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UNIDYNAMICS

RESEARCH AND DEVELOPMENT
DIRECTED TOWARD THE DEVELOPMENT OF

GAS GENERATORS

QUARTERLY REPORT NO. 2

SIGNAL CORPS CONTRACT NO. DA-36-039 SC-87362

SECOND QUARTERLY PROGRESS REPORT
15 OCTOBER 1961 TO 14 JANUARY 1962

U.S. ARMY SIGNAL RESEARCH AND DEVELOPMENT LABORATORY
FORT MONMOUTH, NEW JERSEY

UNIDYNAMICS
A DIVISION OF UNIVERSAL MATCH CORPORATION
CRAB ORCHARD OPERATIONS
MARION, ILLINOIS

UNIDYNAMICS

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CONTRACT NUMBER DA-36-039 SC-87362

SIGNAL CORPS TECHNICAL REQUIREMENT SCL 7564

DATED 11 AUGUST 1960

The object of this program is to develop gas generators covering an output range of 50 to 10,000cc, with means of incorporating delay times from electrical pulse to propellant ignition of 0-2 seconds, with operating temperatures from -65° F to 212° F and storage temperatures from -80° F to 300° F.

Prepared by: B. R. Steele

Approved by:

E. E. Johnson,
Supervisor,
Engineering Laboratories

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PURPOSE

The purpose of this project is to develop improved gas generators to activate zinc-silver oxide batteries employing the Signal Corps metal-tube electrolyte-reservoir activating system. It is desired to replace the gas generators presently employed for this task with a unit which has a longer shelf life over a wider range of environmental conditions.

The project consists of three major tasks: (1) design and development of gas generators, (2) environmental testing, and (3) reports, conferences, and shipment of prototype units.

ABSTRACT

This report describes the work conducted during the second quarter under Contract DA-36-039 SC-87362 with the U. S. Signal Supply Agency and offers conclusions based on the test results. The work consisted of a propellant investigation, test fixture evaluation tests, and pressure-vs-time testing of two propellant formulations.

As a result of the vendor survey UMC will conduct propellant evaluations to determine whether or not the thermal stability of the two commercially available propellants will equal or surpass ~~that of UMC~~ propellant N-1825.

Propellant N-1801 Mod A was found unsuitable for use after storage at 300° F due to decomposition.

Propellant N-1825 prepared by UMC exhibited a small percentage weight loss during thermal stability testing at 300° F for one week. In addition this propellant exhibited low gas evolution when stored at 300° F for one week as compared to other propellants tested. It also displayed satisfactory gas output and more reproducible pressure-vs-time results (time-to-peak pressure and peak pressure) than the N-5 standard propellant. As a result of preliminary testing, it is anticipated that N-1825 propellant will be capable of meeting the Signal Corps technical requirement SCL-7564. This propellant will be investigated further.

The UMC sealed match has proven effective for igniting the gas generators under various temperature conditions, including 300° F for 168 hours.

The redesigned test fixture has proved satisfactory.

CONFERENCES

Progress Review Meeting No. 3 was held at the U. S. Army Signal R & D Laboratory on 8 November 1961 to discuss work accomplishment during the first quarter of the contract. It was mutually agreed that UMC would: (1) utilize a sealed electric match to obtain front end ignition of the propellant grain, (2) continue the propellant investigation with efforts directed toward developing the grain for the gas generator, and (3) continue the market survey for a commercially available propellant.

A conference was held at the U. S. Army Signal R & D Laboratory on 1 December 1961 to discuss future plans. UMC received approval of a propellant work plan.

KEY TECHNICAL PERSONNEL

The following technical personnel have been assigned to this program and have been accredited with the approximate man-hours shown below.

Personnel	Approximate Man-Hours
B. E. Stauder	43
R. E. Williams	70
B. R. Steele	549
C. R. Rittenhouse	69
M. D. Tharp	33

1. FACTUAL DATA

1.1 General. This section describes the work conducted during the second quarter. It consisted of a propellant investigation, test fixture evaluation tests, and pressure-vs-time testing of two propellant formulations.

1.2 Propellant Formulations. Based on the results of the literature survey conducted during the first quarter and in an effort to obtain a composition with greater thermal stability than formulation N-1801 (prepared during the first quarter), UMC formulated composition N-1825 which consists of the following:

Ammonium Perchlorate	-	48.5%
Hycar 1000 x 103	-	15.0%
Guanidine Picrate	-	36.0%
Carbon Black	-	0.5%
		100.0%

1.2.1 Composition N-1825 was subjected to four 168 hour - 300° F thermal stability tests. The propellant lost 2.76 percent and 3.19 percent by weight in open containers and 1.90 percent and 2.14 percent by weight in closed containers. The tests were conducted as follows:

STEP	PROCEDURE
1.	Weigh four samples (approximately 2 grams each).
2.	Place two samples in ointment tubes and crimp the ends.
3.	Place two samples in open containers.
4.	Place all four containers in an oven and let remain for one week at 300° F.
5.	Weigh each of the samples on a Gram-atic balance and determine the percentages of weight loss.

1.2.1.1 Two of the samples were tested in ointment tubes to determine (1) whether or not the test container would be ruptured by pressure, and (2) the effect of limited atmospheric oxygen on decomposition.

1.2.1.2 The thermal stability testing showed that formulation N-1825 loses less weight than any other composition thus far tested (Formulation N-1801 lost 3.35 percent in open containers and 2.04 percent in closed containers).

1.3 Propellant Investigation. The propellant investigation conducted by UMC during this quarter consisted of the following:

a. A vendor survey to determine whether or not a suitable propellant was commercially available.

b. Gas output tests to determine the volume of gas produced by given weights of propellants.

c. Gas evolution tests to determine (1) the pressure of gases evolved and (2) the percent of weight loss and changes in physical qualities after storage at 300° F for seven days in sealed pressure capsules.

d. Pressure-vs-time testing to determine the peak pressure, time-to-peak pressure, ignition time and the effect of temperature variation on each of these parameters.

e. Chromatographic gas analysis to determine the composition of the propellent combustion gases.

1.3.1 A plan for conducting the propellant investigation was prepared by UMC and approved by the Signal Corps (See Appendix A). It was agreed that the propellant investigation would be completed by 15 January 1962.

1.4 Vendor Survey. A new propellant procurement specification was prepared and a request for quotation was submitted to 11 propellant manufacturers. The specification is included in Appendix A. The manufacturers and condensed statements of their replies are listed below:

a. Olin Mathieson has no propellant which meets the specification.

b. Propellex has no propellant which meets the specification.

c. Atlantic Research states that they can tailor an existing propellant to meet the specification during a six-week development program.

d. Hercules Powder has a propellant which might meet the specification. However, all high temperature propellants are in the experimental stage and the compositions are proprietary.

e. Aerojet General has no propellant which meets the specification.

f. Thiokol has propellant TP-J-3000 which should meet the requirements. The bid was received too late, however, to obtain the propellant by the cutoff date of 15 January 1962.

g. Amcel Propulsion has no propellant which meets the specification.

h. Lockheed Propulsion (Formerly Grand Central Rocket) has no propellant which meets the specification.

i. Rocketdyne proposed to develop a propellant to meet the specification during a six-month development program.

j. U. S. Naval Propellant has no propellant which meets the specification.

k. B. F. Goodrich has a propellant which they feel will meet all of the requirements. The composition is classified confidential, but it is similar to UMC propellant formulation N-1801 (described in Quarterly Report No. 1). This propellant will be tested during the third quarter since the bid was received after the 15 January 1962 cutoff date.

1.5 Propellant Preparation. UMC continued with the investigation of a propellant which would meet the specifications. Three types were prepared and tested. These included N-5, which is the standard propellant, and two UMC propellent formulations, N-1825 and N-1801 Mod A.

1.5.1 After transferring N-5 propellant from stock, certification was ascertained and a moisture analysis was conducted.

1.5.2 Propellant N-1801 was mixed in accordance with Preparation Procedure No. M-1 (Appendix B). The propellant was extruded into random lengths with a .390 ± .020-inch diameter in accordance with Procedure No. E-1 (Appendix B). A moisture analysis was conducted.

1.5.3 The preparation of Propellant N-1825 consisted of preparing guanidine picrate in accordance with Preparation Procedure G. P. 1 (Appendix C), mixing the propellant in accordance with Preparation Procedure M-2 (Appendix C), extruding the propellant into random lengths with a .390 ± .020-inch diameter in accordance with Procedure E-1 (Appendix B), and conducting a moisture analysis.

1.6 Moisture Analysis. A moisture analysis was conducted on each of the three propellants to determine the percentage of moisture absorbed during open air storage. The tests were conducted in duplicate, using approximately 1-gram samples of extruded propellant. The original samples were placed in a 212° F oven for one hour and then weighed. The N-5 propellant showed signs of deterioration with a 1.75 percent weight loss, change in color from red to black, and exudate on the surface. The N-1825 and N-1801 Mod A propellants showed less than

.10 percent at this point. As a result of the deterioration of N-5 the tests were conducted at 160° F for three hours. Table I shows the results of the moisture analysis.

TABLE I

MOISTURE ANALYSIS

Propellant	Sample No. 1	Sample No. 2
N-5	.228%	.233%
N-1825	.100%	.140%
N-1801 Mod A	.043%	.036%

1.7 Rated Gas Output. Tests were conducted on compositions N-5, N-1801 Mod A, and N-1825 in order to determine the gas produced by each propellant composition in terms of cubic centimeters gas output per gram of propellant.

