FIELD EXPEDIENT
PREPARATION OF BLACK POWDERS

TABLE OF CONTENTS

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I. APPROACH

The following sequence was pursued in the black powder study.

A. Literature survey.
B. Evaluation of techniques for the preparation of black powder.
C. Performance evaluation of various preparations.
D. Preparation of a field manual outlining preparation procedure(s).

II. DISCUSSION

The fact that the black powder has been known to exist for some 2,000 years does not necessarily imply its current mode of preparation is particularly simple. Although a great number of investigators have independently studied and prepared black powders, all procedures that have resulted in satisfactory products are somewhat involved with respect to the incorporation steps. Simple mixing techniques of either dry or moist ingredients invariably result in inferior products. The purpose of the current program has been the establishment of a method, or methods, for the preparation of suitable black powders which may be accomplished by novice personnel using simple, readily obtainable implements.

A number of basic parameters are all important in the successful blending of black powder. Initially, the sulfur must be intimately incorporated into the cellular structure of the carbon which is usually accomplished by ball milling. Subsequently, the nitrate is mixed with the fuel mixture and requires pressure milling (while moist) in order to achieve proper intimacy. Failure to attain the proper degree of incorporation of the ingredients invariably results in inferior products.

The initial phase of the program dealt with possible means by which the commercial methods could be converted, at least in part, to field expedient procedures. These studies resulted in poor to mediocre products, and inconsistent results were the rule rather than the exception. The lack of any significant degree of success is attributed to substitution of inferior equipment in the operation and lack of experience required throughout the process.

Of necessity, a method meeting the design criteria of the current program must employ available utensils, and require a very limited degree of practice or instruction. This would dictate that principles of preparation be unaffected by a broad range of variation in the procedure, or be a procedure of ultimate simplicity. This goal was achieved through the investigation and development of a unique precipitation procedure. Essentially, this method consists of preparing a hot slurry of charcoal and sulfur suspended in concentrated aqueous potassium nitrate with the subsequent precipitation of black powder by rapid drowning in a common organic solvent such as isopropanol. From this point, filtration, granulation, and drying were facile rapid processes. The resulting products were generally consistent from lot to lot, and approached commercial powders in performance. The method and powder performance test results are considered adequate fulfillment of the requirements of the program.

For illustrative purposes, a flow chart comparing the precipitation method and a typical commercial procedure are shown in Figure 1. As indicated on the chart, commercial methods involve working with black powders containing 4% moisture, or less, with the coming mill step being considered the most hazardous of the operations. By contrast, the precipitation method does not involve working with a low moisture content material until the final product is obtained.

A comparison of the burning rates of the various lots of black powders prepared during the program is presented in Table 1. Lots 1 through 6 represent the powders prepared initially involving the most conventional preparative techniques, while Lots 7 through 14 are powders produced by the precipitation method.

III. EXPERIMENTAL

All approaches to the preparation of black powders emphasized simplicity. Initial studies were a modification of techniques which essentially followed the principles used in commercial prodecures. This included methods for incorporating sulfur with charcoal, subsequent incorporation of nitrate with the blended fuel and pressing operations. Attempts to use field expedient means to simulate commercial black powder processes resulted in products exhibiting poor to fair performance. Realization of the difficulties being encountered in this approach prompted investigation of other means of blending the ingredients, and led to precipitation techniques which offer a simple, novel solution to the problem. The ultimate method chosen involved solvent precipitation of black powder from a hot, aqueous, concentrated potassium nitrate solution containing suspended charcoal and sulfur. Common organic liquids such as methanol, 70% isopropanol (rubbing alcohol) and 80 proof ethanol (vodka) served this purpose successfully. The pyrotechnic properties of black powder prepared in this fashion approached those of commercial black powder.

Simplicity and safety are inherent in the method since no extensive premixing operations are required and the mixture can be handled in a moist form throughout the preparation. It is noteworthy that the method is independent of the prior particle form of the potassium nitrate since total solution of the salt is attained in the procedure. In addition, essentially reproducible products are obtained by this method as compared to the less effective methods initially investigated.

The overall experimental work and results are described in the subsequent portion of this section. The description of the work is divided into three sections: an initial portion concerning the more conventional hand mixing techniques, a second section pertaining to the precipitation method, and a final section reporting field application of powders.

