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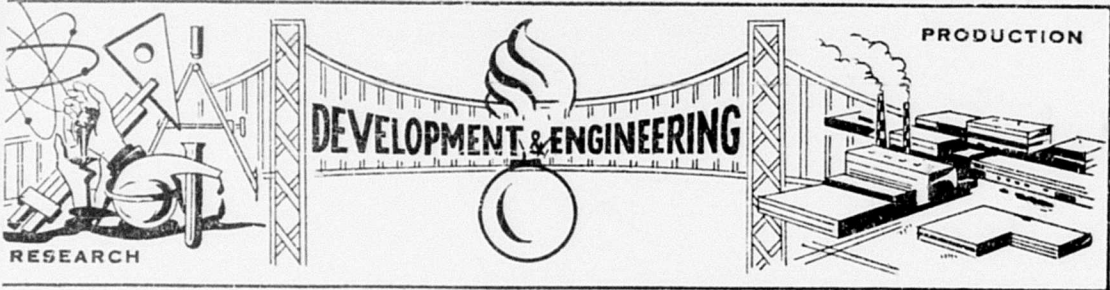
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TECHNICAL REPORT
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EVALUATION TESTS AND PROCESS STUDIES
RELATING TO ESTABLISHMENT OF SUBSTITUTE
FOR
BLACK POWDER

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(IMPROVEMENTS IN MANUFACTURE OF BENITE STRANDS)

BY
E. HUSELTON
S. KAPLOWITZ

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TECHNICAL REPORT

EVALUATION TESTS AND PROCESS STUDIES
RELATING TO ESTABLISHMENT OF SUBSTITUTE FOR BLACK POWDER
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E. HUSELTON

S. KAPLOWITZ

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SUBMITTED BY:

D. Katz

D. KATZ
Chief, Propellants
Section

REVIEWED BY:

S. Fleischnick
S. FLEISCHNICK
Chief, Applications
Branch

APPROVED BY

Aaron Rodkin
AARON RODKIN
Chief, Ammunition
Production & Maint.
Engineering Division

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SECTION I

INTRODUCTION

Picatinny Arsenal Research and Development (R&D) Studies to provide improved igniter compositions as substitutes for black powder in artillery primers led to the development of benite strands. Benite is essentially an extruded form of black powder, containing the ingredients of black powder (potassium nitrate, charcoal and sulfur) in a matrix of nitrocellulose. The ratio of black powder ingredients to nitrocellulose is 60:40.

Benite can be manufactured by a solvent extrusion process, using essentially the same mixing, extrusion and drying facilities employed in the manufacture of double and triple base cannon propellants. However, there is one major difference. Because of the length of the strands -- 6 to 20 inches, depending on the application -- use of cannon powder cutting equipment is not feasible. Handling and cutting equipment developed for production of R&D quantities constitutes a waste of materials and labor and is not considered suitable for scale-up to mass production. In addition, potassium nitrate preparation is expensive because of the recrystallization step believed necessary to yield a less hygroscopic product.

In the product area, the effect of variables such as strand diameter, moisture and volatiles level, incorporation of rework, use of technical grade potassium nitrate (non-recrystallized) and variations in potassium nitrate particle size had not been determined. Only limited data was available on the sensitivity and detonability of in-process benite.

This report covers the production engineering program to overcome these deficiencies in the manufacturing process and to obtain necessary data to base a realistic acceptance specification for benite.

SECTION II

SUMMARY

Production engineering on benite was closely tied to production because of an urgent requirement that arose when a benite-loaded primer was found to be essential for the successful performance of the 90mm T. 500 round. Therefore, a first phase of this program was slanted at solving immediate production problems with minimum modification to existing Ordnance facilities. A second longer range objective was to evolve an optimum process for the manufacture of benite.

The most urgent problem was to develop an acceptance specification practical for production use, since it was soon found that the R&D requirements which were necessarily based on limited manufacture were unsuited for production. It was necessary to relax the requirements for (a) moisture content (b) total volatiles and (c) the 134.5°C heat test. Satisfactory ballistics were used as the criteria for increasing the allowable moisture and volatile content and satisfactory surveillance life, the criteria for permitting the reduced heat test requirement.

Other changes to the R&D specification (PA-PD-1741) included elimination of the hygroscopicity and functioning tests, and the requirement for use of reagent grade potassium nitrate. The resulting modified requirements are contained in the current specification, MIL B-45451, 27 January 1960. The use of technical grade potassium nitrate results in savings in raw material costs of 22 cents per pound.

A product and process variables study was conducted to determine the effect of variables such as strand diameter, particle size of oxidizer, addition of plasticizer, use of rework, use of alternate solvent and stabilizer, and substitution of pyrocellulose for military blend nitrocellulose on the processing and performance characteristics of benite. This study was based on manufacture and testing of 15 pilot lots. It was found that (1) the functioning characteristics of benite are relatively insensitive to minor changes in dimensions and formulation (2) the use of rework has no deleterious effect on the performance characteristics of benite (3) substitution of ethyl acetate for diethyl ether in the solvent system improves processing characteristics (4) direct substitution of diphenylamine for ethyl centralite improves the chemical stability as measured by the short term heat tests.

The efficacy of the benite with the ethyl acetate solvents and the rework was confirmed in ballistic studies in the three-inch gun. Firings using these experimental lots were conducted at -40°F, 70°F and 160°F. In addition, firings conducted using primers environmentally conditioned for four weeks both at 170°F and 122°F and 95% relative humidity, gave completely satisfactory results in regard to velocity dispersion, pressure uniformity, ignition delay and smoothness of the pressure versus time trace.

In an effort to attain a more economical production process for benite strands, pilot scale studies were conducted to determine the feasibility of adapting the high capacity, automated equipment used in the food industry for

the manufacture of spaghetti (long goods extruder and spreader). It was found that particularly good flow characteristics could be achieved with multi-orifice plate extrusion (a 51 way die was used) using the ethyl acetate solvated colloid and that flow uniformity could be achieved by incrementally selecting and reaming holes in the die breaker plate. Also, the ethyl acetate colloided strands after draping on a dowel hung straighter and dried without cracking, warping or other deformation.

It is estimated that with multi-orifice extrusion and spreading, substantial labor savings can be effected. At a production rate of 50,000 pounds per month, it should be possible to amortize the cost of this automated equipment in about one to two months.

Both the friction pendulum and the detonation hazard (solvent wet material) tests showed that benite reacts similarly to single base (M-10) propellant and therefore may be considered a Class 2 explosive hazard when solvent wet.

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SECTION III

CONCLUSIONS

A realistic acceptance specification for benite strands has been prepared based on data from ballistic tests in the 90mm T300E53 round.

Technical grade potassium nitrate is satisfactory for manufacture of benite. The use of this material provides a savings in the manufacturing cost of about 22 cents per pound.

Processing information was developed for use with conventional Ordnance equipment. The major improvements now incorporated in the Description of Manufacture are (a) the drying temperature was increased to 131°F with consequent reduction in drying time (b) the drying time on the reels was reduced from 10 days to 12 hours, and (c) the incorporation of rework with a consequent improvement in yield from 45 to 90% +.

The stability characteristics of benite -- as measured by the standard short-term heat tests -- are improved by the direct substitution of diphenylamine for ethyl centralite in the formulation. If surveillance tests confirm this finding, the substitution will be made.

The processing characteristics of benite are improved by the use of an ethyl acetate and alcohol solvent system in place of the diethyl ether and alcohol system.

The feasibility of using a multi-orifice plate die in extrusion of benite strands, and drying benite strands on a dowel was demonstrated. This

indicates that the use of automated high capacity equipment of the type found in the food and plastics industries may be adapted to benite manufacture. Prototype studies to establish a process with this equipment are now underway at Radford Arsenal.

Benite colloid does not detonate under severe conditions of confinement and therefore should be considered a Class 2 explosive hazard (fire hazard) for extrusion.

The functioning characteristics of benite are relatively insensitive to minor changes in dimensions and formulation.

SECTION IV
RECOMMENDATIONS

None.

SECTION V

STUDY

A. Studies to Establish A Realistic Acceptance Specification For Benite Strands.

The initial requirements for the benite strands acceptance specification (MIL-B-45451) were necessarily based only on a limited amount of chemical and physical test data. Following the start of production at Picatinny, it became evident that some of the requirements were unrealistic and that specification revisions would be required for economic production. Necessary revisions were effected as the result of the work discussed below (Table I).

1. Total Volatiles (T.V.) and Total Moisture (T.M.)

The initial Picatinny production lot of benite (PA-E-29139), made using the same process employed by R&D, failed to meet specification requirements for total volatiles and total moisture, shown in these examples.

	<u>Spec. PA-PD-1741</u>	<u>PA-E-29139</u>
T.V., % max.	0.75	2.11
T.M., % max.	0.50	0.85

To determine whether the specification requirements could be relaxed to permit acceptance of this lot, a series of tests were conducted in the 90mm gun with the T300E53 round to compare the performance of Lot PA-E-29139 with a lot of benite used by R&D in development of the round.

Test results summarized in Table 2, Series I of Appendix A, showed no significant difference between the two lots in velocity, velocity dispersion, maximum pressure, and maximum pressure dispersion. It is noted that the firings were performed at -40°F , which represents the most severe test for the propellant ignition system. The ballistic acceptability of the initial production lot was further confirmed by the results of instrumented static firings in the XM-79 primer (Table 3, Appendix A).

Following this initial work, it was desirable to further evaluate the effects of T. V. and T. M. content on benite performance, to establish limits which could be met without extended drying cycles. This would preclude the necessity for redrying of benite at the loading plant, as is sometimes required with black powder. To accomplish this, a sample of benite (solvent wet strands) was taken from a production mix (lot PA-E-29796) and subjected to this treatment:

- a. Storage for three days at room temperature.
- b. Exposure to an environment of 95% relative humidity and a temperature of 100°F for six hours.

The T. V. and T. M. of the benite at the end of this time was 2.96% and 1.5% respectively. Then the benite was loaded into XM79 primers, with care taken to minimize the loss of volatiles and moisture. For comparison purposes, a second set of primers was loaded with benite representing the first production lot manufactured by Radford Arsenal. The T. V. and T. M. of

the Radford lot was 2.04% and 0.68% respectively. This was done to serve a two-fold purpose: (1) evaluate the effects of "high" T.V. and T.M. (2) evaluate the performance of the first benite made at Radford Arsenal. The tests performed included both instrumented static primer firings at 160°F, 70°F and -65°F, and gun firings with the T300E53 round at 70°F and -40°F.

The primer firings showed that the high T.V. material functioned satisfactorily, without any hangfires or excessive ignition delays at -65°F (Table 3, Appendix A). In the case of the Radford lot, the 160°F and 70°F firings gave normal results. However, at -65°F, one primer gave an excessive ignition delay (greater than 35 milliseconds) while two other primers gave higher than average ignition delays. Typical static firing traces for the two lots are shown at the three conditioning temperatures in Figures 2-4 (Appendix A).

The firings in the 90mm gun (Table 2, Appendix A) showed that the high T.V. benite produced an increase in velocity dispersion at -40°F, but otherwise provided satisfactory ignition and functioning of the round, as can be seen from typical p-t traces for the series (Figure 1, Appendix A).

The Radford lot also gave good ignition as evidenced by the p-t traces, but the delay times were approximately twice that of the high T.V. lot, (32.2 versus 17.4 milliseconds), confirming the behavior in the primer firings. It is noted that the average value obtained by R&D for -40°F ignition lag was 14.5 milliseconds. The higher ignition lag of the Radford material did not appear to cause any deleterious effect. (Since these tests were run, studies performed

with XM79 and XM80 benite primers have shown that ignition delay and hangfire tendencies can be markedly reduced, and uniformity can be improved by a change to the primer head design. This change consists of putting a small (three grain) black powder booster charge in line with the initiator element to provide a stronger flame at the ends of the strands. Since there appears to be variation in the ignitibility of different lots of benite, the black powder booster eliminates the slow or marginal ignition which is occasionally encountered. (A separate report covering this work is being prepared.)

Considering the above data, it was determined that the limit for T. V. should be set at 4.25% max., and 1.0% max. for T. M. These limits removed the problem of long drying times, yet provided an adequate margin of safety to insure satisfactory item performance. It should not be necessary to redry this material at the loading plants because of the relatively low hygroscopicity of benite.

2. Hygroscopicity

The initial specification requirement of hygroscopicity (1.9% max.) was considered a means for assuring that any slight impurity in the raw materials, particularly the potassium nitrate, would not increase the hygroscopicity beyond the R&D program level. In this regard, R&D specified that potassium nitrate used in benite be recrystallized. This was based on prior work with pyrotechnic compositions in which recrystallization was used to reduce the sodium content impurity, which has an affinity for moisture.

Since the recrystallized material, or the equivalent reagent grade, costs about 45 cents per pound more than the technical grade, tests were conducted to determine whether the added cost was justified.

Analysis showed that recrystallization reduced the sodium content of technical grade potassium nitrate from 0.16% (0.19% is the max. allowed by specification MIL-P-256) to 0.02% with a resultant decrease in hygroscopicity from 0.73% to 0.24%. (Table 4, Appendix A). The effect of this difference on berite hygroscopicity was evaluated by comparing hygroscopicities of production lots of berite made with recrystallized potassium nitrate with several pilot batches which were prepared using technical grade potassium nitrate. From the data, summarized in Table 5, Appendix A, it can be seen that use of technical grade potassium nitrate increases the average hygroscopicity 0.13%. From a practical standpoint this difference was not considered to be significant. Further, ballistic tests made with berite prepared with technical grade potassium nitrate showed that performance was not adversely affected even after environmental conditioning (Table 4, Appendix B). Based on this, the requirement for use of recrystallized or reagent grade potassium nitrate and the requirement for hygroscopicity was deleted from the specification. This resulted in a saving of 20 cents per pound in the cost of manufacturing berite.

3. Stability

Soon after berite production was started, it was found that the 134.5°C heat test requirement of 40 minutes (the same as is required for single base

propellants could not be consistently met, as the actual values obtained ranged from 35-45 minutes (Table 6, Appendix A). A study was made to determine the stability characteristics of benite by other rapid methods, as well as the 65.5 and 80°C surveillance tests.

With regard to the latter, a sample of benite (lot PA-E-26467) placed in surveillance during the R&D program had not produced red fumes after 18 months at 80°C. This sample was removed from surveillance and tested for available stabilizer. The ethyl celtrabite content was found to be completely depleted; however, the material showed no visual evidence of physical deterioration. Since the normal 80°C surveillance time for single base propellants is approximately 13 months, the chemical stability of benite was considered equivalent or even somewhat superior to single base propellant.

A sample of PA-E 26467 put into 65.5°C surveillance in March 1958 has not reached an end point at the time of this writing.

Data obtained with various lots of benite using other short term stability tests covered under the Section on Product and Process Variables Studies (the 120°C heat, 90°C and 100°C vacuum stability, and 110°C Taliani) showed this material to have somewhat better stability than double base propellants. Since benite is neither a single nor a double base propellant by conventional definition, it was considered that surveillance data would give the best indication of storage potential. The 134.5°C heat test requirement

was therefore changed to 35 minutes to include the low end of the normal range in stability test values for this material.

4. Specific Gravity

The initial specific gravity requirement of 1.7 ± 0.2 was found to be broader than necessary since the values obtained with the first 12 production lots fell in the range 1.57 to 1.68 gms/ml (Table 6, Appendix A). The requirement was changed accordingly to 1.65 ± 0.15 gms/ml.

5. Functioning Test

The functioning test was included in the original specification as a precautionary measure since there was little data on the lot-to-lot functioning characteristics of berite at the temperature extreme (-65°F and $+160^{\circ}\text{F}$). The requirement, which called for firing 15 primers each at -65°F and $+160^{\circ}\text{F}$ and inspecting the primer tubes for evidence of rupture, was dropped after checking out the first 12 lots and noting no evidence of rupture. This was supported by the fact that instrumented static primer firings demonstrated that the max. pressure developed was well below the hydrostatic test pressure (15,000 pounds per square inch) of the primer body. Also, coverage in this area was provided by including a functioning test in the acceptance specification for loaded primers.