1.7.1 The tests were conducted in triplicate with approximately 0.3 gram samples of each propellant. The samples were burned in a Parr gas evolution bomb fitted with a 0-100 psi pressure gage. After burning each of the propellant samples, the bomb was immersed in boiling water and allowed to pressure stabilize. The pressure at 100° C was then substituted into the following equation and the gas volume calculated:

Equation 1: $$V = \frac{2.32(P)-8.5}{W}$$

Where: V = gas volume, cc/gm
P = pressure, psi
W = sample weight, gm

Derivation of Equation is as follows:

$$V = \frac{1}{W}\left[\frac{P}{P_o}\ \frac{T_o}{T}\ (V_1) - \frac{T_o}{T_1}\ (V_1)\right]$$

where: V = Total gas evolved at STP in cc/gm
W = Sample weight in grams
P = Absolute pressure reading PSIA = $P_g + P_o$
P_s= Pressure reading on gage PSI
P_o= STP Pressure = 14.7 PSIA
T_o= STP Temperature = 273° K
T = Temperature of bomb and fittings = 373° K
T_1= Ambient temperature = 298° K
V_1= Volume of bomb = 46.5 cc
$\frac{T_c}{T_1}(V_1)$ = Volumn of air in bomb at STP

Substitution of known values yields:

$$V = \frac{1}{W}\ \frac{14.7 + P_g}{14.7}\ \frac{273}{373}\ (46.5) - \frac{273}{298}\ (46.5)$$

This equation reduces to:

$$V = \frac{2.32\ P_g - 8.5}{W}$$

Table II shows the results of this testing.

TABLE II

GAS OUTPUT

(cubic centimeters per gram)

Propellant Type	Sample No. 1	Sample No. 2	Sample No. 3	Ave.
N-5	627	658	652	646
N-1801 Mod A	673	729	728	710
N-1825	546	535	541	54[illegible]

1.7.2 The data shown in Table II indicates the superiority of propellant N-1801 Mod A with regard to gas output. Based on this data, the following propellant weights would be required for a 950 cc gas generator assuming 100 percent efficiency:

$$\text{N-5} \quad \frac{950\text{cc}}{646\text{cc/gm}} = 1.47 \text{ gm}$$

$$\text{N-1801 Mod A} \quad \frac{950\text{cc}}{710\text{cc/gm}} = 1.34 \text{ gm}$$

$$\text{N-1825} \quad \frac{950\text{cc}}{541\text{cc/gm}} = 1.75 \text{ gm}$$

1.8 Gas Evolution. Gas evolution tests were conducted on propellants N-5, N-1801 Mod A, and N-1825. The purpose of these tests was to determine (1) the pressure of gases evolved during high temperature storage for seven days, (2) the amount of weight loss during high temperature storage for seven days in sealed units, and (3) the changes in physical characteristics of the propellants.

1.8.1 Tests were conducted in duplicate, using propellant grains of approximately equal weights. The propellant samples were measured, weighed, and then loaded into pressure capsules (Figure 1). As shown by Figure 1 the needle valve is used for bleeding the evolved gas for gas analysis; the opposite end is fitted with a 300 psi rupture disk for safety purposes. Variation in free volume within the capsules due to differences in grain sizes was adjusted by adding metal washers so that all capsules had similar free volumes. The pressure capsule volume was 9.2 cc while the propellant volume was 4 cc, leaving a free volume of 5.2 cc.

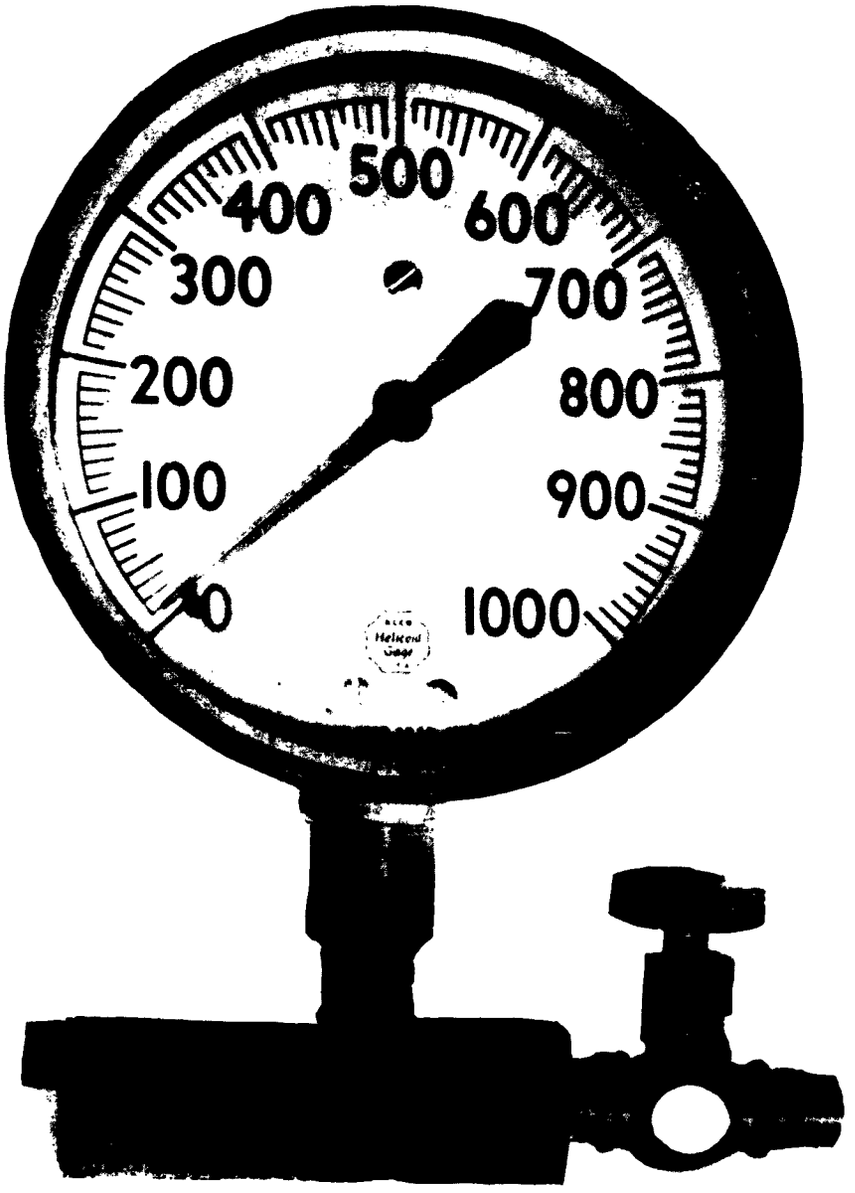


FIGURE 1

1.8.2 The two pressure capsules containing N-5 propellant were placed in a 200° F oven and the capsules containing N-1801 Mod A and N-1825 propellants were placed in a 300° F oven. (NOTE: N-5 propellant was limited to 200° F based on elevated temperature storage test results conducted by the Signal Corps which showed gas generators loaded with N-5 have a useful life of approximately 7.3 days at 220° F and 22 days at 200° F.) Pressure readings were taken after one hour. They were then repeated every 24 hours for 168 hours (See Table III for pressure readings obtained).

1.8.2.1 The readings of 0 psi and 8 psi for capsules 3 and 4 were in error as explained in paragraph 1.8.2.2. The results show that in utilizing N-1825 propellant at 300° F storage pressure buildup within the gas generators is reduced by approximately one-fourth when compared with N-5 at 200° F. These tests also indicate that N-1825 propellant exhibits little tendency for decomposition.

1.8.2.2 At the end of 168 hours storage at elevated temperatures all pressure capsules under test were post-mortemed to determine physical changes of the propellant samples with respect to dimensions and weight. Post-mortems of pressure capsules 3 and 4 showed that the 300 psi burst disks had been ruptured, indicating a decomposition.

1.8.2.3 Table IV shows the physical parameters of the propellant samples before and after temperature storage. This data indicates the superior thermal stability of N-1825 propellant when compared

TABLE III

PRESSURES GENERATED DURING ELEVATED TEMPERATURE

	N-5* (200° F)		N-1801 MOD A (300° F)		N-1825 (300° F)	
Time Hours	Capsule No. 1	Capsule No. 2	Capsule No. 3	Capsule No. 4	Capsule No. 5	Capsule No. 6
1	-	-	-	-	-	-
24	5	3	8*	-*	-	8
48	20	21	8	-	-	10
72	43	50	8	-	-	10
96	55	71	8	-	-	12
120	63	85	8	-	-	12
144	68	98	8	-	-	20
168	70	110	8	-	15	30

*Post-mortem showed 300 psi safety disk was ruptured.

TABLE IV

PHYSICAL DATA ON PROPELLANT SAMPLES

BEFORE AND AFTER EVLATED TEMPERATURE STORAGE FOR 168 HRS.

		N-5 (200° F)		N-1801 MOD A (300°F)		N-1825 (300° F)	
		Capsule No. 1	Capsule No. 2	Capsule No. 3	Capsule No. 4	Capsule No. 5	Capsule No. 6
Grain Diameter Before	(in.)	.392	.391	.392	.392	.390	.390
Grain Diameter After	(in.)	.375	.380	.420	.418	.389	.389
Grain Length Before	(in.)	1.998	1.999	1.913	1.912	1.997	1.992
Grain Length After	(in.)	1.999	1.975	1.895	1.824	1.979	1.954
Weight Before	(gms)	5.3148	5.2965	5.3160	5.3182	5.3125	5.3170
Weight After	(gms)	5.1355	5.1070	4.4196	4.3586	5.2871	5.2890
Weight Loss	(gms)	.1793	.1895	.8964	.9596	.0254	.0280
Percent Weight Loss		3.37	3.57	16.86	18.04	.48	.53

with N-5 and N-1801 Mod A. Figures 2, 3, and 4 show their respective grains in the condition in which they were removed from the pressure capsules.

