



TEST REPORT

BLACK POWDER EQUIVALENCY

Final Report

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U.S. DEPARTMENT OF
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Test Report

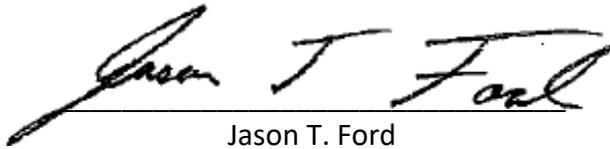
DOT R&D

Black Powder Equivalency

July 2, 2020



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I. OBJECTIVE

The U.S. Department of Transportation and Pipeline and Hazardous Material Safety Administration (PHMSA) has a need to establish criteria and a relatively simple test or series of tests for use in the laboratory or potentially by PHMSA inspectors to measure and compare explosive characteristics (energy profile) of alternative formulations of black powder (i.e., potassium perchlorate, charcoal and sulfur) to conventional and commercially black powder formulations (potassium nitrate, charcoal, and sulfur). The simple test or series of tests will determine if these alternative formulations fare equivalent to conventional or commercially available black powder. Black powder is a major component used to make fireworks approved under APA 87-1 Standard. USDOT PHMSA contracted Safety Management Service, Inc. (SMS®) to develop a test or tests to distinguish the differences between the powders being used as burst powders in aerial fireworks shells.

The U.S. APA requirements currently limit the use of **burst charge to black powder or equivalent non-metallic powder** (limited to 130 mg of metallic powder less than 100 mesh particle size for 1.4G articles). Burst charges for 1.3G articles are limited to black powder or similar pyrotechnic composition with no metallic fuel.

Currently there is no easy field test that determines the equivalency of non-black powder burst charges to black powder (whether these are pyrotechnic compositions that contain non-metallic or metallic fuels). The main concern is that "flash compositions" will be used in burst charge as these types of articles typically have 1.1 behavior

- Objective:** Develop a field test for comparison of burst charges used in aerial fireworks shells to determine if a burst powder is equivalent to black powder
- Easy to operate
 - Easy to interpret

II. EXECUTIVE SUMMARY

A. Program Concept

A Burst Powder Equivalency Test has been developed which allows for simple field testing that is easy to operate and that allows quick comparison of burst charges used in aerial fireworks to black powder and other burst charge powders. The testing method is a modification of the existing UN Koenen Test and consists of a non-reusable steel tube (Photo 1), with its re-usable closing device (threaded collar and nut), orifice plate (through which the gases from the decomposition of the burst powders escape), and a new protective device (Figure 1). Drawings for the new protective device (Figure 2) are provided in the Appendix.

The overall concept for this test is that substances that are more energetic and have burn rates that are relatively fast will require orifices that are much larger than substances that have slower burn rates. If the venting or orifice hole is sufficiently large, then the material will burn within the Koenen tube and vent out the specified orifice hole. If the venting or orifice hole is insufficient, then the Koenen tube will rupture (fragment or burst). This closely imitates the bursting of aerial shells. The confined powder ignites, burns, and bursts the shell open depending on its burn rate. Limiting diameters (which correlate to a vent size) are established when three trials at a given orifice size are performed with no tube rupture or fragmentation. The orifice size at the next smaller orifice diameter is considered the limiting diameter for that substance.



Photo 1: Burst Charge Equivalency Test Tube Assembly - Non-reusable Steel Tube, Re-Usable Closing Device (Threaded Collar And Nut), and Orifice Plates)

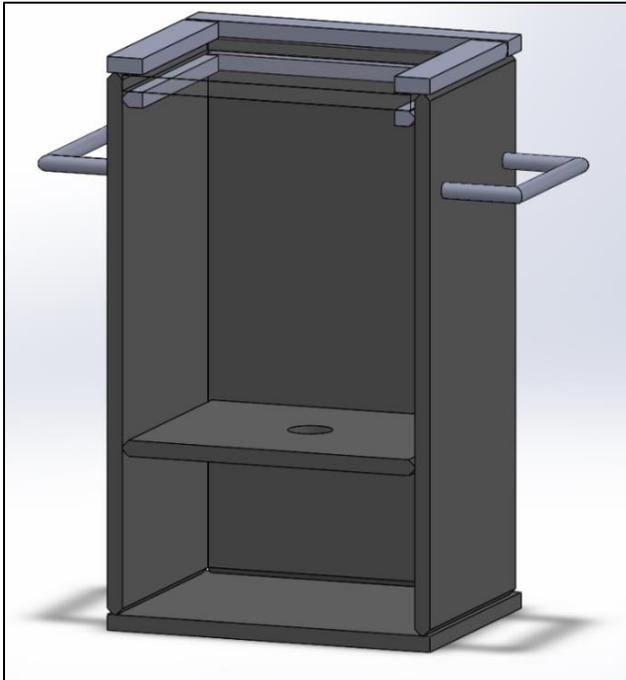


Figure 1: Test Fixture



Photo 2: Burst Charge Equivalency Test Setup

B. Initial Ignition Mode: Auto-Ignition (Std. 4-Burner System)

Initially the standard Koenen ignition source (4-burner propane system) was used, heating the sample up to its autoignition temperature and igniting. After a few phases of progressive

testing, a new ignition method was developed using an electric match as the ignition mode (Photo 2). The spark/ flame ignition mode of an electric match best simulates the initiation source found in pyrotechnic aerial shells and if the orifice is not of sufficient size, the gases rupture, burst, or fragment the tube. The test is repeated at increasing orifice diameters until three trials with no tube rupture occurs or until the limits of the closing device are met (22 mm orifice) .

