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of Engineers**

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**Toxic and Hazardous
Materials Agency**

**ZERO GAP PROPAGATION TESTING OF
PROPELLANT - No. 2 FUEL OIL SLURRIES**

Prepared by:

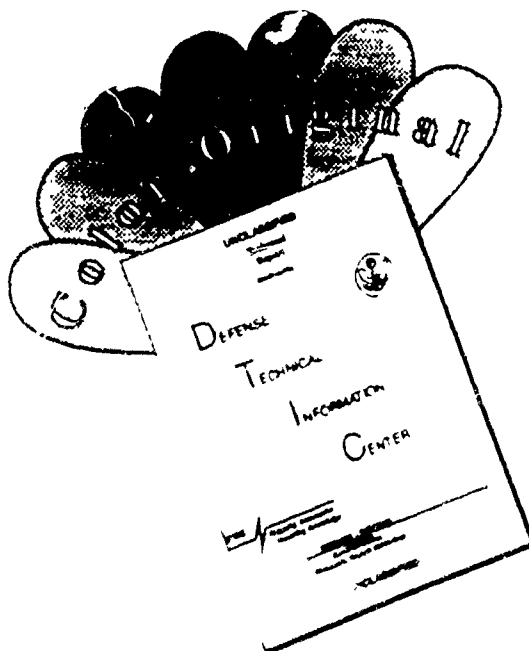
**Tennessee Valley Authority
National Fertilizer and Environmental
Research Center
Muscle Shoals, Alabama 35660
Under TVA Contract No. TV-79416**

**Prepared for:
Commander**

**U.S. Army Toxic and Hazardous Materials Agency
ATTN: CETHA-TS-D
Aberdeen Proving Ground, MD 21010-5401**

January 1992

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FINAL REPORT

ZERO GAP PROPAGATION TESTING OF
PROPELLANT-NO. 2 FUEL OIL SLURRIES

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January 1992

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Prepared for

United States Army Toxic and Hazardous Materials Agency
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ABBREVIATIONS

| | |
|----------|---|
| NFERC | National Fertilizer and Environmental Research Center |
| NC | Nitrocellulose |
| NQ | Nitroguanidine |
| OB/OD | Open-Burning/Open-Detonation |
| TVA | Tennessee Valley Authority |
| USATHAMA | United States Army Toxic and Hazardous Materials Agency |

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I. SUMMARY

The United States Army Toxic and Hazardous Materials Agency (USATHAMA) is currently conducting a program with the TVA to determine the feasibility of utilizing waste propellants as supplemental fuels for the U.S. Army's industrial combustors. Previous laboratory and bench scale research conducted by the TVA has demonstrated that slurring waste propellants with No. 2 fuel oil to form a supplemental fuel for industrial combustors is both a technically feasible and cost-effective disposal technology. However, the safety parameters of the process remain to be delineated.

This report discusses the results of Zero Gap propagation tests that determined the sensitivity of propellant-No. 2 fuel oil slurries to detonation by a shock wave. Two operational modes were studied: the dynamic or pumping mode, and the static or settled slurry mode. Supplemental fuels containing 10 percent by weight nitrocellulose, 15 percent by weight nitroguanidine, and 20 percent by weight AA2 double-base propellants slurried in No. 2 fuel oil did not propagate a detonation in either operational mode. These concentrations of propellant in No. 2 fuel oil are at the maximum found to be both technically feasible and cost-effective in the previous laboratory study. The Zero Gap tests establish that propellant-No. 2 fuel oil slurries could be processed at weight concentrations at or below those specified above without propagating a detonation.

II. INTRODUCTION

2.1 General

The military currently has a large inventory of waste propellants which are contained in conventional munitions that are obsolete or no longer serviceable. Additional quantities of waste propellants, i.e., propellants that do not conform to ballistic, chemical, or physical specifications, are generated during the normal process of manufacturing these materials. Currently available options for disposing of obsolete or out-of-specification propellants are open-burning/open-detonation (OB/OD) or incineration (1,2). However, these options are being severely restricted by federal and state environmental regulations. For example, OB/OD of energetic wastes requires a Subpart X permit under the Resource Conservation and Recovery Act. Subpart X operations remain under interim status until November 1992. At that time, whether or not OB/OD operations will be allowed to continue in their current form is unknown (3). Incineration of waste propellants is costly and does not capitalize on the recovery of energy from these energetic wastes. A technically feasible and cost-effective option to OB/OD or incineration is needed to dispose of waste propellants.

2.2 Review of Previous Projects in the Supplemental Fuels Program

The USATHAMA is currently conducting a program with the TVA to determine the feasibility of utilizing waste propellants as supplemental fuels for the Army's industrial combustors (4). Disposing of obsolete and waste propellants in this manner could be both cost-effective and environmentally sound technology which utilizes the energy value of these materials. Using the energy stored in these wastes reduces fuel consumption while eliminating potential hazardous waste.

In a recently concluded project (5-7), the technical and economic aspects of using propellant-No. 2 fuel oil slurries as supplemental fuels were evaluated. The propellants studied were nitrocellulose, nitroguanidine, and AA2 double-base. Nitroguanidine was supplied as a dry (<1 percent H₂O), finely-divided powder. Nitrocellulose was supplied as a water-wet (28-29 percent H₂O), finely-divided powder, while the AA2 propellant was supplied as paper-thin shavings of various lengths. The nitrocellulose and nitroguanidine propellants were easily dispersed in No. 2 fuel oil to form slurries suitable for physical and chemical testing. The AA2 propellant required wet grinding in No. 2 fuel oil to produce a suitable slurry.

From a technical standpoint of using a conventional oil burner, using 7.5 percent by weight nitrocellulose-, 10 percent by weight nitroguanidine-, and 10 percent by weight AA2 propellant-No. 2 fuel oil slurries as supplemental fuels for the Army's industrial combustors would be feasible. The economic analysis showed that fueling combustors with propellant-supplemented No. 2 fuel oil could be a cost-effective process; costs per ton for burning these slurries averaged \$350, while estimates of the cost per ton for disposal of propellants via OB/OD currently range from \$300-813. Incineration of propellants is estimated to cost \$2100 per ton. Furthermore, the previous project (5) identified the possibility of significantly reducing the overall cost of the process if a burner capable of handling viscous slurries were identified since 10 percent by weight nitrocellulose-, 15 percent by weight nitroguanidine-, and 20 percent by weight AA2 propellant-No. 2 fuel oil slurries could conceivably be used as supplemental fuels in this case.

