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REDSTONE ARSENAL RESEARCH DIVISION

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Report No. S 20

STATUS REPORT ON NIKE ZEUS

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CONTRACT NO. DA 01-021-ORD5135

PROJECT NO. TB5-20-5

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ROHM & HAAS COMPANY

**REDSTONE ARSENAL RESEARCH DIVISION
HUNTSVILLE, ALABAMA**

REPORT NO. S-20

STATUS REPORT ON NIKE-ZEUS (U)

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July 13, 1959

**ARMY ORDNANCE CORPS
Project Number TU2-10
RESEARCH ON ROCKET PROPELLANTS AND ROCKET MOTORS
Contract No. DA 01-021-ORD5135**

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SUMMARY

Work on the Nike-Zeus system has been covered by three supplements to Contract DA-01-021 ORD-5135. The first, supplement No. 3, included the design, loading, and testing of one full-scale Zeus sustainer, using petrin acrylate propellants.

The second, supplement No. 6, was more extensive and included further development of the propellant to determine the most satisfactory formulation for use in the Zeus sustainer. It also included requirements for firing, storage, and physical properties data. In addition, loading and testing of four half-scale and two full-scale motors were required.

The third, supplement No. 16, provided for a continuation of No. 6. In addition the requirements included cycling studies and the loading and testing of four more half-scale motors and four more full-scale motors. The expiration date is January 31, 1960.

Both supplements No. 6 and 16 provided for testing propellant formulations in small scale motors in the 10 to 100 lb. class.

To date a major portion of the requirements have been completed. Several propellant formulations have been investigated to select one with the best characteristics. Composition QZ_{bn} has been selected as the one with the best combination of specific impulse, physical properties, and processing characteristics. This composition or a close approximation has been used in all of the large motor firings to date. Physical properties data have been obtained for several compositions, and an extensive amount of data are available for composition QZ_{bn}.

The advances made in petrin acrylate propellant manufacture last year have indicated the process to be satisfactory for making large motors. Also, further improvements in continuous casting have been uncovered, which will be incorporated in future production facilities. Because of the low initial viscosity of the mix, continuous feeding and mixing of the raw materials appears feasible in readily available equipment. Although reasonable care is required in the processing, loading, and preparation for firing, no seriously difficult

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or unusual problems have developed.

Storage data have been obtained for some compositions, but only recently have motors containing QZ_{bn} been placed in surveillance. One full-scale motor has been placed in ambient storage. After about one month in storage, no apparent change has taken place in the quality of the propellant.

Although limited cycling tests and firings of small motors at temperatures as low as -60°F have been conducted with petrin acrylate propellant, no valid data are available for QZ_{bn} under cycling conditions. Cycling data, using small test motors, will be obtained for QZ_{bn} .

A total of nine half-scale motors have been loaded. Seven have been fired, the last five successfully. Processing difficulties with two of the earlier motors required that the propellant be cut out.

Three full-scale motors have been loaded successfully. One motor which had a batch of propellant from which catalyst was inadvertently omitted has been put in surveillance. The other two motors have been successfully fired.

The demonstrated processibility for large motor manufacture and the superior ballistic properties make petrin acrylate a leading candidate in the solid propellant field.

INTRODUCTION

Approximately two years ago the Redstone Arsenal Research Division of Rohm & Haas Company was asked to determine the feasibility of using a petrin acrylate propellant in the Nike-Zeus sustainer. At that time petrin acrylate propellant was becoming recognized as a potentially useful high energy propellant. In addition to a higher specific impulse, petrin acrylate has certain other advantages. Its burning rate can be varied easily over a considerable range; it is readily castable; it has adequate physical properties.

During the past two years the development program for petrin acrylate propellant has been successful in utilizing these original advantages, improving on them, and demonstrating the propellant in full-scale Nike-Zeus sustainer test hardware. In addition, considerable data were obtained with smaller systems ranging from 10 to 1000 lb.

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Two compositions, QC and QZ, have received most of the attention for use in large motors. (See Table I).

Table I
Composition of QC and QZ Propellant

	<u>QC</u>	<u>QZ</u>
Petrin acrylate	14.60	14.90
Triethylene glycol dinitrate	17.50	17.00
Ammonium perchlorate	57.20	47.85
2-Ethyl hexyl acrylate	1.60	1.60
Polyester-920	0.75	0.45
Aluminum	8.15	18.00
Ethyl centralite	0.20	0.20

The switch to QZ for all later firings was made because of its higher density. Specific impulse was virtually unchanged.

Since indications of good ballistic properties were obtained, it remained for processing techniques to be worked out to provide a simple reliable processing method.

PROCESSING OF PROPELLANT FOR THE NIKE-ZEUS

Processing

Petrin acrylate propellants are relatively easy to load into large motors because of the low concentration of solids (ammonium perchlorate, aluminum) and the low viscosity of the high energy binder system (monomer, plasticizer, etc.). For this reason the propellant can be mixed in a kettle with a low speed agitator at atmospheric pressure. The energy input into the propellant due to mixing is negligibly small. Simple equipment for pumping and deaeration and simple bayonet casting can be used. Petrin acrylate propellants can be produced by a continuous process which has been demonstrated in the loading of half-scale and full-scale Zeus sustainer motors.

The initial viscosity of the propellant can be controlled by varying the type of aluminum powder and the polymerization rate can be controlled by the concentration of inhibitor. These changes have no adverse effects on the ballistic or physical properties of the propellant.

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The polymerization reaction is initiated by the addition of a catalyst (t-butyl perbenzoate) and delayed long enough to permit pumping into the motor by an inhibitor (n-nitroso diphenyl amine). Neither of these materials change the ballistic or physical properties of the propellant.

Experiments have shown that variations of about 5% of each ingredient separately will not have any significant effect on the ballistic or physical properties, simplifying processing control.

The physical properties of the cured propellant have been demonstrated to be satisfactory for use in the Zeus system.

Two problem areas in processing petrin acrylate propellants are an exothermic polymerization reaction during curing, which may require internal cooling of the mandrel for some large motors, and a high freezing point (125°F) which requires jacketing of all process equipment and casting lines. If excessive temperatures (around 210°F) are present for extended periods, degradation and fissuring of the propellant may occur.

This Division has recently designed and put into operation a propellant mixing and casting facility suitable for filling large motors with petrin acrylate propellant. Included are facilities for mixing 1200-lb. batches, continuous inline mixing for incorporation of polymerization catalyst, continuous deaeration, and continuous casting of motors containing up to 10,000 pounds of propellant. The size limitation on the motor is imposed by the quantity-distance restrictions of the existing facilities. Safety features include remote operation when any item of processing equipment is in operation, complete elimination of bearings and stuffing boxes exposed to propellant or inflammable ingredients, and continuous removal of dusts.

The operations included in this facility are outlined below; all of the operations are performed remotely:

1. Weighing of liquid ingredients
2. Charging of both solid and liquid ingredients to the mixer
3. Propellant mixing
4. Metering of propellant and polymerization catalyst in the specified ratio

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5. Mixing of propellant and polymerization catalyst in an inline mixer during casting.
6. Deaeration of catalyzed propellant
7. Bayonet casting at a controlled rate
8. Curing
9. Extraction of mandrel from cured motor

Fig. 1 and 2 show most of the important features of the process. Details of development of this process are given in Appendix A.

Minor ingredients are dissolved in the plasticizer, triethylene glycol dinitrate, and mixed by recirculation through a Vanton pump. The correct weight of liquids is pumped to a tank suspended from a load cell above the mixer and then drained into the mixer. Aluminum and petrin acrylate monomer are weighed into a single cart and charged remotely to a hopper by means of a Cesco skip hoist. Ammonium perchlorate is weighed into another cart and then charged to a second hopper, which is heated to prevent caking. After the liquids have been drained to the mixer, the aluminum and petrin acrylate mixture is charged to the mixer by a pneumatic vibratory feeder. In the mixer the monomer is melted and, with the aluminum, is incorporated into the mix. Ammonium perchlorate is charged by a second vibratory feeder and incorporated into the batch. The mixer is a 100-gallon, jacketed Pfaudler kettle equipped with a flush bottom valve and anchor agitator. A dual vent system, one vent for oxidizer dust and the other for fuel dust mounted over the hoppers and mixer, conveys the dust to a scrubber. The water from the scrubber is filtered and discarded. After mixing for about thirty minutes at 150 to 160°F and sampling the batch, the mix is ready to be charged to the casting system.

Propellant is discharged by gravity from the mixer to a 100-gallon hold tank similar to the mixer. This propellant is cast while another batch is being mixed. Thus, although mixing is done batch-wise, the casting operation operates continuously. Propellant and polymerization catalyst are metered in the correct proportion (approximately 100:1) by means of two positive displacement diaphragm type pumps to a two-gallon capacity inline mixer where the catalyst is mixed into the main propellant stream. Polymerization inhibitor is

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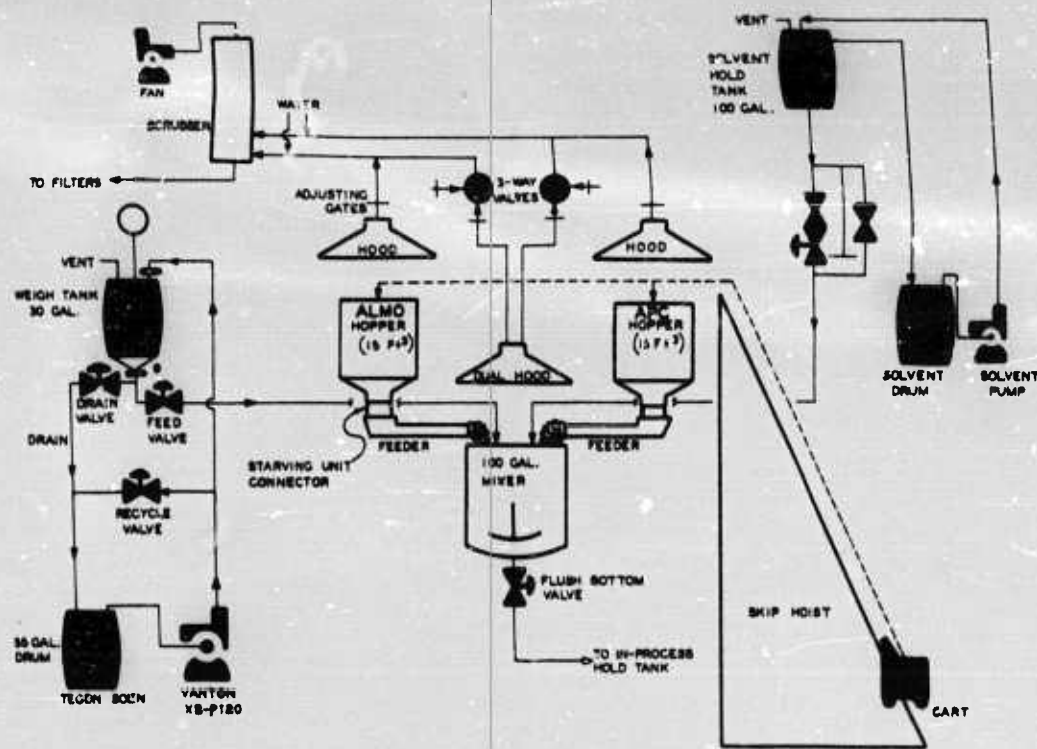


Fig. 1 Feeding and mixing system for 1200 lb. mixer facility.

