FINAL PROJECT REPORT

Contract DAI-23-072-501-ORD-(P)-43

A STUDY OF MODERNIZED TECHNIQUES FOR THE MANUFACTURE OF BLACK POWDER

BY

PROPELLEX CHEMICAL DIVISION

Chromalloy Corporation

Edwardsville Illinois

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Incl 1

ABSTRACT

New approaches to the production of black powder, using both solvents and non-soluting vehicles, followed by hydraulic compacting or tabletizing are investigated. Measurements of dP/dt at different pressures in a closed bomb are used to compare experimental samples and commercial products. The influence of a regular geometry on ballistic performance is considered and a process for yielding a product similar to commercial black powder is developed.

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1. INTRODUCTION

Black powder is essentially a physical mixture of wood charcoal, sulfur and potassium nitrate. Its manufacture has always been looked upon as simple in principle and consisted in the past of a series of milling, mulling and compacting operations to yield a solid irregular grain of intimate mixture of the three constituents. Black powder at a typical powder plant, for instance, consisted of six main operations: pulverizing, incorporating, pressing, corning, glazing, and screening. (See Commercial Black Powder Flow Sheet, Figure 1).

Lumps of willow charcoal and bulk crystalline sulfur are ball-milled together as received in the proper proportions for six to eight hours in a steel mill with steel bolt cuttings at 20 RPM and stored until needed. The potassium nitrate as received is crushed, screened, beaten to a flour fineness and stored for further use. The potassium nitrate beater is a cylindrical steel drum in which several large paddles revolve at high speed to reduce the potassium nitrate to dust.

The pulverized charcoal-sulfur mixture and the beaten potassium nitrate are brought together in the Wheel Mills in the proper proportion. Here the three components are milled together under heavy stones for a period of approximately three hours. During this period the operators, largely by experience, control the moisture to a final moisture content of about 1.75%. An effort is made to keep the moisture at 2.25% for the first two hours after which the moisture content is allowed to drop to 1.75%. This is judged

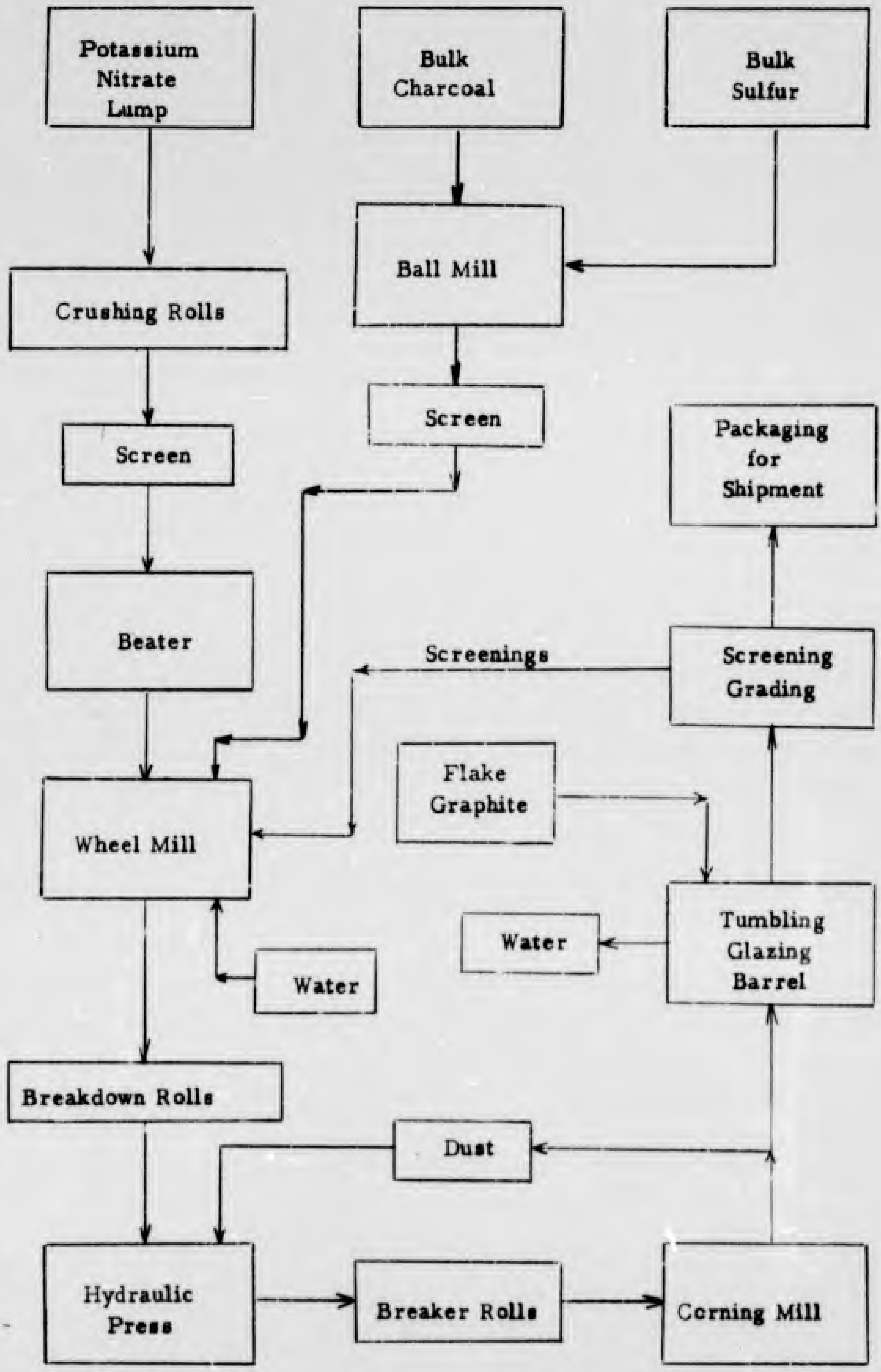


FIGURE 1

COMMERCIAL BLACK POWDER FLOW SHEET

entirely by the caking and balling of the mix being milled. It has been found, however, that above this moisture content the resulting powder will not grain well.

The mill cake from the above operation is broken up between zinc rollers to facilitate later handling. These lumps after various periods of storage are fed through a hydraulic press. It has been found that moisture contents higher than the previously specified 1.75% gave powders too high in density. Ordinarily, pressures of 2000 psi have good cakes of proper density (1.67-1.71 at moisture concentrations between 1.50 and 1.75%. The general range of pressures, however, is 1900 to 3000 psi depending on the range of moisture content, density of charcoal, and other factors, intuitively corrected for by the experienced operator. At the conclusion of the pressing operation, the pressed cakes are broken up into chips and conveyed to the Corning Mill.

Here the chips are broken into smaller pieces by "crack"rolls", brass rolls with diamond-pointed teeth. The resulting cracked chips are passed over a screen to a second set of rolls, and over a second screen. The unscreened powder passes through a third set of rolls and over a third screen. The powder remaining on the screen is fed back to the last set of rolls. The screening operation itself may be so arranged that one or two grades of powder plus the residue of the screening called "dust" may be produced. It should be noted, by the way, that the cracking and rolling operation produces slight increases in density during manufacture. The screened powder from this operation is now ready for glazing.

Glazing is carried on in maple wood barrels turning axially on steel shafts. Initially, the barrels are charged with powder from the Corning Mill and run for about six to as much as thirteen hours at 18 RPM. This step is essentially a drying operation, depending, as does the later graphiting operation, on the frictional heat developed between powder grains and the barrel wall. At a time chosen by the operator, flake graphite is added and rotation is continued at 18 RPM. The graphite is added when the operator detects the lightening of the color of the powder and the odor of sulfur. The drying operation continues after the addition of the graphite, accompanied by the visible signs of steaming as water is lost.

When the operator judges the powder is dry enough, after about six hours from the addition of the graphite, the speed of the barrels is reduced to 5 RPM. The powder, during the high speed rotation, has reached a temperature of 180°F. and the lower speed rotation is largely a cooling-off operation. At the end of five to six hours the powder has cooled sufficiently and may be removed from the barrel. The resulting black powder is commercial "bright grain".

While the various operations of the black powder process are conducted largely by operators who rely on intuition derived from long experience, laboratory controls are applied throughout the process. Purity and physical property controls are, of course, maintained on the incoming raw materials. Moisture control is maintained during the wheel-milling operation to keep the moisture content below the maximum workable 3.75% while the final content of 1.75% or less is held by laboratory control. However, the moisture content in these steps is largely the responsibility of the wheel mill operators and the laboratory functions more to maintain batch to batch uniformity than to dictate the duration and operation of the milling step.

Specific gravity is determined from samples taken from the Corning Mill operation as a guide to the operation of the hydraulic press. Densities of 1.65 to 1.69 at this point will yield, it has been found, final densities within specification (1.72-1.77) after glazing and tumbling. Nevertheless, density control is still largely a matter of personal experience of the operator and the laboratory results serve more as a guide than as an absolute control.

Specific gravity and moisture controls are run on daily composite samples from the Glaze Barrel. Where specifications are not met, the grain is blended with incoming grain from the Corning Mill and returned to the glazing-tumbling operation. Final chemical analyses are made to assist the Superintendent of Operations in holding or altering the composition going into the powder. Where lots of powder fall short of specifications, they are commonly held and blended with later lots at some point in the production process to yield final acceptable lots.

This resume of the black powder process is necessarily brief and leaves many details unstated. It does, however, serve to point out several outstanding disadvantages of the classical process which, it was hoped, the present study would eliminate. Probably, the most outstanding of these disadvantages is the reliance on the intuition of the individual operators, particularly with respect to moisture and density control. At the two most critical steps, wheel milling, and glazing-tumbling, and to a lesser extent at the Hydraulic Press, the success of the run depends largely upon the experience and "process-feel" of the operator.

It was assumed at the start of the present study that physical operations of an improved process should be similar generally to the existing black powder process. However, it was theorized that operations involving particle reduction and incorporation might be better accomplished with newer equipment available on the market.

The present study, as outlined in Propellex Proposal No. 157, was then directed to the investigation of particle reduction, incorporation, and compacting innovations to produce a ballistically reproducible black powder of burning properties similar to commercial bright powder. It was expected that control measures would be developed during the course of the program to allow closer reproduction of properties from batch to batch.

II. EXPERIMENTAL

The general approach adopted to the experimental production of black powder under this contract was

1. Reduction of particle size of charcoal and potassium nitrate by some grinding operation.
2. Incorporation of the three constituents, using some volatile vehicle. This might or might not be capable of dissolving one of the components.
3. Drying under vacuum and compacting.
4. Glazing.

The glazing operation was not studied during the course of this contract. All samples were graphited in the manner developed by the laboratory at Picatinny Arsenal. 1.0% by weight of powdered graphite was added with experimental black powder to the pan of a Ro-Tap Sieve Shaker, and vibrated for six hours.

The composition and relative particle size of black powder was investigated. A microscopic study of the components of commercial black powder was made. Samples of commercial bright powder were leached with both water and carbon disulphide to yield a residue that was potassium nitrate-free and a residue that was sulfur-free. The charcoal fraction could be derived from commercial powder by treatment with both these solvents. It was found that both the potassium nitrate and the charcoal were of very fine particle size. The charcoal was generally in the 10 micron diameter range while the potassium nitrate was slightly larger in the 15-20 micron range. Interestingly, it was found that the carbon particles were all covered with a thin shell of transparent sulfur, suggesting that the application of pressure and friction during the ball milling process had resulted in flowing all of the sulfur about the charcoal particles.

From the above evidence of small particle size and peculiar state of the sulfur in the commercial powder, it was decided that work should concentrate on the use of vehicles which partially or totally dissolve sulfur. Toluene and carbon disulfide were chosen for this investigation. Early work on the possible use of a two component system such as carbon-disulfide-water showed that this approach was cumbersome and offered no particular advantage in the incorporation of components. Generally, mixes produced, using a single vehicle, were as homogeneous. The two solvent system was accordingly abandoned.

Since particle size reduction appeared important to the production of good black powder, it was decided that some study of the screening characteristics of the various components was necessary before continuing to an evaluation of grinding methods. The screening characteristics of components, particularly the hygroscopic potassium nitrate, under various conditions of dryness, was of particular interest.

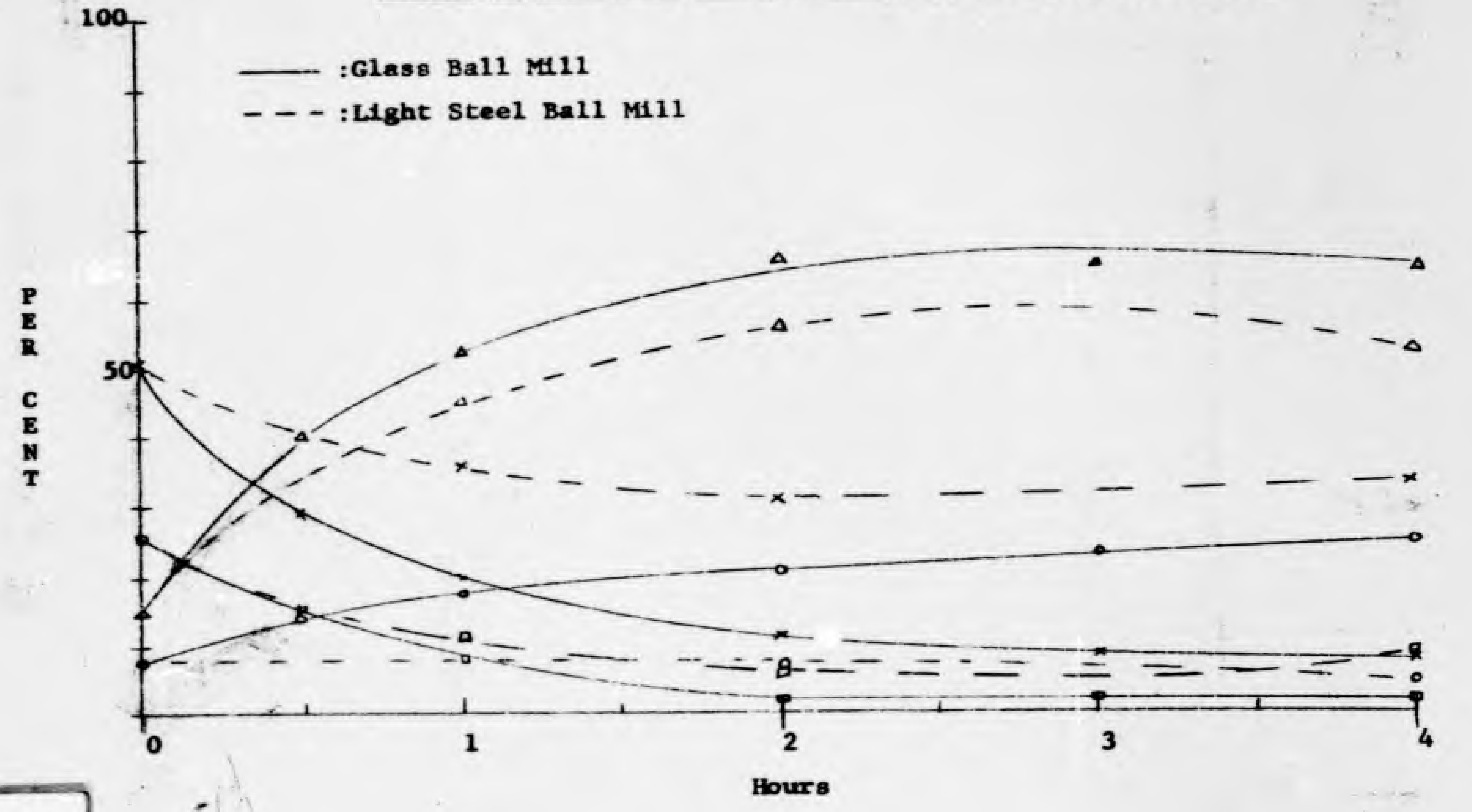
It was found that no reliable size analysis of powdered sulfur could be obtained since the apparent size of sulfur particles appeared to grow during the screening operation by agglomeration. (See Figure 2). No such agglomeration was encountered with charcoal or potassium nitrate. Drying of the sulfur also produced an apparent increase in particle size with the time of drying and temperature of drying. It was found that sulfur tended to agglomerate after being freshly ground even if allowed to stand in a closed container at room temperature. A freshly ground sulfur sample which contained 45% of particles retained on a #140 screen on standing one hour in a sealed bottle at room temperature reverted to a content of 55% of particles retained on a #140 screen.

On the other hand, the moisture content or time of storage of ground charcoal or potassium nitrate appeared to have little effect on the screen analysis, but only on the time necessary for screens to reach equilibrium. Figure 3 shows a typical screen analysis versus time of a charcoal sample containing 0.75% moisture, screened at a relative humidity of 58%. This is compared with screen analysis versus time of the same sample dried at 85°C. overnight.

It may be seen that undried (0.75 - 1.90%) potassium nitrate required 70 minutes to come to weight equilibrium while dried potassium nitrate (moisture less than 0.25%) required 30 minutes. Undried charcoal (moisture 0.75 - 1.0%) required 30 minutes while dried charcoal (moisture less than 0.25%) required 15 minutes to reach weight equilibrium. It became possible with this and other sieving information to control charcoal and potassium nitrate particle size more closely.

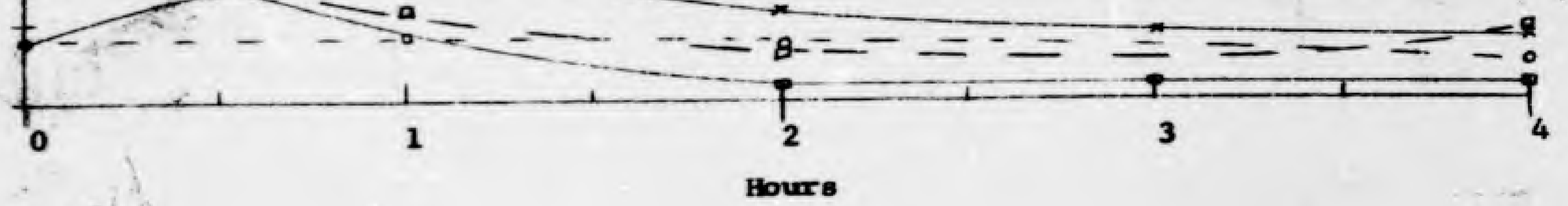
The increase of particle size of sulfur from agglomeration and under various heating conditions suggested some further work with the particle size of sulfur. A study of the effect of friction and impact, such as that encountered in the commercial ball milling operation, upon sulfur was studied. 'Sulfur was ball milled in both glass and light steel mills for one hour, using 0.75 inch

Effect of Ball Mill Construction on Sulfur Particle Size



NOTE: ○ : # 325 sieve; △ : #270 sieve; □ : # 230 sieve; X : # 200 sieve

Sulfur Particle Size Change at 50° C.



NOTE: ○ : # 325 sieve; △ : #270 sieve; ☐ : # 230 sieve; X : # 200 sieve

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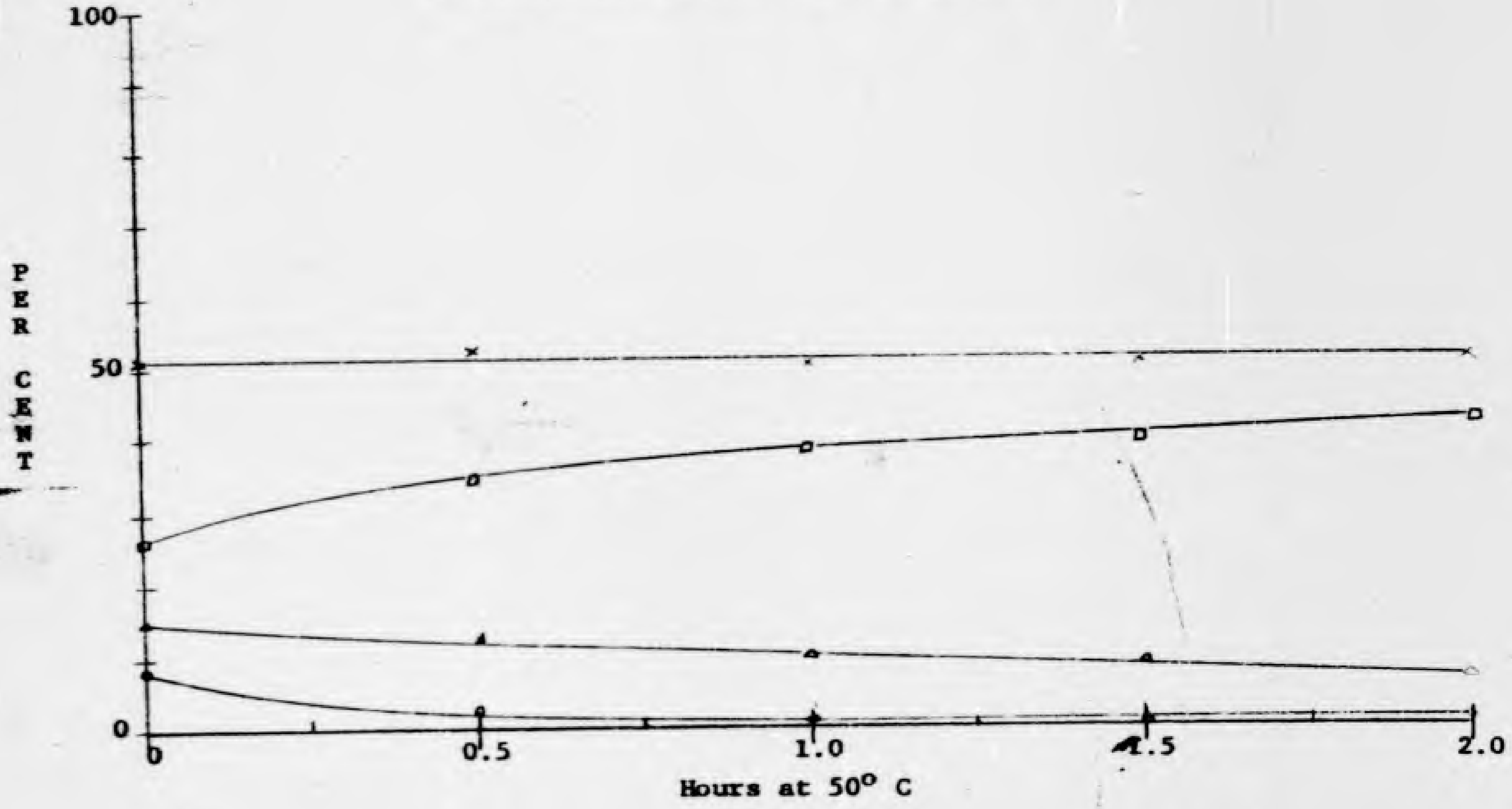


FIGURE 2

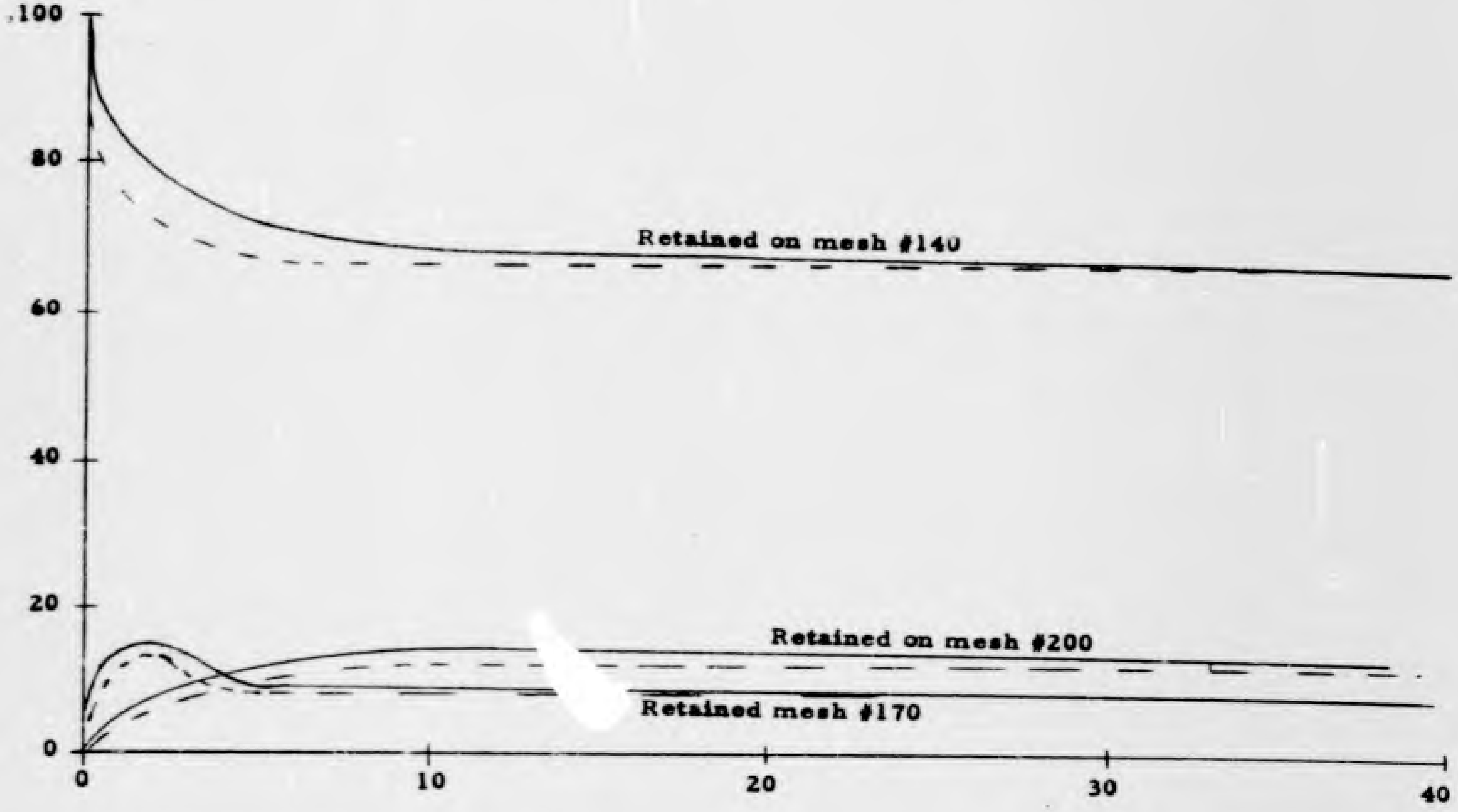


FIGURE 3. Sieving Time, Dried and Undried Charcoal

steel balls. It was found that in a glass, porcelain or similar rigid mill, particle size reduction did occur as milling proceeded for the first one to two hours. However, in the steel wall with more flexible sides, a pronounced tendency occurred for particle size to increase while microscopic study showed definite evidence of flowing of the sulfur. It was found that the same flowing was encountered in both mills at higher RPM, on the order of 25-30 RPM.

From this, it was surmised that the pounding-flexing action of the bolt cuttings against the metal sides of the barrel in the commercial ball milling operation caused the surface of the sulfur to flow. It was probably this agglomeration and surface melting through friction in the ball mill which produces the previously noted effect of a thin sulfur shell coating each particle of charcoal. It was an attempt to duplicate this effect in later work that led us to a consideration of solvents for sulfur as the incorporation vehicle.

Since it was felt that the production time taken by potassium nitrate beater and the ball mill of the conventional black powder plant could be reduced by direct grinding using more modern equipment, the Mikro-Pulverizer of the Metals Disintegrating Company of Summit, New Jersey was evaluated. Three screens were available for this device: 0.013, 0.027, and 0.030." The Mikro-Pulverizer operates by the use of metal flails which force the material to be ground down over a screen, slotted in a herringbone pattern. The flails do not actually contact the screen and there is little chance of sparking. The material to be ground is forced across the screen and through the screen by a pressing and wiping action of the flails, utilizing the shearing effect of this motion against the edge of the screen slots.