B. Product and Process Variables Study

1. Fifteen 15-pound batches of berite were prepared to study the effect on performance and processing characteristics of these variables:

- a. Use of technical grade potassium nitrate (MIL-P-156).

- b. Use of rework.
- c. Use of an alternate solvent system.
- d. Strand diameter 20% over and 20% under nominal.
- e. Use of alternate stabilizer.
- f. Use of single base plasticizer.
- g. Use of carbon black instead of charcoal.
- h. Use of pyrocellulose instead of military blend N/C.
- i. Changes in particle size of potassium nitrate.
- j. Changes in mixing procedure.

2. In general the procedure used to manufacture the benite strands in each of these test batches followed Picatinny Description of Manufacture #35-4-48 (Reference 2).

3. The resultant strands were subjected to chemical and physical tests, and the static functioning characteristics evaluated by instrumented static primer firings. The results of these tests, which are contained in Tables 1-4 of Appendix A are discussed below:

a. Processing Characteristics

Since production difficulties were being experienced in obtaining uniform extrusion rates through all dies and in handling the solvent wet strands without breaking, mixes EP-3 through EP-6 (Table 1, Appendix B) were prepared to study the effect of adding plasticizers such as DNT and DBP to the matrix, and in substituting pyrocellulose for gun cotton. It was found

that a slight improvement in green strength was obtained in mixes EP-3 and EP-5. However, with EP-4 and EP-6 little, if any, improvement was noted. The most significant improvement was obtained with EP-14, in which ethyl acetate was substituted for diethyl ether as the colloid solvent, and diphenylamine directly substituted for ethyl centralite as the stabilizer. (This latter change was not considered to have an effect on processability.) The extrusion uniformity and green strength of EP-14 were noticeably superior to the other 14 batches, probably due to the reduced in-process solvent loss and surface hardening experienced with the less volatile ethyl acetate.

b. Physical Properties

Tensile tests were performed in accordance with specification GOST-3918 Method No. 5100, except that the gage length was set at two inches. Values for tensile strength and elongation ranged from 3320 to 4930 pounds per square inch, and 1.9% to 5.2% respectively, with the highest value for tensile strength being obtained for Lot EP-14. In general, the addition of plasticizers and other formulation changes had a very slight effect on the physical strength of the strand. However, in the case of Lot EP-2 -- which was prepared using coarse potassium nitrate -- a significant reduction in the tensile strength was obtained. This indicated that control of potassium nitrate particle size was an important factor in obtaining good physical properties. In this connection, the benite specification required that the

potassium nitrate be ground to an average particle diameter of 10 to 30 microns.

c. Chemical Properties

The stability of benite was thoroughly investigated in connection with the acceptance specification studies (Section V, Part A). Using these results as the standard, comparison data were obtained for the experimental lots EP-7, 9, 14 and 16. (EP-16 was a replicate of EP-14). The results (Table 2, Appendix B) show that the ethyl acetate colloided benite - using diphenylamine instead of ethyl centralite as the stabilizer (EP-14) - had improved stability as measured by the 90°C vacuum stability, 70°C Tapani and 120°C Heat Tests. Other data shows diphenylamine stabilizer to be superior to ethyl centralite with the 134.5°C heat tests (Table 1, Appendix B). Five out of six lots containing DPA gave salmon pink values of 50 + minutes (one lot, EP-15, where carbon black was substituted for charcoal as another change, gave 45 minutes to salmon pink and exploded in less than 300 minutes), while seven out of eight lots containing EC stabilizer gave values of less than 50 minutes.

Both 65.5°C and 80°C surveillance tests for the experimental lots involving composition variations were still underway at the time of this report.

d. Sensitivity Characteristics

Picatinny Drop Test sensitivity values ranged from seven to 10 inches for standard formulation benite, and from eight to 11 inches

with the modified formulations. The 11 inch values were obtained with the plasticized and the pyrocellulose substituted formulations. Again, the variables selected were shown to be relatively insensitive to the standard tests. The friction pendulum test was run on the standard formulation benite only. In each instance snaps, but no sparks, burning, or detonation with the steel shoe was reported. This test is described in Picatinny Arsenal Testing Manual 7-1.

4. Benite from the experimental lots EP-1 thru EP-15 was static-tested in primers to determine the changes produced by the effect of formulation, strand diameter, etc., on the ballistics. The firings were randomized and included seventeen groups, six primers per group, three primers each, conditioned at -65°F and 70°F respectively. Lot EP-11B, made in accordance with the specification, was the standard, with Lot EP-11A and EP-11C, containing strands 20% undersize and oversize in diameter respectively, making up the additional groups. The data from these firings tabulated in Table 4, Appendix B, indicate:

a. The modified formulation EP-14 with DPA used instead of E. C. as the stabilizer, and ethyl acetate used in place of diethyl ether gave the most desirable primer functioning characteristics -- (1) low ignition delay with high maximum pressures (2) higher maximum pressures at 70°F than at -65°F .

b. Use of rework has no deleterious effect on primer functioning.

c. Use of coarse, 60-80 mesh, potassium nitrate gives long ignition delays and low maximum pressures.

d. Use of carbon black instead of charcoal gives long ignition delays and low maximum pressures.

e. Addition of single base plasticizer dibutylphthalate and dimethyltoluene gives long ignition delays and low maximum pressures.

5. Following this preliminary screening, a second group of M-28B2 primers were loaded with (a) the modified benite EP-14 (b) the rework benite EP-14 (c) the R&D standard benite PAE-28203. This second group was assembled into three-inch test rounds for evaluation in gun firings. This was done to prove out the ballistic acceptability of the two modifications of greatest potential value. The firings in the three-inch gun were considered to provide a severe test of the performance characteristics of the benite for the following reasons:

a. The cartridge case was uncrimped, providing a minimal to no pull value.

b. M-17 propellant, for which low temperature ignition is sensitive, was used.

c. A badly worn gun tube with a history of more than 2,000 previous firings was used.

6. The series included firing of temperature conditioned rounds at -40°F , -70°F and $+160^{\circ}\text{F}$. This was later supplemented with 70°F firings

of rounds containing environmentally conditioned primers. The latter were conditioned by being subjected for 28 days to a temperature of $+170^{\circ}\text{F}$, or a humidity of $+122^{\circ}\text{F}$ and 90% R.H.

7. The results of this ballistic test are in Table 4, Appendix B.

The summary follows:

- a. All of the experimental benite primers performed satisfactorily following temperature or environmental conditioning. All velocity dispersions were less than 100 feet per second; ignition delays were less than 40 milliseconds and shape of the pressure versus time traces showed smooth rise curves (Figure 2, Appendix D).
- b. In general, the values for ignition delay were shortest for the R&D benite and longest for the ethyl acetate - DPA benite, EP-14.
- c. In general, the velocity dispersions were lowest for the R&D benite primed rounds and of approximately the same magnitude for the two experimental benite primed rounds.
- d. Performance of all three lots with respect to average velocity and dispersion were comparable at -40°F , the most critical condition.

Based on the above, it was concluded that (1) use of rework and (2) use of ethyl acetate, instead of diethyl ether as the colloid solvent in the manufacture of benite, caused no significant changes in the functioning characteristics and was an acceptable practice for production.

C. Development of an Improved Process for Mass Production of Benite Strands

Although benite strands are manufactured by essentially the same techniques as malite base cannon propellants, labor costs are considerably higher. This results from the fact that the fine diameter benite strands must remain straight on drying and then be cut into finished lengths of six to 10 inches (depending on the primer). With use of conventional extruders and powder distributors, the strands are coiled in tubs as they leave the press and very quickly develop rigidity owing to solvent evaporation. This requires that each of the long green strands be hung or rest on a rack until sufficient solvent evaporates to stiffen it (about one hour). After stiffening, the cut into rough lengths for final drying. After drying, the strands must be cut to required length. Since existing high speed cannon powder cutters are not suitable for either the green or final cutting operations, improved cutting equipment and associated handling operations result in high labor costs.

Since the length and diameter of benite strands are very similar to those of spaghetti (a product manufactured at a very low cost), a visit was made to a spaghetti plant to witness the processing techniques and equipment used. It was found that the operations of extrusion and preliminary cutting are performed on a continuous basis using a machine called "a long goods spreader". The mix dough (which is prepared continuously) is fed by a screw feeder into the manifold of the long goods spreader to which is

bolted a simple, rectangular plate die containing approximately 2,000 orifices. Strands are continuously extruded through each of the orifices. (Extrusion pressures are approximately 2,000 pounds per square inch and are controlled by varying screw speed and feed rate.) Severing strands from the front and back bank of orifices (a two-stick machine) on the die block is controlled by a timing device which intermittently actuates first the front and then the back strand knife. Each time a rod is simultaneously placed in position to catch the accumulated strands for a preset extrusion period. Each rod, which is 54 inches long, holds 1,000 strands (Figure 2, Appendix C). The timer is set so that all strands on the rod will be full length or longer before trimming through the action of a continuously reciprocating sickle bar cutter. The trimmed strand ends from the faster running dies are dropped to a conveyor and returned for remixing by a blower in the left foreground (Figure 2, Appendix C).

A typical two-stick production machine has a cycle time of 50 seconds, with each cycle yielding 2,000 strands, about 40 inches long. This is equivalent to 900-1,000 pounds of product per hour.

The rods are manually loaded onto a transfer buggy (about 100 rods per buggy), and wheeled into an oven for drying. After drying, the transfer buggy is wheeled to a final cutter. The rods, containing 1,000 strands each, are fed one at a time to the cutter which first removes the rod, next cuts the turns and then by means of three rotary split disc cutters mounted on a single shaft, cuts the strands to the required length while conveying them to

the pack cell station.

Since the use of the above type of equipment (with necessary safety modification for propellant operations) could provide very substantial labor savings in manufacture of benite, a pilot scale investigation was undertaken to determine whether the processing characteristics of benite lends itself to this equipment. Three factors were considered critical in demonstrating feasibility:

- a. Extruder collond must be capable of satisfactory extrusion through a multi-orifice plate type die.
- b. When draped over a rod, the green strands must not curl or deform on drying.
- c. Screw extrusion of benite collond must not involve an undue safety hazard.

The results of the studies performed in each of these areas is discussed in the following sections:

2. Multi-Orifice Plate Die Extrusion Studies

The multi-orifice extrusion studies were performed on a four-inch vertical Logan press. Following discussions with spaghetti equipment and plate die manufacturers, a 51 orifice plate die assembly and adapter for the Logan press was designed and fabricated (Figure 3, Appendix C). The major features of this design are:

- a. Use of a breaker plate located directly above the plate die containing three rows of tapered holes, 17 holes per row, to correspond

with the hole pattern in the plate die) to break up and spread the flow from the distributor manifold.

b. Placement of the hole pattern in the plate die so that the strands, after cutting from the press, can be picked up and draped over a dovel without overlapping.

To evaluate the flow uniformity from the die, the length of the strands from each of the three rows of holes -- designated A and C for the outer rows, and B for the center row -- was measured for each run and a percent yield calculated (Figure 4, Appendix C). This formula was used:

$$\text{Yield per Row, \%} = \frac{\text{Cumulative Length of 17 Strands, inches}}{17 \times \text{Distance from Die exit to floor, inches}} \times 100$$

To provide a common basis for comparison, the run was stopped after three strands reached the floor. An ideal overall yield of 100% would occur if each of the 51 strands reached the floor simultaneously. Figure 5, Appendix C shows this 100% yield or perfect extrusion.

It can be seen from the data that the overall yields in the initial runs, Modification 1 (Table 1, Appendix C) ranged from 39 to 55%, with the lowest per row yields coming from the center row, Row B (Figure 6, Appendix C). Extrusion pressure also was seen to be a factor, since lowering the pressure from 750 pounds per square inch gauge in Extrusion 2 to 650 pounds per square inch gauge in Extrusion 3 caused the overall yield to drop from 54 to 39%.

To improve the yield from the center row, each of the 17 holes in Row B of the breaker plate was opened from .185 inch to .205 inch (Modification 2). This was found to produce an increase in the center row yield, and provided overall yields in Extrusions 10-13 of 54-61%. The wide variation between succeeding runs is attributed to non-reproducible throughput from the individual dies due to intermittent pressure surges with the ram press.

Benzo colloid prepared with an ethyl acetate solvent system was used in all the extrusions except Extrusion 14, where the standard diethyl ether alcohol colloid was used. This extrusion, which was performed with die Modification 2, gave a yield of only 46%. This confirmed the results of work with conventional extrusions reported in Section B of the Study which showed that the use of ethyl acetate in place of diethyl ether provided a marked improvement in the processing characteristics of the colloid.

Since part of the problem in achieving high yields was believed to be in the distribution section, the throat openings into the manifold were increased in diameter by 50% (Modification 3). As seen from the data on extrusions 15-18, this resulted in an increase in overall yield of from 71 to 80% and produced essentially equal yields with all three rows. Further modifications (4 and 5) to the breaker plate hole pattern were ineffective in increasing yield, on the contrary, reductions in yield for the last extrusions were noted.

Therefore, it was concluded that the multi-orifice plate die extrusion of spaghetti was technically feasible, and that by optimizing die design and extrusion conditions, overall yields of 80% or higher could be achieved with a long goods extruder and spreader. These yields are contingent on some μ extrusion and are comparable to those achieved in the manufacture of spaghetti.

3. Rod Drying Studies

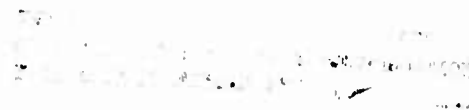
The successful use of a long goods spreader requires that the green strands be draped over a small diameter rod and then dried without deforming. It further requires that the strands have sufficient green strength to be draped over this rod without cracking and breaking at the bend.

From each of 15 mixes made under the **Process and Product Variables Study** discussed in Section B, this was done:

a. The strands from the extrusion were collected in tubs after which they were immediately cut into rough 40-inch lengths and draped over $7/8$ inch wooden dowels.

b. The extrusion was intermittently performed so that as each strand reached the floor, the press was stopped and the strands were cut from the press, and draped over a $7/8$ inch wooden dowel.

It can be seen from the photographs (Figures 7-11) that the strands which were coiled into the tubs (designated by x) had after only a few minutes taken on sufficient set so that they were curled and deformed



after drying. The strands which were cut from the press (designated by S) without having been coiled (Figures 8-14) dried into straight lengths.

As regard to the strength of the strands, this varied from mix to mix. It can be seen in Figure 7, 14 and 15 of Appendix C, that the mix EP-14, prepared using ethyl acetate in place of diethyl ether in the solvent system, showed no evidence of cracking at the bend and exhibited physical characteristics which would be satisfactory for rod drying. As seen from the data in Table 2, Appendix C ethyl acetate compares favorably with the common solvents used in propellant manufacture from the standpoint of ignitability, toxicity and explosive limits.

4. Improvements with Batch Type Process

The existing Picatinny batch type manufacturing process (Flowsheet Figure 16, Appendix C) was scaled up for high capacity production at nominal manufacturing cost with "proveout" and incorporation of these improvements:

a. The drying time was reduced from 10 to 6.5 days by increasing the drying temperature from 104°F to 131°F .

b. The capacity of the facility was increased 13 fold by reducing the time on the strand drying reels to 12 hours.

c. Safety and economy was improved in the cutting operation by utilizing a Diamond Paper Cutter (formerly used for wafering solventless double base propellant) via design and installation of indexing

blocks and scatter guards. The change permitted going from a shielded paper cutter operation working with 1/10 pound quantities to a remote operation working with two pound quantities.

d. The incorporation of rework improved the raw materials utilization from 45 to 90% +.

