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RENOVATED PROGRAM FOR PRODUCTION OF X429 PROPELLANT GRAINS FOR --ETC(U)
SEP 82 H J FRIGAND
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TECHNICAL REPORT ARTSD-TR-82001

**RENOVATED PROGRAM FOR PRODUCTION OF
XM29 PROPELLANT GRAINS FOR SPARROW MISSILE**

HERMAN J. FRIGAND

SEPTEMBER 1982



**US ARMY ARMAMENT RESEARCH AND DEVELOPMENT COMMAND
TECHNICAL SUPPORT DIRECTORATE
DOVER, NEW JERSEY**

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20. ABSTRACT (Continue on reverse side if necessary and identify by block number) In the past, serious problems had arisen relevant to the fast burning characteristics of XM29 propellant grains produced for the Sparrow missile. An exploratory program was therefore conducted to find ways and means of producing slower burning grains which complied with specification requirements of 40 seconds minimum burning time. A renovated program was developed which encompassed the strict surveillance of raw materials and operating procedures in order to assure the production of grains with acceptable ballistic properties.		

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INTRODUCTION

The Sparrow missile, a U.S. Navy item (air-to-air), employs an electrical power unit (EPU) to supply the required energy for the operation of the missile's guidance system. An XM29 propellant grain supplies the energy to operate the EPU. The Sparrow is a single-stage missile, powered by a solid-propellant rocket motor. It is steered by midbody wings and homes onto radar energy which is emitted by the launch aircraft and then reflected back from the target.

During the period from 1979 to early 1980, orders were received at ARRADCOM from the Navy Ships Parts Control Center, Mechanicsburg, Pennsylvania, for a total of 804 propellant grains for this missile. Initial work involved the production of pilot lots prior to full production runs. Sample grains from pilot lots (and later from production lots) were forwarded to the Raytheon Manufacturing Company, Lowell, Massachusetts, for machining and encapsulation. Pilot lot grains were then shipped to the Naval Ordnance Station (NOS), Indian Head, Maryland, for ballistic testing.

In June 1979 at a meeting held at NOS, propellant grain problems were addressed by personnel from ARRADCOM, NOS, and the Navy Pacific Missile Test Center, Point Mugu, California. Two lots of propellant grains had failed ballistic requirements. The groundwork for corrective action was identified at this meeting.

A proposal for a renovated program was introduced by ARRADCOM and accepted at a second meeting held at the Naval Air System Command, Arlington, Virginia, in August 1979, attended by ARRADCOM and Naval personnel.

Two pilot lots (12 grains each) and a production lot (master blend consisting of 880 grains) were later successfully manufactured.

DISCUSSION

The two lots that failed ballistics did not comply with the required burn time of 40 seconds minimum of the Raytheon specification when tested at 89°C (192°F) (ref 1). The lots, AL-20-7 and AL-11-9, were eventually accepted on waiver. Ballistic test results for these lots are shown in table 1. Propellant grains from lot AL-20-7 failed to meet the required burn time by 2.17 and 1.27 seconds below the required 40 second minimum, and grains from lot AL-11-9 failed by 2.55 seconds and 1.50 seconds.

NOS surveillance data (figs. 1 through 4) indicate ageing trends in burning time. For every 3.3 years, there is a decrease in burn time of 1.2 seconds and an increase in maximum pressure of 172 kPa (25 psi) at 89°C. At cold temperatures the grains are differently affected: At -37°C (-35°F) the decrease in burn time for every 3.3 years is 0.18 seconds and the increase in maximum pressure is 117 kPa (17 psi).

WORK PLAN

The work plan entailed (1) the procurement and complete surveillance of new raw materials, (2) the preparation of pilot lots with varied amounts of carbon black in the formula to produce slower burning grains, (3) the implementation of and adherence to established operating procedures, (4) a review of factors for correlation between locally produced strand burning rate data and ballistics for lots under test.

OPERATING PROCEDURES

Handling and Assay of Raw Materials

All of the raw materials used were assayed for compliance with specification requirements (refs 1 through 12) (table 2). The fine HMX (class E) is alcohol washed and dried to a total moisture content of 0.1% maximum. The nitrocellulose is dehydrated, block-broken, and screened (4 mesh screen). Nitroglycerin is added to triacetin in a bubbling vessel prior to transfer to a special container.

Specifications

In addition to the description of manufacture (ref 13), an in-house quality evaluation plan was followed for the pilot lots and the production lot. The grains were 100% inspected as follows:

1. Grain dimensions:

- a. Outside diameter - 37.5 ± 0.13 mm (1.475 ± 0.005 in.)
- b. Length - 308 ± 1.3 mm (12.125 ± 0.050 in.)

c. Straightness - Each accepted grain was passed freely (without encumbrances) through a Rollorama gage for the entire length of the grain. [The gage consisted of two parallel metallic surfaces, 37.92 to 37.97 mm (1.493 to 1.495 in.) apart, which were lying on a horizontal plane.] Grains were supported for the entire length during inspection and handling.

2. Radiographic examination: A standard test grain was prepared that contained two holes [0.79 mm (1/32 in.) in diameter] for comparison with the test grains during radiographic examination. The test grains were x-rayed at 0° and 90° positions. The grains were inspected to assure that they were free of fissures, were free of foreign material greater than 0.79 mm in any direction, and did not contain more than five foreign particles regardless of size.

3. Visual examination: Lateral and end surfaces of each grain were inspected to assure that they were reasonably free of tool marks and that they

appeared smooth to the naked eye. Grains were also inspected to assure they did not show any grease, smears, or oil spots.

Preparation of Pilot Lots

A single pilot lot was prepared first [lot RDD80K000E070 (IB-8950)]. Two 22.7 kg (50 lb) mixers were used for the lot and the ingredients were mixed in a 0.08 m³ (20 gal.) mixer. Because of an inoperative brine system, the mixer jacket was cooled by means of two 0.21 m³ (55 gal.) drums fitted with 13 mm (0.5 in.) copper tubing and filled with sodium chloride and ice. This improvised unit was hooked up to the circulatory lines leading to the mixer. Makeup solutions were added to the drums as required.

The mix sequence is shown below. When properly mixed and ready for extrusion, the colloid mix was sandy and loose (no lumps and not sticky in appearance).

<u>Order of addition of ingredients to mixer</u>	<u>Mixer action</u>	<u>Mixing time</u>
Nitrocellulose Carbon black N-methyl-p-nitroaniline Ethyl centralite in alcohol solution	Mix	1 hr
Lead stearate	Mix	30 min
Sucrose-octa-acetate	Mix	10 min
HMX in alcohol solution (1 increment)	Agitate	10 min
Nitroglycerin-triacetin-ether solution (via a nitroglycerin distributor)	Blend Mix	30 min 1 hr minimum (longer if required)
Solvents as required to provide a well-colloided and workable mix	---	---

The colloided mix (solvent wet) was then transferred to a 102 mm (4 in.) Logan press, where it was screened through 12 and 24 mesh screens at pressures between 4827 and 5516 kPa (700 and 800 psi). The resulting screened material was placed into a Logan extruder equipped with three dies, 3 mm (0.117 in.) in diameter, and 12 and 24 mesh screens. The screened material was then extruded as strands (three strands per press) into containers for transfer to the McKiernan-Terry cutter operation. The strands were cut into 3 mm (0.120 in.) long granules.

The granules were spread onto trays in preparation for drying. Each mix was identified as to ingredients and was kept separate. The granules were washed with water and covered with a cotton-canvas cloth. After remaining at ambient temperature [about 21°C (70°F)] for 3 days, the granules were forced-air dried for 3 days at temperatures of 40° to 48°C (104° to 118.4°F) to reduce the total volatiles content to 0.4% maximum.

The mix was then blended in a Sweetie Barrel for 20 minutes. The blended granules were then screened by hand to achieve optimum granulation and to remove possible contaminants.

Burning-rate samples were prepared in a 51 mm (2 in.) Logan press. The extruded strand, with a diameter of 3 mm (0.125 in.) was cut to a length of 181 mm (7.125 in.). A total of 30 strands were used for the burning rate test (ref 14). Strand burning rate data (table 3) were received and plotted on log-log graph paper (table 4, fig. 5) to determine if the burning rate characteristics of the propellant were satisfactory. A test was also conducted for heat of explosion (ref 14). Twelve acceptable propellant grains were then manufactured for ballistic testing (in accordance with the description of manufacture) using the 286 mm (11.25 in.) solventless extrusion press. Prior to the extrusion, the granules are preconditioned at a temperature of 71°C (160°F). This press accepted dried and blended granules as press feed (see operating parameters below). Approximately 15.9 kg (35 lb) of press feed was introduced into the basket of the press. Then, upon the attainment of proper vacuum of mercury [711 mm (28 in.)], the ram moved forward and became operable.

The extruded strand was cut at the exit end of the die by the flying cutter to rough lengths of 330 mm (13 in.). This operation was viewed from the control room via closed circuit television. Cut strands (grains) were identified with the extrusion number and strand number prior to their transfer to flat-bottomed tote boxes for subsequent annealing.

The grains were annealed in a dry house for a minimum of 8 hours at 50°C (122°F). After being cooled at 35°C (95°F), the grains were x-rayed in the 0° position for compliance with the quality evaluation plan. Acceptable grains were then conditioned for 24 hours at 18° to 24°C (65° to 75°F) prior to lathework at similar temperatures. Each grain was faced off at both ends to a final length of 308 ± 1.3 mm (12.125 ± 0.050 in.) and was turned to an outside diameter of 37.5 ± 0.13 mm (1.475 ± 0.005 in.). A tungsten carbide-tipped tool bit was used on the lathe.

Acceptable grains were x-rayed at 0° and 90° positions for compliance with the quality evaluation plan. Grains were then passed through the Rollorama gage and visually examined in accordance with the quality evaluation plan.

These samples were forwarded to Raytheon for finishing operations and later sent to NOS for ballistic test work. Acceptable ballistic results were received from NOS (table 1).

A second pilot lot [RDD80K000E071(1B-8951)] of 12 grains was prepared in the same manner as the first one and was tested by NOS for ballistics (table 1, fig. 6). Acceptable firing values were received.

Based on ballistic data evaluation, the first pilot lot was chosen for the production run.

Preparation of Production Lot

The production lot was produced in the same manner as the pilot lots and subjected to similar testing (ref 13, table 3, figs. 7 through 13). In addition, a complete chemical assay (representative of a composite sample of a master blend identified as Lot RDD81F000E094) and a stability assay (ref 14) were conducted for the lot (tables 5 and 6). For the production lot the exceptions were as follows:

1. A total of six mixes were used.
2. The mix size was 136.4 kg (300 lb).
3. The mixer size was 0.38 m^3 (100 gal.).
4. The HMX in alcohol solution was added in four increments and the mix was agitated for 10 minutes after each increment.
5. A master blend was prepared constituting the six mixes.