While laboratory and bench scale research verified the principle of using propellant-supplemented No. 2 fuel oil as a feed to an industrial combustor, and an economic analysis showed a positive advantage using this approach, safety would also be of paramount

importance in using propellants as fuel supplements. The very nature of propellants requires special handling during their intended use and even stricter controls during combustion in an industrial combustor. Consequently, the likelihood of detonations occurring must be addressed before this process could be recommended for further pilot-scale studies (8).

III. DISCUSSION OF RESULTS

3.1 General

This report summarizes the results from tests performed by the Allegany Ballistics Laboratory, Hercules, Inc., Rocket Center, West Virginia, under the technical direction of the TVA's NFERC located in Muscle Shoals, Alabama. The propagation of reaction characteristics of nitrocellulose-, nitroguanidine-, and AA2 double-base propellant-No. 2 fuel oil slurries were investigated using a Zero Gap test protocol (9). The Zero Gap propagation test determines the sensitivity of propellant formulations to detonation by a shock wave.

3.2 Zero Gap Test Results

The Allegany Ballistics Laboratory, Hercules, Inc., prepared samples and conducted Zero Gap propagation tests on slurries of water-wet nitrocellulose, dry nitrocellulose, nitroguanidine, and AA2 double-base propellant in No. 2 fuel oil. The Zero Gap test is described in a subsequent section. Two modes of operating conditions were simulated. The first mode consisted of a dynamic flow system condition in which the particulate propellants were in a homogeneous suspension; the second, a static condition to simulate a flow stoppage that would allow the propellant particles to settle (10). Static tests were conducted in a horizontal 16-inch long pipe (2 inch, Sched 40, 304 SS) in which the propellant-No. 2 fuel oil slurry was allowed to settle for a duration of 2 to 4 hours. Dynamic tests were conducted in a vertical pipe of the same parameters and specifications in which the mixture was agitated to form a suspension and then immediately tested for detonation potential. A blank control test was also conducted on No. 2 fuel oil. Each test consisted of three replicate trials.

None of the Zero Gap tests conducted resulted in a propagation reaction. A propagation is defined as a shattered or deformed witness plate or damage to the containment tubing significantly greater than that of the No. 2 fuel oil blank. Propellant-supplemented fuels containing 10 percent by weight nitrocellulose (dried and water-wet material), 15 percent by weight nitroguanidine, and 20 percent AA2 propellant slurried in No. 2 fuel oil were tested. For the Zero Gap tests involving the AA2 propellant-No. 2 fuel oil slurries, a thickening agent (Cab-o-Sil) was added to the No. 2 fuel oil to prevent rapid settling of the AA2 propellant and thereby allow both dynamic and static operational mode testing (10).

Table 3-1 presents the results of the Zero Gap tests. In all cases, including the test of No. 2 fuel oil alone, the containment tube peeled and there was no change in the witness plate. There was no apparent difference between the dynamic and static operational modes in the sensitivity to propagate a detonation. Photographs of the containment tubes which show the extent of tube peeling for each type of propellant-supplemented fuel are shown in Figures 3-1 through 3-10.

Table 3-1. Results From Zero Gap Tests

| <u>Sample</u> | <u>Composition</u> | <u>Test Configuration</u> | <u>Special Conditions</u> | <u>Results</u> |
|----------------|----------------------------------|---------------------------|--|---|
| Fuel Oil | 100% Fuel Oil | Vertical | None | Tube peeled; No change in witness plate; True for each of three replicate tests |
| Dry NC | 10% NC; 90% Fuel Oil | Vertical | Dry NC to <0.5% moisture; Slurry with fuel oil | Tube peeled; No change in witness plate; True for each of three replicate tests |
| Dry NC | 10% NC; 90% Fuel Oil | Horizontal | Dry NC to <0.5% moisture; Slurry with fuel oil | Tube peeled; No change in witness plate; True for each of three replicate tests |
| Wet NC | 10% NC (wet basis); 90% Fuel Oil | Vertical | Dry NC; Re-wet & slurry with fuel oil | Tube peeled; No change in witness plate; True for each of three replicate tests |
| Wet NC | 10% NC (wet basis); 90% Fuel Oil | Horizontal | Dry NC; Re-wet & slurry with fuel oil | Tube peeled; No change in witness plate; True for each of three replicate tests |
| Nitroguanidine | 15% NQ; 85% Fuel Oil | Vertical | Grind NQ <200 mesh; Slurry with fuel oil | Tube peeled; No change in witness plate; True for each of three replicate tests |

Table 3-1. (Continued)

| Sample | Composition | Test Configuration | Special Conditions | Results |
|----------------|---|--------------------|--|---|
| Nitroguanidine | 15% NQ; 85% Fuel Oil | Horizontal | Grind NQ <200 mesh; Slurry with fuel oil | Tube peeled; No change in witness plate; True for each of three replicate tests |
| AA2 Propellant | 20% AA2; 3% Cab-O-Sil; 77% Fuel Oil | Vertical | Wet Grind AA2; Reslurry with fuel oil | Tube peeled; No change in witness plate; True for each of three replicate tests |
| AA2 Propellant | 20% AA2; 3% Cab-O-Sil; 77% Fuel Oil | Horizontal | Wet Grind AA2; Reslurry with fuel oil | Tube peeled; No change in witness plate; True for each of three replicate tests |

Figure 3-1. Typical
Containment Tube Prior
to Testing.

