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FRL-TN-83

EVALUATION OF NITROCELLULOSE-BASE  
SPONGE PROPELLANTS PREPARED WITH THE  
USE OF BLOWING AGENTS

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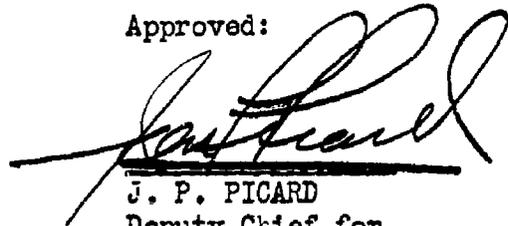
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## SUMMARY

The preparation of sponge propellants by inclusion of a chemical blowing agent in a nitrocellulose-solvent system was investigated. Using fibrous nitrocellulose very poor grains were obtained due to the difficulty in removing the solvent. The use of nitrocellulose in the form of ball powder permitted the inclusion of all the ingredients with suitable (non-explosive) plasticizer in a mold and thorough dispersion of the blowing agent in the mix. The grains were then formed by simultaneously foaming and curing at 120°C.

Although a certain degree of porosity was obtained the finished grains were unsatisfactory due to the presence of large voids within the matrix. Limited closed bomb data indicated that porosity had no apparent effect on the burning rate. The thermal stability obtained by the 134.5°C Heat Test was unsatisfactory. This has been attributed to the high temperature to which the grains were subjected during the foaming and curing process.

## CONCLUSIONS

The preparation of nitrocellulose-base sponge propellants by the use of blowing agents is not promising. It appears to be difficult to obtain sponge grains having a uniform pore structure. In addition extensive work would have to be done to evaluate blowing agents which are compatible with nitrocellulose and which have blowing action at a relatively low temperature.

## RECOMMENDATIONS

It is recommended that work on the preparation of nitrocellulose-base sponge propellant grains by the use of blowing agents be terminated.

## INTRODUCTION

Under Contract DAI-33-019-ORD-(P)-23 the Defense Research Division of the Firestone Tire and Rubber Company investigated various methods for the preparation of a nitrocellulose-base sponge propellant. As the name indicates this type of propellant deals with a composition that has been given a cellular structure similar to sponge rubber. The obvious objective in using sponge propellant for fast burning is the utilization of the large exposed surface area. For the adjustment of this surface area, control of the size and type of cell is required.

The objectives of this project were:

- (a). The development of a sponge propellant with uniformity of pore structure and composition, a density above 0.70 g./cc., and reproducibility of burning characteristics.
- (b). The development of means of varying and controlling the "apparent burning" rate of the sponge propellant.

Various methods for producing a sponge structure in cellulose nitrate were evaluated as follows (Ref 1):

- (1). Incorporating a water soluble material in a nitrocellulose-solvent system and extracting the water soluble material and solvent by means of a leaching bath.
- (2). Dissolving a gas or low boiling liquid in a nitrocellulose-solvent system and foaming by reducing the pressure or evaporating the liquid.
- (3). Entrapping air in a nitrocellulose-solvent system by means of a rapid mixing such as used in some commercial foaming techniques.
- (4). Using bifunctional agents such as tolylene diisocyanate to achieve a molecular cross-linkage of a gelled nitrocellulose matrix while simultaneously entrapping a gas.
- (5). The inclusion of a chemical blowing agent in a nitrocellulose-solvent system which on heating decomposes with the evolution of a gas to produce a cellular structure.

Techniques for foaming nitrocellulose are limited because of the inherent properties of this material. For example, nitrocellulose is not thermoplastic and the decomposition temperature is relatively low. Initial surveys of the above methods resulted in the selection of a salt-solvent system as the most promising method of producing the desired product.

A procedure was developed by Firestone for preparing sponge grains by the "nitrocellulose-salt-solvent" system (Method 1) using sodium chloride as the salt and acetone as the solvent. Ballistic and stability evaluation of the sponge grains were carried out by Picatinny Arsenal. The results obtained have been reported elsewhere and need not be repeated here (Ref 1, 2, 3).

The grains formed by Method 2, 3 and 4, had cells or voids which were irregular and difficult to control and, on removing the solvent, a distorted (and frequently degraded) grain was obtained. The development of a sponge propellant by these methods did not appear feasible and work was abandoned.

The incorporation of a blowing agent in a nitrocellulose-solvent system (Method 5) showed some merit. A blowing technique as compared to salt extraction would be faster. It would permit a greater range of product density, and it would provide a choice of closed cell structure or open, connected cell structure. This technique was extensively evaluated and forms the subject of this report.

