLANT (PCDE LOT 6+8)



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Figure 19

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TABLE 37

COMPOSITION AND PROPERTIES OF PCDE-BDNPA/F PROPELLANTS WITH VARYING CROSSLINKER CONTENT (U)

<u>Component^a</u>	<u>PCDE No.</u>			
	<u>287</u>	<u>288</u>	<u>289</u>	<u>290</u>
AP, 0.5 μ		5.0		
AP, 6 μ		25.0		
AP, 180 μ , RRD		21.0		
AL, H-60		22.0		
FeAA		0.05		
HAA		0.05		
BDNPA/F	11.406	11.585	11.728	11.844
PCDE	12.546	12.743	12.900	13.029
HT	0.689	0.565	0.466	0.385
IPDI	2.258	2.007	1.806	1.642
PCDE/HT	35/65	40/60	45/55	50/50
<u>Safety Properties^b</u>				
Impact, cm/2kg	6.4	8.7	-	-
Friction, g/3000 rpm	750	460	-	-
Onset/Ignition Temp., °F	305/630	317/632	-	-
<u>Mechanical Properties at 77°F^c</u>				
σ_m , psi	91	85	DID	DID
ϵ , %	21	31	NOT	NOT
E_o , psi	519	346	CURE	CURE

a PCDE and BDNPA/F passed through molecular sieves. NCO to OH ratio maintained at 85/100 for all batches.

b Cured specimens

c Minibone specimens

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TABLE 38

COMPOSITION AND PROPERTIES OF PCDE-BDNPA/F PROPELLANTS CONTAINING TREATED AND UNTREATED ALUMINUM (U)

Components ^a	PCDE No.			
	314	315	316	317
AP, 0.5μ		5.0		
AP, 6.0μ		27.5		
AP, 180μ, RRD		18.5		
Al	22.0 ^b	22.0 ^c	22.0 ^d	22.0 ^e
S-8		0.20		
FeAA		0.05		
HAA		0.05		
BDNPA/F		11.549		
PCDE		12.704		
HT		0.459		
IPDI		1.988		
<u>Mechanical Properties at 77°F</u>				
σ_m , psi	140	51	121	65
ϵ_m , %	22	33	18	38
E_m , psi	735	226	732	263
<u>Swelling Ratio in Acetone</u>	3.62	4.09	3.57	3.86

a - Lot 6+8 PCDE and BDNPA/F passed through molecular sieves. Equivalents ratio, PCDE/HT/IPDI = 45/55/95.

b - MDX-65, untreated

c - H-60, untreated

d - MDX-65, treated (see text)

e - H-60, treated (see text)

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(U) It is apparent that the propellants containing MDX-65, treated or untreated, are better cured than those containing H-60. The effect is, therefore, not the result of reactions involving aluminum and the isocyanate. Further experiments must be done to clarify the significance of the effect.

(3) With HT + DEA (U)

(U) A series of propellants was made in which the crosslinker HT was replaced by DEA. The composition and properties of the propellants are given in Table 39. The properties of the propellants were generally good indicating that DEA should be used. PCDE Nos. 303, 304 and 305 have elongations adequate for the goals of this program. It should be noted that MDX-65 was used in these batches. Experience indicates that propellants using H-60 will require more crosslinker.

8. Processing (U)

(U) Processing studies of PCDE-BDNPA/F propellants have begun. The effect of processing aids and of HAA content on the viscosity of the propellant has been investigated. The composition and viscosity of the propellants are recorded in Tables 40 and 41. Graphic representations are shown in Figures 20 through 27. The data presented are tentative in nature because the viscosity measurements were made in small tubes containing about 50-g of propellant. This procedure is, however, adequate as a screening method. Techniques or formulations that are promising will be investigated further by more reliable measurements.

(U) The control propellant has a potlife in excess of four hours, the limit of the measurement. FC-189 decreases the potlife. On the other hand, P711, which is also an antioxidant, decreases propellant viscosity both at 5000 dynes/cm² and at infinite shear stress. The effect of DC-200 is difficult to assess. The propellant containing it has a lower viscosity at 5000 dynes/cm² at four hours than at two, because of the change of the viscosity-shear stress curve with time. At infinite shear stress, the viscosity of the DC-200 propellant is the same as that of the control.