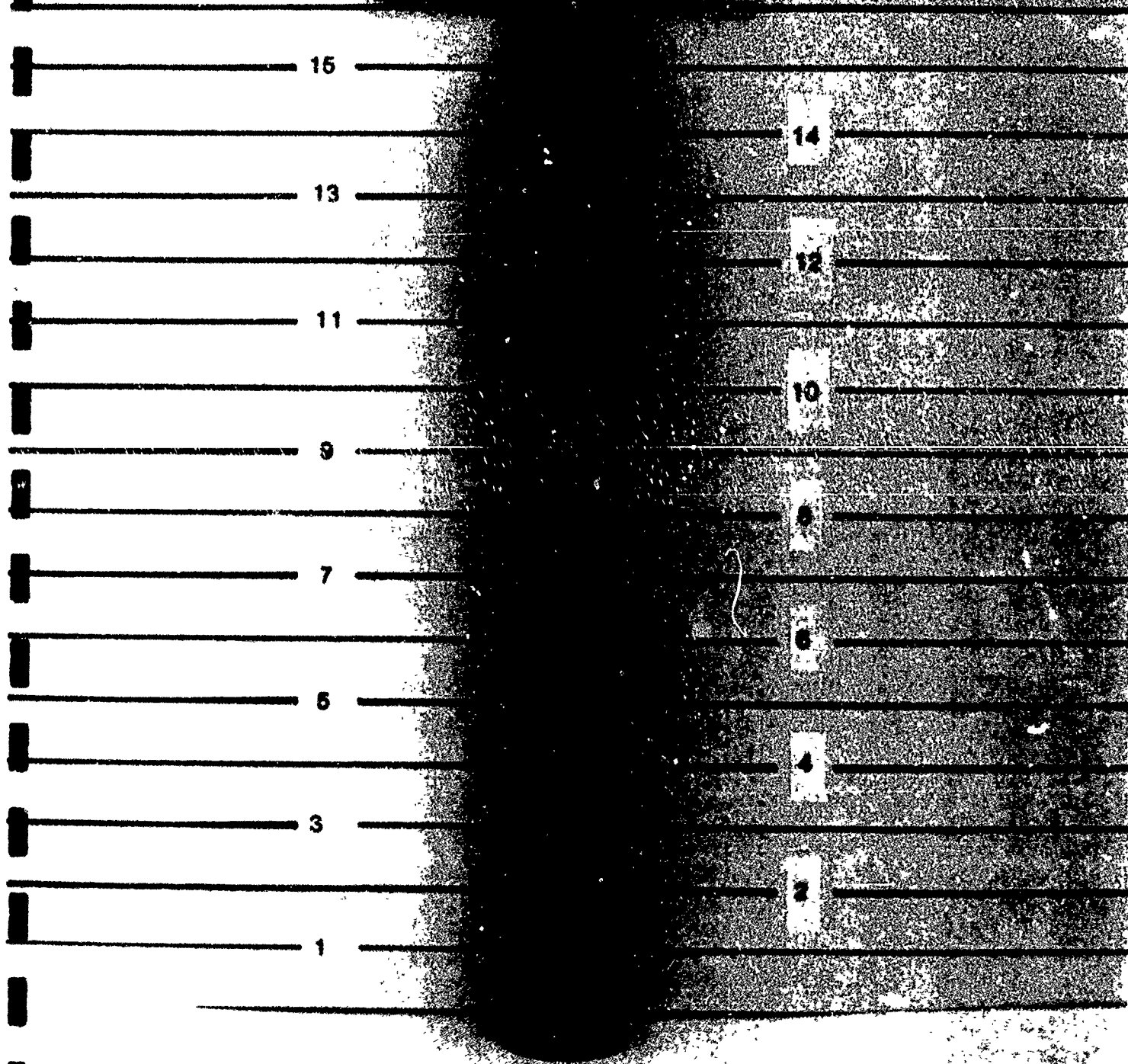


Figure 3-2. No. 2 Fuel Oil
Blank Reference Shot.