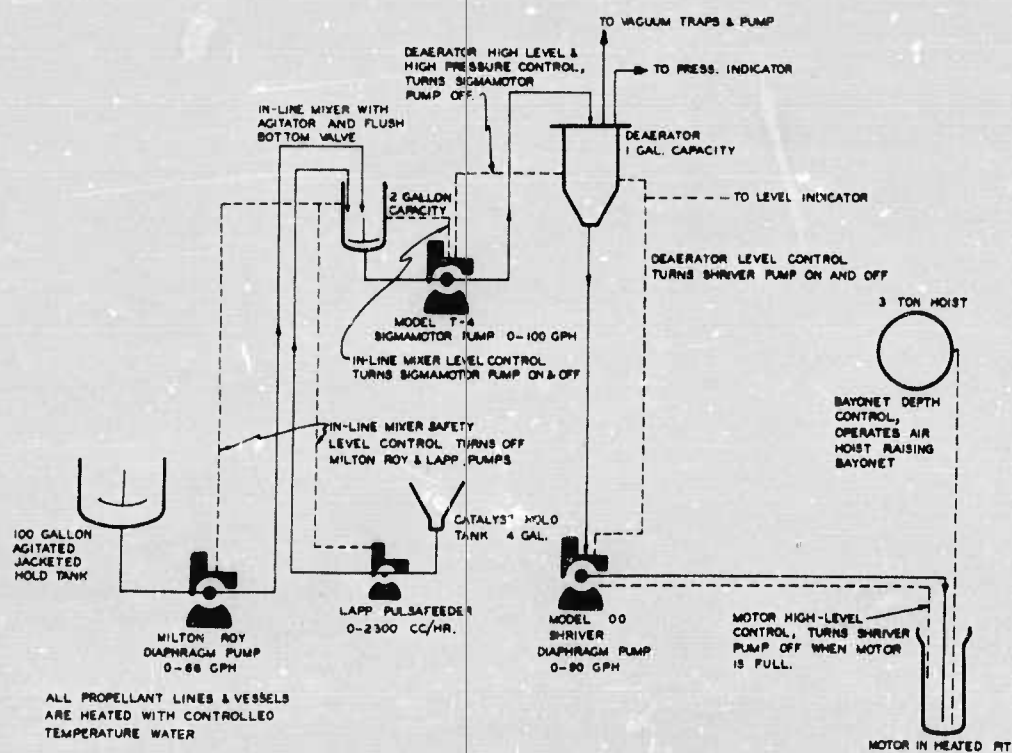


Fig. 2 Continuous casting system for 1200-lb. mixer facilities.

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added with the catalyst at a concentration adjusted to give the correct gel time for the propellant. This concentration is determined from suitable tests performed with the sample previously taken from the 100-gallon mixer. The catalyzed propellant is pumped by means of a Sigmamotor pump to a slit deaerator. The deaerator is elevated approximately 18 feet to allow the propellant to be pumped from vacuum by a diaphragm pump which pumps the mix through the final portion of casting line into the motor. The depth of the bayonet below the propellant surface is automatically controlled by a level sensor which raises the bayonet as the propellant level rises in the motor.

To compensate for differences in the pumping rates of the three propellant pumps in the system, each pump is sized so that its capacity under normal operating conditions is greater than the pumps upstream of it. The pumps are then controlled by level controls in the inline mixer and deaerator, which turn the pumps on and off to maintain a fixed, predetermined level in the respective vessels. The pumping rate of each pump is remotely variable (except for the propellant metering pump for which the stroke length must be manually adjusted) so that the rates can be adjusted to give essentially full time operation of each pump. Other controls for the pumps include safety high-level controls for the inline mixer and deaerator and a high pressure control for the deaerator.

The facilities and process described above have now been in use since December, 1958 to cast approximately 30,000 lbs. of petrin acrylate propellant without any serious difficulty, including three 7000-lb. motors and four 1000-lb. motors. This number of motors was adequate for programs underway; the proportion of available facility time used was low.

Raw Material Quality Control

It was expected that monomer and TEGDN quality control problems could cause large variations in the final propellant properties. This expectation was fortunately not realized and to date no propellant failure has been traced to variations in the raw materials as long as specifications were met (see Table II). The preparation of about 15,000

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lbs. of satisfactory monomer at Rohm & Haas Company through 1958 and 1959 (to date) is an indication that the present process is under control. TEGDN must, of course, be dried to prevent erratic polymerizations.

Table II
Specifications on Raw Materials

	<u>Petrin acrylate</u>	<u>Triethylene glycol dinitrate</u>
Assay ¹	97% Min.	99 - 103%
H ₂ O	0.25% Max.	0.25% Max.
Total Volatiles		0.50% Max.
Diacrylate (chromatography)	2.0% Max.	
Acid		0.01% Max.
Stability	Pass	

The other purchased raw materials, meeting manufacturer's specifications, were all found to be satisfactory.

Raw Material Logistics for Petrin Acrylate Propellants

All raw materials for petrin acrylate propellants are now in commercial production except petrin acrylate monomer. At present, Picatinny Arsenal is supplying petrin which is converted to the acrylate at this Division. The present capacity here is approximately 10,000 lb./month which will produce about 60,000 lbs. of propellant. The Explosives Division of E. I. duPont deNemours Company is currently undertaking process studies for the production of petrin and petrin acrylate. They are reported to have in operation a continuous process pilot plant for the production of petrin acrylate.

Triethylene glycol dinitrate has been purchased from Propellex Chemical Company and from Hercules Powder Company. However, E. I duPont deNemours and Olin-Mathieson are also interested in the manufacture of this nitrate plasticizer which can be produced in a commercial continuous Biazzi unit. The other raw materials are either used in small quantities (2-Ethyl hexyl acrylate, Plaskon 920,

¹Petrin Acrylate - chromatography

TEGDN - Nitrate ester determination

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Magnesium Oxide, nitroso diphenyl amine, ethyl centralite) and are commercially available in the required quantity, even for very large scale production, or are identical with the raw materials used in other propellants (aluminum and ammonium perchlorate).

BALLISTICS DEVELOPMENT

Full-Scale Nike-Zeus Loadings and Firings

Ballistic development details leading to full-scale motor loading and firing are given in Appendix B.

The 36 x 140 STM is a workhorse case of the Nike-Zeus sustainer, designed to hold 6,800 lbs. of propellant. A modified AG star, (Fig. 3) with flattened outer points, was designed and purchased. The surface-time trace, (Fig. 4) shows the effect of the grain shortening due to the rounded ends of the motor case. Note how the actual firing traces duplicate this theoretical curve. A new mandrel could easily be designed to eliminate this regressivity if desired. Very little erosivity has been found for QZ_{bn} even at $A_p/A_t = \frac{1}{0.8} = 1.25$.

Motor RH No. 9 - Processing and Ballistics (Fig. 5)

The first 36 in. motor was cast on February 10, 11, 1959 from seven batches of QZ_{bn} propellant. The processing proceeded with remarkable smoothness. A full length grain of excellent quality resulted. Mandrel cooling was used to limit the exotherm. The trimmed grain weight was 6,945 lbs. and the propellant density was 0.0647 lbm/cu in.

The firing was highly successful. Ignition was good and no erosive spike occurred. The pressure trace was smooth, but regressive throughout most of the shot. The total impulse exceeded specifications for the sustainer. The Planeton IV insulation was completely burned through in the converging face of the nozzle in line with each of the six star valleys. The exit cone delaminated causing periodic thrust disturbances. Stripping of the expansion cone insulation was readily seen in the motion pictures of the firing. Excellent agreement was obtained between Rohm & Haas Company and ARGMA instrumentation.

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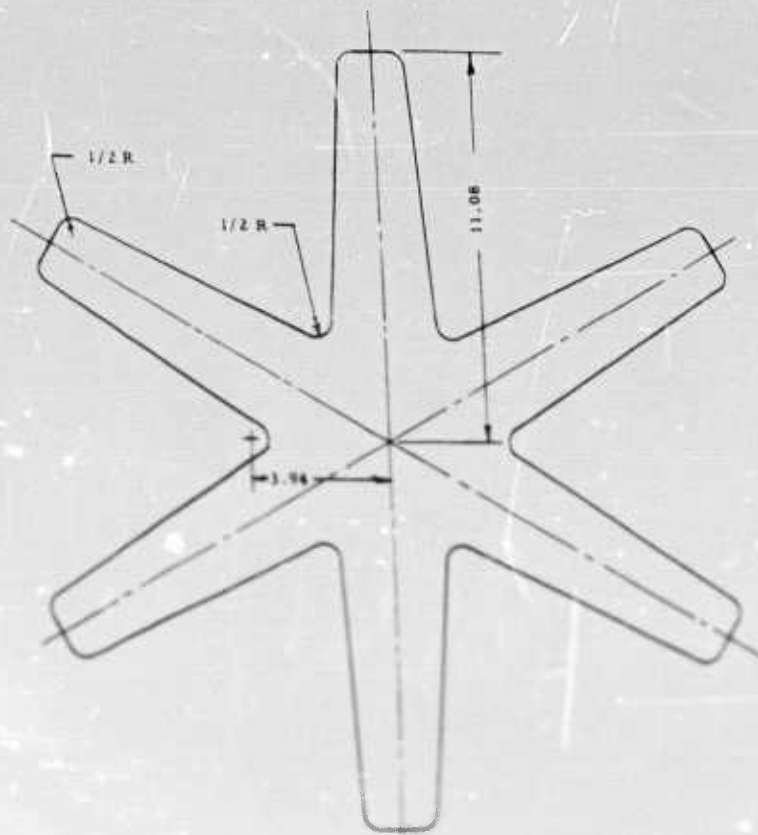


Fig. 3 Cross section of the modified AG mandrel for the full-scale Nike-Zeus.

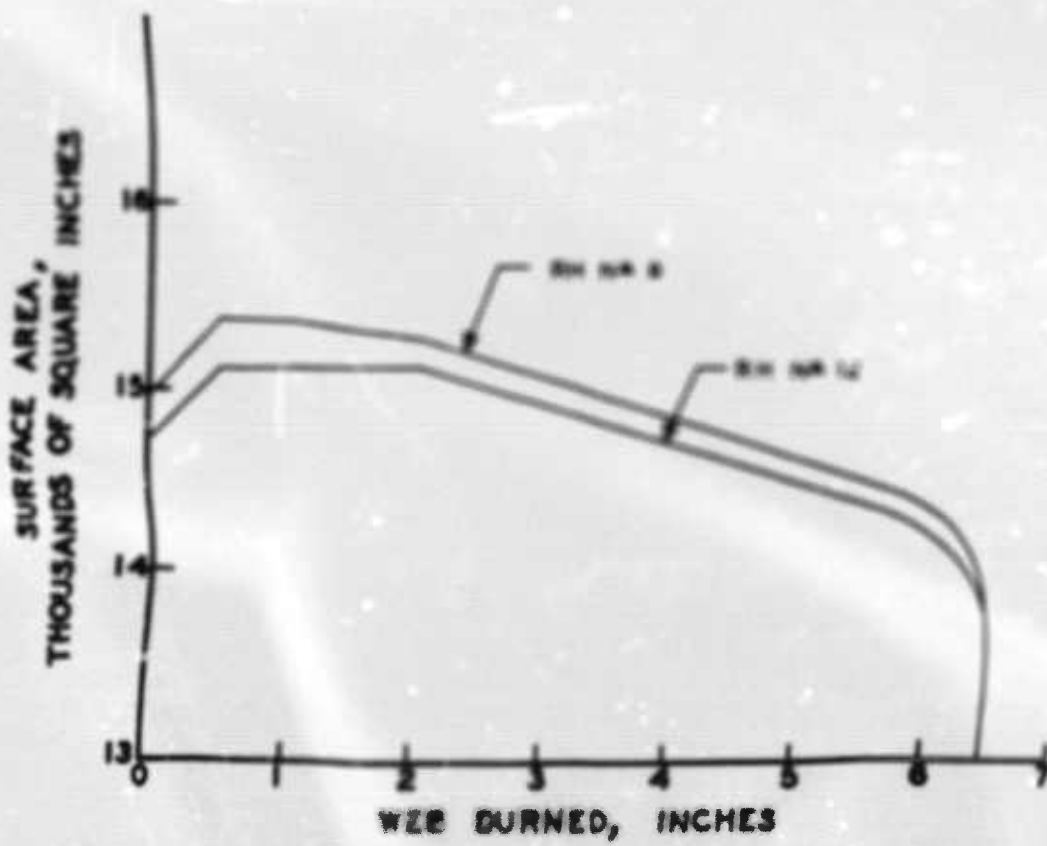


Fig. 4 Calculated surface as a function of distance burned for the full-scale Nike-Zeus