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TABLE 39

COMPOSITION AND PROPERTIES OF PCDE-BDNPA/F PROPELLANTS WITH VARYING AMOUNTS OF DEA (U)

Components ^a	PCDE No.						
	299	300	301	302	303	304	305
AP, 6μ				30.0			
AP, 180μ, RRD				21.0			
Al, MDX-65				22.0			
FeAA				0.05			
HAA				0.05			
BDNPA/F	11.636	11.634	11.632	11.631	11.630	11.628	11.627
PCDE	12.799	12.798	12.796	12.794	12.792	12.791	12.789
HT	0.463	0.442	0.421	0.399	0.378	0.357	0.336
DEA	-0-	0.024	0.049	0.073	0.098	0.122	0.147
IPDI	2.003	2.002	2.002	2.002	2.002	2.001	2.001
<u>Safety Properties ^b</u>							
Impact, cm/2kg	10.7	-	-	-	-	-	12.5
Friction, g/3000 rpm	385	-	-	-	-	-	350
Onset/Ignition Temp., °F	317/632	-	-	-	-	-	297/631
<u>Mechanical Properties at 77°F ^c</u>							
σ _m , psi	138	136	118	126	118	124	106
ε _m , %	26	26	27	26	29	28	30
E _m , psi	674	667	533	613	502	556	446
Swelling Ratio in Acetone	4.02	3.77	3.79	3.80	3.84	3.83	3.88

a - PCDE Lot 6+8 and BDNPA/F passed through molecular sieves. Equivalents ratio, PCDE/HT/DEA/IPDI = 45/55/0/95, 45/52.5/2.5/95, 45/50/5/95, 45/47.5/7.5/95, 45/45/10/95, 45/42.5/12.5/95, and 45/40/15/95 for PCDE No. 299 to 305, respectively.

b - Cured specimen

c - Minibone specimen

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TABLE 40

COMPOSITION AND PROPERTIES OF PCDE-BDNPA/F PROPELLANTS CONTAINING PROCESSING AIDS (U)

Components ^a	PCDE No.			
	342	343	344	345
AP, 0.5μ		5.0		
AP, 6.0μ		27.5		
AP, 180μ, RRD		18.5		
Al, H-60		22.0		
S-8		0.20		
P711	-0-	0.10	-0-	-0-
FC-189	-0-	-0-	0.10	-0-
DC-200	-0-	-0-	-0-	0.01
FeAA		0.05		
HAA		0.05		
BDNPA/F	11.395	11.352	11.352	11.390
PCDE	12.534	12.487	12.487	12.530
HT	0.510	0.508	0.508	0.510
DEA	0.054	0.054	0.054	0.054
IPDI	2.206	2.198	2.198	2.205

Rotovisko Properties at 125°F

At 5000 dynes/cm ²				
kpoise at 2 hr	38	33	45	79
kpoise at 4 hr.	46	35	66	52
Estimated potlife, hr	>4	>4	2.6	-
At infinite shear stress				
kpoise at 2 hr	14	12.2	11.5	14
kpoise at 4 hr	20	15	25	20

a - PCDE Lot 6+8 and BDNPA/F passed through molecular sieves. Equivalents ratio, PCDE/HT/DEA/IPDI = 40/55/95 for all batches.

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TABLE 41

COMPOSITION AND PROPERTIES OF PCDE-BDNPA/F
PROPELLANTS CONTAINING VARYING AMOUNTS OF HAA (U)

Components ^a	PCDE No.			
	346	347	348	349
AP, 0.5μ		5.0		
AP, 6.0μ		27.5		
AP, 180μ, RRD		18.5		
Al, H-60		22.0		
S-8		0.20		
FeAA		0.05		
HAA	-0-	0.015	0.03	0.05
BDNPA/F	11.416	11.410	11.404	11.395
PCDE	12.558	12.551	12.543	12.534
HT	0.511	0.510	0.510	0.510
DEA	0.055	0.054	0.054	0.054
IPDI	2.210	2.209	2.208	2.206

Rotovisko Properties at 125°F

At 5000 dynes/cm ²				
kpoise at 2 hr	45	44	24	39
kpoise at 4 hr	59	56	38	45
Estimated potlife, hr	2.7	3.3	>4	>4
At infinite shear stress				
kpoise at 2 hr	16.5	17.6	12.4	12.9
kpoise at 4 hr	22.3	23.0	16.2	18.7

a - PCDE Lot 6+8 and BDNPA/F passed through molecular sieves. Equivalents ratio, PCDE/HT/DEA/IPDI = 40/55/5/95 for all batches

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PROPELLANT VISCOSITY AS A FUNCTION OF SHEAR STRESS AND TIME FROM CURING AGENT ADDITION