5. Detonation Hazards Studies

To assess the detonability of benite colloid, a series of pipe detonation tests were performed using a test set-up which has been developed at Picatinny for this purpose. Colloid in crumb form as taken from the mixer, as well as consolidated blocks, was subjected to tests. Wet sand was used as inert simulant and M10 colloid as a control.

It can be seen from the drawing, (Figure 18, Appendix C) and the photograph, (Figure 19, Appendix C) that the test material is initiated by a relatively heavy charge (J-2 Engineer Blasting Cap and .47 pounds of Teteryl) under conditions of severe confinement. The major criterion in assessing the results is the amount of indentation produced in the one-inch mild steel witness plate located approximately three charge diameters from the initiating explosive. Other data collected is the weight of unconsumed material, and the number and size of the pipe fragments.

Tests results are summarized in Table 7 and photograph Figures 20-26 (Appendix C). The photographs and data indicate that consolidated blocks of both benite and M-10 colloid behaved similarly, being

only partially consumed and producing no evidence of detonation. However, the loose grains were completely consumed for both M10 and benite. In the case of benite, a .125 inch indentation is viewed to be evidence of a low order detonation, and duplicates almost exactly the value obtained for M6 colloid in a test series previously reported (Reference 3). For comparison purposes, a tabulation of data from this test and previous test series with various propellants is given below:

<u>Propellant</u>	<u>Witness Plate Indentation, in</u>	<u>Explosive Hazard Classification</u>
M16 (Double Base Solventless)	Complete shattering	9
M15 colloid	0.532	9
M6 colloid	0.117	2
M10 colloid	0	2
CBS-125K (low energy, ammonium nitrate- composite)	0.123	2(Recommended)
Benite	0.125	

Based on the above comparison, benite colloid appears no more sensitive to detonation than single base or low energy composite propellants and can be considered as a Class 2 material during extrusion.

6. Economic Analysis of Proposed Production Engineered Process

A mill cost analysis was made for the manufacture of benite strands at the rate of 50,000 pounds per month by (1) scale up of the current R&D process, and (2) a proposed production engineered process based on spaghetti-type processing techniques involving continuous extrusion thru a plate die long goods spreader combination, and final cutting by means

of a rotary split disc cutter. Flowsheets for the R&D and Production Engineered Processes are in Figure 16 and 17 respectively (Appendix C).

A breakdown of manufacturing costs for the two processes, as well as a cost estimate for the proposed spaghetti type equipment are contained in Tables 3-6, Appendix C. The analysis is summarized below:

	<u>R&D Process</u>	<u>Prod. Engr'd Process</u>
Mat'l costs/100 lbs.	\$ 70.84	\$ 64.00
M-hrs/100 lbs.	21.28	7.84
Total mill cost/100 lbs.	227.78	121.82

Amortization Period 1.1 months

It can be seen from these results that a savings in manufacturing costs of \$106 per 100 pounds is projected by using the proposed production engineering process -- which at a rate of 50,000 pounds per month would permit a payout period for the prototype equipment of 1.1 months.

REFERENCES

1. H. H. Hassman, Evaluation of Benite, an Extruded Form of Black Powder in Separate Loaded Ammunition (U) Reprint #1, August 1958, Confidential.
2. Portsmouth Arsenal Description of Manufacture for Benite Strands (U) #5-4-48.
3. E. P. Haselton et al., Industrial Engineering for Dart XM-23, JATC Propellant and Igniter (C) DB-TR4-59, April 1959.

APPENDICES

APPENDIX A

TABLE 1

REVISIONS EFFECTED TO BENITE SPECIFICATION REQUIREMENTS

<u>TEST</u>	<u>PA-PD-1741</u> <u>(9 April 1959)</u>	<u>MIL-P-45451</u> <u>(27 Jan 1960)</u>
Total Moisture	0.5%	1.0%
Total Volatiles	0.75%	2.25%
Specific Gravity	1.7 $\pm$ 0.2	1.65 $\pm$.15
Hygroscopicity	1.9% Max.	Eliminated
Functioning	Fire 15 ea. at -65°F and +160°F	Eliminated
Potassium Nitrate Ingredient	Recrystallize or Reagent Grade	Commercial Grade
134.5°C Heat Test (Time for Methyl Violet Test Paper To Change to Salmon Pink)	Not Less Than 40 Min.	Not Less Than 35 Min.
Straightness (200 $\pm$ 20 Grain Bundle)	Thru 0.420" D Ring Gage	Thru 0.413" D Ring Gage

TABLE 2

FIRING RESULTS WITH HIGH MOISTURE AND VOLATILES
BENITE IGNITER MATERIAL, XM-79, T300E53 (90mm) ROUND

Lot No. 1	No. of Rds. (1)	Round Conditioning Temp. ° F.	TV "	TM "	Instr. Velocity (ft./sec.)		Max. Press. (PSI)			Std. Dev.	Time Element (ms.)		
					Avg.	Dispers.	Cu. Gage	H.F. Gage	Cu. Dispers.		Ignition Delay	Total Functioning	
SERIES I													
PAE29139	9	-40	2.11	0.85	3927	40	13.5	44700	-----	1300	470	-----	-----
PAE28*03	10	-40	1.59	0.43	3922	40	15.8	44100	-----	2400	479	-----	-----
SERIES II													
PAE29796	4	-40	2.96	1.5	3922	71	29.1	46160	53200	2700	1030	17.4	35.3
RADSR-31B	6	-40	1.86	0.55	3902	48	17.2	45270	51960	3400	400	32.2	48.7
PAE29796	3	70	2.96	1.5	4069	53	22.1	49400	60000	200	100	9.9	32.5

Round - T300 E 53 Round-Stab crimped 1" Stabs for bullet full 5000 lbs.

Propellant - M-17 RAD #60654 (web 0.0481" M.P.) Chg. wt. 8 lb, 4 oz.

Projectile - M353 (wt. 24.1 lbs.)

Case - T24 B1 2)

Primer - XM-79E1 (benite - 380 grains)

Vehicle - 90mm Gun, T139 No. 1087 Tube #48530 with 49 (Series 1) and 92 (Series 2) rounds previously fired.

NOTE:

- 1) Following the warming and standardization round firings, the two series (shown above) were each fired in alternating order.
- 2) The steel cartridge cases were deformed at the mouth due to heavy crimping. Chambering was achieved via machine grinding of metal from the shoulder of the crimp.

TABLE 3

RESULTS OF STATIC FIRINGS IN THE XM - 79E1 PRIMERS

Benite Lot No.	Condition- ing Temp. F	TV	TM	Max. Press. PSIG	Ignition 1st Flash Hole	Delay, ms. Last Flash Hole	Burning Delay ms.	Remarks
SR-31B	70	1.86	0.55	5024	1.0	1.9	.9	1st Radford
"	"	"	"	4429	.7	1.7	1.0	Prod. Lot
"	"	"	"	5233	.5	1.0	.5	"
"	"	"	"	4270	1.0	1.7	.7	"
"	"	"	"	4898	2.3	2.2	--	"
"	"	"	"	7850	2.0	2.1	.1	"
Average				5284	1.25	1.77	.52	"
PAE29796	70	2.96	1.5	4643	1.2	1.2	0	High
"	"	"	"	6707	1.3	1.4	.1	Moisture
"	"	"	"	5854	.8	1.7	.9	Volatiles
"	"	"	"	7913	22.2*	22.4*	.2	Lot
"	"	"	"	7262	1.2	1.3	.1	"
"	"	"	"	4773	.9	1.2	.3	"
Average				6192	1.08	1.36	.28	"
SR-31B	160	1.86	0.55	3842	.6	1.2	.6	1st Radford
"	"	"	"	4647	.5	1.2	.7	Prod. Lot
"	"	"	"	4773	.6	1.7	.9	"
"	"	"	"	4152	.9	1.5	.6	"
"	"	"	"	4002	1.0	1.6	.6	"
Average				4283	.7	1.44	.74	"
PAE29796	160	2.96	1.5	5349	.6	1.3	.7	High TV &
"	"	"	"	6773	.8	1.3	.5	TM Lot
"	"	"	"	7174	.2	1.0	.8	"
"	"	"	"	5950	.8	1.3	.5	"
"	"	"	"	6072	.9	1.1	.2	"
Average				6264	.66	1.2	.54	"

TABLE 3

(CONTINUED)

Benite Lot No.	Condition- ing Temp. ° F	TV %	TM %	Max. Press. PSIG	Ignition Delay, ms.		Burning Delay ms.	Remarks
					1st Flash Hole	Last Flash Hole		
SR-31B	-65°	1.86	0.55	No Trace	Excessive Delay			1st Radford
"	"	"	"	4180	.7	1.7	1.0	Prod. Lot
"	"	"	"	4037	.7	2.5	1.8	"
"	"	"	"	4984	11.6	12.3	.7	"
"	"	"	"	4242	5.4	6.2	.8	"
"	"	"	"	4270	3.4	24.4	1.0	"
Average				4342	8.4	9.42	1.02	"
PAE29796	-65°	2.96	1.5	4524	4.9	5.4	.5	High TV &
"	"	"	"	5864	.5	1.4	.9	TM Lot
"	"	"	"	9043	1.8	1.9	.1	"
"	"	"	"	7049	2.7	2.6	---	"
"	"	"	"	6012	1.3	1.3	0	"
"	"	"	"	7375	2.0	2.1	.1	"
Average				6645	2.2	2.45	.25	"
PAE28203	-65°	1.59	0.43	6235	.7	1.1	.4	R&D St'd.
"	"	"	"	7471	.5	1.2	.7	"
"	"	"	"	7262	1.5	2.0	.5	"
"	"	"	"	5054	.6	1.2	.6	"
Average				6505	.78	1.4	.55	"
PAE29139	-65°	2.11	0.85	7073	.6	1.4	.8	1st Prod.
"	"	"	"	6686	.8	1.3	.5	Lot P.A.
"	"	"	"	6000	.5	1.2	.7	"
Average				6587	.6	1.3	.7	"
PAE29139	70°	2.11	0.85	4702	.9	1.2	.3	"
PAE28203	70°	1.59	0.43	6337	.5	1.0	.5	R&D St'd.

TABLE 4

TABULATION OF MOISTURE ABSORPTION CHARACTERISTICS AND
COST FOR POTASSIUM NITRATE FROM SEVERAL SOURCES

<u>Source or Type</u>	<u>MIL-P-156 Class 1 or 2 (Technical Grade)</u>	<u>Recrystallized</u>	<u>Reagent Grade (J. T. Baker Company)*</u>
1. Percent Hygroscopicity	0.73	0.24	
No. of Lots Averaged	5	4	
2. Sodium Content (Max.)	0.19% **		0.02%
Sodium Content (Analyzed)	0.16%	0.02%	
3. Cost per pound	\$.10	\$.50+	\$.53

Notes

* J. T. Baker's Reagent Grade was used by Radford Arsenal as an alternate for recrystallization.

** This value is obtained from MIL-P-156 in which the percent as sodium oxide is specified as 0.25%.

TABLE 5

EFFECT OF REAGENT GRADE POTASSIUM NITRATE AND PRESENCE OF CHARCOAL
INGREDIENT ON HYGROSCOPICITY AND MOISTURE ANALYSIS OF BENITE

<u>Summary</u>	<u>No. of Lots</u>	<u>Percent Hygro- scopicity</u>	<u>Total Volatiles</u>	<u>Total Moisture</u>	<u>Calc. Residual Solvent</u>	<u>Lot No's</u>
Use of Tech. Grade KNO_3	6	1.60	1.72	.65	1.07	EP-1, 2, 7, 8, 9, 12
Use of Reagent Grade (i. e. Recrystallized) KNO_3	11*	1.47	1.69	.67	1.02	See below
Use of Bakers Reagent Grade (i. e. 0.027% Sodium) KNO_3	1	1.51	2.04	.55	1.49	Radford Lot SR-31B
Use of Ethyl Acetate-DPA in lieu of Diethyl Ether-EC	1	1.03	2.16	.70	1.46	EP-14
Use of Carbon Black & Di-Butyl Phthalate in lieu of Charcoal	1	1.21	0.61**	.50	0.11	EP-15

NOTE* These data were summarized from the PA-PD-1741 Benite Lots listed in Table 6.

NOTE** This value indicates that the characteristic unexplainably high volatiles content of specification benite which is the sum of the total moisture and residual solvent, is due to the charcoal.

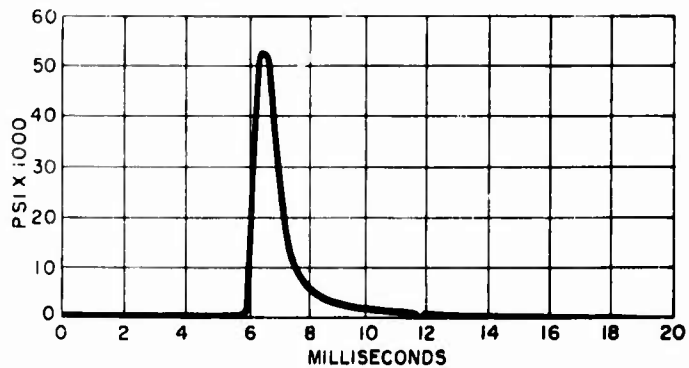
TABLE 6

ANALYSIS OF EARLY BENITE LOTS

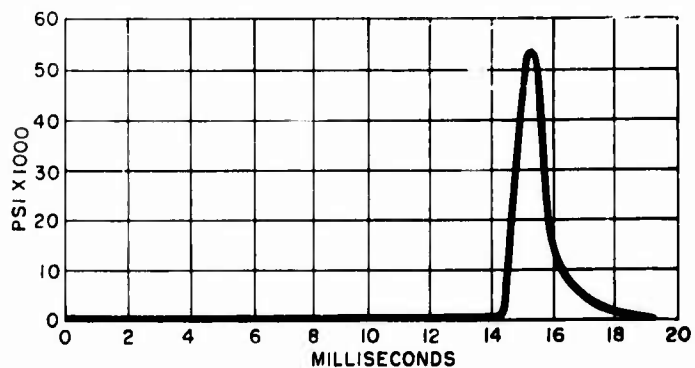
Lot No. PAE	N	C	KNO ₃	S	C	EC	Gran. Dimen.	Var. D.	Sp. Gr.	De. Dimen. in.	Hygroscopicity	Total Moisture	Total Volatiles	Ht. Test 134.5 C	Stability Surveillance 65.5 C	Lab. Report No.	Remarks
23203	38.68	44.41	6.08	10.83	0.55	0.0810	1.48	1.58	1.02	1.28	.43	1.59	45	59-H1-314			
26468	40.92	43.35	6.23	9.5	0.6												
29139	39.91	44.18	6.32	9.59	0.53	0.0775	1.20	1.58		1.20	.85	2.11	40	59-H1-451			
29155	39.21	44.94	6.39	9.46	0.52	0.0750	4.05	1.59		1.41	.60	1.27	45	59-H1-549			
29171	39.04	44.82	6.47	9.67	0.50	0.0764	2.14	1.57		1.51	.85	1.33	45 35	59-H1-528			
29203	40.26	43.74	6.47	9.33	0.55	0.0782	1.07	1.59		1.54	.75	1.60	40	59-H1-562			
29406	40.40	44.32	6.13	9.15	0.60			1.62		1.26	.50	1.69	35	AI-11-59			
29251	40.49	43.86	6.17	9.48	0.58	0.0754	1.28	1.60		1.50	.75	1.99	40	59-H1-591			
29534	39.76	44.36	6.45	9.43	0.53			1.65		1.60	.35	1.55	35	AI-P-26-59			
29796	39.73	44.13	6.51	9.63	0.47	0.0759	1.03	1.62		1.58	.80	2.12	35	AI-P-70-59			
SR-31A	39.60	44.10	6.07	9.64	.59	0.079		1.66	0.0992		.65	4.13		Radford Results			
SR-31B	40.43	43.54	6.04	9.34	.65	0.079		1.64									
SR-31B	39.49	44.76	6.44	9.31	.55	0.077	2.01	1.66		1.51	.55	1.86	45	AL-F-56-59			
EP-8	39.18	44.50	6.58	9.74	.57	0.0801		1.68	1.02	1.61	.70	1.93	45	59-H1-560	25% Rework		
EP-13	39.34	44.56	6.47	9.63	.43	0.0731	3.29	1.62		1.33	.70	2.14	40		100%		

Spec. 40 ± 1 44.3 $\pm$ 1 6.3 $\pm$.3 9.4 $\pm$.3 0.5 $\pm$.1 0.03 $\pm$.005

* Note: Samples into Surveillance ovens November 1959.