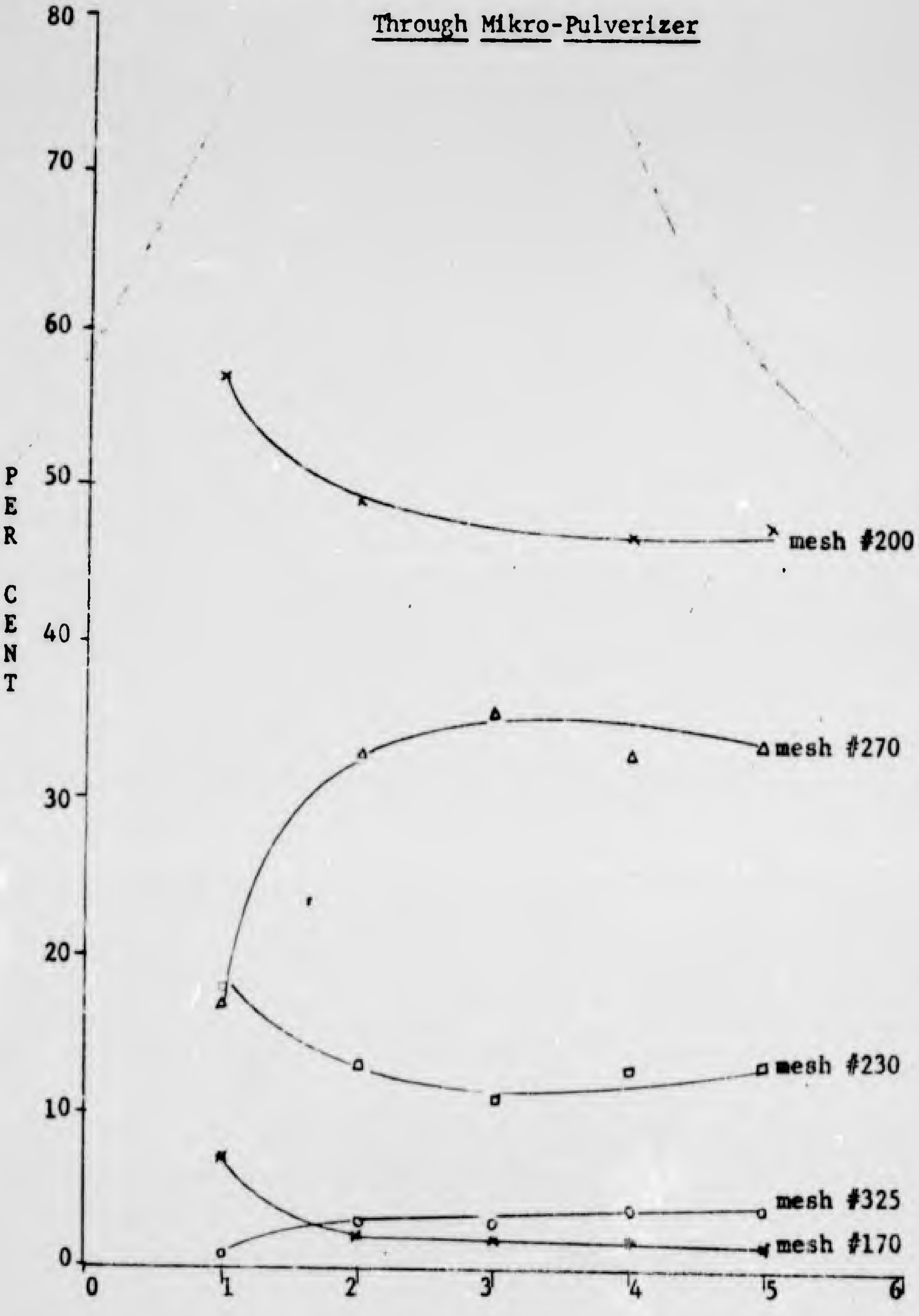
It was found that the 0.013 screen was capable in one pass of the material through the Mikro-Pulverizer of reducing a sample of potassium nitrate, having passed 2.9% of its weight by a U.S. Standard Sieve #100, to a material 86.8% of which was passed by a mesh #200 screen. A more detailed study of size reduction on repeated grindings with the 0.013 screen of the Mikro-Pulverizer was conducted. It was found that the finest material was obtained with two grindings of the potassium nitrate. After this particle size was not significantly reduced to justify further passes through the Mikro-Pulverizer, and, indeed, in some instances agglomeration appeared to occur. (See Figure 4, KNO_3 Particle Size Distribution versus Number of Grindings).

Samples of charcoal were ground and studied in the same manner using the Mikro-Pulverizer. The finest particle size, using the 0.013 screen, was obtained after four passes through the Mikro-Pulverizer. At this point approximately 40 weight-per cent of the charcoal was passed by a U.S. Standard mesh #325 screen. (See Figure 5, Charcoal Particlze Size Distribution versus Number of Grindings).

From the above results it was recognized that, even with repeated passes through the screen, the Mikro-Pulverizer could not achieve the particle sizes observed in commercial black powder. Thus, in grinding potassium nitrate, the smallest significant fraction which could be achieved after two grindings was 74 microns and below while the particle size observed in commercial powder was 10-15 microns. In the same manner, the significant fraction of charcoal after four grindings was only 44 microns and below while charcoal in the 10 micron range was observed in commercial powder. This suggested that the search for

FIGURE 4

Reduction of KNO_3 Particle Size vs Passes Through Mikro-Pulverizer

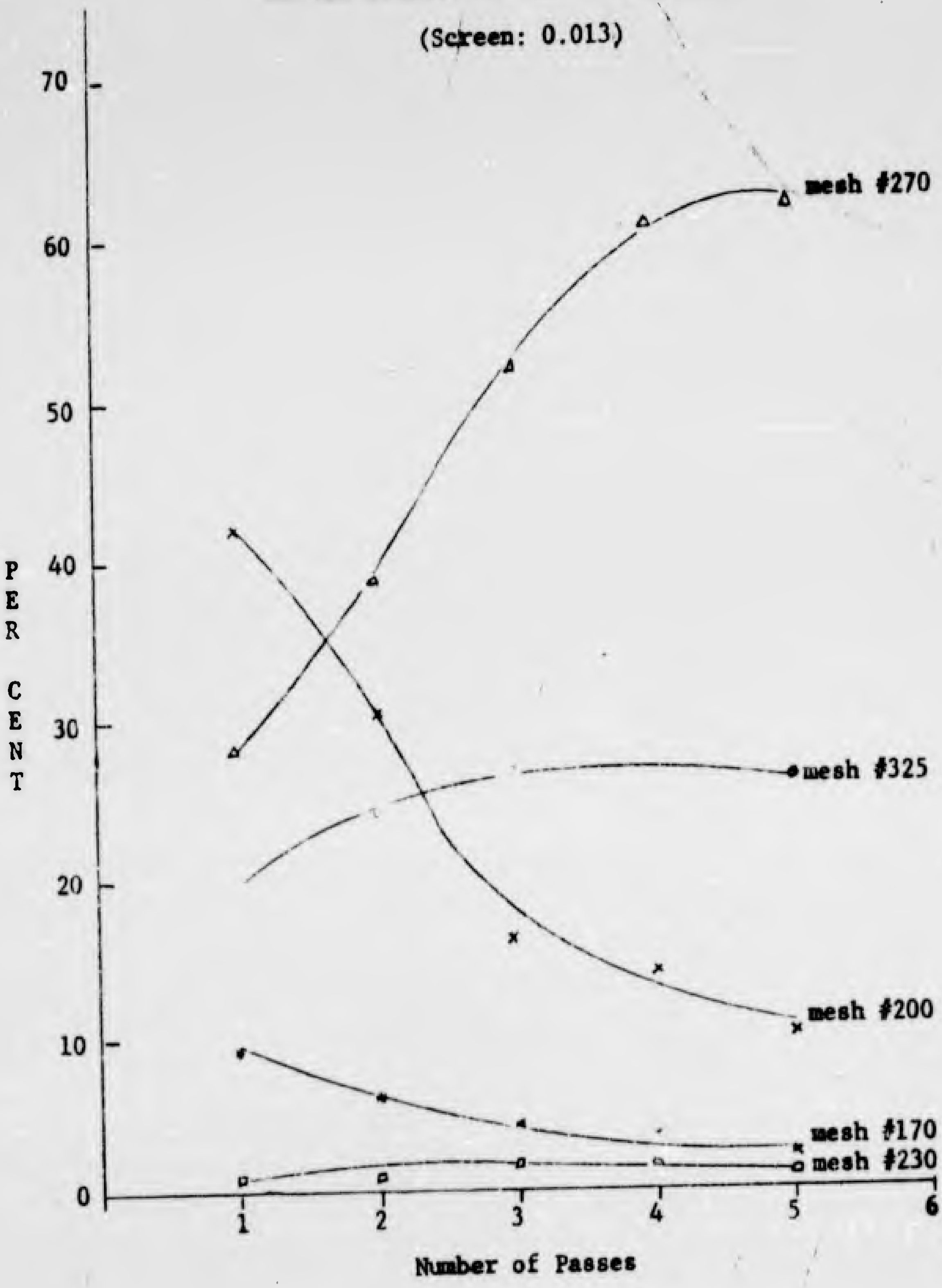


Passes Through Mikro-Pulverizer (0.013)

FIGURE 5

Reduction of Charcoal Particle Size vs Passes Through the Mikro-Pulverizer

(Screen: 0.013)



size reduction equipment should be continued.

In the meantime early experimental samples of a crude black powder were made, using these larger particle sizes, and closed bomb measurements of pressure versus time were made in an attempt to compare these samples with standard commercial black powder. Since it had been found that sulfur coated the charcoal particles as a result of their being ball-milled together, it was decided that this condition should be approximated by the use of a sulfur-dissolving vehicle. Both toluene and carbon disulfide were considered. The carbon disulfide was the less desirable of the two because of its inflammability and toxicity. (Toluene and carbon disulfide properties are compared in Figure 6, A Comparison of the Hazardous Properties of Toluene and Carbon Disulfide). After repeated tests, however, it was found that toluene, with its lower ability to dissolve sulfur, did not give materials capable of being well compacted. Samples made with toluene were generally crumbly and densities were quite low. Toluene was accordingly discarded and the initial phase of the study centered around carbon disulfide, in spite of its obvious safety disadvantages.

Extrusion as a method of densifying was investigated, but, in spite of the pasting of the sulfur by carbon disulfide, it was found that samples could not be extruded. Thus, until the use of the Stokes Tabletizer later in this contract, the extrusion press was used as a hydraulic press to produce cakes of black powder which were then broken up for further processing. Because of the nature of this press, control of density was somewhat difficult. The cake produced was one inch in diameter and two to three inches long.

A COMPARISON OF THE HAZARDOUS PROPERTIES OF TOLUENE AND CARBON DISULFIDE

Property	Toluene	CS_2	Source
Flash Point (°F. closed cup)	40	-22	Perry, Chem. Eng. Handbook, 3rd Edition
Autoignition Point (°F.)	1026	212	Ditto
Explosive Range (% in air)	1.27 - 6.80	10-50	Ditto
Vapor Density (air = 1)	3.14	2.64	Ditto
Vapor Pressure at 68°F. (in mm hg)	22	365	Ditto
Boiling Point (°F.)	232	114	Lange, Chemical Handbook, 8th Edition
Maximum allowable concentration (ppm in air)	200	15	Sax, Handbook of Dangerous Materials, 1st Edition

FIGURE NO. 6

It was felt that much of the problem of density control stemmed from gradations of density from the edge of the cake to the center. In an attempt to increase density control, thinner cakes, on the order of one inch, were pressed. A search continued for a better processing machine to increase production while exercising good density control. Initially, the following samples were made, using the screen #325 charcoal and the screen #200 potassium nitrate: (The proportions of major ingredients, neglecting final graphiting, were: potassium nitrate: 74.0%; charcoal: 15.6%; sulfur: 10.4%).

Sample X-1: Sulfur and charcoal were pasted with carbon disulfide, using 1.0 ml of CS_2 per gram of solids. The resulting paste was dried with stirring at 40°C. Potassium nitrate was added and the resulting mixture pasted with water, 0.25 grams water per gram of solids. The thick paste was compacted, dried at 60°C. and crushed. The sample was screened but not graphited.

Sample X-2: Sulfur and charcoal were ball-milled together as a carbon disulfide paste (1 ml CS_2/gram of solids) for 65 hours. The paste was dried at 40°C. with constant stirring. Potassium nitrate and water were added (0.25 grams H_2O/gram solids) and the resulting paste compacted, dried at 60°C., broken up and screened.

Sample X-3: Sulfur, potassium nitrate and charcoal are pasted together with carbon disulfide (0.7 ml CS_2/gm dry solids) and ball milled for 15 hours. The resulting paste was dried at 40°C. with stirring and .25 gm water per gram solids added to produce a stiff paste, capable of pressing. The resulting cake was dried at 60°C., broken up, and screened.

Sample 4: The charcoal was mixed with azeotropic ethanol, 1 ml ethanol per gram of charcoal. Sulfur, dissolved 1 gram in 0.5 gram of carbon disulfide, was added. The sulfur precipitated and the resulting mixture was dried with stirring at 60°C. Potassium nitrate and 0.25 gram of water per gram solids were added and the resulting paste compacted, dried at 60°C., broken up and screened.

Densities were determined on each of the above samples and 12.0 grams of each were ignited in a closed bomb of 545 ml capacity to yield pressure-time curves for comparison with standard commercial black powder. (The curves are reproduced in Report V of this contract). Figure 7 tabulates data from this series of tests, giving maximum pressure for a 12 gram sample, time to peak pressure, moisture content, and density of sample. Generally speaking, the curves for all four samples were almost congruent with slopes of tangents drawn to the curve at a particular pressure to yield dP/dt giving almost identical results. The samples were as a group much slower in burning rate than the commercial black powder sample.

It was decided that the slowness of the above samples might stem from the size of carbon particles, potassium nitrate particles, dispersion of sulfur, moisture content, or poor control of density. It was decided that a closely controlled sample with a finer charcoal would be made and shipped to Picatinny Arsenal for evaluation. In the meantime a search continued for processing equipment capable of reducing the particle size of the charcoal and potassium nitrate still further.

The sample, hereafter referred to as PCD "A" , for evaluation by Picatinny Arsenal was prepared in the following manner. Hardwood charcoal (obtained from the H. W. Pleitner Charcoal of St. Louis, Missouri was run through the 0.013 screen of the Mikro-Pulverizer twice and screened to obtain particles passing mesh #325 (44 microns or less) screen. This charcoal was further ball-milled for 65 hours and screened to yield charcoal passing a mesh #625 screen (less than 20 microns). Since it was planned that water still be used as the final pasting medium, no further attempt was made to reduce particle size of the potassium nitrate. The potassium nitrate used was thus passable by a mesh #200 screen (less than 74 microns).

The sulfur for the sample was dissolved in carbon disulfide (100 grams of carbon disulfide per 40 grams of rhombic sulfur) and the ground charcoal was incorporated into the mixer. The mixture was dried with constant stirring at 50°C. at atmospheric pressure. At the end of this time the potassium nitrate was added with water, 0.5 ml of water per gram of dried powder. This paste was dried with constant stirring for 12 hours at 60°C. to a final moisture content of 10%. Although some particle growth of potassium nitrate was expected during this period, none was observed, suggesting that the potassium nitrate particles were of fairly uniform size.

The 10% aqueous paste was compressed under 5000 psig to yield a cake which was dried for 5 hours at 60°C. The cake was then crushed and further dried at 60°C. for 15 more hours. The chips were then completely crushed and screened to yield a range of grain sizes comparable to commercial A-1 powder. These particles of density 1.71 were added to the pan of the Ro-Tap Sieve Shaker with

FIGURE 7.

Results of Preliminary Testing of Commercial Black Powder and Various Samples

Type	Sample Size	Maximum Pressure	Time between ign. and peak press.	Approx. Density
Commercial A-1 Black Powder	12 gm	940 psig	.035 sec	1.77 gms/cc
Sample I	12 gm	780 psig	.095 sec	1.61 gms/cc
Sample II	12 gm	800 psig	.085 sec	1.80 gms/cc
Sample III	12 gm	846 psig	.085 sec	1.78 gms/cc
Sample IV	12 gm	810 psig	.090 sec	1.72 gms/cc

one per cent by weight of powdered graphite (Fisher Scientific Company) and shaken for six hours. At the end of this time, excess graphite was removed by screening. This sample was sent to Picatinny Arsenal for evaluation. (See Appendix "C" and III Technical Discussion).

Attempts were made to find commercial sources of charcoal of finer particle size than used in this study. Although a number of commercial suppliers were contacted, all commercial charcoal powder was of larger particle size than that used in this study to date.

Evaluation of the Eppenbach Division, Gifford-Wood Company Model QV-6 colloid mill indicated that this machine might be applicable to the reduction of charcoal and potassium nitrate particle sizes. One major disadvantage of the machine, however, is that a liquid vehicle for the solid materials is needed. This is not true of the Mikro-Atomizer, a device manufactured by the Metals Disintegrating Company of Summit, New Jersey, manufacturers of the Mikro-Pulverizer previously used in this study. This device, widely used in industry, consists of a screw-fed grinder and air-classifier capable of giving small particle sizes (down to the 2-4 micron range) with close control. A quotation on the #5 device together with an air classifier is presented in Appendix "A" together with a drawing of the device. The #5 machine weighs 650 pounds. The largest machine, the #8 in this series, is capable of processing materials to 5 microns average particle size as fast as 2000 pounds per hour.

Samples of charcoal and potassium nitrate, ground by the Mikro-Atomizer were obtained for evaluation. Potassium nitrate ground by the Mikro-Pulverizer yielded material, 99.95% of which passed a mesh #325 (less than 44 microns) screen. Samples of potassium nitrate passing the #325 screen yielded a material with a 35 micron maximum particle size and an average size of 6-8 microns. Charcoal so ground yielded material, 99.98% of which passed a mesh #325 screen. This charcoal, passing the #325 screen, yielded a maximum particle size of 40 microns and an average particle size of 5-6 microns. From this analysis, it may be seen that the Mikro-Atomizer yields particle sizes in the range now found in black powder.

Preparation of a series of samples with both small charcoal and potassium nitrate particle sizes was begun. To avoid possible growth of potassium nitrate crystals during processing, the final pasting medium chosen was azeotropic ethanol in place of water. Preliminary closed bomb tests indicated that this was a good approach and accordingly a sample, hereafter designated PCD "B", was prepared for evaluation by Picatinny Arsenal.

PCD "B" was prepared in the following manner: The sulfur was dissolved in carbon disulfide (40 grams of sulfur per 100 grams of carbon disulfide) and 5-6 micron charcoal added. The resulting paste was dried at 60°C. with thorough mixing. The resulting dried material was very compact and was crushed before proceeding. 6-8 micron potassium nitrate was added with azeotropic ethanol (88 ml of ethanol per 100 grams solids). Using the high speed shearing action of the Eppenbach Homo-mixer, a complete dispersion was achieved after 45 minutes of mixing. The resulting mixture was dried with mixing at 60°C. for seven hours. The dried paste was compressed at

5000 psig and the resulting cake dried further at 60°C. for 15 hours followed by crushing and further drying under vacuum to less than 0.50% moisture. The small grains were then screened to yield powder in the range of commercial A-1 powder and graphited by shaking for seven hours in the pan of the Ro-Tap Shaker with 1% charcoal.

It was found that density control was very difficult with the hydraulic press in use and production was quite slow with no distinct advantage over that found in the present commercial press. Since methods of particle size reduction had been successfully achieved, it was decided that compacting machines should be investigated.

After extensive investigation, two machines were considered for evaluation. The first was Allis-Chalmers Compacting Mill, a high pressure continuous hydraulic press with close control of density. The low pressure version of this machine, operating at 10,000 to 25,000 psi, seemed most applicable. This machine, capable of delivering 5-10 tons per hour costs approximately $30,000. It is available with a granulating mill, vibrating screens and a provision for returning fines to the mill. This device has already been used with safety, according to the Allis-Chalmers sales department, to compact black powder. Unfortunately, we were unable at the time of evaluation to rent this machine or to procure their laboratory services in compacting a black sample since they were not set up to handle hazardous materials. Moreover, there were no funds available for the purchase of this machine. Pelletizers and tabletizers were therefore valuated.

No pelletizer could be found on the market which offered hope of compacting experimental black powder mixtures. All of the pelletizers of promise were either hazardous or used extrusion techniques which had been found unsuitable to this operation. The Stokes Tabletizer, however, operating as a series of miniature hydraulic presses, offered hope of compacting with good control of density. Further, this machine was available at Kingsbury Arsenal where some work had been conducted previously on the compacting of black powder. While it was understood that this work had met with indifferent success because of the critical moisture control necessary to obtain good feeding characteristics, a Propellex representative visited Kingsbury Arsenal to evaluate the machine in situ.

The library of Kingsbury Arsenal yielded reports on the tabletizing of black powder, using the Stokes Tabletizer Model RD-4 press. Black powder grade A-5 was used in these tests. The tabletizing pressure was 10,000 psi apparently using special dies and punches, and a binder was found to be necessary. The binder, present at from 0.25% to 2.0%, was either graphite, barium stearate, or calcium stearate. The specific gravity of the tabletized samples was between 1.45 and 1.60. The necessity of a binder, the high pressures used and the low densities achieved are particularly surprising in view of subsequent results with the RD-4 machine in tabletizing experimental samples under this study.

Dies and punches capable of producing a grain 5/32" in diameter by 3/32" long were obtained from the Stokes Company and the LD-4 Tabletizer, delivered from Kingsbury Arsenal, was set up for evaluation. Initial adjustments were made using an inert material, sodium carbonate. Approximately 10,000 psi was needed to deliver a pellet in the density range of black powder. The production capacity, under the experimental set-up, was about 130 pellets per minutes.

Initially, densities of experimental black powder samples varied widely. This variation was traced to unequal feeding of coarse material into dies. Any attempt to correct this problem by local adjustments resulted in thinner pellets with no improvement in density control. After some investigation, a modification was made to the tabletizer to allow a broader area of feed. This resulted in a smoother feed to the dies and the problems of density control were eliminated from this source. A drawing of this device and a photograph of the device in place is included in Appendix "B". (The drawing holds to the empirical dimensions and angles found effective. There is probably wide latitude in these.)

One further problem was encountered in control of density, using the Stokes tabletizer. It was found that after a short period densities began to vary from their previous set. It was found that all of the Stokes punches had become deformed. Upper punches were found to vary in length from 1.600" to 1.754" while the lower punches varied from 3.212" to 3.270". On further investigation, it was found that the operating pressure ranges in use, 1600 pounds to 2000 pounds per punch, to achieve densities in the range of 1.70 to 1.80 were close to the limit recommended by the Stokes Company. It was assumed that adjustments during the early installation of the machine had deformed the punches.

A new set of punches were ordered from the Schafner Machine Company of St. Louis, Missouri as a result of several conferences with this company which serviced Stokes machines in the area. The punches were, on the advice of the Schafner Company, slightly redesigned. Where the Stokes' punches were rounded on the bottom, the Schafner punches were flat. When these punches were installed, it was found that the edges of pellets obtained were much sharper than with the Stokes' punches. Moreover, the surface of the pellet was very glossy and hard, and density control was, with the help of the Propellex-designed Wiper Assembly, quite reliable.

When the problems of tabletizing to a reliable density and pellet size had been solved with the Stokes LD-4 tabletizer, a series of samples were prepared and shipped to Picatinny Arsenal for evaluation. As a result of a conference with Mr. J. Cussens and later Mr. Maurice Baer, the samples were finally prepared in the manner hereafter detailed and labeled respectively PCD #1 through PCD #7.

Sample PCD #1: 125 grams of sulfur were dissolved in carbon disulfide (20 gm/100 ml) 187 grams of 5-6 micron charcoal were added and the mixture dried at 40°C. The dried material was crushed and slurried in ethyl alcohol with an Eppenbach Homo-mixer. 88 grams of potassium nitrate were added and thoroughly incorporated into the slurry. This material was dried at 60°C. and crushed to a fine powder. The powder was further dried under vacuum (8-10 mm Hg) to a moisture content of less than 1.0% by Karl Fischer method. The dried powder was screened through a 40 mesh screen and tabletized under controlled humidity. Densities were held to an average of 1.77. The pellets were graphited by vibration for 24 hours in the Ro-Tap pan in contact with 1.0% of graphite. Karl Fischer analysis gave a moisture content of less than 0.70%.

Sample PCD #2: This sample was prepared in the same manner as Sample #1, but using in place of the 5-6 micron carbon, a carbon passed by a mesh 270 screen. Final moisture content was less than 0.70%. Average density was 1.76.

Sample PCD #3: This sample was prepared in a manner similar to Sample #1, but in place of ethyl alcohol in the step where potassium nitrate is added, a 50% aqueous ethanol was used. Final average density was 1.76.

Sample PCD #4: 125 grams of sulfur, 187 grams of fine (5-6 micron) charcoal, and 888 grams of fine KNO_3 were mixed, using distilled water as a vehicle. (1000 ml H_2O for 1200 grams). It was impossible to mix the paste with any lower concentration of water. The paste was mixed for 45 minutes with an Eppenbach Homo-mixer and dried at 60°C. The resulting material was crushed to a fine powder and further dried at 60°C. under 8-10 mm Hg vacuum to a moisture content of less than 0.70%. The powder was then passed through a 40 mesh screen and tabletized to an average density of 1.74. The pellets were graphited in the Ro-tap pan.

Sample PCD #5: This sample was prepared in the same manner as Sample #4, with the exception that coarse charcoal (passed through a mesh 270 screen) was used. Average density of the pellets was 1.77.

Sample PCD #6: This sample was prepared in the same manner as Sample #1. It was felt that this sample should be repeated because of the wide variation in density from particle to particle observed during #1's manufacture. (This variation was due to the deformed punches referred to earlier.)

Sample PCD #7: 125 grams of sulfur, 187 grams of 5-6 micron charcoal and 888 grams of fine potassium nitrate were slurried in 1000 ml of ethanol for one hour, using the Eppenbach Homo-mixer. The material was dried at 60°C. and crushed to a fine powder. The material was further dried at 60°C. under 8-10 mmHg vacuum to a moisture content of less than 1.0%. The dried powder was passed through a mesh 40 screen and tabletized to an average density of 1.73. The pellets were graphited with 1.0% graphite in a Ro-tap pan and the moisture determined to be less than 0.70%.

These samples were sent to Picatinny Arsenal for evaluation in the closed ballistic bomb and the M-58 Primer. Preliminary evaluations in a low pressure ballistic bomb were conducted at Propellex. In addition, all measurements were conducted at pressures less than 1000 psi. It was hoped that such low pressure data would avoid the variations introduced by difference in geometries. Data including burning time, peak pressure, and dP/dt at 100 psi was reported for all samples versus a typical commercial black powder sample. These results together with traces are given in Appendix "B".

III. TECHNICAL DISCUSSION

During the course of this contract, nine samples of experimental black powder were transmitted to Picatinny Arsenal for evaluation in the Closed Bomb Test and the M-58 Primer Test. (The method of preparation of each of these samples has been previously reviewed in detail in Part II, EXPERIMENTAL WORK PERFORMED. The samples designated as PCD "A" and PCD "B" were those transmitted in November, 1958 and January 1959 and reported by Picatinny Arsenal in letters dated January 20, 1959 and March 6, 1959. Samples prepared subsequent to those were numbered serially #1 through #7 and so shown in this report as in earlier correspondence.

All data transmitted by Picatinny Arsenal on samples "A", "B", and #1 through #7, have been tabulated in the table in Figure 8. Relative Quickness was not reported as such on samples "A" and "B" but rather a series of dP/dt values with parallel values at parallel chamber pressures for a commercial black powder. Taking the dP/dt value of the standard black powder as 100 for each reported chamber pressure, a relative dP/dt value, Relative Quickness by definition, was calculated for each reported point on the Closed Bomb trace of samples "A" and "B". Since no information was given on the conditions under which each sample was run and no information was available on how the conditions of these tests compared with those subsequently run on samples #1 through #7, maximum pressures for samples "A" and "B" were reported in a separate column in the tabulation in Figure 8. It should be noted (see Picatinny Arsenal letters dated January 20 and March 6 in Appendix "C") that the dP/dt values at identical pressures for the two commercial black powder samples varied significantly. From this, it may be inferred that a similar variation was encountered in the DuPont Lot #30-139 and Egyptian Lot #8-1 black powder samples used as standards in subsequent tests. While this variation, as we shall see, is probably not as significant as it first appears, the variation among standard samples nevertheless points out that values for Relative Quickness should not be interpretted too closely since several large sources of error exist in determining dP/dt values quite apart from the inherent variation of production batches of black powder.

Laboratory analyses were run by Picatinny Arsenal on only a few of the total number of black powder samples. The specific gravity of sample "B" was found to be quite lot, 1.42 as against the required 1.72. As might be expected, this lower density found a reflection later in a higher linear burning rate.

Sample #1 was found to have a high ash content. This analysis was confirmed by a rerun. No explanation for the high ash could be found. Sample #2 was found to be slightly below specifications in density, giving on two trials an average density of 1.66. It was reported during the manufacture of this sample that densities might run below specification since trouble was being encountered with deformed Stokes punches. This condition was subsequently corrected and lab analyses by Propellex showed samples #3 through #7 to lie within specific gravity specifications. The significance of specific gravity as an analytical criterion will be discussed later in this section.

The significance of Maximum Pressure and Relative Force <u>per se</u> in the Closed Bomb Test is debatable. Theoretically, a fixed mixture of sulfur, charcoal, and potassium nitrate will yield a constant number of calories per gram and a constant number of moles of gas of the same average molecular weight. One would expect, therefore, that all mixtures of these components following the black powder proportions would give essentially the same heat evolution per gram of mixture and the same pressure within a given volume. That a variation in pressure does exist from batch to batch probably stems from true differences in composition, differences in moisture content and incomplete combustion. However, probably the most significant factor in the difference of Maximum Pressure and of Relative Force reported in the tabulated results is the factor of burning time. It should be noted that the lower values for Relative Force in samples #3, #4, and #5 are accompanied by higher burning times: 79.5, 75, and 73.5 milliseconds respectively, all values three to eight times as long as the samples exhibiting Relative Forces in the mid 90's. While one might plot exponential curves showing the relationship of Maximum Pressure and Relative Force to burning time, it is probable that such relationships are unique with the test set-up being used and are not easily generalized.

The values reported for heat of explosion and heat of combustion for the two black powder standards and for Propellex samples #1 and #2 also vary among themselves by large factors. While PCD #1 displays a heat of explosion almost identical with DuPont #30-139, its heat of combustion appears quite a bit below either the DuPont or the Egyptian samples. The variation in heat of explosion might be explained by the relatively high potassium nitrate content compared with the Egyptian sample while the high ash and lowered charcoal content might explain the lowered heat of combustion.

PCD #2 on the other hand shows a very low heat of explosion with a heat of combustion higher than the Egyptian sample and close to the DuPont sample. There seems no ready explanation for this variation. The lack of any significant composition variation to explain the variation in heats of explosion and combustion in the case of PCD #2 suggests the variation in PCD #1 and PCD #2 both may be a result of the method rather than any detectable differences in composition.

The ignition delays reported in the Closed Bomb Test vary from as little as 18.5 msec to as long as 119 msec. It has been generally theorized that ignition delay is influenced strongly by both composition and physical state of the surface. The tabulated data seems to indicate a correlation between burning time, decline in relative quickness with pressure, and ignition delay. It appears that those tabletized samples showing the highest burning rates and greatest relative quickness at any pressure were most easily ignited. Interestingly enough, the irregular samples "A" and "B" did not show any major advantage in ease of ignition over the best of the tabletized samples. Production Black Powder showed greater ease of ignition though this decreased ignition delay was not more than one-half of the delay encountered with the best of the experimental samples. It is suggested that some of the difference in ignition delay might stem from the loose, less compact nature of the surface graphiting. The production samples were graphited by continuous abrasion with flake graphite in a revolving barrel while the experimental samples were graphited by shaking the sample in a vibrating pan with powdered graphite.