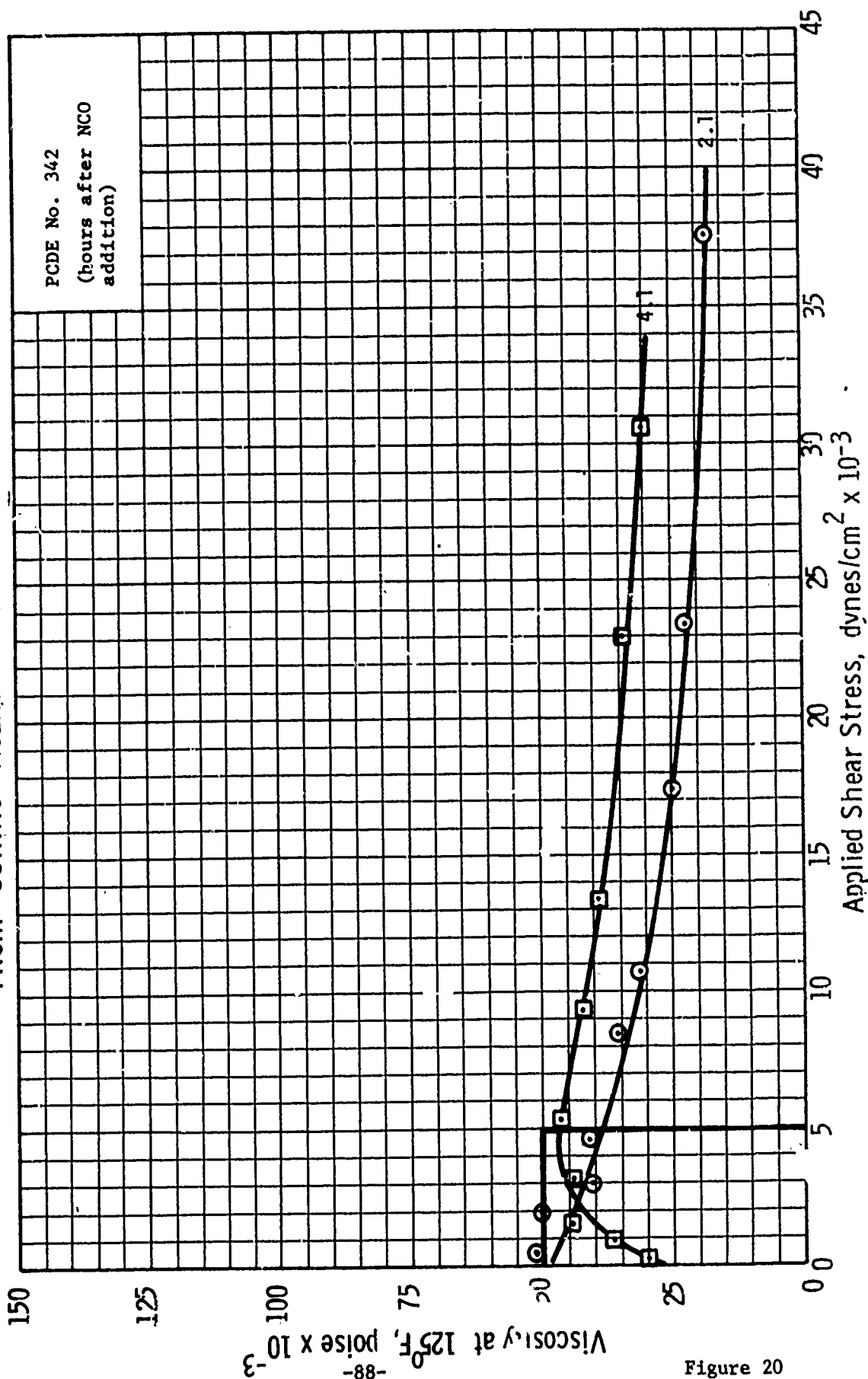


Figure 20

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PROPELLANT VISCOSITY AS A FUNCTION OF SHEAR STRESS AND TIME
FROM CURING AGENT ADDITION