COMMERCIAL PROCEDURE FLOW CHART

(A or B may be the initial stage)

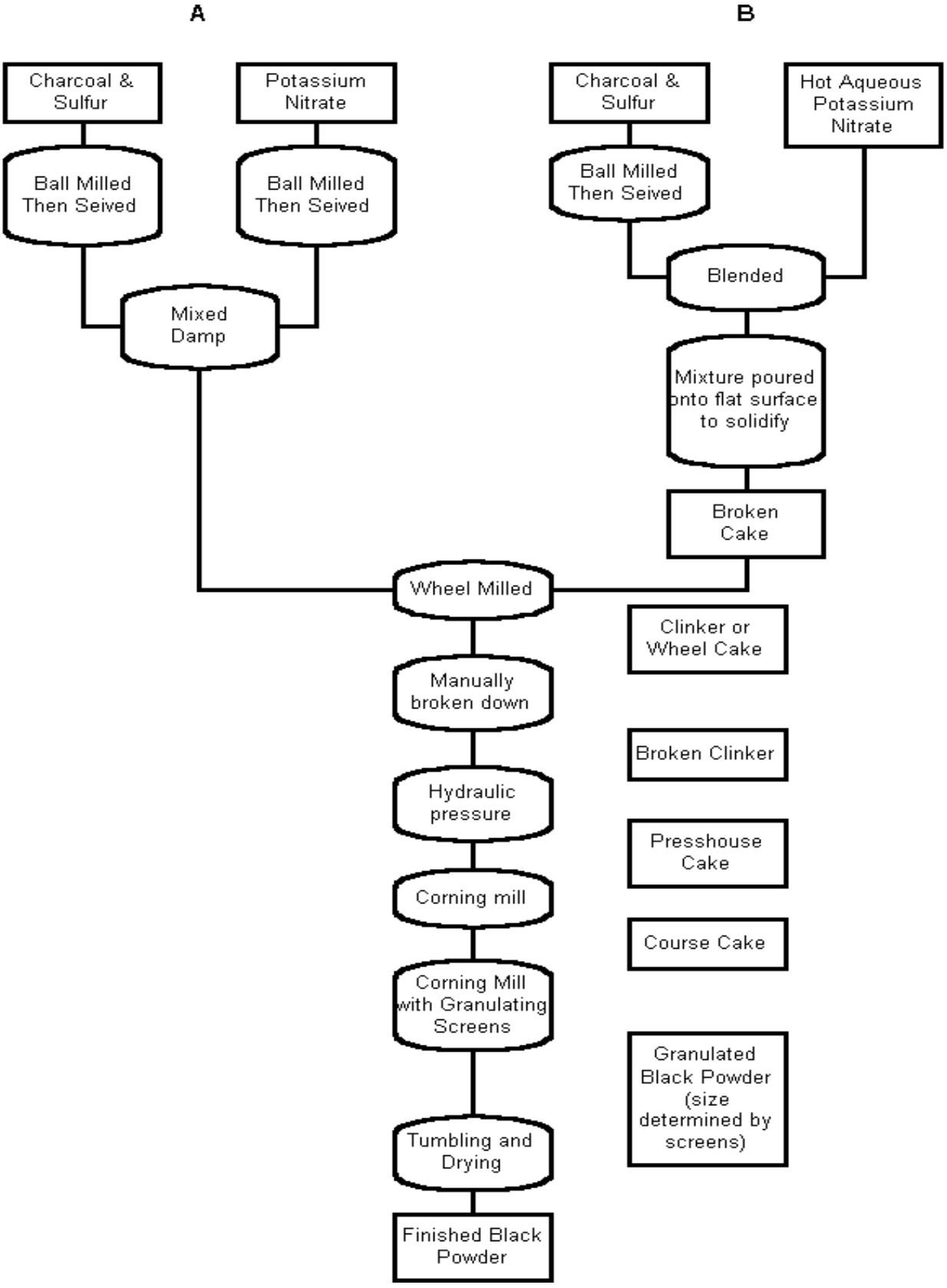


FIGURE 1

PRECIPITATION PROCEDURE FLOW CHART

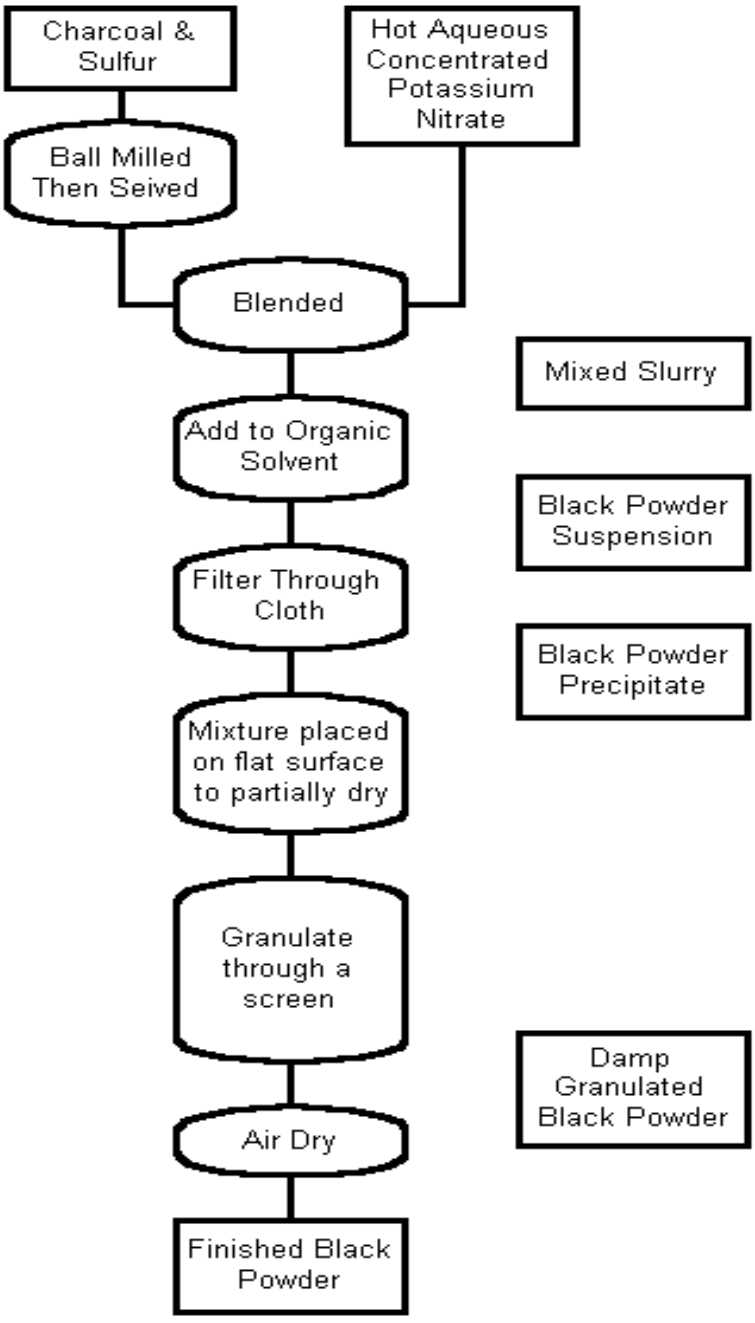


FIGURE 1 cont.

TABLE I

Burning Rates of the Various Black Powder Preparations (Note 1)

Lot No.	Granulation	Comments	Burning Rate cm/sec
1	10-16 mesh		12.6
2	10-16 mesh		7.7
3	Similar in size to grain of sand	Sodium nitrate base	0.96
4	Dry mixed powder; no granular structure	Sodium nitrate base	0.22
5	3 1/2-16 mesh		6.1
5	16-80 mesh		2.0
5	Through 80 mesh		0.95
6	10-16 mesh	Cake pressed manually	2.2
6	10-16 mesh	Cake pressed at 4 tons	7.7
6	10-16 mesh	Cake pressed at 5 tons	8.6
6	10-16 mesh	Cake pressed at 6 tons	10.7
7	10-16 mesh	100% IPA (Note 2) precipitated	16.2
8	10-16 mesh	70% IPA precipitated	18.4
9A	10-16 mesh	70% IPA precipitated	10.5
9A	Riced grains	70% IPA precipitated	Instantaneous