ITEM NO.	PART NUMBER	DESCRIPTION	QTY.
1	KNNSHLD-005	BACK	1
2	KNNSHLD-004	BOTTOM	1
3	KNNSHLD-003	SIDES	2
4	KNNSHLD-007	LEXAN SUPPORT RAIL	1
5	KNNSHLD-008	LEXAN SUPPORT RAIL	2
6	KNNSHLD-009	LEXAN SUPPORT RAIL	2
7	KNNSHLD-011	LEXAN SUPPORT RAIL	1
8	KNNSHLD-012	TOP (3/8" LEXAN)	1
9	KNNSHLD-006	TUBE SUPPORT	1
10	KNNSHLD-013	HANDLES	2

Figure 2: Test Fixture Drawing (Protective Device)

The first item determined for the new method was the sample size for testing. Due to the high confinement and possibility of fragmentation, a smaller quantity will provide the highest safety from an exposure consideration. Using the propane burner system, the sample size was determined as outlined in the test results in Table 1. A sample size of 5 grams was ultimately determined to be the best fit for the developed method. In addition, during this phase of the project, the Threshold Initiation Levels (TILs) for the limiting diameters was determined for the standard commercial 4Fg black powder for 3-gram and 5-gram sample sizes of 5 mm and 10 mm, respectively. These values matched the limiting diameters for each of these sample sizes further substantiating this test method as a valid method for burst charge powder comparison. Additional testing on some known whistle compositions were also performed to see if the method could differentiate these faster burning compositions. Initial data indicated that the method would be able to differentiate the upper levels of the orifice diameters.

Table 1: Summary of Burst Charge Equivalency Test – Ignition Mode: Auto-ignition

<u>Compositions Subjected to Auto-Ignition Mode</u>	<u>Limiting Diameter (mm)</u>
3 g - FFFFg Goex Black Powder (USA)	3
3 g - FFFFg Goex Black Powder (duplicate set) (USA)	≥3
5 g - FFFFg Goex Black Powder (USA)	≥5
5 g – 95% FFFFg 5% (0.88-0.105mm Al. Powder) (SMS)	5
5 g – 95% FFFFg 5% (0.125-0.149mm Al. Powder) (SMS)	5
5 g – 90% FFFFg 10% (0.088-0.105mm Al. Powder) (SMS)	5
5 g – 90% FFFFg 10% (0.125-0.149mm Al. Powder) (SMS)	5
5 g – 85% FFFFg 15% (5μ Al. Powder) (SMS)	5

<u>Compositions Subjected to Auto-Ignition Mode</u>	<u>Limiting Diameter (mm)</u>
5 g – 90% FFFFg 10% (0.088-0.105mm Al. Powder) (SMS)	5
5 g – 85% FFFFg 15% (0.125-0.149mm Al. Powder) (SMS)	5
5 g – 97% FFFFg/ 3% 5µ Al. Powder (German Black) (SMS)	<8
5 g - FFFFg Goex Black Powder (USA)	8
5 g – 95% FFFFg, 5% (5µ Al. Powder) (SMS)	8
5 g – 90% FFFFg 10% (5µ Al. Powder) (SMS)	8
7 g – FFFFg Goex Black Powder (USA)	8
5 g – AR2210 (flaked smokeless powder) (Australia)	<10
5 g – 94% FFFFg/ 6% 5µ Al. Powder (German Black) (SMS)	<15
7 g – 94% FFFFg/ 6% 5µ Al. Powder (German Black) (SMS)	<18
5 g – 57% Potassium perchlorate, 19% Sulfur, 13.5% Aluminum, 10.5% Charcoal - Canister Shell #2 (Burst Powder) (China)	<20
5 g – 69% Potassium perchlorate, 20% Sulfur, 11% Charcoal - Canister Shell #3 (Burst Powder) (China)	<20
5 g – Pure Flash Comp	>20
5 g – Canister Shell #1 (Burst Powder) (China)	>20
3 g – 70% Potassium Perchlorate, 30% Sodium Benzoate - Whistle Composition #1 (USA)	> 22
5 g – 70% Potassium Perchlorate, 30% Sodium Benzoate - Whistle Composition #1 (USA)	> 22
3 g-70% Potassium Perchlorate, 10% Sodium Benzoate, 20% Corn Starch - Whistle #3 (USA)	> 22
5 g-70% Potassium Perchlorate, 10% Sodium Benzoate, 20% Corn Starch - Whistle #3 (USA)	> 22

C. Finalized Ignition Mode: Electric Match

As the method evolved to using the electric match ignition mode, several samples were tested from the traditional rice hull formulations, to exotic non-metallic benzoates. During the duration of this project, testing was performed at SMS’s test facility at the Tooele Army Depot in Tooele, Utah, at a pyrotechnic manufacturer in the United States, and at a pyrotechnic manufacturer in China. The apparatus was taken to China and tested on formulations right out of production from current aerial shells. The results from all these tests using the electric match ignition mode are presented in Table 2.