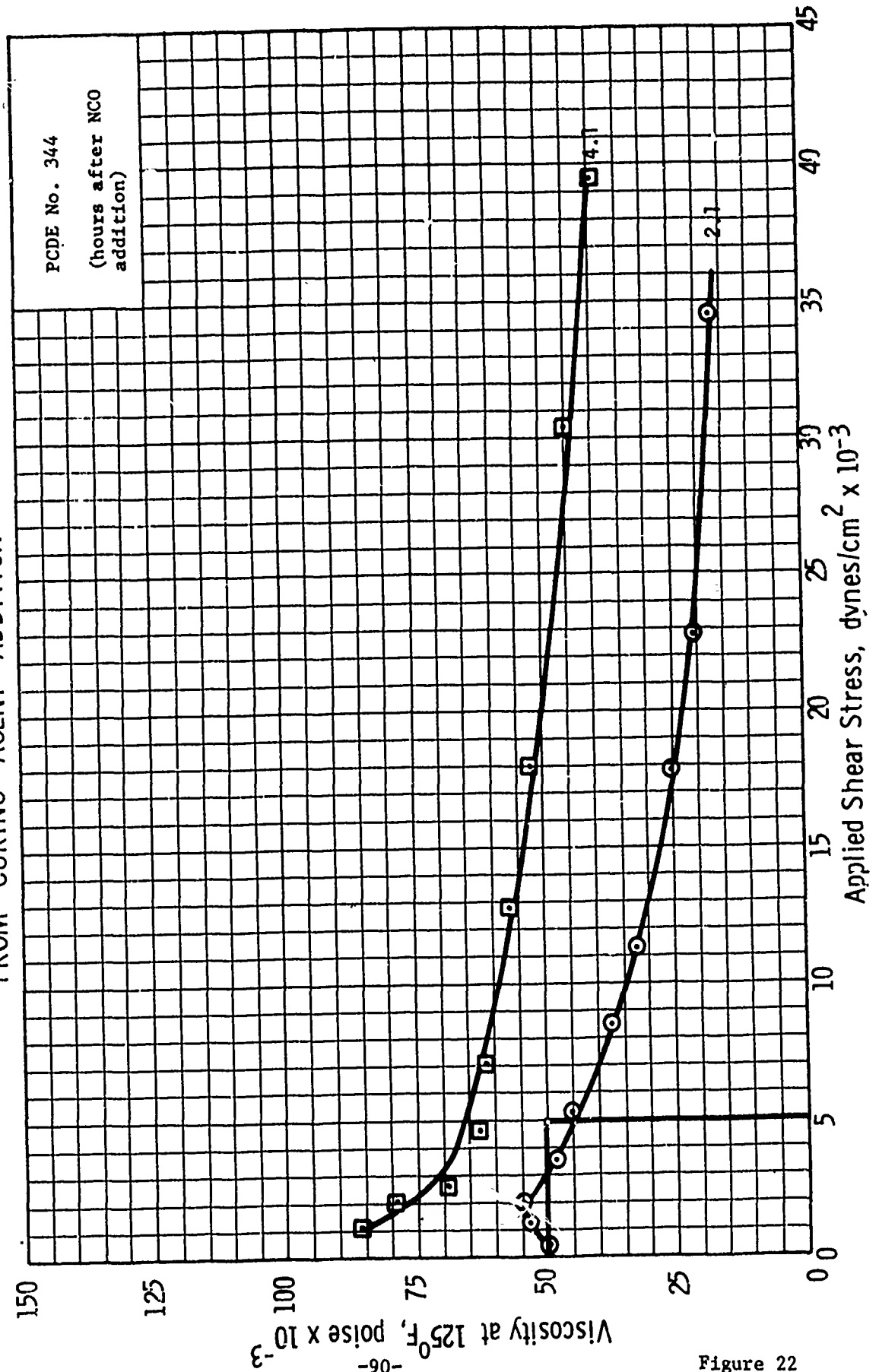


Figure 22

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PROPELLANT VISCOSITY AS A FUNCTION OF SHEAR STRESS AND TIME FROM CURING AGENT ADDITION