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Project No. T41-3060A		RH Project No. 62-24.1		Static Test No. 7		Report No. R44 8		Test Date 2/18/59	
MOTOR DATA				PROPELLANT DATA					
Type of Case - 16 in. Workhorse		Loaded Wt., lb. 5314		Batch No. C-21, 22, 23, 24, 25, 26, 27		Composition: QZ ₈₀		Instrumentation By: Rohm & Haas, ARGMA	
Serial No. 5681912 E		Fired Wt., lb. 2317		Configuration - Modified AG Star		Grain Wt., lb. 6945		C _D , lbm/lbf-sec 0.046	
		Wt. Consumed, lb. 6997		OD, in. 15.104		Grain Length, in. 155.8		C _{Do} , lbm/lbf-sec 10.856	
				ID, in. 22.484		Density, lbm/cu.in. 0.8047		c _w , ft/s-c 17.456	
				Web, in. 6.410		Temp., °F Amb.		t ₀ , sec. (0 thrust) 0.59	
				Port Area, sq. in. 123.946		Igniter Type Jelly Roll		r _n , lbm/sec 438	
				Mean Surface, sq. in. 15.004		Igniter Wt., gm. 1800		F _{max} , lbf 91.571	
								F _{av} , lbf. 1,597,449	
								I _{av} , lbf-sec 230.0	
								I _m , lbf-sec/lbm 244.8	
								F _{time} , lbf-sec/lbm 1.48	
								C _F 0.77	
								C _{F1000} 121.1	
								K _m	
NOZZLE DATA				LINER AND RESTRICTION DATA					
D _t (av. before), in. 12.465		A _t (before), sq. in. 122.032		Liner Comp. LR7-73		Restrictor Comp. LR7-73		∫ p _{dt} , psi-sec 9159	
D _t (av. after), in. 12.659		A _t (after), sq. in. 125.861		Liner Wt., lb. 150		Restrictor wt., lb. 7.0		(A _t /A _v) ^{1/2} initial 0.77	
D _e (av.), in. 31.83		A _t (av.), sq. in. 123.946		Liner Thickness, in. 0.22		Location: Aft End.		C _{F1000} 121.1	
Expansion Ratio 6.4		Closure 0.063 Aluminum							
Insert Mat. Graphite		Insulation Mat. Planeton IV							

Motor RH No. 11 - Processing and Ballistics

The second 36 x 140 STM was successfully cast from seven batches of QZ_{bn}. However, the catalyst was inadvertently left out of one batch so that the grain could not be fired in the normal manner. This motor has been stored in an Army Igloo at ambient temperature for surveillance purposes. No major changes have resulted from one month of storage.

Motor RH No. 12 - Processing and Ballistics (Fig. 6)

The third 36 x 140 STM was successfully cast from seven batches of QZ_{bn} propellant. A small amount of fissuring and some case bond separation was noted at the aft-end of the motor after cure; but the propellant was successfully repaired. The firing was intended to be at an average pressure of 1,000 psi and was actually 1,018 psi. The nozzle condition was somewhat better than on motor RH No. 9, particularly in the converging section. Although the remaining insulation in the expansion cone was about 1/8 inch more than in RH No. 9, the original thickness was 1/8 inch greater, indicating an equivalent amount of delamination for the two tests.

The relationship of the P-K-r data from the latest large motor firings is shown on the P-K-r curves for QZ_{bn} propellant (See Fig. 7).

Surveillance

Only a very limited amount of data have been obtained on surveillance. Some data have been obtained on an older composition, but these data are not presented here because the information may not be applicable to the current composition, QZ_{bn}. At the present time, ten 10-lb. motors are in surveillance at 135°F and one full-scale motor is in ambient storage. Inspection of the full-scale motor after about one month storage has indicated no significant change in propellant quality.

Cycling

No cycling data have been obtained on the current composition.

Detonability

As is expected from any high energy propellant, composition QZ_{bn} can be detonated under the proper conditions. Limited tests have

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Project No. T01-0000A	Alt Project No. 62-20-1	Static Test No. 9	Round No. RH 12	Test Date 5/20/59
MOTOR DATA				
Type of Case - 20 in. Double-base	Loaded Wt., lb. 9500	Batch No. C-10, 39, 40, 41, 42, 43, 44	Composition: QZ ₁₀	Instrumentation By: Rohm & Haas, ARGMA
Serial No. 544-712-001 RD-1102	Filed Wt., lb. 2355	Configuration Modified AG Star	Grain Wt., lb. 6900	$t_{\text{ign}}^{\text{sec.}}$ 0.101
	Wt. Contained, lb. 6515	CD, in. 35.246	Grain Length, in. 134.1	$t_{\text{b}}^{\text{sec.}}$ 9.294
MEASURE DATA				
P_1 (in. barbed), in. 11.710	A_1 (barbed), sq. in. 96.410	TD, in. 22.295	Density, lbm/cu. in. 0.0646	$t_{\text{a}}^{\text{sec.}}$ (0 thrust) 16.762
P_2 (in. barbed), in. 11.497	A_2 (barbed), sq. in. 101.015	Web, in. 6.476	Temp., °F +83 (Amb)	$r_{\text{b}}^{\text{in/sec}}$ 0.70
P_3 (in. barbed), in. 11.493	A_3 (barbed), sq. in. 101.120	Port Area, sq. in. 15.6	Igniter Type Jelly Roll	$P_{\text{ign}}^{\text{psi}}$ 294
P_4 (in. barbed), in. 11.493	A_4 (barbed), sq. in. 101.120	Mean Surface, sq. in. 14.784	Igniter Wt., gm. 1800	$P_{\text{max}}^{\text{psi}}$ 1140
Expendable Motor 7.8	Chamber 4 in. Frictioned plastic			$P_{\text{b}}^{\text{psi}}$ 1020
Igniter type: Composite	Insulation Mat. Phenolic IV			$P_{\text{a}}^{\text{psi}}$ 840
		LINER AND RESTRICTION DATA		$t_{\text{m}}^{\text{lb-sec/lbm}}$ 240.0
		Linear Comp. LR7-73	Restrictor Comp. LR7-73	$\int P dt, \text{psi-sec}$ 11,139
		Linear Wt., lb. 170	Restrictor Wt., lb. 10	$F_{\text{max}}^{\text{lb-sec/lbm}}$ 247.8
		Linear Thickness, in. 0.25	Location: Aft End	C_F 1.52
				$C_{F_{\text{max}}}$ 1.58

REMARKS: Purge of firing was to obtain a 1000 psi average pressure in the Nike Zeus container.

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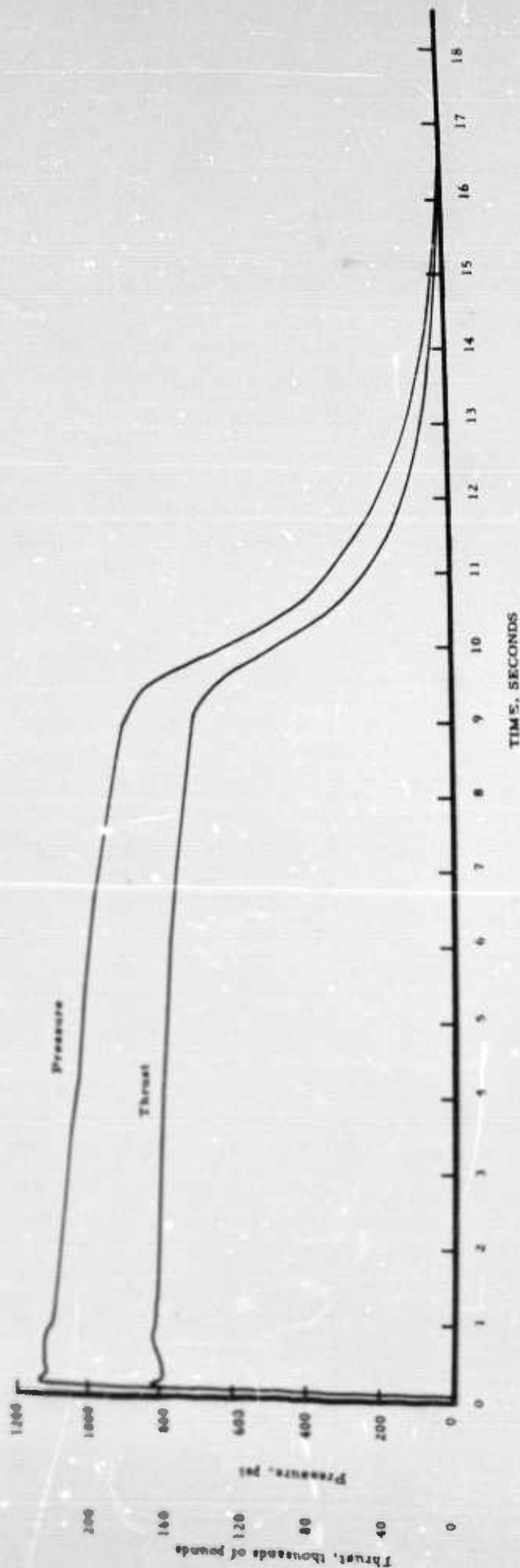