High TV Benite, PAE 29796



Standard 1st. Radford Lot, RAD SR-31B Charged Primer

Figure 1. Typical Pressure Vs. Time Traces From 90 mm. Gun Firing At -40°F. With The T-300E 53 Round (Series 2).

- Note:
1. The calibration factor for determining time (milliseconds) from the above traces is 2.5 times the value shown.
 2. The discontinuity of the P Vs. T trace location, the point at which the projectile leaves the muzzle of the gun.

(31) 52927 XM-79E1 PRIMER
P-35 Cal. 5000
P-53 Cal. 9420 P/S4166
P-55 Cal. 5000 8/31/59
Lot PAE 29796 Temp 70⁰F

(32) 52928 XM-79E1 PRIMER
P-35 Cal. 5000
P-53 Cal. 9420 P/S4166
P-55 Cal. 5000 8/31/59
PAE 29796 Temp -65⁰F



Figure 2. Typical Static Firings Pressure Vs. Time Trace For -65⁰F.
Conditioned High Moisture Benite Primer

(1) 52897 XM-79E1 PRIMER

P-35 Cal. = 5K Ch 1

P-53 Cal. 9420 Ch 2 P/S4166

P-55 Cal. = 5K Ch 3 8/31/59

+70 Lot 29796

(2) 52898 XM-79E1 PRIMER

Ch 1 P-35 Cal. = 5K

Ch 2 P-53 Cal. 9420 P/S4166

Ch 3 P-55 Cal. 5K 8/31/59

+160 Lot 29796



Figure 3. Typical Static Firings Pressure Vs. Time Trace For 70°F And +160°F Conditioned High Moisture Benite Primer

(17) 52913 XM-79E1 PRIMER

P-35 Cal. = 5K P/S4166

P-53 Cal. 9420

P-55 Cal. 5K 8/31/59

+160 SRB-31

(18) 52914 XM-79E1 PRIMER

P-35 Cal. = 5K

P-53 Cal. = 9420 P/S4166

P-55 Cal. = 5K 8/31/59

65 SRB-31



Figure 4. Typical Static Primer Pressure Vs. Time Trace For Standard Benite, Lot SR-31B, Conditioned At +160°F. & -65°F.

APPENDIX B

TABLE I

EFFECT OF FORMULATION AND OTHER VARIABLES ON PROCESSABILITY AND END PRODUCT CHARACTERISTICS

Lot No.	Major Variables	Solvent		In-Process Characteristics			Physical and Chemical Test Results														
		Composition (lbs. per lb. NC)	Ratio ²⁾ Ether or ET to Solvent	Screen Press., psi	Extrusion Press., psi	Green Strength	Extrusion Uniformity	Hygroscopicity, %	Total Volatiles, %	Total Moisture, %	Specific Gravity	Diameter, inches	Shrinkage, %	Tensile Strength, (lbs. in ²)	Elongation, %	Drop Test Sensit., in.	Friction Pendulum	134.5°C Heat Test, min.	60°C Surveillance ³⁾	65.5°C Surveillance ³⁾	Heat of Expl. (cal. cm ³)
Standard Formulation Except																					
Composition (lbs. per lb. NC)																					
Ratio ²⁾ Ether or ET to Solvent																					
Screen Press., psi																					
Extrusion Press., psi																					
Green Strength																					
Extrusion Uniformity																					
Hygroscopicity, %																					
Total Volatiles, %																					
Total Moisture, %																					
Specific Gravity																					
Diameter, inches																					
Shrinkage, %																					
Tensile Strength, (lbs. in ²)																					
Elongation, %																					
Drop Test Sensit., in.																					
Friction Pendulum																					
134.5°C Heat Test, min.																					
60°C Surveillance ³⁾																					
65.5°C Surveillance ³⁾																					
Heat of Expl. (cal. cm ³)																					
Notes:																					
1) Average particle diameter of Potassium Nitrate 10-30 microns except as noted.																					
2) Re-work added 15 minutes after Charcoal and Sulfur addition.																					
3) EP-14 Surveillance Samples started November 1959, other samples started July 1959.																					
4) With EP-2 the No. 24 and 35 Screens were omitted; with EP-10 the No. 35 Screen was omitted.																					
5) Omitted fine Grinding and Screening to measure effect due to major variable.																					
6) Friction Pendulum Test using Steel Shoe gave Crackles 8 out of 10 tries, with no sparks, burning, detonation.																					
7) This sample failed the 300 min. (134.5°C) Explosion Test, samples failing after 183, 196 and 206 min.																					
8) Letter OXDBB-TM4 dated 8 October 1959, "Test of Explosion Test."																					

Notes: 1) Average particle diameter of Potassium Nitrate 10-30 microns except as noted.
 2) Re-work added 15 minutes after Charcoal and Sulfur addition.
 3) EP-14 Surveillance Samples started November 1959, other samples started July 1959.
 4) With EP-2 the No. 24 and 35 Screens were omitted; with EP-10 the No. 35 Screen was omitted.
 5) Omitted fine Grinding and Screening to measure effect due to major variable.
 6) Friction Pendulum Test using Steel Shoe gave Crackles 8 out of 10 tries, with no sparks, burning, detonation.
 7) This sample failed the 300 min. (134.5°C) Explosion Test, samples failing after 183, 196 and 206 min.
 8) Letter OXDBB-TM4 dated 8 October 1959, "Test of Explosion Test."

TABLE 2
COMPARISON OF STABILITY TEST RESULTS FOR VARIOUS BENITE LOTS

Benite Lot No.	Heat Test (min.)		Vacuum Stability (40 hrs.) in ml. of gas		110° C Taliani Test			Surveillance	
	134.5 C	120 C	90° C	100° C	Time to 100mm.	Slope @ 100mm.	Slope @ 100 min.	80° C	65° C

I Standard Formulation Benite

PAE									
26467	15 ¹	20 ¹	-	-	-	-	-	-	160mo+
28203	45	140	1.60	11.0 ²	Neg.	Neg.	Neg.		
29171	35	145		3.49	"	"	0.65		
29406	35	115		4.91	164	3.53	0.55		
29534	35	135		4.54	160	0.52	0.60		
RADSR-31B ³	40	135	1.87	6.59	Neg.	Neg.	0.10		
EP-7 ⁴	35	145		-	-	-	-		
EP-9 ⁵	60	300		-	-	-	-		

II Modified Benite using DPA in lieu of EC (Stabilizer) and Ethyl Acetate Solvent in lieu of Ether.

EP-14	50	245	1.74	2.73	-	-	-	-	6
EP-16B	55	300+		1.06	Neg.	Neg.	Neg.		

NOTE:

1. The values shown were obtained from testing the material removed from the 80° Surveillance oven. With the 134.5° C test, explosion resulted in less than 300 minutes of continued exposure. The sample was found to be depleted of Ethyl Centralite Stabilizer.
2. The high vacuum stability value was checked. The indicated impaired stability was not confirmed by a low stabilizer analysis value (the material analyzed 0.51% Ethyl Centralite).
3. A rerun on vacuum stability gave a value of 5.76 ml. The analysis for Stabilizer gave a value of 0.42% E.C.
4. Analysis for Stabilizer 0.45% E.C.
5. Analysis for Stabilizer 0.21% E.C.
6. In addition to surveillance tests on this non-standard benite, surveillance testing is underway on non-standard benite involving lots: EP-3, 4, 5, 8, 10, 12.

TABLE 3

SUMMARY OF BALLISTIC DATA FROM

THREE INCH GUN FIRINGS

No. of rounds.	Round Conditioning Temp. °F	Launcher Material	Lot No.	Primer Environment Conditioning	Muzzle ft sec Av.	Velocity		Cu. gage	Max. Pressure, (psi)		Time Element	
						Muzzle Dispersion	Std. Dev.		H. F. gage Dispersion	Std. Dev.	Ignition Delay (ms)	Total Funct. (ms)
5	70	Bl. Pdr	KOP 7-309	None	3086 (1)	9	3.7	41400	47850	2000	740	26.6
5	70	Bl. Pdr	KOP 7-309	None	3019	28	13.1	40400	46710	800	410	29.0
5	70	Bl. Pdr	KOP 7-309	None	2998	31	11.3	39600	47000	1500	700	28.5
3	70	Bl. Pdr	KOP 7-309	None	3014	7	4.4	39200	51000	1200	730	28.5
5	70	Bl. Pdr	KOP 7-309	None	2991	82	31.7	37000	46750	3600	1440	28.8

NOTE: - The following rounds were fired in Alternating Series Order.												
4	70	Benite-R&D	PAE 28203	None	3049	3	2.0	39400	46210	1800	760	25.2
6	70	Benite-R&D	PAE 28203	4 wks-122°F & 90% RH	2984	52	18.9	37300	47400	1800	690	28.8
6	70	Benite-R&D	PAE 28203	4 wks-170°F	2945	51	17.3	34600	44250	3300	1200	27.7
7	-160	Benite-R&D	PAE 28203	None	3018	18	8.1	43900	51450	2700	950	25.6
7	-40	Benite-R&D	PAE 28203	None	2880	94	39.5	34500	39425	4000	1560	40.6
4	70	Benite-100 Rework	EP-13	None	3014	12	5.3	39200	45300	2700	1140	27.6
6	70	Benite-100 Rework	EP-13	4 wks-122°F & 90% RH	2973	69	26.7	36800	46450	2800	1290	32.5
6	70	Rework	EP-13	4 wks-170°F	2950	81	28.3	35100	44000	3000	1200	29.1
7	-160	Rework	EP-13	None	3091	63	22.7	45300	53250	4100	1310	25.6
7	-40	Rework	EP-13	None	2855	67	25.8	33300	38600	3360	1040	40.4
5	70	Benite-ETAC	EP-14	None	3010	55	25.0	38800	42900	2900	1090	34.3
6	70	Benite-ETAC	EP-14	4 wks-122°F & 90% RH	2987	28	12.0	37300	46900	1700	620	37.3
6	70	Benite-ETAC	EP-14	4 wks-170°F	2980	57	20.8	36300	45700	2100	800	33.8
7	-160	Benite-ETAC	EP-14	None	3085	44	15.8	44900	55100	3600	1140	29.8
7	-40	Benite-ETAC	EP-14	None	2875	79	29.4	33300	39200	3500	1360	55.3

Round - (Non Standard round used) Propellant M-17 RAD #6054 (web 0.0481 in. M. P.) Chg. wt. 4 lb., 12 oz.

Projectile M-42A1 Inert loaded to 12.8 lbs.

Case MK 2 M2 Brass (resized). Not crimped; drilled 10 in. from base

Primer M28 B2, Benite 280 gr., M-61 Element; M1 B1 A2 Head

3 in. Gun #788 Model M-5

Tube - 3 in. #7220; Drilled for P-t work; condition: Worn (2068 previous firings)

NOTES: (1) Chronograph readings found to be approx. 60 ft sec high after these 5 rounds had been fired.

TABLE 4

A TABULATION OF STATIC FIRING RESULTS WITH EXPERIMENTAL LOTS OF BENITE

Lot No.	Lot Identity	Avg. Maximum Pressure		Avg. Ignition Delay		No. Rounds Fired					
		-65 F	70 F	-65 F	70 F	-65 F	70 F				
		lbs./in. ² order*	lbs./in. ² order*	ms. order*	ms. order*						
Lots Involving Composition Variations.											
EP-14	Ethyl Acetate & DPA	3996	1	4261	1	1.3	1	1.7	5	3	
- 5	4% DNT; DBP, DPA	2912	11	2860	7	4.9	11	5.6	14	2	3
- 6	2% DBP; DPA	2748	15	2756	10	8.4	15	5.4	13	3	2
- 3	40% Pyrocellulose, DPA	3180	8	3316	4	2.6	5	1.3	2	3	3
- 4	33.5% Pyrocellulose, DPA	3591	4	3204	5	7.2	14	1.6	4	3	3
-15	Carbon Black, 2% DBP, DPA	2860	13	2189	17	13.3	16	7.2	16	3	3
Lots Involving Use of Coarse Potassium Nitrate.											
EP-2	60 - 80 Mesh	2740	16	2240	15	17.0	17	6.8	15	2	3
-10	100 - 200 Mesh	3251	7	2502	14	3.3	8	1.5	3	3	3
Lots Involving Use of Rework.											
EP-8	25% Level	3363	5	3056	6	2.7	7	1.9	9	3	2
-13	100% Level	3705	2	4143	2	1.8	3	2.4	10	3	3
Lots Involving Standard Formulation.											
EP-1	Std Formulation	2856	14	2697	11	1.6	2	1.2	1	2	3
-7	Nitrate & Solvent	3063	10	2800	9	4.3	10	1.8	7	3	3
-9	Chgrd Together	3119	9	2812	8	2.7	5	2.4	11	3	3
-11A	Nitrate & Solvent	2870	12	2670	12	5.0	12	1.8	8	2	3
-11B	Used 0.080" Dies	3274	6	2592	13	6.8	13	3.3	12	3	3
-11C	Used 0.102" Dies	2681	17	2212	16	3.3	9	7.7	17	3	3
-12	Used 0.117" Dies	3688	3	3382	3	2.4	4	1.8	6	3	2
	Linters Type NC										

* Order refers to ranking of each of 17 lots considering them as a group and based on the following: Lot having highest pressure considered #1; Lot having shortest ignition delay considered #1.

NOTE:

1. The benite strands were tested in M-28B2 primer hardware using M-61 percussion elements and T88E1 Primer heads.
2. The test set-up included clamping the loaded primer containing 250-270 grs of benite strands into static firing fixture, attaching pressure sensing cells to the 1st and last flashhole, and remotely functioning same. A cathode ray oscilloscope with drum camera records the pressure cell trace and thiotron pulse shift at firing pin impact. Typical static traces are shown, see Appendix A, figures 1-4 incl.
3. An Ignition Delay greater than 50 milliseconds with -65° F conditioned primers was experienced with a single primer from each of lots EP-1, 5, 11A, also a 70° F conditioned primer from Lot EP-8 gave a 50 plus millisecond delay.
4. Of the 102 rounds one percussion element, M-61, was a dud.