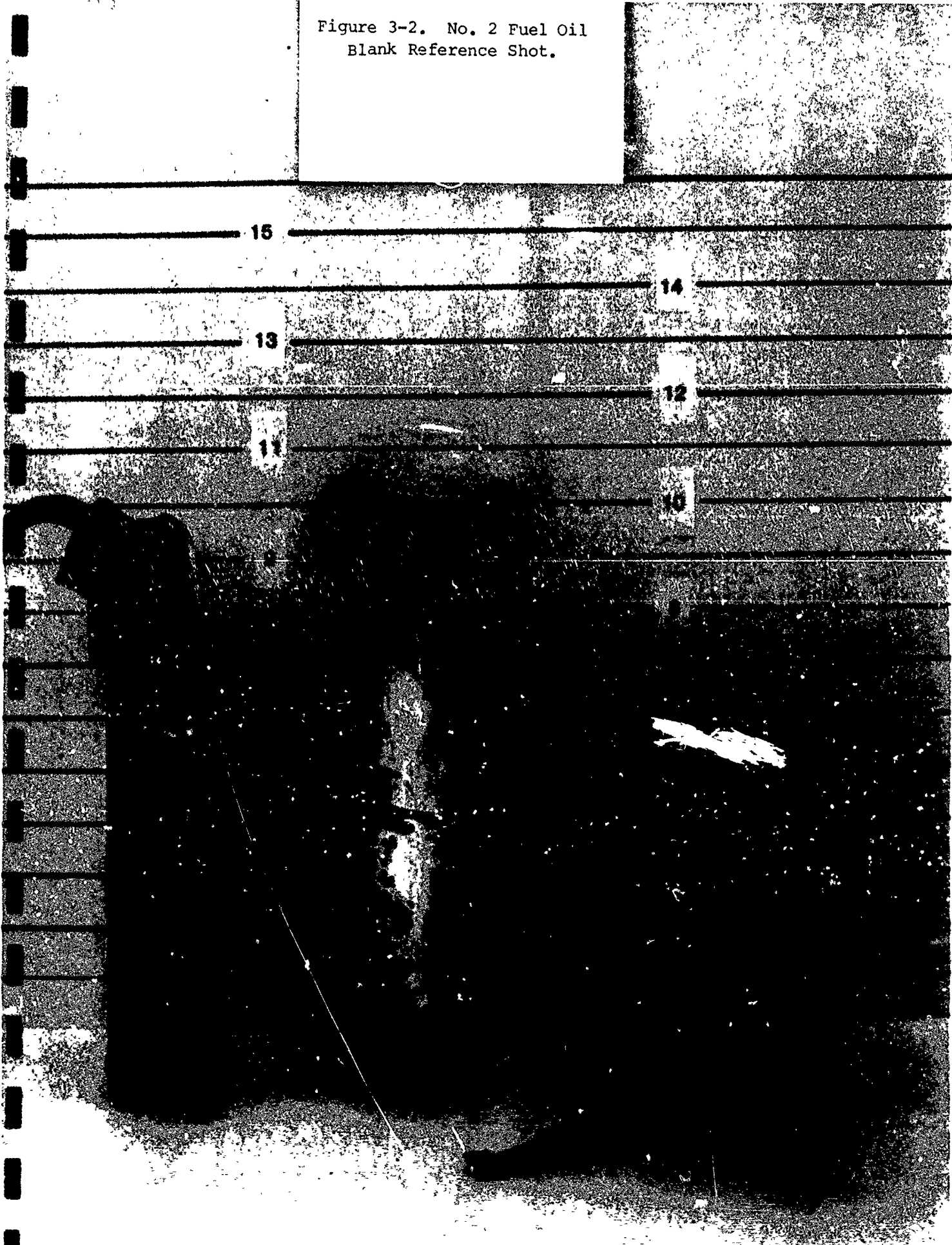


Figure 3-3. Zero Gap Test in
Vertical Configuration: 10%
NC (Dried) Suspended in
No. 2 Fuel Oil.

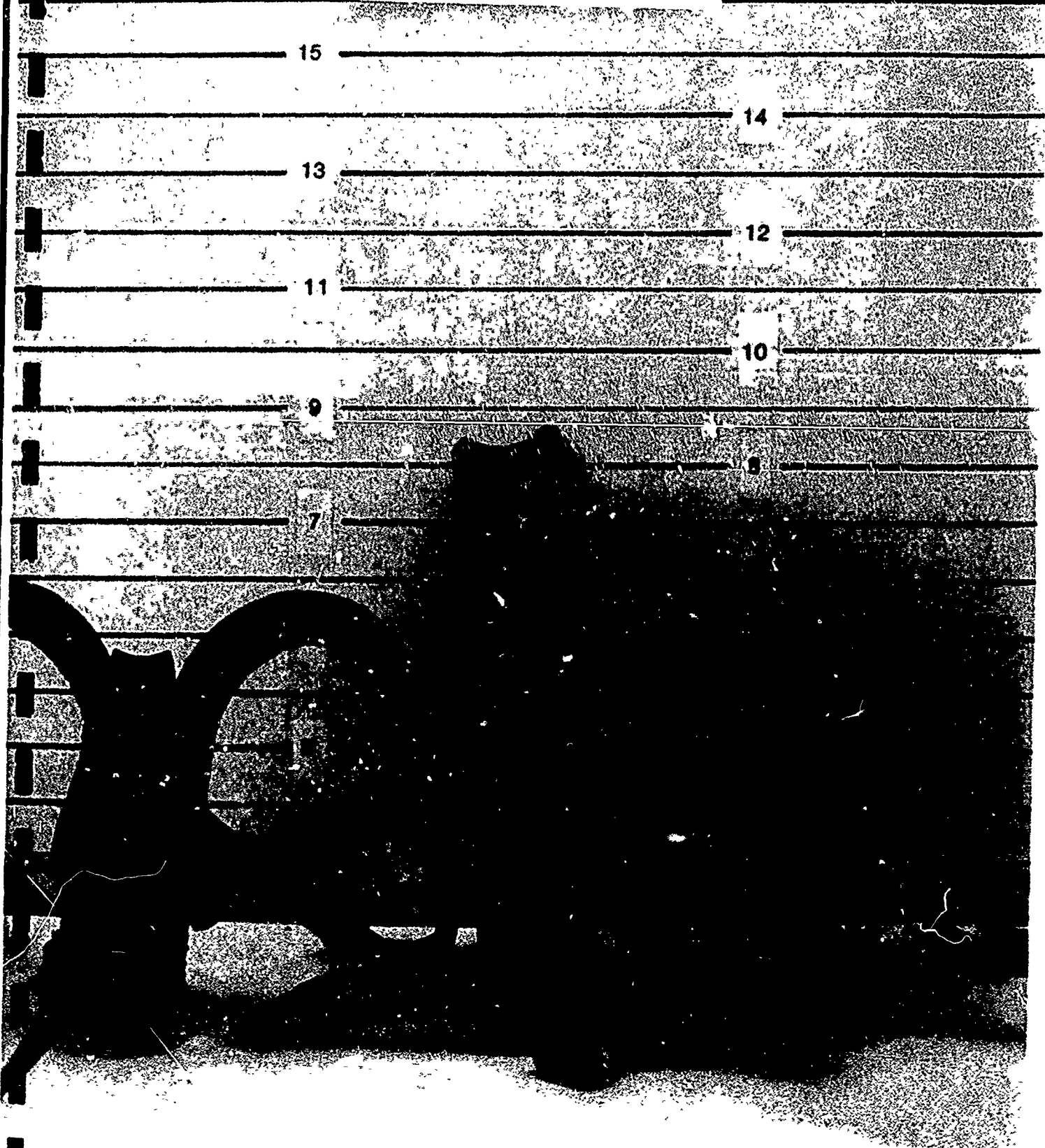


Figure 3-4. Zero Gap Test in
Horizontal Configuration:
10% NC (Dried) Settled in
No. 2 Fuel Oil.



Figure 3-5. Zero Gap Test in
Vertical Configuration:
10% NC (Water-Wet)
Suspended in No. 2 Fuel
Oil.



Figure 3-6. Zero Gap Test in
Horizontal Configuration:
10% NC (Water-Wet)
Settled in No. 2 Fuel Oil.



Figure 3-7. Zero Gap Test in
Vertical Configuration:
15% Nitroguanidine
Suspended in No. 2 Fuel Oil.