Fig. 6 Ballistic data for the full-scale Nike-Zeus motor, RH No 12

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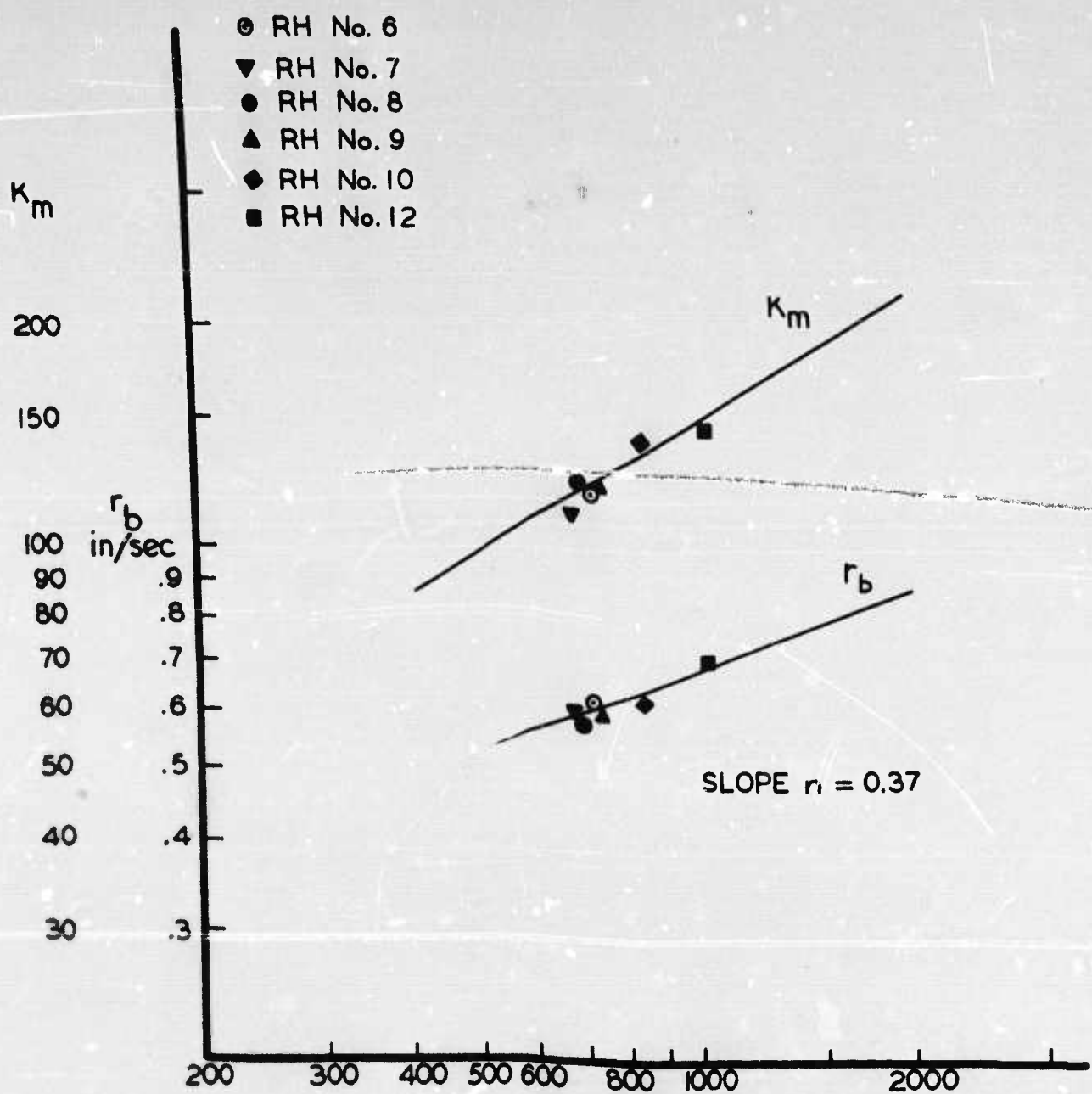


Fig. 7 Pressure, mean K, and burning rate variation for large motor firings

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indicated a minimum or critical diameter of between two and two and one-half inches below which detonation will not occur. For these tests approximately one pound of composition C-4 was used as a booster which was initiated by a number 8 blasting cap. The propellant samples, confined in a wrapping equivalent to an ice cream carton, detonated at 2-1/2 inches diameter, but failed to detonate at 2 inches diameter. It is not known what minimum size of booster could cause the sample to detonate.

METAL PARTS DEVELOPMENT

To test petrin acrylate in the Nike-Zeus system, it became necessary to develop a nozzle and liner material which would withstand the higher temperature of this high energy propellant and which could be used with the Zeus workhorse motor, supplied by Douglas Aircraft Company. Therefore an investigation was made to determine a list of possible materials to use in areas exposed to the flame. To conserve materials in the early part of development, it was also necessary to design and build smaller-scale test motors and nozzles. One-half and one-fifth scale motors were built.

The half-scale motors contain approximately 1000 lb. of propellant and require identical preparation to the full-scale motor. The one-fifth scale motors contain only 50 lb. of propellant and because of their shorter burning time, less insulation is required. Discussion will be limited to the one-half and full-scale motors.

Half-Scale Motors and Nozzles

The half-scale motors (see Fig. 8) were designed to contain approximately 1000-lb. of propellant, based on the original estimated sustainer weight of 8000 lb. Since the primary interest at Rohm & Haas Company is the development of propellants, the motor was designed as simply as possible for repeated firings. The motor was fabricated from non-heat treated steel of rolled and welded construction and hydrostatically tested to 1800 psi. The nozzle was fabricated from low carbon steel with a graphite nozzle insert. After obtaining severe erosion in the first firing, the successive nozzles were lined with a plastic,

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either Planeton IV¹ or Rocketon², in both the converging and diverging sections. The first motor and nozzle assembly was purchased from Diversey Engineering Company. Three additional motors were purchased from Diversey, while the nozzles were obtained from Joe Brady's Shop.

Planeton IV has been used in all the firings to date, although other nozzles have been prepared with Rocketon for future tests. The Planeton IV has prevented the nozzle from being destroyed with each firing. However, since the Planeton is a laminated material, successive layers are heated and stripped out of the nozzle during burning. Although this does not affect the propellant, slight irregularities are periodically indicated on the thrust trace as each layer is stripped from the nozzle. For this reason, Rocketon, which is a molded plastic consisting of asbestos and a phenolic resin, will be tried in future tests.

Erosion or ablation of the surface should be more uniform.

Full-Scale Motors and Nozzles

Three full-scale workhorse type motors (see Fig. 9) have been received from Douglas Aircraft Company. The nozzle which was supplied with the first motor was not adequate for use with petrin acrylate propellant. Therefore, a new design was made and the nozzle metal parts were purchased by this Division from Richardson Machine Company. (See Fig. 10). After firing the first full-scale motor, the nozzle design was modified and two more sets of metal parts were ordered from Richardson. All motors and nozzles were plastic lined similar to the half-scale components. Planeton IV was formed into the converging rear portion of the motors as well as into both the converging and diverging parts of the nozzles.

Both full-scale firings have been successful, both from a ballistic standpoint and from a component standpoint. The 3/8-inch Planeton IV in the expansion cone of the first nozzle delaminated during firing until only one or two layers remained. To be more conservative, the nozzle for the second firing had the Planeton IV liner thickness increased to

¹A product of Reinhold Engineering and Plastics Co., Norwalk, Calif.

²A product of Haveg Corporation, Wilmington, Delaware.

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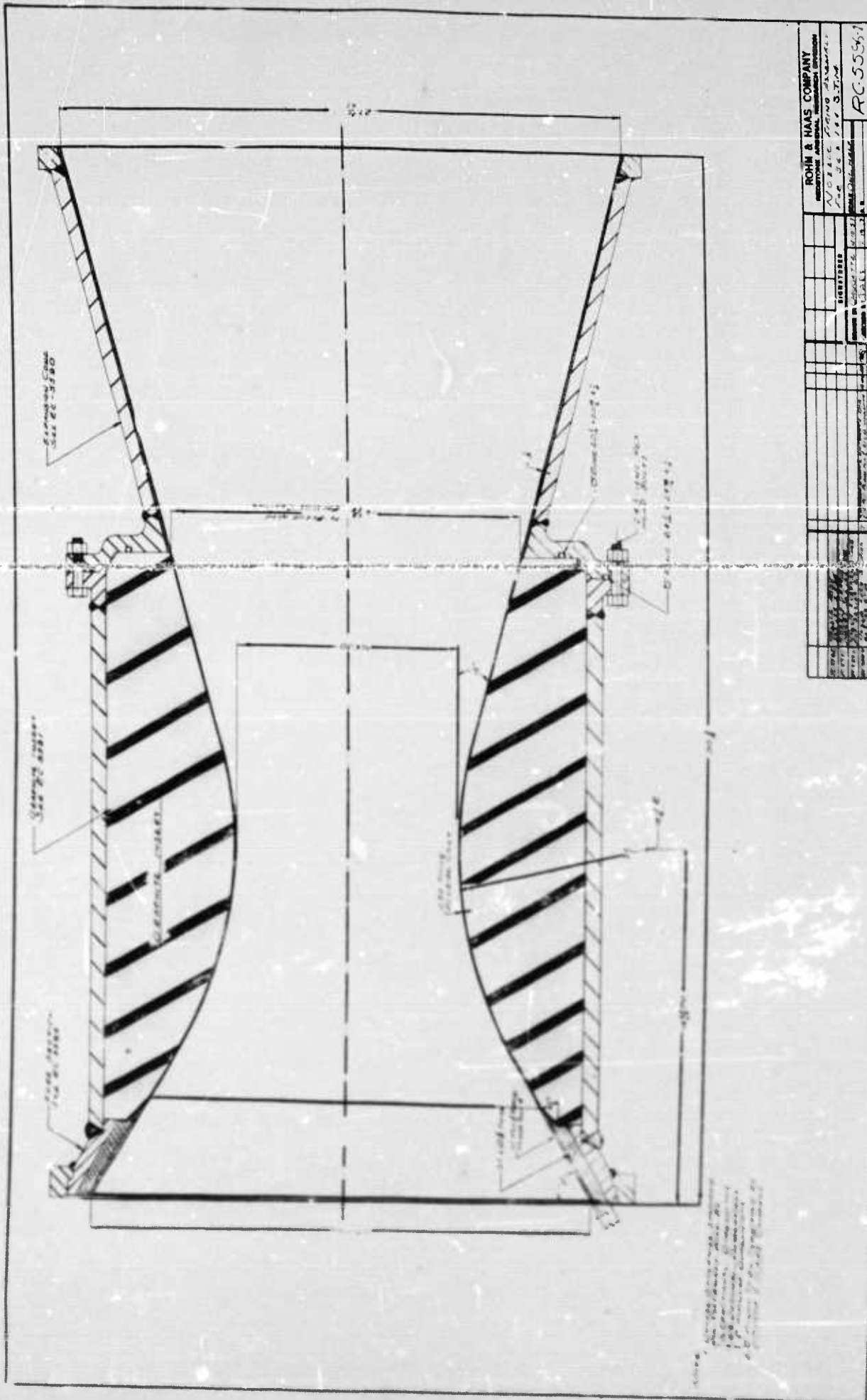


Fig. 10 Nozzle firing assembly for Nike-Zeus Workhorse static test motor

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1/2-inch in the expansion cone. After firing, only 1/8-inch remained, resulting in the same amount of delamination for both firings.

For future firings, molded insulator rings will be used in the converging section of the nozzle, the diverging section will continue to use the laminated Planeton IV for economic reasons.

To date, extruded ATL graphite has been used for all nozzle throat sections. The increase in area due to erosion has been 3% and 5% in the two full-scale firings, but it has varied from 2% to 6% in the half-scale firings. This variation could probably be reduced by using a better quality molded graphite for the nozzle throats. However, the cost would be higher and the increase in cost did not appear justified for initial firings of a static test vehicle.

Motor Liner

A mixture of chopped asbestos, polyester P-13 resin, and polyester P-43 was selected as the liner material for the large motors. This material has been spread by hand, completely covering the inside of the motor case, for all firings conducted to date. However, it has been determined that this material can be sprayed successfully and the spray method would probably be used for lining several motors of a "production" run. Purchase of the spray equipment could be justified if several motors were to be lined. Compatibility of the propellant and polyester liner has been satisfactory as far as initial bonding is concerned. The possibility of liner deterioration due to plasticizer migration is being checked on motors which are stored for surveillance.

An investigation is currently under way to develop a liner, pre-plasticized with triethylene glycol dinitrate (TEGDN), which will have enough plasticizer in the liner, so that no further migration will occur. Results of current tests are very encouraging.

Since the normal liner is not sufficient insulation in the converging rear section of the motor, an additional layer of insulating or heat resistant material was required. Planeton IV, a laminated glass cloth impregnated with a phenolic resin was selected. A layer approximately 3/4 inch thick, overlayed with a normal layer of P-13, asbestos liner material, has proved to be adequate for full-scale firings.

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APPENDIX A

Processing

Composition QZ_{bn} (see Table I) was selected as the most promising propellant for the full-scale Zeus sustainer. This composition has been demonstrated in half-scale and full-scale static test motors.