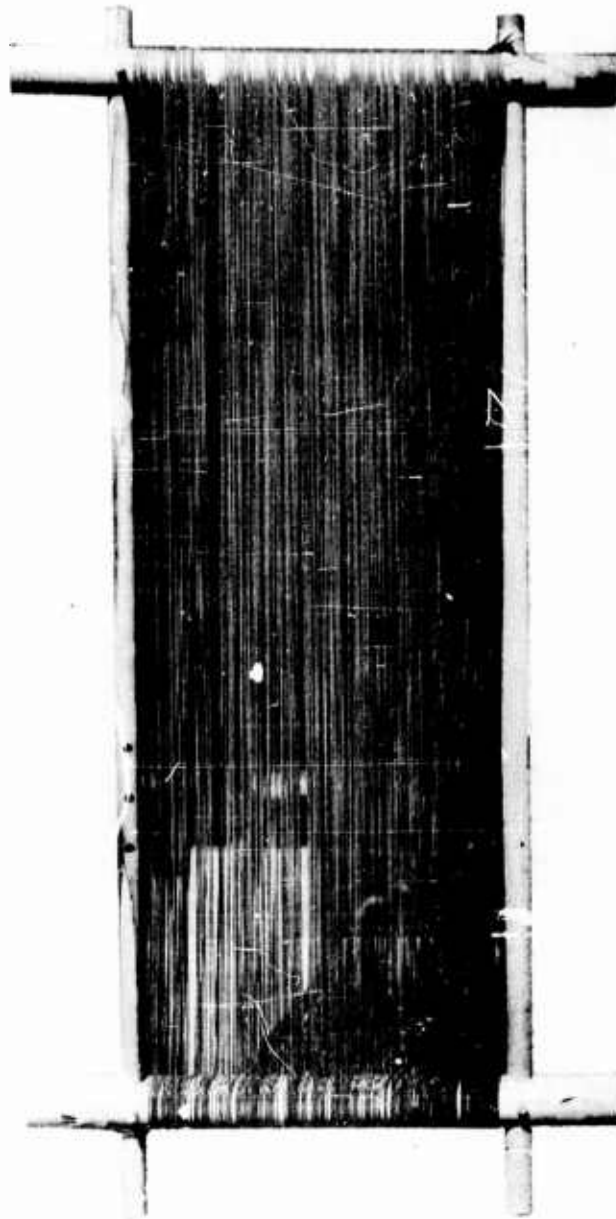
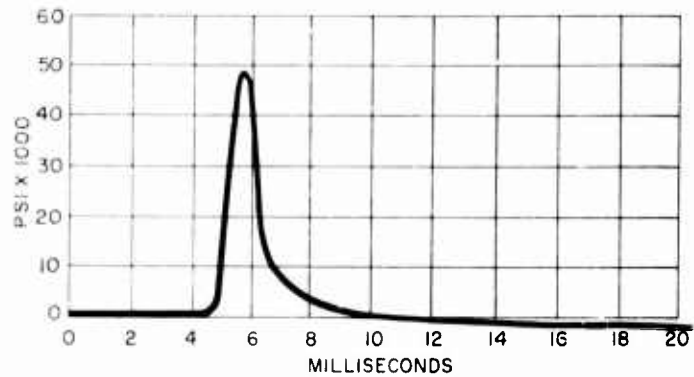
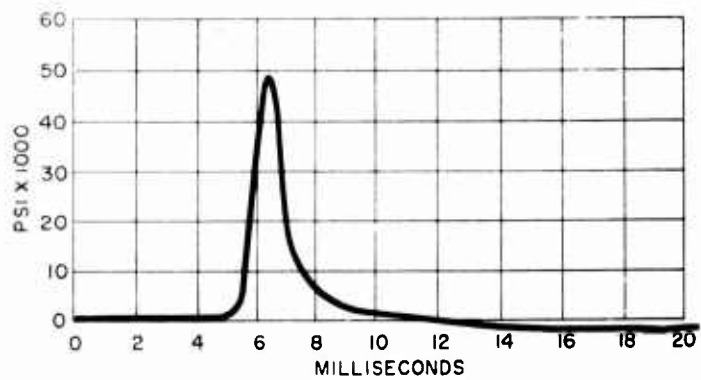


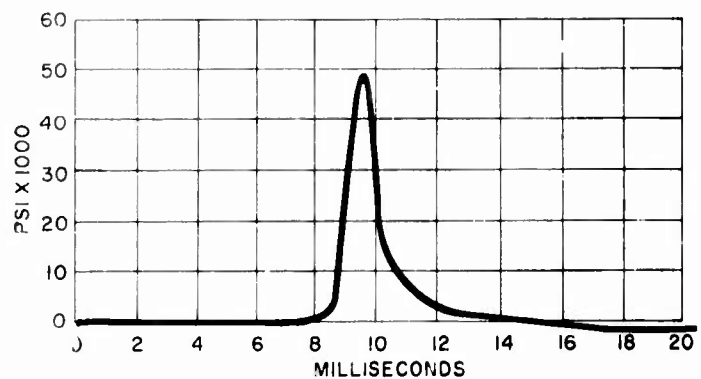
Figure 1. Benite Strands On The Picatinny
Benite Tension Drying Rack



Black Powder As Ignition Material



100% Rework Benite As Ignition Material



Ethyl Acetate Colloided Benite As Ignition Material

Figure 2. Typical Pressure Vs. Time Traces From Three-Inch Gun Firings For Several Benite Lots

APPENDIX C

TABLE 1
YIELD RESULTS WITH PILOT PLANT EXTRUSION PLATE DIE

Extrusion No.	Modification No.	Mix. No.	Percent Yield, %			Av. 55.5	Ext. Press, psig	Remarks
			Row A	Row B	Row C			
1.	1	16A	61	45	61	55.5	750	Breaker Plate Holes
2.	1	"	57	37	60	54.1	750	0.185 in. Das Shown
3.	1	"	47	23	48	39.2	650	on Dwg. - EP-101866,
4.	1	"	47	44	54	48.5	750	Sheet 4
5.	1	"	41	56	48	48.1	750	"
6.	1	"	45	45	71	54.0	1100	"
7.	1	"	47	45	65	52.5	1100	"
8.	1	"	63	37	51	50.7	1100	"
9.	1	"	54	27	61	48.2	1100	"
10.	2	"	58	55	69	60.9	1050	Middle Row of holes
11.	2	"	51	51	60	53.8	1050	opened to .205 in.
12.	2	"	71	52	59	60.6	1100	"
13.	2	"	68	46	60	57.6	1100	"
14.	2	PAL 30237	51	37	50	46.4	1000	Ether-Alc colloid

12/24 screens on top
of Breaker plate.

15.	3	16B	83	78	78	80	700	Opened in manifold throats. DX 1, 5 D
16.	3	"	75	76	76	76	750	"
17.	3	"	69	71	72	71	750	"
18.	3	"	72	68	74	72	750	"
19.	4	"	71	64	70	69	800	#24 screen omitted
20.	4	"	52	55	58	55	700	"
21.	4	"	77.3	67.9	73.9	73.0	1000	(Breaker Plate holes (as follows increased (0.020 in. D: A-14, (C-8, 10, 16, B-7, 8, 9, (10, 11, 16, 17
22.	4	"	75.3	65	71	70.5	1000	"
23.	4	"	76.5	61	76	71.5	1000	"
24.	5	16C	82	70	74.5	76	500	(Holes B-7, 8, 9, 11, 16 (17 opened an additional (0.020 in.
25.	5	"	61.5	51.5	51	55	500	"
26.	5	"	69.5	62	56	62.5	600	"
27.	5	"	70	62.5	69	67	600	"
28.	5	"	68.5	70	67	68	600	"
29.	5	"	81	68.5	71.5	74	600	"

TABLE 2

COMPARISON OF TOXIC AND EXPLOSIVE
PROPERTIES OF SEVERAL SOLVENTS

I. MAC - Maximum allowable concentration in air for an eight-hour period.

Ethyl Acetate	- 400 ppm
Diethyl Ether	- 400 ppm
Acetone	-1, 000 ppm
Ethyl Alcohol	-1, 000 ppm

II. Flash Point - Temperature at which vapors from an open or closed cup of solvent will ignite.

	Closed Cup	Open Cup
Diethyl Ether	-20°F	---
Acetone	0°F	15°F
Ethyl Acetate	24°F	30°F
Ethyl Alcohol	55°F	---

III. Explosive Limits (Percent Volume in Air)

	<u>Lower</u>	<u>Upper</u>
Diethyl Ether	1.7 percent	48.0 percent
Acetone	2.15 percent	13.0 percent
Ethyl Acetate	2.18 percent	11.5 percent
Ethyl Alcohol	3.28 percent	19.0 percent

IV. Reference

I (above): NI Sax Dangerous Properties of Industrial Materials

II & III (above): Lange, Handbook of Chemistry

TABLE 3
BENITE RAW MATERIALS COST BREAKDOWN

Ingredient	Specification	Cost \$/100 lbs.	Nominal Formula Percent	Cost for R&D Process, \$/100 lbs.	Cost for Prod. Engr'd Process, \$/100 lbs.
1. Nitrocellulose	JAN-N-244				
	GR C, Type 1	30.00	40.0	\$12.00	12.00
2. Potass Nitrate	MIL-P-156	9.50	44.3	4.21	4.21
	Class 1 or 2				
3. Sulfur	JAN-S-487	5.85	6.3	.57	.37
	Grade A				
4. Charcoal	JAN-C-178	6.00	9.4	.56	.56
	Class D				
5. Ethyl Centralite	JAN-E-255	92.80	0.5	.46	--
	Grade 2				
6. Diphenylamine	JAN-D-98	38.70	added	--	.12
7. Ethyl Alcohol	MIL-E-462A	9.70	19. lbs.	1.84	1.84
	Grade 2				
8. Diethyl Ether	JAN-E-199	15.00	45. lbs.	6.75	--
	Grade A				
9. Ethyl Acetate	MIL-E-11266 (Grd C)	17.00	32. lbs.	--	5.44
Book Cost per 100 lbs.				26.19	24.61
Est. Yield				80%	95%
Actual Cost per 100 lbs.				32.74	25.90

TABLE 4

LABOR REQUIREMENT COMPARISON IN MANHOURS FOR R&D AND
PRODUCTION ENGINEERED PROCESS FOR 50,000 LBS. OF BENITE
STRANDS PER MONTH, 20 DAY, TWO SHIFT PER DAY OPERATION

<u>Operation</u>	Man Hours	
	<u>R&D Process</u>	<u>Per Shift Prod. Engr'd Process</u>
1. Ingredient Preparation	16	16
a. Grind potass. nitrate, 600 lbs.		
b. Dehy & break Nitrocellulose, 500 lbs.		
c. Weigh out mat'ls for 250 lb. mixes		
2. Mix	16	16
3. Block	8	--
4. Extrude	56	8
5. Straighten & Pre-dry	80	8
6. Cut to Length	48	8
7. Final Air Dry	16	16
8. Pack (26-48 lb cartons)	<u>26</u>	<u>26</u>
Total (M. H. per Shift)	266	98
Total (M. H. per 100 lbs)	21.28	7.84

TABLE 5

A PROJECTED MILL COST COMPARISON IN DOLLARS PER 100 LBS.
FOR A 50,000 LB PER MONTH BENITE FACILITY SHOWING THE R&D
PROCESS COST VS. THE PRODUCTION ENGINEERED PROCESS COST

<u>Item</u>	<u>Basis</u>	<u>R&D Process</u>	<u>Production Engineered Process</u>
Material, Raw	See Table #2	\$32.74	\$25.90
Material, Packing Actual Cost incurred at P. A.		38.10	38.10
Labor	\$2.50/rmb (See Table #3)	53.20	17.60
Supervision & Clerical	10% of D. L.	5.32	1.96
Maintenance	25% of D. L.	13.30	4.90
Utilities	10% of D. L.	5.32	1.96
Overhead	150% of D. L.	<u>79.80</u>	<u>29.40</u>
	Total	\$227.78	\$121.82

TABLE 6

PROJECTED COST SAVINGS USING SCREW EXTRUDER, SPREADER AND
LONG GOODS CUTTER FOR 50,000 LB. PER MONTH FACILITY

1. Installed Cost of Prototype "Long Goods" Equipment

a. Extruder and Spreader -----	\$16,000
b. Long Goods Stick Remover & Cutter-----	\$11,250
Installation (40%)-----	\$10,900

Sub Total \$38,150

2. Modification Cost for Use With Explosive Materials

a. Design and Shop -----	\$10,000
*b. Instrumentation (10%)-----	\$ 4,000
*c. Conveying Equipment (15%)-----	\$ 6,000

Sub Total \$20,000
\$58,150

Total

3. Manufacturing Savings Anticipated
(from Table #4)

a. Cost per 100 lbs with R&D Process -----	\$227.78
b. Cost per 100 lbs with Prod. Engr'd Process--	\$121.82
c. Savings per 100 lbs (a-b)-----	\$105.96

4. Amortization Period Based Upon Labor and
Materials Savings-----

\$58,150
\$105.96 x 500 = 1.1 mo.

*Required For Remote Operation

TABLE 7

DETONATION TESTS WITH SOLVENT WET BENITE
IN LOOSE AND CONSOLIDATED FORM

Item	Benite			MJO-Propellant			Wet Sand
Test Number (i.e., see marked enclosed photographs)	1	2	4	5	6	7	3
Condition of Material	Blocks	Blocks	Crumbs	Blocks	Blocks	Crumbs	Packed
Wt. of Material Charged, (lbs.)	11.78	11.94	8.00	10.05	9.97	6.52	--
Wt. of Material Recovered, (lbs.)	5.47	3.13	0	3.25	3.30	0	--
Pipe Fragments Recovered, (%)	30	25	25	25	25	30	95
Fragments 5 in.	4	3	1	4	2	4	2
Fragments 5 in.	1	1	8	2	3	9	None
Indentation, Witness Plate, (in)	None	None	0.125	None	None	None	None

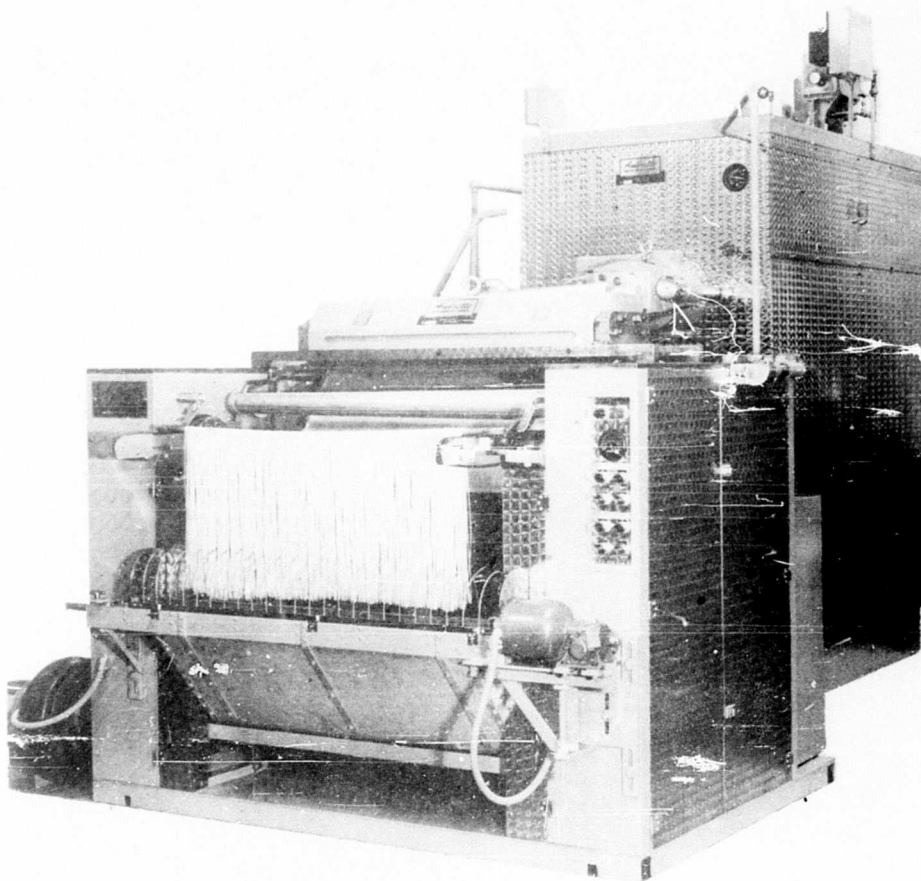


Figure 1. Typical Long Goods Extruder - Spreader

Equipment arrangement and trimming operation.