Packing of Production Lot

Grains which complied with the specifications and quality evaluation plan were packed out as follows: Each grain was wrapped in single-faced corrugated fiberboard, with tape positioned in the middle and at both ends of the grain. Fifty grains were placed in a carton in five layers (10 grains per layer). Each layer was separated by a filler. The carton was then sealed and overwrapped with barrier material and Kraft paper. The carton was then placed in a wooden packing box fitted with filler material at the ends, sides, and top. The packing box, with wooden cover in place, was strapped with two thick steel straps and identified for shipment.

Shipment of Production Lot

In September 1981 a total of 804 grains were shipped to Raytheon, which shipment satisfied ARRADCOM's commitment. However, when the production run was completely finished, 76 more acceptable grains had been manufactured, and these additional grains were shipped to Raytheon in October 1981. Therefore a grand total of 880 grains were shipped.

OPERATING PARAMETERS

In order to strictly control the process and to minimize operating variables for the pilot lots and the production lot, the following parameters were used in most instances in the manufacture of the grains:

Mixer size:

Pilot lot - 0.08 m^3 (20 gal.)
Production lot - 0.38 m^3 (100 gal.)

Mix size:

Pilot lot - 22.7 kg (50 lb)
Production lot - 136.4 kg (300 lb)

Total mix time: 4 hr

Mix temperature: 8° to 9°C (46° to 49°F)

Extrusion - solvent [102 mm press (4 in. press)]

Die size - 2.97 mm (0.117 in.)
No. of dies - 3
Type of die - AB

McKiernan-Terry cutter

No. of knives - 28
Teeth:

A-gear - 71
B-gear - 180
C-gear - 160
D-gear - 80

Front, center, and back plate hole diameter - 4.1 mm (0.160 in.)
Length of cut - 3 mm (0.120 in.)

Forced-air dry:

Cycle:

Preliminary - 3 days minimum
Final - 3 days minimum

Temperature:

Preliminary - approx 21°C (70°F)
Final - 45°C (113°F)

Extrusion - solventless [286 mm press (11.25 in. press)]

Die size - 39.4 mm (1.550 in.)
No. of dies - 1
Propellant, die, and basket temperature - 70° to 72.2°C (158° to 162°F)
Ram rate - 12.7 mm (0.5 in.) per minute

Cutter - flying

Length of cut - 330 mm (13.0 in.)

Annealing (dry house)

Cycle - 8 hr
Temperature - 50°C (122°F)

COMPARISON OF ARRADCOM AND NOS DATA

An attempt was made to arrive at a correlation factor between ARRADCOM strand burning rate data and NOS grain firings at equivalent pressures (table 7). Higher burn values were indicated for NOS firings at hot temperatures when compared to ARRADCOM burn values, and vice versa at cold temperatures. However, data were limited and therefore no conclusions could be drawn at that time.

CONCLUSIONS

1. Acceptable XM29 propellant grains for the Sparrow missile can be produced with adoption of the following conditions:

a. Strict surveillance of raw materials, including control of carbon black content.

b. Strict process controls, especially as related to the temperatures of the colloid mix and the conditions of extrusion.

c. Use of forced-air drying.

d. Release of production grains based strictly on actual firing results of pilot lot test samples.

2. Through future work it may be possible for a correlation factor to be established between the results of the strand burning rate tests and the results of ballistic testing of the grains.

3. The establishment of a correlation factor between the results of the strand burning rate tests and the results of ballistic testing of the grains would provide economic advantages, because ballistic testing could then be deleted as part of the acceptance process.

RECOMMENDATIONS

1. XM29 propellant grains for the Sparrow missile should be released for use only under the following conditions:

a. Strict surveillance of raw materials, including control of carbon black content.

b. Strict process controls, especially as related to the temperatures of the colloid mix and the conditions of extrusion.

c. Use of forced-air drying.

d. Release of production grains based strictly on actual firing results of pilot lot test samples.

2. A cost feasibility analysis should be conducted, on a lot-to-lot basis, involving the burning rate data procured from ballistic firings versus strand burning rates for each lot of propellant grains for the purpose of establishing a correlation factor. The establishment of a correlation factor could obviate the need for ballistic testing and could result in huge savings.

REFERENCES

1. Purchase Specification, Raytheon Manufacturing Co. Lowell, MA, Solid Propellant, Cylindrical Billet, 399MR029P001, approved 31 May 1961.
2. Military Specification, Nitrocellulose, MIL-N-244A, dated 13 February 1962.
3. Military Specification, Nitroglycerin, MIL-N-246-B, dated 19 February 1962 (Manufactured by the Biazzi process under carefully controlled conditions.).
4. Military Specification, HMX, MIL-H-45444B, (AR), Amendment 3, dated 1 May 1978.
5. Military Specification, Triacetin (Glyceryl Triacetate), MIL-T-301A, dated 5 December 1961.
6. Military Specification, Ethyl Centralite (carbamate), MIL-E-255A, Amendment 1, dated 8 June 1966.
7. Purchase Description, N-Methyl-P-Nitroaniline, PA-PD-450, dated 26 January 1956 (declared obsolete pending the promulgation of a MIL specification).
8. Military Specification, Lead Stearate, MIL-L-758A, dated 25 May 1962.
9. Military Specification, Carbon Black, Powdered, Dry, MIL-C-306C, dated 4 August 1978 (use of a heat absorbing grade to promote ignition).
10. Military Specification, Ether, Diethyl Technical, MIL-E-199A, Amendment 3, dated 15 June 1974.
11. Military Specification, Ethyl Alcohol (For Ordnance Use), MIL-E-463B, dated 14 May 1962.
12. Military Specification, Sucrose Octa Acetate, MIL-S-23117, dated 23 February 1962 (canceled as of 25 January 1980).
13. Description of Manufacture, Electric Power Unit Propelling Charge, XM29 Propellant, File No. 35-3-122, ARRADCOM, 1961.
14. Military Standard, Propellants, Solid Sampling, Examination and Testing, MIL-STD-286B, dated 1 December 1967.

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Table 1. Ballistic test data

Grain no.	Igniter		Propellant				Conditioning		Nozzle size	
	Peak pressure kPa	Time to peak (sec)	Max pressure kPa	Min pressure kPa	Burn time (sec)	psi	°C	°F	mm	inch
10-60B	3413	0.320	3227	468	43.74	400	-37	-35	1.7323	0.0682
16-60B	3379	0.282	3213	466	43.72	389	-37	-35	1.7323	0.0682
8-60B	5075	0.178	3599	522	38.73*	436	89	192	1.7247	0.0679
1-60B	6426	0.152	3827	555	37.83*	442	89	192	1.7247	0.0679
2-A	3392	0.252	3103	450	44.02	404	-37	-35	1.702	0.067
3-A	3434	0.275	3165	459	44.03	397	-37	-35	1.676	0.066
4-A	3434	0.292	3185	462	44.12	408	-37	-35	1.702	0.067
7-B	3392	0.216	3165	459	43.90	408	-37	-35	1.727	0.068
8-B	3427	0.345	3185	462	43.18	416	-37	-35	1.702	0.067
9-B	3558	0.199	3213	466	43.53	400	-37	-35	1.727	0.068
10-B	3710	0.329	3448	500	43.72	410	-37	-35	1.727	0.068
1-A	3358	0.406	3185	462	43.94	411	-37	-35	1.702	0.067
1-B	7005	0.171	3572	518	38.13*	426	89	192	1.702	0.067
2-B	6778	0.149	3785	549	38.32*	444	89	192	1.702	0.067
3-B	9363	0.096	3710	538	38.22*	436	89	192	1.727	0.068
4-B	6640	0.165	3758	545	38.38*	442	89	192	1.727	0.068

* Failed Raytheon spec S399MR029 P001 (solid propellant); burning time - 40 seconds minimum.

Table 1. (cont)

Grain no.	Igniter		Propellant				Conditioning		Nozzle size	
	Peak pressure kPa	Time to peak (sec)	Max pressure kPa	Min pressure kPa	psi	psi	°C	°F	mm	inch
7-A	8633	0.121	3806	3075	552	446	89	192	1.727	0.068
8-A	7129	0.144	3820	3123	554	453	89	192	1.702	0.067
9-A	8881	0.111	3834	3130	556	454	89	192	1.727	0.068
10-A	8508	0.104	3792	3075	550	446	89	192	1.727	0.068
FIRST PILOT LOT (RDD80K000E070)										
1	2841	0.270	2689	2137	390	310	-37	-35	1.75	0.069
2	2799	0.282	2668	2151	387	312	-37	-35	1.75	0.069
3	3089	0.116	2744	2248	398	326	-37	-35	1.73	0.068
4	2923	0.229	2744	2206	398	320	-37	-35	1.73	0.068
5	2717	0.756	2703	2220	392	322	-37	-35	1.73	0.068
6	2937	0.183	2751	2275	399	330	-37	-35	1.73	0.068
7	6137	0.129	3144	2510	456	364	89	192	1.73	0.068
8	6054	0.158	3061	2475	444	359	89	192	1.73	0.068
9	7653	0.158	3137	2475	455	359	89	192	1.73	0.068
10	6840	0.138	3075	2524	446	366	89	192	1.70	0.067
11	5833	0.195	3130	2517	454	365	89	192	1.70	0.067
12	6454	0.145	3096	2455	449	356	89	192	1.73	0.068

Table 1. (cont)

Grain no.	Igniter		Propellant				Conditioning		Nozzle size	
	Peak pressure kPa	Time to peak (sec)	Max pressure kPa	Min pressure kPa	Max pressure psi	Min pressure psi	°C	°F	mm	inch
SECOND PILOT LOT (RDD80K000E071)										
1 1A	2965	0.206	2710	2096	393	304	-37	-35	1.67	0.0659
2 3A	3972	0.066	2703	2144	392	311	-37	-35	1.67	0.0657
3 5A	2992	0.214	2744	2158	398	313	-37	-35	1.74	0.0687
4 2B	3034	0.179	2689	2193	390	318	-37	-35	1.69	0.0667
5 4B	2951	0.211	2661	2144	386	311	-37	-35	1.70	0.0670
6 7B	3020	0.239	2772	2124	402	308	-37	-35	1.71	0.0672
7 2A	6785	0.138	2999	2448	435	355	89	192	1.71	0.0673
8 4A	7605	0.099	2951	2427	428	352	89	192	1.7	0.0675
9 7A	5578	0.180	3137	2468	455	358	89	192	1.71	0.0675
10 1B	6840	0.119	3041	2462	441	357	89	192	1.72	0.0676
11 3B	5923	0.154	3048	2455	442	356	89	192	1.72	0.0676
12 5B	5447	0.172	2786	2399	404	348	89	192	1.74	0.0685

Table 2. Analytical data for raw materials

Raw material	Vendor analysis		ARRADCOM analysis	
	Characteristic	Value	Characteristic	Value
Nitrocellulose	Lot RAD80A001-003:*		(See footnote below)	
	Nitrogen content, %	12.54	Total volatile material, %	22.97
	Ether/alcohol solubility, %	99+	water, %	2.37
	Acetone insolubles, %	Trace	alcohol, %	20.60
	Ash, %	0.04	Ash content, %	0.10
	Viscosity, seconds	10	Acetone insoluble, %	0.19
	Fineness, milliliters	96	Nitrogen, %	12.57
	65.5°C heat test with KI	45+	134.5°C heat test, minutes	35
	starch paper, minutes		65.5°C heat test, minutes	60+
	134.5°C heat test, minutes	30+		
	Lot 2217L:*			
	Nitrogen, %	12.53		
	Solubility			
	Grades A, D, E (%)	99+		
	Acetone insoluble, %	Trace		
RMX	Ash, %	0.02	Melting point, °C	278.0
	134.5°C heat test, minutes	30	Acetone insoluble, %	0.05
	Solubility		Inorganic insoluble, %	0.01
	Grades B, C (%)	—	Insoluble particles	
	Viscosity, seconds	8	retained on no. 40 sieve	none
	Fineness, milliliters	98	retained on no. 60 sieve	none
	% moisture	29.9	Acidity, %	0.002
			Granulation	
			through no. 325 sieve, %	99.8
			RDX, %	< 0.02

* Vendor lots blended together at ARRADCOM in the ratio of 1125 lb for lot RAD80A001-003 and 300 lb for lot 2217L.