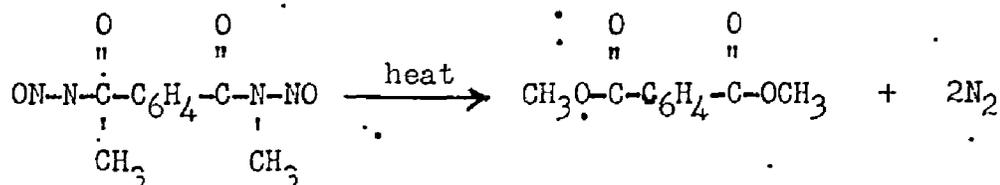
## RESULTS AND DISCUSSION

The use of a blowing agent to produce a cellular structure within a nitrocellulose grain introduces two problems. The process utilizes the evolution of gas by some material on heating or other activation method. Due to the low thermal stability of nitrocellulose it is essential that the activation energy of the blowing agent be as low as possible. The second problem is that both the blowing agent and its decomposition products must be compatible with nitrocellulose and any explosive plasticizer which may be used.

Most commercial blowing agents have a fairly high activation energy, and are, therefore, not suited for use in a nitrocellulose system. However, two compounds, N,N'-dimethyl-N,N'-dinitrosoterephthalamide (BL-353) and benzene disulfohydrazide (Porofor 13/CE) were found which showed strong blowing action at 95-100°C in about twenty minutes.

To determine the effect of these two blowing agents on the stability of nitrocellulose, compatibility evaluation was carried out by Picatinny Arsenal using samples of the blowing agent obtained from Firestone (Ref 4). Nitrocellulose films were prepared (from acetone solution) containing 1% blowing agent, with and without stabilizer, and evaluated by the standard 134.5°C Heat Test. The data reported in Table I shows that the products remaining after decomposition of the BL-353 were compatible with nitrocellulose, while the residue from Porofor 13/CP had a marked degrading effect on the nitrocellulose. In this evaluation it was assumed that the temperature of the Heat Test (134.5°C) was high enough to insure complete blowing of the compounds under investigation. Subsequent work by the contractor and this Laboratory showed that this was not true, and that the blowing action in simple films was quite unpredictable.

BL-353 (N,N'-dimethyl-N,N'-dinitrosoterephthalamide) is a blowing agent that decomposes on heating to give gaseous products consisting of 99% nitrogen plus some carbon dioxide (Ref 5,6). The compound is considered a weak ignition explosive, and is somewhat sensitive to impact and friction. For use as a blowing agent it is coated with approximately 30% by weight of white mineral oil which adequately desensitizes it with minimal lowering of storage stability or its blowing performance. In the course of the blowing action in an inert medium the decomposition takes the following course:



The solid product remaining after blowing, methyl terephthalate, would not be expected to cause instability of nitrocellulose or other nitrate esters.

The nature of the products formed during blowing of Porofor 13/CP (benzene disulfohydrazide) are unknown to the writers. However, on the basis of its structural formula, C<sub>6</sub>H<sub>4</sub>(SO<sub>2</sub>NNH<sub>2</sub>)<sub>2</sub>, it is believed that these decomposition products would be acidic compounds of sulfur. Such compounds are known to be incompatible with nitrate esters. Therefore, no further work was carried out with this blowing agent.

As previously stated, in nitrocellulose film studies with BL-353 it was difficult to control the blowing action which was quite unpredictable. These films were prepared by dissolving the nitrocellulose in a solvent, pouring the viscous solution on a glass plate, and evaporating the solvent at ambient temperature. The results indicated that the blowing action was lost during drying of the solvent from the film. An attempt was then made to use high boiling point solvents such as the cellosolves and carbittols. A procedure was devised in which the ingredients were thoroughly colloided in a Baker-Perkins Mixer, and then molded in a polyethylene-lined glass cylinder. After removing the sample from the mold it was placed in a boiling water bath to initiate the blowing action. This technique resulted in the preparation of a porous grain, but it was difficult to prevent distortion of the grain.

The work described above was carried out using either fibrous military grade (12.6 %N) or lacquer grade (11.7 %N) nitrocellulose. Attention was then directed to the use of nitrocellulose in the form of single-base ball powder. It was proposed that this form of nitrocellulose might be foamed by a method similar to that used with commercial plastics. This technique would permit the inclusion of all the ingredients with suitable plasticizers in a polyethylene-lined mold and thorough dispersion of the blowing agent throughout the fluid mix. Since the curing step involves heating it would be possible to foam the mixture during the curing phase. The simultaneous gas evolution and curing of the plastisol particles would result in encapsulation of gas cells providing a foamed structure.