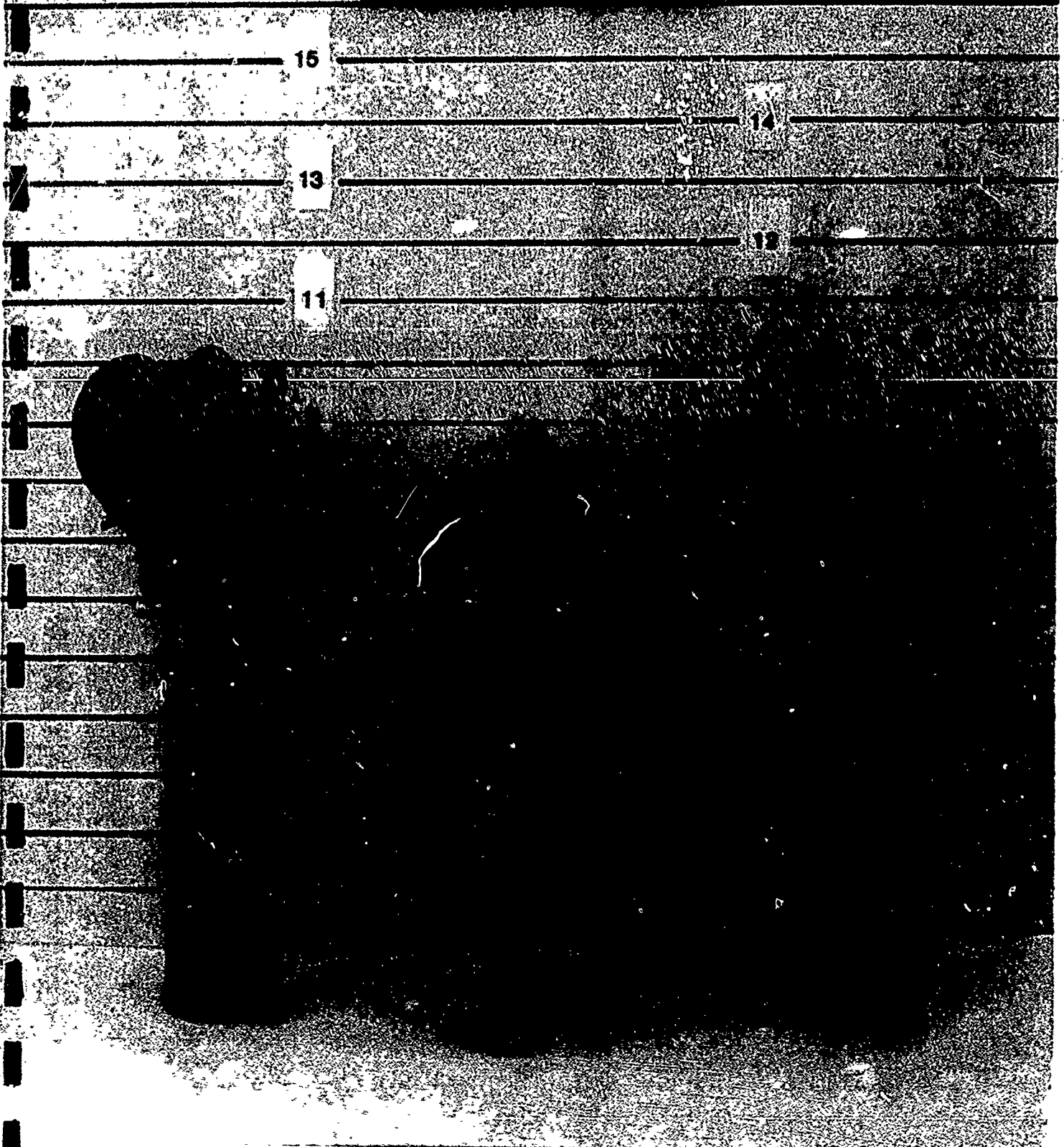


Figure 3-8. Zero Gap Test in
Horizontal Configuration:
15% Nitroguanidine Settled
in No. 2 Fuel Oil.