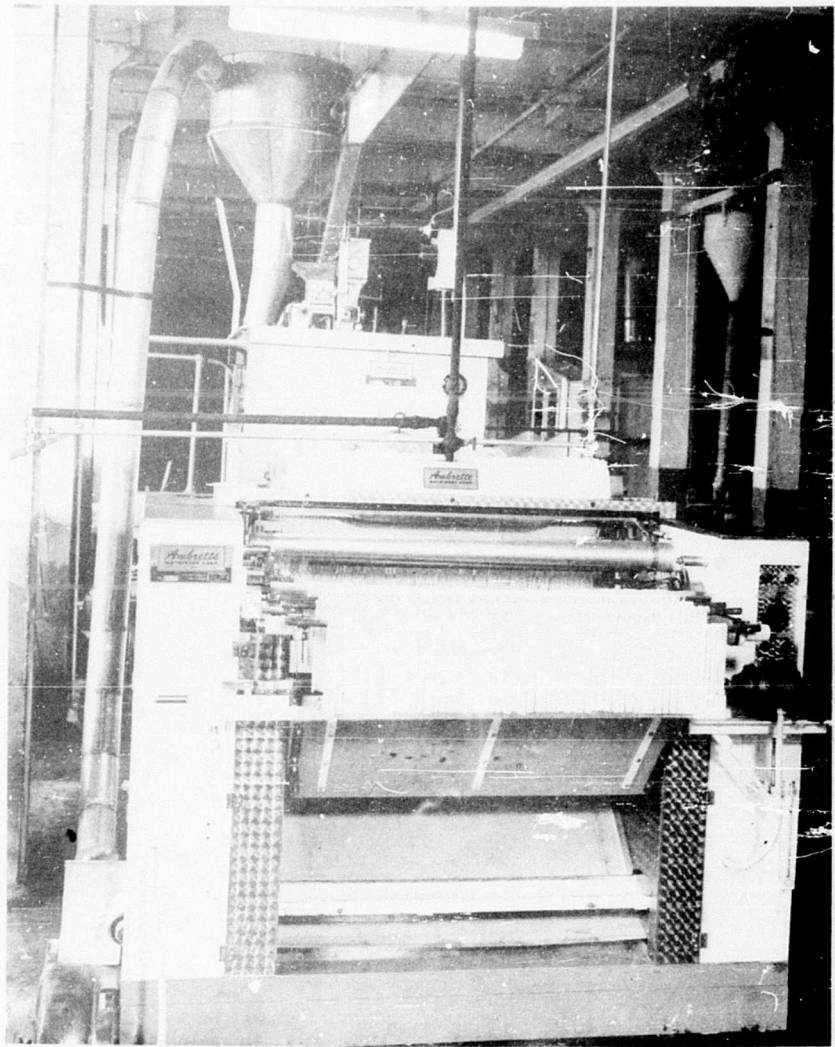


Figure 2. Typical Spaghetti Spreader

Equipment showing hopper, conveyor and blower-return for trimmings.

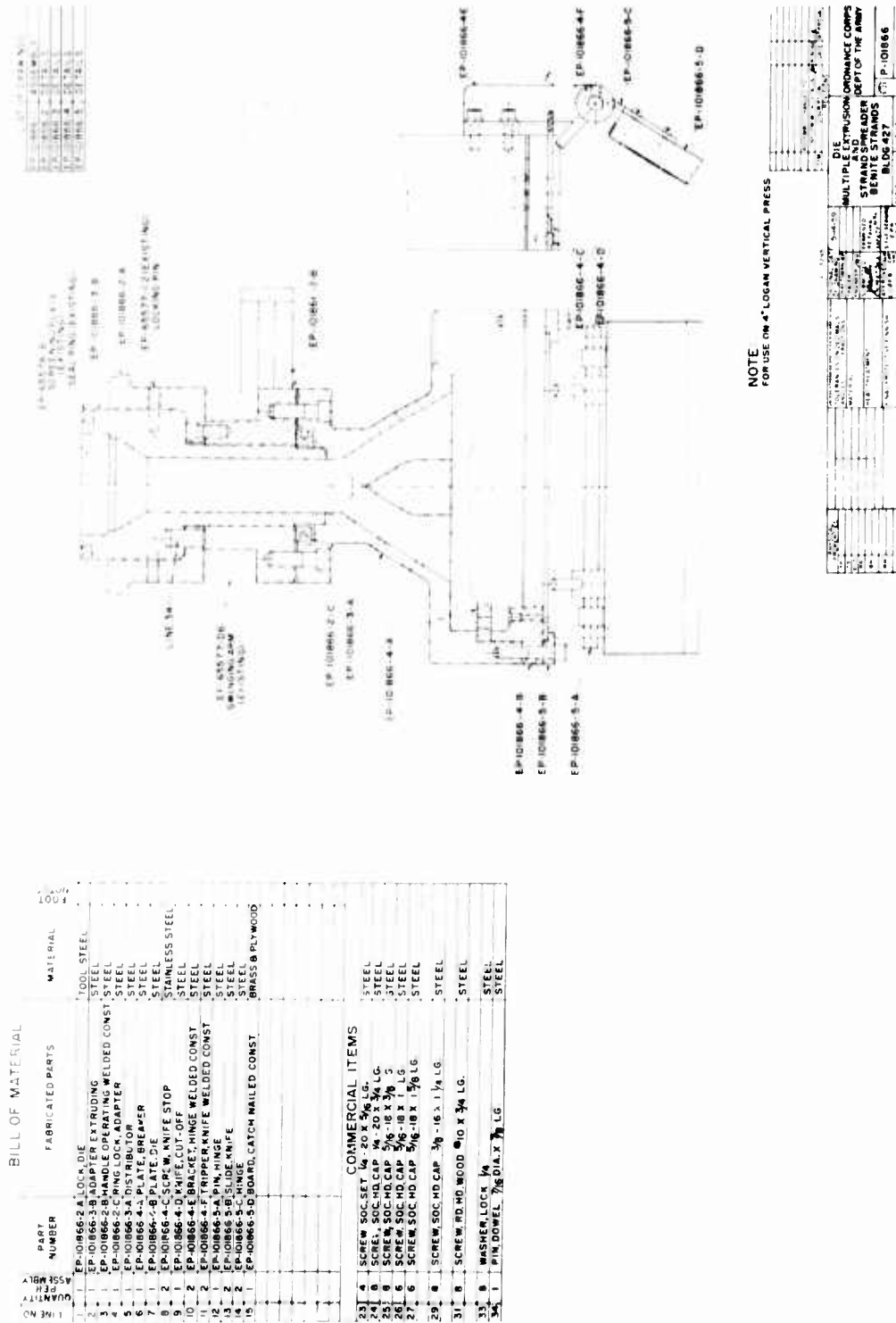


Figure 3. Die, Multiple Extrusion And Strand Spreader For Benite Strands

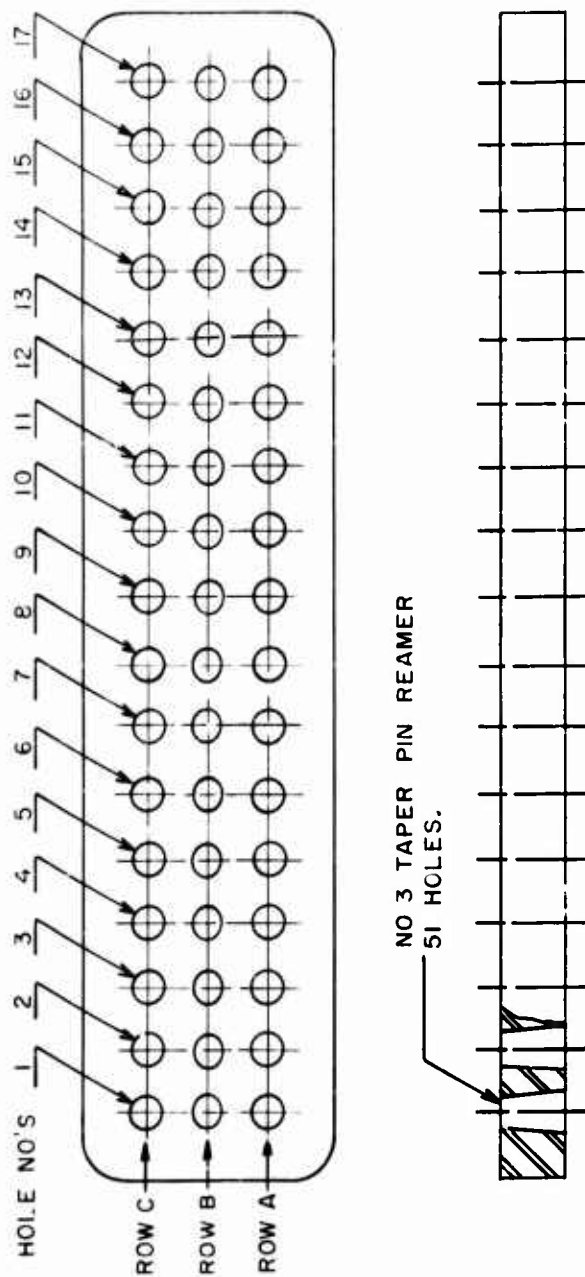


Figure 4. Breaker Plate For 51 Way Multi-Orifice Extrusion Die

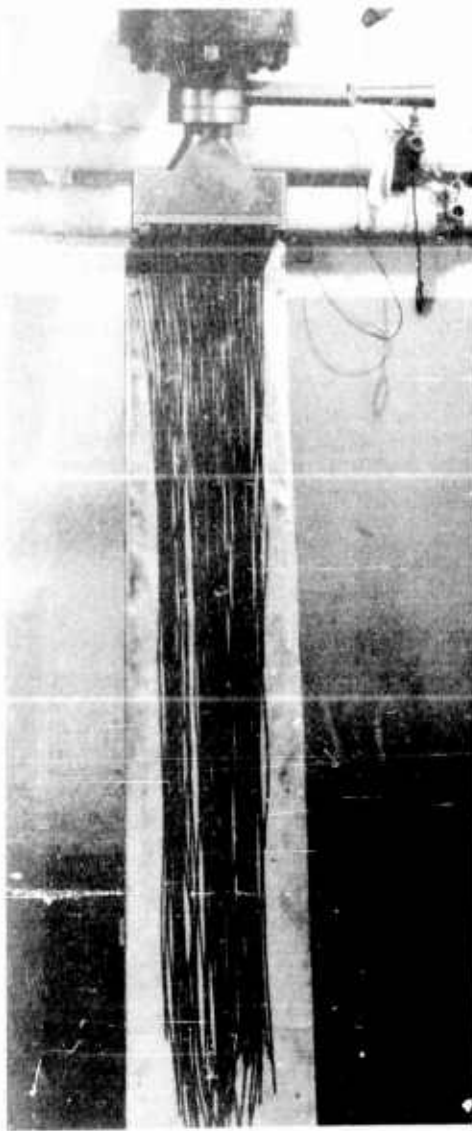


Figure 5. Perfect Flow Pattern

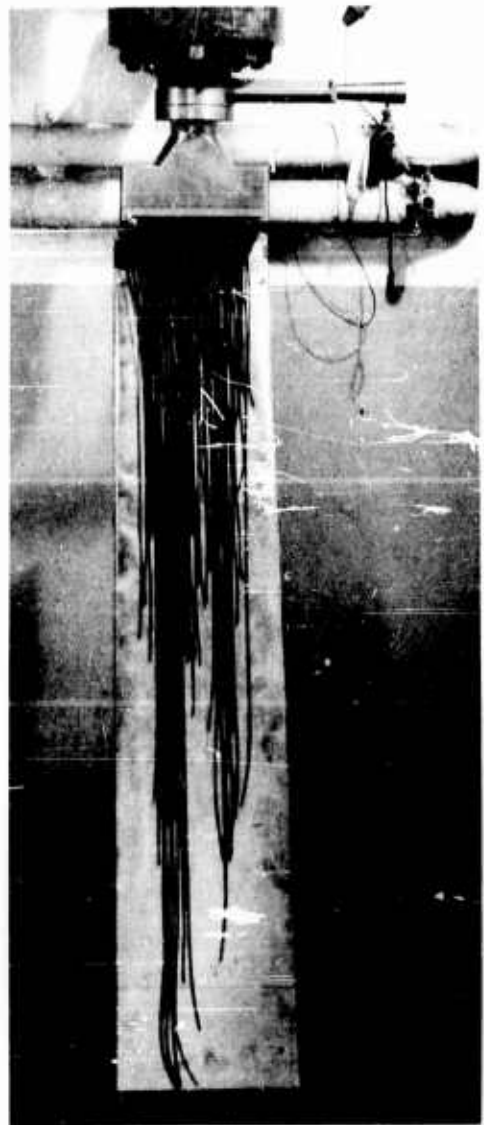


Figure 6. Flow Pattern Of Modified Benite Through The 51 Way Die

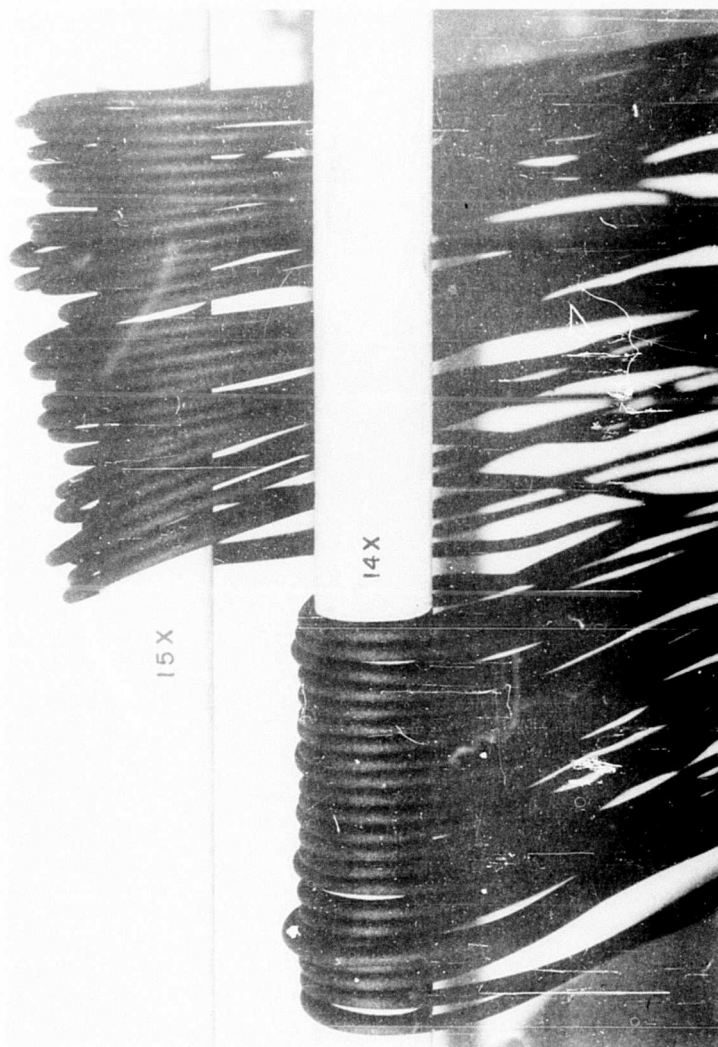


Figure 7. Comparison Of Strand Straightness (Top View)

For ethyl acetate colloided benite (Lot EP-14) Vs. diethyl ether colloided benite having carbon black in lieu of charcoal (Lot EP-15).