It is suggested that the latter treatment yielded a less compact surface, offering more insulation to the heat from the ignition flame.

Generally speaking, those samples (PCD #3, #4, and #5) which showed the longest ignition delays in the closed bomb test showed the greatest variation in ignition in the M-58 Igniter Test. PCD #3, for instance, showed ignition delays varying from as low as 3.1 msec to as high as 24.7 msec. PCD #7 sample, on the other hand, with an average delay of 1.8 msec showed a range of ignition delays between 1.3 and 2.5 msec.

It may be observed that, with the notable exceptions of PCD #3, #4, and #5, ignition delays in both the Closed Bomb Test and the M-58 Primer Test for commercial black powder and the various Propellex experimental samples were comparable. The larger and more erratic ignition delays encountered with samples #3, #4 and #5 were accompanied by significantly longer burning times in both the Closed Bomb and the M-58 Primer together lower Relative Quickness values and a sharper decline of Relative Quickness with Pressure in the Closed Bomb Test. From these data, it may be inferred that ignition delay is less influenced by method of graphiting and the geometry of the particle, i.e. whether a smooth cylinder or jagged particle, than by the surface characteristics of the particle stemming from the method of grinding and incorporating the components.

What appears most significant of all the data collected in the M-58 Primer Test is the maximum pressure data before rupture for a standard weight of propellant. At first glance it might be expected that this rupture pressure

Figure 8.	DuPont Lot #30-139	Egyptian Lot #8-1	PCD Sample "A" *	PCD Sample "B" *	PCD Sample #1	PCD
ANALYSIS (Requirements Given)						
Moisture, % (max: 0.70)	0.50	0.32			0.31	
Specific Gravity(1.72 - 1.77)	1.74	1.74		1.42	1.77	
Ash, % (Max: 0.80)	0.17	0.51			1.87	
Potassium Nitrate,%(74.0)	73.50	74.22			73.75	7
Charcoal, % (15.6)	16.34	15.46			13.14	1
Sulfur, % (10.4)	9.94	10.32			10.35	1
Total Volatiles				0.08		
CLOSED BOMB TESTS						
Maximum Pressure, PSI	6101	5965	4470	4600	5089	
Maximum Pressure, Standards for "A" and "B" ONLY: **			4740	4600		
Relative Force:	102.2	100.0	94.4	100.0	90.3	
Relative Quickness,						
650 PSI:	99.4	100.0	102.5	181.0	40.3	
1000 PSI:	99.4	100.0			40.3	
1300 PSI:	94.8	100.0	105.8	198.8	34.3	
1900 PSI:			103.5			
1950 PSI:	90.9	100.0		209.5	28.6	
2000 PSI:	94.8	100.0			34.3	
2600 PSI:	89.8	100.0		206.8		
3000 PSI:	90.3	100.0			28.6	
4000 PSI:	89.8	100.0				
		(7.3)				
Burning Time, msec.	9.3	11.3			34.2	
Ignition Delay, msec.	18.5	30.7	56.8	19.6	57.4	
		20.3	55.0	49.2		
Heat of Explosion, cal/gm	673.8	729.6			674.1	
Heat of Combustion, cal/gm	1471.0	1263.0			1076.0	1
M-58 PRIMER TESTS						
Ignition Delay Average, Msec.	2.7	5.7			5.8	
Burning Time, msec.	1.7	2.2			4.0	
Maximum Pressure to Rupture	16180	13230			6140	
Ignition Delay Range, msec.						
Burning Time Range, msec.						
Max. Rupture Pressure Range,PSI						

* Sample "A": See Picatinny Arsenal Letter Jan. 20, 1959
Sample "B": See Picatinny Arsenal Letter March 6, 1959

** Separate samples of production black powder were run for tested. See appropriate Picatinny Arsenal Letter.

m Lot 1	PCD Sample "A" *	PCD Sample "B" *	PCD Sample #1	PCD Sample #2	PCD Sample #3	PCD Sample #4	PCD Sample #5	PCD Sample #6	PCD Sample #7
32			0,31	0,38					
74		1.42	1.77	1,66					
31			1,87	0,36					
22			73,75	73,90					
56			15,14	15,98					
32			10.35	10,13					
		0.08							
35	4470	4600	5089	5509					
Y1 **	4740	4600							
0	94,4	100,0	90,3	98,0	84,4	78,3	88,5	95,4	96,8
0	102.5	181.0	40.3	56.1	37.7	41.2	47.1	106.7	107.3
0			40.3	56,1					
0	105.8	198.8	34.3	41,9	29.3	29.9	37.9	71.8	76.6
	103,5								
0		209.5	28.6	31.0	22.8	20.9	30.4	52.9	56.1
0			34.3	41.9					
0		206.8			17.9	15.3	23.7	41.9	44.0
0			28.6	31,0					
0									
5)									
3			34.2	28.4	79.5	75.0	73.5	32.5	29,5
7	56,8	19.6	57.4	35.0	107.0	119.0	106.0	40.5	50.5
3	55.0	49.2							
6			674,1	603,2					
0			1076.0	1455,0					
7			5.8	6.0	10.6	7.5	6,9	2.8	1.8
2			4.0	3.3	8.0	7.4	7.4	3,6	3.2
0			6140	2840	1693	1542	1662	3091	3780
					3.1-24.7	2.0-11.0	2,6-15.1	1.7-3.6	1.3-2.5
					6,7-9,7	5,0-10,7	6,4-8,3	3,0-4.3	2.8-3.6
					1279-1905	1243-2175	1398-1864	2019-3961	2796-5487

de Picatinny Arsenal Letter Jan. 20, 1959

ee Picatinny Arsenal Letter March 6, 1959

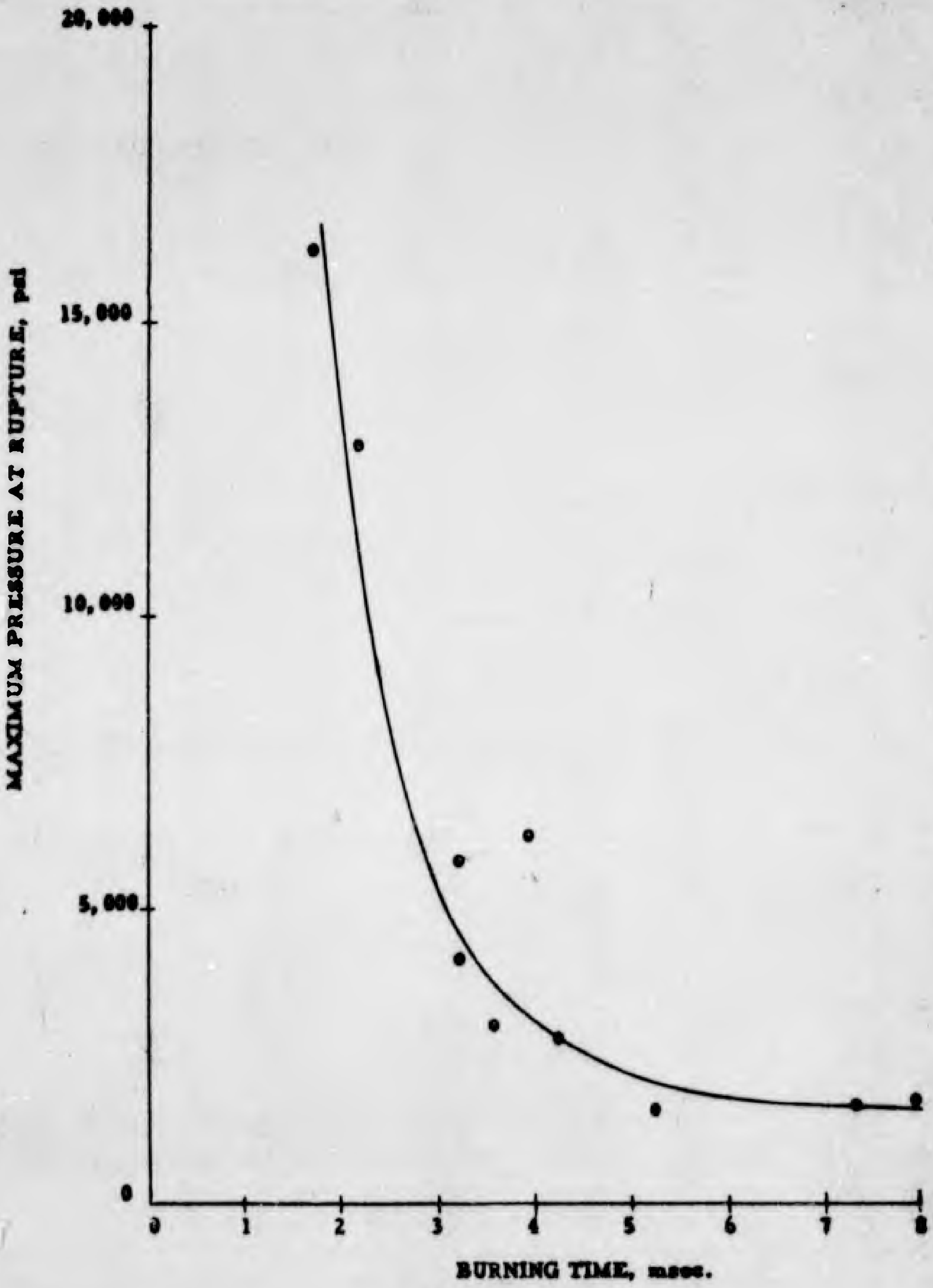
les of production black powder were run for each sample A and B
appropriate Picatinny Arsenal Letter.

would be generally the same for all propellants and dependent almost completely on the structural parameters of the primer case. However, such is not the case as the tabulated data in Figure 8 demonstrates. While the production samples of black powder gave maximum pressures of 13,230 to 16,180 psi, PCD samples #1 through #7 gave pressure values an order of magnitude lower! (Unfortunately, no values are available for the performance of samples "A" and "B" in the M-58 Primer.)

There does, however, appear to be some correlation between burning time and maximum pressure. If burning time in msec is plotted as abscissa with maximum pressure as ordinate as in Figure 9, a clearer relationship emerges. The more likely relationship is probably between maximum pressure and time to reach maximum pressure, but the latter data is not available. Burning time probably bears a fairly linear relationship to time to maximum pressure, however. With obvious deviations noted, an exponential relationship between maximum pressure and burning time seems demonstrated by the plot of the former against the latter in Figure 9. From this relationship, it would appear that the pressures developed in the M-58 Primer before rupture are directly related to the rate of onset of pressure, i. e. the development and maintenance of a high quickness (dP/dt) value. This phenomenon might be explained by postulating a failure pressure of about 1,500 psi, the horizontal asymptote of the Maximum Pressure vs Burning Time Plot, together with a fixed period of mechanical opening of the primer case and a fairly reproducible rate of gas effusion from the ruptured case, regardless of the propellant.

FIGURE 9

MAXIMUM PRESSURE VS BURNING TIME IN M-58 PRIMER



Under such conditions, after the rupture pressure of 1,500 psi had been reached, the maximum pressure recorded results from the difference between mass flow from the burning propellant and mass loss from the rupture in the primer wall. If the situation is truly as described, the faster burning propellant will naturally produce the largest over pressurization between the initial rupture of the primer case and the final complete failure of the case as a pressure vessel.

All of the deviations from the performance of commercial black powder in both the Closed Bomb Test and the M-58 Primer Test have been reflected by the behavior of the pressure-time trace in the closed bomb test and more specifically by the slope of the tangent line to the trace at various pressures, the quantity designated as Quickness and defined mathematically as dP/dt. In the tabulated data in Figure 8, all quickness data is reported as relative quickness, that is quickness relative to a chosen commercial black powder trace whose dP/dt values at various pressures are taken as 100. Samples "A" and "B" have been compared each with a separate commercial black powder sample run at the same time; all other samples... PCD #1 through #7 and DuPont #30-139... have been compared with Egyptian Lot #8-1 whose dP/dt values at various pressures have been taken as the base 100.

From the tabulation it may be seen that the relative quickness values of DuPont #30-139 declined slightly as the chamber pressure increased while all of the PCD numbered samples showed a sharp decline of relative quickness with increasing chamber pressure. Interestingly enough, no such decline of relative quickness is noted in the traces of samples PCD "A" and PCD "B" and indeed, in the case of sample PCD "B", a definite increase in relative quickness occurs between 650 and 1950 psi. Of all of the numbered PCD samples, only samples PCD #6 and PCD #7, the latter a sample made with only alcohol as a liquid vehicle, demonstrate a quickness in the early history of the pressure-time trace comparable with commercial samples.

The influence of density upon the ballistic trace of the various samples is interesting. Only two samples are known to deviate sharply from specifications. These samples are sample "B" with a specific gravity of 1.42 and sample PCD #2 with a specific gravity of 1.66. As might be expected, sample "B" shows a much higher burning rate than samples of higher density. All dP/dt values reported are approximately twice those of commercial black powder. However, in spite of this wide variation in burning rate and density between Egyptian Lot #8-1 and PCD "B", the same ratio of dP/dt values for the two samples holds over a range of 2600 psi, that is, the pressure-time traces are essentially parallel.

On the other hand, the lowered specific gravity of PCD #2 does not noticeably affect the burning rate of this sample which in every other respect than density is identical with PCD #1. Moreover, the ratio of dP/dt values between #1 and #2 remains approximately the same as evidenced by the parallel decline of relative quickness values. Thus, in the instance of PCD #2 as with PCD "B", the density of the propellant appears to have little effect upon the slope of tangents to the pressure-time trace at given pressures, even though the burning rate of the propellant may be profoundly affected by this change in density.

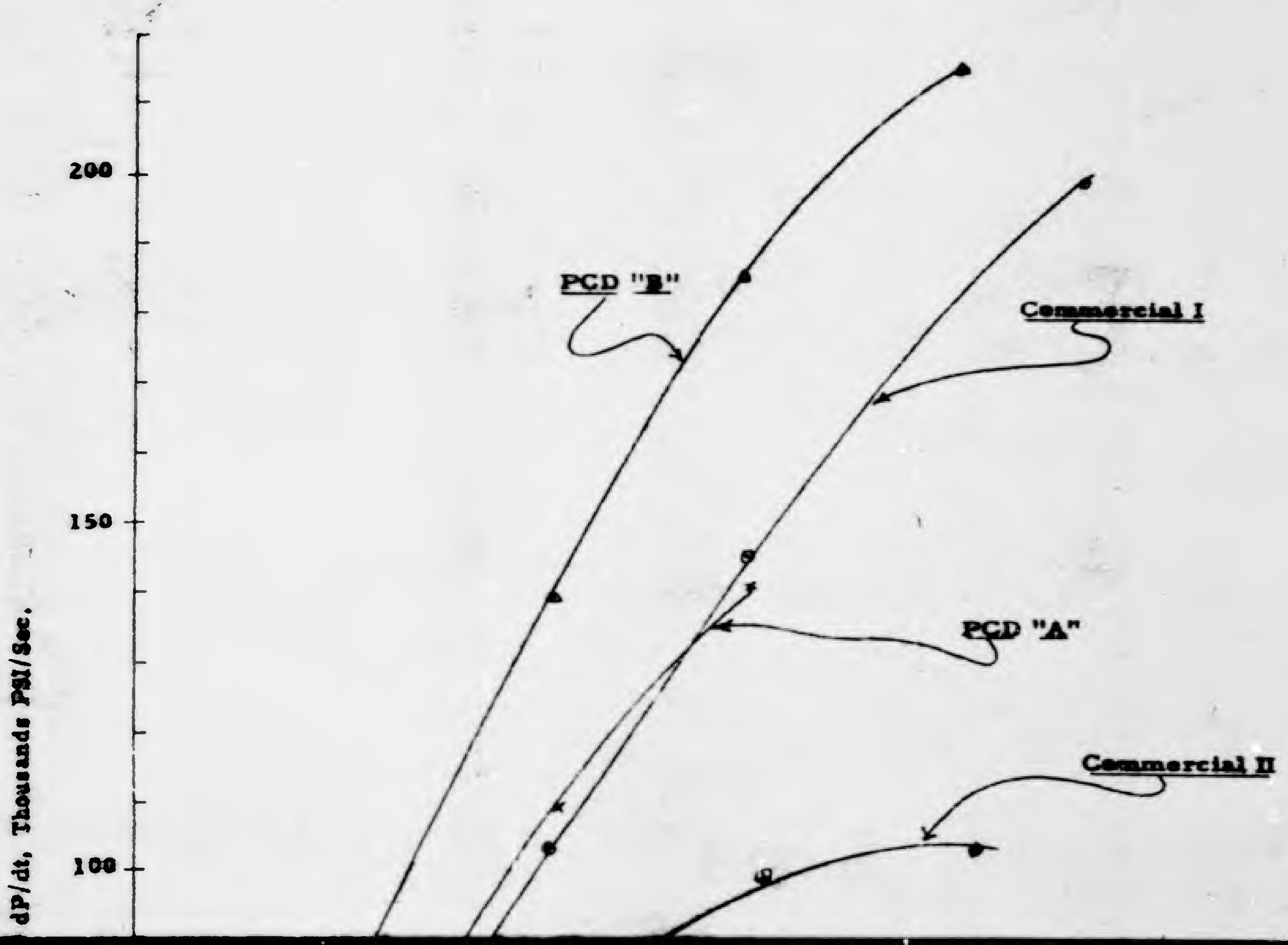
This peculiarity of behavior of the quickness values of the various propellant samples is most clearly seen from Figure 10. Here the relative quickness values have been converted to actual dP/dt values based on curve I, the plot of dP/dt vs Pressure for the commercial black powder sample reported in the Picatinny Arsenal letter of January 20, 1959. All dP/dt values for samples PCD #1 through #7 were calculated in this manner and plotted. Actual values for samples "A" and "B" were available for plotting as were the values for another commercial black powder sample, plotted as II. The black powder samples used for deriving curves I and II were not otherwise identified in the Picatinny Arsenal letters referred to.

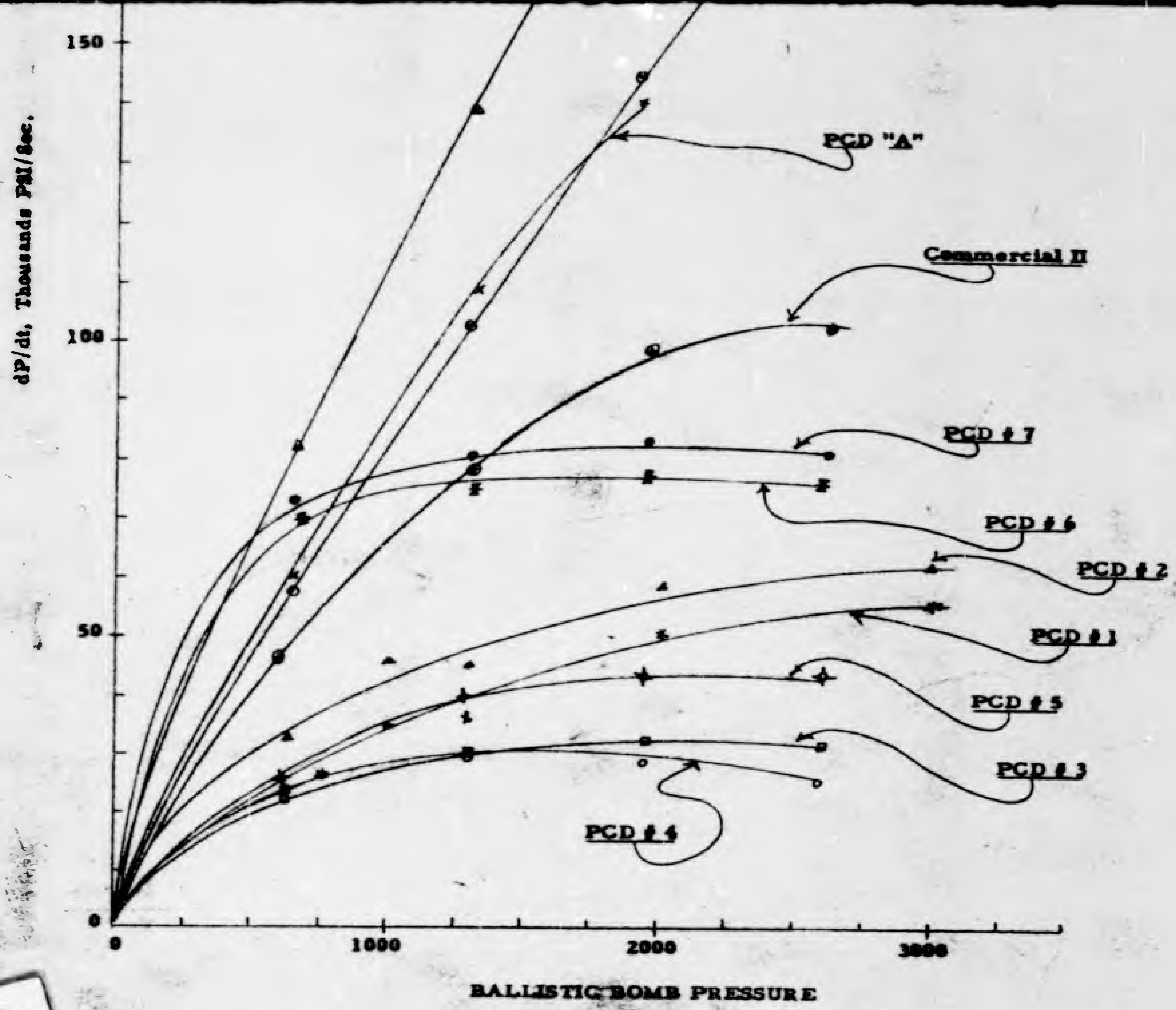
From the plot in Figure 10, it becomes immediately apparent that all plots fall into one of two categories. Plots I, II, A and B are all of general unmodified exponential shape. All other plots, i.e. PCD #1, #2, #3, #4, #5, #6 and #7 show a tendency to change slope after the first 500 psi and all curves exhibit plateaus, those in curves #3, #4, #5, #6 and #7 dipping downward again above 2000 psi.

No ready explanation for the grouping of number samples into one family of curves with the lettered samples and commercial samples in another is available on the basis of manufacturing methods, unless one considers the final compacting process. All numbered samples were, of course, compacted with a Stokes tabletizer and exhibited a regular cylindrical geometry. On the other hand, samples "A" and "B" were compacted by a hydraulic press, then broken up and screened so that an irregular surface, similar to that found in commercial powder, should have resulted.

FIGURE 10

VARIATION OF QUICKNESS, dP/dt, WITH PRESSURE
BALLISTIC BOMB TEST




dP/dt, Thousands PSI/Sec.
150
100
50
0
1000
2000
3000
BALLISTIC BOMB PRESSURE
PCD "A"
Commercial II
PCD # 7
PCD # 6
PCD # 2
PCD # 1
PCD # 5
PCD # 3
PCD # 4

It was decided that, if such a sharp difference in curve shapes resulted from the above noted differences in geometry, this difference could be predicted on the basis of a mathematical treatment of different geometries of black powder burning in a closed ballistic bomb. It was decided to explore mathematically the behavior of two general types of black powder: (1) powder with an irregular surface such that the resulting grains were essentially neutral burning, and the total burning surface did not change appreciably, and (2) black powder of a general regular geometry whose surface was some squared function of a linear dimension of the grain and whose burning history should be strongly regressive as the burning surface decreased during burning.

In a closed bomb, if heat loss is negligible and perfect adiabatic gas processes are assumed, the classical pressure-volume-temperature relationship holds:

$$PV = n\,R\,T \tag{1}$$

or

$$P = n\,\frac{(R)\,(T)}{(V)} \tag{2}$$

n, the number of moles of gas, is defined as the mass of gas, X, divided by the average molecular weight of the gas, $\overline{M}$. Thus:

$$P = X\,\frac{(R)\,(T)}{(\overline{M})\,(V)} \tag{3}$$

However, X, the weight of gas, is identical with the weight of propellant consumed which is defined as the volume, Vo, times the density, p, of the propellant. However, the volume of propellant may be generally described as the burning surface, S, times the web, w, times some constant, A, where the term "web" has its usual significance as the shortest burning dimension of a propellant geometry. For most purposes, the approximation inherent in this treatment is served if the web is assumed to be the mean radius of any black powder particle, treated as if the grain were a true sphere.

From the above relationships, we arrive at an equation:

$$P = \frac{(A)(w)(S)(p)(R)(T)}{(\bar{M})(V)} \qquad (4)$$

However, of the above terms, A is a constant as is p, as is R and V. We may assume that, with no heat loss, all gases generated have the same temperature, which makes T a constant. We may also assume that, under identical conditions of pressure and temperature, gas compositions will be identical such that $\bar{M}$ may be considered a constant. We may thus combine all of these into a larger constant, K, such that:

$$K = \frac{(A)(p)(R)(T)}{(\bar{M})(V)} \qquad (5)$$

The equation then becomes:

$$P = (K)(w)(S) \qquad (6)$$

However, the web, w, can be redefined as the burning rate, r_B, times the time of burning, t, such that:

$$P = (K)(r_B)(t)(S) \qquad (7)$$

Since the rate of burning can be defined as:

$$r_B = a\ P^n \qquad (8)$$

we find:

$$P = (a)(K)(t)(S)(P^n) \qquad (9)$$

or, combining a and K into B:

$$P^{(1-n)} = (B)(S)(t) \qquad (10)$$

Differentiating with respect to t:

$$(1-n)\, P^{-n} \frac{dP}{dt} = (B)(t)\frac{dS}{dt} + (B)(S) \qquad (11)$$

and:

$$\frac{dP}{dt} = \left\{\frac{B}{(1-n)}\frac{dS}{dt} + \frac{(B)(S)}{(1-n)}\right\} P^n \qquad (12)$$

Defining a new constant, $C = \frac{B}{(1-n)}$

$$\frac{dP}{dt} = \left\{(C)\frac{dS}{dt} + (C)(S)\right\} P^n \qquad (13)$$

We may assume two general conditions resulting from the different geometries postulated for black powder grains. If the surface of the grain is highly irregular, the initial burning surface is quite large and the initial dP/dt values are high. With an irregular grain the burning does not decrease by simple geometric progression and with an extremely irregular grain the burning surface may remain essentially unchanged throughout the complete burning history of the sample. In such an extreme case, we may say that:

$$\frac{dS}{dt} = 0$$

In such a situation, equation (13) above becomes:

$$\frac{dP}{dt} = (C)(S)\, P^n \qquad (14)$$

However, since we have said that S in this instance is a constant, (C) and (S) may be combined into a new constant, E:

$$\frac{dP}{dt} = E\, P^n \qquad (15)$$

n, in equation (15) is, of course, the pressure exponent encountered in the burning rate equation and is less than 1.0. It may be seen that the form of

(15) may be taken as an unmodified parabolic curve of the form $Y^2 = 2PX$, i. e. :

$$\left\{\left(\frac{dP}{dt}\right)^{\frac{(1-n)}{2}}\right\}^2 = 2\left|\frac{E^{(1-n)}}{2}\right|P \tag{16}$$

A more complicated situation emerges where the black powder grains are of a regular geometry, such that the following equation is satisfied:

$$S = D X^2 \tag{17}$$

D in the above is the appropriate proportionality constant which in the specific case of a sphere is 0.75π. X is a linear dimension. In the case of a sphere, this is the radius. In the case of a cylinder, X becomes the radius of the base while equation (17) expands to two terms of the form, DX^2 but with different proportionality constants. Any regular geometry may, however, be represented by the general equation (3).

If (17) is differentiated with respect to t:

$$\frac{dS}{dt} = 2 X D \frac{dX}{dt} \tag{18}$$

Substituting (18) in the general equation (13):

$$\frac{dP}{dt} = \left|2\ C D X \frac{dX}{dt}\ P^n\right| + \left|C\ D\ X^2\ P^n\right| \tag{19}$$

However, the rate of change of the linear dimension of the particle is the negative of the burning rate at the particular instant, i. e. :

$$\frac{dX}{dt} = -r_B = -a\,P^n \tag{20}$$

Substituting (20) in (19) we get:

$$\frac{dP}{dt} = (X^2\ P^n - 2aX\ P^{2n})\ D\ C \tag{21}$$

(21) is, of course, a more complex curve than (13) since it embodies two opposed influences. Increasing pressure serves to increase the burning rate by the relationship described in (8), thus increasing the mass flow rate and increasing dP/dt values. On the other hand, the decrease of burning surface described by (18) has the opposite effect. (21) shows that the shape of the dP/dt vs P trace is not a simple function but one dependent on the size of the grain as represented by X and the shape as represented by D, the geometric proportionality constant.