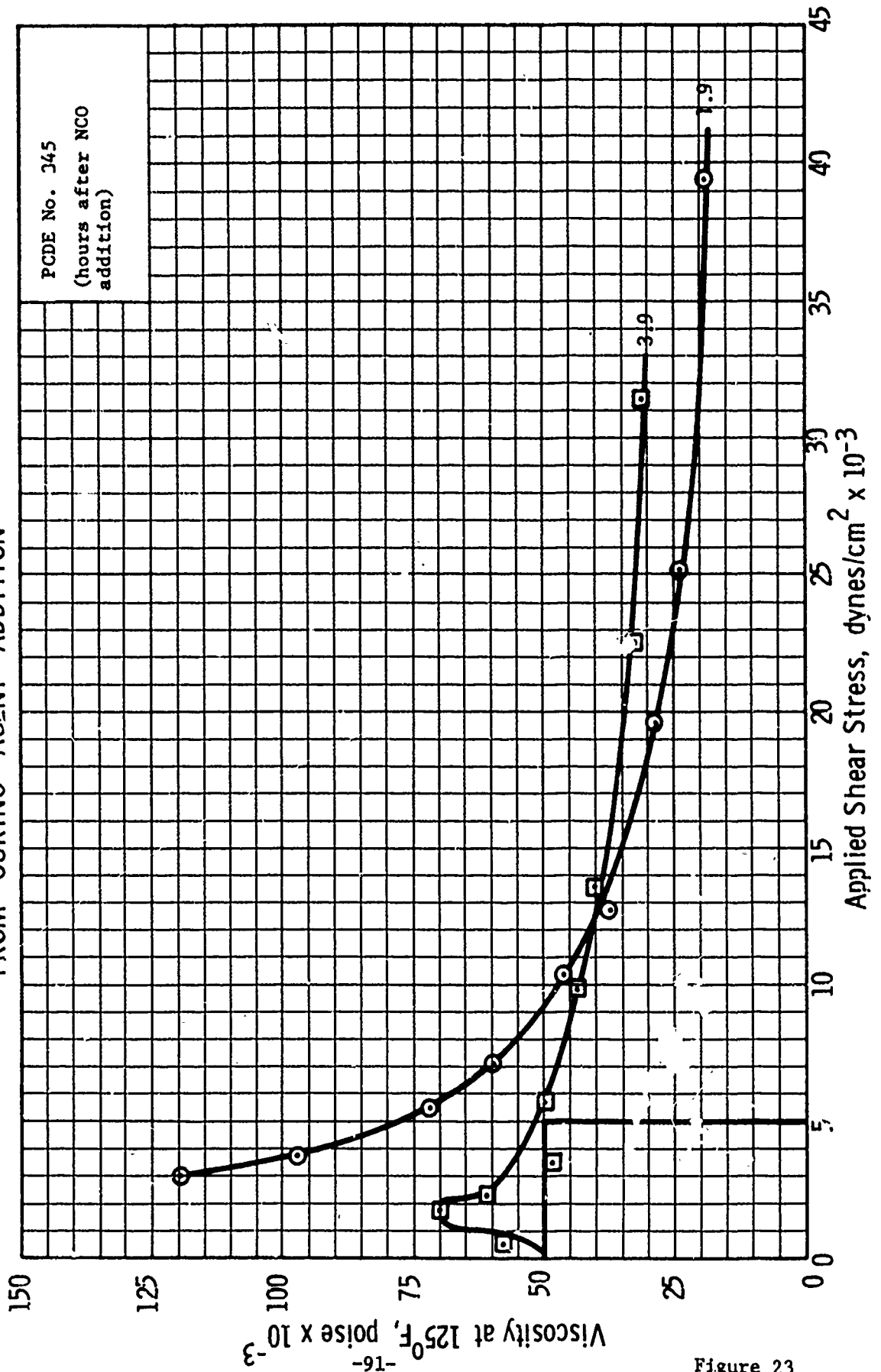


Figure 23

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PROPELLANT VISCOSITY AS A FUNCTION OF SHEAR STRESS AND TIME
FROM CURING AGENT ADDITION