Table 2: Summary of Burst Charge Equivalency Test – Ignition Mode: Electric Match (Flame)

<u>Compositions Subjected to Electric Match Ignition Mode</u>	<u>Limiting Diameter (mm)</u>
Theatrical Flash (from HSL Testing)	< 3
GOEX Meal Powder (USA)	3
GOEX 5FBP (unglazed) (USA)	3
6” Shell Lift Charge (China)	3
6” Shell Burst Charge – Rice Hulls (China)	3
15’ Sparkblast (from HSL Testing) (France)	3
Black Powder, 5% Al Powder (GB) optimized (USA)	< 5
5 g - FFFFg Goex Black Powder (USA)	5

<u>Compositions Subjected to Electric Match Ignition Mode</u>	<u>Limiting Diameter (mm)</u>
Black Powder with 10% Al Optimized (USA)	5
35% Potassium Nitrate, 23% Potassium Perchlorate, 13% Potassium Benzoate, 10% Charcoal (USA)	5
74% Potassium Perchlorate, 22% Potassium Benzoate, 4% Charcoal (SMS)	8
GOEX Meal Powder 90%, Al Powder (GB) 10% (USA)	8
68% Barium Nitrate, 9% Sulfur, 23% Aluminum (GB) (USA)	8
80% Potassium Perchlorate, 20% Potassium Benzoate - "Benzoate Break" (China)	8
69% Potassium Perchlorate, 17% Sulfur, 14% Charcoal- "Standard Break" (China)	> 8
Standard Break Charge with 5% aluminum powder (China)	> 8
Waterfall (from HSL Testing) (France)	10
50' - 60' Yellow Mine (from HSL Testing) (France)	10
70% Potassium Perchlorate, 20% Potassium Benzoate, 10% Air Float Charcoal (USA)	10
70% Potassium Perchlorate, 30% Potassium Benzoate - "Whistle Comp" (China)	> 10
68.5% Potassium Perchlorate, 12.5% Aluminum, 11.4% Charcoal, 7.6% Sulfur (SMS)	12
70% Potassium Perchlorate, 30% Potassium Benzoate (USA)	12
60% Potassium Perchlorate, 40% German Dark Pyro Aluminum (USA)	15
70% Potassium Perchlorate, 10% Sodium Benzoate, 20% Corn Starch - Whistle #3 (USA)	15
62% Potassium Perchlorate, 26% Sodium Salicylate, 1% Red Iron Oxide, 9% Titanium (60-100 mesh), 2% Mineral Oil Whistle Composition #5 (USA)	15
57% Potassium perchlorate, 19% Sulfur, 13.5% Aluminum, 10.5% Charcoal (China)	≥ 15
50% Potassium Perchlorate, 50 % Magnesium (44 microns or smaller)	< 18
Standard Break Charge with 15% aluminum powder (China)	> 10 / < 18
Standard Break Charge with 10% aluminum powder (China)	> 18
Le Maitre Whistle Comp (from HSL Testing) (France)	20
Super Gunshot Red (from HSL Testing) (France)	20
Flash Report (from HSL Testing) (France)	> 22
65% Potassium Perchlorate, 35% German Dark Pyro Aluminum (USA)	> 22
70% Potassium Perchlorate, 30% German Dark Pyro Aluminum (USA)	> 22
Spanish Salute Powder (#7) (USA)	> 22
70% Potassium Perchlorate, 30% Sodium Benzoate - Whistle Composition #1 (USA)	> 22
70% Potassium Perchlorate, 20% Sodium Benzoate, 10% Corn Starch - Whistle #2 (USA)	> 22
63.6% Potassium Perchlorate, 27.3% Sodium Benzoate, 9.1% Titanium - Whistle #4 (USA)	> 22

The results clearly show the potential for this test method. Several samples can easily be tested in a short amount time and compared.

The current standard vent sizes are as follows:

- 3.0 mm
- 5.0 mm
- 8.0 mm
- 10.0 mm
- 12.0 mm

- 15.0 mm
- 18.0 mm
- 20.0 mm
- 22.0 mm

Standard 4Fg black powder has a limiting diameter of 5 mm. The Rice Hulls composition and 2Fg type lift charges have a limiting diameter of 3 mm. The limiting diameter for flash compositions could not be determined (> 22 mm), however, many known formulations were tested, and a limiting diameter assigned. At the onset of this project, the goal was to develop a test that would establish the relative burst charge power of black powder compositions compared to other pyrotechnic powders with enough resolution to distinguish the differences between these powders. Based on the data, it appears this test method accomplishes this goal.

D. Sample Test Results

The following photos show the typical test setup for the final configuration and typical test results for both “NO-GO” or Passing criteria and “GO” or non-conforming reactions.