One may test for the existence of a plateau in both (15) and (21) by setting up the following condition:

$$\frac{d(dP/dt)}{dP} = 0 \qquad (22)$$

When (15) is handled in this manner, the result is:

$$n\,E\,P^{n-1} = 0 \qquad (23)$$

which has only the solution one might expect for a parabolic function centered at X=0; Y=0 : P = 0

An attempt to treat (21) in a similar manner yields the complex expression:

$$0 = \left[nX^2P^{n-1} + 2P^nX\frac{dX}{dP} - 2aP^{2n}\frac{dX}{dP} - 4anXP^{2n-1}\right]DC \qquad (24)$$

where $dX/dP = (dX/dt)(dt/dP) = (-r_B)(dt/dP) = (-aP^n)(dt/dP)$

so that:

$$0 = \left[nX^2P^{n-1} - 2aP^{2n}X\frac{dt}{dP} + 2a^2P^{3n}\frac{dt}{dP} - 4anXP^{2n-1}\right]DC \qquad (25)$$

$$(2\,aP^{2n})(X-aP^n)\frac{dt}{dP} = nX\,(XP^{n-1} - 4aP^{2n-1}) \qquad (26)$$

and:

$$\frac{dP}{dt} = \frac{(2aP^{2n})(X-aP^{n})}{n(X^{2}P^{n-1} - 4aXP^{2n-1})} \qquad (27)$$

Extracting (1/n) or (n^{-1}), the right hand term on simple algebraic long division yields a solution in terms of an infinite series:

$$\frac{dP}{dt} = n^{-1}\left\{\frac{1}{2}aX^{-1}P^{n+1} - \frac{3}{8}P - \frac{3}{32}a^{-1}XP^{1-n} - \frac{3}{128}a^{-2}X^{2}P^{1-2n} \ldots \frac{3}{2(4)^{m}}a^{1-m}X^{m-1}P^{(1-n[m-1])}\right\} \qquad (28)$$

Since dP/dt approaches a finite term only as the sum of the series is extended to include the term where m is infinite, (28) may be expressed as the limit of a sum:

$$\frac{dP}{dt} = n^{-1}\left\{\left(\frac{1}{2}aX^{-1}P^{n+1}\right) - \left(\lim_{m\to\infty}\sum_{k=1}^{m}\frac{3}{2(4)^{m}}a^{1-m}X^{m-1}P^{1-mn+n}\right)\right\} \qquad (29)$$

From the above it may be seen that d(dP/dt)/dP yields a finite solution other than O for (21) since:

$$\lim_{m\to\infty}\left(\frac{3}{2(4)^{m}}a^{1-m}X^{m-1}P^{1-mn+n}\right) = 0 \qquad (30)$$

It may thus be seen that for black powder of regular geometry, a part of the dP/dt versus P curve exists where d(dP/dt)/dP = O exists and that the conditions specifying this inflection point or segment are found in (29). This situation is quite different from the situation with black powder of an irregular geometry where a parabolic form for dP/dt versus P is approached. In this latter case, no such inflection point (other than P=O) or plateau may be found. It is worth noting that the solution of d(dP/dt)/dP for grains of regular geometry is independent of the constant, D. Thus, the solution of (24) is quite independent of the geometry being treated, provided only that it may be treated by (17), i.e. that the geometry is "regular".

The preceding treatment demonstrates the theoretical basis of the data observed and plotted in Figure 10. The deviation of the plots of cP/dt vs P for commercial powder from true parabolic form is, of course, explainable in that it was assumed that dS/dt for these grains was 0. A further conclusion from this treatment may be reached with respect to the M-58 Primer. A consideration of the graph in Figure 9 plus the data in Figure 10 suggests that a powder whose grains are of regular geometry is incapable of giving the pressure onset required for the high maximum pressures and low burning times for which the M-58 primer was designed.

To minimize the influence of geometry on ballistic properties in experimental samples, an attempt was made to use a low pressure ballistic bomb for measurements. Traces for commercial black powder and for samples PCD #1 through #7 are given in Appendix "D". The data from these traces are tabulated in Figure 11.

Relative force correlates qualitatively with the same quantity in Figure 8. However, the difference in pressures is more pronounced than with the high pressure ballistic bomb, probably due to the longer burning times involved. (The high pressure bomb usually gives burning times circa 11 msec.) With longer burning times, chance for heat transfer to the body of the bomb increases with a consequent drop of pressure.

dP/dt's and relative quickness values were derived graphically for only one pressure: 100 psi. Because of the longer burning time in the low pressure test, it is felt that dP/dt values are more realistic in relation to each other since the values are more sensitive to small fluctuations in onset. The relationship of relative quickness among the samples generally preserves the relationship found in the high pressure bomb.

Samples #1 and #6 have changed their relative positions in the low pressure bomb. Both of these samples were prepared in the same manner, but PCD #1 was more irregular in surface since it was compacted with deformed Stokes punches with round surfaces. One would expect the trace of PCD #1 to be steeper than that of PCD #6. This is not true in the data from Picatinny Arsenal's high pressure bomb but is true in the low pressure data in Figure 11.

Samples PCD #3, #4, and #5 show very low burning rates in both tests. This is significant since sample PCD #3 was processed in the final incorporation and compacting with 50% aqueous alcohol while PCD #4 and #5 were processed using only water as the vehicle. Visual observation indicated the growth of potassium nitrate particles in #4 and #5. The growth of such particles in these samples and in PCD #3 would explain the lowered burning rates.

PCD "A", however, was processed with water in the final incorporation stage after sulfur had been added via a carbon disulfide vehicle. Here no crystal growth was observed and resulting ballistic properties of PCD "A" were very close to commercial powder. We may assume that the KNO_3 in PCD "A" remained essentially below mesh 200. (Below 74 microns).

The KNO_3 used in samples PCD #1, PCD #6, and PCD "B" was nominally 6-8 micron (the average particle size with a maximum particle size of 35 microns).

The charcoal used in PCD "A" was mesh 325 (less than 44 microns) while later charcoals were often 5-6 microns average size with a maximum particle size of 40 microns. The use of either did not noticeably affect the ballistic data from Picatinny Arsenal. The same statement may be made with reference to the use of mesh #200 KNO_3 versus 6-8 micron KNO_3. Probably the major arguments in favor of using finer charcoal and KNO_3 are:

(1) ease of grinding and elimination of screening by use of Mikro-Atomizer

(2) better fluidity of mixtures, and

(3) greater ease of compression.

From the slope of the dP/dt versus P trace of both samples PCD #6 and #7 during the first 500 psi of the closed bomb test (see Figure 10), it would appear that these samples might have equaled PCD "A" and I, given an irregular geometry. This is borne out by relative quickness data from the low pressure bomb. (See Figure 11.) This is particularly interesting in view of the fact that PCD #7 was made by compressing the three components (both KNO_3 and charcoal being as fine as possible) using only azeotropic alcohol. If reproducible results similar to PCD "A" can be achieved with alcohol alone, this should certainly be an improvement over earlier approaches.

Proposed processes for the manufacture of black powder, based on the use of both carbon disulfide and alcohol and on alcohol alone are discussed in IV, CONCLUSIONS AND RECOMMENDATIONS. For the reasons previously discussed, these processes are based on the use of 5-6 micron charcoal and 6-8 micron KNO_3.

FIGURE 11

LOW PRESSURE BALLISTIC BOMB DATA

Sample *	Burning Time	Peak Pressure	Relative Force	dP/dt at 100 psi	Relative Quickness
Commercial	0.046 sec	226 psi	100.0	7550	100.0
PCD #1	0.049 "	220 "	97.4	7150	94.7
PCD #2	0.058 "	164 "	72.7	3740	49.5
PCD #3	0.105 "	157 "	69.6	3650	48.4
PCD #4	0.130 "	162 "	71.7	2300	30.5
PCD #5	0.250 "	144 "	63.8	1070	14.2
PCD #6	0.047 "	180 "	79.7	4250	56.4
PCD #7	0.047 "	216 "	95.6	7100	94.2

*Each sample: 10.0000 ± 0.0005 gm

IV. CONCLUSIONS AND RECOMMENDATIONS

1. Conclusions from Experimental Work:

a. Vehicles and Solvents:

During the course of this study various solvents and vehicles have been considered. Toluene was discarded early in the study since its ability to dissolve sulfur was comparatively low. In those instances where the coating of the charcoal with sulfur was desired, carbon disulfide was used. The use of this material has three major disadvantges, however. Carbon disulfide is an inherently dangerous material with a low ignition temperature and high vapor pressure. (See Figure 6, page 17). Moreover, it is fairly expensive, selling for from 7.1 to 9.1 cents per pound in less than tank car quantities and for 5.45 cents per pound in tank car quantities. The use of carbon disulfide together with another solvent complicates the solvent recovery picture.

The use of water in several connections was investigated during the course of the study. While water was used as the final incorporation medium in sample PCD"A", results from samples PCD #4 and #5 where only water was used as the vehicle and from PCD #3 where 50% ethanol was used, indicate that control of potassium nitrate crystal growth becomes more difficult where water is present. This seems particularly true where many small particles are present in a KNO_3 sample where the particle range is large. The use of water, it appears, might necessitate extensive regrinding and screening, even if other factors were equal.

The use of fine particle materials mixed in azeotropic alcohol and compressed after the vehicle was removed led to sample PCD #7. This technique, from closed bomb and M-58 primer data, seemed equal to the use of both carbon disulfide and ethanol. The trace for PCD #7 during the first 500 psi of the closed bomb test in Figure 10, page 41, conforms closely to that for commercial black powder and the data from the low pressure bomb confirms this.

It seems therefore that two approaches to the use of vehicles are of interest in further process investigations:

(1) The use of carbon disulfide for dispersing the sulfur around the carbon particles followed by the use of azeotropic ethanol for the final mixing vehicle.

(2) The use of azeotropic alcohol for the complete mixing of all three components.

Considering the excellent pellets formed using only the alcohol, it would appear that, under the pressure of compacting, there is extensive flowing and redistribution of the sulfur. Microscopic evidence confirms this. It is suggested, therefore, that (2) above is the more desirable procedure and the one most likely to yield reliable black powder at a reasonable cost.

b. Particle Size of Components:

The ballistic data from PCD "A" and from samples PCD #1 through #7 suggest that the use of potassium nitrate particle sizes below mesh #200 has little effect on ballistic properties. The reduction of charcoal seems to be more critical. It was found generally that any black made with charcoal particle size greater than that passing mesh #325 (sieve opening: 44 micron) was invariably too slow in the low pressure bomb or high pressure bomb.

It was found, however, that the finer charcoal and potassium nitrate both contributed to easier compacting and better control of density. It is recommended, therefore, that charcoal be held to below mesh #625 (sieve opening: 20 micron) and potassium nitrate to below mesh #325. Charcoal and potassium nitrate ground by the Mikro-Atomizer to the fineness used in this study is felt to be excellent for further work.

c. Grain Geometry:

The data from Picatinny Arsenal on both the High Pressure Ballistic Bomb and the M-58 Primer agree generally with the theoretical treatment in the previous pages. It had been demonstrated that black powder of a regular geometry cannot be used in the M-58 Primer and will not generally give ballistic results comparable with regular commercial powder.

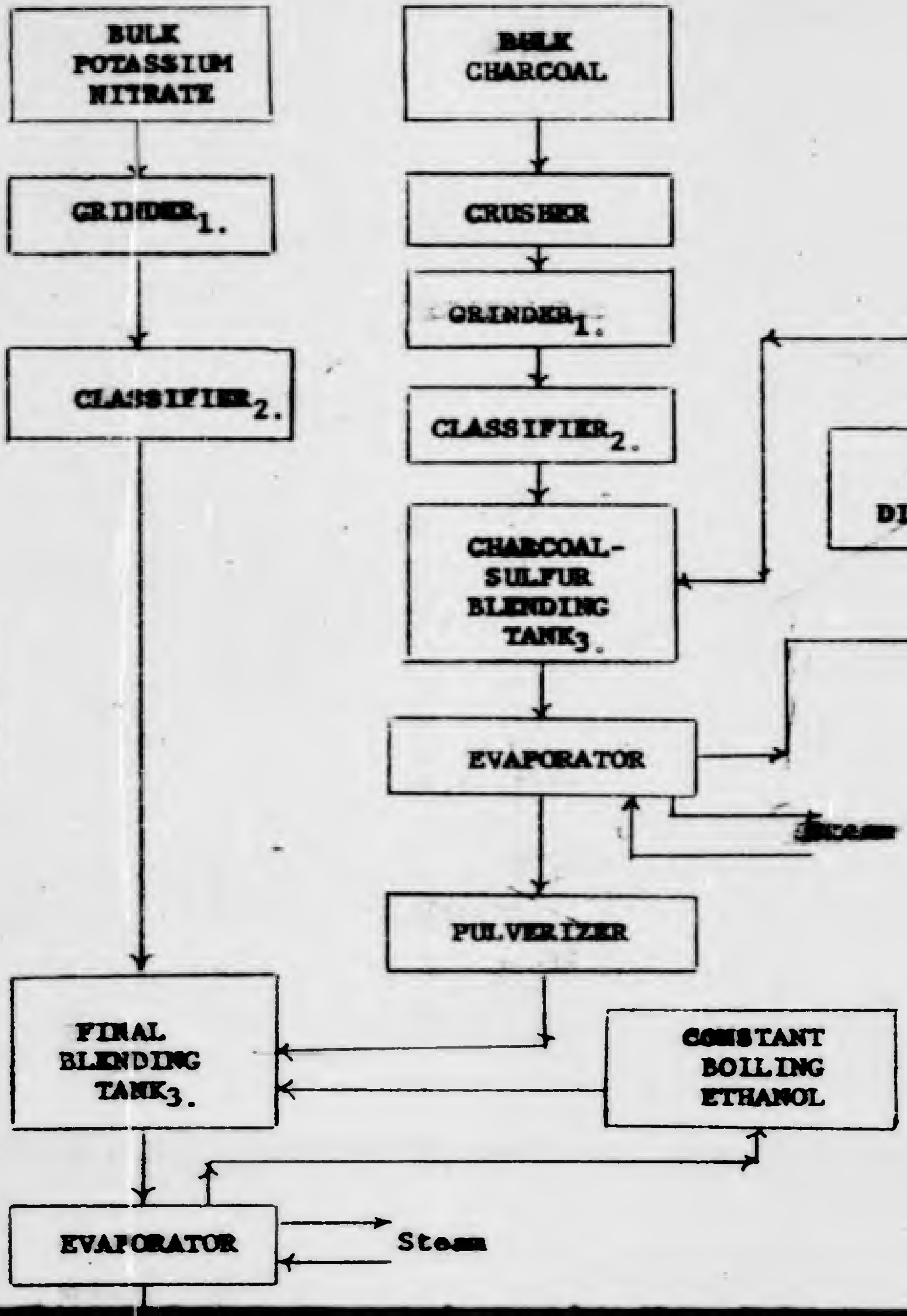
It is, therefore, recommended that all future work on duplicating black powder be confined to irregular geometries. The Stokes Tabletizer is not suited to the manufacture of black powder of various grades in production quantities.

2. Recommended Process and Process Equipment:

Figures 12 and 13 give two general flow diagrams for the production of black powder, using the techniques investigated under this contract. The first and less desirable approach (Figure 12) is that using carbon disulfide for the initial incorporation of the sulfur. The more desirable (Figure 13) is the second process which consists simply of the appropriate reduction of particle size followed by combination in an azeotropic ethanol vehicle, drying and compacting.

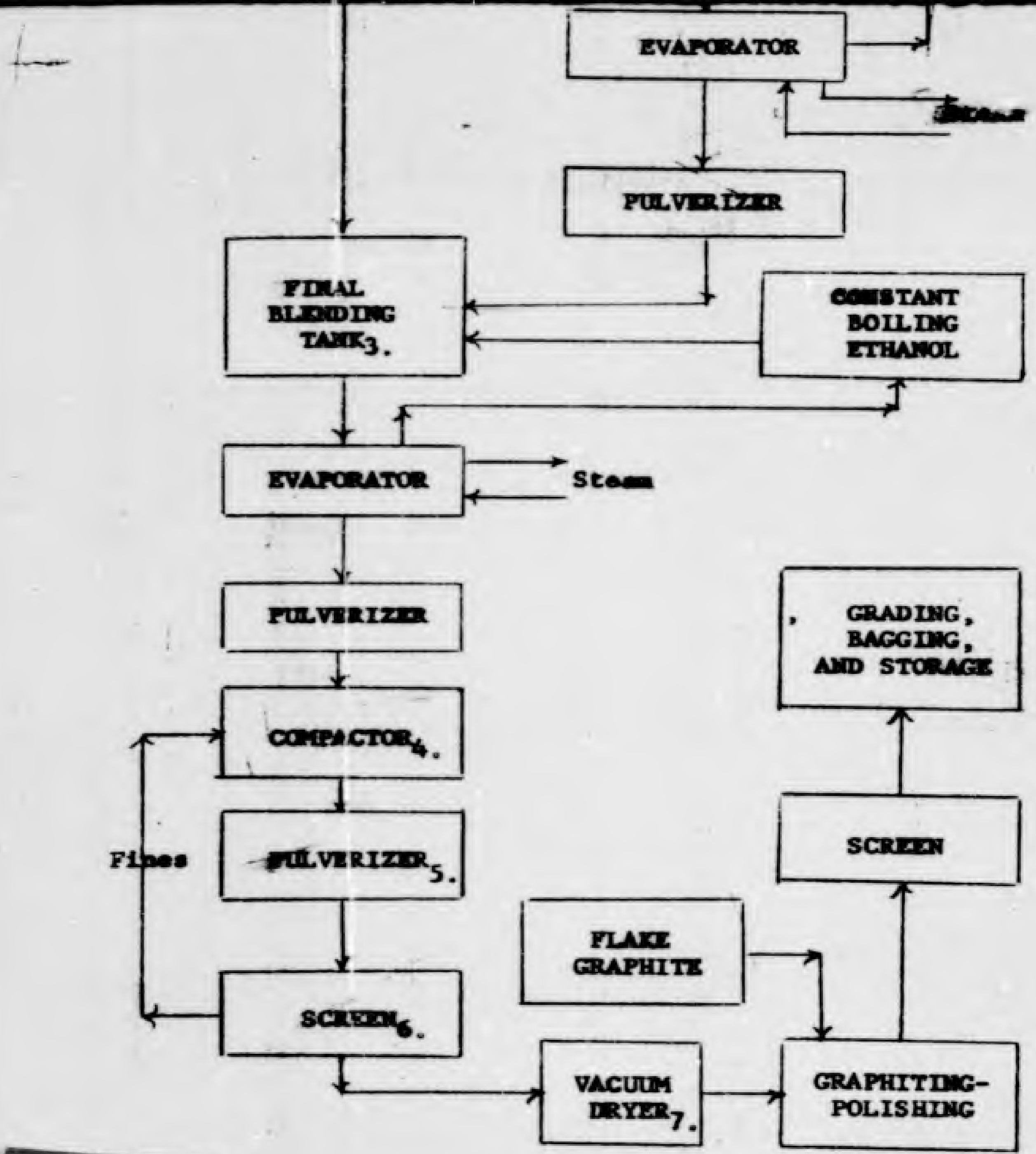
If the scheme in Figure 12 is used, fairly elaborate precautions must be taken to avoid sparks and friction which might set off the carbon disulfide vapors. Care must also be taken in storing the material to avoid sources of heat, such as direct sunlight which might ignite this easily flammable material.

1



RECOMMENDED EQUIPMENT

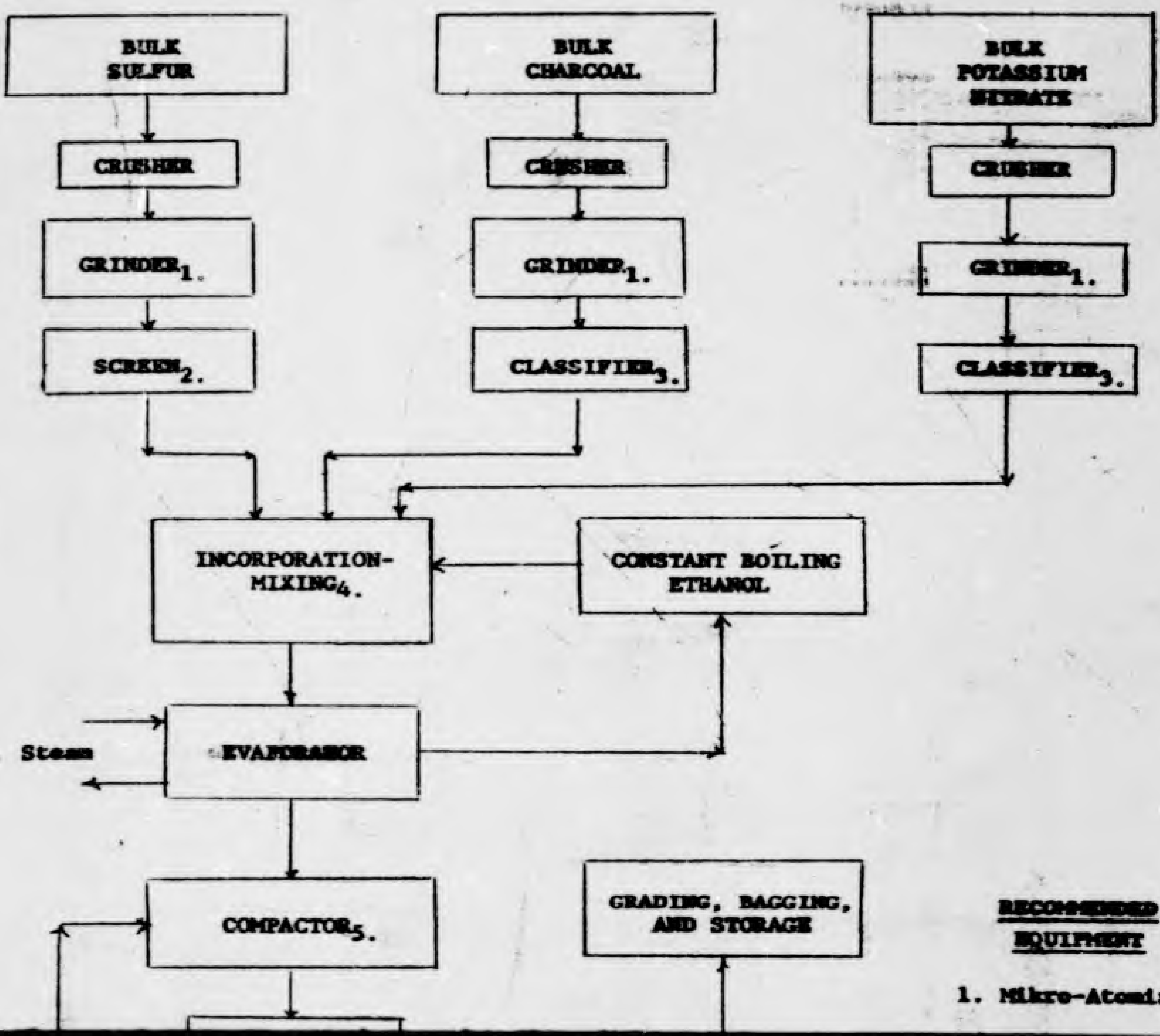
1. Mikro-Atomizer
2. Auxiliary of 1. above
3. Steam-jacketed tanks with Eppenbach Homo-mixer (Alter-



RECOMMENDED EQUIPMENT

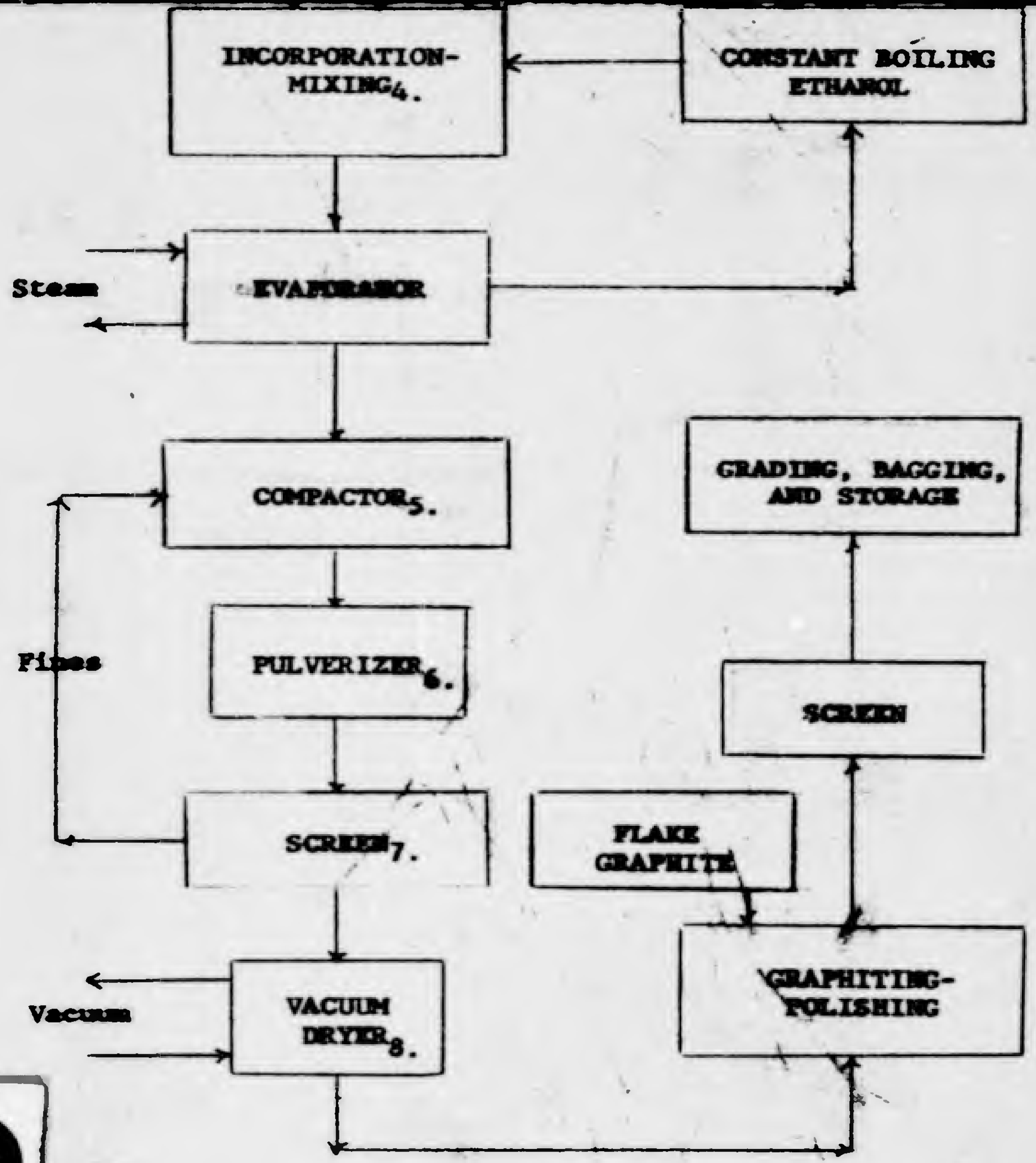
1. Mikro-Atomizer
2. Auxiliary of 1. above
3. Steam-jacketed tanks with Eppenbach Homo-mixer (Alternate: Sigma Blade Mixer
4. Allis-Chalmers Compacting System.
5. Included as auxiliary of 4.
6. See 5. above
7. Vacuum style optional

FIGURE 12: PROPOSED BLACK POWDER PROCESS, SCHEME I



RECOMMENDED EQUIPMENT

1. Mikro-Atomizer



RECOMMENDED EQUIPMENT

1. Mikro-Atomizer
2. Mesh #200
3. See 1. above
4. Heated vat plus ribbon mixer or Eppenbach Homo-mixer
5. Allis-Chalmers Compacting Process
6. See 5.
7. See 5.
8. Vacuum style optional

FIGURE 13: PROPOSED BLACK POWDER PROCESS, SCHEME II

a) Particle Size Reduction

A number of grinding and mailing machines have been considered for the reduction of the three constituents of black powder. Generally, the requirements for the reduction of lump sulfur are not nearly so stringent as with the potassium nitrate and charcoal. The Mikro-Pulverizer of the Metals Disintegrating Company of Summit, New Jersey has been successfully used and fits well into a production set-up, either batch or continuous.

A number of ball mills have been considered, most notably the Ball and Pebble Mills manufactured by the Paul O. Abbe Company. While ball milling does accomplish the size reduction required, the process is not adaptable to high production rates or to continuous processing. Ball and pebble mills *per se* were therefore not considered for use in any future pilot operation.

The use of the Allis-Chalmers Vibrating Mill, a modification of the ball mill, does suggest itself. This device, essentially a vibrating ball mill, is capable of continuous processing, handling up to a ton an hour of material. By suitable control of "stay" within the mill, it is possible to carry particle reduction down to a material, 90% of which is 20 microns or less. Separate air classification equipment is available with this apparatus.

However, for reduction of charcoal and potassium nitrate, the Mikro-Atomizer of the Metals Disintegrating Company, Pulverizing Machinery Division seems most suited to the particle sizes needed and the production volume desired. This device (see Appendix "A") includes air classification column such that a narrow range of particle sizes is automatically obtained. Using this device, no subsequent screening of the grind is necessary. For more complete classification of particles an air classifier is available.

The listed capacity of the Mikro-Atomizers for a representative substance, carbon black pellets, is:

Model	Average Size	Capacity, Lb/Hr
#5	7-10 microns	150
#6	" "	600
#8	" "	2100

Because of its general utility and continuous capacity, the Mikro-Atomizer offers the best features for incorporation into a black powder production set-up and is the recommended equipment.

b) Screening and Classification

In the particle size reduction stage of raw materials, the effluent grind from the Mikro-Atomizer is sufficiently classified for most purposes. However, an air classifier is available for further separation if necessary. A similar air classifier is available with the Allis-Chalmers Vibrating Mill.

For later screening of green black powder grain, vibrating screens of various types are applicable. In general, the single shaft vibrating screens, manufactured by the Dravo Corporation, seem adaptable to redesign in non-sparking metals. The Aero-Vibe vibrating screen Model AVS of Allis-Chalmers is similar in operating principle but seems even more adaptable to the screening of hazardous materials.

Because of their inapplicability to continuous processing, gyratory screens have not been considered for further pilot work.

c) Mixing and Incorporation

For batch-type production, some type of steam jacketed kettle equipped with a mixer is essential in either Scheme I or II for each incorporation. A variety of such kettles suggest themselves. One of particular interest is a twin motion pony mixer with dust cover manufactured by the J. H. Day Company, Division of Cleveland Automatic Machine Company.

It has been found that the Homo-Mixer of the Eppenbach Company, a high-speed shearer-impeller mixer, is of particular value in working with coarse pastes of the type encountered after the removal of carbon disulfide and addition of potassium nitrate and alcohol in Scheme I. This same mixer is similarly of value in the one-step incorporation in Scheme II. The Eppenbach offers the same type of impeller-shearer blade in a flow unit with variable return such that this type of mixing might be applied to continuous process.

For pilot plant work, the possible use of a sigma blade mixer has been considered in both processes. A variety of such mixers are available from such companies as the J. H. Day Company, Paul O. Abbe Company, etc. While these mixers are of limited value in a full scale production set-up, in a pilot plant they offer the advantage of being equipped for application of both heat and vacuum, allowing the combination of incorporation, evaporation, pulverizing and vacuum drying steps.