Table 2. (cont)

Raw material	Vendor analysis		ARRADCOM analysis	
	Characteristic	Value	Characteristic	Value
Triacetin	--		Color	Satisfactory
			Specific gravity, 25°C	1.153
Sucrose octaacetate	Description	White, burned sugar odor	Acidity	0.000
	Solubility	Haze in CHCl ₃ and in ether	Ash content, %	0.001
	Melting range, °C	85.5 (does not all melt)	Ester content, %	99.85
	Acidity	Less than 0.03% HAc	Purity, %	99.3
	Water	0.053%	Free acidity, %	0.007
	Identification	OK	Melting point, °C	85.5
	Residue on ignition	0.23%	Insoluble in alcohol, %	0.00
	Insoluble matter	OK	Specific gravity, 20°C/20°C	1.275
	Color	OK		
	Chemical marketing color test	OK		
Ethyl centralite	Assay	100.18%	Solidification point, °C	72.0
	Solidification point (801013-2)	72.1°C	Melted material	Satisfactory
	Volatile matter (801013-1)	0.13%	Volatile content, %	0.00
	Secondary amines (801014-1)	0.15%	Ash content, %	0.02
	Tertiary amines (800926-2)	<0.02%	Secondary amines, %	0.06
	Hydrolyzable chlorides (800926-1)	<0.001%	Acidity, %	0.00
	Acidity (800925-1)	0.0015%	Hydrolyzable chlorine compounds, %	<0.001
	Ash (800929-1)	0.023%	Particle form through no. 30	100
	Screen test	99.9%	STD sieve, %	

Table 2. (cont)

Raw material	Vendor analysis		ARRADCOM analysis	
	Characteristic	Value	Characteristic	Value
N-methyl-p-nitroaniline	Color/acetone solution	Lighter than standard	Purity, %	99.3
	Purity/nitrite	99.3 PCT	Moisture, %	0.00
	DMNA/HPLC	0.3 PCT	Acidity, mg KOH/g	0.014
	PNCB/HPLC	0	Acetone insolubles, %	0.04
	Moisture/K.F.	0.27 PCT	Freezing point (melting point), °C	151.6
	Acidity/Mg. KOH/G	0.064 PCT		
	Acetone insolubles	0.021 PCT		
	Iron	0.0003 PCT		
	Chloride	0.0002 PCT		
	Melting point	153.5°C		
Lead stearate	---	---	Moisture, %	0.12
			Water - soluble material, %	0.16
			Acidity to phenolphthalein to methyl orange, %	0.003
			Alkalinity	0.000
			Purity:	0.000
			Lead content, %	28.2
			Stearic acid, %	73.1
			Melting point of lead stearate, °C	107.5
			Melting point of fatty acid, °C	60.5
			Iodine number of fatty acid Granulation	4.5
			Retained on no. 100 sieve, %	5.0
			Retained on no. 200 sieve, %	31.0
			Retained on no. 325 sieve, %	36.0

Table 2. (cont)

Raw material	Vendor analysis		ARRADCOM analysis	
	Characteristic	Value	Characteristic	Value
Carbon black	Iodine adsorption ng., mg/g	9.4	pH of water solution	7.0
	BET surface area, m ² /g	8.4	Pour density, lb/ft ³	16.8
	Electron microscope surface area, m ² /g	8.5	Bulk density, gm/cc	0.27
	DBP absorption no., cc/100 g	43	Heat loss at 105°C, %	0.03
	Pour density, lb/ft ³	41.4	Water insoluble material, %	0.09
	Discoloration of benzene	1	Ash, %	0.12
	pH	10.1	Acetone extractable material, %	0.21
	Volatile content, %	0.09	Iodine absorption no.	30.04
	Ash content, %	0.09	Discoloration of orthodi-chlorobenzene transmittance at 460 nm, %	43.7
	Heat loss at 105°C (ASTM), %	0.015		
	Mass pellet strength, lb	<10		
	Pines, 5', %	12.4	Coarse particles retained on no. 325 sieve	none
	20', %	13.2		
	Sieve residue, % - US #325 mesh	0.0059		
	Sulfur, %	0.15		
Diethyl ether	Acetone extract	0.40	Specific gravity at 20/20	0.721
	Carbon, %	98.0	Non-volatile residue, %	0.007
			Acidity, %	0.001
			Acetylene, %	0.000
			Peroxides, %	0.000
Ethyl alcohol, grade 2			Chlorides, %	0.000
			Aldehydes, %	0.000
			Ethyl alcohol, %	95.58
			Benzene, %	<0.75
			Acidity, %	0.000
			Non-volatile matter, %	0.002

Table 3. ARRADCOM strand burning rate data ^{a,b}

<u>Lot</u>	<u>Temp °C</u>	<u>Pressure</u>		<u>Burning rate</u>	
		<u>psi</u>	<u>kPa</u>	<u>mm/sec</u>	<u>In./s</u>
FIRST PILOT LOT (RDD80K000E070)					
IB-8950 (RDD80K000E070)	99 (210°F)	200	1379	2.77	0.109
		300	2069	3.00	0.118
		400	2758	3.05	0.120
		500	3448	3.56	0.140
		750	5171	4.09	0.161
	-31.7 (-25°F)	200	1379	2.36	0.093
		300	2069	2.46	0.097
		400	2758	2.54	0.100
		500	3448	2.34	0.092
		750	5171	2.67	0.105
SECOND PILOT LOT (RDD80K000E071)					
IB-8951 (RDD80K000E071)	99 (210°F)	200	1379	2.87	0.113
		300	2069	2.79	0.110
		400	2758	3.00	0.118
		500	3448	3.40	0.134
		750	5171	4.39	0.173
	-31.7 (-25°F)	200	1379	2.16	0.085
		300	2069	2.44	0.096
		400	2758	2.31	0.091
		500	3448	2.21	0.087
		750	5171	2.77	0.109

^a The burning rate strands are inhibited with a bituminous compound (solvent type, black) that furthers a cigarette-type burning. Each strand is subjected to individual dip coats of paint, followed by an air-dry cure per dip. Burning is accomplished in the Crawford bomb. The procedure was conducted in accordance with reference 14, method T803.1.

^b The burning rate data are the average of duplicate tests per mix determined at the noted temperatures and pressures.

Table 3. (cont)

Lot	Temp °C	Pressure		Burning rate	
		psi	kPa	mm/sec	In./s
PRODUCTION LOT (SIX INDIVIDUAL MIXES) IB-8952 (RDD81F000E094)					
IB-8952 (Representative of lot RDD80K000E070)					
Mix 1	99 (210°F)	200	1379	2.54	0.100
		300	2069	2.77	0.109
		400	2758	2.57	0.101
		500	3448	2.77	0.109
		750	5171	3.86	0.152
	-31.7 (-25°F)	200	1379	2.18	0.086
		300	2069	2.41	0.095
		400	2758	2.29	0.090
		500	3448	1.91	0.075
		750	5171	2.54	0.100
Mix 2	99 (210°F)	200	1379	2.62	0.103
		300	2069	2.67	0.105
		400	2758	2.59	0.102
		500	3448	2.87	0.113
		750	5171	3.84	0.151
	-31.7 (-25°F)	200	1379	2.29	0.090
		300	2069	2.46	0.097
		400	2758	2.39	0.094
		500	3448	2.13	0.084
		750	5171	2.54	0.100
Mix 3	99 (210°F)	200	1379	2.59	0.102
		300	2069	2.72	0.107
		400	2758	2.77	0.109
		500	3448	2.92	0.115
		750	5171	3.81	0.150
	-31.7 (-25°F)	200	1379	2.34	0.092
		300	2069	2.44	0.096
		400	2758	2.57	0.101
		500	3448	2.29	0.090
		750	5171	2.57	0.101
Mix 4	99 (210°F)	200	1379	2.62	0.103
		300	2069	2.77	0.109
		400	2758	2.79	0.110
		500	3448	2.79	0.110
		750	5171	3.76	0.148

Table 3. (cont)

Lot	Temp °C	Pressure		Burning rate	
		psi	kPa	mm/sec	In./s
Mix 4 (cont)	-31.7 (-25°F)	200	1379	2.21	0.087
		300	2069	2.34	0.092
		400	2758	2.41	0.095
		500	3448	2.03	0.080
		750	5171	2.57	0.101
Mix 5	99 (210°F)	200	1379	2.51	0.099
		300	2069	2.64	0.104
		400	2758	2.97	0.117
		500	3448	2.97	0.117
		750	5171	3.99	0.157
	-31.7 (-25°F)	200	1379	2.24	0.088
		300	2069	2.44	0.096
		400	2758	2.46	0.097
		500	3448	2.21	0.087
		750	5171	2.54	0.100
Mix 6	99 (210°F)	200	1379	2.64	0.104
		300	2069	2.69	0.106
		400	2758	2.77	0.109
		500	3448	2.90	0.114
		750	5171	3.99	0.157
	-31.7 (-25°F)	200	1379	2.21	0.087
		300	2069	2.36	0.093
		400	2758	2.36	0.093
		500	3448	2.06	0.081
		750	5171	2.49	0.098
RDD81F000E094 (master blend, composite of mixes 1 through 6)	99 (210°F)	200	1379	2.72	0.107
		300	2069	2.77	0.109
		400	2758	2.77	0.109
		500	3448	3.20	0.126
		750	5171	3.86	0.152
	-31.7 (-25°F)	200	1379	2.29	0.090
		300	2069	2.44	0.096
		400	2758	2.44	0.096
		500	3448	2.18	0.086
		750	5171	2.54	0.100

Table 4. Key to figures 5 through 13

The burn time requirements of Raytheon spec 399MR029P001 shall be as follows:

A minimum burn time of 40 seconds per grain or 3.53 mm/sec (0.139 in./s) maximum.

The data obtained were determined at pressures of 1379, 2069, 2758, 3448, and 5171 kPa (200, 300, 400, 500, and 750 psi) at -31.7°C (-25°F) and 99°C (210°F).

The burning rate figures were plotted on log log graph paper where the burning rate-pressure points are joined by a straight line for each temperature. A 45° line intersects the point of 1895 kPa (275) psi and 0.1 in./s. This represents a constant P/r comparable to 2750 which is equal to a ratio used in motor test firings.