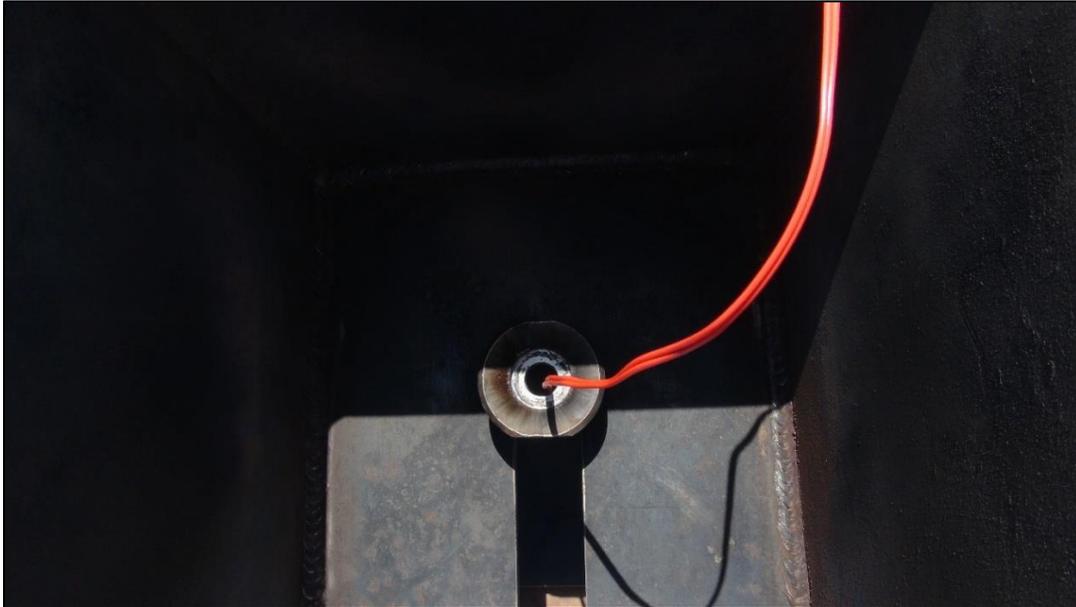


Photo 3: Burst Charge Equivalency Test Setup



Photo 4: Typical “No-Go” Results (PASS)



Photo 5: Typical “No-Go” Results (PASS)



Photo 6: Typical “Go” Results (FAIL)



Photo 7: Typical “Go” Results (FAIL)



Photo 8: Typical “Go” Results (FAIL)

E. Resolution of the Test Method

Analyzing the data, it can be seen that the tradition black powders, the standard potassium perchlorate/potassium benzoate (both from the USA and China), and some even metal containing compositions have limiting diameters from 3 to 10 mm. It is evident that when the formulations are optimized, the burn rate and burst power increase, and the result is an increased limiting diameter. It was also interesting to note that when the China standard break charge formulation was optimized with aluminum powder, the limiting diameter increased from 8 mm to 18 mm or greater. However, simply cutting out metallic fuels will not capture all

the pyrotechnic compositions that might not be equivalent to black powder (i.e., have limiting diameters much greater). Figure 3 shows the range of data the breadth of this method.