1.8.2.4 Figure 2 shows a gummy substance on the N-5 propellant grain which is attributed to the exudation of nitroglycerin. The N-5 grains were soft and had changed in color from red-orange to black.

1.8.2.5 Figure 3 shows the deformation of the N-1801 Mod A propellant grains. The surfaces of these grains were covered with a granular substance which chemical analysis proved to be principally ammonium perchlorate and some nitroguanidine. It is postulated that the ammonium perchlorate was forced to the surface as the nitroguanidine sublimed.

1.8.2.6 Figure 4 shows the smooth surface and excellent condition of the N-1825 propellant grain. Although the surface color of the grains had changed from green to black, cross-sectioning showed that the color change was only on the surface. The grains had increased in hardness from a durometer 50 to a durometer 95. The hardening is attributed to the curing of the Hycar 1000 x 103 rubber fuel binder.

1.8.2.7 These tests have shown that N-1825 propellant exhibits excellent thermal stability when stored at 300° F for seven days. The .48 and .53 percent weight loss is attributed to the loss of residual solvents and is deemed insignificant when compared to the 3.37 and 3.57 percent weight loss exhibited by the standard N-5 propellant.

N-5

FIGURE 2

N-1801 MOD-A

FIGURE 3

[redacted]

N-1825

FIGURE 4

1.9 Pressure-vs-Time Tests. Pressure-vs-time tests were conducted on each of the three propellant compositions (N-5, N-1801 Mod A, and N-1825) to determine the effect of temperature variation on peak pressure, time-to-peak pressure, and ignition time.

1.9.1 A total of 66 gas generators were tested. Twenty-two units were loaded with N-5; 22 units were loaded with N-1801 Mod A; and 22 units were loaded with N-1825. Each generator was loaded as follows:

a. Propellant grain - (1) diameter - .390 ± .020 inch
(2) length - 1.00 ± .020 inch

b. Ignition material - 1.5 gram of standard ignition material, 666

c. Igniter - One UMC sealed electric match.

1.9.2 The units were tested in the test fixture shown in Figure 5, and the pressure-vs-time test results are presented in Tables V, VI, and VII.

1.9.3 The chart below shows the variations of the three propellant compositions over the temperature range shown in Tables V, VI and VII.

Percent Variation from Mean

Composition	Temperature Range	Peak Pressure	Time-to-Peak Pressure
N-5	-65 to 200° F	31.7	39
N-1801 Mod A*	-65 to 300° F	16.3	22.6
N-1825	-65 to 300° F	30.4	14.1

*Results do not include 300° F temperature storage

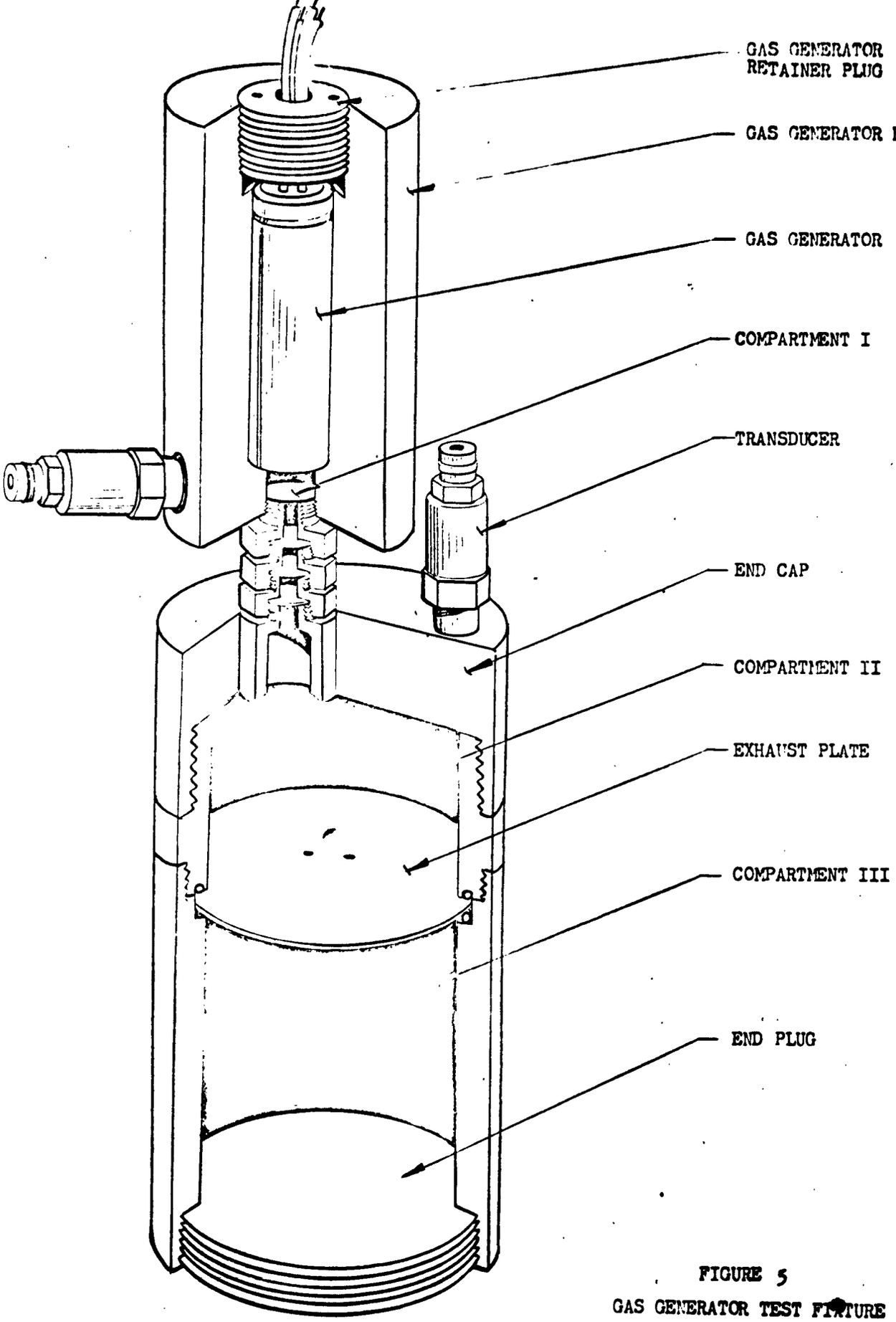


FIGURE 5
GAS GENERATOR TEST FIXTURE

TABLE V

PRESSURE-VS-TIME DATA FOR PROPELLANT N-5

Unit No.	Storage Temp. (°F)	Storage Time (Hrs.)	Ignition Time (μsec.)	Peak Pressure Chamber I (psi)	Time-to-Peak Chamber I (ms)	Peak Pressure Chamber II (psi)	Time-to-Peak Chamber II (ms)
1.	Ambient	-	150	1130	48	170	625
2.	Ambient	-	120	1351	49	- (1)	- (1)
3.	Ambient	-	170	1020	51	170	730
4.	Ambient	-	100	- (2)	- (2)	200	600
5.	-65	4	150	1200	56	160	710
6.	-65	4	150	- (2)	- (2)	140	720
7.	-65	4	140	810	165	200	680
8.	-65	4	100	900	50	200	640
9.	212	4	120	1180	35	- (3)	- (3)
10.	212	4	120	480	41	195	590
11.	212	4	100	900	34	230	480
12.	212	4	100	690	44	300	460
13.	TS (4)	30	90	- (5)	- (5)	205	560
14.	TS (4)	30	100	1200	33	220	460
15.	TS (4)	30	100	1170	72	260	460
16.	TS (4)	30	100	1200	31	230	420
17.	200	72	125	1[illegible]	27	220	480
18.	200	72	125	600	27	200	460
19.	200	120	120	1140	70	290	340
20.	200	120	150	1020	45	270	420
21.	200	168	40	900	35	250	460
22.		168	60	- (5)	0 (5)	370	320
AVERAGES				1003	51	224	531

(1) Camera shutter not opened
(2) Scope did not trigger
(3) Defective Film
(4) Thermal Shock - 5 cycles, each cycle consisting of 3 hours at -80° F followed immediately by 3 hours at 212° F.
(5) Rupture diaphrams apparently ruptured by hot particle rather than pressure rise.

TABLE VI

PRESSURE-VS-TIME DATA FOR PROPELLANT N-1801 MOD A

Unit No.	Storage Temp. (° F)	Storage Time (Hrs.)	Ignition Time (μsec.)	Peak Pressure Chamber I (psi)	Time-to-Peak Chamber I (ms)	Peak Pressure Chamber II (psi)	Time-to-Peak Chamber II (ms)
1.	Ambient	-	100	-(1)	-(1)	360	360
2.	Ambient	-	125	750	18	500	350
3.	Ambient	-	100	-(2)	-(2)	405	325
4.	Ambient	-	150	800	20	-(3)	-(3)
5.	-65	4	120	930	72	420	380
6.	-65	4	120	1200	27	390	380
7.	-65	4	80	900	25	380	340
8.	-65	4	80	900	20	400	360
9.	212	4	120	960	19	430	320
10.	212	4	120	1200	64	370	360
11.	212	4	40	540	20	460	240
12.	212	4	60	510	28	410	320
13.	TS(4)	30	85	1500	20	440	320
14.	TS(4)	30	120	960	24	370	380
15.	TS(4)	30	40	960	27	420	360
16.	TS(4)	30	110	960	21	430	300
17.	300	72	120	1800+	3	-(5)	-(5)
18.	300	72	100	1800+	3	-(5)	-(5)
19.	300	120	120	3000+	9	-(6)	-(6)
20.	300	120	-	-	-	-(7)	-(7)
21.	300	168	-	-	-	-(7)	-(7)
22.	300	168	-	-	-	-(7)	-(7)
AVERAGES				1157	25	412	340

(1) Intensity was to low
(2) Scope triggered late
(3) Wrong vertical sensitivity on scope
(4) Thermal shock - 5 cycles, each cyle consisting of 3 hours at -80 F followed immediately by 3 hours at 212 F.
(5) Base plugs blew out.
(6) Leadwires were blown out of base plug.
(7) Not fired because of anticipated similar results to Units 17, 18, and 19.