Since uncoated ammonium perchlorate tended to cake and become difficult to charge to the mixer, an effective anti-caking agent (see Fig. A-1) was sought. Magnesium oxide was selected. A concentration of 1% magnesium oxide coated on the 65 μ ammonium perchlorate eliminated the caking problem and also resulted in improved thermal stability and improved physical strength of the propellant. The effects of magnesium oxide on the thermal stability and physical properties of QZ_{bn} are presented in Tables A-I and

A-II.

Table A-I

Effect of MgO on Thermal Stability of QZ Propellant

% MgO Based on Ammonium Perchlorate	Test Temperature °F	Auto Ignition Time Hours
0	248 ¹	7
1	248 ¹	25
2	248 ¹	44
0	212	33, 40
0.5	212	>115, 790
1	212	>115
0	195	140

¹Accelerated test using small samples. All other samples were about 14 lb., 6 inch dia. cylinders.

Table A-II

Effect of MgO on Physical Properties of QZ Propellant

Oxidizer ¹	Average Physical Properties at 75°F	
	Tensile Strength, psi	Elongation, %
Uncoated	28	22
Coated with 1% MgO ²	45 to 65	33 to 40

¹Ammonium perchlorate ground to 65 μ medium particle size.

²A USP light magnesium oxide is used. A reagent grade magnesium oxide is slightly less effective in terms of physical properties improvement, giving values on the low side of the range reported.

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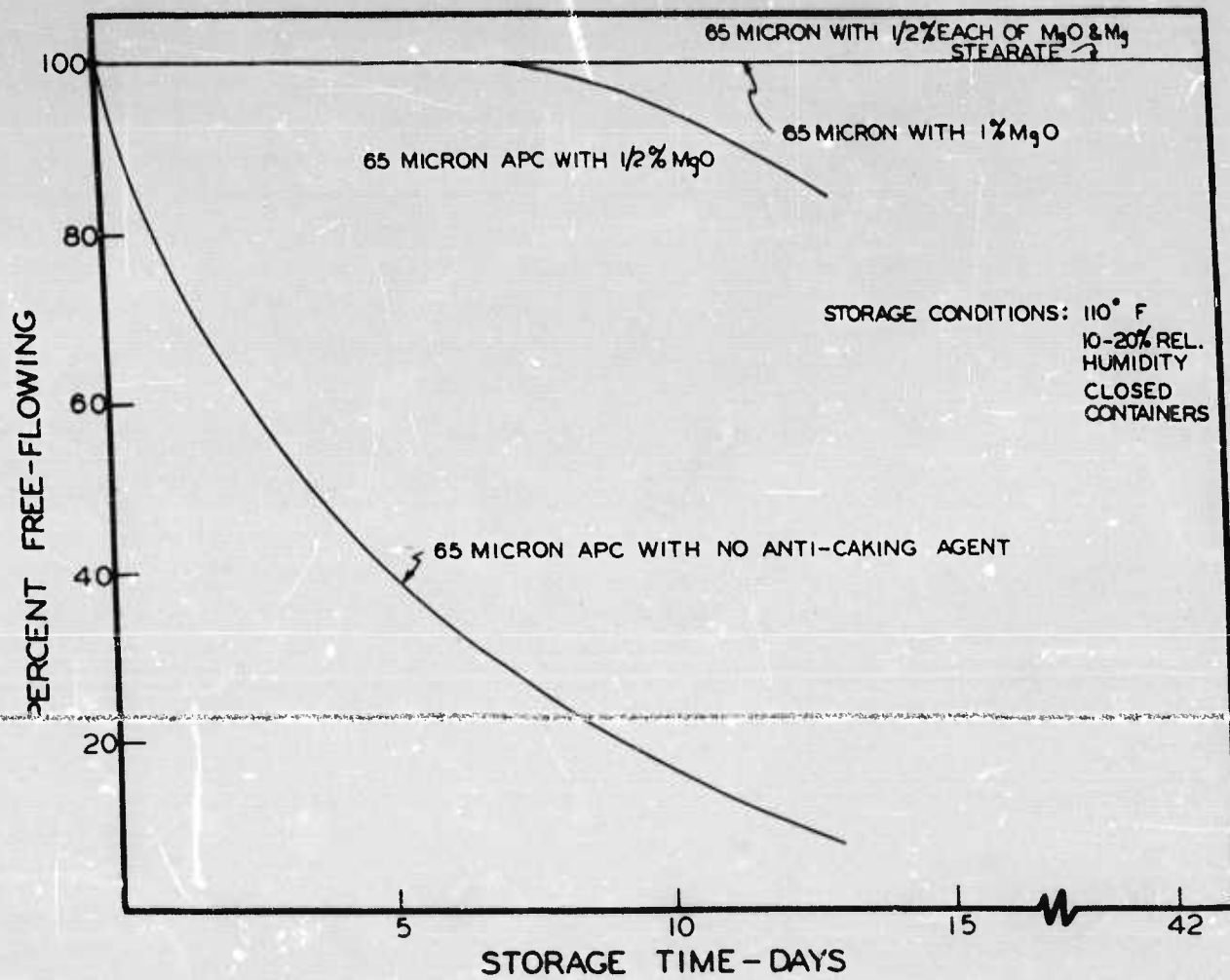


Fig. A-1 Effect of anti-caking agents on the caking characteristics of ammonium perchlorate.

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The rate of increase of viscosity is controlled to result in a viscosity of 10,000 poises¹ after 60 to 180 minutes from an initial viscosity of about 30 poises¹. A time of less than 60 minutes may result in solidification in the casting lines, while a time greater than 180 minutes results in settling of the solids in the motor.

It has been determined that any appreciable polymerization reaction occurs only after catalyst addition and after deaeration. To prevent decomposition of the inhibitor during mixing, it is added in the casting line with the catalyst as late as possible in the process.

To determine the desired amount of inhibitor a small sample of propellant is withdrawn as soon as the propellant is mixed, catalyzed, and tested for viscosity increase. From this test the inhibitor level for the batch is chosen. This technique assures an adequate gel time, even with gross variations in the proportioning of the catalyst-inhibitor solution in the propellant (see Table A-III).

Table A-III
Effect of catalyst-Inhibitor Solution Proportioning Variations on Gel Time of QZ Propellant

<u>Catalyst-Inhibitor Solution</u>	<u>Gel Time, minutes</u>
Nominal	90
Twice Nominal	120
Half Nominal	75

The polymerization rate is also increased appreciably by traces of water (see Fig. A-2). Slight variations in the amount of water in the batch are virtually impossible to avoid due to the hygroscopic nature of the ammonium perchlorate. The effect of water and other unclassified factors are successfully overcome by using a suitable level of inhibitor (see Fig. A-3). This level of inhibitor is determined by the method described above. Although this technique has been very effective and has permitted successful continuous operation, a more powerful polymerization control system that will tend to override the effect of uncontrolled polymerization variables is highly desirable, and an investigation of more powerful catalyst-inhibitor systems is in progress.

¹Apparent viscosity in a Brookfield Viscometer using a T spindle at 5 R.P.M

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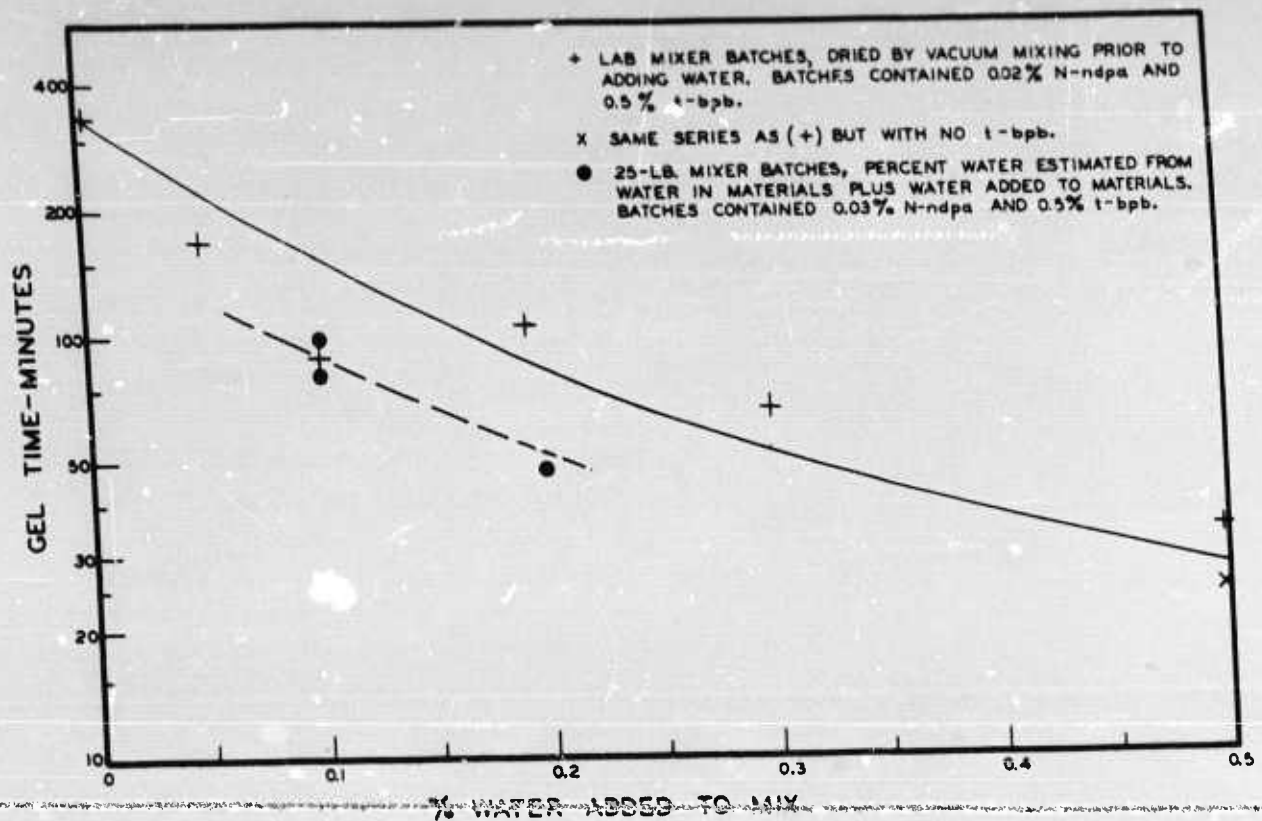


Fig. A-2 The effect of water on QZ propellant gel time.