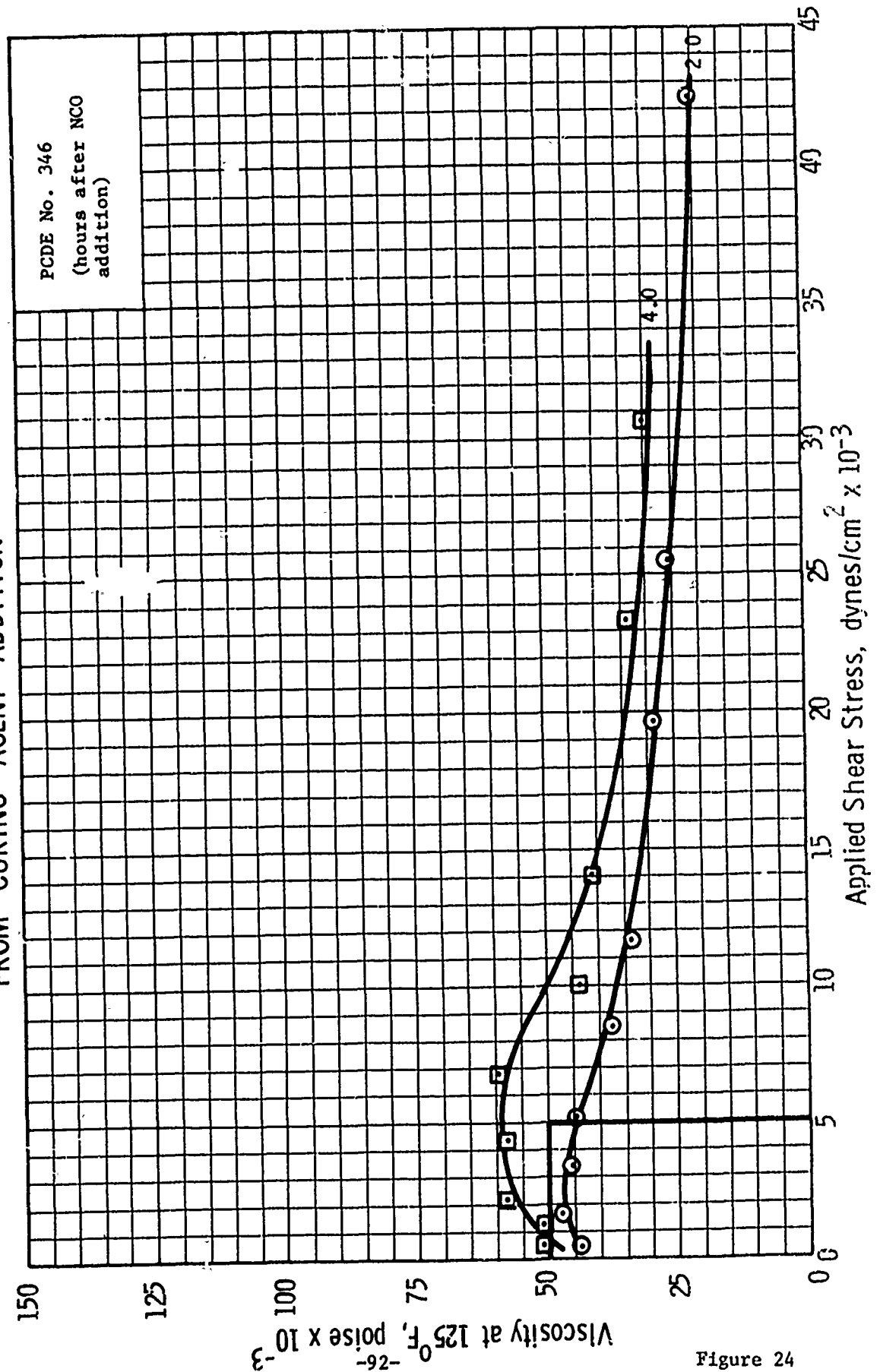


Figure 24

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PROPELLANT VISCOSITY AS A FUNCTION OF SHEAR STRESS AND TIME FROM CURING AGENT ADDITION