TABLE VII

PRESSURE-VS-TIME DATA FOR PROPELLANT N-1825

Unit No.	Storage Temp. (° F)	Storage Time (Hrs.)	Ignition Time (μsec.)	Peak Pressure Chamber I (psi)	Time-to-Peak Chamber I (ms)	Peak Pressure Chamber II (psi)	Time-to-Peak Chamber II (ms)
1.	Ambient	-	110	1080	33	- (1)	- (1)
2.	Ambient	-	110	1110	39	200	800
3.	Ambient	-	100	900	24	160	850
4.	Ambient	-	100	930	27	230	800
5.	-65	4	80	900	85	270	760
6.	-65	4	120	870	46	200	840
7.	-65	4	120	990	80	240	760
8.	-65	4	80	900	41	230	800
9.	212	4	120	900	31	290	720
10.	212	4	120	870	24	280	700
11.	212	4	80	1080	20	290	600
12.	212	4	120	920	68	300	660
13.	TS(2)	30	120	990	72	240	700
14.	TS(2)	30	100	1020	30	280	700
15.	TS(2)	30	120	1080	23	280	640
16.	TS(2)	30	120	900	34	260	700
17.	300	72	80	1380	-(3)	200	640
18.	300	72	100	900	45	230	800
19.	300	120	120	960	35	260	640
20.	300	120	90	1140	77	250	600
21.	300	168	120	930	39	240	740
22.	300	168	100	1320	37	250	640
AVERAGES				1003	43	246	719

(1) Defective film
(2) Thermal Shock - 5 cycles, each cycle consiting of 3 hours at -80° F followed immediately by 3 hours at 212° F.
(3) Electronic counter did not stop

1.9.4 All units containing compositions N-5 and N-1825 functioned normally. Composition N-1801 Mod A functioned properly except when it was stored at 300° F. The two units stored for 72 hours at 300° F blew out the base plugs. One unit was fired after 120 hours at 300° F with the base plug physically contained. The leadwires were blown from this unit, thus allowing leakage. During each of these tests the pressure ahead of the burst diaphragms was in excess of 3,500 psi. The three remaining gas generators which had been stored at 300° F were not fired because the test fixture was not designed to withstand such high pressures.

1.9.5 The pressure-vs-time test results show that:

a. Composition N-1825 gives more reproducible results with respect to pressure-vs-time than composition N-5.

b. Composition N-1801 Mod A is unsuitable for 300° F applications.

c. Time-to-peak pressure on N-1825 is 719 ms average compared to 531 ms average for N-5.

d. Gas output of N-1825 is 541 cc/gm compared to 646 cc/gm for N-5.

1.10 Combustion Gas Analysis. A gas analysis to determine the gases produced upon the combustion of the propellants was conducted since the gas generators utilizing the propellant will be used in the automatically activated zinc-silver oxide batteries employing the Signal Corps metal tube electrolyte reservoir activating system. It was considered that large quantities of acidic gases would be detrimental to this system. The total quantitative analysis of the combustion

TABLE VIII

PERCENTAGE AND COMPOSITION OF COMBUSTION GASES

Percentage of Propellant Formulations

Gases	N-5	N-1801 Mod A	N-1825
H_2	9.5	11.5	12.9
H_2O	16.7	10.9	17.4
CO	40.5	16.8	21.4
CO_2	16.9	10.8	9.7
N_2	14.7	31.4	20.7
*HCl	-----	18.8	15.5
CH_4	1.5	-----	1.8
O_2	0.2	trace	0.6

* Total acidity expressed as HCl

gases produced by compositions N-1801 Mod A and N-1825 was obtained by integrating the results of three separate analyses. The three contributing analyses were: (1) the H_2O determination, (2) the HCl determination, and (3) the chromatographic analysis of the other component gases (H_2, N_2, CO, CO_2, CH_4, and O_2). H_2O and HCl were determined separately due to the fact that they could not be clearly identified on the chromatograph. Other methods such as bubbling the combustion gases through KOH, NaOH, and precipitation tests using $AgNO_3$, will be used to confirm the HCl content of the gases. The analysis of N-5 combustion gases was conducted in the same manner with the exception of the HCl determination which was not required. A detailed description of the three analyses and the manner in which the results were integrated to obtain the complete analysis can be found in Appendix D. Table VIII gives the results of the combustion gas analyses for the three propellant compositions.

1.11 Test Fixture Evaluation.

1.11.1 Standard 950 cc gas generators were tested in the test fixture shown in Figure 6. The purpose of these tests was to establish the instrumentation setup and pressure-versus-time traces for standard units in this test fixture in order that test data from gas generators loaded with new propellants could be compared with that of standard units.

1.11.1.1 Figure 7 is a schematic diagram of the instrumentation setup established during the firing of the first five units. The initial