Using this technique the Firestone Company prepared a number of grains and submitted them to Picatinny Arsenal for closed bomb evaluation, measurement of pore size distribution, and stability determination. Although these foamed samples were prepared with inert plasticizers resulting in poor thermochemical properties, it was anticipated that in future work the inert plasticizer would be replaced, at least in part, by high energy plasticizers such as nitroglycerin to permit their adoption as propellants. The composition of these grains is given in Table II. It is evident from a consideration of the data that a wide variety of foam densities can be obtained by judicious selection of plasticizer system and concentration of blowing agent.

#### Closed Bomb Evaluation:

As explained above the grains were prepared with inert plasticizers. This was necessary for reasons of safety, and until operating procedures and stability parameters could be evaluated. Therefore, these grains were not expected to have favorable thermochemical properties. However, closed bomb

evaluation was carried out to determine the effect, if any, of grain porosity and to have data for comparison with grains prepared with explosive plasticizers.

Table III gives data for the closed bomb evaluation of propellant grains No. 62 and 63 in comparison with standard M-10 propellant. In view of the high percentage of unoxidized carbon in the sponge grains, it is not surprising that the relative force is so low. No valid data is available for comparison, but it appears that the porosity of the grains had no marked effect on the burning rate.

#### Pore Size Distribution:

In order to determine the pore size and homogeneity of the propellant samples, Grains No. 64 and 65 were cross sectioned into increments approximately 1/8 inch thick. From these cross-sectioned slices wafers, 0.55 inch in diameter, were cut with a cork borer and examined microscopically. Photographs of representative samples of these wafers are shown in Figure 1.

The microscopic examination revealed a grain composed of a combination of open and closed pore structure. Though irregular the geometry and distribution of the small pores were similar throughout any one grain. However, both grains contained a number of widely scattered large voids up to approximately 1/4 x 1/8 inch in size. Similar voids were observed in other grains (Table IV). Whether these voids were formed during the blowing process or by entrapped air during casting is unknown. It is possible that the gas produced during the blowing action migrated into pockets forming large fissures rather than the desirable uniformly encapsulated gas cells.

Each of the grains examined contained a considerable amount of green colored impurity having a melting point range of 110-120°C. (Item 4, Fig 1). The nature of this impurity is unknown. Methyl terephthalate, the solid product formed by the thermal decomposition of BL-353, has a melting point of 140°C.

#### Thermal Stability:

The thermal stability data for the sponge propellant grains, as determined by the standard 134.5°C Heat Test, are given in Table V. It is interesting to note that the data falls into two distinct groups. One set of grains (No. 63-65) showed 20 minutes to Salmon Pink and about 130 minutes

to Red Fumes. The other grains showed 25 minutes to Salmon Pink and 30 minutes to Red Fumes. The primary difference is that grains No. 63-65 had a lower percentage of plasticizer than the other grains. The nature of the plasticizer, or the concentration of the blowing agent, appeared to have no effect on the stability.

For standard single-base propellants containing 1% diphenylamine as stabilizer, the minimum test period to Salmon Pink at 134.5°C is 40 minutes. Therefore, on the basis of their low Salmon Pink value (20-25 minutes) the sponge propellant grains are considered unstable, even though there was no explosion in 300 minutes. The low stability data for these grains was surprising since prior work in this Laboratory (Table I) had shown that nitrocellulose films containing 1% BL-353, with or without stabilizer, were stable at this elevated temperature. The grains were prepared by Firestone without the addition of stabilizer (Table II). However, the ball powder used in these grains contained 2% nitrodiphenylamine, which is equivalent to 1.5% stabilizer in the finished propellant grain.

To resolve the discrepancy in the stability data between the simple nitrocellulose films and the sponge propellant grains a few experiments were carried out to evaluate the stability of ball powder in contact with the blowing agent at 135°C. The results are listed in Table VI.

Since BL-353 is classified as a weak initiator, its incompatibility with ball powder in the dry state is not unexpected. When the ingredients are dispersed in acetone and cast into a film, satisfactory stability is obtained at a low concentration of the blowing agent. Substitution of a high boiling point plasticizer for acetone makes it possible to increase the BL-353 concentration up to 30% without deleterious effect on the Salmon Pink value. The data thus indicates that ball powder and BL-353 are compatible over a wide range of concentrations provided they are dispersed in a suitable matrix. It, therefore, appears that the low Salmon Pink value for the sponge propellant grains is inherent in the propellant system and not due to the composition.