9B	10-16 mesh	NaCl sat'd. 70% IPA precipitated	10.5
9B	Riced grains	NaCl sat'd. 70% IPA precipitated	10.5
9C	10-16 mesh	KNO3 sat'd. 70% IPA precipitated	Instantaneous
9C	Riced grains	KNO3 sat'd. 70% IPA precipitated	Instantaneous
10	On 10 mesh	KN03 sat'd. 70% IPA precipitated	14.0
10	10-16 mesh	KNO3 sat'd. 70% IPA precipitated	8.4
10	16-25 mesh	KNO3 sat'd. 70% IPA precipitated	6.5
10	Through 25 mesh	KNO3 sat'd. 70% IPA precipitated	1.4
10	Rewet riced grains	KN03 sat'd. 70% IPA precipitated	21.0
11	10-16 mesh	70% IPA precipitated	15.0
12A	10-16 mesh	80 proof vodka precipitated	12.8
12A	16-25 mesh	80 proof vodka precipitated	14.0
12A	Rewet riced grains	80 proof vodka precipitated	24.8
12B	10-16 mesh	KN03 sat'd. 80 proof vodka precipitated	13.2

12B	16-25 mesh	KNO3 sat'd. 80 proof vodka precipitated	13.1
12B	Rewet riced grains	KNO3 sat'd. 80 proof vodka precipitated	12.1
13A	Riced grains	70% IPA precipitated	8.3
13B	Riced grains	Absolute methanol precipitated	11.1
14	Riced grains	70% IPA precipitated	15.0
14	Rewet riced grains	70% IPA precipitated	14.0

NOTE 1: All preparations were KNO3 based unless designated otherwise

NOTE 2: IPA - isopropyl alcohol

A. Black Powder Preparations -- Initial Phase

The preliminary black powder formulations were made using both potassium and sodium nitrates and employed simple laboratory procedures. In the initial three lots the weighed or volume measured ingredients (column 3, Table II) were moistened with water, thoroughly blended in a mortar and pressed between two metal plates using moderate hydraulic pressure. The resulting cake, which was approximately one-sixteenth of an inch thick, was oven-dried at 600C. and granulated by gentle crushing on a hard, flat surface with a length of pipe. A fourth lot of black powder containing sodium nitrate was prepared by a dry blending method. The volume of ingredients given in column 3, Table II, were placed in a three pound coffee can, fitted with a polyethylene cover, and the can rotated in all directions until an apparently uniform mixture was obtained. The four powders were evaluated for burning rate and impact sensitivity; results are given in Table II. The burning rates of the four compositions varied considerably; the sodium nitrate composition was the slowest burning, which is in accord with studies made by previous workers.

The impact sensitivities were determined using a Bureau of Mines two-kilogram impact apparatus which was standardized with RDX (32 cml 10% fire level). The burning rates were determined in paper tubes 5/16-inch in diameter and 8 inches in length. The powders were poured into weighed tubes, reweighed, and ignited by means of a black powder fuse. The bum time was measured by means of a manual stopwatch.

At this point, techniques were altered to emphasize the use of common household utensils in the preparation of black powder formulations.

TABLE II

Properties of Various Black Powder Formulations

Powder	Ingredients	Percentages	Impact Sensitivity	Burn Rate cm/sec	Particle Size for burn Rate
Lot 1	KNO3 C S	75 by wt. 15 by wt. 10 by wt.	No fire at 103 cm	12.6	Through #10 sieve, but not through a #16
Lot 2	KNO3 C S	58.2 by vol. 32.4 by vol. 9.4 by vol.	No fire at 103 cm	7.7	Same as Lot 1
Lot 3	NaNO3 C S	72 by wt. 17 by wt. 11 by wt.	No fire at 103 cm	0.96	Similar in size to grains of sand
Lot 4	NaNO3 C S	42.4 by vol. 46.9 by vol. 10.9 by vol.	10% fire level at 23 cm No fire level at 22 cm	0.22	Dry mixed powder; no granular structure