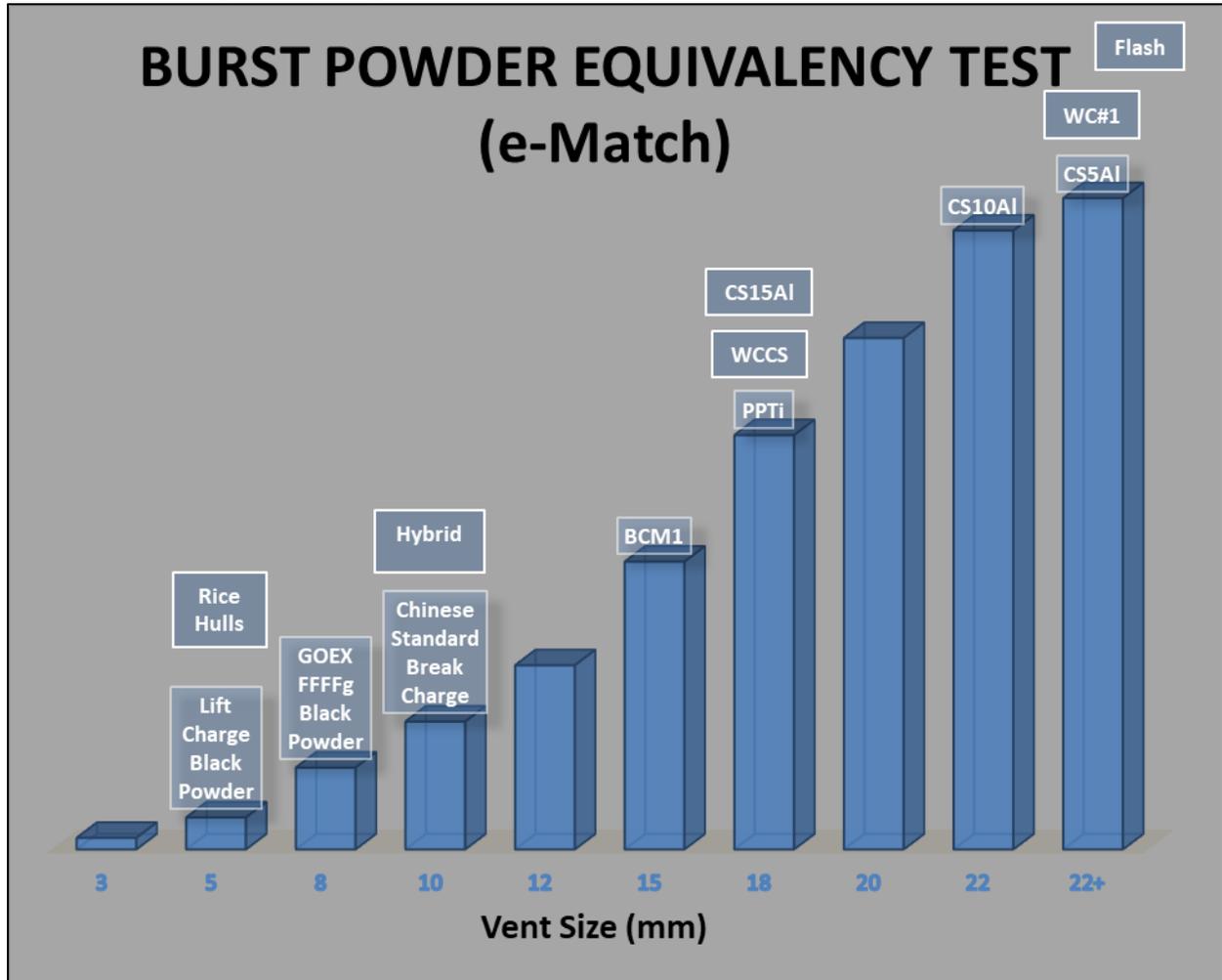


Figure 3: Burst Powder Equivalency Test – Range of Limiting Diameters

F. Defining “Black Powder Equivalency” – Limiting Diameter Range

The next main conclusion was to establish the acceptable level for what range of limiting diameters would be considered equivalent to black powder. After analyzing the data and consultation with PHMSA personnel, a range of 3 mm to 8 mm would be an acceptable range for powders that are clearly similar or equivalent in burst power to black powder. Powders exhibiting limiting diameters greater than 8 mm would be considered not equivalent to black powder and not allowed, for example, to be used as a burst powder for Consumer 1.4G fireworks. This range may increase slightly with the establishment of additional data.

G. Path Forward – Additional Testing

Now that the method has been established, additional testing is necessary to vet the method and allow for discussion on ease of use, etc. It is proposed that additional trials be performed to help substantiate the proposed hypothesis. The following is list of potential future efforts that would add to this body of work:

- Select approximately 10 compositions from Table 2 and perform complete Threshold Initiation Level (TIL) testing on each composition to confirm the limiting diameter that has been established.
- Select 20 more different compositions and perform the Burst Powder Equivalency Test on these powders to enlarge the database for this test method.
- Build a protective device that has a tapered slot and determine if this would aid in increasing efficiencies. It is estimated that move from a hole to a slotted hole in the protective device reduced the trial time by 30%.
- Complete drawing package for a slotted and taper slotted protective device.
- Select 5 or 6 of the tested compositions and contract with an pyrotechnic manufacturer (preferably in China, as this is where the majority of the fireworks in the U.S. originate from) to manufacture a few complete aerial shells containing these compositions the burst charges. Function the shells individually and record the overpressure and noise generated to see if a difference does exist in the final product.
- Investigate further the effect the match wire thickness has on the test results. It is current hypothesized that electric match leads are inconsequential as the reduction is constant (same for all powders) as long as the lead wire diameter is specified/ maintained. Does this hold true for the smaller diameters versus the larger diameters?
- Investigate further the energetic contribution of the electric match to the energy released by the burst charge sample. It is currently hypothesized that this contribution is insignificant as the mass is less than 2% of the system and would have little effect on the test results

III. BACKGROUND – APA 87-1 STANDARD

PHMSA’s primary mission under the Hazardous Materials Transportation Uniform Safety Act of 1990, P.L. 101-615, is to protect people and the environment from the risks inherent in the transportation of hazardous materials (HM), which include the classification and transportation of fireworks. Division 1.4G consumer fireworks may be classed and approved by the Approvals and Permits Division, within PHMSA’s OHMS or an established Department of transportation (DOT) approved fireworks certification agency (FCA). Each 1.4G consumer firework must be evaluated for conformance to the requirements in S173.64 of 40 CFR Hazardous Materials Regulations (HMR), which requires a firework device be manufactured in accordance with the American Pyrotechnic Association (APA) 87-1 Standard. Black powder is a major component used to make fireworks approved under APA 87-1 Standard. Requirements under the APA 87-1

standard, UN recommendations, and 49 CFR all defined black powder as mixtures of potassium nitrate, charcoal, and sulfur but the APA 87-1 standard allows similar / equivalent pyrotechnic composition to be used as black powder. PHMSA requires assistance in defining “similar / equivalent pyrotechnic composition” by performing research to measure the properties of black powder with one or more test(s) that measure the energy profile of alternative formulations of black powder (for example potassium perchlorate, charcoal and sulfur) and compares them to conventional (potassium nitrate, charcoal and sulfur) and commercially available black powder formulations. The test or tests will be used to determine if these alternative formulations are “equivalent” to conventional or commercially available black powder. To understand the how to approach this effort, the following areas of understanding are required:

- How is black powder produced?
- How are Aerial Fireworks Shells constructed?
- What is the role of burst charge powders in Aerial Fireworks Shells?
- What are the established limits of APA 87-1 Standard?
- What are Potential Existing Tests?