However, for full scale production, a continuous heavy duty mixer of the Eppenbach type is desirable. The Type GPM Heavy Duty Continuous Mixer of the Patterson Foundry & Machinery Company is considered an excellent approach as is the Paul O. Abbe Double Shaft Continuous Mixer.

d) Compacting

No form of extrusion machine, pelletizer nor tabletizer seems applicable to the black powder process. Forgetting for the moment the problems associated with a regular geometry, the production rates of these machines are generally too low. The available equipment for compacting appears to be limited to some form of hydraulic press, either bed or roll.

After evaluation of most of the equipment now available, it is recommended that future pilot plant compacting systems be based upon the Allis-Chalmers Compacting Process. The low bearing load compacting mill seems most applicable to the needs of black powder processing. This device, selling for approximately $30,000, has a pressure range of 10,000 to 25,000 psi. This device is, according to Allis-Chalmers, currently in use at the Badger Ordnance Works for compacting and flaking black powder.

A typical flow sequence which is most applicable to the black powder process is given in the Allis-Chalmers literature which is reproduced in Appendix "E".

APPENDIX "A"

MIKRO-ATOMIZER

Pulverizing Machinery Division
Metals Disintegrating Company, Inc.
Summit, New Jersey

Phone: *CRestview 3-6360*

QUOTATION

No. -352
Page 1

PULVERIZING MACHINERY DIVISION
METALS DISINTEGRATING COMPANY, INC.
SUMMIT, NEW JERSEY

DATE: December 30, 1958

TO: Propellex Chemical Corporation
P. O. Box 187
Edwardsville, Illinois

FOR:
Mr. T. N. Scortia, Asst. Mgr.
Chemical Development

YOUR REF:

ITEM	QUANTITY	DESCRIPTION	UNIT PRICE	TOTAL
1	One (1)	#5 Mikro-Atomizer mounted on welded structural steel base as shown on attached Dimension Print S-672. The main drive will be through guarded multi V-belt, and feed drive will be of the guarded variable speed type. Main drive motor will be 5 H.P., 3450 RPM, explosion-proof type, and the feed drive motor will be fractional horsepower, gear head, explosion-proof type. The mill construction is of Dairy metal housing with rotor shaft and interior parts of 18-8 stainless steel. Discharge is through stainless steel duct to stainless steel cyclone. Cyclone exhaust is connected to a large nylon woven cloth bag. One extra nylon bag is provided. Motors are for operation on 220 Volt, 3 Phase, 60 Cycle current and for Class I, Group D, hazardous location.		$4453.00

FOB: Summit, New Jersey

TERMS: Net 30 Days

SHIPMENT: 7 weeks after receipt of order and all details necessary to complete fabrication.

SHIPPING WEIGHT: 700 Lbs.

The price quoted is a firm quotation for a period of 30 days after quotation date except that we reserve the right to increase the price at time of shipment in the amount of any increase to us in the cost of purchased equipment not of our manufacture.

PULVERIZING MACHINERY DIVISION

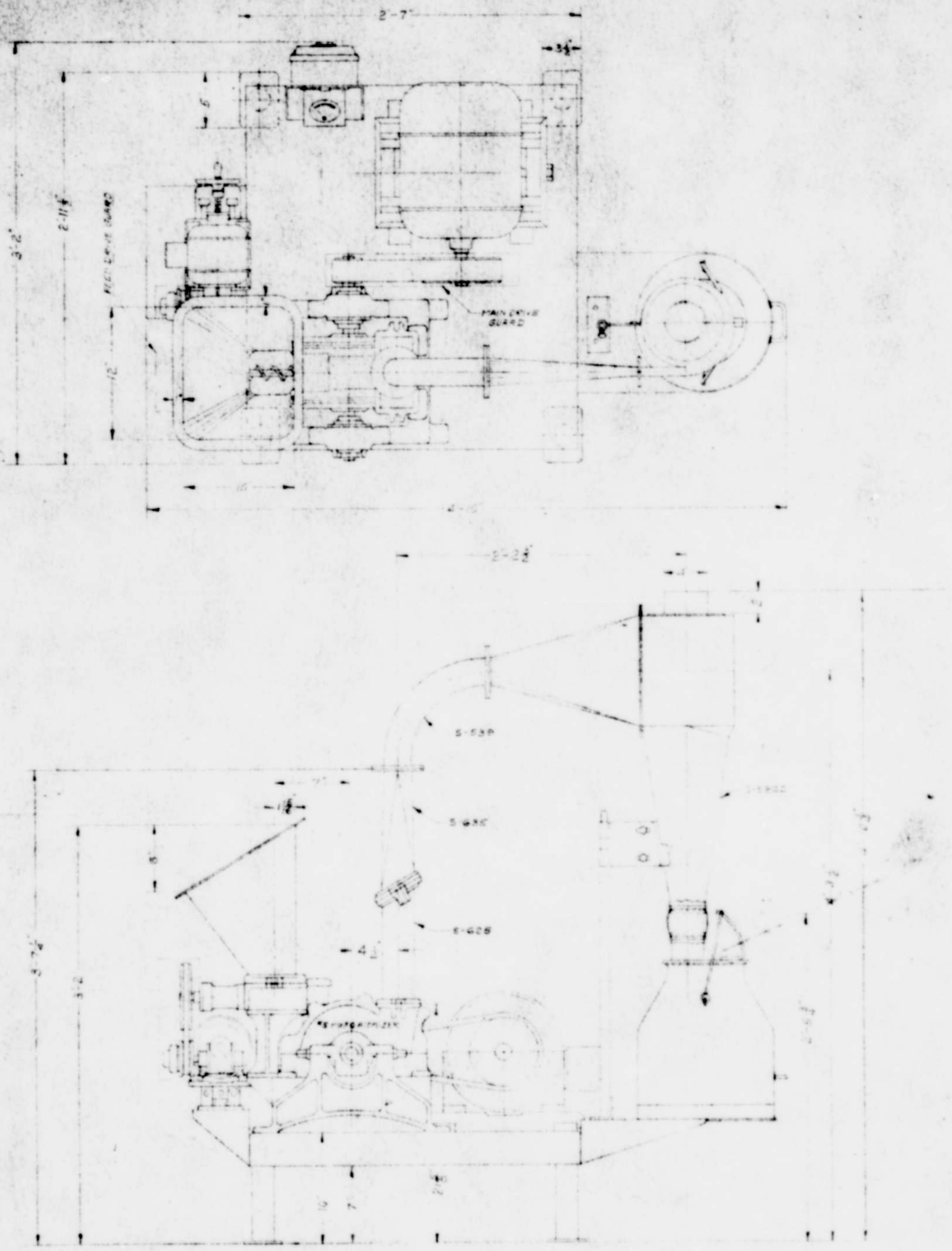
"The contents included in the above quotation are subject to approval by home office."

G. E. Howkinson

G. E. Howkinson
Representative

SEE OVER FOR CONDITIONS OF SALE

8M-8-7-2-58



PULVERIZING MACHINERY CO.
SUMMIT, N.J.

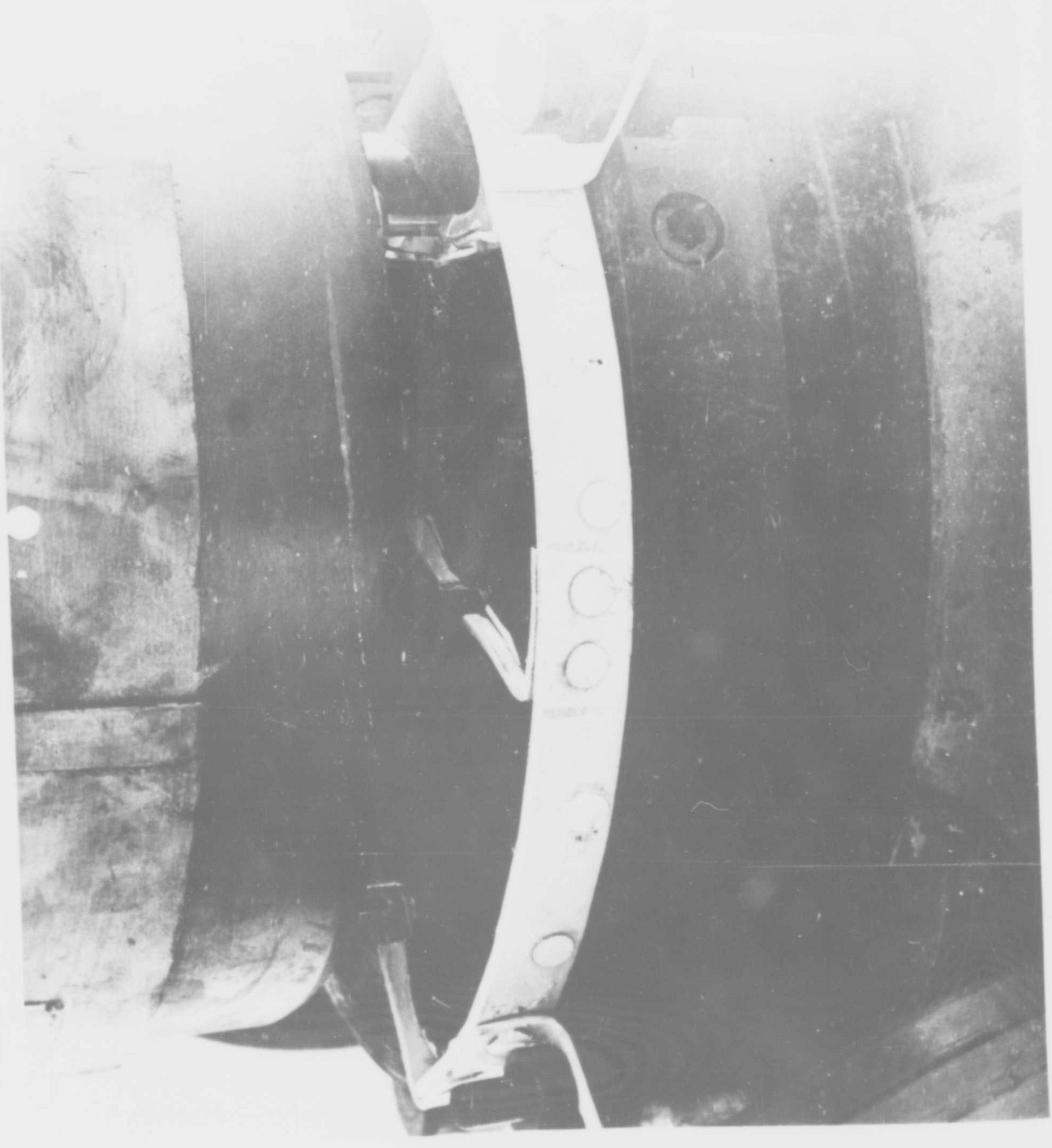
ARRANGEMENT OF #5 MIKRO-ATOMIZER
AND CYCLONE

DIMENSIONS ARE APPROXIMATE	9-6-50		S-672

THIS PRINT FOR REFERENCE ONLY UNLESS CERTIFIED

APPENDIX "B"

Wiper Assembly for Stokes Tabletizer



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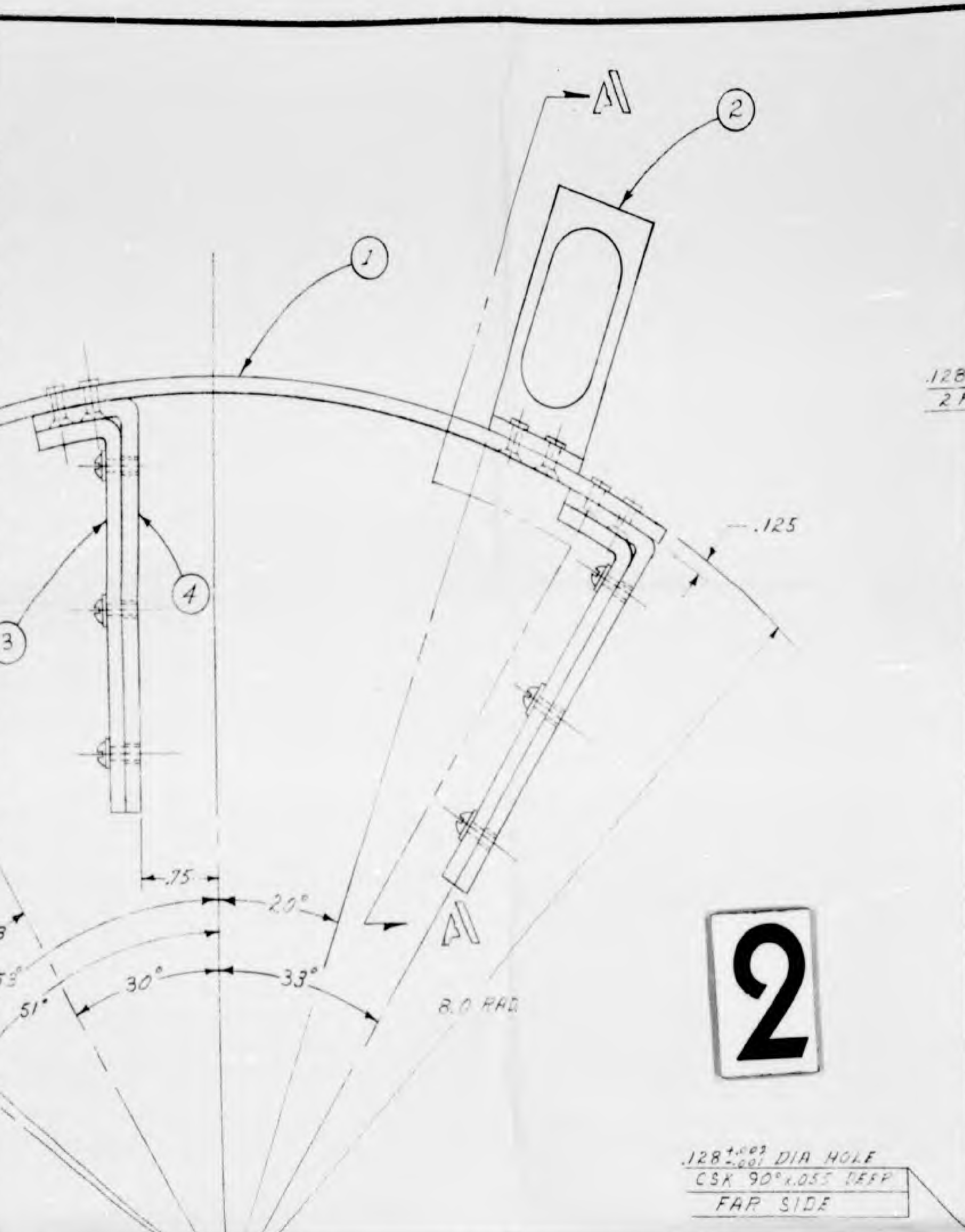
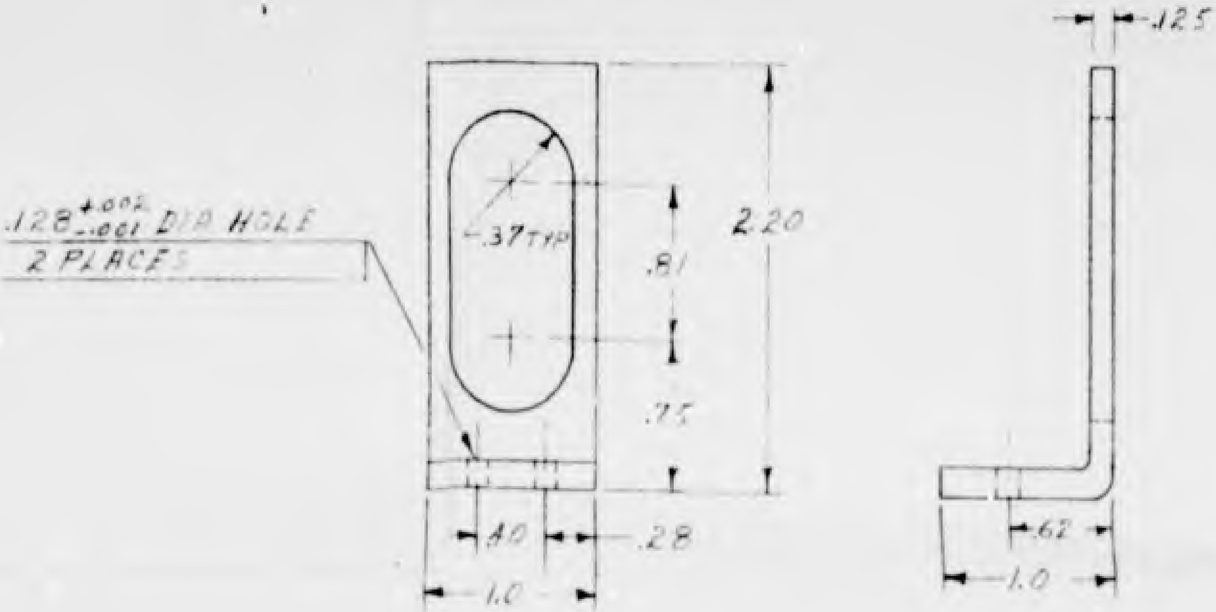
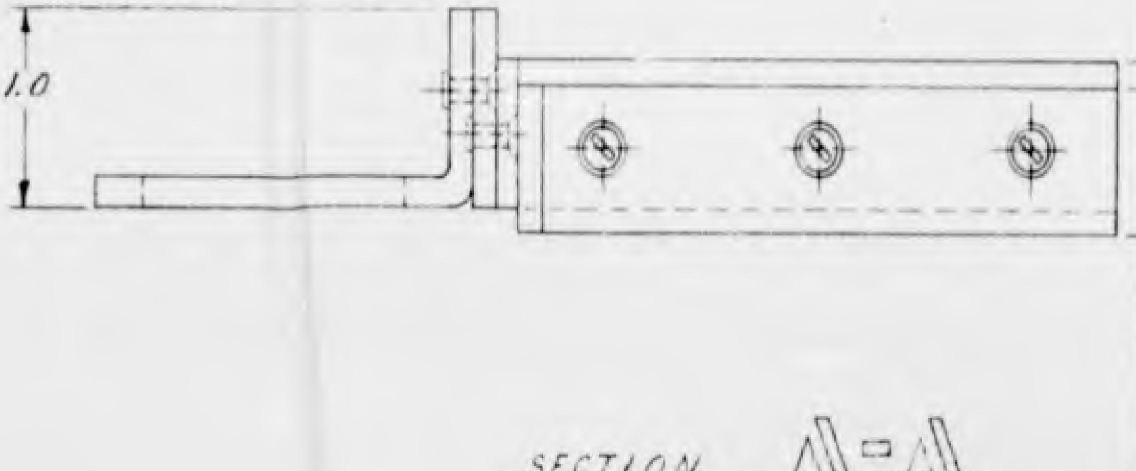

A
2
1
.128
.125
4
3
.75
20°
53°
51°
30°
33°
8.0 RAD.
A
2
.128 +.003 -.001 DIA HOLE
CSK 90° x .055 DEEP
FAR SIDE

3



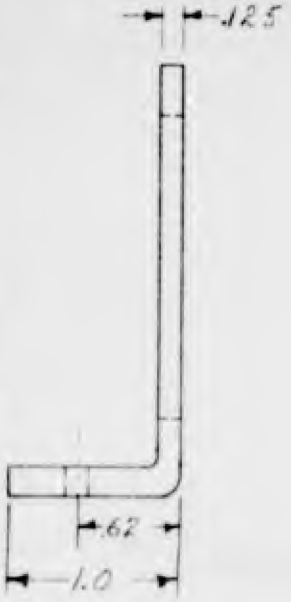
DETAIL 2
TWO REQD

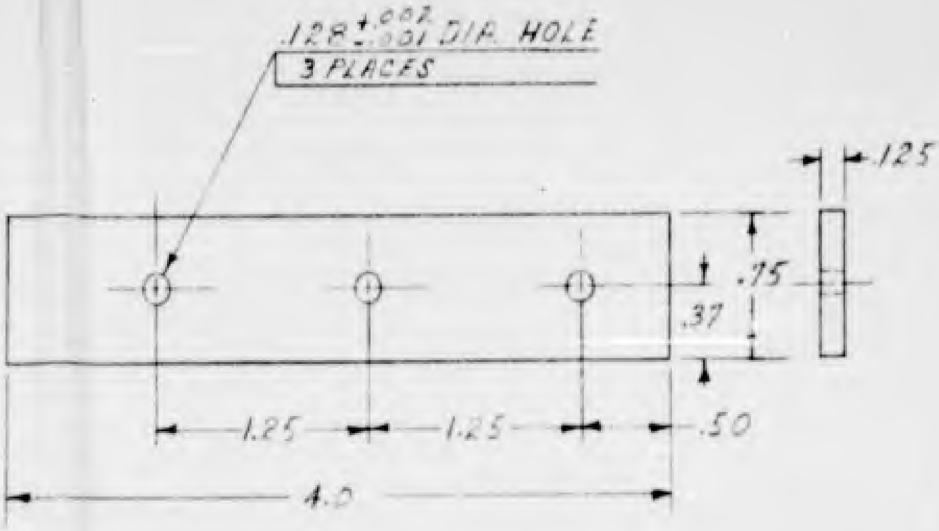


SECTION A-A

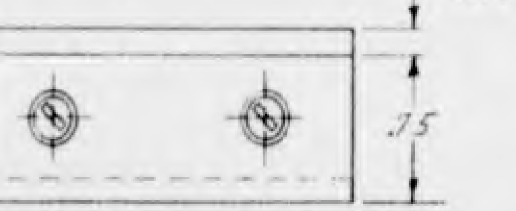
REVISIONS

SYM.	DESCRIPTION	DATE	APPROVAL





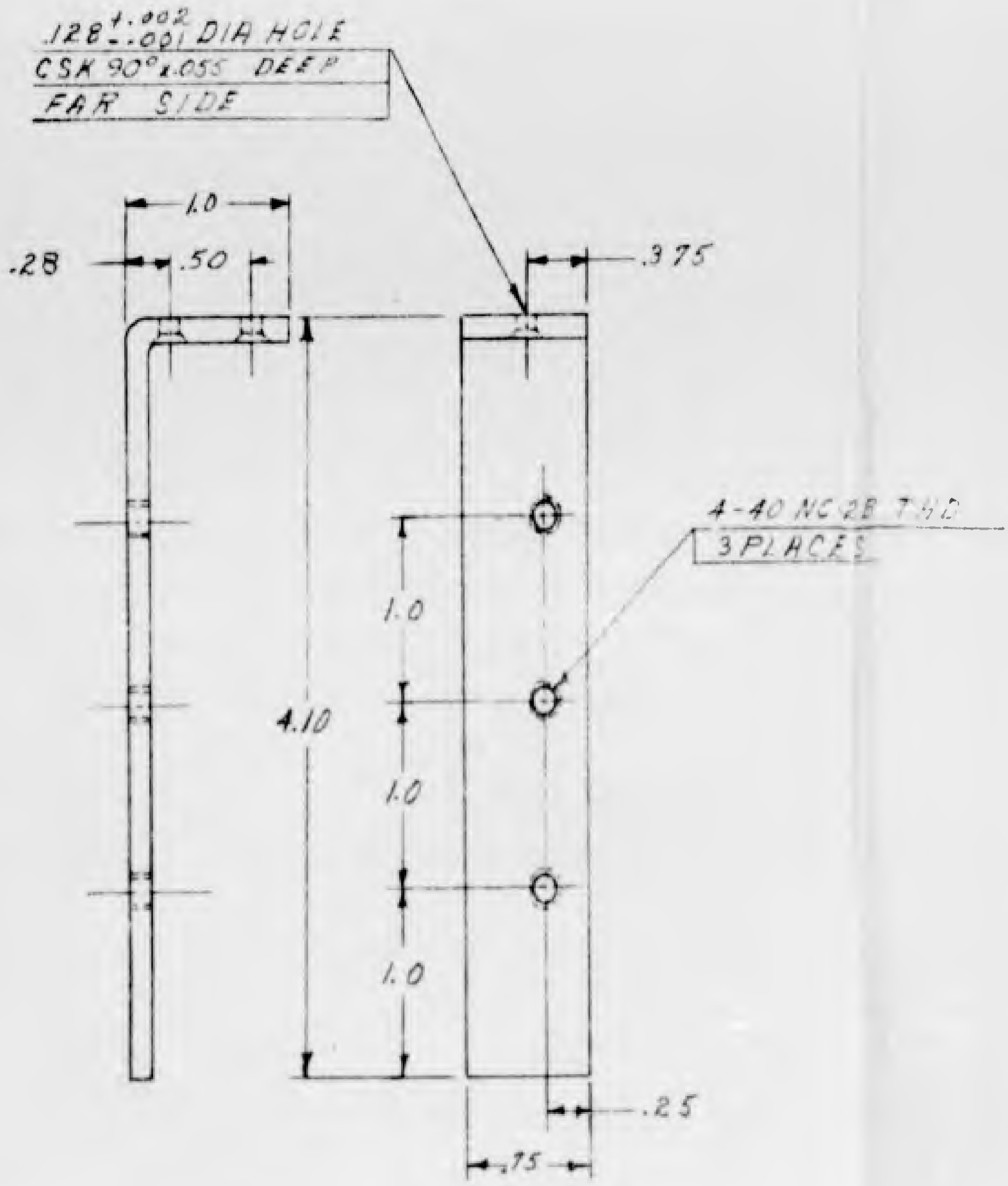
DETAIL 3
THREE REQD



A-A

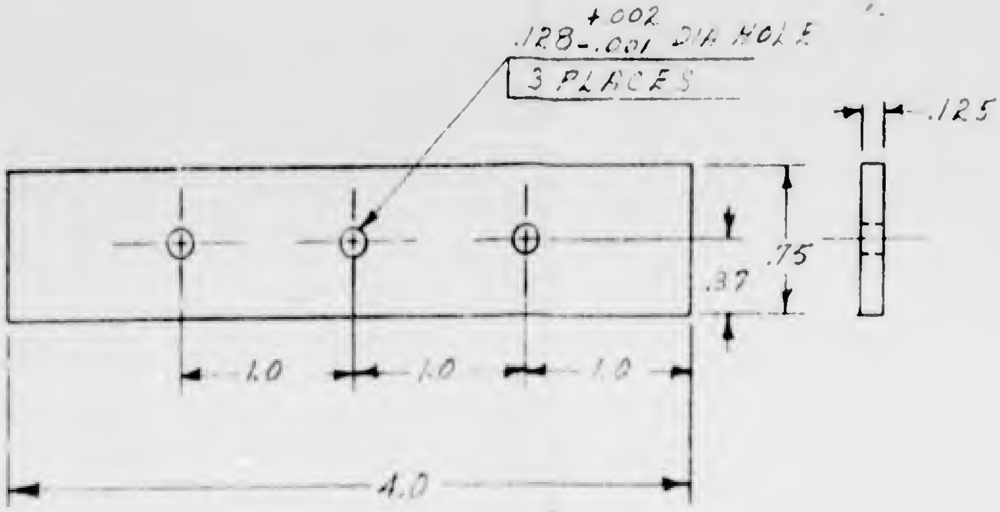
4

(6)



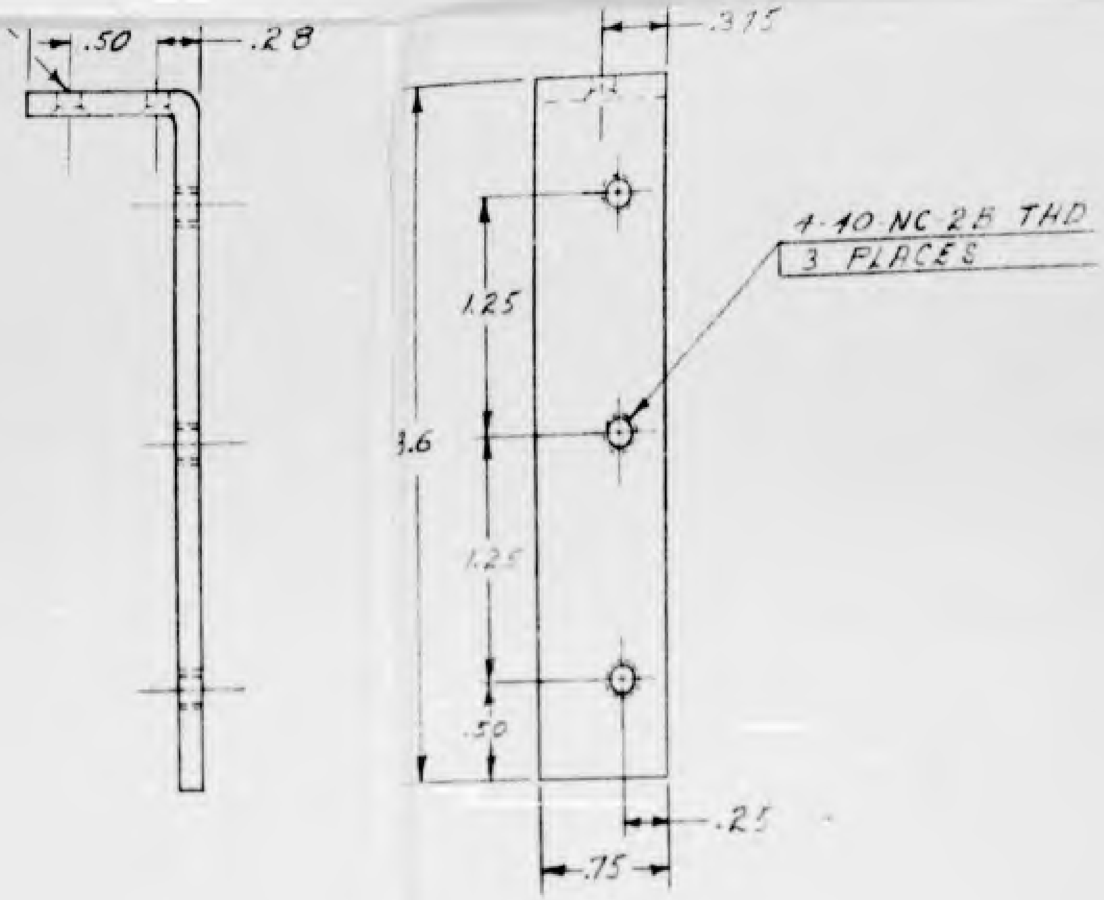
DETAIL 5
ONE REQ'D

5



DETAIL 6
ONE REQ'D

6



DETAIL 4

THREE REQ'D

7

9	12	RIVET
8	12	WASHE
7	12	SCREW
6	1	WIPE
5	1	ARM
4	3	ARM
3	3	WIPE
2	2	DOG
1	1	RIM
DET	QTY.	NAM

NO. REQ'D	NEXT ASSEMBLY

UNLESS OTHERWISE SPECIFIED

TOLERANCES:
.X = ± .1
.XX = ± .03 ANGLES ± ½°
.XXX = ± .010
ALL DIMENSIONS IN INCHES
BREAK ALL SHARP EDGES .005 X .005 (MAX.)
REMOVE ALL BURRS | DO NOT SCALE DWG.