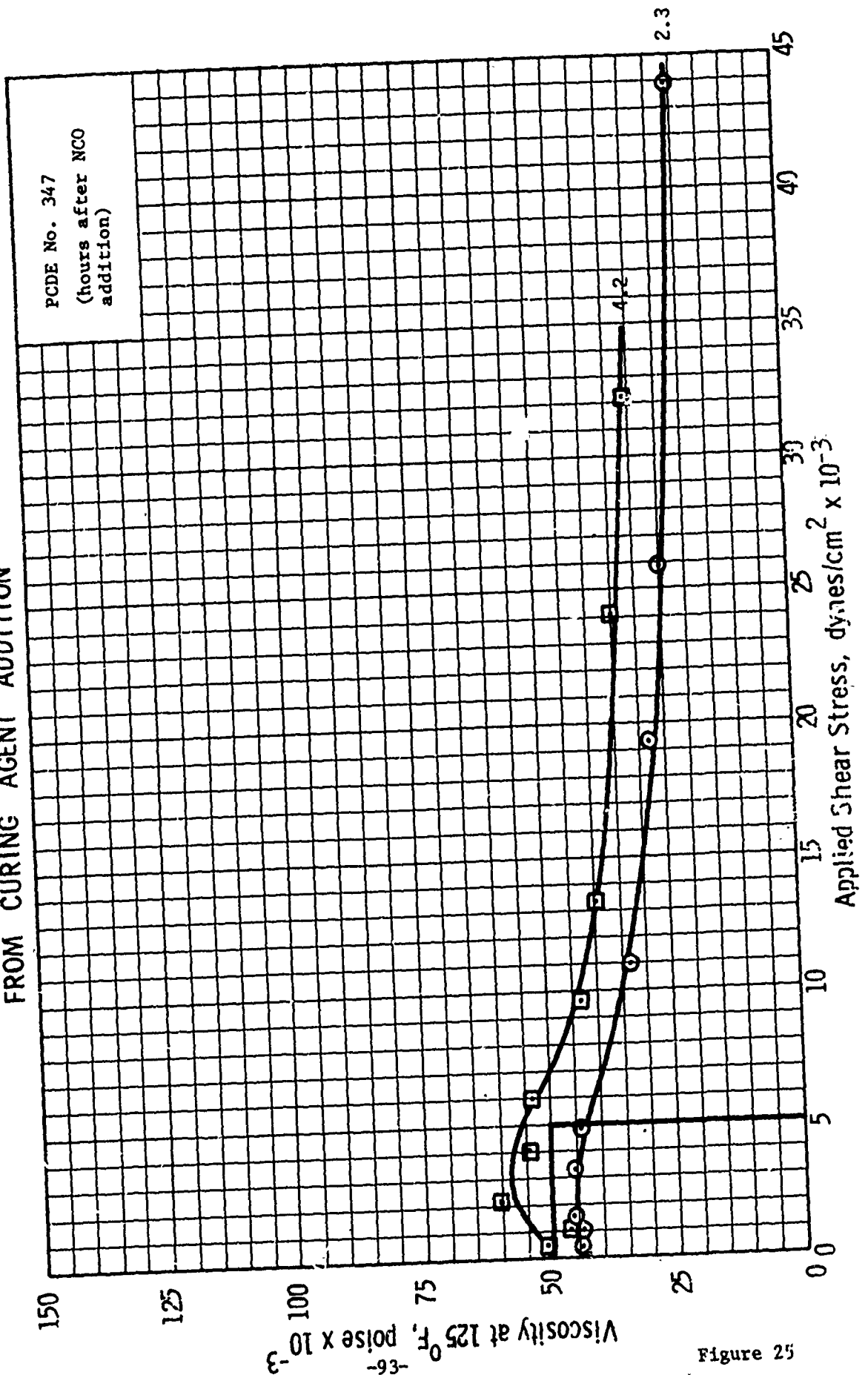


Figure 25

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PROPELLANT VISCOSITY AS A FUNCTION OF SHEAR STRESS AND TIME
FROM CURING AGENT ADDITION

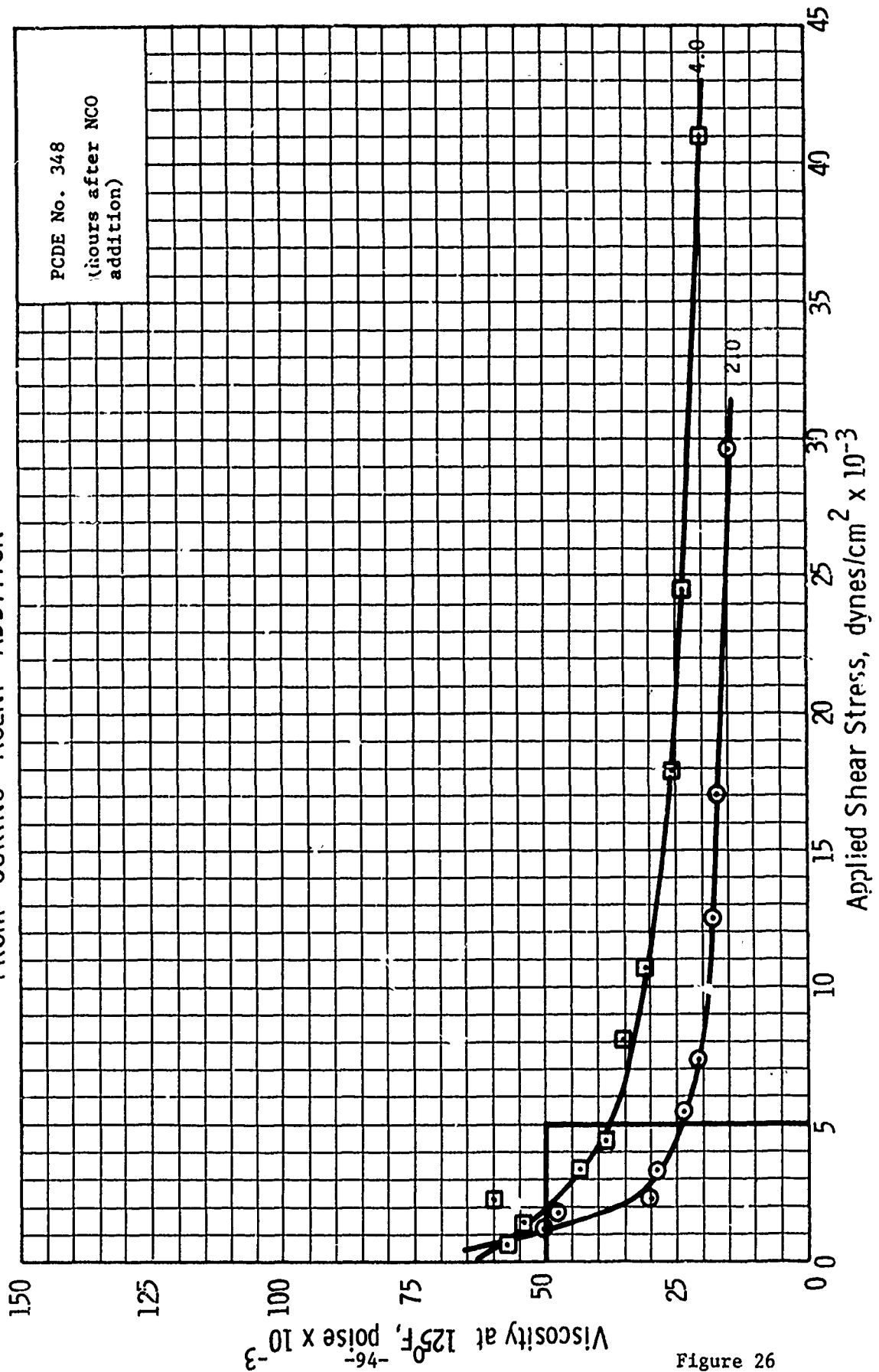


Figure 26

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PROPELLANT VISCOSITY AS A FUNCTION OF SHEAR STRESS AND TIME
FROM CURING AGENT ADDITION