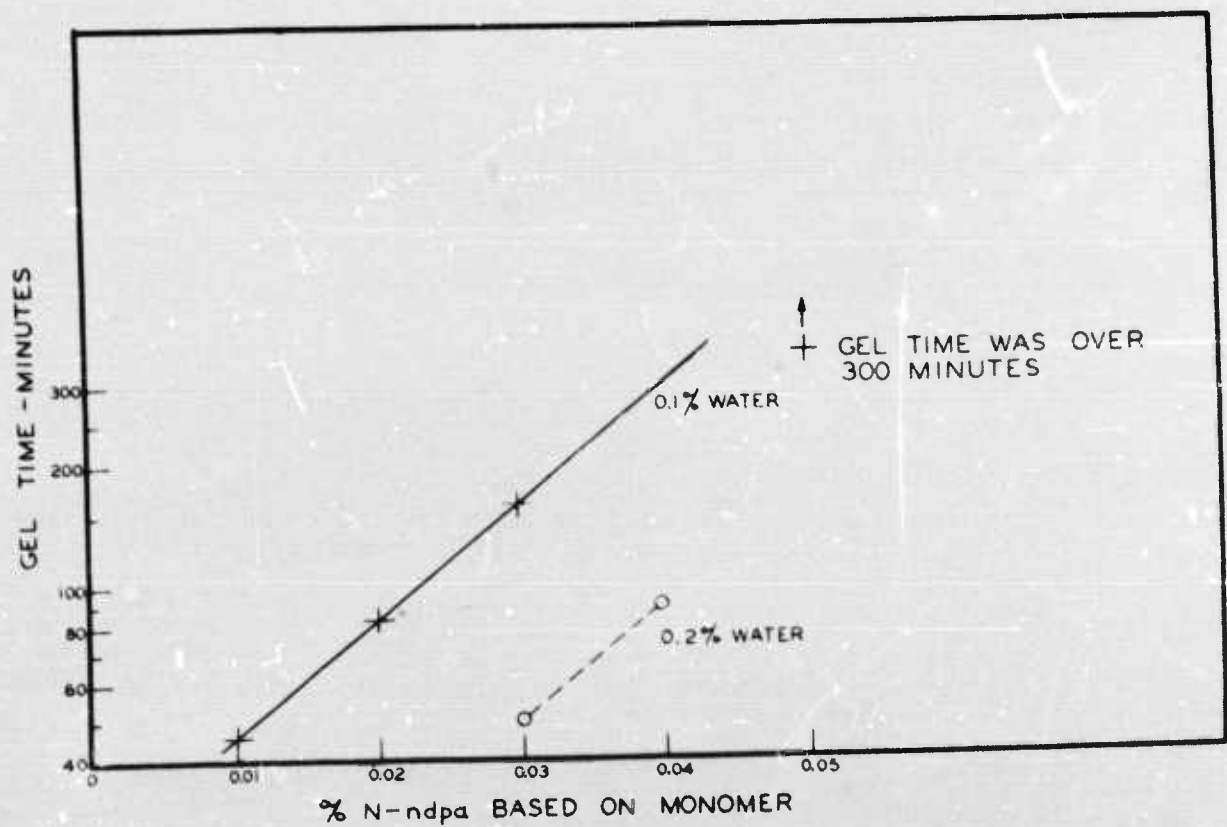


Fig. A-3 The effect of inhibitor concentration on gel time of QZ propellant containing traces of water.

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APPENDIX B

BALLISTICS DEVELOPMENT

Burning Rate Studies

The initial burning time for the Zeus sustainer was 17 seconds at 650 psi. To meet this requirement two charge configurations were considered; the AG star (see Fig. 3, B-1) which required a burning rate of 0.44 inches per second, and a slotted tube which required a burning rate of 0.77 inches per second, both at 650 psi.

Composition QC_d, which was the most likely candidate for use in the Zeus system at the beginning of the development program, was a compromise, with a burning rate of 0.53 inches per second at 650 psi. The first three 1000-lb. motors were loaded with this composition because its characteristics were reasonably well established.

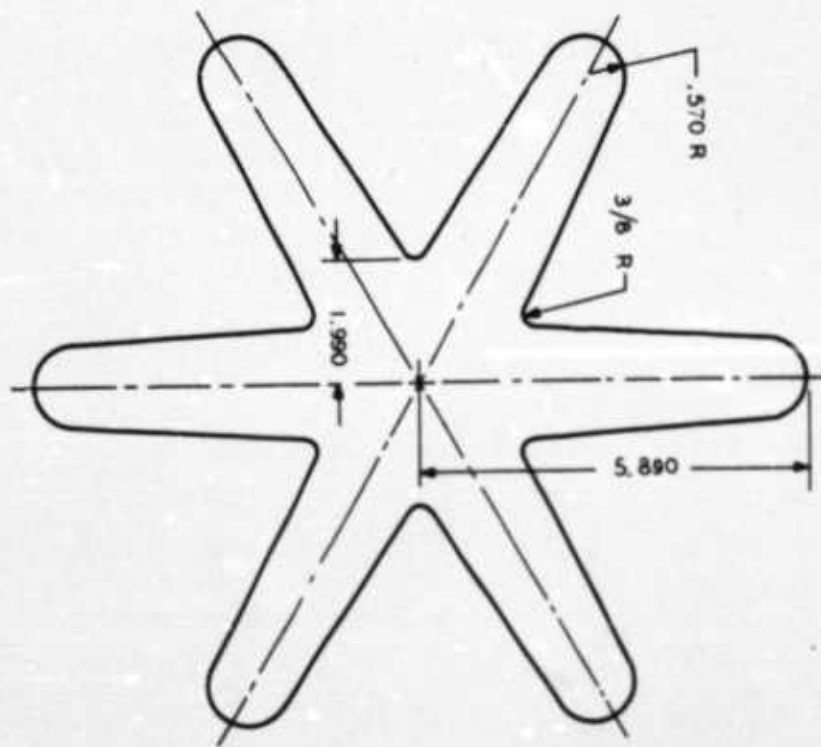


Fig. B-1 Cross section of the AG mandrel for the half-scale Nike-Zeus

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Because of its improved physical and ballistic properties and its simpler processing requirements, composition QZ_s replaced QC_a.

Since two charge geometries were considered and two different burning rates were required, a series of tests, using 6C5-11.4 static test motors (STM (charge = 6" OD x 5" ID x 11.4" long), was conducted to determine the range of burning rates which could be expected with QZ composition, using various oxidizer grinds. Seventy-six 6C5-11.4 motors,* each containing seven pounds of propellant, were fired to obtain burning rate modification data. A burning rate range of from 0.42 inches per second to 0.81 inches per second at 650 psi was established.

Before these results could be utilized, the sustainer burning time was decreased to 12 seconds. At this time the slotted tube configuration was dropped, since QZ_s came close to providing the correct burning rate for the AG star charge configuration. Additional 6C5-11.4 motors were fired to check out minor modifications of QZ_s burning rate and specific impulse.

To simplify the oxidizer handling during the propellant processing, a small amount of magnesium oxide (1% based on oxidizer) was added to the ammonium perchlorate to prevent caking in the hopper. Two secondary effects of MgO were the improved thermal stability and substantially improved physical properties. The resulting composition (QZ_s with 1% MgO) was designated QZ_{bn}. Although the burning rate of QZ_{bn} was about 10 - 15% higher than that of QZ_s, it was satisfactory for ballistic evaluation of the propellant.

The latest changes in requirements, namely a 9 second burning time and 1000 psi chamber pressure, fit in almost exactly with the properties of QZ_{bn} which has a burning rate of about 0.7 inches per second at 1000 psi. The most recent full-scale firing resulted in a burning time of 9.275 seconds at an average pressure over the burning time of 1020 psi. The calculated burning rate was 0.70 inches per second.

At the present time, as far as burning rate is concerned, the requirements of the Zeus sustainer are met almost exactly.

¹Ballistics Section Progress Report No. 73, "Burning Rate Studies for Nike-Zeus Application", L. M. Brown

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Specific Impulse

Compositions QC_d, QZ_s and QZ_{bn} have consistently delivered a specific impulse (F_{1000}^0) of 244 to 248 lbf-sec/lbm. However, it was believed that the specific impulse could be increased somewhat by changing the composition slightly to obtain the optimum combination of components. The percentage of aluminum was varied between 13% and 23%. The aluminum powder size was varied between 6 μ and 26 μ average particle size. The proportions of flake to atomized aluminum were varied. The quantity of total solids was varied between 63% and 69%. QZ composition contains 65.85% total solids.

Resulting specific impulse values varied from 239 to 250 lbf-sec/lbm obtained from a series of over 125 test firings. Variations from shot to shot were great enough to result in an average specific impulse the same as for QZ_{bn}. Therefore it was apparent that composition QZ_{bn} was very close to optimum. Also, it was observed that even fairly large changes in composition had very little effect on the specific impulse. For that reason, QZ_{bn} has been used for all the recent large motor firings.

STATIC TEST FIRINGS

Six-Inch (7 lb.) Motors

The 6C5-11.4 STM is the standard test motor for evaluating the burning rate and specific impulse of propellant compositions. A substantial amount of processing information has also been obtained from casting these motors.

For the Zeus program in particular, a total of 76 6C5-11.4 motors were cast and fired for burning rate studies. In addition, 163 motors containing QZ_s and QZ_{bn} compositions were fired to establish normal batch-to-batch variations in specific impulse and burning rate. These include sample motors which were cast from the same propellant batches used for each large motor. It has been established that these 6-inch sample motors exhibit performance comparable to the large motors made from the same batches of propellant, as relating to specific impulse and burning rate.

An additional 125 6-inch test motors were fired with modified QZ_{bn} compositions to determine the maximum specific impulse which

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is likely to be attained without drastic changes in propellant composition.

Two 6-inch static test motors have been fired successfully at each 0°F and -40°F for the Zeus program, using QZ_{bn} composition. A limited number of additional motors have been successfully fired at temperatures as low as -60°F, using QZ compositions.

One-Fifth Scale Motors (50 lb.)

A 7 x 28 static test motor was designed as an exact scale of the 36-inch Zeus sustainer to establish the charge geometry (shape of pressure and thrust traces) of the sustainer, using QZ compositions.

Eighteen motors were loaded and fired with various propellant compositions. The modified AG star (same shape as Fig. B-1) proved to have about the proper web and surface. Some of the traces showed slight erosion peaks, but not when using the final composition, QZ_{bn}. The pressure was neutral over the first half of the burning time and slightly regressive over the last half. One motor was successfully fired at -20°F.

100-lb. Motor Firings

Nine Jato motors were fired with the slotted tube design. Although this motor was primarily a propellant process development vehicle, valuable ballistic data were obtained. Burning rate and impulse data verified the results of the 6-inch motor firings.

Half-Scale Nike-Zeus Loadings and Firings

The 19 x 75 STM was designed to provide a test case for petrin acrylate propellants that would hold approximately 1,000 lbs. of propellant. This was the capacity of the large mixer. The motors for RH No. 1, 2, 3 and 5 were slightly longer than the final series. The final series was an exact scale of the 35.75 in. diameter workhorse motor, including 1/8 in. liner in both motors. The forward-end and aft-end closures were duplicated also. A six-point, AG star mandrel was fabricated for this motor. (See Fig. B-1). The AG geometry provides a constant surface during burning in a right circular cylinder motor, so that the pressure trace is regressive toward the end of burning when the effective grain length shortens due to the curved forward and aft closures. This regressivity, which is shown in Fig. 5, 6, B-2, B-3, B-4, B-5 and B-6 was not bothersome. The regressive nature

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was also accentuated by the taper of the mandrel.

The loadings and firings of this motor have not always been exact scales of expected 36 in. firings. Instead, they have been used to test processing techniques, grain designs, and nozzle materials.

RH No. 1 - Ballistic Design and Processing

The first 19 x 75 STM was successfully cast on September 4, 1957, from propellant batches QC_d-7, 9. A bag of silica gel was inadvertently left in the ammonium perchlorate charged to the second batch, and silica gel particles were distributed throughout the rear half of the grain. Each silica gel particle was a nucleus for a small crack. After the propellant was closely examined, it was cut out rather than risk a blow-up.

RH No. 2 - Ballistic Design and Processing

The second 19 x 75 STM was successfully cast during the week of November 7 - 13, 1957 of QC_d-128, 129. Several small cracks developed during the curing and cooling cycle. These were patched before firing. The firing was successful. Extensive erosion of the steel and graphite nozzle indicated problem areas in the test motor materials.

RH No. 3 - Processing

A third 19 x 75 STM was cast on December 31, 1957, with QC_d-142, 143. The grain cracked badly during the curing and cooling period. The interface between the two batches was not well bonded. In all, 13 cracks were patched with P-13 polyester resin before the firing.