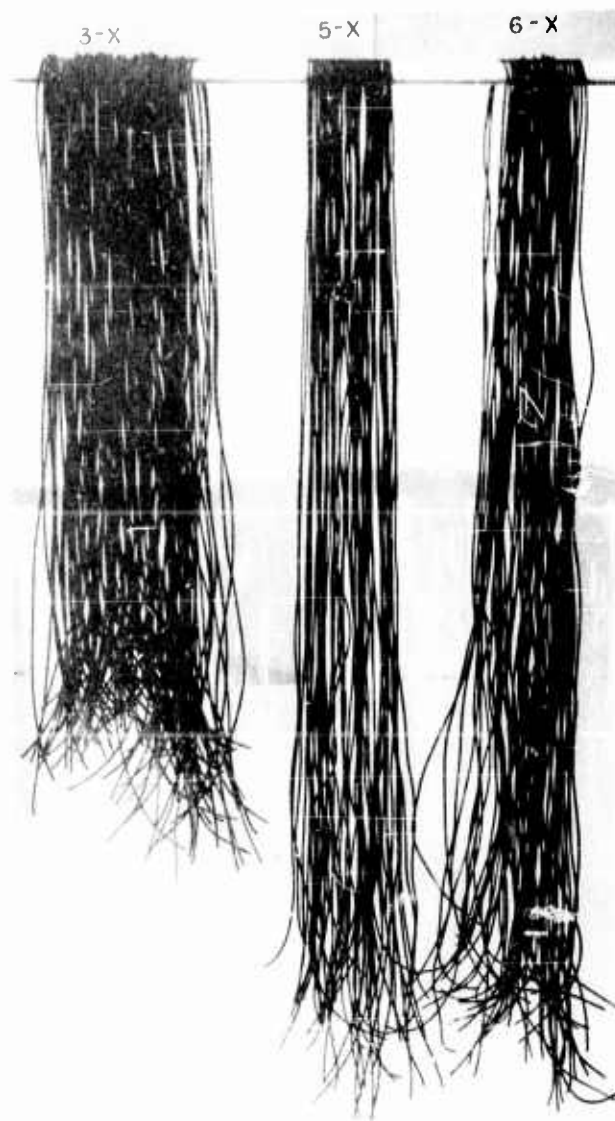


Figure 8. Comparison Of Three Modified Formulation Benite Lots For Straightness After Drying (Front View)



Figure 9. Straightness Comparison Of Dried Strands (Front View)

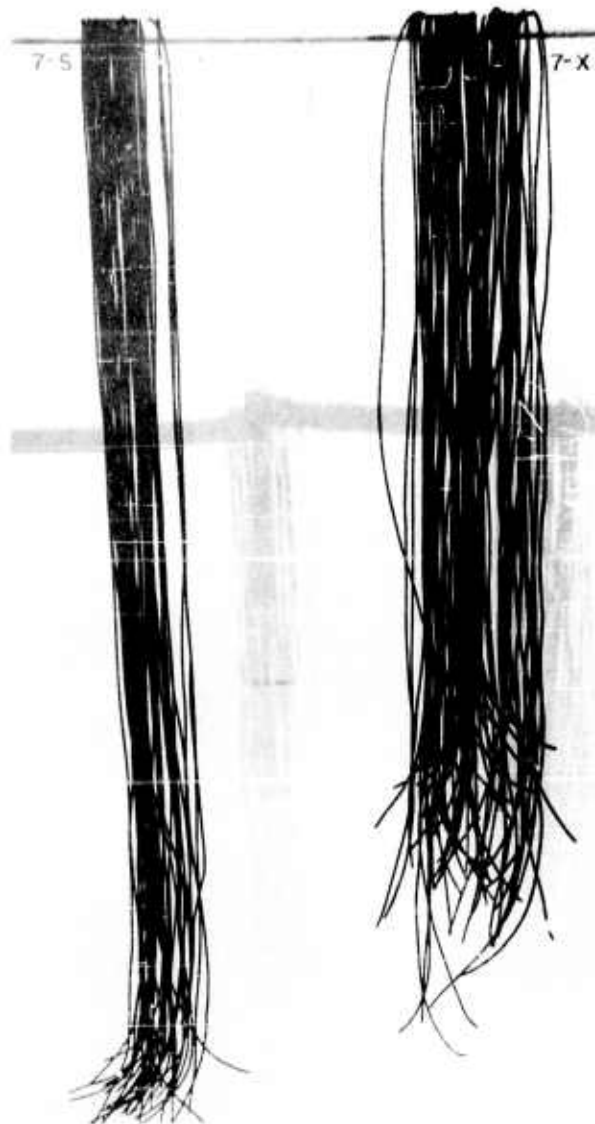


Figure 10. Straightness Comparison Of Inverted
Mix Cycle Benite (Front View)

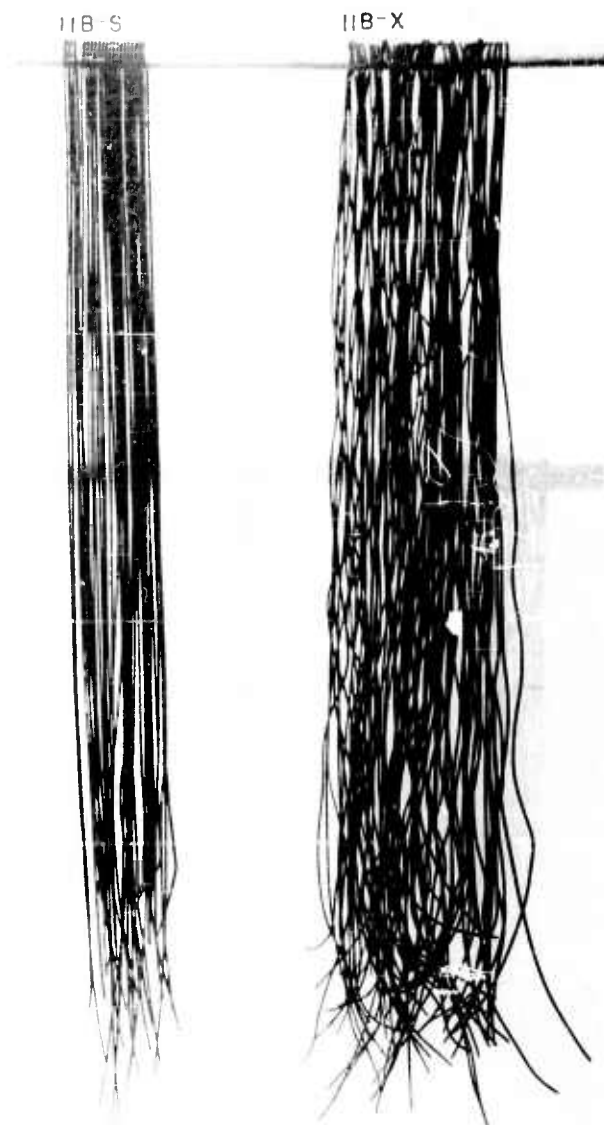


Figure 11. Straightness Comparison Of Standard Benite (Front View)



Figure 12. Comparison Of Three Modified Formulation Eenite Lots For Solvent Wet Plasticity And Strength (Top View)



Figure 13. Comparison Of Three Modified Formulation Benite Lots For Straightness After Drying (Front View)

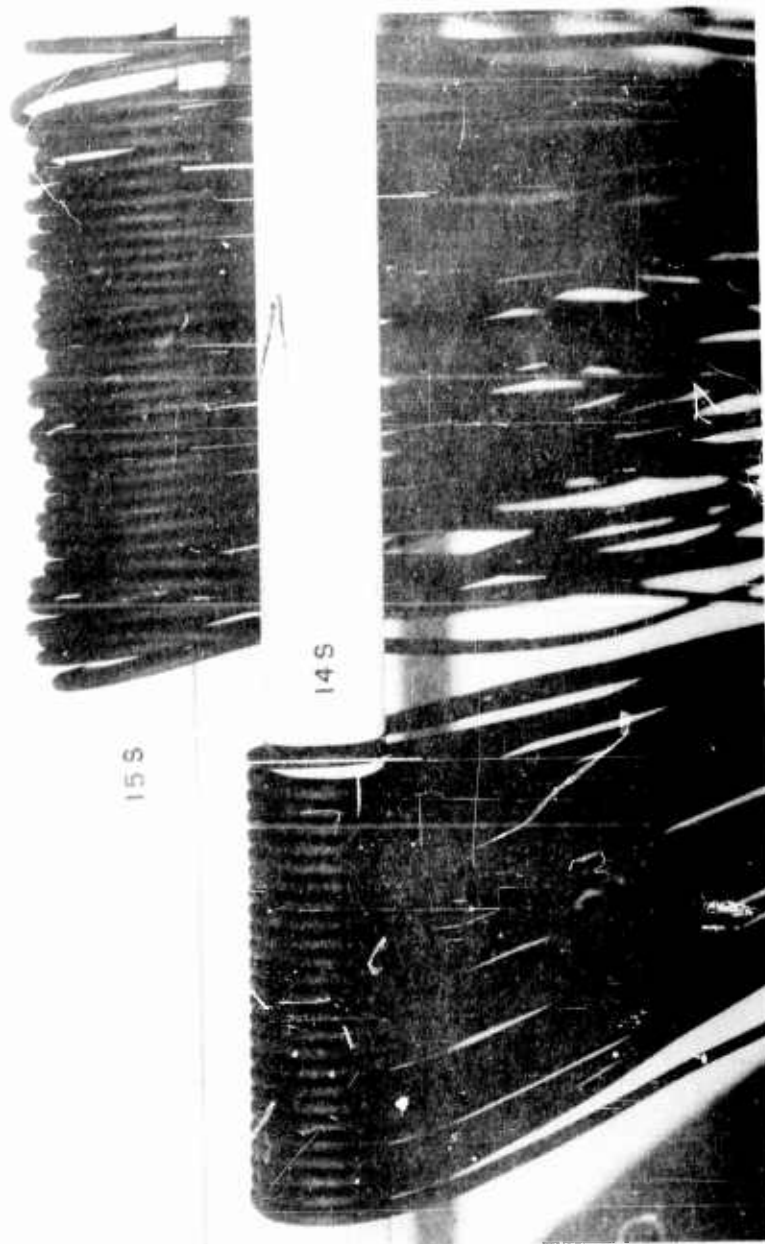


Figure 14. Comparison Of Strand Straightness (Top View)

For ethyl acetate colloided benite (Lot EP-14) vs. diethyl ether colloided benite using carbon black in lieu of charcoal (Lot EP-15).



Figure 15. Improved Straightness (Front View)

Results from immediate spreading of benite collioded with less volatile ethyl acetate solvent (Lot EP-14) vs. diethyl ether collioded benite containing carbon black (Lot EP-15).

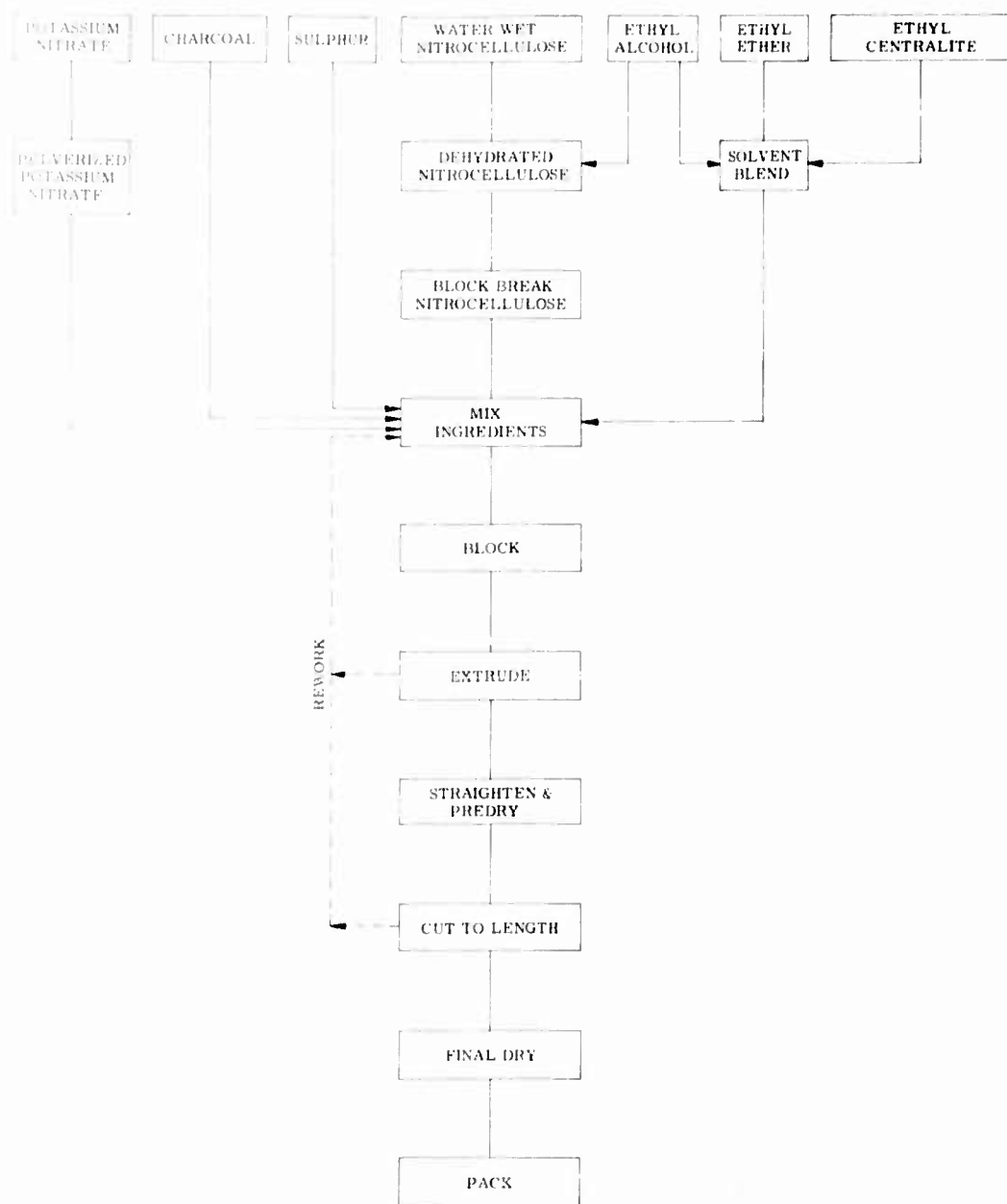


Figure 16. Flowsheet, R&D Process For Manufacture Of Benite Strand

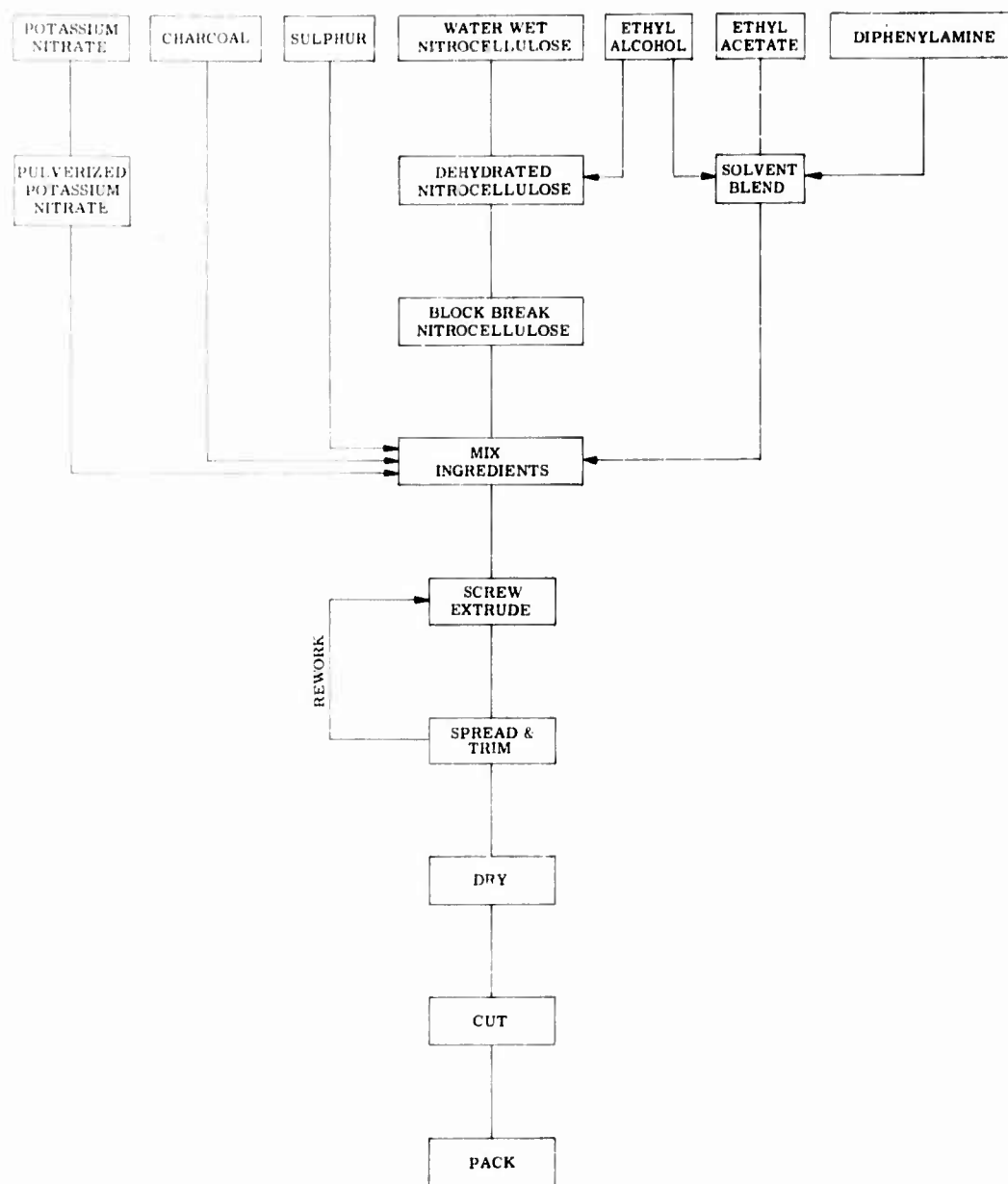


Figure 17. Flowsheet, Production Engineered Process With Continuous Extrusion And Spreading Using Modified Benite