The sponge propellant grains were prepared by curing at either 100°C for one-hour or at 120°C for 30 minutes. They were then subjected to the elevated temperature (134.5°C) of the heat test. It is probable that the initial heating period, required for efficient blowing action, caused some degradation of the nitrocellulose. The standard heat test was, therefore, carried out on a propellant grain in which the nitrocellulose was partly degraded. Therefore, the 20-25 minutes Salmon Pink value is not realistic, since the propellant grains were subjected to much more strenuous heating conditions than is required by heat test specifications. Therefore, due to the necessity of curing

the sponge propellant grains at an elevated temperature, a certain degree of instability will be present in all grains prepared by this procedure.

Although plastisol nitrocellulose propellant grains can be cured at 60°C., a temperature of at least 100°C is necessary to insure decomposition of the blowing agent. Therefore, unless a blowing agent (or activator) can be obtained which has a pronounced blowing action at low temperatures, it does not appear possible to obtain "stable" nitrocellulose sponge propellant grains by this procedure. The few known blowing agents which decompose at low temperatures (Ref 10) do so with the liberation of ammonia which will cause fast decomposition of the nitrocellulose.

## EXPERIMENTAL PROCEDURE

### Preparation of Grains: (Ref 7)

The plasticizer and blowing agent were thoroughly mixed in a beaker after which the ball powder was added and the ingredients thoroughly mixed by hand. Depending on the consistency, the final mix was either molded or poured into a polyethylene lined aluminum tube and cured. Grains Nos. 103 and 104 which were very fluid were cast into a glass mold. Grain No. 87 was kept at room temperature for one hour and then cured for one hour at 100°C. All the other grains were cured at 120°C for 30 minutes.

### Testing of Grains:

The grains were cut longitudinally and transversely for visual examination of pore size distribution. Shavings obtained from the cut surfaces were submitted for stability and close bomb determination.

The 134.5°C Heat Test was carried out in accordance with standard procedures used for single-base propellants (Ref 8).

The closed bomb determination (Ref 9) was carried out by firing the standard and sponge propellants under the same conditions. The burning time and pressures were obtained from pressure-time traces using a Baldwin Strain Gauge as the pressure transducer. A piezoelectric gauge was used for obtaining the relative force and relative quickness values. Both sets of data were obtained simultaneously.

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TABLE I

COMPATIBILITY OF NITROCELLULOSE WITH BLOWING AGENTS

<u>Formulation No.</u>	<u>1</u>	<u>2</u>	<u>3</u>	<u>4</u>	<u>5</u>	<u>6</u>
Nitrocellulose (13.15%N), %	99.0	99.0	99.0	99.0	99.0	99.0
BL-353, %	1.0	1.0	1.0	-	-	-
Porofor 13/CP, %	-	-	-	1.0	1.0	1.0
Diphenylamine (added)	-	1.0	-	-	1.0	-
Ethyl Centralite (added)	-	-	1.0	-	-	1.0
<u>134.5°C Heat Test, min to:</u>						
Salmon Pink	45	45	45	*	40	*
Explosion	300+	300+	300+	10	65	18

\*Sample exploded before methyl violet paper turned Salmon Pink.

TABLE II

COMPOSITION OF SPONGE PROPELLANT GRAINS

<u>Propellant Grain No. (a)</u>	<u>62</u>	<u>63</u>	<u>64</u>	<u>65</u>	<u>72</u>	<u>84</u>	<u>85</u>	<u>87</u>	<u>103</u>	<u>104</u>
Ball Powder, Lot XU-1164, (b) %	75.2	75.2	75.2	75.2	66.6	64.5	64.5	65.4	65.9	65.9
Dibutyl Phthlate, %	7.5	7.5	7.5	7.5	10.5	10.8	10.8	10.9	-	-
Dipropylene Glycol, %	7.5	7.5	7.5	7.5	10.5	10.8	10.8	10.9	-	-
Cellosolve, %	7.5	7.5	7.5	7.5	10.5	10.8	10.8	10.9	16.5	16.5
Triacetin, %	-	-	-	-	-	-	-	-	16.5	16.5
Blowing Agent BL-353, (c) %	2.3	2.3	2.3	2.3	2.0	3.2	3.2	2.0	1.1	1.1
Initial Weight of Ingredients, gm.	53.2	53.2	53.2	53.2	60.1	46.5	46.5	46.5	45.5	45.5
Final Weight of Grain, gm.	52.2	51.7	51.6	51.6	55.5	39.0	38.4	41.2	43.9	43.9
Apparent Density, gm./cc	0.78	0.74	0.74	0.76	0.745	0.757	0.792	0.752	0.72	0.70

(a) Condition of Cure: Grain No. 87: Room temperature for one hour, then 100°C for one hour.  
All the other grains were cured at 120°C for one hour.