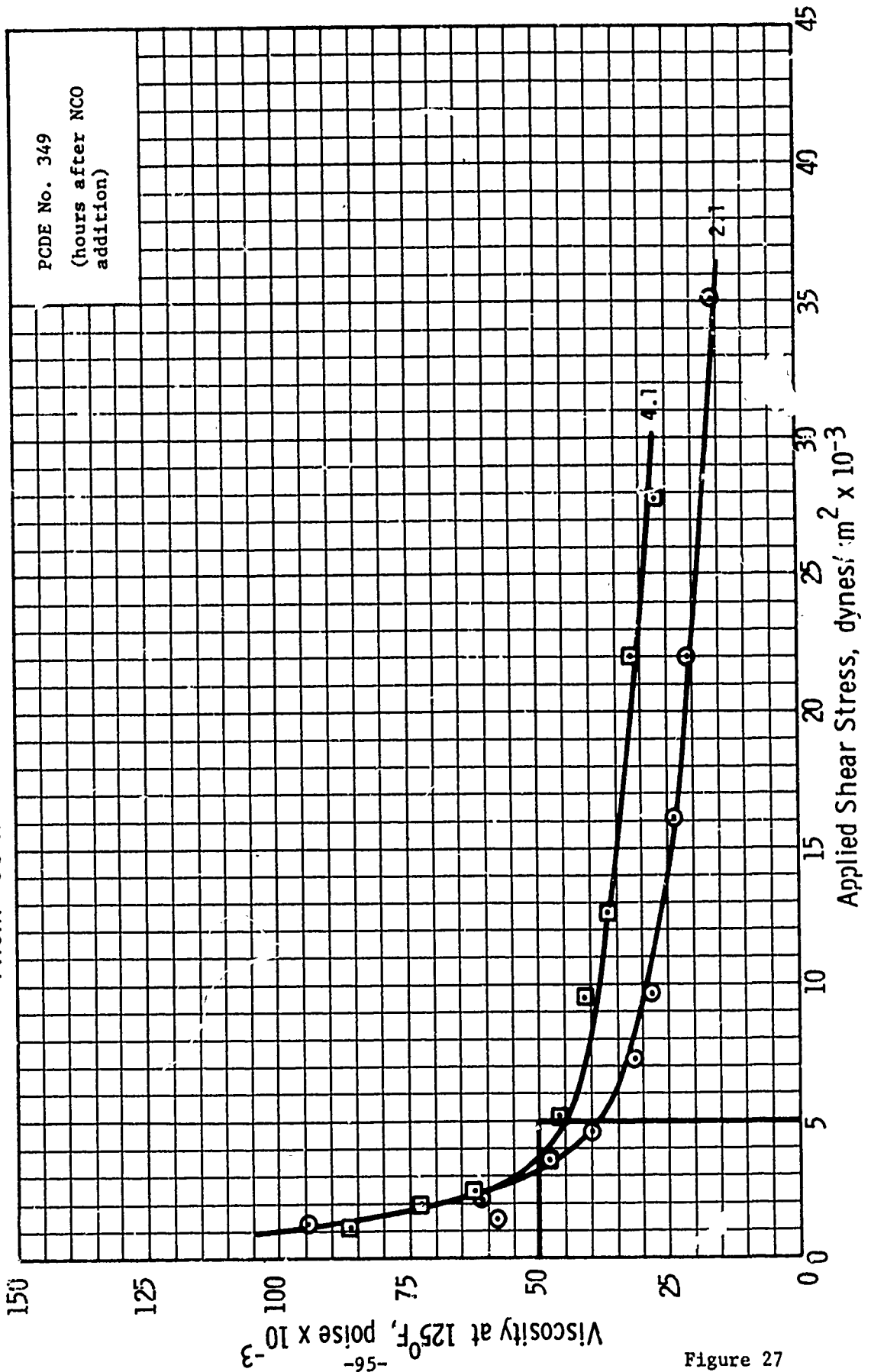


Figure 27

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(U) If the FeAA level is maintained at 0.05%, the best propellant viscosity is obtained with 0.03% HAA. With 0.05% HAA the viscosity is just a little higher. In both cases, the potlife exceeds four hours.

(U) The processing studies are continuing, and improvements are being incorporated in the propellant formulation.

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VI. REFERENCES (U)

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2. C. J. Rogers and P. L. Smith, FACTORS INFLUENCING SOLID PROPELLANT FLOW CHARACTERISTICS, Proceedings of the 26th JANNAF Solid Propulsion Meeting, July 14-16, 1970, p. 53.

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