MATL. SPECIFICATIONS:
YELLOW BRASS HALF HARD
ASTM B36-52 ALLOY NO.8

DRAWN	R. SEYBERT	7-13
TRACED		
CHECK	D. JENSEN	7
STRESS		
GROUP LEADER		
PROJECT ENGR.		
APPR.		
APPR.		

-40 NC-2B THD
PLACES

8

DRAWING NO.
157-3
REV.

9	12	RIVETS	AN-420-C4-6 COPPER COUNTERSUNK HEAD
8	12	WASHERS	#4 PLAIN
7	12	SCREWS	#4-40 1/4 LG. ROUND HEAD MACHINE
6	1	WIPER	RUBBER
5	1	ARM	BRASS
4	3	ARM	BRASS
3	3	WIPER	RUBBER
2	2	DOG	BRASS
1	1	RIM	BRASS
DET	QTY.	NAME	MAT'L

BILL OF MATERIALS

SPECIFIED
NGLES ± 1/2°
NCHES
.005 X .005 (MAX.)
NOT SCALE DWG.
S:
S HALF HARD
ALLOY NO.8

DRAWN	R. SEYBERT	DATE 7-13-59
TRACED		
CHECK	D.JENSEN	7-13-59
STRESS		
GROUP LEADER		
PROJECT ENGR.		
APPR.		
APPR.		

TITLE: WIPER ASS'Y

SCALE 1/1
WT.

PROPELLEX
CHEMICAL CORP.
EDWARDSVILLE, ILLINOIS

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DRAWING NO. 157-3
REV.

SHEET 1 OF 1 SHEETS

APPENDIX "C"

Picatinny Arsenal Ballistic Data

ORDNANCE CORPS
PICATINNY ARSENAL
DOVER, NEW JERSEY

Mr. JCussen/ef/1257

IN REPLY
REFER TO
INDUSTRIAL ENGINEERING DIVISION
ORDBB- DR1

Propellex Chemical Corporation
P. O. Box 187
Edwardsville, Illinois

Attention: Mr. [illegible]

SUBJECT: Contract No. DA-[illegible]-072-5[illegible]1-ORD-(P)-[illegible]

Gentlemen:

Reference is made to the black powder sample submitted by your organization to Picatinny Arsenal for evaluation. Results obtained with the sample as compared to standard black powder in closed bomb tests are as listed:

	Standard Sample		Propellex Sample	
	dp/dt (PSI/SEC)	P(PSI)	dp/dt (PSI/SEC)	P(PSI)
	58.5×10^3	650	59.4×10^3	650
	$1.0[illegible] \times 10^5$	1300	1.09×10^5	1300
	1.45×10^5	1900	1.40×10^5	1900
Maximum Pressure	4740 PSI		4470 PSI	
Relative Quickness	100		102	
Relative Force	100		94.2	
Ignition Delay	55 microseconds		56.8 microseconds	

Relative quickness is defined as the ratio of the average rate (dp/dt) at which pressure changes as compared to the standard. Relative force is the ratio of the maximum pressure of the test sample as compared to the standard. Ignition delay is a measure of the time interval between initial ignition and the first indication that the sample is burning. It would appear based upon these preliminary results, that your sample is similar with respect to burning characteristics to standard black powder.

FOR THE COMMANDER:

Sincerely yours,

S Fleischn

S. FLEISCHNICK
Assistant

RAC, 157 file

ORDNANCE CORPS
PICATINNY ARSENAL
DOVER, NEW JERSEY

Mr. JCussen/ef/4267

IN REPLY
REFER TO:
INDUSTRIAL ENGINEERING DIVISION
ORDBB- DB1

Propellex
MAR 9 1953
RECEIVED

55 -8 AM

Propellex Chemical Corporation
P. O. Box 187
Edwardsville, Illinois

ATTENTION: Mr. T. N. Scortia

SUBJECT: Contract No. DA-23-072-501-ORD-(P)-43

Gentlemen:

Listed below are the results obtained on evaluation of the second sample of black powder submitted to this Arsenal:

	Standard Black Powder		Propellex Black Powder
	dp/dt (psi/sec)	psi	dp/dt (psi/sec)
	45.1×10^3	650	81.6×10^3
	$70. \times 10^3$	1300	1.39×10^5
	88.4×10^3	1950	1.85×10^5
	1.035×10^5	2600	2.14×10^5
Maximum Pressure	4600 psi		4600 psi
Relative Quickness	100		205
Relative Force	100		99.9
Ignition Delay	49.2 microseconds		19.6 microseconds
Total Volatiles			0.08%
Specific Gravity			1.42

Of the results, relative quickness, and specific gravity are not considered acceptable. It is felt that the fine particle size charcoal and potassium nitrate, which would produce a porous product causing rapid burning and low specific gravity, are responsible for the poor results. The low ignition delay, which is considered desirable, may also be attributed to this factor.

FOR THE COMMANDER:

Sincerely yours,

L. Saffian

L. SAFFIAN
Assistant

ORDNANCE CORPS
PICATINNY ARSENAL
DOVER, NEW JERSEY

Mr. JCussen/ef/4267

IN REPLY
REFER TO:
Industrial Engineering Division
ORDBB-DB1

Propellex Chemical Corporation
P. O. Box 187
Edwardsville, Illinois

ATTENTION: Mr. T. W. Scortia

SUBJECT: Contract No. DA-23-072-501-ORD-(P)-43

Gentlemen:

Inclosed are results obtained on two samples of black powder submitted by you to this Arsenal for evaluation. It is felt that the poor burning characteristics of the experimental lots were caused by the smaller surface area of pellets as opposed to the irregular granular type powder.

FOR THE COMMANDER:

1 Incl
1. Table

TABLE I

		du Pont Lot	Egyptian Lot	Propellex Lot	
Lab. Analysis	Spec. Req't	#30-139	#8-1	#1	#2
Moisture %	Max. 0.70	0.50	0.32	0.31	0.38
Sp. Gravity	1.72 - 1.77	1.74	1.74	1.77	1.66* 1.67*
Potassium Nitrate %	74.0	73.5	74.22	73.75	73.9
Charcoal %	15.6	16.34	15.46	15.14	15.98
Sulfur %	10.4	9.94	10.32	10.35	10.13
Ash %	Max. 0.80	0.17	0.51	1.91* 1.83*	0.36
* Fails					
Close Bomb Results					
Max. Press. psi		6101	*5965	5089	5509
Relative Force		104.1	100	90.3	98.0
Rel. Quickness @ 1000 psi		99.4	100	40.3	56.1
" " @ 2000 psi		94.8	100	34.3	41.9
" " @ 3000 psi		90.3	100	28.6	31.0
" " @ 4000 psi		89.8	100	- -	- -
Ignition Delay Milliseconds		18.5	20.3	57.4	35
Burning Time Milliseconds		9.3	7.5	34.2	28.4
Heat of Combustion, cal/gm		1471	1263	1076	1455
Heat of Explosion, cal/gm		673.8	729.6	674.1	603.2
*Standard					
*Static Firing - M-58 Primer					
Ignition Delay Milliseconds		27	57	58	60
Burning Time Milliseconds		17	22	40	53
Max. Pressure psi		16180	13230	6140	2840

*All results in the static firing are the average of 5 tests.

ORDNANCE CORPS
PICATINNY ARSENAL
DOVER, NEW JERSEY

Mr. MBaer/ef/4267

Propellex

IN REPLY
REFER TO:
Industrial Engineering Division
ORDBB-IB1

Propellex Chemical Corp.
P.O. Box 1[illegible]7
Edwardsville, Illinois

ATTENTION: Mr. T. W. Scortia

SUBJECT: Contract No. DA-23-072-501-ORD-(P)-43

Gentlemen:

Inclosed are the results obtained by closed bomb and static firing tests of seven samples of black powder submitted to this Arsenal under subject contract. The results indicate that the experimental samples have a slower relative quickness than standard black powder, which may be attributed to the smaller surface area of pellets as opposed to the irregular granular type material.

The static firing test results for samples 1 and 2 as well as the standards were sent to you by letter dated 15 September 1959.

Since these results complete our testing program for data for inclusion in your final report on subject contract, it would be appreciated if this Arsenal were advised when we may expect a copy for review.

FOR THE COMMANDER:

Yours truly,

S Fleischer

S. FLEISCHER
[illegible] Branch

2 Incl
1. Table I
2. Table II

Table I

Closed Bomb Results

Sample No.	Relative Quickness, % at Pressure, Psi				Relative Force % at Max Pressure	Delay Time Milliseconds	Burning Time Milliseconds
	650	1300	1950	2600			
EGY-8-1	100	100	100	100	100	30.7	11.3
DUP-30-139	99.4	94.8	90.9	89.8	104.1	18.5	9.3
Propellex 1	40.3	34.3	28.6	——	90.3	57.4	34.2
Propellex 2	56.1	41.9	31.0	——	98.0	35.0	28.4
Propellex 3	37.7	29.3	22.8	17.9	86.4	107.0	79.5
Propellex 4	41.2	29.9	20.9	15.3	78.3	119.0	75.0
Propellex 5	47.1	37.9	30.4	23.7	88.5	106.0	73.5
Propellex 6	106.7	71.8	52.9	41.9	95.4	40.5	32.5
Propellex 7	107.3	76.6	55.1	44.0	96.9	50.5	29.5

Incl 1

Table II

Static Firing - M58 Primer

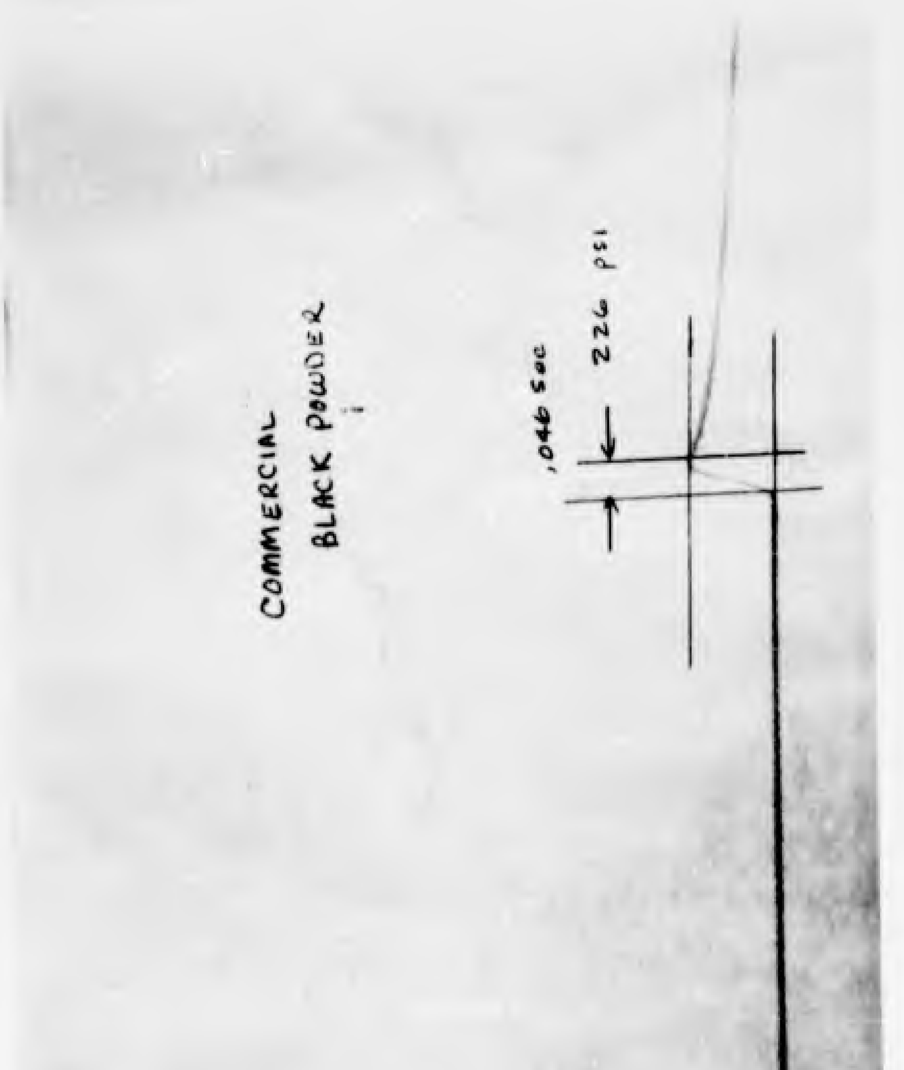
	Sample 3					Sample 4				
	1	2	3	4	5	1	2	3	4	5
Max Pressure, Psi	1905	1786	1279	1786	1709	2175	1243	1476	1398	1320
Burning Time, Sec.	———	.0071	.0067	.0083	.0097	.0050	.0071	.0107	.0079	.0064
Ignition Delay, Sec.	———	.0031	.0075	.0247	.0069	.0061	.0093	.0020	.0093	.0110

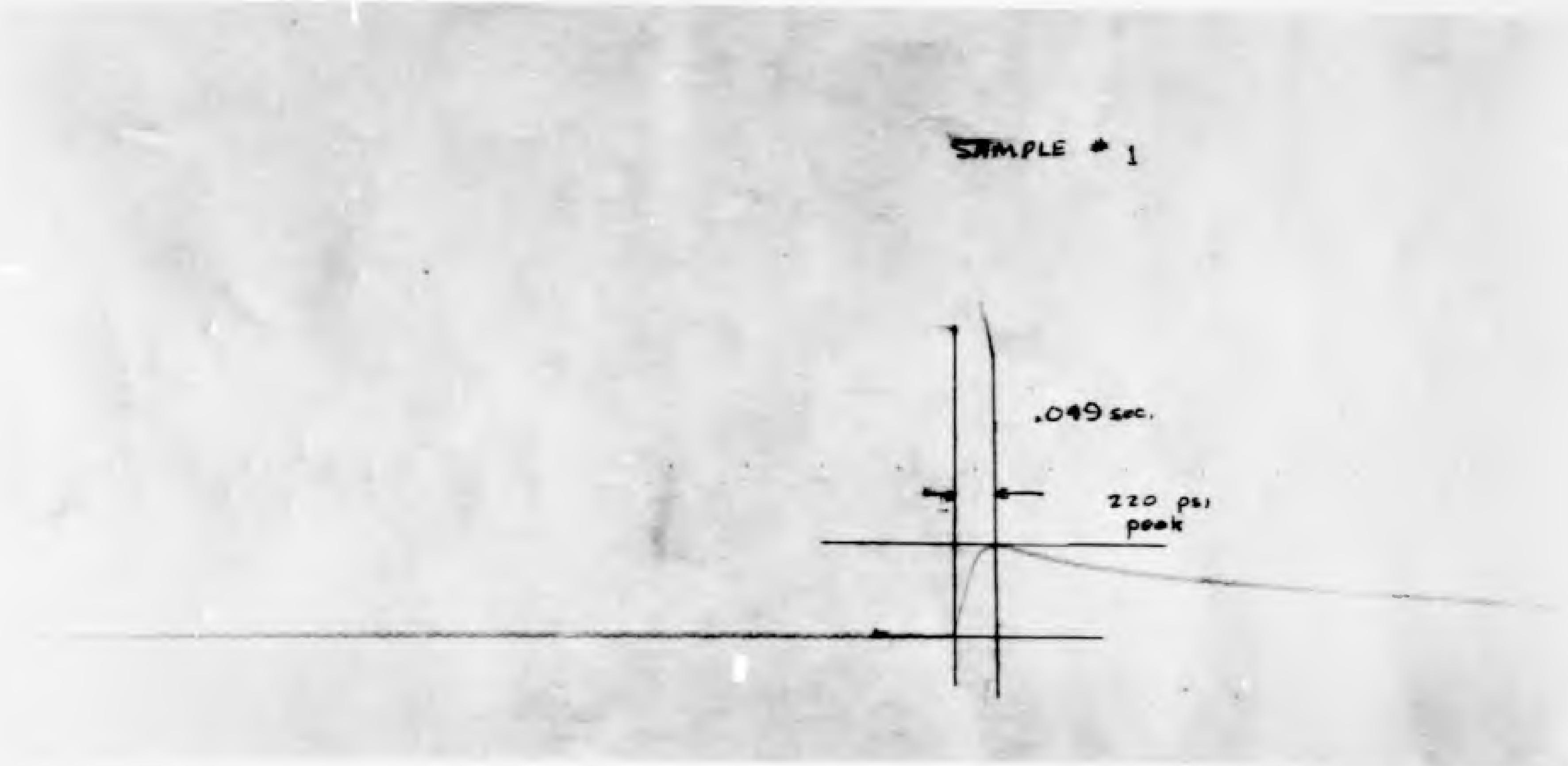
	Sample 5					Sample 6				
	1	2	3	4	5	1	2	3	4	5
Max Pressure, Psi	1864	1398	1786	1631	1631	3961	2019	3884	2330	3262
Burning Time, Sec.	.0064	.0083	.0075	.0079	.0071	.0033	.0045	.0030	.0040	.0031
Ignition Delay, Sec.	.0119	.0026	.0067	.0083	.0151	.0034	.0017	.0036	.0021	.0034

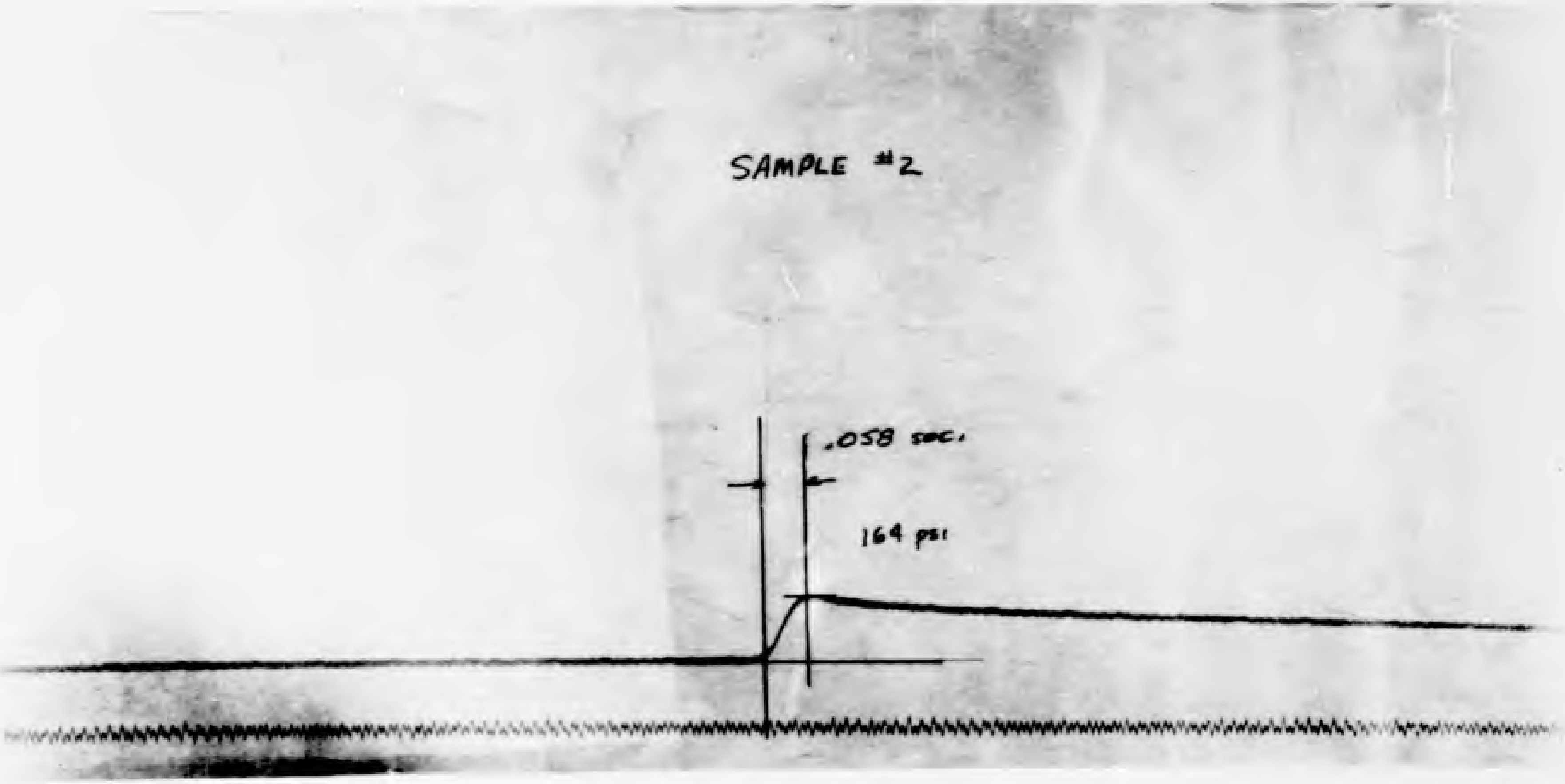
	Sample 7				
	1	2	3	4	5
Max Pressure, Psi	3573	5487	3806	2796	3240
Burning Time, Sec.	.0031	.0028	.0032	.0036	.0036
Ignition Delay, Sec.	.0013	.0023	.0017	.0014	.0025

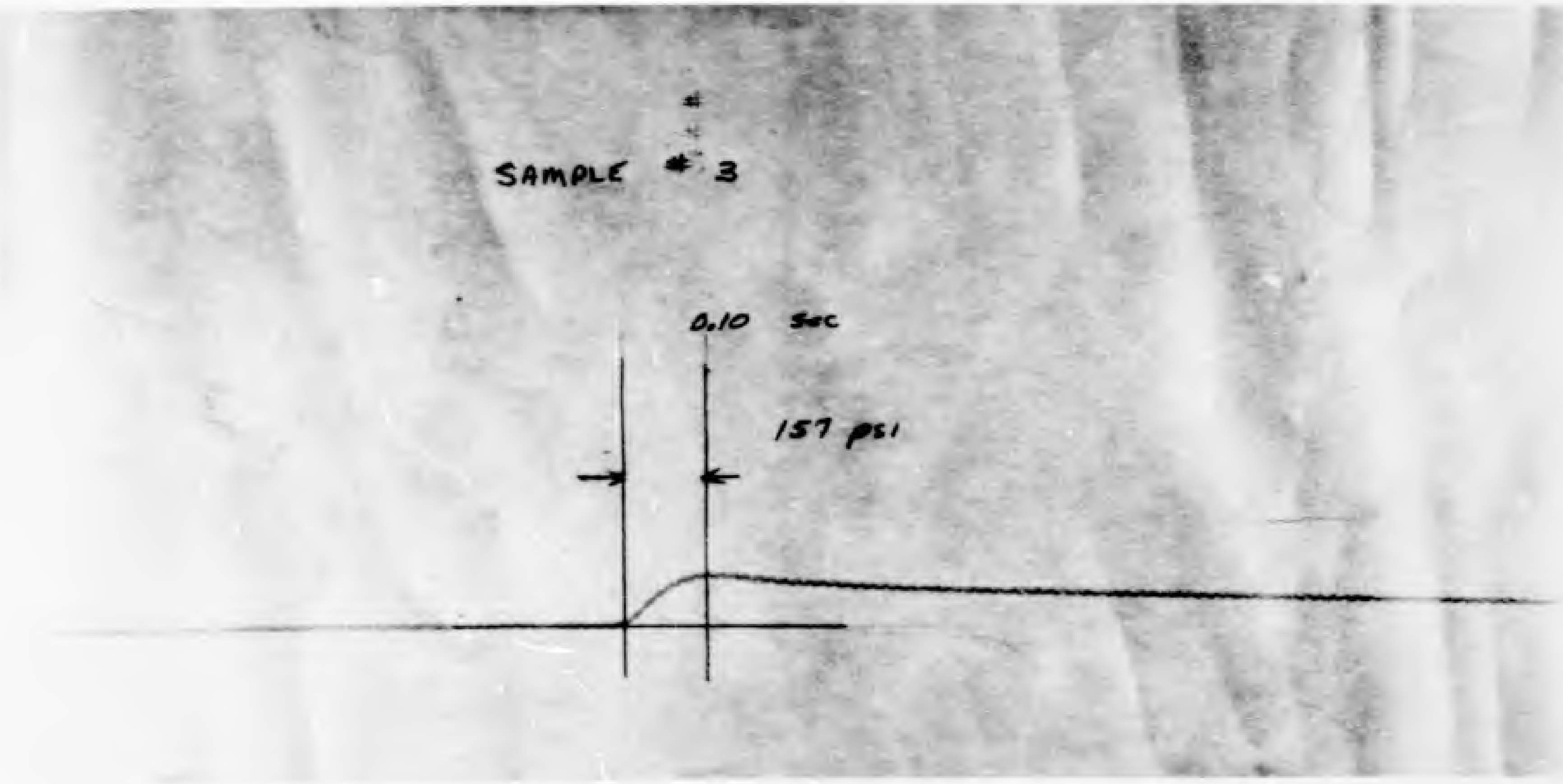
APPENDIX "D"

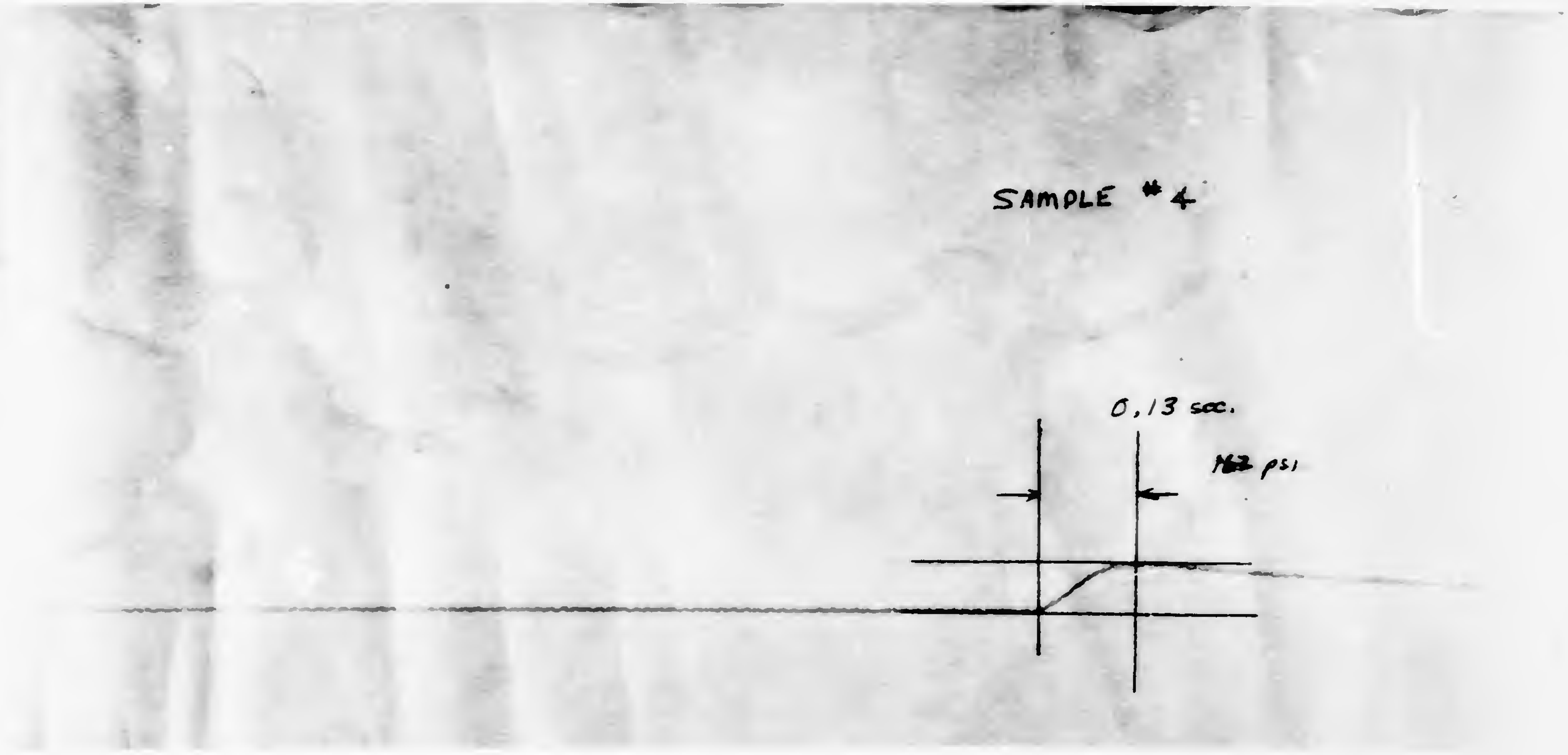
Pressure-Time Traces Low Pressure Ballistic Bomb
Samples PCD #1 through #7