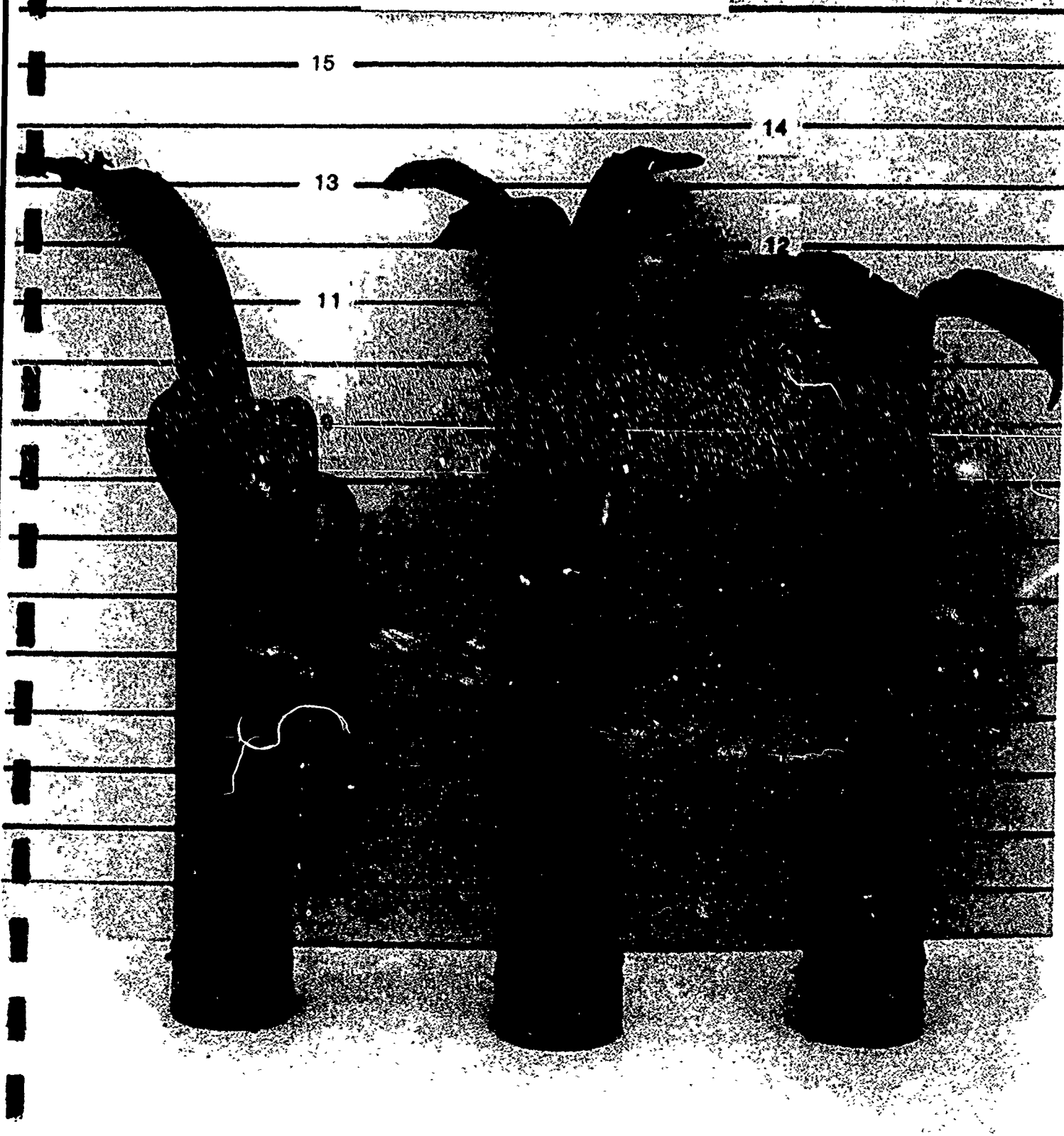


Figure 3-9. Zero Gap Test in
Vertical Configuration:
20% AA2 Propellant/3%
Cab-o-Sil Suspended in
No. 2 Fuel Oil.



Figure 3-10. Zero Gap Test
in Horizontal
Configuration: 20% AA2
Propellant Settled in
No. 2 Fuel Oil.



IV. EXPERIMENTAL PROCEDURES

4.1 Characterization and Composition of Propellants

Propellant samples were shipped to the Allegany Ballistics Laboratory from the Naval Ordnance Station in Indian Head, Maryland. Nitroguanidine was supplied as a dry (<1 percent H₂O), finely-divided white solid. As Figure 4-1 shows, some aggregation of the nitroguanidine occurred during shipping and handling, however, these aggregates were easily broken up when the nitroguanidine was dispersed in No. 2 fuel oil.

Nitrocellulose was supplied as a water-wet (approximately 26-29 percent H₂O), finely-divided white solid. A photograph of this material at 2X magnification shows the finely-divided nature of this propellant (Figure 4-2). Finally, the AA2 double-base propellant was supplied as paper-thin shavings of various sizes and lengths (Figure 4-3), resulting from the extrusion of propellant blocks through a die to form large grains for use in rockets. The composition of this propellant was supplied by Hercules, Inc. (Table 4-1).

Table 4-1. Composition of the AA2 Propellant Formulation

| <u>Ingredient</u> | <u>Weight Percent in the Formulation</u> |
|---|--|
| Nitrocellulose (12.2 percent nitrogen) | 51.0 |
| Nitroglycerin | 38.6 |
| Triacetin | 2.7 |
| Lead Salt | 4.0 |
| Dinitrophenylamine | 1.6 |
| 2-Nitrodiphenylamine | 2.0 |
| Wax | 0.1 |

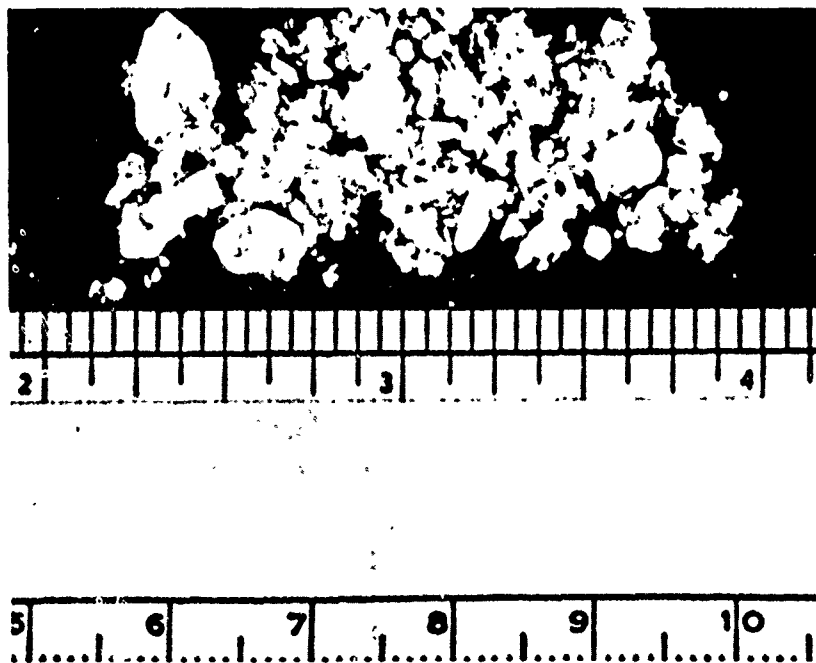


Figure 4-1. Photograph of Nitroguanidine Propellant (2 x Magnification).

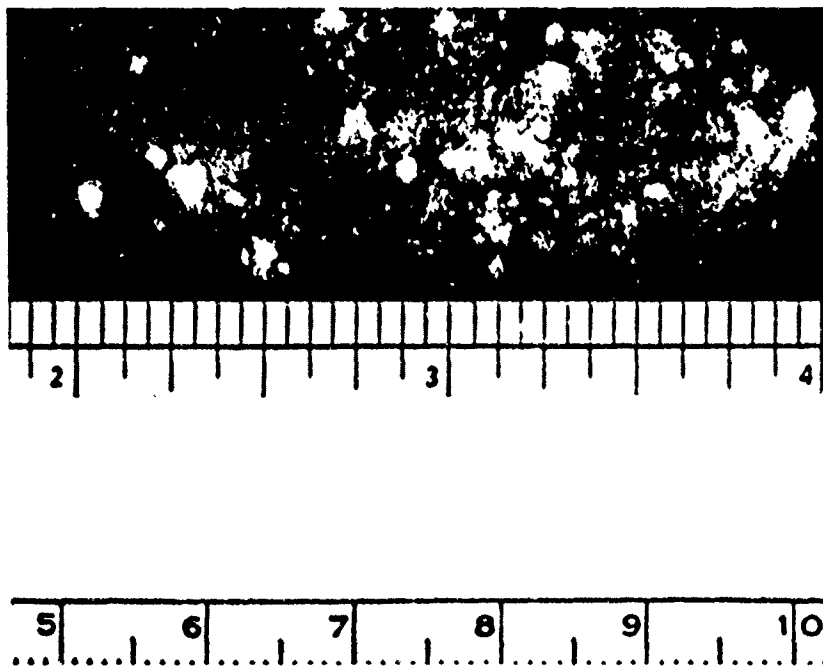


Figure 4-2. Photograph of Nitrocellulose (Dried) Propellant (2x Magnification).