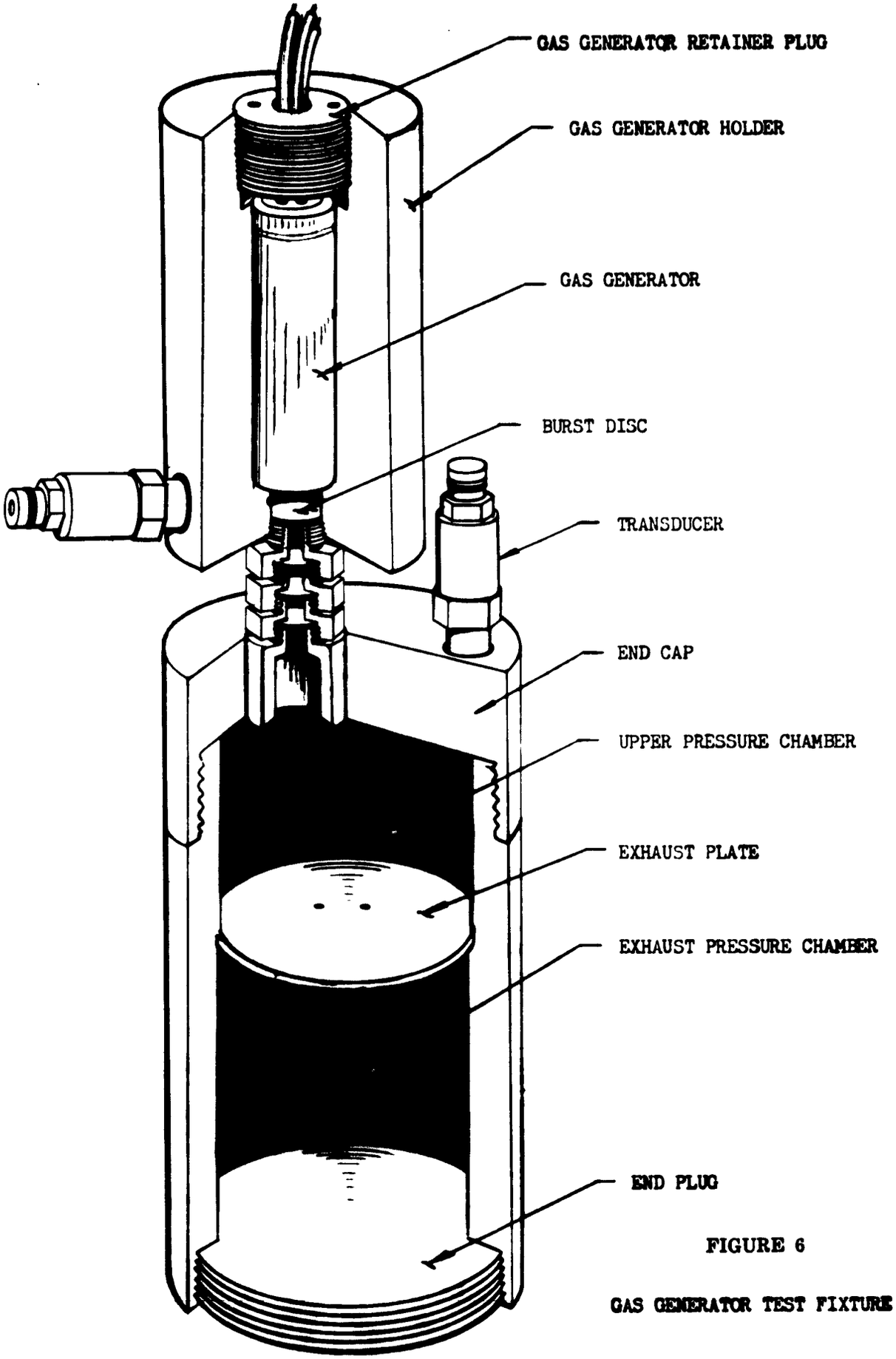


FIGURE 6

GAS GENERATOR TEST FIXTURE

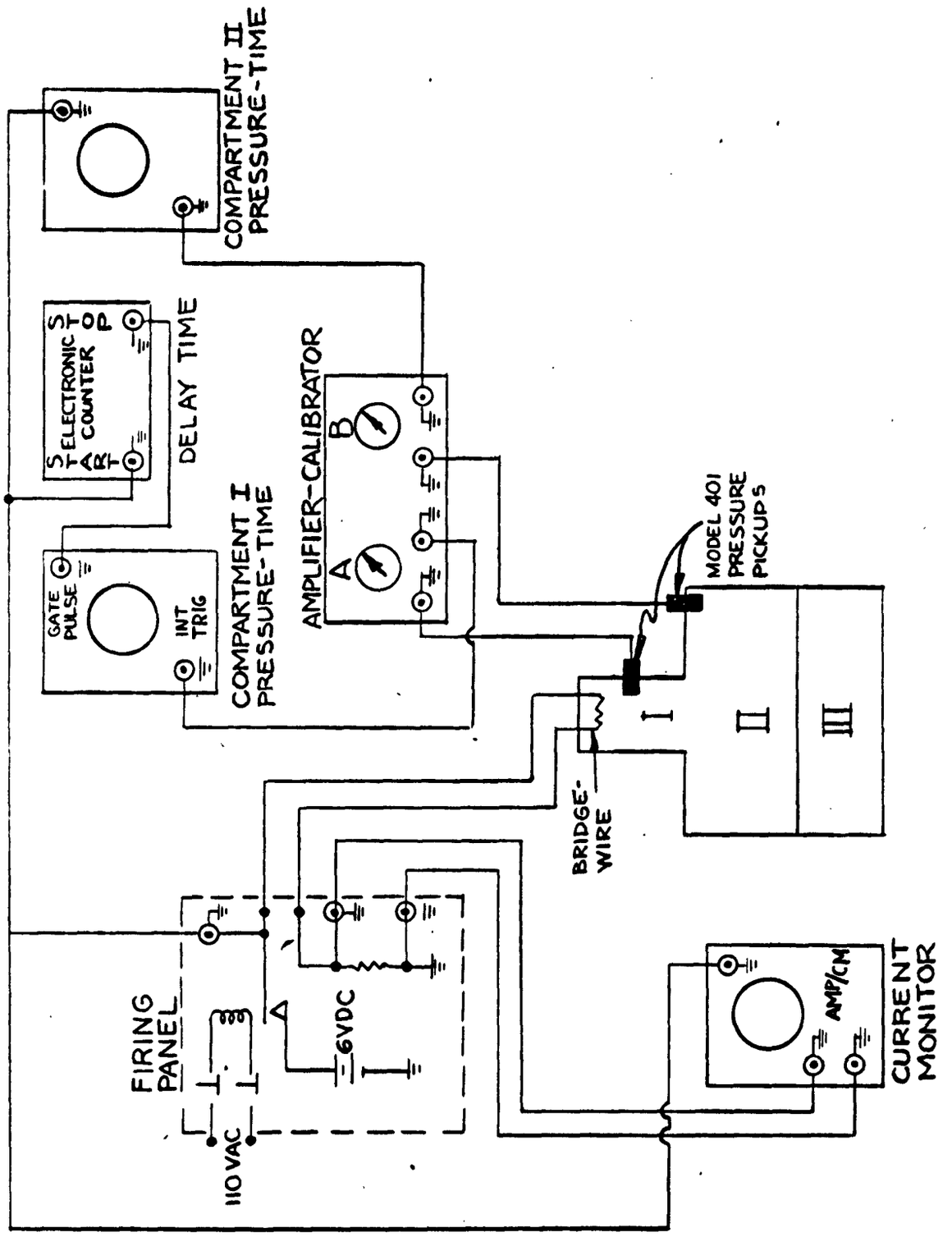


FIGURE 7

INSTRUMENTATION SCHEMATIC

five units did not yield pressure-versus-time curves on scope No. 1. The pressure rise and rupture of the disks occurred at such rapid rates after ignition that the sweep speed on the scope must be fast (5 ms/cm). An electronic counter was, therefore, placed in the instrumentation setup. The counter is triggered by the firing pulse. Oscilloscope No. 1 is triggered by the pressure rise in compartment 1 of the test fixture and a pulse from the oscilloscope then stops the counter. The time on the counter is then added to the time reading from the trace on oscilloscope No. 1 to determine time from firing pulse.

1.11.1.2 Ten standard 950 cc gas generators were tested at ambient conditions. Subsequent testing showed that the test fixture leaked at the welded joint which holds the exhaust plate in place (See Figure 6). This leakage voided the data which had been obtained previously.

1.11.1.3 As a result of the above testing, the test fixture was redesigned and the welded joint was replaced with O-rings on end side of the exhaust plate as shown in Figure 5. The redesigned fixture was checked by filling compartment III with water and pressurizing compartment II with 150 psi air. No leaks were detected.

1.11.1.4 Ten additional standard 950 cc gas generators were tested to characterize the test fixture pressure-versus-time data. Table IX shows the results of this testing. These tests show that an

average peak pressure of 113 psi in an average time to peak of 850 milliseconds is required to duplicate the standard gas generators in this test fixture.

1.11.2 In order to more closely simulate with the test fixture the actual conditions experienced in the zinc-silver oxide batteries, the same type of copper burst diaphragms as used in the BA-472/u batteries were procured from Eagle-Picher, along with information concerning the burst pressures of the diaphragms. These diaphragms burst at the following pressures when used in the BA-472/u batteries: Nos. 1 and 2 burst at 800 psi and Nos. 3 and 4 burst at 1000 psi with an overall range of burst pressures from 600 to 1300 psi. Based on this information, the burst disk adaptors were machined to allow the copper diaphragms to burst at similar pressures. Using nitrogen pressure and the test fixture (Figure 5), the diaphragms burst as shown in Table X.

TABLE IX

PRESSURE-VS-TIME FOR TEST FIXTURE EVALUATION

USING STANDARD 950 CC GAS GENERATOR

Propellant - N-5
Ignition - From 6 V DC
Temperature - Ambient

Unit No.	Ignition Time (μsec.)	Peak Pressure Chamber I (psi)	Time-to-Peak Chamber I (ms)	Peak Pressure Chamber II (psi)	Time-to-Peak Chamber II (ms)
1.	300	(1)	(1)	50(2)	1600
2.	340	1140	33	140	800
3.	300	1120	82	95	800
4.	500	1190	34	110	950
5.	400	1200	29	110	800
6.	300	1230	27	130	750
7.	220	1300	28	(1)	(1)
8.	(1)	1330	26	(3)	(3)
9.	760	1175	22	70	1100
10.	780	870	22	140	750
Average	433	1173	34	101	969

(1) Scope did not trigger
(2) Test fixture leaked
(3) Wrong sweep speed on scope

TABLE X

BURST PRESSURES FOR COPPER DIAPHRAGM

Test No.	Adaptor No. 1	Adaptor No. 2	Adaptor No. 3	Adaptor No. 4
1.	650	725	1075	1125
2.	650	750	1075	1175
3.	685	790	1075	1090
4.	650	790	1075	1175
5.	650	725	1075	1025
6.	650	750	1075	1090
7.	*	*	1075	1000
8.	*	*	1075	1050
9.	*	*	1075	1200
10.	*	*	1100	1050
Average	656	755	1077	1098

* No test

2. CONCLUSIONS.

2.1 General. The following conclusions have been reached from the work conducted during the second quarter:

a. As a result of the vendor survey UMC will conduct propellant evaluations to determine whether or not the thermal stability of the two commercially available propellants (Thiokol and B. F. Goodrich) will equal or surpass that of UMC propellant N-1825 (See paragraph 1.4).

b. Propellant N-1801 Mod A is unsuitable for use after storage at 300° F.

c. Propellant N-1825, prepared by UMC, has exhibited a small percentage weight loss during thermal stability testing at 300° F for one week. In addition, this propellant has exhibited low gas evolution when stored at 300° F for one week as compared to other propellants tested, satisfactory gas output, and more reproducible pressure-versus-time results than the N-5 standard propellant. As a result of the preliminary tests of propellant N-1825, it is anticipated that this basic formulation will be capable of meeting the requirements of the Signal Corps Technical Requirement SCL-7564 and, therefore, will be investigated further.

d. The UMC sealed match has proven effective for igniting the gas generators under various temperature conditions, including 300° F storage for 168 hours.

e. The redesigned test fixture has proved satisfactory.

3. PROGRAM FOR NEXT INTERVAL.

3.1 Propellant Investigation. During the next interval, it is planned to continue the propellant investigation in order to evaluate Thiokol and B. F. Goodrich propellants which have been located through the vendor survey. These propellants will be tested in accordance with the propellant work plan in Appendix A of this report.

3.2 Ignition Tests. Tests will be conducted on delay electric matches to determine charge weights and delay column heights.

3.3 Gas Evolution. Gas evolution tests will be conducted on larger propellant grains (N-1825) theoretically capable of producing 10,000 cc of gas. These tests will be conducted in sealed pressure capsules.

3.4 N-1825. Propellant formulation N-1825 will be modified and tested to determine the effect of the particle size of the ammonium perchlorate on time to peak pressure and peak pressure. In addition the amounts of ammonium perchlorate and guanidine picrate will be varied to ascertain the effect for manufacturing control purposes and time to peak pressure and peak pressure.

3.5 Extended Storage. Unit will be tested after storage at 300° F for 30 days to further characterize the propellant. Sixteen units will be loaded and stored at 300° F. Four units will be tested each week to determine the effects on pressure versus time characteristics.

3.6 Shock and Vibration. Units will be subjected to shock and vibration to ascertain the effects on the propellant after storage at 300° F and -80° F for seven days.

3.7 Temperature Change. Tests will be conducted to determine the rate of propellant grain temperature change upon removing from high or low temperature storage. This will be accomplished by inserting a thermocouple into the propellant grain in a loaded gas generator and recording grain temperature change and time after removal from elevated or reduced temperatures.

3.8 Exhaust Gas. Tests will be conducted to establish a procedure for determining the amount of HCL in the combustion gases. Methods will include bubbling the combustion gases through standardized KOH or NaOH solutions in addition to precipatation methods using $AgNo_3$.