(b) Composition of Ball Powder, Lot XU-1164: 98% nitrocellulose (12.6%N), 2% nitrodiphenylamine.

(c) Contains 30% white mineral oil.

TABLE III

CLOSED BOMB DATA FOR SPONGE PROPELLANTS

<u>Comparison Propellant</u>	<u>Sponge Grain Number</u>	<u>Loading Density (gm/cc)</u>	<u>Weight (gm)</u>	<u>Burning Time (millisec)</u>	<u>Max Pressure (psi)</u>	<u>Relative Quickness, % at Pressures, psi.</u>	<u>Relative Force, % at Max. Pressure</u>
M10 (PA-30187)	-	0.1	20	3	17,100	<u>3000</u> 100.0	<u>5400</u> 100.0
	62A	0.1	20	25	10,600	18.4	28.7 60.8
	62B	0.1	20	25	10,600	18.4	28.7 62.5
M10 (PA-30187)	-	0.2	40	6	36,650	<u>6000</u> 100.0	<u>11,500</u> 100.0
	63	0.2	40	17	23,600	29.8	64.0 59.3

NOTE: Samples 62A and 62B were taken from different parts of the same grain.

Unoxidized carbon: Sponge grains, 22.5%  
M10, 4.0%

TABLE IV

MICROSCOPIC EXAMINATION OF SPONGE PROPELLANT GRAINS

<u>Grain Number</u>	<u>Top</u>	<u>Center</u>	<u>Bottom</u>
72	Voids 1/16" in diameter and greater. More porous than center or bottom.	Voids 1/16" in diameter.	Small voids 1/32" in diameter.
85	More porous than bottom. No large voids.	Voids 1/8" x 1/16" and smaller.	Void 1/2" x 1/2" x 1/4" found 1/8" from end of grain.
87	Much more porous than bottom.	One larger void, 1/4" x 1/8" x 3/8".	Hollow cavity towards the center of the grain.
103	Numerous voids 1/16" diameter and larger.	Voids 1/16" and greater. Very porous.	Voids larger than 1/16" present near end of grain.
104	Voids 1/16", 1/8" and 1/4" diameter, depth 1/8".	Same as 103	Same as 103

NOTE: The terms Top and Bottom refer to different ends of the same grain. All measurements are only approximate. A green colored impurity (m.p. 110-120°C) was present in varying degree in all grains.

TABLE V

134.5° C HEAT TEST DATA FOR SPONGE PROPELLANT GRAINS

<u>Grain Number</u>	<u>Minutes to:</u>		
	<u>Salmon Pink</u>	<u>Red Fumes</u>	<u>Explosion</u>
63	20	110	300+
64	20(20)	130(135)	300+(300+)
65	20(20)	135(135)	300+(300+)
72	25	30	300+
85	25	30	300+
87	25	30	300+
103	25	30	300+
104	25	30	300+