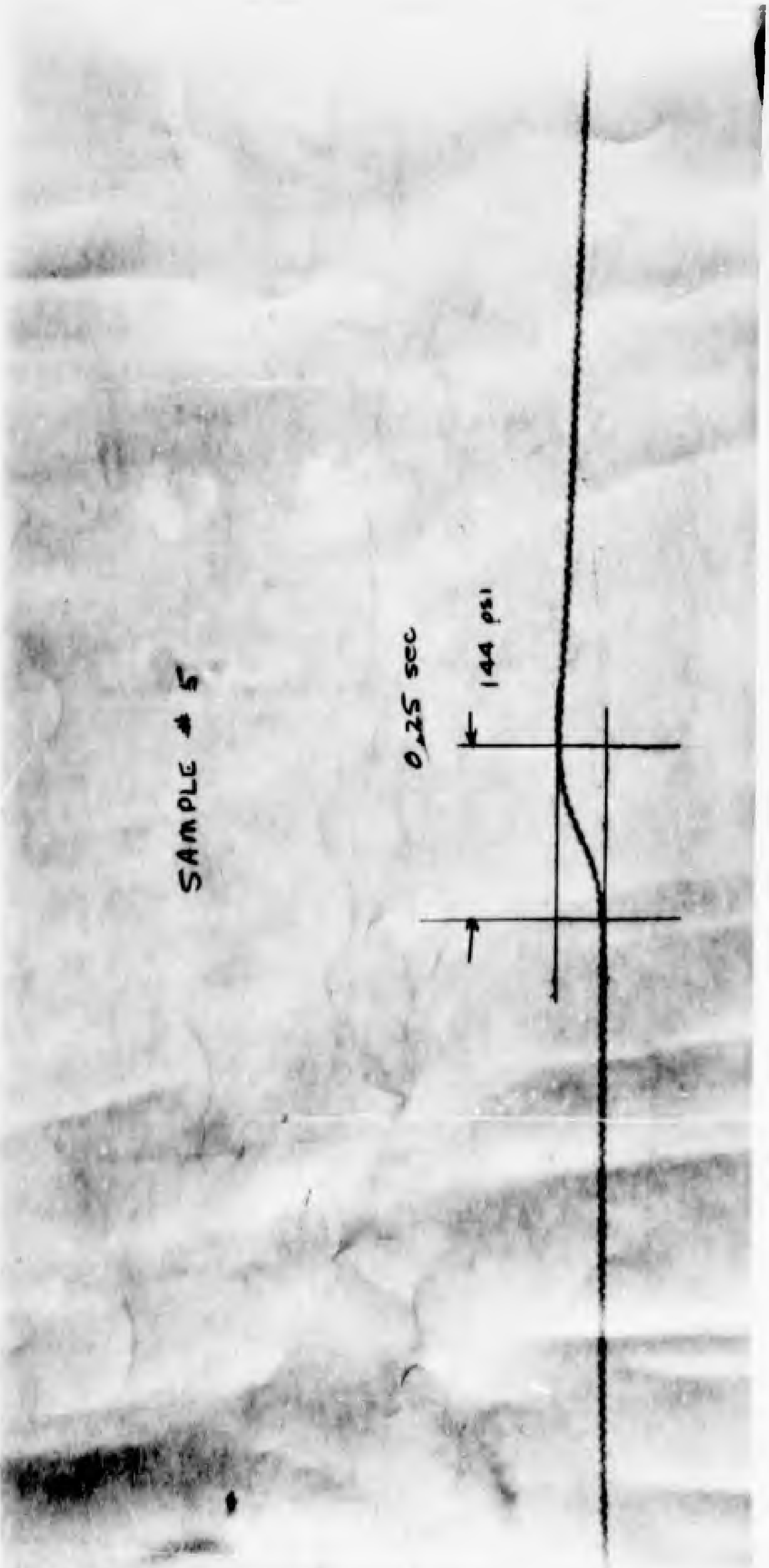

COMMERCIAL
BLACK POWDER
.046 sec
226 psi

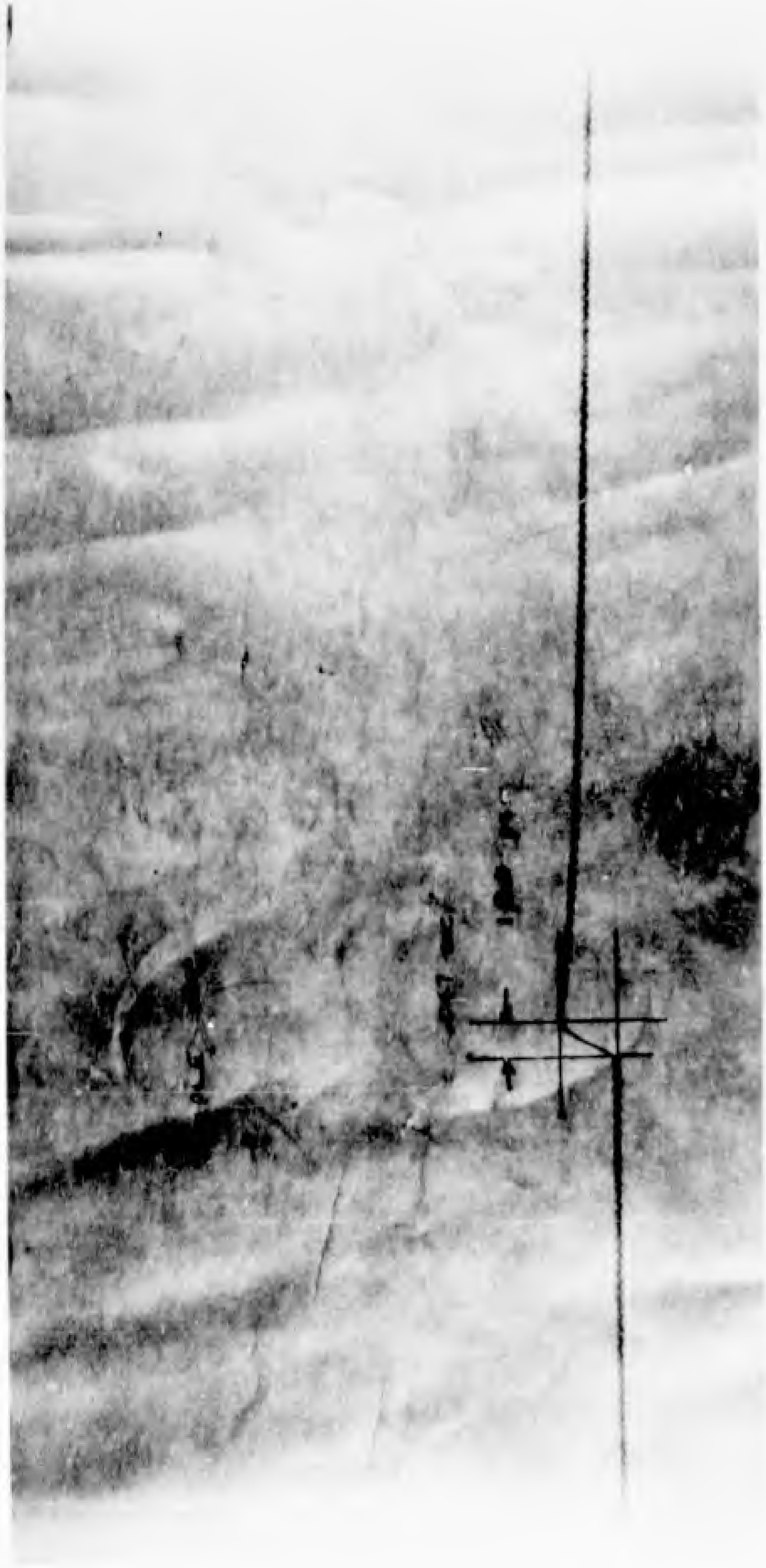

SAMPLE # 1
.049 sec.
220 psi peak

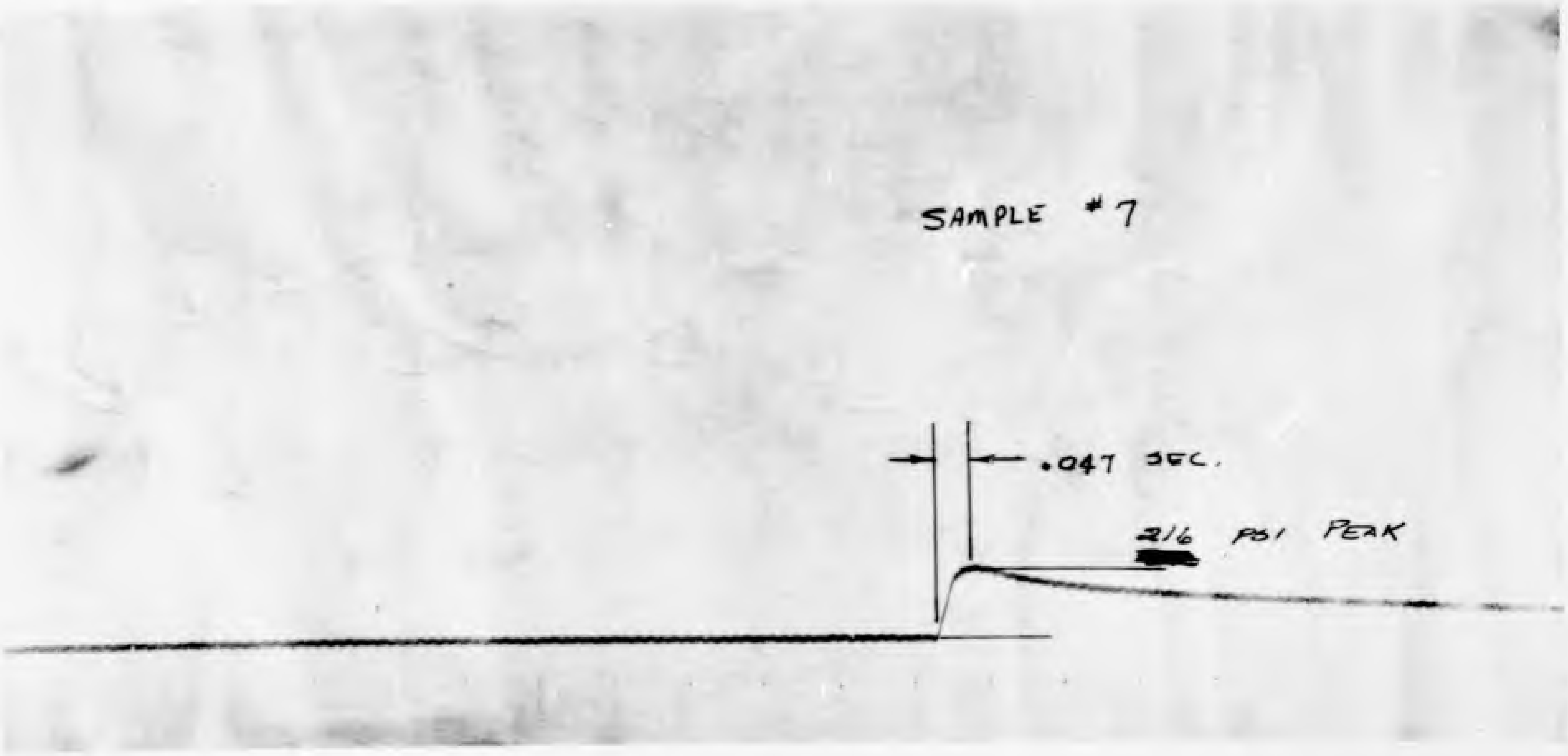

SAMPLE #2
.058 sec.
164 psi


SAMPLE # 3
0.10 sec
157 psi


SAMPLE #4
0,13 sec.
[illegible] psi


SAMPLE # 5
0.25 sec
144 psi




SAMPLE #7
.047 SEC.
216 PSI PEAK

APPENDIX "E"

ALLIS-CHALMERS

COMPACTING PROCESS

ALLIS-CHALMERS

SALES NEWS

Marketing Supplement

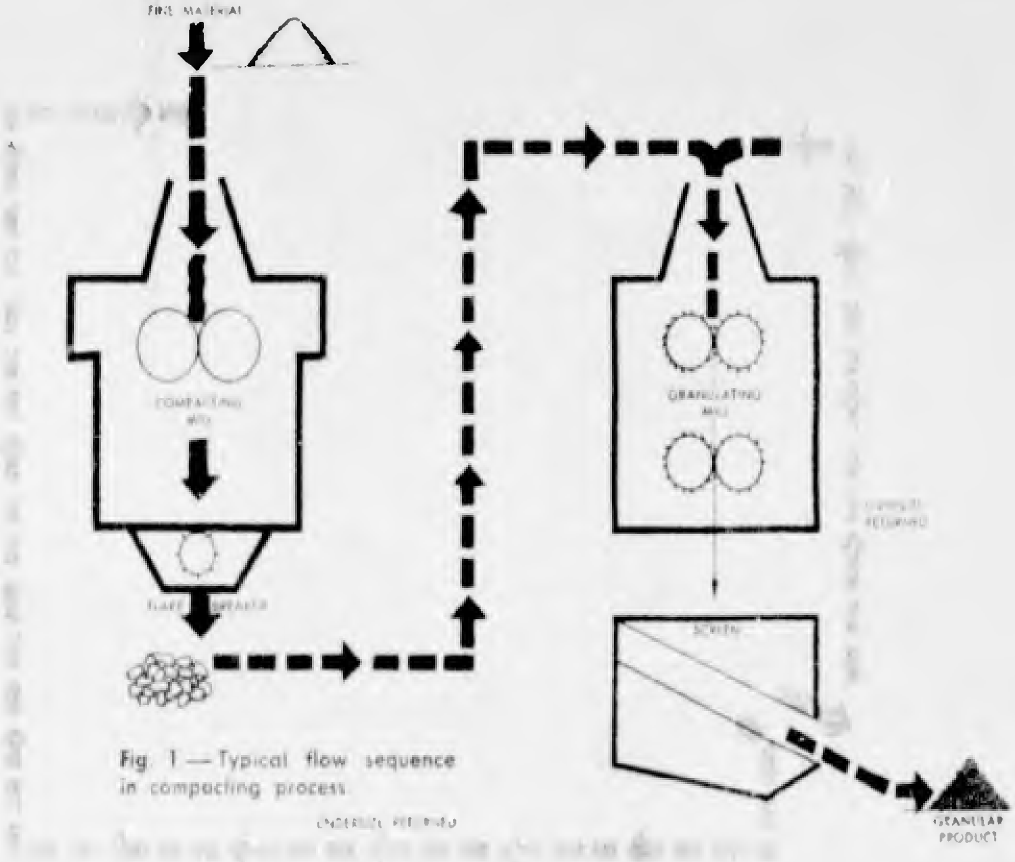


Fig. 1 — Typical flow sequence in compacting process.

APPLICATIONS UNLIMITED

With A-C's COMPACTING PROCESS

The increasing demand for a close control of product specification is being profitably answered by the Allis-Chalmers continuous mechanical compacting process — a process for converting or upgrading particle size, for controlling density and for controlling the solubility factor of chemical salts.

The Allis-Chalmers process has several marked advantages over other processes, including:

1) The Allis-Chalmers process is a simplified, continuous process requiring less equipment, less BTU, lower capital investment — and, produces a more readily marketable product with fewer square feet of floor space.

2) Operating and maintenance costs are comparatively low.

3) A controlled process that produces a stable product.

4) Less power is required.

5) Varying degrees of granulation can be obtained simply by granulator roll adjustment.

6) When necessary, additives (binding agents) can be added without disturbing production rate of process.

7) Products of varying densities can be readily produced.

8) Compacting rolls are smooth . . require less costly and less frequent maintenance.

Processing Machinery Department

Fig. 2 — Feed or fine material.

The application for this process or for the equipment used individually, is unlimited. Any producer of organic or inorganic salts is a possible user. The process has been successfully demonstrated for upgrading and densifying tile mix, soda ash, sodium tripoly phosphate, and milk mix. The process has been used successfully for upgrading salt, sodium meta silicate, sodium nitrite, "Methionine", organics, fertilizer mix, lactose, coal, dextrose, cement, urea, calcium phosphate, cyanamid and flake caustic. The process has also been used successfully in densifying "Daldow", cellulose acetate, detergent, titanium oxide, aluminum powder, "Pentek" ($C(CH_2OH)$, used in paint, resins), plastic, sterate, calcium gluconate, mining reagent, and drugs.

The most active users at present are producers of such fertilizer salts as ammonium sulphate, diammonium phosphate and potash. The primary reason is that the fertilizer salt manufacturers have a marketing problem brought about by the increased production of nitrogen bearing fertilizers produced by the petro-chemical plants (i.e. prilled urea).

A typical Allis-Chalmers compacting process flow is shown in Fig. 1. The feed material (Fig. 2) can be organic or inorganic material. Inverted, gravity type feed hopper prevents bridging and maintains the necessary positive head of feed to compactor (Fig. 3). Smooth faced compactor rolls pick up the feed, apply a rolling pressure, produce a continuous compacted sheet, full width of the rolls. As the continuous sheet comes from the compacting mill it is broken into pieces (Fig. 4) by the flake breaker. Pieces measure approximately ¾ x ¾ x compacted sheet or flake thickness to provide proper feed for the granulator (Style "N" granulating roller mill).

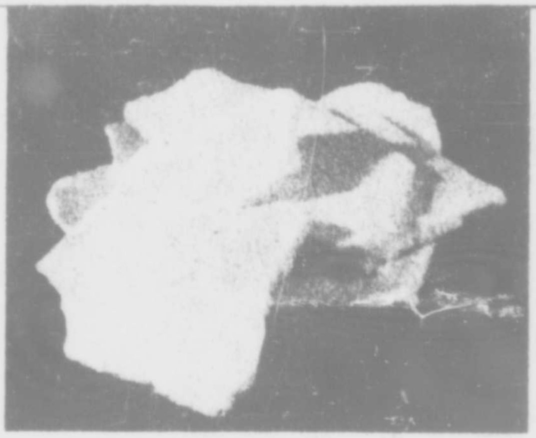


Fig. 4 — Compacted flakes broken by flake breaker produced desired granulating mill feed.

At this point in processing, some materials require drying or curing (either natural or forced) to relieve internal molecular stresses, to harden and strengthen flake character in order to obtain the greatest overall production from the granulator.

The Allis-Chalmers granulator (Style "N" granulating roller mill) (Fig. 5) may have one, two or three pair of rolls, the number of rolls and type of roll corrugations are dependent on the final product desired. The principle of corrugated rolls and gradual reduction is a big factor in obtaining high product yield. Following the granulator, the product is screened on an Allis-Chalmers gyrating screen (Fig. 6) or vibrating screen (Fig. 7).

From the screen, all fines are recirculated to the compactor, all oversize is recirculated to the granulator and all desired product (Fig. 8) sent to stock or to packing. In the case of ammonium sulphate, pilot plant work has indicated that starting with the original ammonium sulphate crystal feed (all passing 30 mesh) a 60 to 70 percent yield of 6 x 30 mesh granulated material could be expected.

A successful compacting process involves: (1) proper feed, (2) ample compression force, (3) low power requirements, (4) good capacity per unit of capital investment.

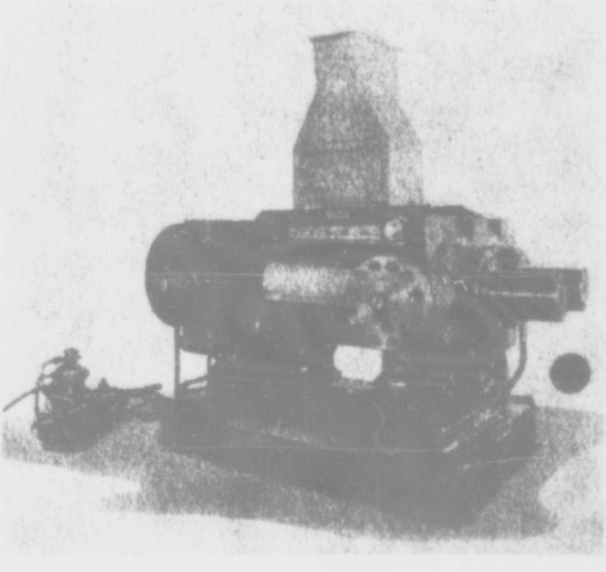


Fig. 3 — The heart of the process is the compacting mill. Compacting rolls are cast iron face with steel shafts (depending on application requirements). They may be equipped with either antifriction or sleeve bearings. In the case of antifriction bearings, they may be water cooled and equipped with the circulating water system. Roll pressure is regulated by hydraulic adjustment. Prestress force is exerted through the front thrust bar to hold the roll in a fixed predetermined position. The hydraulic pressure mechanism is shown to the left in the above photo. Gravity feed type hopper and fabricated discharge hopper is also furnished.

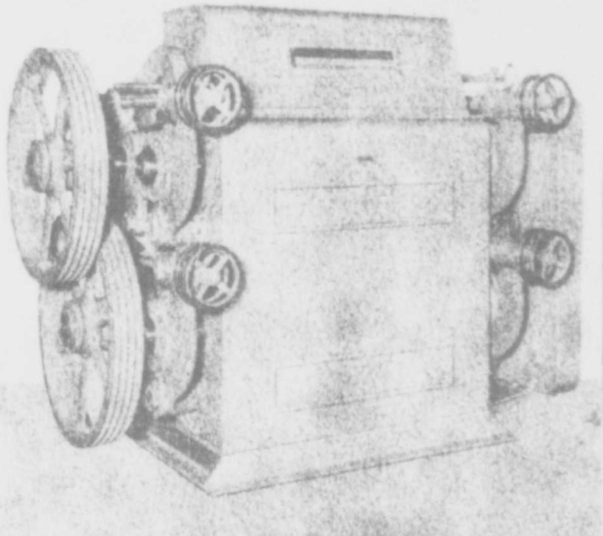


Fig. 5 — Granulating roller mills are equipped with centrifugally cast circle chill grinding rolls, antifriction bearings, and a roller chain differential as described in Bulletin 06S8056. They may be equipped with either roll or vibrating type feeder. The unit shown above is a two-stage unit. Other models are one- and three-stage units.

Feed

Feed factors involve: (1) Particle size. (2) Moisture content. (3) Temperature of material. (4) Specific gravity. (5) Binding agents.

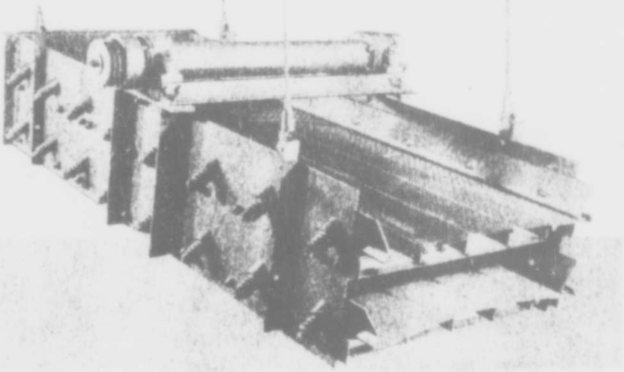
Usually materials will compact more readily and produce a higher quality of material if the material is fed to the compactor at elevated temperatures. Compacting rolls can be heated to assist in the compacting process. Generally, some moisture addition is desirable to aid in the compacting of the material. The advantage of proper moisture is to lower the bearing load requirements, thereby facilitating a higher feed rate. Binding agents may or may not be added, depending on the character of feed crystals. The particle size of the feed material will affect the feed rate and capacity of the compacting unit. For example: A wide range of particle sizes in the feed material is more desirable than a closely graded feed material. The largest particle which can be introduced is determined by the hardness of the materials and the angle of nip of the compacting rolls. In general, the larger the particle size to start with, the higher the capacity.

Compression Forces

A-C compacting mills are supplied with bearing capacities of 9,000, 80,000 and 150,000 maximum pounds of force. Units are classified in the following types of operation: (1) High bearing load, (2) medium bearing load, (3) low bearing load.

The low bearing load unit (9,000 lbs) has 10-in. diameter by 42-in. long contact rolls. It is capable of producing a separating force of 428 lbs. per lineal inch. On *special* limited load applications, A-C can furnish a compactor-granulator, (Fig. 9) where two pair of granulating rolls (10-in. diameter by 42-in. long) are installed below the compactor rolls in the same frame.

The medium bearing load unit (80,000 lbs) has 20-in. diameter by 24-in. long contact rolls with a 20-in. working face. It is capable of producing a separating force of 8,000 lbs per inch. This unit can be applied on some organic or inorganic compounds. Its field of application is much broader than the low bearing load unit, but not as broad as the high bearing load unit.



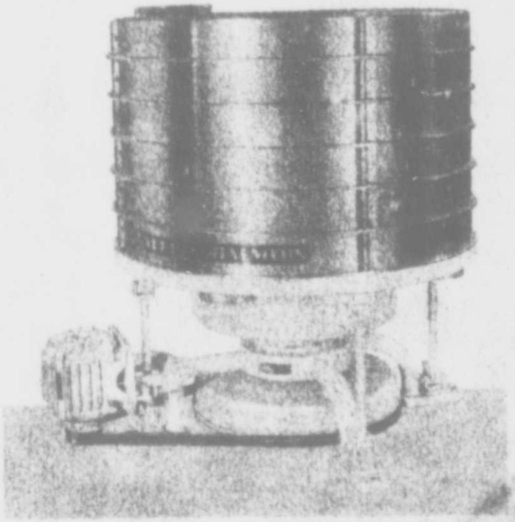


Fig. 6 & Fig. 7 — Two types of screens covering a complete range of sizes and capacities are available. Fig. 6 (right) shows a stainless steel gyratory screen model SS-1 and is described in Bulletin 07B8446. Gyratory screens, Model CS-1 (Bulletin 07B7625) and Models LB and HB (Bulletin 07B6124) are also available. Fig. 7 (left) shows the widely used ***Aero-Vibe*** vibrating screen Model AVS as described in Bulletin 07B6099; available with cable suspension or floor mounting.

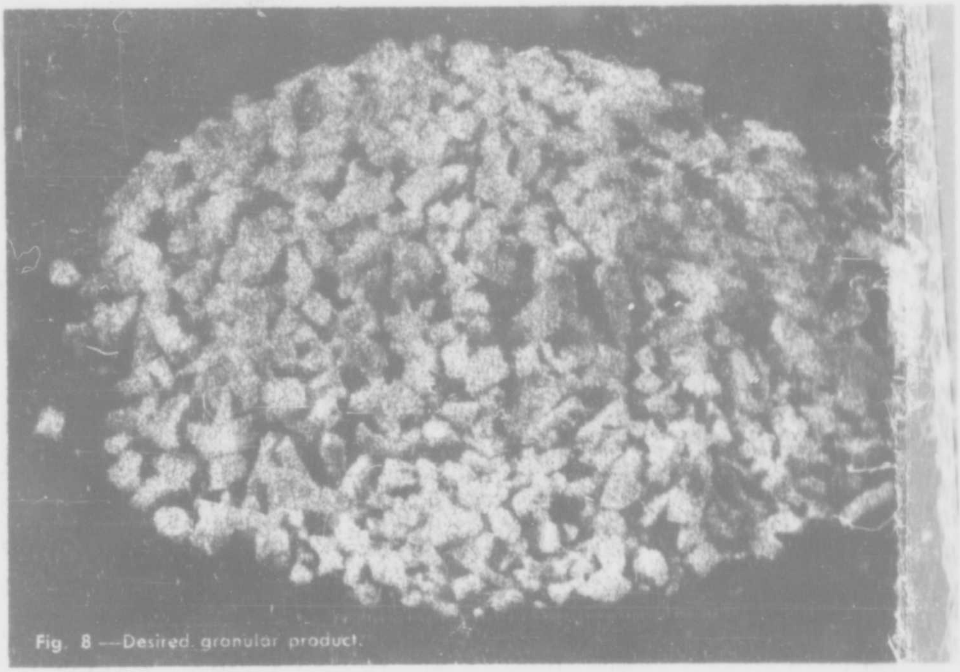


Fig. 8 —Desired granular product.

The heavy bearing load unit (150,000 lbs) has 18-in. diameter by 16, 20, or 24-in. long rolls. This unit can produce separating forces of 18,750, 15,000 or 12,500 lbs per inch respectively. This unit is applied to most applications due to its ability to exert the greater force.

Power Requirements

For compacting: The power required depends on the material characteristics as mentioned under feed plus the roll speed, the lineal roll contact, and thickness of flake produced. Compacting machines use from 50 to 100 horsepower. The speed range of the compactors is from 25 to 36 rpm. Lineal contact of the rolls will range from 16 to 42 depending on the bearing force per lineal inch needed to compact particular material.

For granulating: Generally 10 horsepower per pair of rolls is required.

For screening: Horsepower requirements range from 1 to 1½ per unit for gyratory screens and 5 horsepower per unit for vibrating screens.

Note: Maximum connected horsepower for the three-unit process is approximately 150.

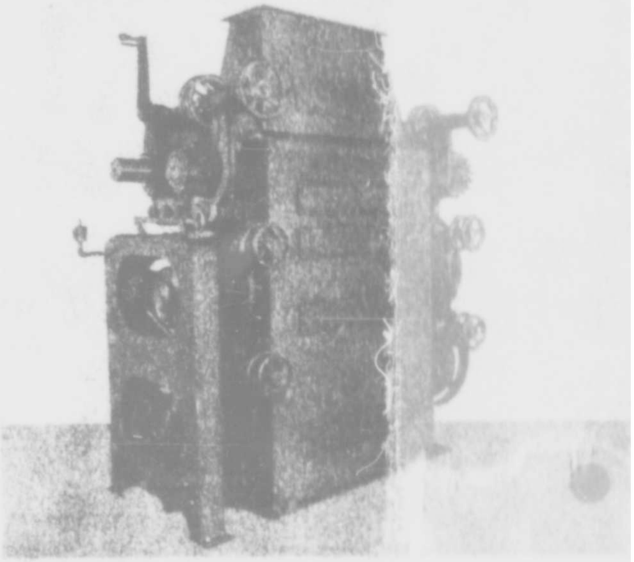
Production Capacity

Compacting mill: Capacity, of course, is dependent on the same condition as the power requirement. Capacity of compacting mills vary from a few tons per hour up to approximately 10 tons per hour.

Granulating mill: The Style "N" granulating roller mill will vary up to approximately six tons per hour depending on the mesh size of product desired.

Screening equipment: Capacity follows closely that of the granulating mills.