APPENDIX A

PROPELLANT WORK PLAN

REFERENCE: QUARTERLY PROGRESS REPORT NO. 1

Page 41, Paragraph 2.4.2

SIGNAL CORPS CONTRACT NO. DA-36-039 SC-87362

U. S. ARMY SIGNAL RESEARCH AND DEVELOPMENT LABORATORY

FT. MONMOUTH, NEW JERSEY

UNIVERSAL MATCH CORPORATION

ARMAMENT DIVISION

CRAB ORCHARD OPERATIONS

MARION, ILLINOIS

PROPELLANT WORK PLAN

1. INTRODUCTION

This work plan is to be used for the procurement, preparation, testing, and characterization of candidate propellant formulations which are theoretically capable of meeting the specification requirements of Signal Corps Technical Requirement SCL-7564.

This work plan is to be incorporated into and become a part of the work plan which appears in the appendix of Quarterly Report No. 1 on Signal Corps Contract No. DA-36-039 SC-87362. Refer to Paragraph 2.4, Page 41 of the referenced report.

To characterize the chemical and physical properties of a propellant based on comparative analysis, assuming all test conditions to be equal, some standard must be selected as the reference point. Since N-5 propellant has a proven history in the same gas generator application for which the high temperature stable propellant developed under this contract will be used. N-5 propellant has been selected as the standard. Hence, N-5 propellant will be subjected to all tests cited herein.

It is anticipated that up to five different propellant compositions will be subjected to the tests as outlined herein for comparative analysis.

On completion of the propellant investigation, all information collected will be tabulated and analyzed and a meeting held between UMC and the Signal Corps personnel to discuss the results.

2. PROPELLANT PROCUREMENT REQUIREMENTS

a. The propellant must be capable of withstanding, without degradation, 168 hours (7 days) storage at 300° F.

b. Burn rates to peak pressure must be reproducible within plus or minus 5% over a temperature range of -65° F to 212° F and after (a).

c. Propellant must give reproducible peak pressures and time-to-peak pressures within plus or minus 5% after being subjected to 5 cycles of thermal shock, each cycle consisting of 3 hours at -80° F followed immediately by 3 hours at 212° F.

d. Propellant must produce the rated volume of gas plus or minus 5% after being subjected to any or all of the temperatures discussed above.

e. Propellant must have physical characteristics such that it will withstand vibration of 35 g's, 5-2000 cps, after being stabilized at -80° F to 212° F, when loaded in gas generators, in addition to shock of 250 g's with a rise time of 6 to 11 milliseconds under the same conditions.

f. Propellant must produce a minimum amount of slag.

g. Propellant must have a minimum of 300 cc/gm gas output.

h. Flame temperature must be no greater than 3600° F. (Isobaric)(T_p)

i. Composition of propellant must be supplied and certified.

j. Propellant must be capable of being supplied in grain sizes of .390- to .700-inch diameter and a minimum of 6 inches in length.

k. The use of materials which do not conform to MIL-Spec should be avoided when possible.

3. PROPELLANT BATCH PREPARATION

A sufficient batch of each type propellant selected for tests will be prepared and/or purchased to conduct those tests as required by Sections 4 through 7 of this work plan. The following information will be collected and recorded for each batch of material prepared and/or purchased:

a. Manufacturers Certification of propellant constituents.

b. Chemical analysis of propellant

c. Moisture analysis of propellant

d. Preparation procedures if UMC manufactured

4. RATED GAS OUTPUT

To determine the rated volume of gas produced by the propellant in terms of cc/gram at ambient conditions, the following test will be conducted:

Number of Samples to be Tested	Weight of Sample to be Tested	Method of Test	Measurement to be Taken
3	0.3 gram	Place sample in Parr gas-evolution bomb and ignite	Output of sample cc/gram

f. Compare the results of the candidate propellant samples with the results of N-5 propellant. If candidate propellant samples have similar pressure rises equal to N-5 for the seven day period, the candidate shall be considered to be suitable for further evaluation.

(1) Reference 7 (this work plan)

5. GAS EVOLUTION @ 300° F

The purpose of the tests is to determine the following:

a. Pressure of gases evolved during 300° F temperature soak at various time intervals.

b. The amount of propellant weight loss during 300° F temperature soak for one week in pressure sealed units.

c. The changes in physical qualities of the propellant.

Procedure

a. Load candidate propellant samples (2 each type propellant) in pressure capsules. The pressure capsules are shown in Figure 1. The propellant samples loaded into the capsules shall have: (1) equal diameters, (2) equal weights, (3) length varying to give equal weight, and (4) approximately equal free volume. Any excess volume due to short grains shall be taken up by inserting metal slugs into the capsule.

*b. Place pressure capsules in 300° F oven and record pressures at the end of the first hour and then every 24 hours for 7 days. *N-5 propellant to be subjected to 200° F.

c. Remove the capsules from oven and bleed evolved gas into a closed bomb for transport to the chromatograph. (1)

d. Remove the propellant sample and record weight, length, and diameter and compare with original figures.

e. Examine propellant sample under microscope for porosity and other physical changes, and take photo micrographs of the sample in addition to photo micrographs of propellant samples which have not been temperature soaked.

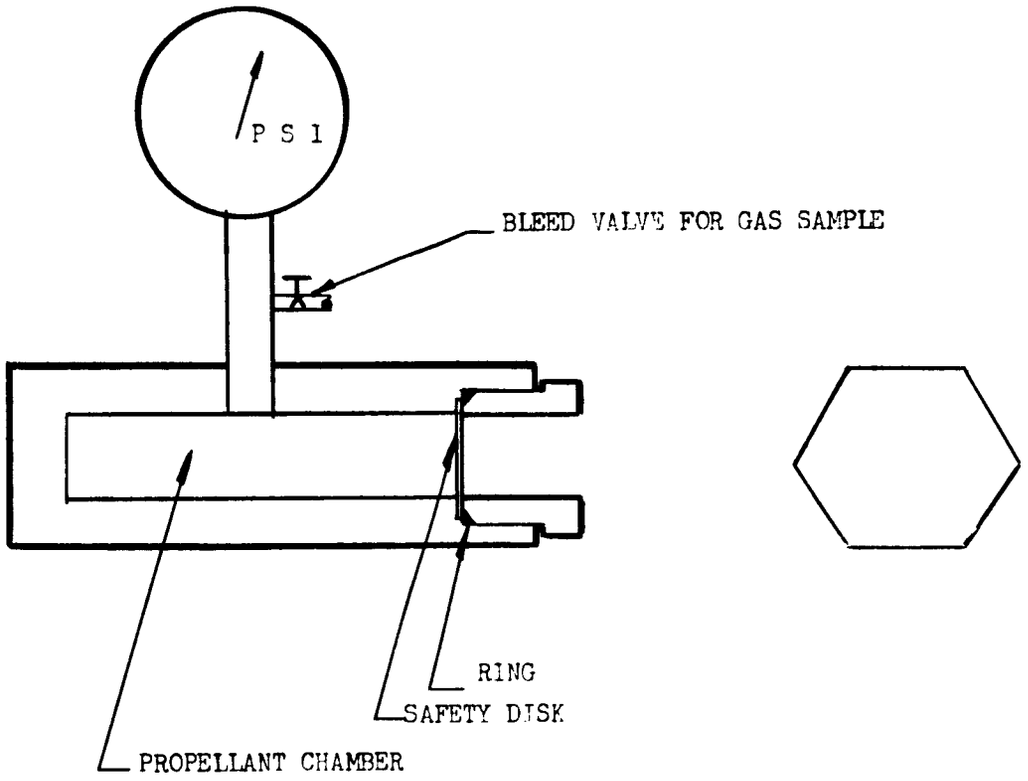


FIGURE 1

PRESSURE CAPSULE

6. PRESSURE-VS-TIME TESTING.

Tests will be conducted to establish the following characteristics of the propellant:

a. Peak pressure

b. Time-to-peak pressure

c. Ignition time

d. Affect of temperature variation on Items a-c.

These tests will be conducted using the test hardware design as shown by Figure 2 and will be loaded as follows:

a. Physical dimension of propellant grain - .390 ± .020 dia. x 1.0 ± .020 length

b. Ignition material - 666 equal amount for all tests

c. Ignition element - one sealed match

The units fabricated will be subjected to the environmental test conditions as shown by Table I.

TABLE I

Number of Units to be Tested	Conditioning Temperature	Time at Temperature
4	Ambient	---
4	-65° F	4 hours
4	212° F	4 hours
2	300° F	72 hours
2	300° F	120 hours
2	300° F	168 hours
4	thermal shock between -80° F and 212° F	3 hrs. each temperature 5 cycles

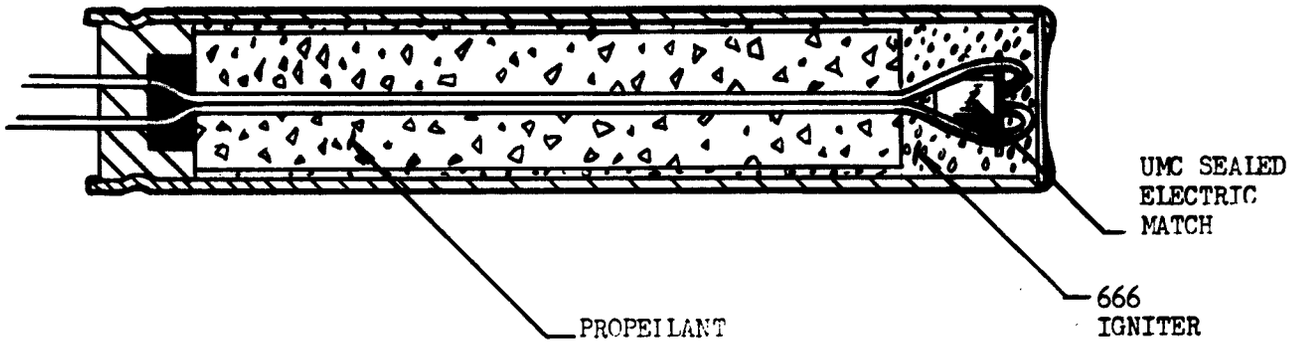


FIGURE 2

GAS GENERATOR

Upon completion of the environmental test conditions, the units will be test fired in the 950 cc test fixture and the following information recorded:

a. Ignition time

b. Time-to-peak pressure

c. Peak pressure

d. Condition of test hardware resulting from (1) temperature, (2) erosion, and (3) slag deposit.