A. Black Powder Manufacturing

The earliest formulation for Black powder or its earliest predecessor, dates back to 900 B.C. China were three formulations of saltpeter, sulfur, and charcoal were being used for fire bombs or flame throwers, however, the saltpeter content was closer to 50% for all three formulations³. However, the formulation varied mainly with the percentage of saltpeter (the original potassium nitrate) depending on the application (bombs, grenades, mines, incendiary articles, etc.). Purification techniques of saltpeter by Chinese and Islamic alchemist brought about a more optimized formulation. Hasan al-Rammah⁴ listed over 100 Arabic recipes as far back as the 13th century. The most ideal formulation of black powder was refined over the years to 75% potassium nitrate, 10% sulfur, and 15% charcoal.

Currently there are around 9 types of commercial Black Powders available in the U.S., however some may no longer be being produced or imported:

- (1) GOEX (made in America);
- (2) Swiss (made in Switzerland by Swiss Black Powder Factory);
- (3) Wano and Schuetzen (made in Germany by Wano);
- (4) KIK Kamnik (made in Slovenia); no longer imported
- (5) Elephant Black Powder (made in Brazil- no longer in operation)
- (6) Diamondback Powder (made in Brazil; equipment purchased from Elephant plant); no longer imported
- (7) Skirmish, a low grade GOEX powder (made in America by GOEX but repackaged by Powder Inc.).
- (8) Jack’s Powder Keg Black Powder (made in America by GOEX but repackaged by Jack’s Powder Keg; this is GOEX 5FA and is a 3FFF equivalent).

(9) Lidu Black Powder (made in China – no longer available)

All black powders are not created equally. Rifle powders require a very specific recipe along with a meticulous manufacturing process to generate a good sporting powder. Most sporting powders are made from charcoal from alder wood but the most powerful is made from Pacific willow. Any missteps in the manufacturing process will result in an underperforming powder. For example, one issue with underperforming black powder is ensuring that the grains are polished properly and cleaned after polishing. If not, they will retain a powder dust clinging to the grains that will cause issues with accuracy. In addition, if the bark is not removed completely, the powder can lose the desired “moist-burning” property due to the charcoal being over-burnt during the high charring temperature preparation step.

In the United States, black powder grains were designated F (for fine) or C (for coarse) depending on how finely granulated it is. Grains are produced by corning the mixture. Corning is a method for processing the meal powder into grains of black powder. This is done by pressing the wetted meal into a cake. The cake is then fractured into grains and screened into the different grain sizes. Grain diameter decreases as the number of Fs increase (finer granulation) and increased with a larger number of Cs, ranging from about 2 mm for 7F to 15 mm for 7C. The more "F's", the finer the Black Powder is, and the finer the Black Powder is, the faster it burns. For sporting powders, the smaller (finer) granulations burn faster and are used in small bore guns, where larger (coarser) granulations, are used for larger bore guns and cannons. The coarser powders are also used as lift powders for launching aerial firework shells out of mortars. Sizes may be identified by the manufacturer as well. For bore applications based on granulation, sizes are typically as follows:

- **Fg:** Coarse grain typically used in cannons, muskets, rifles larger than .75 caliber, and shotguns that are 10-gauge or larger. These may also be used as lift powders for aerial firework shells.
- **FFg:** Medium grain typically used in larger rifles between .50 and .75 caliber, 20-gauge to 12-gauge shotguns, and pistols larger than .50 caliber. These may also be used as lift powders for aerial firework shells.
- **FFFg:** Fine grain typically used in smaller rifles and pistols under .50 caliber and smaller shotguns.
- **FFFFg:** Extra-fine grain used as a priming powder in the pans of flintlocks. Never use FFFFg black powder as the primary powder charge in a rifle, pistol, or shotgun.

GOEX uses the following designations for their Blasting Grade “A” Black Powder:

- FA -3 mesh / +5 mesh
- 2FA -4 mesh / +12 mesh

- 3FA -10 mesh / +16 mesh
- 4FA -12 mesh / +20 mesh
- 5FA -20 mesh / +50 mesh
- 6FA -30 mesh / +50 mesh
- 7FA -40 mesh / +100 mesh
- Meal D -40 mesh to dust
- Meal F -100 mesh to dust
- Meal XF -140 mesh to dust

Meal powder is the fine dust produced as a byproduct during the milling high-quality black powder, where the powder is corned and screened to separate it into different grain sizes. Meal powder is used in various pyrotechnic applications, usually acting as a prime to other compositions. It is also used in many fireworks to add power to the lift charge increasing the height of the firework.