Fig. 9 — This compacting-granulating mill, as the name implies, carries on the function of compacting and granulating in one single unit. Compacting rolls are on top; granulating rolls are below in the single frame. This type of unit, naturally, is limited to low bearing load forces and cannot be used where intermediate curing or drying between compacting and granulating is involved.



CHANGES TO FINAL REPORT

Contract DAI-23-072-501-ORD(P)-43

A STUDY OF MODERNIZED TECHNIQUES FOR THE MANUFACTURE OF BLACK POWDER

Please destroy pages 42 through 48 of the subject report and substitute for them the attached pages.

PROPELLEX CHEMICAL DIVISION
CHROMALLOY CORPORATION

APR 27 1960

A consideration of the graph in Figure 9 plus the data in Figure 10 suggests that a powder whose grains of regular geometry is incapable of giving the pressure onset required for the high maximum pressure and low burning times for which the M-58 primer was designed. From a consideration of these data, it becomes more obvious that geometry has a profound influence upon the ballistic performance of black powder. All ballistic traces and primer designs using commercial black powder demonstrate high onset compared to the tabletized black powder developed during the course of this study. It would seem, therefore, that an attempt must be made to evaluate the black powder produced under this contract independently of the influence of geometry upon ballistic characteristics.

To minimize the influence of geometry on ballistic properties in experimental samples, an attempt was made to use a low pressure ballistic bomb for measurements. Traces for commercial black powder and for samples PCD #1 through #7 are given in Appendix "D". The data from these traces are tabulated in Figure 11.

Relative force correlates qualitatively with the same quantity in Figure 8. However, the difference in pressures is more pronounced than with the high pressure ballistic bomb, probably due to the longer burning times involved. (The high pressure bomb usually gives burning times circa 11 msec). With longer burning times, chance for heat transfer to the body of the bomb increases with a consequent drop of pressure.

dP/dt's and relative quickness values were derived graphically for only one pressure: 100 psi. Because of the longer burning time in the low pressure test, it is felt that dP/dt values are more realistic in relation to each other since the values are more sensitive to small fluctuations in onset. The relationship of relative quickness among the samples generally preserves the relationship found in the high pressure bomb.

Samples #1 and #6 have changed their relative positions in the low pressure bomb. Both of these samples were prepared in the same manner, but PCD #1 was more irregular in surface since it was compacted with deformed Stokes punches with round surfaces. One would expect the trace of PCD #1 to be steeper than that of PCD #6. This is not true in the data from Picatinny Arsenal's high pressure bomb but is true in the low pressure data in Figure 11.

Samples PCD #3, #4, and #5 show very low burning rates in both tests. This is significant since sample PCD #3 was processed in the final incorporation and compacting with 50% aqueous alcohol while PCD #4 and #5 were processed using only water as the vehicle. Visual observation indicated the growth of potassium nitrate particles in #4 and #5. The growth of such particles in these samples and in PCD #3 would explain the lowered burning rates.

PCD "A", however, was processed with water in the final incorporation stage after sulfur had been added via a carbon disulfide vehicle. Here no crystal growth was observed and resulting ballistic properties of PCD "A" were very close to commercial powder. We may assume that the KNO_3 in PCD "A" remained essentially below mesh 200. (Below 74 microns).

The KNO_3 used in samples PCD #1, PCD #6, and PCD "B" was nominally 6-8 micron (the average particle size with a maximum particle size of 35 microns).

The charcoal used in PCD "A" was mesh 325 (less than 44 microns) while later charcoals were often 5-6 microns average size with a maximum particle size of 40 microns. The use of either did not noticeably affect the ballistic data from Picatinny Arsenal. The same statement may be made with reference to the use of mesh #200 KNO_3 versus 6-8 micron KNO_3. Probably the major arguments in favor of using finer charcoal and KNO_3 are:

(1) ease of grinding and elimination of screening by use of Mikro-Atomizer

(2) better fluidity of mixtures, and

(3) greater ease of compression.

From the slope of the dP/dt versus P trace of both samples PCD #6 and #7 during the first 500 psi of the closed bomb test (see Figure 10), it would appear that these samples might have equaled PCD "A" and I, given an irregular geometry. This is borne out by relative quickness data from the low pressure bomb. (See Figure 11.) This is particularly interesting in view of the fact that PCD #7 was made by compressing the three components (both KNO_3 and charcoal being as fine as possible) using only azeotropic alcohol. If reproducible results similar to PCD "A" can be achieved with alcohol alone, this should certainly be an improvement over earlier approaches.

Proposed processes for the manufacture of black powder, based on the use of both carbon disulfide and alcohol and on alcohol alone are discussed in IV, CONCLUSIONS AND RECOMMENDATIONS. For the reasons previously discussed, these processes are based on the use of 5-6 micron charcoal and 6-8 micron KNO_3.

FIGURE 11

LOW PRESSURE BALLISTIC BOMB DATA

Sample *	Burning Time	Peak Pressure	Relative Force	dP/dt at 100 psi	Relative Quickness
Commercial	0.046 sec	226 psi	100.0	7550	100.0
PCD #1	0.049 "	220 "	97.4	7150	94.7
PCD #2	0.058 "	164 "	72.7	3740	49.5
PCD #3	0.105 "	157 "	69.6	3650	48.4
PCD #4	0.130 "	162 "	71.7	2300	30.5
PCD #5	0.250 "	144 "	63.8	1070	14.2
PCD #6	0.047 "	180 "	79.7	4250	56.4
PCD #7	0.047 "	216 "	95.6	7100	94.2

*Each sample: 10.0000 ± 0.0005 gm

IV. CONCLUSIONS AND RECOMMENDATIONS

1. Conclusions from Experimental Work:

a. Vehicles and Solvents:

During the course of this study various solvents and vehicles have been considered. Toluene was discarded early in the study since its ability to dissolve sulfur was comparatively low. In those instances where the coating of the charcoal with sulfur was desired, carbon disulfide was used. The use of this material has three major disadvantges, however. Carbon disulfide is an inherently dangerous material with a low ignition temperature and high vapor pressure. (See Figure 6, page 17). Moreover, it is fairly expensive, selling for from 7.1 to 9.1 cents per pound in less than tank car quantities and for 5.45 cents per pound in tank car quantities. The use of carbon disulfide together with another solvent complicates the solvent recovery picture.

The use of water in several connections was investigated during the course of the study. While water was used as the final incorporation medium in sample PCD"A", results from samples PCD #4 and #5 where only water was used as the vehicle and from PCD #3 where 50% ethanol was used, indicate that control of potassium nitrate crystal growth becomes more difficult where water is present. This seems particularly true where many small particles are present in a KNO_3 sample where the particle range is large. The use of water, it appears, might necessitate extensive regrinding and screening, even if other factors were equal.

The use of fine particle materials mixed in azeotropic alcohol and compressed after the vehicle was removed led to sample PCD #7. This technique, from closed bomb and M-58 primer data, seemed equal to the use of both carbon disulfide and ethanol. The trace for PCD #7 during the first 500 psi of the closed bomb test in Figure 10, page 41, conforms closely to that for commercial black powder and the data from the low pressure bomb confirms this.

It seems therefore that two approaches to the use of vehicles are of interest in further process investigations:

(1) The use of carbon disulfide for dispersing the sulfur around the carbon particles followed by the use of azeotropic ethanol for the final mixing vehicle.

(2) The use of azeotropic alcohol for the complete mixing of all three components.

Considering the excellent pellets formed using only the alcohol, it would appear that, under the pressure of compacting, there is extensive flowing and redistribution of the sulfur. Microscopic evidence confirms this. It is suggested, therefore, that (2) above is the more desirable procedure and the one most likely to yield reliable black powder at a reasonable cost.

b. Particle Size of Components:

The ballistic data from PCD "A and from samples PCD #1 through #7 suggest that the use of potassium nitrate particle sizes below mesh #200 has little effect on ballistic properties. The reduction of charcoal seems to be more critical. It was found generally that any black made with charcoal particle size greater than that passing mesh #325 (sieve opening: 44 micron) was invariably too slow in the low pressure bomb or high pressure bomb.

It was found, however, that the finer charcoal and potassium nitrate both contributed to easier compacting and better control of density. It is recommended, therefore, that charcoal be held to below mesh #625 (sieve opening: 20 micron) and potassium nitrate to below mesh #325. Charcoal and potassium nitrate ground by the Mikro-Atomizer to the fineness used in this study is felt to be excellent for further work.

c. Grain Geometry:

The data from Picatinny Arsenal on both the High Pressure Ballistic Bomb and the M-58 Primer agree generally with the theoretical treatment in the previous pages. It had been demonstrated that black powder of a regular geometry cannot be used in the M-58 Primer and will not generally give ballistic results comparable with regular commercial powder.

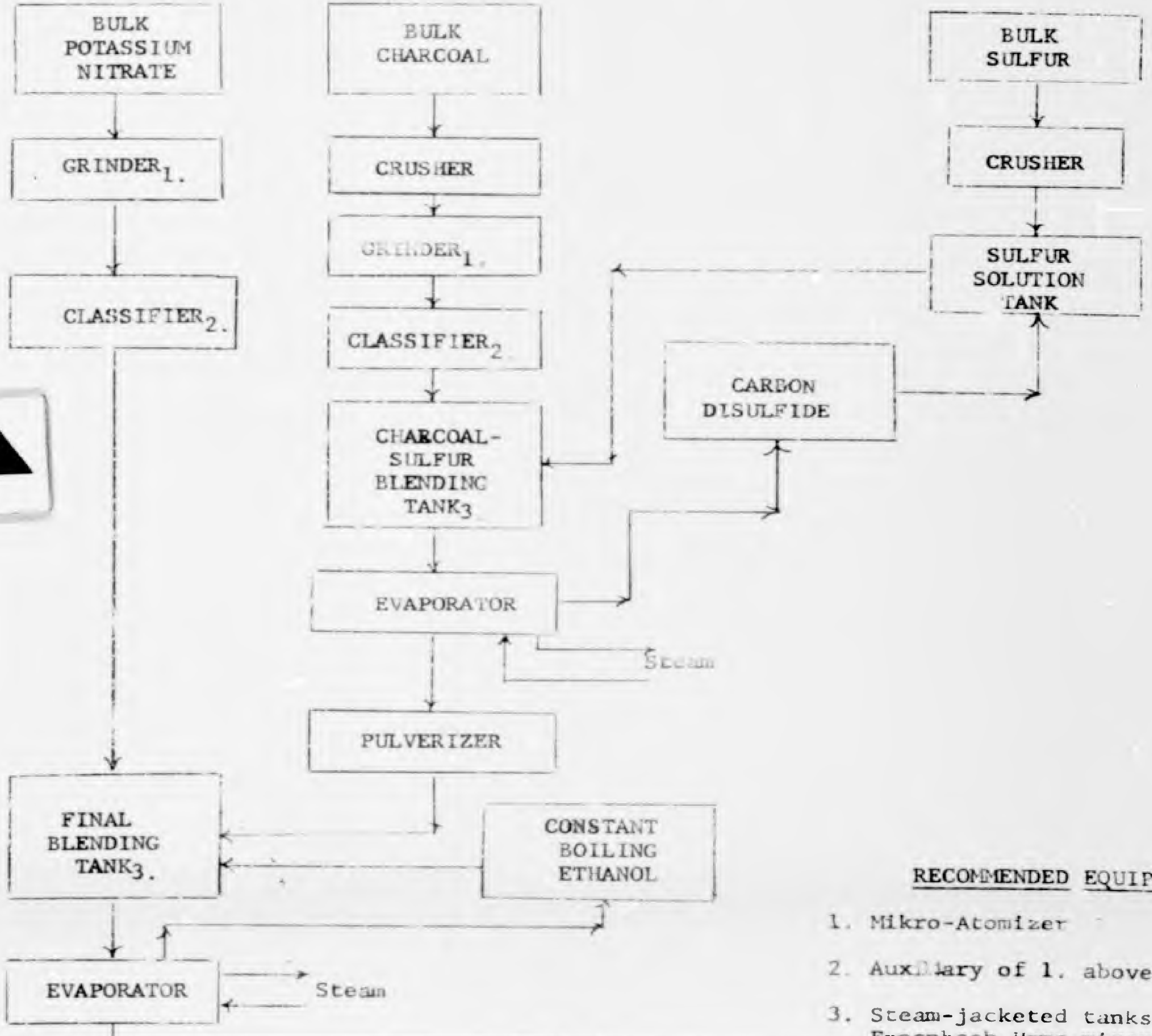
It is, therefore, recommended that all future work on duplicating black powder be confined to irregular geometries. The Stokes Tabletizer is not suited to the manufacture of black powder of various grades in production quantities.

2. Recommended Process and Process Equipment:

Figures 12 and 13 give two general flow diagrams for the production of black powder, using the techniques investigated under this contract. The first and less desirable approach (Figure 12) is that using carbon disulfide for the initial incorporation of the sulfur. The more desirable (Figure 13) is the second process which consists simply of the appropriate reduction of particle size followed by combination in an azeotropic ethanol vehicle, drying and compacting.

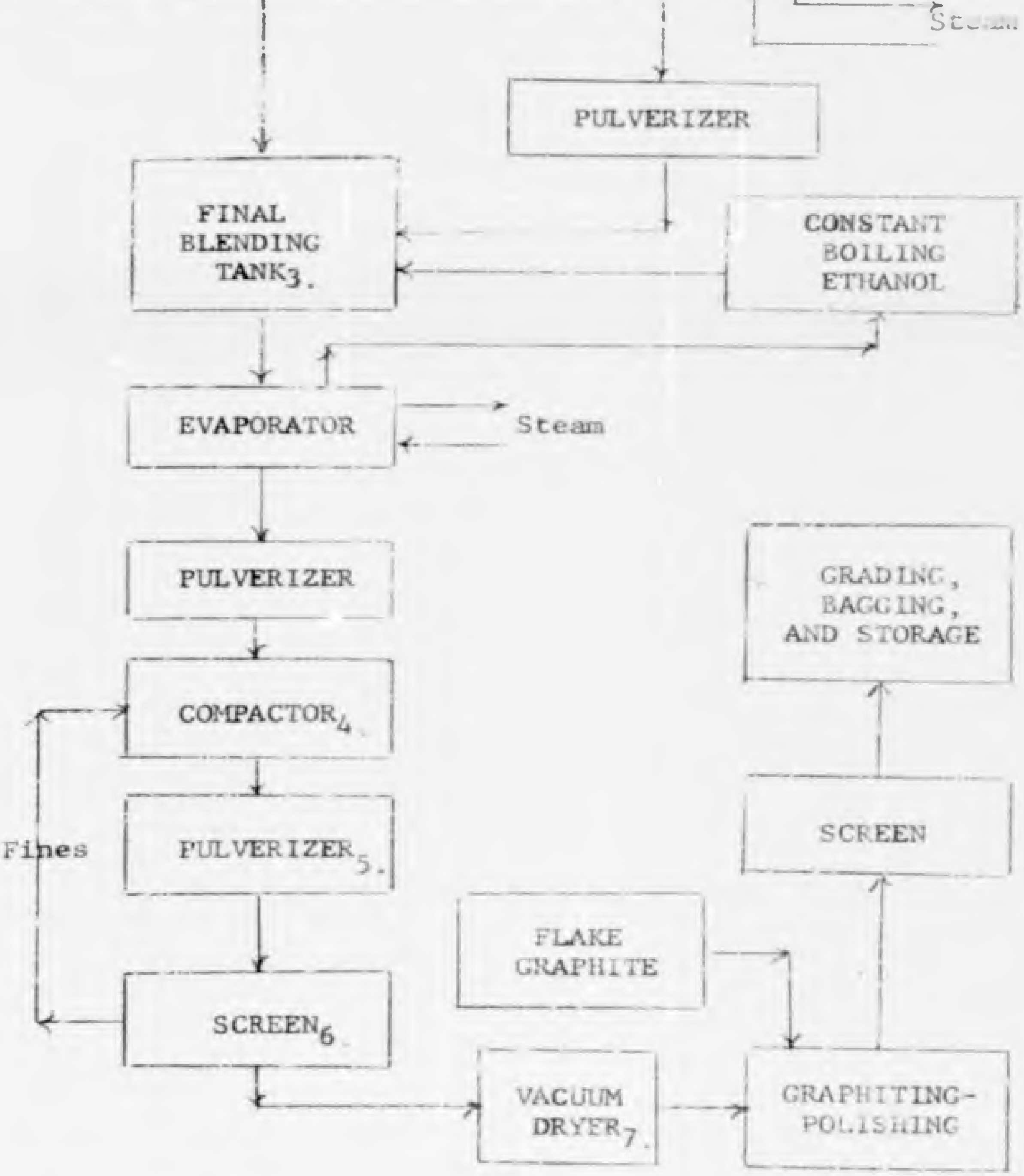
If the scheme in Figure 12 is used, fairly elaborate precautions must be taken to avoid sparks and friction which might set off the carbon disulfide vapors. Care must also be taken in storing the material to avoid sources of heat, such as direct sunlight which might ignite this easily flammable material.

1



RECOMMENDED EQUIPMENT

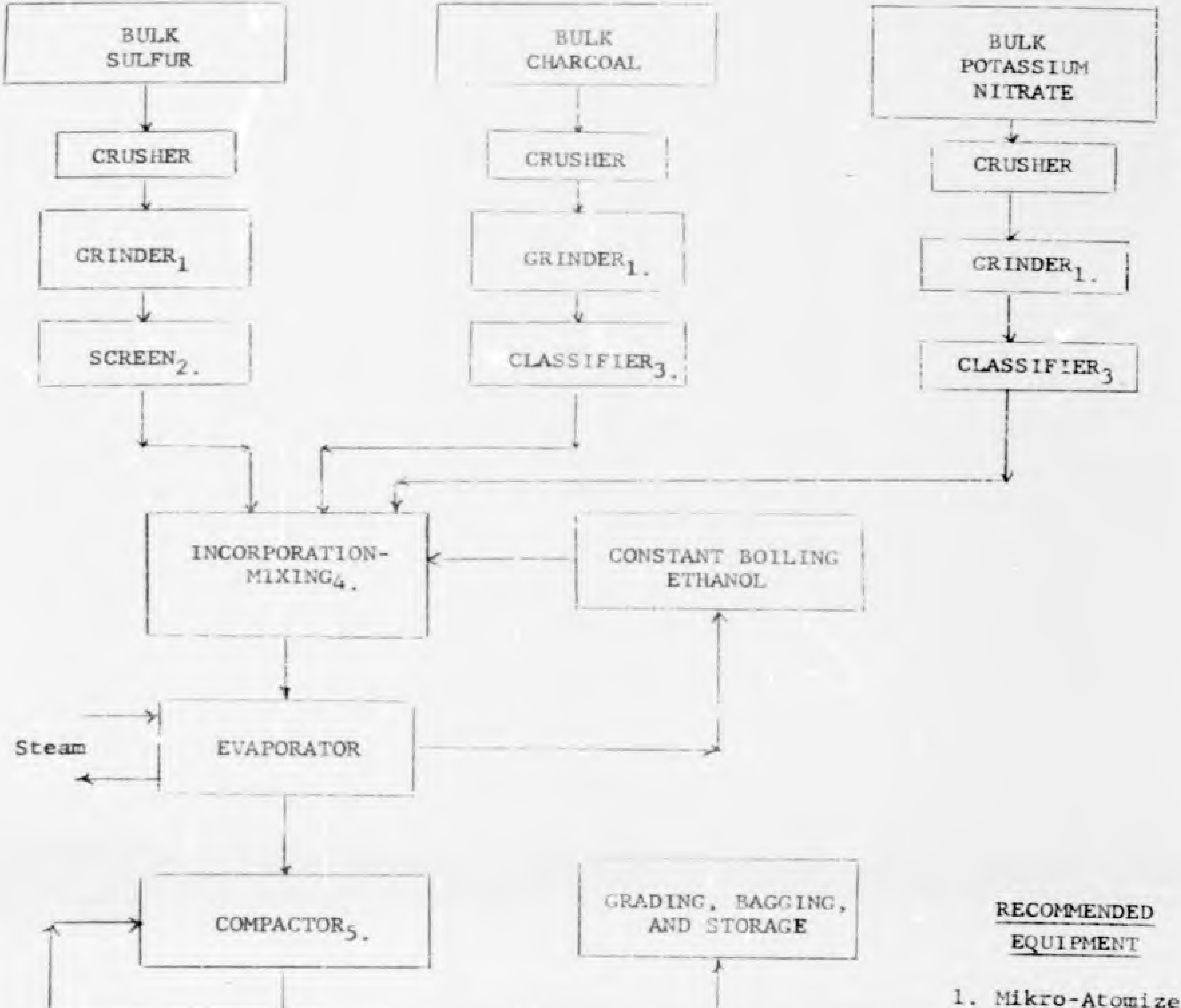
1. Mikro-Atomizer
2. Auxiliary of 1. above
3. Steam-jacketed tanks with



RECOMMENDED EQUIPMENT

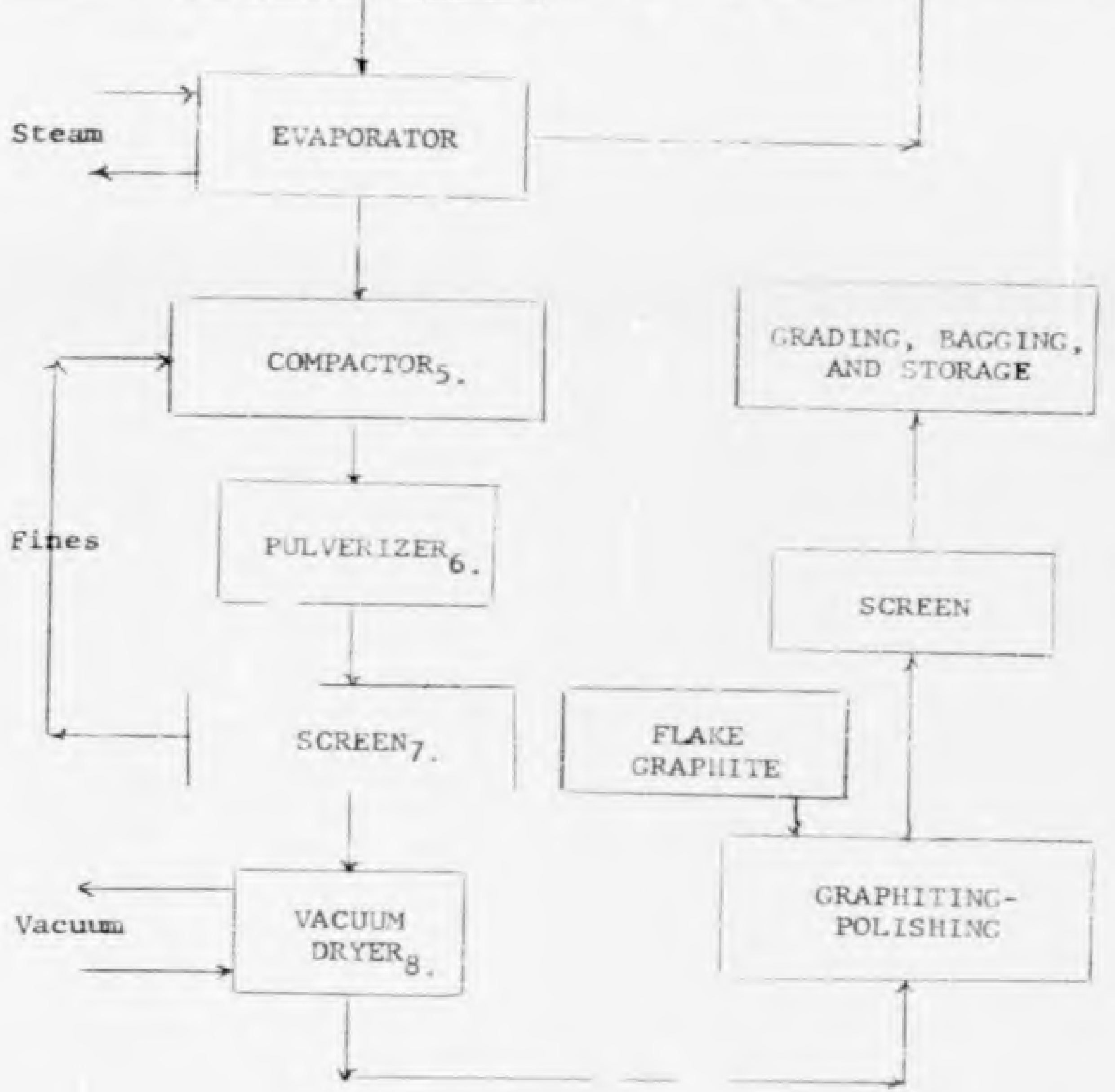
1. Mikro-Atomizer
2. Auxiliary of 1. above
3. Steam-jacketed tanks with Eppenbach Homo-mixer (Alternate: Sigma Blade Mixer
4. Allis-Chalmers Compacting System.
5. Included as auxiliary of 4.
6. See 5. above
7. Vacuum style optional

FIGURE 12: PROPOSED BLACK POWDER PROCESS, SCHEME I



RECOMMENDED
EQUIPMENT

1. Mikro-Atomizer



RECOMMENDED EQUIPMENT

1. Mikro-Atomizer
2. Mesh #200
3. See 1. above
4. Heated vat plus ribbon mixer or Eppenbach Homo-mixer
5. Allis-Chalmers Compacting Process
6. See 5.
7. See 5
8. Vacuum style optional

FIGURE 13: PROPOSED BLACK POWDER PROCESS, SCHEME II

a) Particle Size Reduction

A number of grinding and mailing machines have been considered for the reduction of the three constituents of black powder. Generally, the requirements for the reduction of lump sulfur are not nearly so stringent as with the potassium nitrate and charcoal. The Mikro-Pulverizer of the Metals Disintegrating Company of Summit, New Jersey has been successfully used and fits well into a production set-up, either batch or continuous.

A number of ball mills have been considered, most notably the Ball and Pebble Mills manufactured by the Paul O. Abbe Company. While ball milling does accomplish the size reduction required, the process is not adaptable to high production rates or to continuous processing. Ball and pebble mills *per se* were therefore not considered for use in any future pilot operation.

The use of the Allis-Chalmers Vibrating Mill, a modification of the ball mill, does suggest itself. This device, essentially a vibrating ball mill, is capable of continuous processing, handling up to a ton an hour of material. By suitable control of "stay" within the mill, it is possible to carry particle reduction down to a material, 90% of which is 20 microns or less. Separate air classification equipment is available with this apparatus.

However, for reduction of charcoal and potassium nitrate, the Mikro-Atomizer of the Metals Disintegrating Company, Pulverizing Machinery Division seems most suited to the particle sizes needed and the production volume desired. This device (see Appendix "A") includes air classification column such that a narrow range of particle sizes is automatically obtained. Using this device, no subsequent screening of the grind is necessary. For more complete classification of particles an air classifier is available.

The listed capacity of the Mikro-Atomizers for a representative substance, carbon black pellets, is:

Model	Average Size	Capacity, Lb/Hr
#5	7-10 microns	150
#6	" "	600
#8	" "	2100

Because of its general utility and continuous capacity, the Mikro-Atomizer offers the best features for incorporation into a black powder production set-up and is the recommended equipment.

b) Screening and Classification

In the particle size reduction stage of raw materials, the effluent grind from the Mikro-Atomizer is sufficiently classified for most purposes. However, an air classifier is available for further separation if necessary. A similar air classifier is available with the Allis-Chalmers Vibrating Mill.

For later screening of green black powder grain, vibrating screens of various types are applicable. In general, the single shaft vibrating screens, manufactured by the Dravo Corporation, seem adaptable to redesign in non-sparking metals. The Aero-Vibe vibrating screen Model AVS of Allis-Chalmers is similar in operating principle but seems even more adaptable to the screening of hazardous materials.

Because of their inapplicability to continuous processing, gyratory screens have not been considered for further pilot work.

c) Mixing and Incorporation

For batch-type production, some type of steam jacketed kettle equipped with a mixer is essential in either Scheme I or II for each incorporation. A variety of such kettles suggest themselves. One of particular interest is a twin motion pony mixer with dust cover manufactured by the J. H. Day Company, Division of Cleveland Automatic Machine Company.

It has been found that the Homo-Mixer of the Eppenbach Company, a high-speed shearer-impeller mixer, is of particular value in working with coarse pastes of the type encountered after the removal of carbon disulfide and addition of potassium nitrate and alcohol in Scheme I. This same mixer is similarly of value in the one-step incorporation in Scheme II. The Eppenbach offers the same type of impeller-shearer blade in a flow unit with variable return such that this type of mixing might be applied to continuous process.

For pilot plant work, the possible use of a sigma blade mixer has been considered in both processes. A variety of such mixers are available from such companies as the J. H. Day Company, Paul O. Abbe Company, etc. While these mixers are of limited value in a full scale production set-up, in a pilot plant they offer the advantage of being equipped for application of both heat and vacuum, allowing the combination of incorporation, evaporation, pulverizing and vacuum drying steps.

However, for full scale production, a continuous heavy duty mixer of the Eppenbach type is desirable. The Type GPM Heavy Duty Continuous Mixer of the Patterson Foundry & Machinery Company is considered an excellent approach as is the Paul O. Abbe Double Shaft Continuous Mixer.

d) Compacting

No form of extrusion machine, pelletizer nor tabletizer seems applicable to the black powder process. Forgetting for the moment the problems associated with a regular geometry, the production rates of these machines are generally too low. The available equipment for compacting appears to be limited to some form of hydraulic press, either bed or roll.

After evaluation of most of the equipment now available, it is recommended that future pilot plant compacting systems be based upon the Allis-Chalmers Compacting Process. The low bearing load compacting mill seems most applicable to the needs of black powder processing. This device, selling for approximately $30,000, has a pressure range of 10,000 to 25,000 psi. This device is, according to Allis-Chalmers, currently in use at the Badger Ordnance Works for compacting and flaking black powder.

A typical flow sequence which is most applicable to the black powder process is given in the Allis-Chalmers literature which is reproduced in Appendix "E".