7. CHROMATOGRAPHIC ANALYSIS OF COMBUSTION GASES

A chromatographic analysis of combustion gases will be conducted on those propellants which fulfill the thermal stability, burning rate tolerance, and gas output tolerance requirements to determine the actual gas analysis. The analysis will be conducted as follows:

a. Three-tenths gram of the propellant will be burned in a closed bomb fitted with a valve for removal of the gas.

b. A sufficient quantity of gas to fill the gas chromatograph sampling tube will be released from the bomb while it is immersed in boiling water. (The bomb is immersed in boiling water to vaporize the H_2O.)

c. Each gas expected to be in the combustion products will be passed through the gas chromatograph for calibration of the instruments. Commercially obtained gases will be used. It is anticipated that the following gases will be used in the calibration:

(1) Nitrogen

(2) Carbon monoxide

(3) Carbon dioxide

(4) Hydrogen

(5) Water (distilled)

(6) Hydrogen chloride

(7) Methane

(8) Ammonia

(9) Oxygen

The above procedure will also be used in determining the gas

evolved during the 300° F temperature soak. This test will also be conducted on N-5 propellant and the results compared to the theoretical values given in the SPIA manual.

APPENDIX B

PROCEDURE NO. M-1

STANDARD PREPARATION PROCEDURE FOR FORMULATION N-1801, MOD A

See Quarterly Report No. 1 for photographs of the extruder mixer and associated assemblies. To prepare a 400-gram lot of propellant N-1801, MOD A, the following composition and procedures are used:

Ingredients:

Ammonium Perchlorate	66.5% -	266 gm.
Hycar 1000 X 103	- 15.0% -	60 gm.
Nitroguanidine	- 18.0% -	72 gm.
Carbon Black	- 0.5% -	2 gm.

Ingredient Certifications:

Ammonium Perchlorate - Certified to JAN-A-192, Grade I, Class B

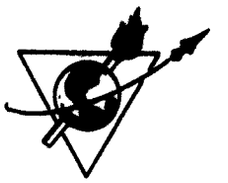
Hycar 1000 X 103 - Certified by the B. F. Goodrich Chemical Company

Nitroguanidine - Certified to JAN-N-494-d-HD-5307

Carbon Black - Certified to JAN-C-306

Preparation of Ingredients:

STEP	PROCEDURE
1.	Dry the ammonium perchlorate for 16 hours at 160° F, then pass through a 50-mesh sieve.
2.	Place the Hycar 1000 X 103 under 200 ml of hexane and allow to soak for 24 hours.
3.	Dry the nitroguanidine for 16 hours at 160° F.
4.	Dry the carbon black for 16 hours at 160° F.



Mixing Procedure:

STEP	PROCEDURE
1.	Place the 60 gm. of pre-softened Hycar 1000 X 103 in a one-quart sigma blade mixer with the 200 ml of hexane in which it was soaked.
2.	Operate the mixer for five minutes to break up the Hycar.
3.	Add the 2 gm. of carbon black and operate the mixer for two minutes to blend this material with the Hycar.
4.	Add the 72 gm. of nitroguanidine and operate the mixer for five minutes to blend this material with the Hycar and carbon black.
5.	Add approximately one half of the ammonium perchlorate and operate the mixer for two minutes.
6.	Add the remaining ammonium perchlorate, and operate the mixer for an additional two minutes.
7.	Scrape down any ingredients which have collected on the mixer walls using a rubber spatula, and incorporate these into the main bulk of the propellant.
8.	Operate the mixer continuously for 30 minutes, then visually inspect the mixture for uniformity.
9.	If uniform in appearance, remove the hexane solvent from the mixture in the following manner: a. Apply a vacuum to the mixer using a water aspirator. b. Operate the mixer for ten minutes with vacuum, then visually inspect the mixture for uniformity

STEP	PROCEDURE
	and degree of dryness.
	c. If hexane is still present, operate the mixer for an additional five minutes while vacuum is applied; then visually inspect the mixture for uniformity and degree of dryness.
	d. If necessary, operate the mixer with vacuum for one-minute intervals. Continue the one-minute intervals until the composition is solvent-free and of a uniform, well-granulated appearance.
10.	Transfer the composition from the mixer to an evaporating dish. Place the material in a 160° F oven for 16 hours to remove residual solvent.

PROCEDURE E-1

Standard Procedure for Extruding Propellant Grains

The following is a guide for extruding propellant grains.

STEP	PROCEDURE
	NOTE: Prior to entering the extrusion bay shut off extrusion press and bleed all hydraulic lines.
1.	Place the metal blank in the bottom of the extrusion chamber and fill the chamber with propellant.
2.	Press the propellant to 560 ± 20 psi and hold the pressure for one minute.
3.	Refill the chamber with propellant and repeat Step 2.
4.	Repeat Step 3 until the desired amount of propellant is in the chamber.
5.	Remove the metal blank from the bottom of the chamber.
6.	Place the die with the monoperforated element in the bottom of the extrusion chamber.
7.	Extrude the propellant by applying 475 ± 25 psi.
8.	Cut in random lengths of 12 to 30 inches for transporting purposes.

APPENDIX C

PROCEDURE G.P.1

Standard Preparation Procedure of Guanidine Picrate

The following procedure will be used to prepare guanidine picrate in approximately a 400 gram batch.

Material

1. Guanidine Nitrate, Reagent Grade
2. Picric Acid with 10 percent H_2O, Reagent Grade

STEP	PROCEDURE
1.	Dissolve 350 grams of picric acid (containing 10 percent water) in 1000 ml. of methyl alcohol with heating and stirring.
2.	Dissolve 170 grams of guanidine nitrate in 200 ml. of distilled water with heating and stirring.
3.	While the picric acid solution is stirring vigorously and almost to the boiling temperature, add the hot guanidine nitrate solution in a fine stream.
4.	After all of the guanidine nitrate solution has been added, continue to heat and stir the guanidine picrate suspension for five minutes, then cool.
5.	Filter the guanidine picrate on a Buchner funnel using No. 1 filter paper.
6.	Wash the product three times with hot distilled water, then three times with hot methyl alcohol.

STEP	PROCEDURE
7.	Remove the guanidine picrate from the filter and place in a 160° F oven for 16 hours to dry.
8.	When the guanidine picrate is thoroughly dry, determine its melting point. To be acceptable the melting point must be $342^{\circ} \pm 2^{\circ}$ C.

PROCEDURE NO. M-2

STANDARD PREPARATION PROCEDURE FOR FORMULATION N-1825

To prepare a 400-gram lot of propellant N-1825, the following ingredients and procedures are used:

Ingredients

Ammonium Perchlorate	- 48.5%	- 194 gm.
Hycar 1000 X 103	- 15.0%	- 60 gm.
Guanidine Picrate	- 36.0%	- 144 gm.
Carbon Black	- 0.5%	- 2 gm.

Ingredient Certifications:

Ammonium Perchlorate - Certified to JAN-A-192, Grade I, Class B

Hycar 1000 X 103 - Certified by the B. F. Goodrich Chemical Company

Guanidine Picrate - Prepared by UMC. There is no specification available for certification.

Carbon Black - Certified to JAN-C-306

Preparation of Ingredients:

STEP	PROCEDURE
1.	Dry the ammonium perchlorate for 16 hours at 160°F, then pass through a 50-mesh sieve.
2.	Place the Hycar 1000 X 103 under 200 ml of hexane and allow to soak for 24 hours.

STEP	PROCEDURE
3.	Dry the guanidine picrate for 16 hours at 160°F, then pass through a 50-mesh sieve.
4.	Dry the carbon black for 16 hours at 160°F.

Mixing Procedure:

STEP	PROCEDURE
1.	Place the 60 gm. of pre-softened Hycar 1000 X 103 in a one-quart sigma blade mixer with the 200 ml of hexane in which it was soaked.
2.	Operate the mixer for five minutes to break up the Hycar.
3.	Add the 2 gm. of carbon black and operate the mixer for two minutes to blend this material with the Hycar.
4.	Add approximately one third of the guanidine picrate, and operate the mixer for two minutes.
5.	Add another third of the guanidine picrate, and operate the mixer for two minutes.
6.	Add the remaining guanidine picrate and operate the mixer for two minutes.
7.	Add approximately one third of the ammonium perchlorate and operate the mixer for two minutes.
8.	Add another third of the ammonium perchlorate, and operate the mixer for two minutes.

STEP	PROCEDURE
9.	Add the remaining ammonium perchlorate and operate the mixer for two minutes.
10.	Scrape down any ingredients which have collected on the mixer walls using a rubber spatula, and incorporate into the main bulk of propellant.
11.	Operate the mixer continuously for 30 minutes, then visually inspect the mixture for uniformity.
12.	If uniform in appearance, remove the hexane solvent from the mixture in the following manner: a. Apply a vacuum to the mixer using a water aspirator. b. Operate the mixer for ten minutes with vacuum, then visually inspect the mixture for uniformity and degree of dryness. c. If hexane is still present, operate the mixer for an additional five minutes while vacuum is applied; then visually inspect the mixture for uniformity and degree of dryness. d. If necessary, operate the mixer with vacuum for one-minute intervals. Continue the one-minute intervals until the composition is solvent-free and of a uniform, well-granulated appearance.
13.	Transfer the composition from the mixer to an evaporating dish. Place the material in a 160°F oven for 16 hours to remove residual solvent.