After an ignition delay of 0.020 sec., the grain ignited smoothly and burned normally for one second; the pressure was 600 psi and the thrust 30,000 lbs. as expected. Then the propellant surface increased and a small amount of propellant was lost. About 0.25 sec. after the first break-up, the case was overpressured and ruptured. Subsequent examination of the burnout pattern on the motor liner indicated that the trouble spot was near the interface region between the two batches.

Matador Ballistic Design (Fig. B-2)

A Matador booster workhorse case was loaded with 1,030 lbs. of composition QZ_s-103 to check out a slotted tube grain design in a

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Project No. TU2-17	RH Project No. 62-24.1	Static Test No. 3	Round No. Matador
MOTOR DATA			
Type of Case - 23.25 in. Heavy Wall			
NOZZLE DATA			
D _i (av. before), in.	6.664	A _t (before), sq. in.	34.879
D _t (av. after), in.	6.848	A _t (after), sq. in.	36.831
D _e (av.), in.	18.13	A _t (av.), sq. in.	35.855
Expansion ratio	7.40	Closure	0.040 Aluminum
Insert Mat.	Graphite	Insulation Mat.	P-13 Asbestos
PROPELLANT DATA			
Batch No. QZ-103	Composition: QZ	Grain Wt., lb.	1031
Configuration - SF Slotted Tube	22.916	Grain Length, in.	75.0
OD, in.	15.957	Density, lbm/cu. in.	0.0648
ID, in.	3.480	Temp., °F	Amb.
Web, in.	226.5	Igniter Type	Tube
Port Area, sq. in.	4347	Igniter Wt., gm.	500
Mean Surface, sq. in.	LINER AND RESTRICTION DATA		
Liner Comp. - P-13 Asbestos	Restrictor Comp.	Restrictor Wt., lb.	None
Liner Wt., lb.	Restrictor Wt., lb.	Location:	None
Liner Thickness, in.	0.17		
STATIC FIRING SUMMARY			
Test Date 2/27/58	Instrumentation By: ARGMA		
t _{ign} , sec.	0.017	C _D , lbm/lbf-sec.	6.3 x 10 ⁻³
t _b , sec.	7.782	C _{Do} , lbm/lbf-sec.	6.3 x 10 ⁻³
t _a , sec. (0 thrust)	8.515	c*, ft/sec.	5076
r _b , in/sec.	0.447	m, lbm/sec	121.063
P _{ign} , psi	99	F _{max} , lbf.	37,535
P _{max} , psi	670	F _{av} , lbf	27,325
P _b , psi	555	I, lbf-sec.	232,686
P _a , psi	535	I _m , lbf-sec/lbm	225.7
∫ p dt, psi-sec	4553	F _{lim} , lbf-sec/lbm	240.6
(A _t /A _e) initial	0.154	C _F	1.44
K _m	122.8	C _{F_{lim}}	1.53

REMARKS: Hole burned in exit cone just back of insert at 3.6 sec.

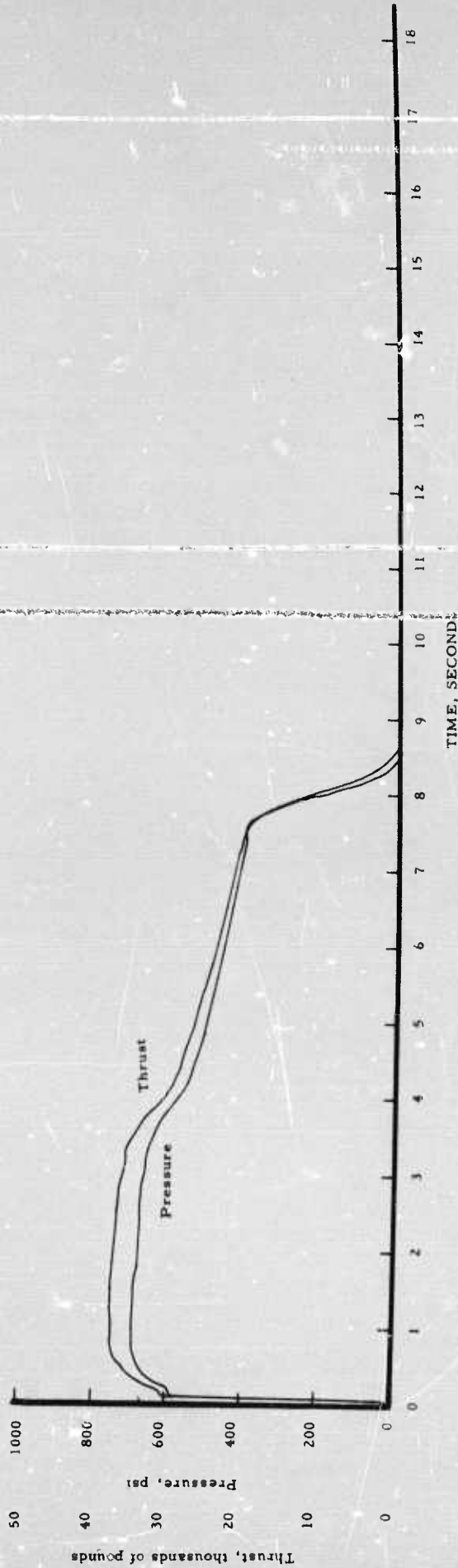


Fig. B-2 Ballistic data for the Matador motor

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1,000 lb. motor. Fissuring occurred during the curing, but it was not serious enough to prevent firing. The firing was successful although the nozzle expansion cone burned through about halfway through the shot.

RH No. 4 - Processing and Ballistic Design

The fourth 19 x 75 STM was cast on February 28, 1959, with QZ_s-107. Bad fissuring during cure necessitated cutting out the propellant.

RH No. 5 - Processing and Ballistic Design

Only 200 lbs. of propellant were cast into the fifth 19 x 75 STM before the propellant polymerized in the casting lines. The motor was fired, using a nozzle throat diameter of 6.5 inches. The smooth regressive trace indicated that petrin acrylate propellant will burn stably at low pressures; P_{max} was 43 psi.

RH No. 6 - Processing and Ballistic Design (Fig. B-3)

The sixth 19 x 75 STM was cast on December 23, 1958 using QZ_{bn} C-15 composition. The case was the first of the new series which was slightly shorter than the earlier motors. The case was not completely filled with propellant because of trouble during casting. However, the propellant was of excellent quality.

The motor was fired successfully. All metal parts stood up well, especially the Planeton IV* in the nozzle exit cone. The small pressure spike just after ignition was not expected to be, and is not believed to be the result of erosivity. Therefore, there may have been some slight surface increase at ignition.

RH No. 7 - Processing and High Temperature Firing (Fig. B-4)

The seventh 19 x 75 STM was cast on January 19, 1959. Again processing difficulties resulted in a slightly underfilled motor. After trimming and inhibiting, the motor was conditioned over the week end at 120°F. The firing was successful although a very bad pressure spike occurred when all 13 lbs. of the grain inhibitor was ejected through the nozzle shortly after ignition. Some break-up and surface change also

*A glass cloth laminate impregnated with a phenolic resin. A product of Reinhold Engineering and Plastics Company, Norwalk, California.

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REMARKS: Purpose of firing was to check out Q7. In 1960 to 1962.

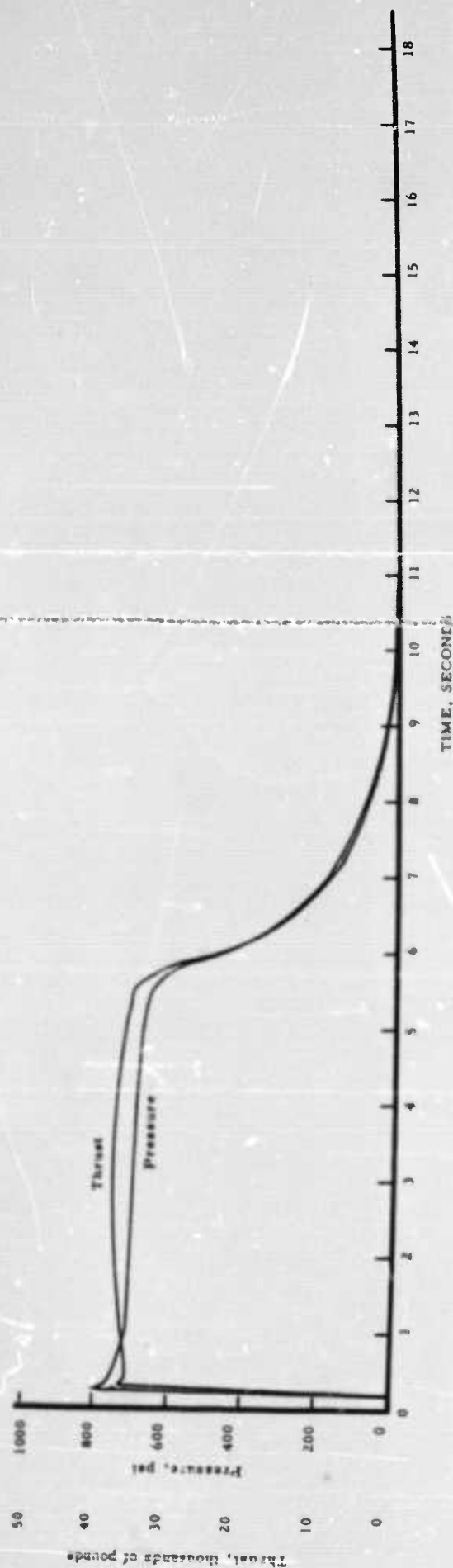


Fig. B-3 Ballistic data for the half-scale Nike-Zeus motor RH No. 6

Project No. TUI-3060A	RH Project No. 62-24.1	Static Test No. 5	Round No. RH
MOTOR DATA			
Type of Case - 19 in. Heavy Wall	Loaded Wt., lb.	Batch No. C17	Composition Q
Serial No. 2	Fired Wt., lb.	Configuration - AG Star	Grain Wt., lb.
	Wt. Consumed., lb.	OD in.	Grain Length, in.
NOZZLE DATA			
D_t (av. before), in.	6.558	ID in.	Denatly, lbm/cu in.
D_t (av. after), in.	6.617	Wt., in.	Temp., °F
D_n (av.), in.	16.97	Port Area, sq. in.	Igniter Type
Expansion ratio	6.6	Mean Surface, sq. in.	Igniter Wt., gm
Insert Mat. Graphite		LINER AND RESTRICTION DATA	
		Linear Comp.	1.87-73
		Linear Wt., lb.	29.3
		Linear Thickness, in.	0.14
		Restrictor Corn.	1.87-73
		Restrictor Wt., lb.	13.2
		Location	Alt End
•REMARKS: These values were taken at 0.5 seconds, not at the extreme. The bad pressure spikes occurred when the 13 lb. of restrictor broke loose from the end of the grain and momentarily plugged the nozzle. Purpose of firing was to test QZ ₅₀ at elevated temperature in 1000 lb charge.			