In Lot Number 5, fifteen grams of finely ground carbon were mixed intimately with 10 grams of ground sulfur flour and placed in a heavy iron skillet on low heat. The mixture was heated and stirred until the constituents were uniform in appearance. The carbon/sulfur mixture was removed from the skillet and stirred until cool. This mixture was added to a previously heated solution of 75 grams of potassium nitrate in 40 milliliters of water. The skillet and contents were removed from the heat and stirred until a uniformly wet paste was obtained. The slightly moist powder was poured onto a hard, flat surface and pressed manually into a thin cake with a rolling pin. The powder was dried at 600C. The dried lumps were broken into uniform granules and sieved through cheesecloth onto a nylon scarf. The portion on the scarf was shaken until all the fine powder was sifted out. The three portions were evaluated or the burning rate as described in the previous section with the following results:

	cm/sec.
Powdered portion through scarf (<80 mesh)	0.95
Portion remaining on scarf (80 -- 16 mesh)	2.0
Portion retained on cheesecloth (16 - 3 1/2 mesh)	6.1

Separation of the various particle sizes aided in demonstrating effectiveness of particle size on ultimate performance in field applications.

Lot Number 6 was a 500-gram quantity of powder prepared in a manner similar to that described for evaluation of pressure effects on the moist powder. Burning rate tests similar to those previously described were used as the criteria for evaluation. The moist powder was divided into four approximately equal portions and treated in the following manner: one portion was pressed manually on a flat surface with a rolling pin as described above, and the remaining three portions of the wet powder were pressed between two flat metal plates using four, five, and six tons of pressure. The actual pressure area on the powders corresponded to a circle about three inches in diameter. The four samples of powder were dried, broken and sieved to produce particles passing through a number 10 sieve, but retained on a number 16 sieve. The results were as follows:

	Burn Rate cm/sec.
Powder pressed manually	2.2
Powder pressed at 4 tons pressure	7.7
Powder pressed at 5 tons pressure	8.6
Powder pressed at 6 tons pressure	10.7

Pressure tends to incorporate the ingredients and, as indicated above, increases the burning rate considerably.

B. Black Powder Preparation by the Precipitation Method

Difficulty in obtaining an intimate mixture of components in the preparation of various black powders using modified commercial methods led to the investigation of techniques incorporating a salting-out procedure employing organic solvents. This procedure, in conjunction with the use of several common variety store items, resulted in the production of a black powder considered to be nearly equivalent to commercial black powder.

Lot No. 7

Potassium nitrate (tech. grade)	150 g.
Charcoal	30 g.
Sulfur	20 g.

The components were intimately mixed and added to 100 mi. of hot water in an iron skillet. Heating was continued and water added in small increments until the potassium nitrate was dissolved. The hot mixture was poured into approximately 300 mi. of isopropanol, allowed to cool, and the solids separated by filtration through a nylon stocking. The moist solid was rolled into a flat cake, approximately 1/4" thick, allowed to dry overnight, and crushed with a rolling pin. The burning rate of the 10-16 mesh product was 16.2 cm per second.

Lot No. 8

Potassium nitrate (tech. grade)	75 g.
Charcoal	15 g.
Sulfur	10 g.
Water	75 ml
Isopropanol, 70%	200 ml

The procedure employed was essentially identical to that used for Lot No. 7 with the exception that the volume of water was measured and 70% isopropanol was employed. The burning rate of the 10-16 mesh powder was 18.4 cm per second.

Lot No. 9

Potassium nitrate	340 g.
Charcoal	68 g.
Sulfur	45 g.
Water	325 ml
Isopropanol, 70%	500 ml
Isopropanol, 70% (saturated with NaCl)	250 ml
Isopropanol, 70% (saturated with KNO3)	250 ml

Preparatory procedure, as previously described, was followed with the exception that the lot was divided into three portions prior to precipitation:

9A - 1/2 of lot precipitated in 500 mi. 70% isopropanol
9B - 1/4 of lot precipitated in 250 mi. 70% isopropanol saturated with NaCl
9C - 5/4 Oflot precipitated in 250 mi. 70% isopropanol saturated with KNO3

Each sub-lot was ultimately divided into quarters to accomplish air and oven drying and compare granulation methods as outlined in Table III.