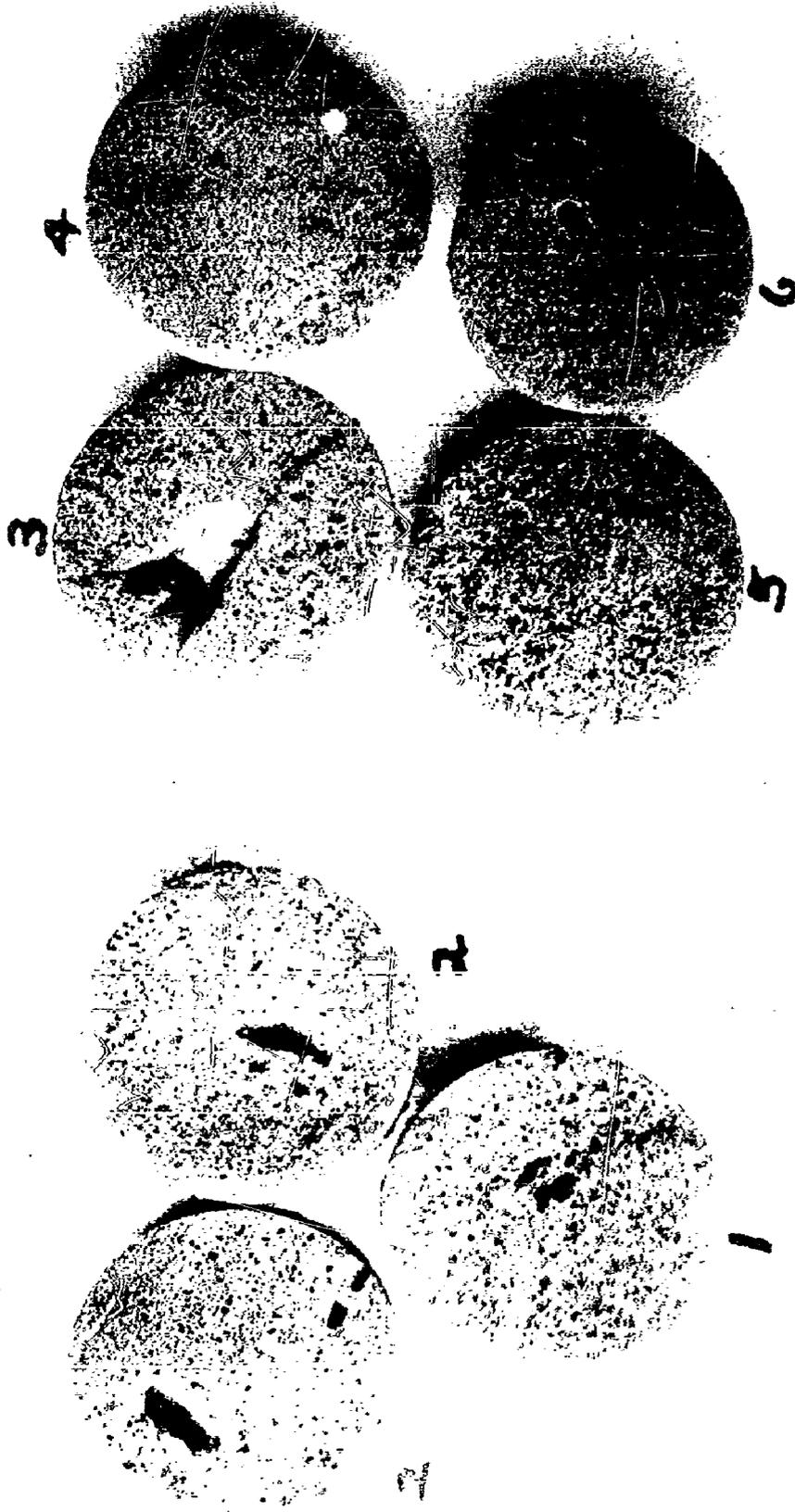
NOTE: With Grains No. 64 and 65 duplicate tests were made with samples taken from the end and center of the grain. With the other grains the samples used for testing were randomly selected.

TABLE VI

COMPATIBILITY OF BALL POWDER WITH BL-353 AT 135°C.

<u>System</u>	<u>% Composition</u>	<u>Remarks</u>
1. BL-353	-	Deflagrated immediately.
2. Mixture: Ball powder BL-353	98 2	Deflagrated immediately.
3. Film cast from acetone: Ball powder BL-353	98 2	No deflagration. Time to Salmon Pink - 45 minutes.
4. Film cast from acetone: Ball powder BL-353	60 40	Deflagrated immediately.
5. Gel: Ball powder Dibutylphthalate BL-353	30 40 30	No deflagration. Time to Salmon Pink - 50 minutes. Ball powder in solution but no blowing action.
6. Gel: Ball powder Ethylene glycol BL-353	30 40 30	No deflagration. Time to Salmon Pink - 55 minutes. Blowing action obtained.
7. Sponge grain cast into film from acetone solution	-	No deflagration. Time to Salmon Pink - 25 minutes.

FIGURE 1



M-51848

Feb 1959

Picatinny Arsenal

Ordnance Corps

Sponge Propellant (Firestone)

1. End of Grain #65
2. Center of Grain #65
3. Center of Grain #65 (near outer periphery)
4. Impurity in Center of Grain #65
5. Standard-Normal Structure of Grain #65
6. Center of Grain #64

Microscopy Laboratory

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