It was observed, however, that most fireworks companies in China are not utilizing these types of commercial black powders and each fireworks plant manufacturers their own break charge powder which they call “black powder.” These black powders follow the general formulation for traditional sporting black powder, but the potassium nitrate is exchanged for potassium perchlorate in many instances. The current “standard” break charge powder for aerial shells is 69% potassium perchlorate, 17% sulfur, and 14% charcoal. These “black powders” do not have to be as clean in the manufacturing process as they do for the sporting powder industry and the variations on the formulations for these “black powders” are numberless. In addition, the burst powders may also have effects as part of the powder output.

B. Aerial Shell Construction

The fireworks shells have varying compositions. The specific chemicals would be those approved from within APA 87-1.

Aerial fireworks shells consist of the following components as seen in Figures 4-6:

- Fast-acting fuse
- Lift charge – propels the shell into the air
- Time-delay fuse
- Ignition charge
- Bursting charge – ruptures case and ignites/propels stars
- Shell case
- Stars – effects (produces sound, light, and colors)

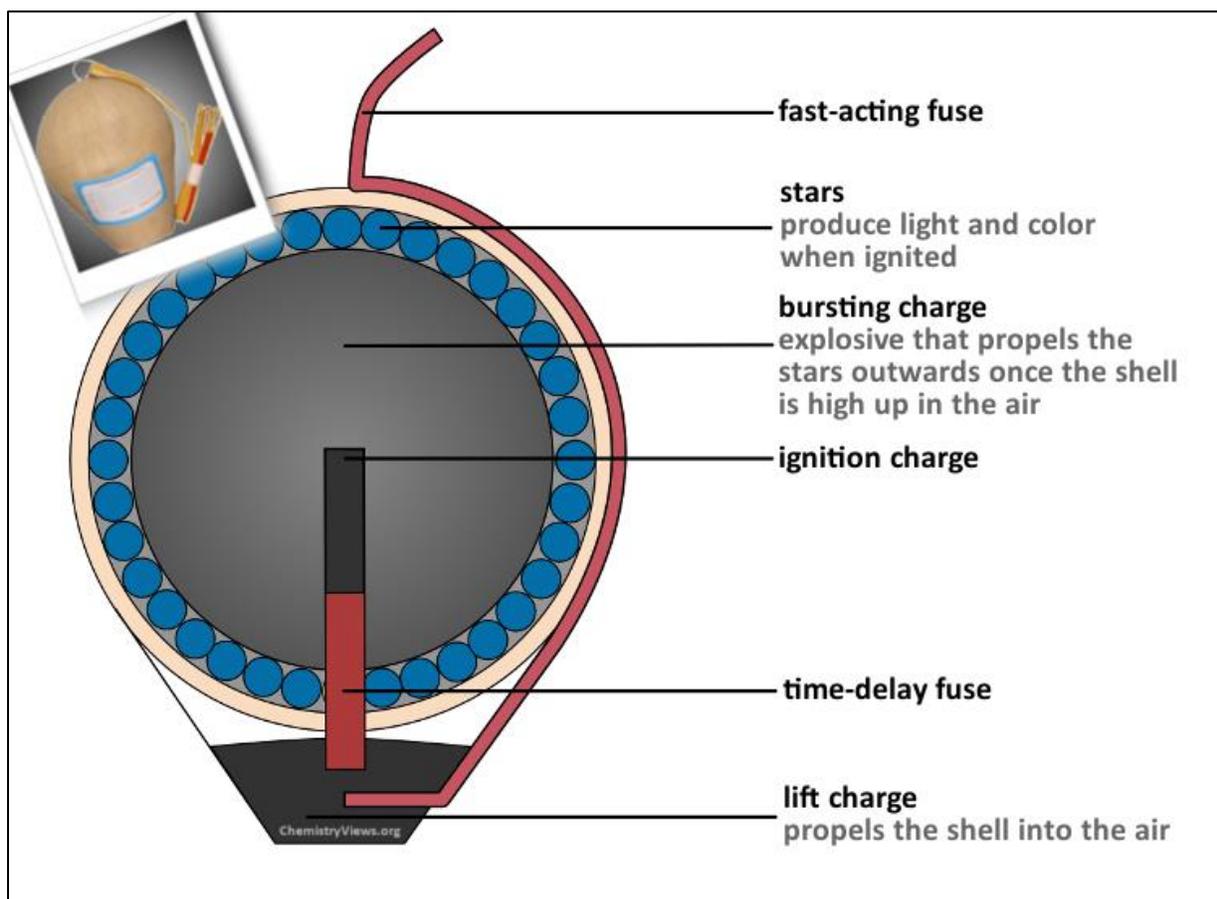


Figure 4: Sample Aerial Fireworks Shell Diagram (Spherical)