Test Date 1/26/59			
STATIC FIRING SUMMARY			
Instrumentation By: Rohm & Haas, ARGMA			
t_{gr} , sec.	0.160	C_D , lbm/lbf-sec	6.21×10^{-3}
t_b , sec.	5.616	C_D , lbm/lbf-sec.	6.51×10^{-3}
t_a , sec. (0 thrust)	9.200	C_F , ft/sec.	4941
\dot{m} , lbm/sec.	0.60	\dot{m} , lbm/sec.	103.31
P_{gr} , psi	51	F_{max} , lbf.	36,905*
P_{max} , psi	763*	F_{av} , lbf.	22,896
P_b , psi	675	F_r , lbf-sec	210,558
P_a , psi	555	I_m , lbf-sec/lbm	221.6
$\int P dt$, psi-sec	4491	F_{1000} , lbf-sec/lbm	238.0
(A_t/A_p) initial	0.76	C_F	1.44
K_m	111.1	C_{F1000}	1.55

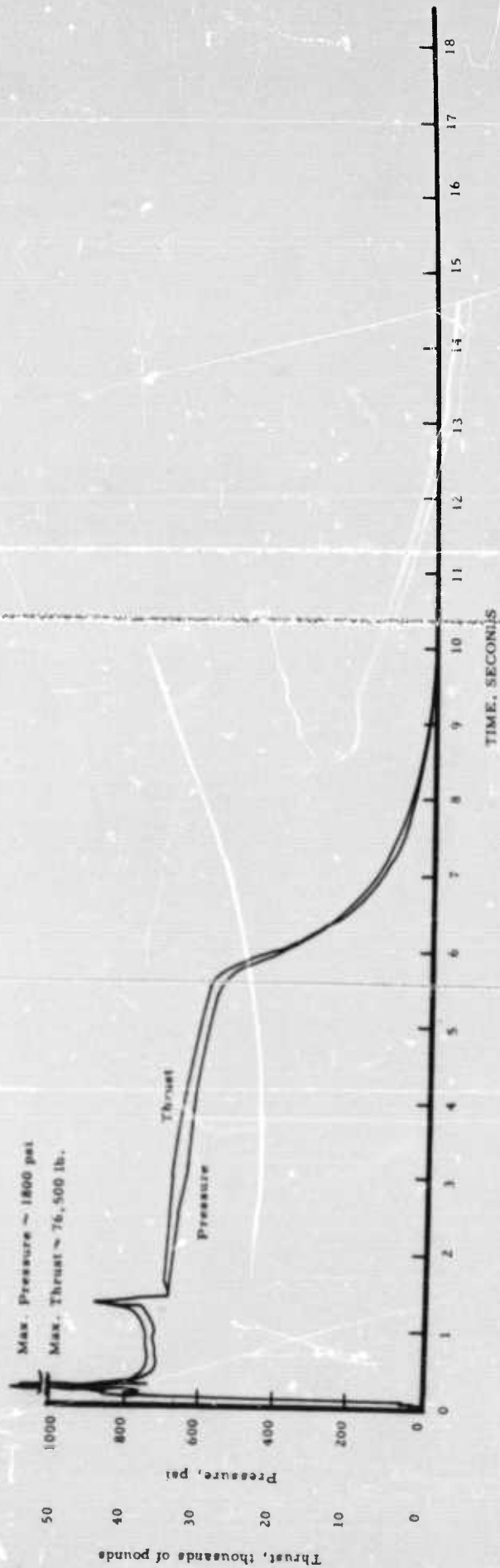


Fig. B-4 Ballistic data for the half-scale Nike-Zeus motor RH No. 7

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occurred at 1.25 sec. The case and nozzle performed well. Both Rohm & Haas Company and ARGMA took pressure and thrust records with excellent agreement.

RH No. 8 - Processing and Low Temperature Firing (Fig. B-5)

The eighth 19 x 75 STM was cast from three batches of QZ_{bn} to develop the multiple batch technique needed for casting the full scale sustainer. A full motor of good quality propellant was obtained. The firing after 48 hours conditioning at +20°F was highly successful. Good impulse and smooth pressure and thrust traces were obtained. No grain cracking or distortion occurred.

RH No. 10 - Ballistics and Processing (Fig. B-6)

This motor was cast to determine the mean K necessary for a 1,000 psi firing as well as for a check on processing techniques, after modifications to the processing building and equipment. The casting was made from two batches, QZ_{bn}, C-28, C-29, and a full length grain was obtained. The firing at ambient conditions was highly successful. The Planeton IV exit cone insulation delaminated during the firing, causing small perturbations in the thrust trace. A lower than expected pressure resulted from using too low a mean K. Good specific impulse was obtained.

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Project No. TUL-3060	RH Project No. 62-24.1	Static Test No. 6	Round No. RH 8	Test Date 2/6/59	STATIC FIRING SUMMARY			
Type of Case: 155 mm. B. A. 7.5	Loaded Wt., lb. 1693	P-actl No. C-18, 19, 20	Composition: QZ ^{Br}		Instrumentation by: Rohm & Haas, ARGMA			
Serial No. 3	Fired Wt., lb. 687	Configuration - AG Star	Grain Wt., lb.	1002	t_{ign} , sec.	C_D , lbm/lbf-sec	6.20×10^{-3}	
	Wt. Consumed, lb. 1006	OD, in.	Grain Length, in.	70.95	t_b , sec.	C_D , lbm/lbf-sec	6.51×10^{-3}	
		ID, in.	Density, lbm/cu.in.	0.065	t_b , sec. (0 thrust)	c^* , ft/sec	4943	
		Web, in.	Temp., °F	420	\dot{r}_b , in./sec.	\dot{m} , lbm/sec.	99.73	
		Port Area, sq. in.	Igniter Type	Jelly Roll	P_{igo} , psi	F_{max} , lbf.	37,095	
		Mean Surface, sq. in.	Igiter Wt., gm.	300	P_{max} , psi	F_{av} , lbf.	22,678	
					P_b , psi	I , lbf-sec.	227,906	
					P_s , psi	\dot{m} , lbf-sec/lbm	227.4	
					$\int P_{\text{dtd}}$, psi-sec	F_{meas} , lbf-sec/lbm	244.3	
					$(A/A_p)^{1/2}$ initial	C_F	1.48	
					K_m	$C_{F_{\text{meas}}}$	1.59	
<p>NOZZLE DATA</p> <p>D_t (av. before) 4.0</p> <p>D_t (av. at 1000 in. 4.28</p> <p>D_e (av. 4.08</p> <p>Exp. Jet Flow</p> <p>Ins. Jet Mat. Graphite</p>								
<p>LINER AND RESTRICTION DATA</p> <p>Lines Comp. 1 R7-73 Restrictor Comp.</p> <p>Lines Wt., lb. 18 Restrictor Wt., lb</p> <p>Lines Thickness, in. 0.19 Restrictor</p>								

Figure 1. The effect of the concentration of the polymer solution on the surface free energy of the polymer film.

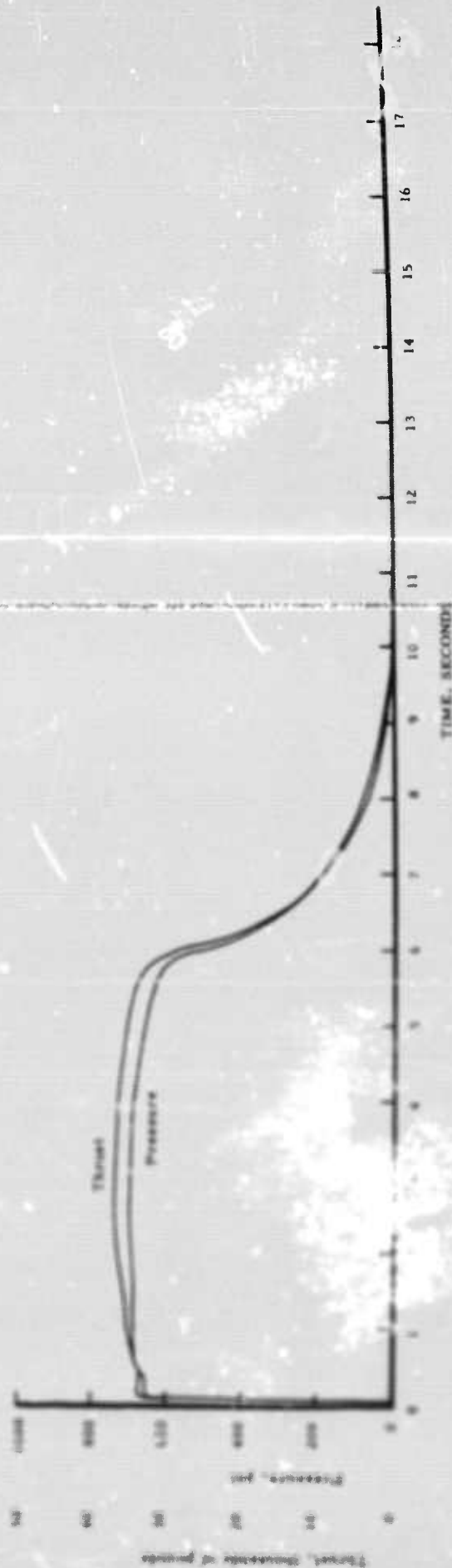


Fig. B-5 Ballistic data for the half-scale Nike-Zeus motor RH No. 8

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Static Firing Summary			
Test No. 6124/56	By	Station & Name, ARMA	
		C_D , lbm/lbf-sec	6.24×10^{-3}
		C_{D0} , lbm/lbf-sec	6.46×10^{-3}
		C_{D0} , lbm/lbf-sec	4982
		C_{D0} , lbm/lbf-sec	110.40
		C_{D0} , lbm/lbf-sec	39,810
		C_{D0} , lbm/lbf-sec	25,631
		C_{D0} , lbm/lbf-sec	238,956
		C_{D0} , lbm/lbf-sec	232.0
		C_{D0} , lbm/lbf-sec	234.3
		C_{D0} , lbm/lbf-sec	1.50
		C_{D0} , lbm/lbf-sec	1.58

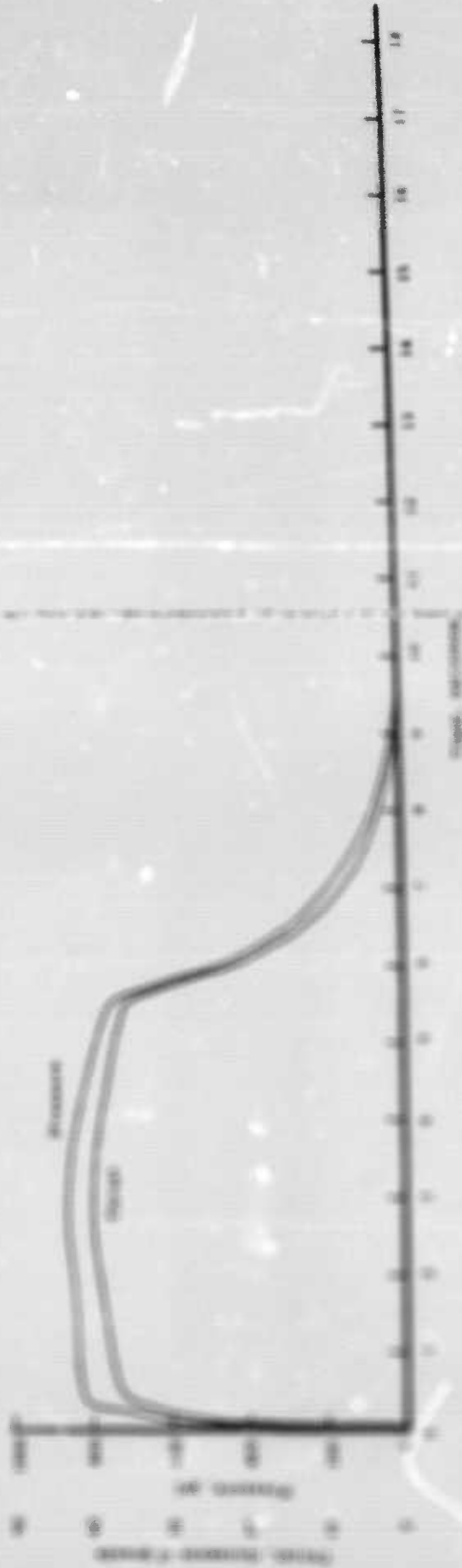


Fig. B-6 Static data for the half-scale Nike-Zeus motor RM No 10

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