The burning rate values are then drawn and established at the points where the 45° line intersects the -31.7°C and 99°C isotherms.

kp, $^{\circ}\text{F}$, represents the temperature coefficient (a value of 0.14%/°F or less is desirable for optimum ballistics).

The seconds/grain is derived by dividing the in./s into the finished length of the billet (5.56 in.).

Table 5. Chemical assay for production lot IB-8952 (RDD81F000E094)

<u>Ingredients</u>	<u>Percent nominal in formula</u>	<u>Percent determined by assay</u>	<u>Method</u>
Nitrocellulose (12.6%N)	38.0	37.60	AL-P-164-62
Nitroglycerin	16.5	16.31	AL-P-164-62
HMX	25.0	24.44	AL-P-164-62
Sucrose octaacetate	7.8	7.61	AL-P-164-62
Triacetin	7.7	8.77	AL-P-164-62
Ethyl centralite	1.0	1.00	AL-P-110-63
N-methyl-p-nitroaniline	1.0	0.98	AL-P-164A-62
Lead stearate	3.0	3.29	AL-P-164-62
Total		100.00	
Carbon black (added) ^a	varies	0.09	MIL-STD-286B, 309.1.2
Total volatiles	--	0.15 ^b	MIL-STD-286B, 103.3.3
		<u>Value determined by assay</u>	
Specific gravity at 15.6°C/15.6°C	--	1.593	MIL-STD-286B, 510.1.1

^a On a carbon black plus total volatiles-free basis

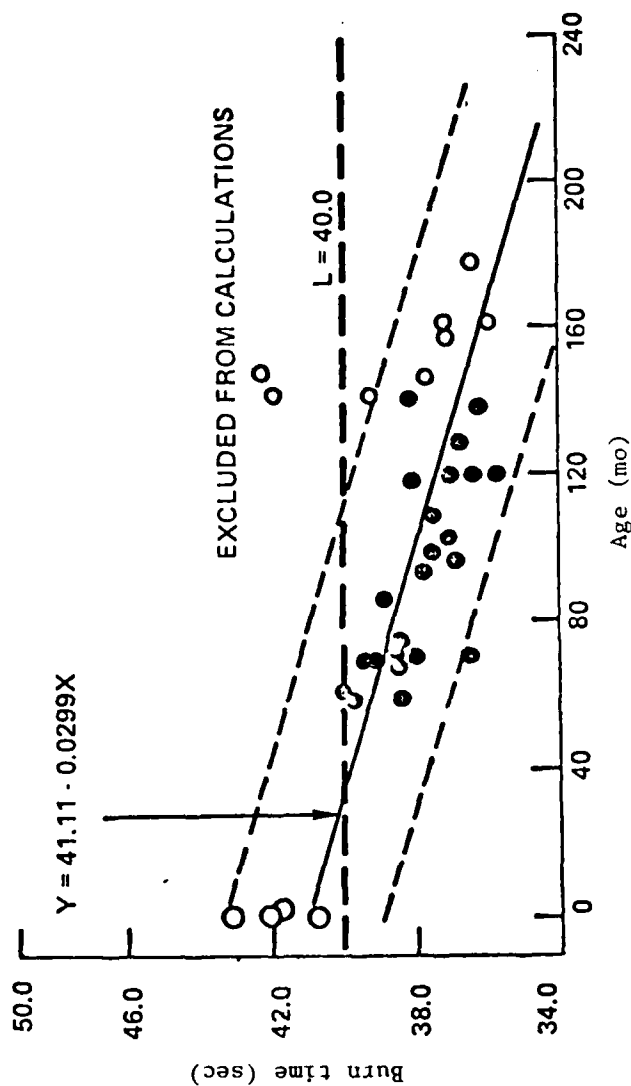
^b Average of values for six mixes: 0.14, 0.14, 0.08, 0.15, 0.18, and 0.21

Table 6. Stability assay for production lot IB-8952 (RDD81F000E094)

<u>Test</u>	<u>Results</u>	<u>Method</u>
Vacuum stability at 90°C (194°F) (mL gas)	0.98	MIL-STD-286B (403.1.2)
Heat test at 120°C (248°F)		MIL-STD-286B (404.1.2)
Time to explosion (min)	300+	
Time to salmon pink (min)	220	
Red Fumes	None	

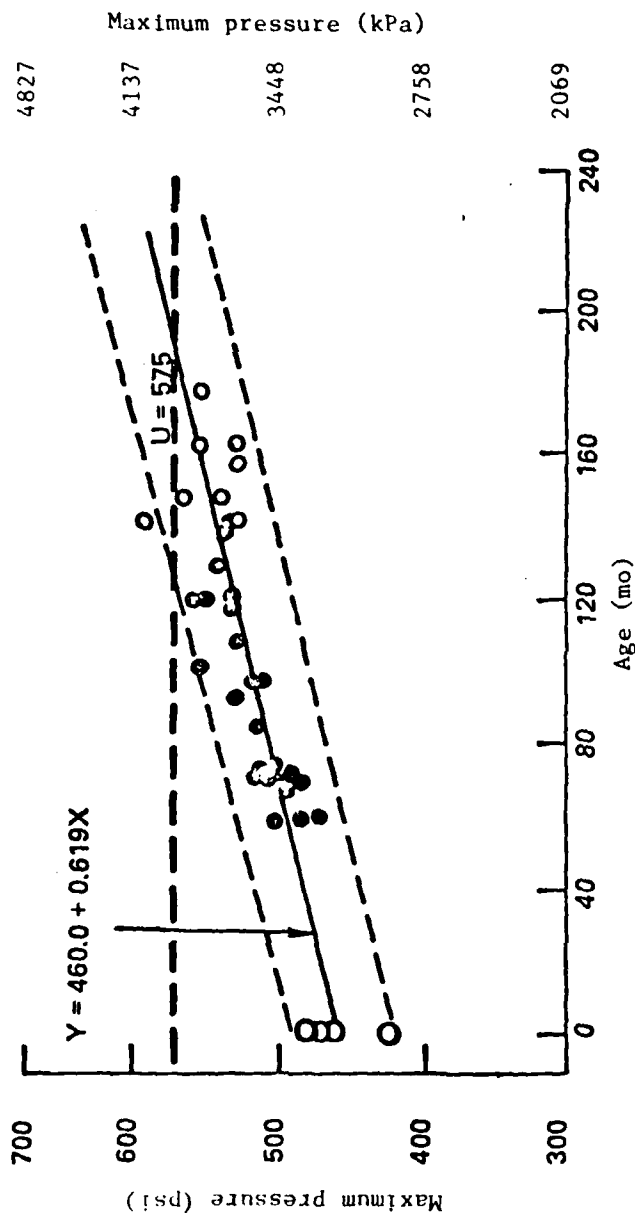
Table 7. ARRADCOM strand burning rate data versus NOS grain firing data

Lot no.	ARRADCOM data			NOS data				Percent difference between burning rates
	Burning rates obtained from plotted graphs (sec/grain)			Average burning rates (sec/grain)	Pressure			
	Temp °C	Temp °F	psi					
					kPa			
IB-8950 (RDD80K000E070)	99	210	89	192	46.5	3110	451	0.43
	-31.7	-25	-37.2	-35	52.8	2717	394	-5.30
IB-8951 (RDD80K000E071)	99	210	89	192	47.7	2992	434	1.26
	-31.7	-25	-37.2	35	56.4	2717	394	-8.33



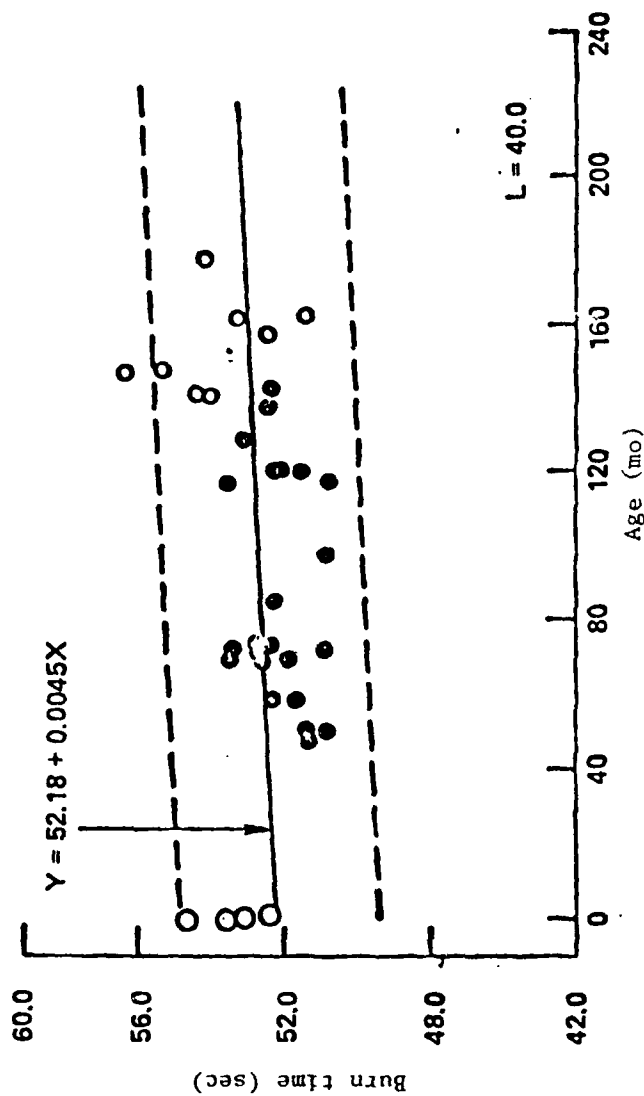
$Y = 41.11 - 0.0299X$ represents the least-squares regression equation.
 $L = 40.0$ represents the specification limit of 40 seconds minimum for burn time.
 The solid line represents the relationship between propellant age and burn time.
 ○ - Open circle represents surveillance data for 1979 per motor (one grain per motor).
 ● - Shaded circle represents surveillance data prior to 1979 per motor (one grain per motor).