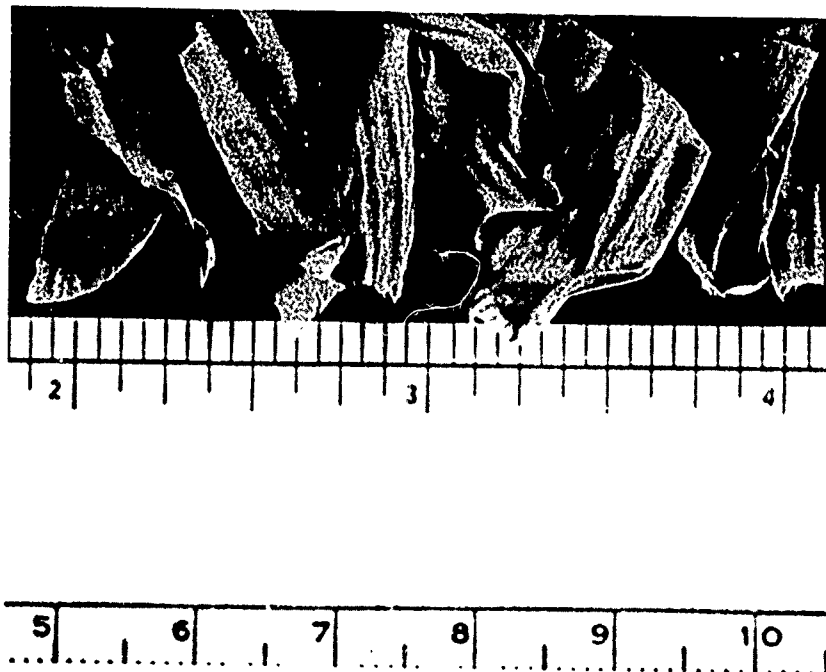


Figure 4-3. Photograph of AA2 Propellant Shavings (2x Magnification).

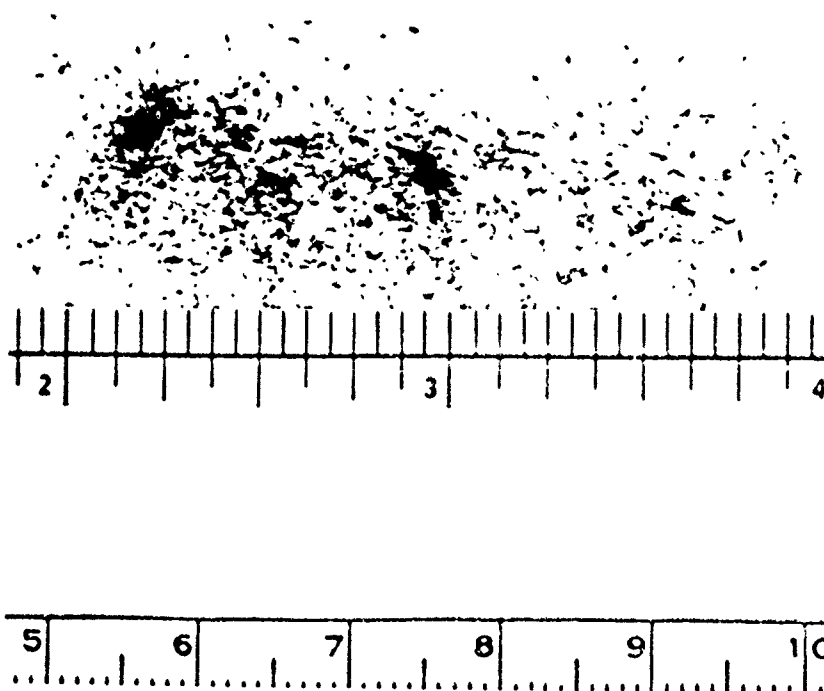


Figure 4-4. Photograph of AA2 Propellant Shavings After Grinding in Fuel Oil (2x Magnification).

4.2 Preparation of Propellant-No. 2 Fuel Oil Slurries

The appropriate amounts of No. 2 fuel oil and propellant to prepare the desired slurry composition were each weighed out on an analytical balance. The No. 2 fuel oil and propellant were then combined in a Waring blender and mixed at low speed for 5-10 minutes. In the case of the nitrocellulose and nitroguanidine propellants, this procedure formed a homogeneous slurry with No. 2 fuel oil. For the AA2 propellant, this operation resulted in a size reduction of the as-supplied propellant shavings. To ensure the safety of the technicians performing this operation, the blender was remotely controlled.

In a previous project (5), the settling rates of various propellant-No. 2 fuel oil slurries were measured. For a nitroguanidine-No. 2 fuel oil slurry containing 15 percent by weight propellant, 2 hours elapsed before all the nitroguanidine settled out. On average, for both the nitrocellulose (dried)- and nitrocellulose (water-wet)-No. 2 fuel oil slurries, 1 hour was required for the nitrocellulose in the slurries to settle out, irrespective of concentration. However, for the AA2 propellant-No. 2 fuel oil slurries, the elapsed time for the propellant to settle out averaged less than 15 minutes. Therefore, a fumed silica (Cab-o-Sil) was specified by the TVA as a thickening agent for the AA2 propellant-No. 2 fuel oil slurries to assist in maintaining a suspension.