APPENDIX D

DESCRIPTION OF COMBUSTION

GAS ANALYSES

H_2O Determination

The mole percent of H_2O in the combustion gases of the propellants was determined through a modification of the gas output test (paragraph 1.7). The manner in which this data was obtained consisted of burning an accurately weighed propellant sample in the gas evolution bomb, immersing the bomb in boiling water, and recording the gas pressure at 100° C, then immersing the bomb in ice water and recording the gas pressure at 0° C. The pressure differential resulting from the 100° C - 0° C temperature reduction was attributed to condensation of the H_2O present in the gases and was used to determine the mole percent of H_2O present in the gases. The determination was obtained by using the equation:

$$PV = nRT$$

The above equation was solved for n (moles) at 100° C and at 0° C, using the recorded pressures at each temperature and the known volume of the bomb. The difference in the number of moles of gas at the two temperatures was considered to be the number of moles of H_2O present. Using the calculated value for the total moles at 100° C and the reduction in moles at 0° C due to H_2O condensation, the mole percent of H_2O was determined.

HCl Determination

The HCl content in the combustion gases of N-1801 MOD A and N-1825 was determined by titrating solutions of the gases produced by known weights of

propellant against a standardized sodium hydroxide solution. During the neutralization, the pH of the solution was continually monitored on a Beckman Zeromatic pH meter. The determination consisted of burning accurately weighed samples of each propellant in a Parr calorimeter bomb containing 30 ml of water in the bottom to dissolve the HCl produced. The bomb was allowed to stand for approximately 15 minutes, then opened and the solution filtered. The solution was then titrated against a standard sodium hydroxide solution while the pH was monitored. The pH was then plotted against the milliliters of sodium hydroxide solution to determine the end-point of the titration. The number of moles of HCl present in the solution was then calculated. The mole percent of HCl could not be determined from the data obtained in this test alone, since the total number of moles of gas produced by the propellant sample was not known.

Chromotographic Analysis

Apparatus

Perkin Elmer Model 154-D Gas Chromatograph Column

Perkin Elmer - Column I - 1/4"-2 meter filled with calcium aluminum silicate

Perkin Elmer - Column J - 1/4"-2 meter filled with silica gel

Carrier Gas - Helium

Detector Voltage - 7 volts

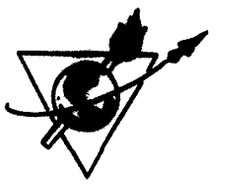
Temperature Column - 28° C

Gas Flow Rate:

Column I - 200 cc/min-p = 15 psi

Column J - 75 ml/min-p = 7 psi

Sample Size - 0.25 ml



Qualitative Analysis

Standardization was accomplished by measuring retention time from injection of sample to center of election peak.

Column I

Gas	Retention Time-short div.
O_2	3
H_2	1
CO	42
N_2	8.5
CH_4	13
HCl	Could not be determined, never elected

Column J

Gas	Retention Time-short div.
H_2	1.5
O_2, N_2	3
CO	4
CH_4	6
NO	Not tested(1)
CO_2	59

(1) Literature indicates it would peak between CH_4 and CO_2.

Quantitative Analysis

Quantitative estimation of the components found in the burned gases was accomplished by the area ratio method. The area under each peak was

determined by multiplying the peak height times 1/2 peak width at 1/2 peak height. Each area was then divided by its respective thermal conductivity difference (difference between thermal conductivity of respective gas and thermal conductivity of helium). This corrected the areas for any difference in the conductivity between the gases. This correction factor was not applicable by itself to hydrogen though. The corrected area of a 0.25 ml sample of hydrogen was determined to be 6.3 times the calculated area. This factor makes H_2 area approximately equal to area 0.25 ml of other gas yields.

Area H_2 (corr for) = 0.92 sq mm

Area CO (corr for) = 5.78 sq mm

$\frac{5.78}{.92}$ = 6.3 (correction factor for H_2 area to make it approximately equal in area to other gases)

Thermal Conductivity of Several Gases

Gas	$\lambda \times 10^5$
CO_2	3.39
CO	5.42
He	33.60
H_2	39.60
CH_4	7.20
N_2	5.68
P_2	5.70

λ = g cal/(sec)(sq cm)($^\circ$C/cm)

Experimental Data on Propellants

N-5

Area (mm^2)	Gas	Mole %
0.84	H_2	11.4
1.30	N_2	17.7
3.58	CO	48.6
1.49	CO_2	20.2
0.14	CH_4	1.9
0.02	O_2	0.3

Areas for H_2, N_2-O_2, CO, CO_2, CH_4 determined on Column J. O_2-N_2 was resolved and ratio determined on Column I. Ratio was then applied to the combined area from Column J to yield the percent N_2 and O_2.

N-1825

Area (mm^2)	Gas	Mole %
1.57	H_2	19.3
2.52	N_2	30.9
0.07	O_2	0.9
2.59	CO	31.8
1.18	CO_2	14.5
0.22	CH_4	2.7

Areas for H_2, O_2-N_2, CO, CO_2, and CH_4 were determined on Column J. Ratio of O_2 to N_2 was determined on Column I.

N-1801 MOD A

Area (mm^2)	Gas	Mole %
1.68	H_2	16.4
4.58	N_2	44.6
2.43	CO	23.6
1.59	CO_2	15.5

Areas for all gases determined on Column J.

Sample Calculations

N-1801

Gas - H_2

Peak Height - 11 mm

1/2 peak width at 1/2 peak height - .3 mm

Area at sensitivity 8 = 3.3 mm^2. As other areas were recorded at a sensitivity of 16, it is necessary to divide 3.3 by 2. The area at a sensitivity of 16 would then be 1.6 mm^2. To correct for differences in λ, subtract λ of He from λ of H_2.

39.6 of H_2 x 10^5

33.6 of H_2 x 10^5

and divide the area by this value

$$\frac{1.6\ mm^2}{6} = 0.266\ mm^2$$

and due to instrumental effects, multiply this by the area correction factor of H_2, 6.3.

$0.266\ mm^2 \times 6.3 = 1.68\ mm^2$

Now, the total of areas for N-1801 is 10.28 mm^2.

$$\text{Mole \%} = \frac{1.68}{10.28} \times 100 = 16.4$$

Integration of Data from H_2O, HCl, and Chromatographic Determinations

In order to integrate the data from the three analyses, it was necessary to consider that all three had been conducted on the same sample of propellant, thereby giving a constant value for the total moles of gas. Since the total molar gas evolution was determined by the H_2O analysis, this sample was selected as representative and was used throughout the calculations on each propellant. The complete series of calculations used to determine the mole percent of each component in the combustion gas from N-1825 is presented in the following paragraphs as an example of the method.

N-1825 Combustion Gas Analysis

Percent H_2O

The pressures recorded in gas output test No. 1 were the following:

a. At 100° C - 76 psi (5.17 atmosphere)

b. At 0° C - 46 psi (3.13 atmosphere)

A sample weight of 0.30753 gm was burned in a bomb with 40 ml volume. The equation

$$PV = nRT$$

is solved for n at 100° C and at 0° C.

100° C - $PV=nRT$

$(5.17)(40) = n(82.057)(373)$

$n = 0.00677$ moles

0° C - $PV = nRT$

$(3.13)(40) = n(82.057)(273)$

$n = 0.00559$ moles

Therefore: 0.00677
-0.00559
0.00118 moles of H_2O

$\frac{0.00118}{0.00677}$ X 100 = 17.4 mole percent H_2O

Percent HCl

24.9 ml of 0.1198 normal sodium hydroxide was required to neutralize the NCl produced by 0.85332 gm of N-1825. The number of moles of HCl present is determined as follows:

n = (ml)(N)

n = (24.9)(0.1198)

n = 0.00292 moles of HCl

Determining the number of moles of HCl produced by 0.30753 gm of N-1825 (the sample weight used in the H_2O determination):

$\frac{0.00292}{X} = \frac{0.85332}{0.30753}$

X = 0.00105 moles of HCl

$\frac{0.00105}{0.00677}$ x 100 = 15.5 mole percent HCl

Percent H_2, N_2, CO, CO_2, CH_4, and O_2. The molar ratio of H_2, N_2, CO, CO_2, CH_4, and O_2 was determined by the chromatographic analysis found to be:

H_2 = 1.5 X 6.3 = 1.57 = 19.3% of total gases analyzed by chromatogr

N_2 = $\frac{70.3}{27.92}$ = 2.52 = 30.9% of total gases analyzed by chromatogr

CO = $\frac{73.0}{28.18}$ = 2.59 = 31.8% of total gases analyzed by chromatogr

CO_2 = $\frac{35.7}{30.21}$ = 1.18 = 14.5% of total gases analyzed by chromatogr

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As a result of the vendor survey UMC will conduct propellant evaluations to determine whether or not the thermal stability of the two commercially available propellants will equal or surpass that of UMC propellant H-1825.

Propellant H-1801 Mod A was found unsuitable for use after storage at 300° F due to decomposition.

Propellant H-1825 prepared by UMC exhibited a small percentage weight loss during thermal stability testing at 300° F for one week. In addition this propellant exhibited low gas evolution when stored at 300° F for one week as compared to other propellants tested. It also displayed satisfactory gas output and more reproducible pressure-vs-time results (time-to-peak pressure and peak pressure) than the H-5 standard propellant. As a result of preliminary testing, it is anticipated that H-1825 propellant will be capable of meeting the Signal Corps technical requirement SCL-7564. This propellant will be investigated further.

The UMC sealed match has proven effective for igniting the gas generators under various temperature conditions, including 300° F for 168 hours. The redesigned test fixture has proved satisfactory.

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