Figure 1. Aging trends in burn time at 89°C (192°F)



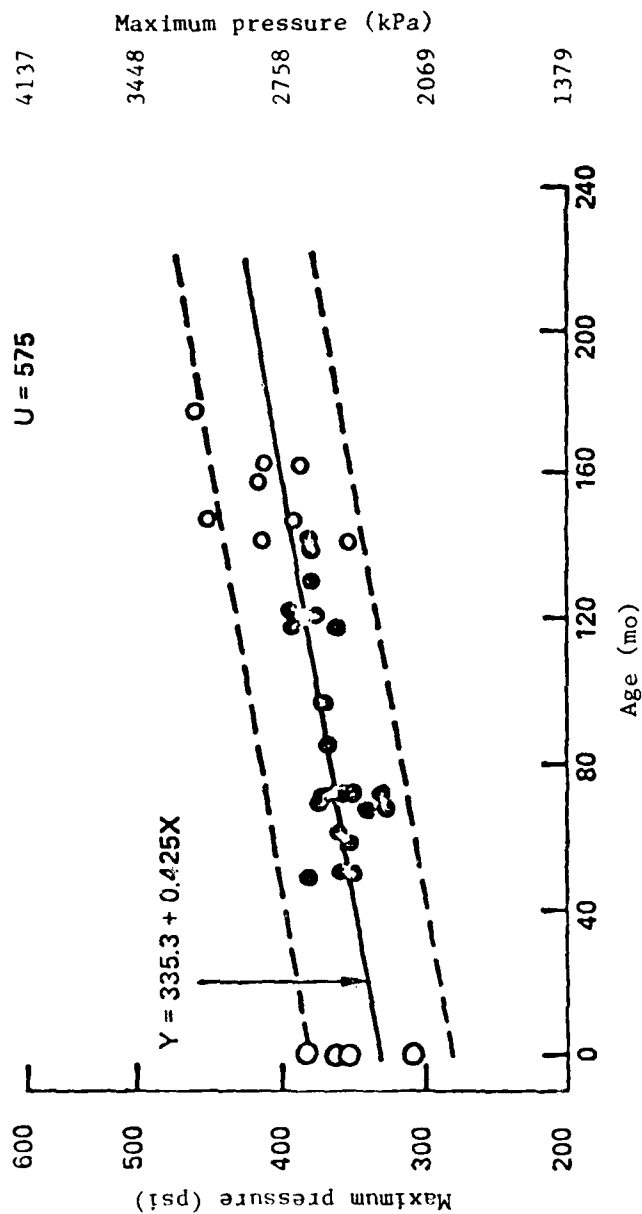
$Y = 460.0 + 0.619X$ represents the least-squares regression equation.
 $U = 575$ represents the acceptance limit of 575 psi (3965 kPa) maximum for pressure.
 The solid line represents the relationship between propellant age and pressure.
 O - Open circle represents surveillance data for 1979 per motor (one grain per motor).
 ● - Shaded circle represents surveillance data prior to 1979 per motor (one grain per motor).

Figure 2. Aging trends in maximum pressure at 89°C (192°F)



$Y = 52.18 + 0.0045X$ represents the least-squares regression equation.
 $L = 40.0$ represents the specification limit of 40 seconds minimum for burn time.
 The solid line represents the relationship between propellant age and burn time.
 O - Open circle represents surveillance data for 1979 per motor (one grain per motor).
 ● - Shaded circle represents surveillance data prior to 1979 per motor (one grain per motor).

Figure 3. Aging trends in burn time at -37°C (-35°F)



Y = 335.3 + 0.425X represents the least-squares regression equation.
 U = 575 represents the acceptance limit of 575 psi (3965 kPa) maximum for pressure.
 The solid line represents the relationship between propellant age and pressure.
 - Open circle represents surveillance data for 1979 per motor (one grain per motor).
 - Shaded circle represents surveillance data prior to 1979 per motor (one grain per motor).

Figure 4. Aging trends in maximum pressure at -37°C (-35°F)

Propellant Lot RDD80K000E070
(IB-8950)

Composition, % XM29
(with 0.08% carbon black
added)

NOTE: See table 4.

Heat of Explosion, cal/gm

Calc 750

Expt 769.4

Ref: MIL STD 286B dated 1 Dec 1967 Method 802.1

γ_p at Constant p/r

From $^{\circ}\text{F}$ to $^{\circ}\text{F}$

Press, at 70 $^{\circ}\text{F}$ $\frac{p/r}{2750}$ $\gamma_p, \%/^{\circ}\text{F}$
0.09

Burning rate values

Temperature		mm/sec	In./sec	Sec/grain
$^{\circ}\text{C}$	$^{\circ}\text{F}$			
99	210	3.02	0.119	46.7
-31.7	-25	2.44	0.096	57.9

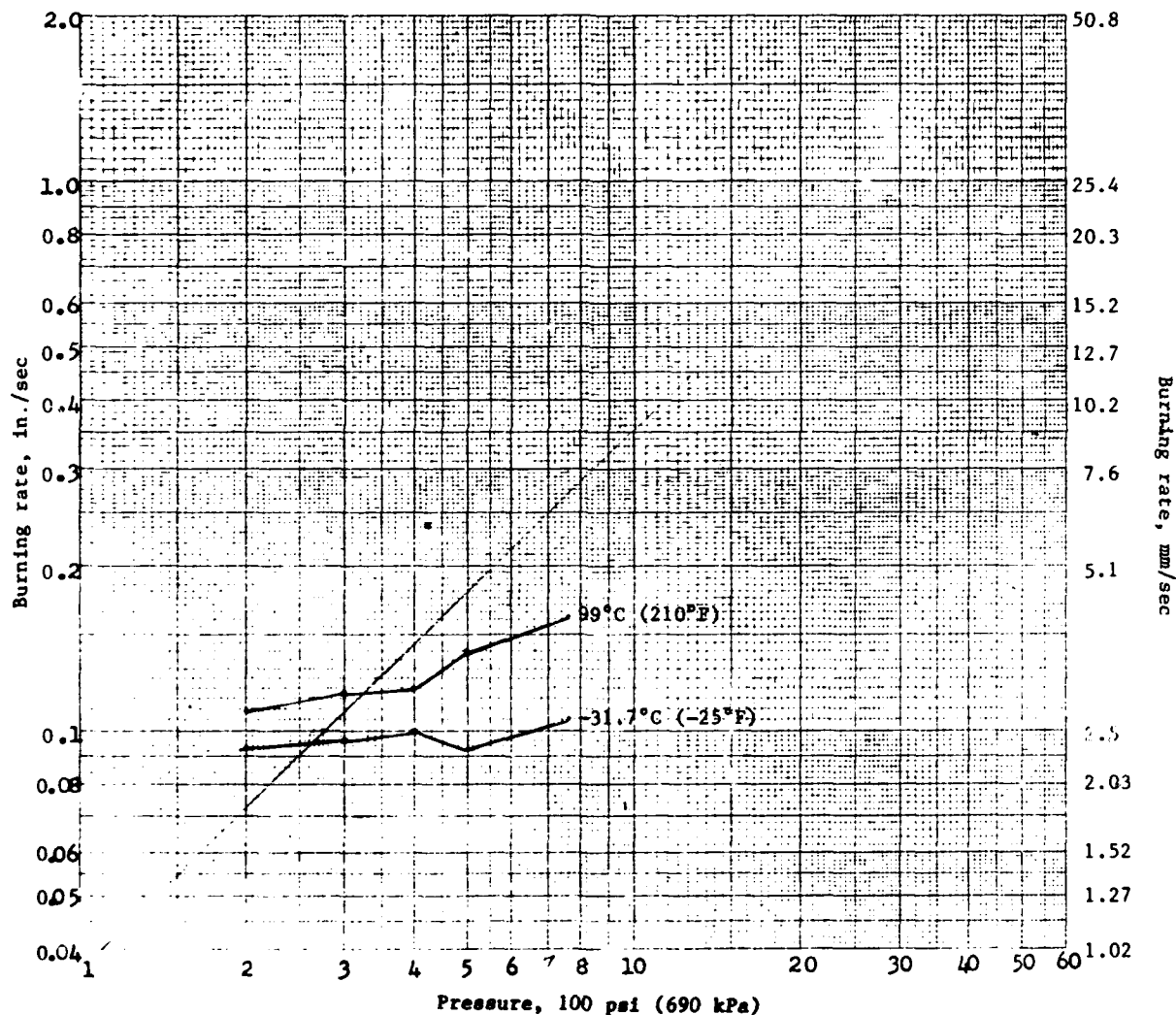


Figure 5. Plots of burning rate values for lot IB-8950 (RDD80K000E070)

Propellant Lot RDD80K000E071
(IB-8951)

Composition, % XM29

(with 0.06% carbon black added) Ref: MIL STD 286B dated 1 Dec 1967, Method 802.1

NOTE: See table 4.

Heat of Explosion, cal/gm

Calc 750

Expt 772.5

γ_p at Constant p/r

From $^{\circ}\text{F}$ to $^{\circ}\text{F}$

Press, at 70°F

p/r

2750

γ_p , %/ $^{\circ}\text{F}$

0.08

Burning rate values

Temperature		mm/sec	In./sec	Sec/grain
$^{\circ}\text{C}$	$^{\circ}\text{F}$			
99	210	2.84	0.112	49.6
-31.7	-25	2.34	0.092	60.4

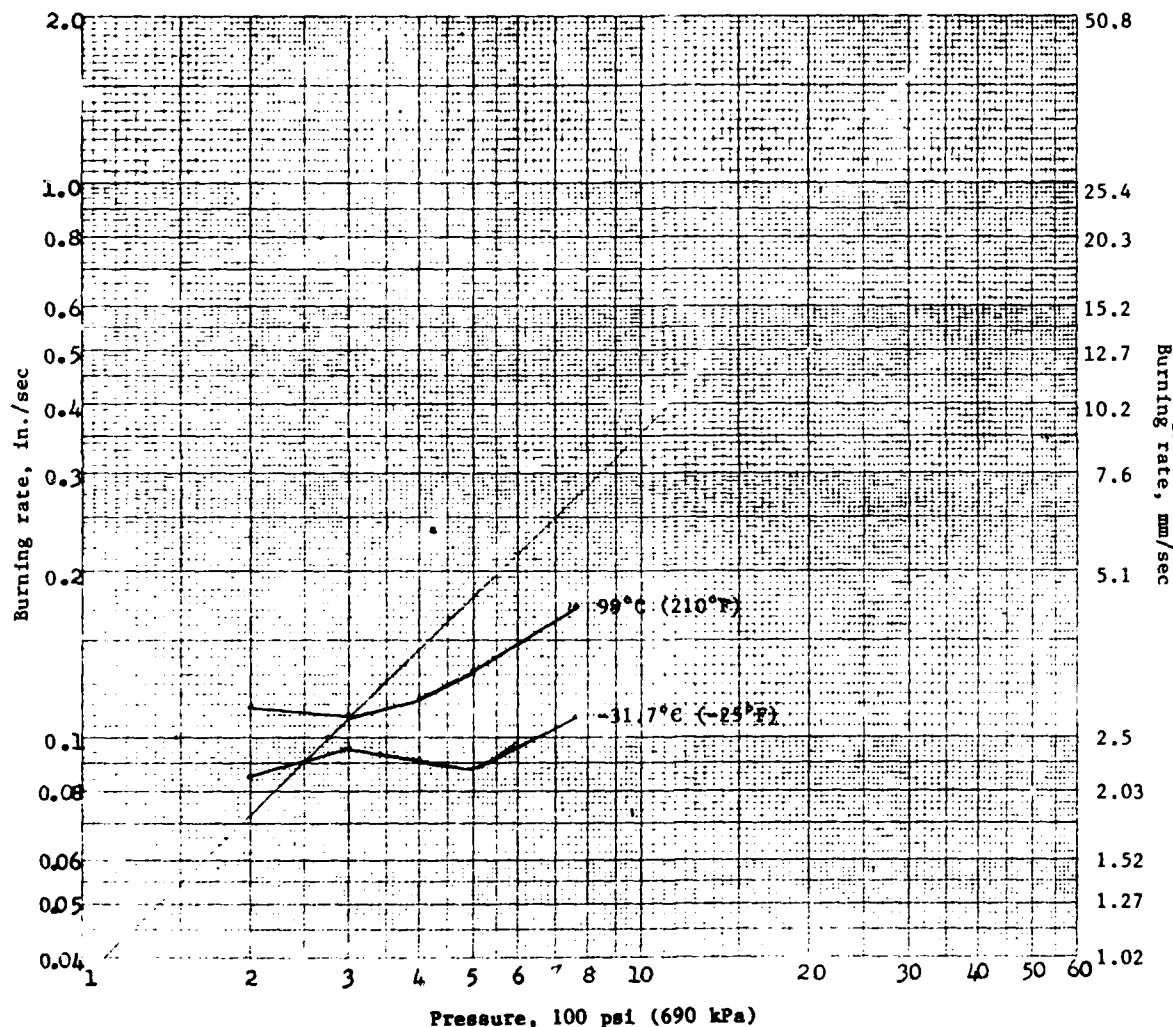


Figure 6. Plots of burning rate values for lot IB-8951 (RDD80K000E071)

Propellant Lot IB-8952

Composition, % XM29

(with 0.08% carbon black added)

Mix. No. 1

NOTE: See table 4.