TABLE III

Burn Rate (cm/sec)

Sub-Batch	Granulation (mesh)	Air Dried	Oven Dried (2 hrs. at 900C)
9A	10-16	10.5	11.0
9B	10-16	10.5	8.0
9C	10-16	Instantaneous	11.0
9A	Riced grains*	Instantaneous	11.1
9B	Riced grains*	10.5	8. 1
9C	Riced grains*	Instantaneous	11.0

*Granulation accomplished using a household potato ricer (Figure 2). A typical particle range of such a powder is 75%, 10 -16 mesh and 25%, 16-25 mesh admired with a small quantity of fines.

(Potato Ricer)
FIGURE 2

Lot No. 10	
Potassium nitrate	1020 g.
Charcoal	204 g.
Sulfur	136 g.
Water	900 ml
Isopropanol (sat. w. KNO3)	3000 ml

Composition and procedure were identical to that employed in the previous lot. The primary objective was the determination of burning rates of the powder in various size ranges.

TABLE IV

Granulation (mesh)	Burning Rate (cm/sec)
10	14.0
10 - 16	8.4
16 - 25	6.5
25l	1.4
Riced, Rewet*	21.0

*This was accomplished by moistening the original powder with 70% isopropanol, granulating with a ricer and air drying.

Lot No. 11

Potassium nitrate	340 g.
Charcoal	68 g.
Sulfur	44 g.
Water	300 ml
Isopropanol	1000 ml

Untreated 70% isopropanol was employed since the salt-saturated material used in Lot No. 9 and Lot No. 10 did not produce a powder with any significant superiority. In this, and subsequent lots, cotton cloth was used as a filter material rather than nylon. The precipitate was divided into two portions, half of which was air dried and half oven dried. Results of burning rate tests were as follows:

Air dried	10-16 mesh	15 cm/sec.
Oven dried (2 hrs. @ 90 deg. C.)	10-16 mesh	13.1 cm/sec.

Lot No. 12

Potassium nitrate	340 g.
Charcoal	68 g.
Sulfur	44 g.
Water	300 ml
Vodka (80 proof)	500 ml
Vodka (80 proof) Sat. w/KNO3	500 ml

Universal availability dictated that vodka be given consideration as a precipitation medium in the field preparation of black powder. Initial preparation was as previously outlined; the lot divided into two equal parts and precipitated in vodka with (12A) and without nitrate saturation (12B). One half of each sub-lot was rolled, air dried, crushed and screened into definite granulation ranges; and the second half forced through a potato ricer and allowed to air dry. Burning rates of the resulting powders are given in Table V.

TABLE V

	Burning Rates (cm/sec)		
Sub-Batch	Air Dried	& Crushed	Riced (rewet*)
Granulation (mesh)	10-16	16-25	
12A	12.8	14.0	24.8
12B	13.2	13.1	12.1

*See footnote on Table IV.

A question of nitrate loss through solubility in the precipitation method prompted the retention of solvent after removal of solids in a lot similar in composition to Lot No. 9. Evaporation of the isopropanol yielded 61.7 g. of KNO3 representing a weight loss in the powder of 18.1%. Compensation for this loss results in a 78/13.2/8.8 composition.

Lot No. 13	
Potassium nitrate	880 g.
Charcoal	150 g.
Sulfur	100 g.
Water	600 ml

The composition adjusted to account for nitrate solubility loss as determined above, was mixed as previously described and divided into two equal parts. 13A was precipitated in 2400 ml. of 70% isopropanol, and 13B was precipitated in 2400 mi. of absolute methanol, and both sub-lots were put through a potato ricer prior to air drying. The burning rates of 13A and 13B were 8.3 and 11.1 cm/sec respectively.

Lot No. 14	Parts by Volume	
Potasssium nitrate	3 cups	(6)
Charcoal	2 cups	(4)
Sulfur	0.5 cups	(1)
Water	3 cups	(6)
Rubbing alcohol (70% isopropanol)	5 pints	(10)

As indicated, volume measurements were made of the ingredients. Blending was performed in the normal fashion with the exception that the fuels were added to previously dampened nitrate to eliminate working with the oxidizer-fuel system in the dry state. The filtration was accomplished using a linen towel. After air drying to a slightly moist consistency, the material was granulated using a potato ricer and sun dried for one hour. The bum rate was determined to be 15 cm/sec. Re-wetting and re-ricing did not appreciably alter this particular product (14 cm/sec.). Weight-volume relationships for the various ingredients are given in Table VI.