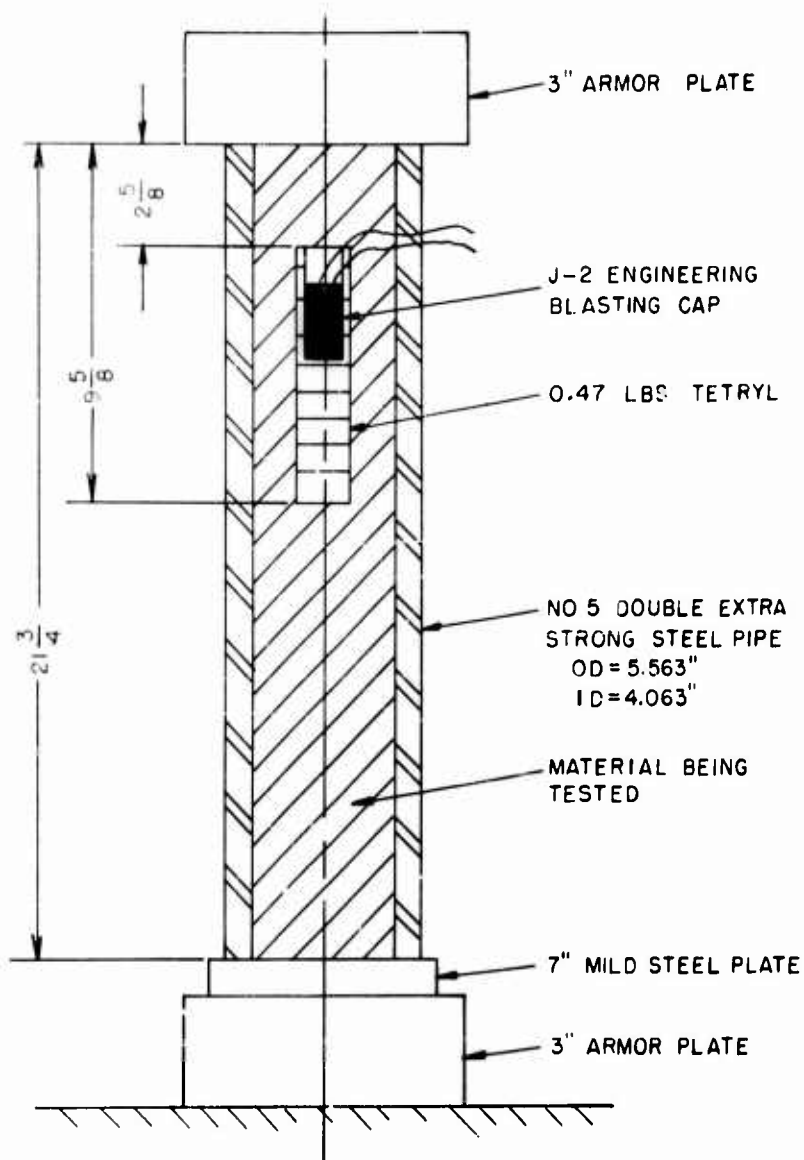


Figure 18. Detonation Test Set-Up

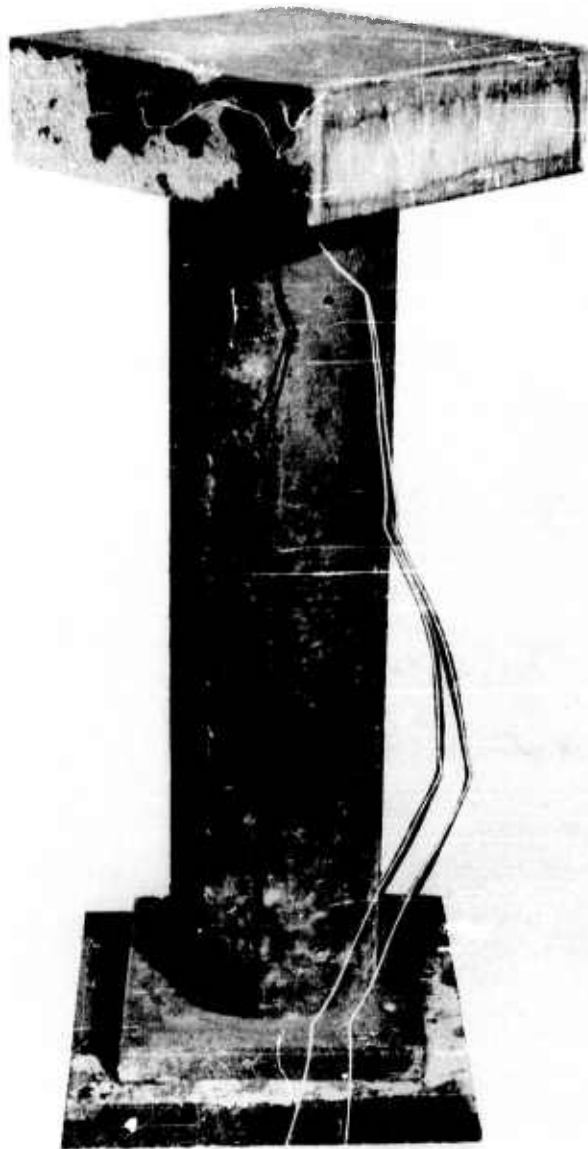


Figure 19. Set-Up For Test 1 Prior To Detonation



Figure 20. Fragments And Unburned Benite After Detonation
With Consolidated Benite, (Test 1.)



Figure 21. Fragments And Unburned Benite After Detonation
With Consolidated Benite, (Test 2.)



Figure 22. Fragments After Detonation With
Wet Sand, (Test 4.)



Figure 23. Fragments After Detonation With
Benite Crumbs, (Test 3.)



Figure 24. Fragments And Unburned Propellant After
Detonation Of Consolidated M-10 (Test 5.)

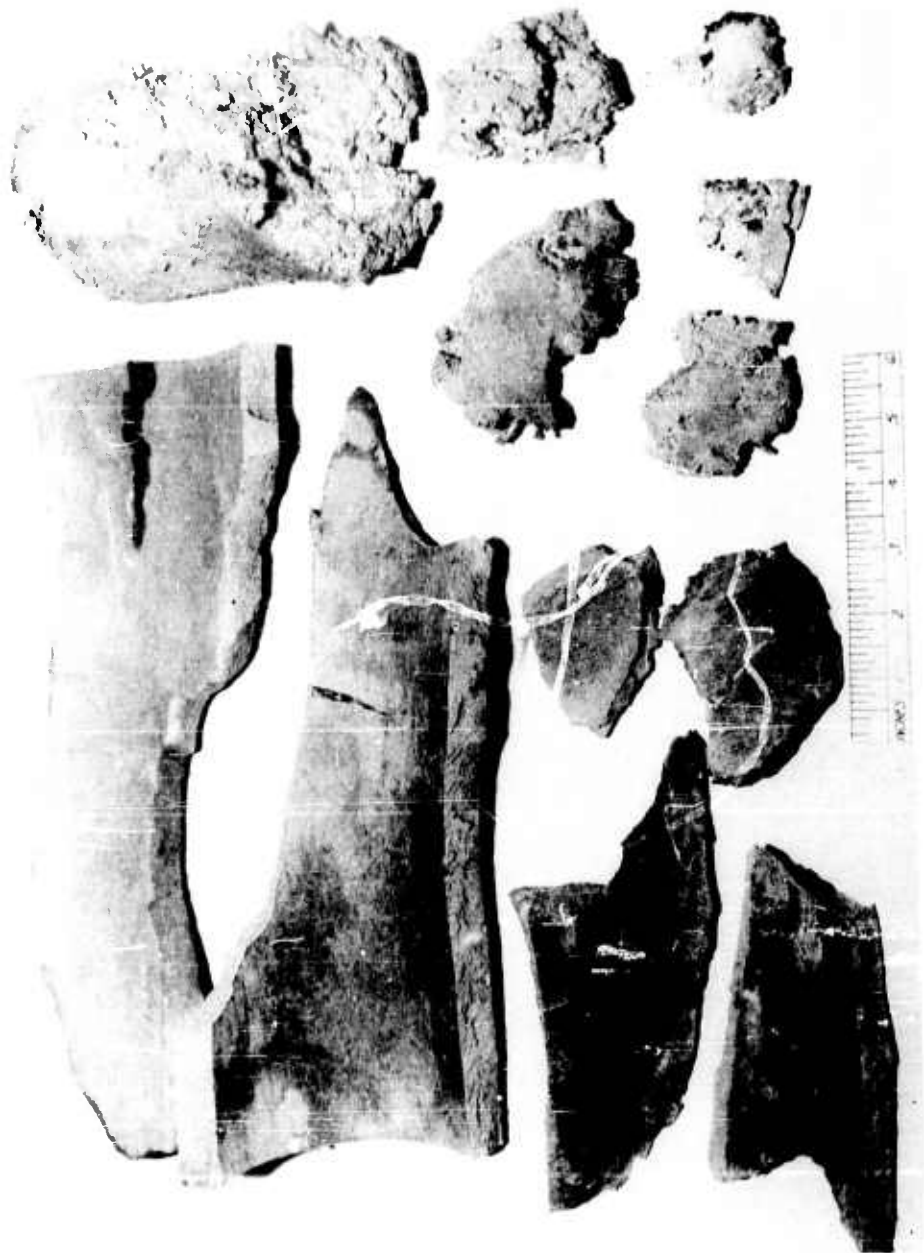


Figure 25. Fragments And Unburned Propellant After Detonation Of Consolidated M-10 (Test 6.)

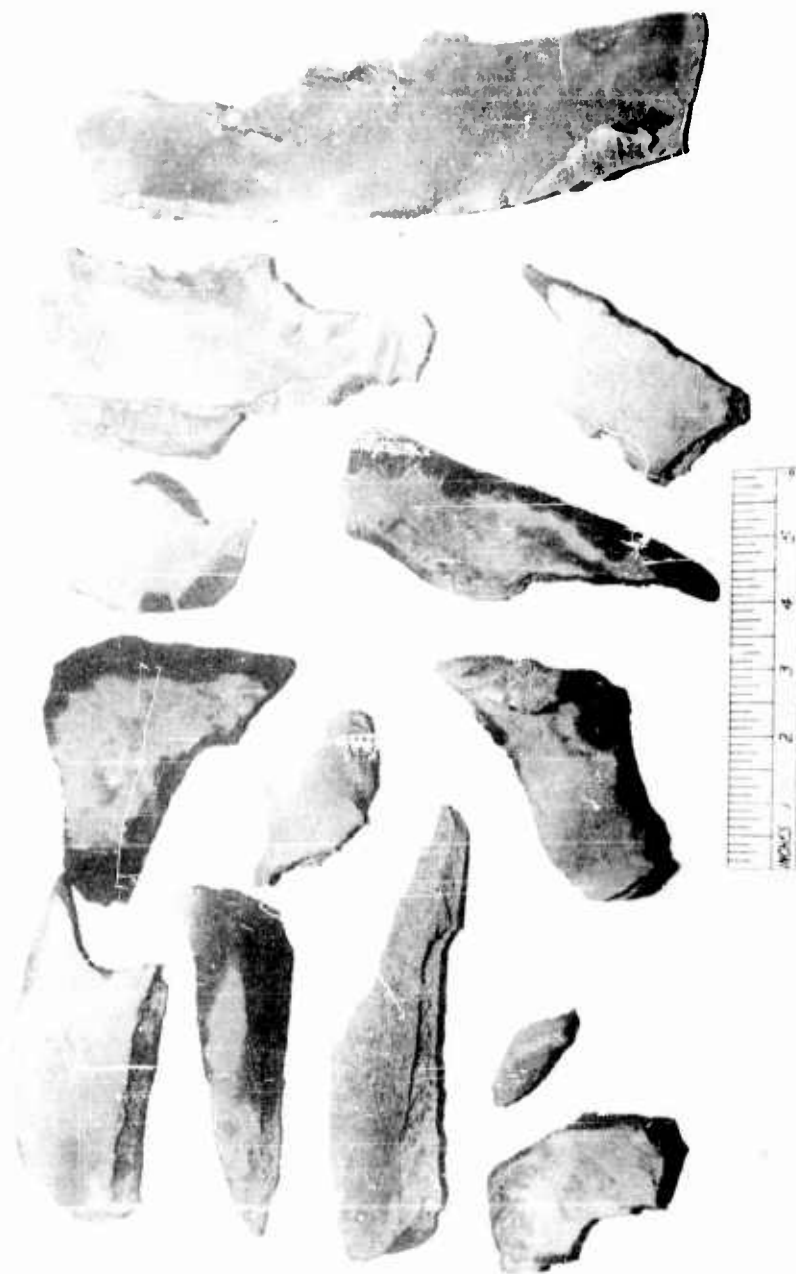


Figure 26. Fragments After Detonation Of M-10
Propellant Crumbs (Test 7.)

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ABSTRACT

AD

Accession No.

Picatinny Arsenal, Ammunition Group,
Dover, New Jersey

EVALUATION TESTS AND PROCESS STUDIES RELATING TO ESTABLISHMENT OF SUBSTITUTE FOR BLACK POWDER (IMPROVEMENTS IN MANUFACTURE OF BENITE STRANDS)

E. Huselton and S. Kaplowitz

Technical Report DB-TR: 5-60, April 1961, 34 pp.
Tables, Figures.
Unclassified Report

A realistic acceptance specification for benite strands was prepared. Conventional Ordnance manufacturing equipment may be used. Technical grade potassium nitrate is satisfactory. Increasing the drying temperature to 131°F. reduces drying time from 10 days to 12 hours. Reworking doubles the yield. Stability is improved by substituting diphenylamine for ethyl acetate and alcohol as solvents instead of diethyl ether and alcohol. Multiple-orifice plates for extrusion and dowels for drying of the strands are recommended for high capacity production equipment.

Benite colloid does not detonate under severe conditions of confinement; it is accordingly considered a Class 2 explosive hazard (fire hazard) for extrusion. The performance characteristics of benite are relatively insensitive to minor changes in formula and dimensions.

UNCLASSIFIED

1. Black powder....
Substitutes.
2. Benite--Extrusion.

I. Huselton, E.
S. Kaplowitz, S.
III. Project No.
252. 7507. 0402. 311.

UNITERMS

Black powder
Substitutes
Benite
Extrusion
Drying
Dowel
Hazards
Solvent
Diphenylamine
Ethyl acetate
Alcohol
Huselton, E.
Kaplowitz, S.
Project No.
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