Heat of Explosion, cal/gm

Calc 750

Expt 768.5

Ref: MIL-STD-286B dated 1 Dec 1967, Method 802.1

η_p at Constant p/r

From $^{\circ}F$ to $^{\circ}F$

Press, at 70 $^{\circ}F$

p/r

η_p , %/ $^{\circ}F$

2750

0.07

Burning rate values

Temperature		mm/sec	In./sec	Sec/grain
$^{\circ}C$	$^{\circ}F$			
99	210	2.77	0.109	51.0
-31.7	-25	2.34	0.092	60.4

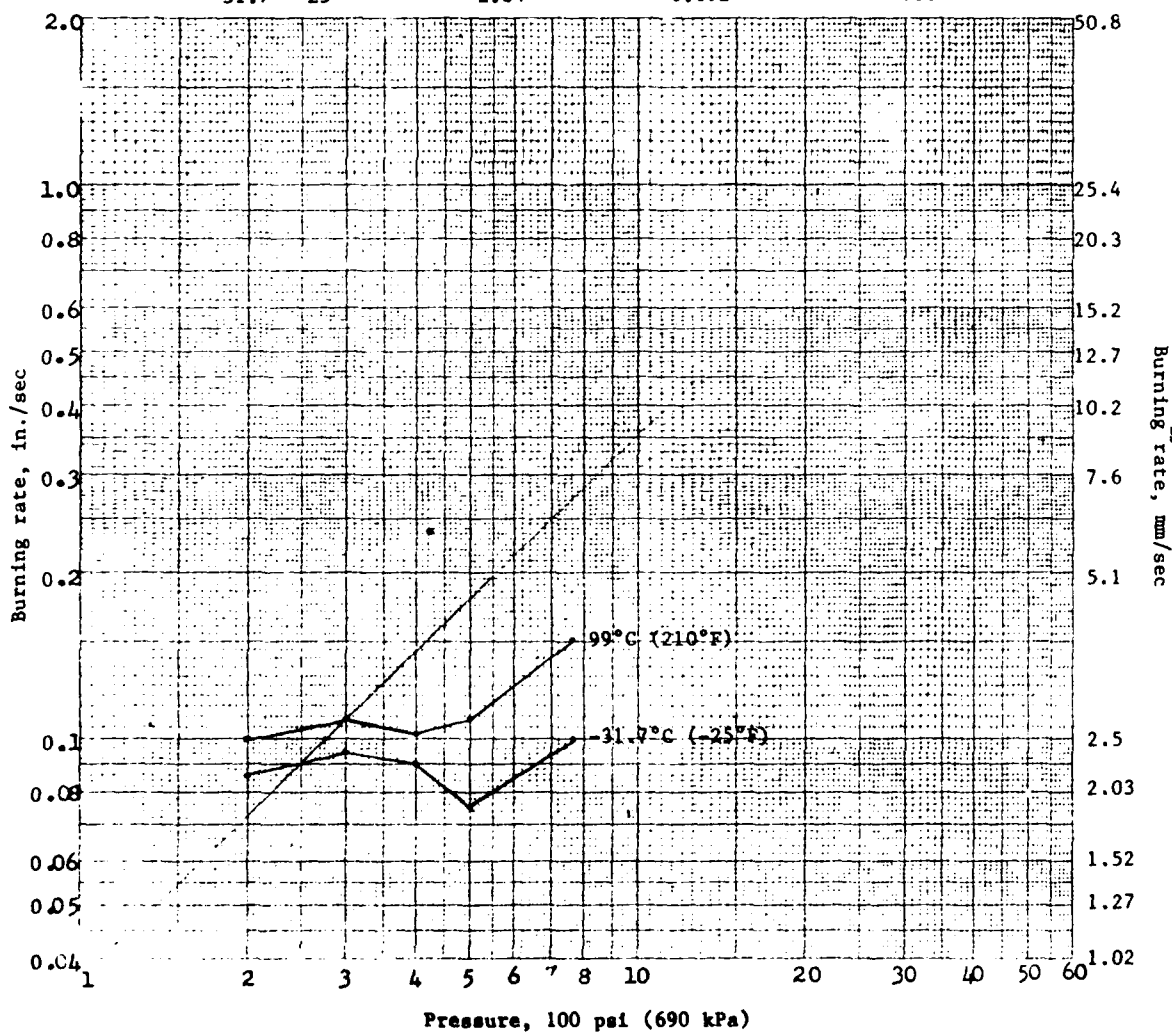


Figure 7. Plots of burning rate values for lot IB-8952 - Mix 1

Propellant Lot IB-8952

Composition, % XM29

(with 0.08% carbon black added)

Mix No. 2

NOTE: See table 4.

Heat of Explosion, cal/gm

Calc 750

Expt 770.3

Ref: MIL-STD-286B dated 1 Dec 1967, Method 802.1

γ_p at Constant p/r

From $^{\circ}\text{F}$ to $^{\circ}\text{F}$

Press, at 70 $^{\circ}\text{F}$

p/r
2750

γ_p , %/ $^{\circ}\text{F}$
0.05

Burning rate values

Temperature		mm/sec	In./sec	Sec/grain
$^{\circ}\text{C}$	$^{\circ}\text{F}$			
99	210	2.67	0.105	53.0
-31.7	-25	2.44	0.096	57.9

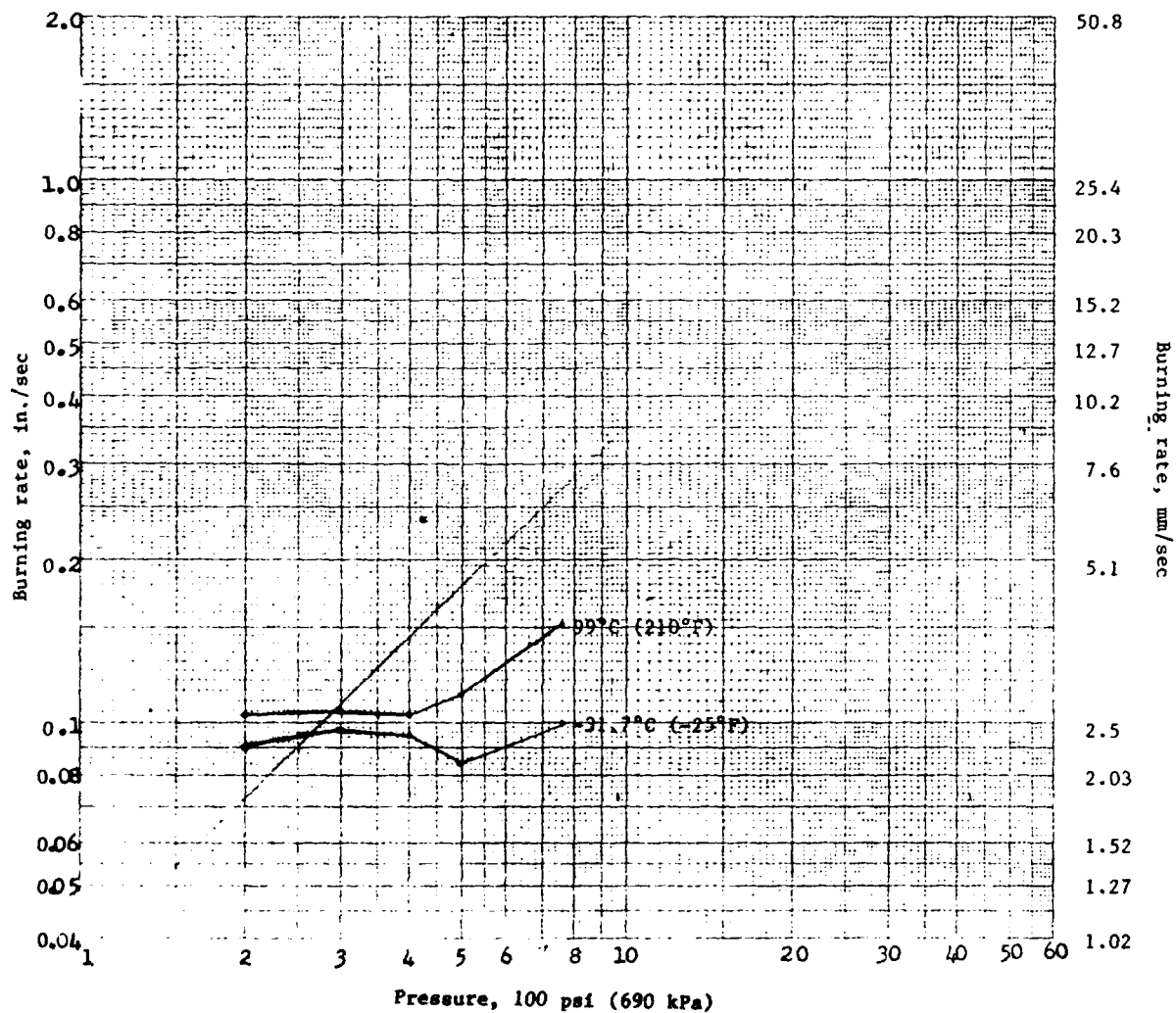


Figure 8. Plots of burning rate values for lot IB-8952 - Mix 2

Propellant Lot 1B-8952

Composition, % XM29
(with 0.08% carbon black added)

Mix No. 3

NOTE: See table 4.