TABLE VI

Weight-Volume Relationship of Ingredient of Black Powder

Ingredient	Approximate Weight (gms) of one cup (8 fluid ounces)
Potassium nitrate (N.F. granulated)	270
Charcoal (wood, powdered)	75
Sulfur (U.S.P., precipitated powder)	200

C. Application Tests

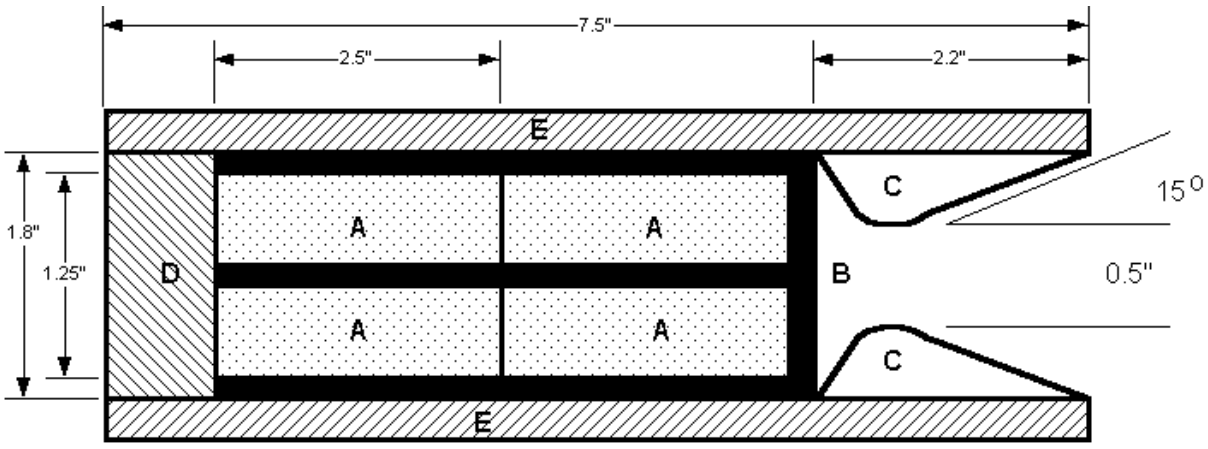
One pound of black powder, as produced for Lot No. 5, contained in a 12" x 2-1/2" plastic tube was placed in a 36" x 2-3/4" hole, fused and covered with soil. On ignition, a hole approximately 20 inches in diameter was produced.

Three pounds of black powder, as produced for Lot No. 13A was contained in a coffee can and buried 42" deep. Ignition produced a crater six feet in diameter.

While actual force or impulse measurements were not taken, powders prepared by the precipitation method are considered acceptable for blasting purposes. Extensive testing is considered essential to completely delineate their capabilities under all environmental conditions.

Several propellant grains were prepared from powders similar to Lot No. 11 and Lot No. 13A, both loosely packed and pressed to several tons. In each case the powder exploded when ignited in a nozzled chamber indicating the need for a modifier to control the burning rate.

Three pounds of powder were prepared in a manner similar to that described in 13A except that 270 grams of soluble starch was intimately mixed with the wet Powder prior to ricing. Subsequent to air drying, the composition was re-wet with 10% water, and two 1-'/4"-cylinders having 1/8" center holes were pressed at five tons. The grains weighed 50 grams each and after air dr3iing were incorporated into rocket configuration shown in Figure 3. After additon of fuse and ignition material, the unit, which weighed 580 grams, functioned as anticipated and attained an altitude of 400 feet.



A. Propellant Grains (50 g each)
B. Ignition Material
C. Plaster of Paris Nozzle
D. Epoxy Plug
E. Fiber Board Tube

FIGURE 3

While the allotted time did not allow for extensive studies involving areas other than nitrate black powders, a brief investigation was made of chlorate as an oxidizer and sugar as a fuel. Chlorate powders are inherently sensitive and, therefore, pose a problem especially where inexperienced personnel are concerned.