Some additional steps were taken by the Allegany Ballistics Laboratory, Hercules, Inc., in order to assure that the desired testing conditions were consistently obtained:

- (1) The nitrocellulose was received water-wet (tested to be 26.5 percent moisture) and was dried to approximately 0.3 percent moisture;

- (2) For the "dry" nitrocellulose test, the dried nitrocellulose was slurried with the No. 2 fuel oil to obtain the proper ratio and then tested;
- (3) For the "wet" nitrocellulose test, the dried nitrocellulose was re-wet with a known amount of water before slurrying with the No. 2 fuel oil to the desired ratio and tested;
- (4) Three percent Cab-o-Sil was added to the AA2 slurry to assist in maintaining a suspension. No. 2 fuel oil was added to make the final ratio 20 percent AA2 propellant, 77 percent No. 2 fuel oil, and 3 percent Cab-o-Sil.

4.3 Overall Test Plan

Because the number of tests required to establish a GO/NO-GO slurry concentration within the range of interest could not be predicted, an initial base program was conducted at the maximum concentrations of interest (5). If no propagation occurred in any of the base program tests, then no further tests would be conducted. However, if propagation did occur in any of the base program tests, then additional tests to establish the NO-GO concentration for each particular propellant-No. 2 fuel oil slurry would have to be conducted. The base program test plan scope is delineated in Table 4-2. As discussed previously, the experiments outlined in the base program test plan were sufficient to establish that propellant-No. 2 fuel oil slurries did not propagate a detonation at the maximum concentrations of interest.

Table 4-2. Base Program Test Plan

| <u>Material</u> | <u>Condition</u> |
|---------------------|---|
| No. 2 Fuel Oil | Tested Alone as a Blank |
| Dry Nitrocellulose | 10% NC (Dry) + 90% Fuel Oil* |
| Wet Nitrocellulose | 10% NC (Wet) + 90% Fuel Oil* |
| Nitroguanidine (NQ) | 15% NQ + 85% Fuel Oil* |
| AA2 Propellant | 20% AA2 + 77% Fuel Oil + 3% Cab-o-Sil* |

* Samples tested in the vertical position to simulate a suspended slurry and in a horizontal position to simulate settled solids in a pipe. There were three replicates at each condition.

4.4 Zero Gap Test Protocol (9)

In the Zero Gap propagation test, the propellant-No. 2 fuel oil slurry was contained in a cylinder consisting of a 16-inch length of cold-drawn seamless carbon steel mechanical tubing, 1.875 inches O.D. with a wall thickness of 0.219 inches. The bottom of the cylinder was closed with two layers of 3 mil polyethylene sheet tied on with gum rubber bands and PVC electrical tape. Tests in the horizontal configuration had both ends closed similarly. A 3/8-inch thick mild steel witness plate six inches square was placed over the top of the tube, separated from it by 1/16-inch spacers. A shock donor consisting of a Pentolite pellet 2 inches in diameter and 2 inches thick was abutted to the bottom of the cylinder. The Pentolite pellet was initiated by a J-2 cap.

Criteria for a positive result was specified by Hercules, Inc., as deformation of the witness plate or damage to the containment tubing significantly greater than that of the No. 2 fuel oil blank.

The Zero Gap propagation test protocol may be compared to the Card Gap test (11). The Card Gap test also measures the sensitivity of a propellant formulation to detonation by a shock wave. A sample of the propellant is placed in a cardboard tube with a booster explosive. The explosive and sample are separated by a series of 0.254 millimeter (0.01 inch) cellulose acetate cards. The test results are reported as the number of cards necessary to prevent detonation of the sample. Three successive trials with no detonation are required. Seventy cards represent the dividing line between an explosive and fire hazard material. Therefore, based on this information, it is clear that the Zero Gap test is the most severe example of a Card Gap test, i.e., a test where no cellulose acetate cards are present.

4.5 Size Reduction and Particle-Size Distribution Tests

As stated earlier, the AA2 propellant was supplied as paper-thin shavings of various sizes and lengths. In order to produce slurries suitable for Zero Gap propagation testing, the AA2 propellant shavings were wet ground with No. 2 fuel oil in a Waring blender operated from a remote location. The particle-size distribution from a representative AA2 propellant-No. 2 fuel oil slurry is given in Table 4-3. This particle-size distribution compared favorably with data published previously where AA2 propellant shavings were wet-ground in No. 2 fuel oil using an Ultra-Turrax grinder (5). Figure 4-4 shows a photograph taken at 2X magnification of the ground AA2 propellant after it had been filtered from the No. 2 fuel oil, washed thoroughly with kerosene, and dried. This photograph clearly illustrates the reduction in particle size for the AA2 propellant shavings.

Table 4-3. Particle-Size Distribution of AA2 Propellant
After Wet-Grinding in No. 2 Fuel Oil

| <u>Percent Greater Than</u> | | |
|-----------------------------|-------------------|---------------------|
| <u>Sieve Size (micron)</u> | <u>Sieve Size</u> | <u>Sieve Number</u> |
| 600 | 0.27 | 30 |
| 500 | 0.40 | 35 |
| 420 | 0.53 | 40 |
| 300 | 1.33 | 50 |
| 212 | 13.02 | 70 |
| 147 | 47.68 | 100 |
| 63 | 93.41 | 230 |
| 1 | 100.00 | [On Filter] |

V. CONCLUSIONS

As a result of the total of twenty-four Zero Gap propagation test firings performed on No. 2 fuel oil slurries containing nitrocellulose (dried and water-wet), nitroguanidine, and AA2 propellant which were confined in either horizontal (static mode) or vertical (dynamic mode), 2-inch diameter, schedule 40, stainless steel pipes 16 inches in length, it is possible to conclude the following:

- Slurries of 10 percent by weight dried nitrocellulose in No. 2 fuel oil did not propagate a detonation in either a dynamic or a static operational mode.
- Slurries of 10 percent by weight water-wet nitrocellulose in No. 2 fuel oil did not propagate a detonation in either a dynamic or a static operational mode.
- Slurries of 15 percent by weight nitroguanidine in No. 2 fuel oil did not propagate a detonation in either a dynamic or a static operational mode.
- Slurries of 20 percent by weight AA2 double-base propellant in No. 2 fuel oil containing 3 percent by weight Cab-o-Sil did not propagate a detonation in either a dynamic or a static operational mode.

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