Heat of Explosion, cal/gm

Calc 750

Expt 771.0

Ref: MIL-STD-286B dated 1 Dec 1967, Method 802.1

γ_p at Constant p/r

From $^{\circ}F$ to $^{\circ}F$

Press, at $70^{\circ}F$

p/r

2750

γ_p , %/ $^{\circ}F$

0.05

Burning rate values

Temperature		mm/sec	In./sec	Sec/grain
$^{\circ}C$	$^{\circ}F$			
99	210	2.72	0.107	52.0
-31.7	-25	2.44	0.096	57.9

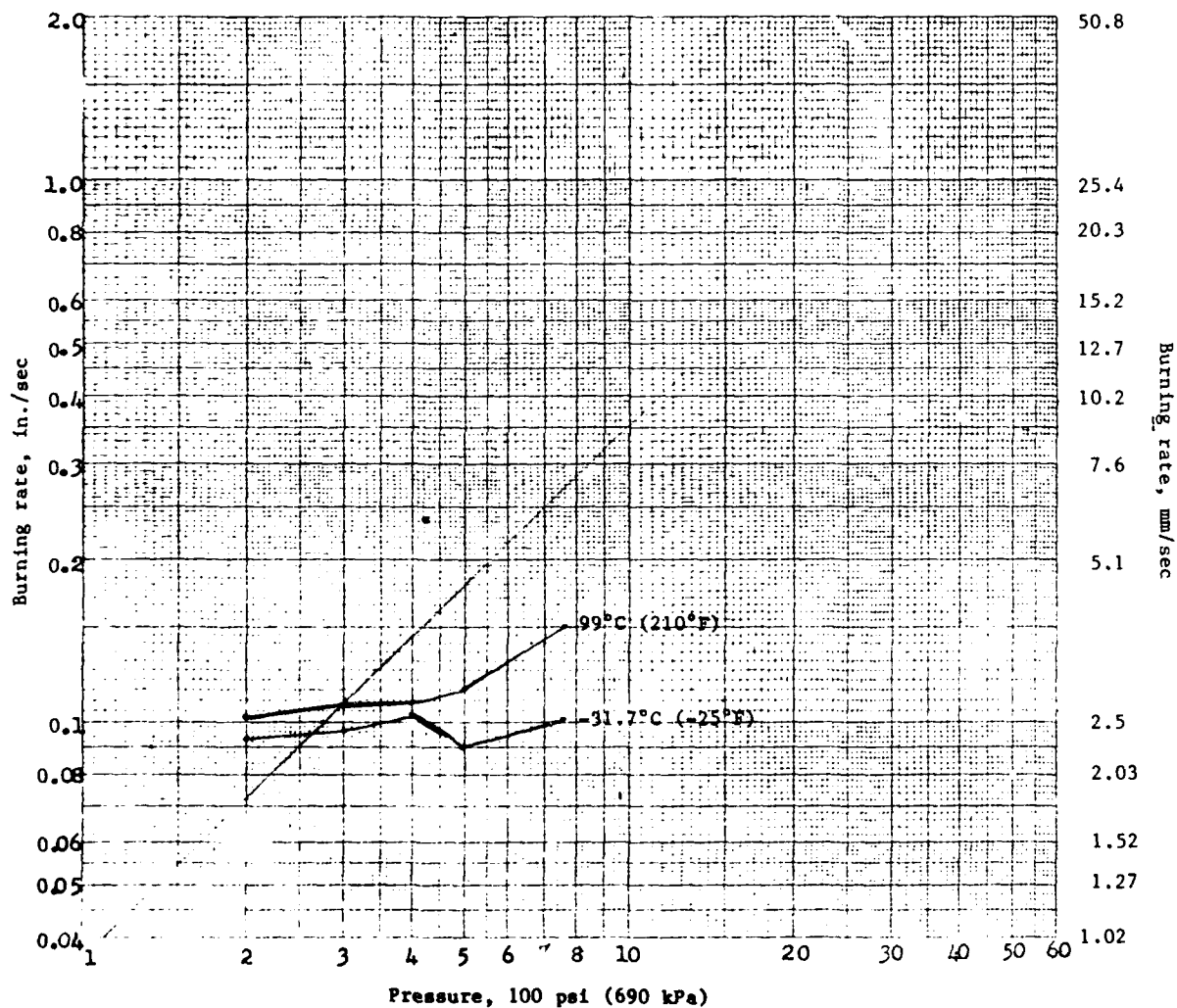


Figure 9. Plots of burning rate values for lot 1B-8952 - Mix 3

Propellant Lot IB-8952

Composition, % HT29

(with 0.08% carbon black added) Ref: MIL-STD-286B dated 1 Dec 1967, Method 802.1

Mix No. 4

NOTE: See table 4.

Heat of Explosion, cal/gm

Calc 750

Expt 770.7

Ref: MIL-STD-286B dated 1 Dec 1967, Method 802.1

η_o at Constant p/r

From $^{\circ}F$ to $^{\circ}F$

Press, at 70 $^{\circ}F$

p/r
2750

η_D , %/°F
0.08

Burning rate values

Temperature		mm/sec	In./sec	Sec/grain
$^{\circ}C$	$^{\circ}F$			
99	210	2.79	0.110	50.5
-31.7	-25	2.29	0.090	61.8

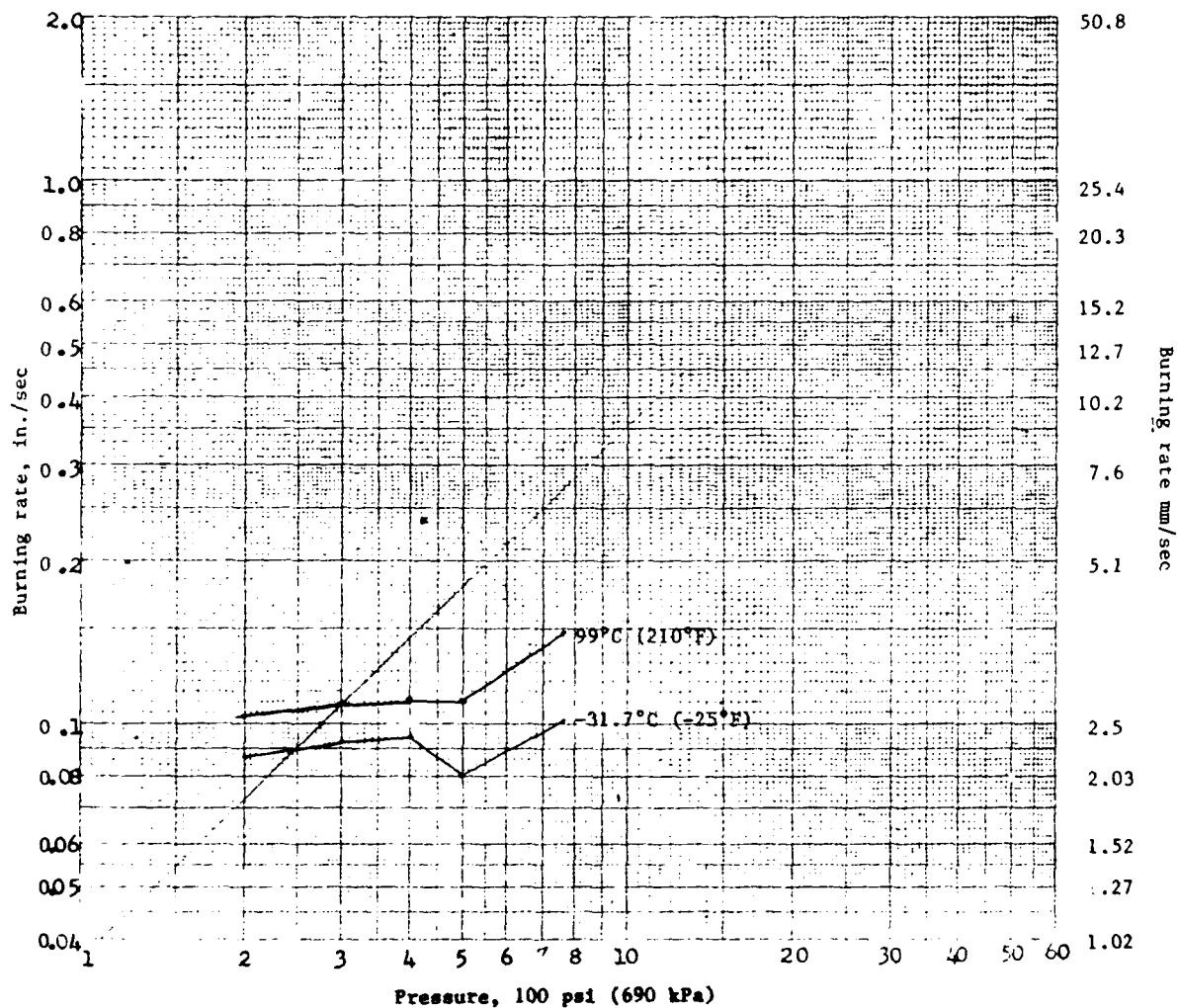


Figure 10. Plots of burning rate values for lot IB-8952 - Mix 4

Propellant Lot IB-8952

Composition, % XM29
(with 0.08% carbon black added)

Mix No. 5

NOTE: See table 4.