For instance, one small batch of 75% KClO3, 12.5% S, 12.5% C, prepared by wet blending in isopropanol exhibited an impact sensitivity of 15 cm (10% fire level). RDX gave a value of 32 cm. Other compositions prepared include 75% KCl03, 25% sugar, 20 cm; 60% KCl03, 40% sugar, 46 cm; 60% KClO3, 20% C, 20% S, 7 cm; and 60% KClO3, 35% sugar, 50% C, 10 cm. Also, these mixtures were prepared using solvent blending. In addition, a promising "white powder," namely potassium nitrate-sugar, was briefly investigated. Such a composition was prepared and evaluated in the following manner: Sixty-five grams of potassium nitrate and 35 grams of granulated cane sugar were placed in an iron skillet and sufficient water added in increments with heating and stirring until solution was affected. Shortly after the mixture began to boil, a precipitate formed resulting in a white slurry. Stirring was continued until the mass changed with additional heating from a crystalline slurry to a smooth homogeneous mass. At this point, the material was poured onto a flat surface and worked by rubbing with the wooden stirring rod into small lumps. If the mixture is allowed to cool without stirring, a single mass is formed which is very difficult to granulate. The burning rate of the 10-16 mesh product was 9.3 cm/sec.

This approach to indigenous pyrotechnics should warrant further study from a standpoint of fuel selection, oxidizer selection and fabrication techniques. Previous investigations have shown KN03-sugar compositions to be satisfactory propellants.

IV. CONCLUSIONS

1. Black powders prepared in the field by simple mechanical mixing are of inferior quality with respect to burning rate. Attempts to use simple utensils for preparing black powder in accordance with commercial processes results in, at best, a mediocre product.

2. A simple, facile precipitation process involving the salting out of nitrate oxidizer onto the charcoal-sulfur fuel using common organic solvents affords a satisfactory black powder. This product exhibits a burning rate approaching that of commercial powder, produces favorable cratering effects and, with modification, can be successfully used as a rocket propellant.

3. Attempts to adjust the precipitation method to account for nitrate solubility in the solvents did not improve the product and, in certain cases, yielded inferior materials. In part, this might be attributed to an oxidizer-rich surface of the black powder being formed as a result of the adjustments.

Appendix A

FIELD EXPEDIENT
PREPARATION OF BLACK POWDERS

Preparation of Black Powder:

Potassium nitrate black powder may be prepared in a simple, safe manner. The formulation described below will result in approximately 1-1/2 pounds of black powder, which may be used as blasting or rifle powder.

Material Required:

- Heat source such as a kitchen stove (or an open fire, if it is the only available source)
- Two-gallon bucket (metal or plastic)
- Cooking pan or skillet; 4 quart capacity
- Flat window screen, at least i-foot square
- Large wooden spoon or stick
- Plain weave cloth sheet (at least 2 feet square)
- Measuring cup (8 ounces)
- Potassium nitrate (granulated)
- Powdered wood charcoal
- Powdered sulfur
- Rubbing alcohol (70% isopropyl alcohol) or wood (methyl) alcohol
- Water

Procedure:

1. Measure by volume, 3 cups of granulated potassium nitrate, 2 cups of powdered charcoal, and 1/2 cup of powdered sulfur into the 4-quart pan (or skillet), and moisten with 1 cup of water. Using a wooden stick or spoon, throroughiy mix the ingredients.

2. Add 2 additional cups of water to the mixture and place the pan on the heating source. Allow the liquid to come to a simmer with sufficient stirring to obtain an evenly mixed blend. With vigorous stirring, rapidly pour this mixture into five pints of alcohol contained in the two-gallon bucket.

3. After the alcohol mixture has been allowed to stand about 5 minutes, collect the black powder by straining the entire contents through the cloth. Remove as much liquid as possible by wrapping the cloth around the powder and squeezing the resulting bag.

4. Spread the wet powder in a thin layer (1/2 inch thick) on a flat surface and allow to dry to a slightly moist solid. Place the screen over the bucket which has been cleaned and dried from the operation described in Step 2. Place a workable amount of the moist powder on the screen and granulate by hand, rubbing the solid through the screen. If the particles collected in the bucket appear to stick together and change in shape, recombine the entire batch, redry, and repeat the granulation operation.

5. Dry the granulated black powder by spreading on a flat surface in about a '/2-inch layer. Sun drying is preferred for this step.