Heat of Explosion, cal/gm

Calc 750

Expt 775.9

Ref: MIL-STD-286B dated 1 Dec 1967, Method 802.1

γ_p at Constant p/r

From $^{\circ}F$ to $^{\circ}F$

Press, at 70 $^{\circ}F$

p/r

2750

γ_p , %/ $^{\circ}F$

0.04

Burning rate values

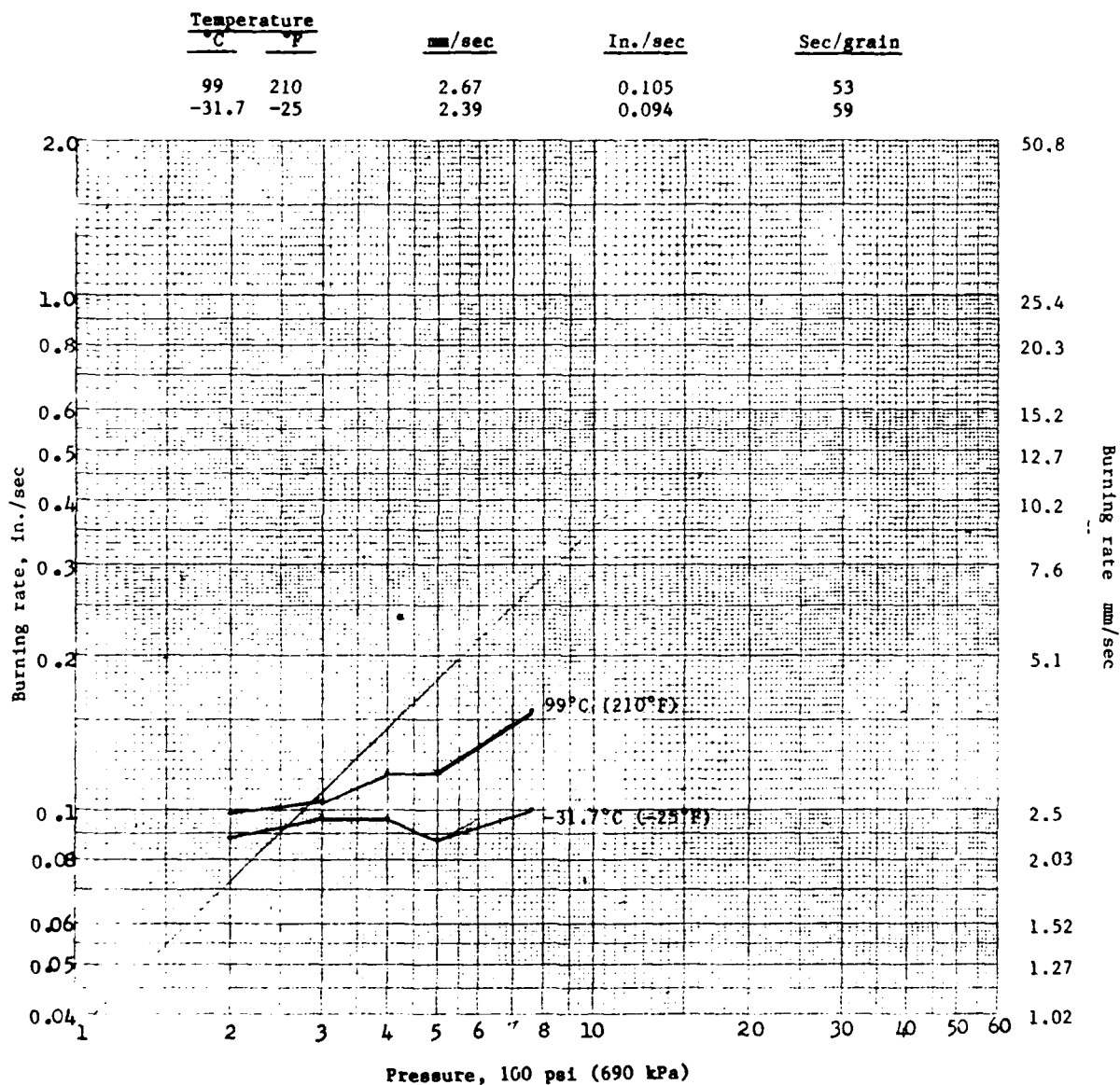


Figure 11. Plots of burning rate values for lot IB-8952 - Mix 5

Propellant Lot IB-8952

Composition, % XM29

(with 0.08% carbon black added) Ref: MIL-STD-286B dated 1 Dec 1967, Method 802.1

Mix No. 6

NOTE: See table 4.

Heat of Explosion, cal/gm

Calc 750

Expt 770.6

γ_p at Constant p/r

From $^{\circ}F$ to $^{\circ}F$

Press, at $70^{\circ}F$

p/r

2750

γ_p , %/ $^{\circ}F$

0.07

Burning rate values

Temperature		mm/sec	In./sec	Sec/grain
$^{\circ}C$	$^{\circ}F$			
99	210	2.69	0.106	52.5
-31.7	-25	2.29	0.090	61.8

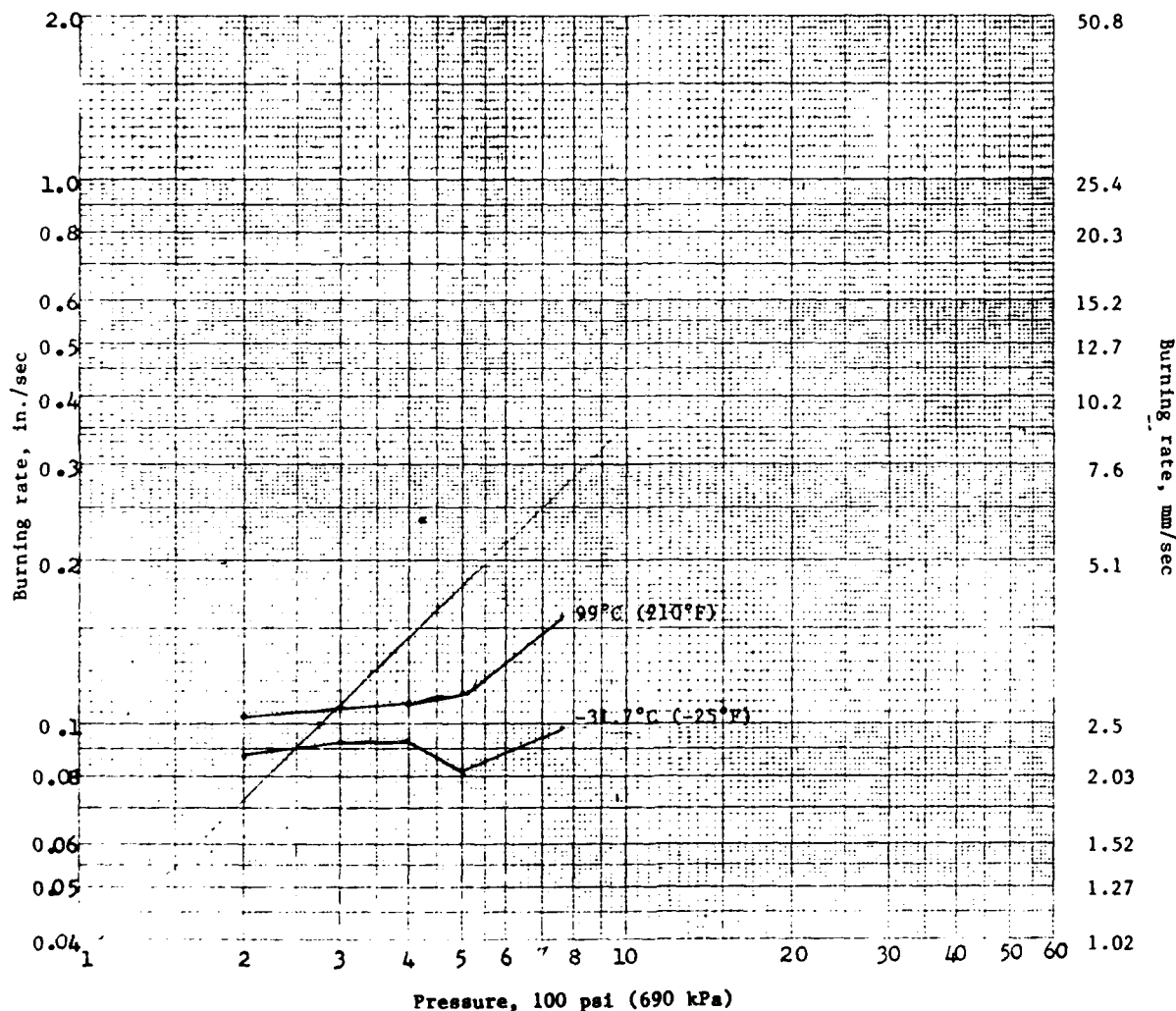


Figure 12. Plots of burning rate values for lot IB-8952 - Mix 6

Propellant Lot ~~XXXXXXXXXX~~
(IB-8952)

Composition, % RDD8 --Master Blend
(with 0.004 carbon black added)

Heat of Explosion, cal/gm

Calc 790

Expt 742

Ref: MIL-STD-286B dated 1 Dec 1967, Method 802.1

γ_p at Constant p/r

From $^{\circ}F$ to $^{\circ}F$

Press, at 70 $^{\circ}F$

p/r

γ_p , %/ $^{\circ}F$

2750

8.06

NOTE: See table 4.

Burning rate values

Temperature		mm/sec	In./sec	Sec/grain
$^{\circ}C$	$^{\circ}F$			
99	210	2.79	0.110	50.5
-31.7	-25	2.39	0.094	59.1

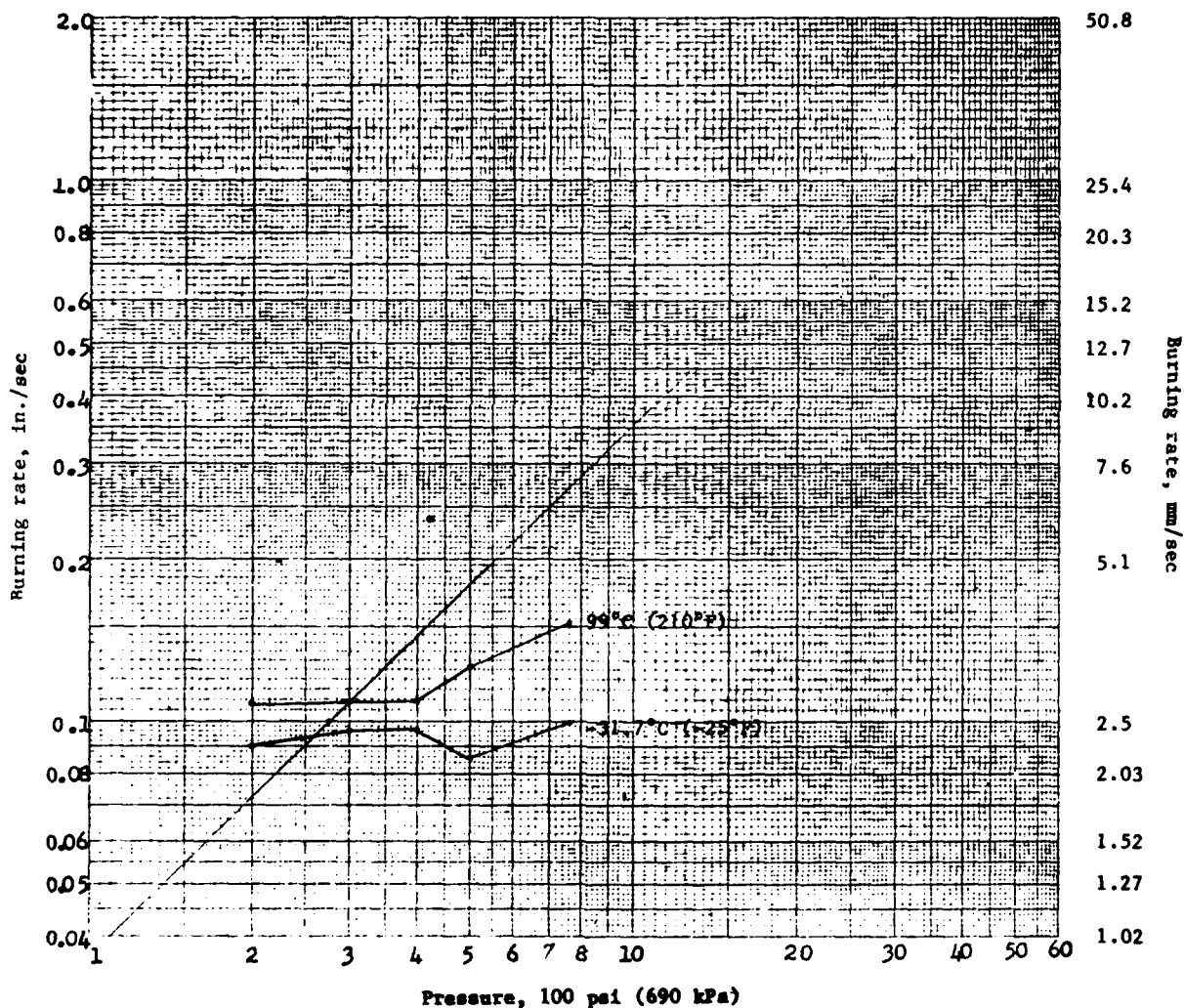


Figure 13. Plots of burning rate values for lot RDD81F000E094 (IB-8952) - Master Blend

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