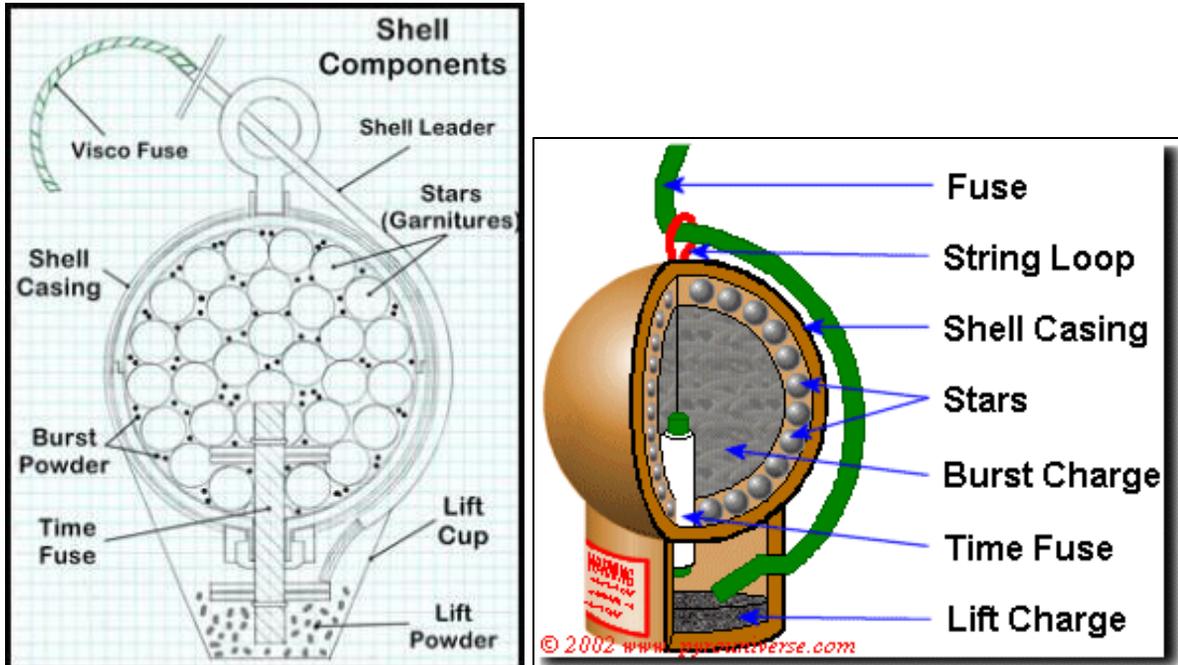


Figure 5: Sample Aerial Fireworks Shell Diagram (Spherical)

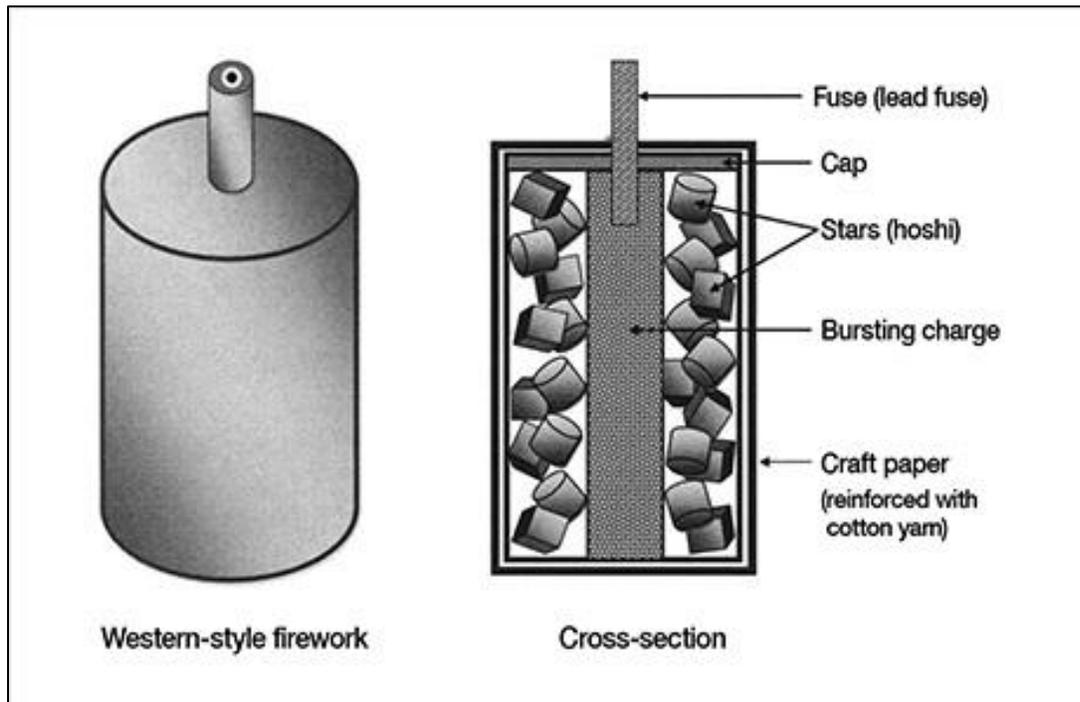


Figure 6: Sample Aerial Fireworks Shell Diagram (Cylindrical)

Photos 9-24 show some actual spherical and cylindrical shells and their internal composition as they were dissected.



Photo 9: Example Aerial Fireworks Shell – Spherical and Cylindrical



Photo 10: Example Aerial Fireworks Shell – Stars

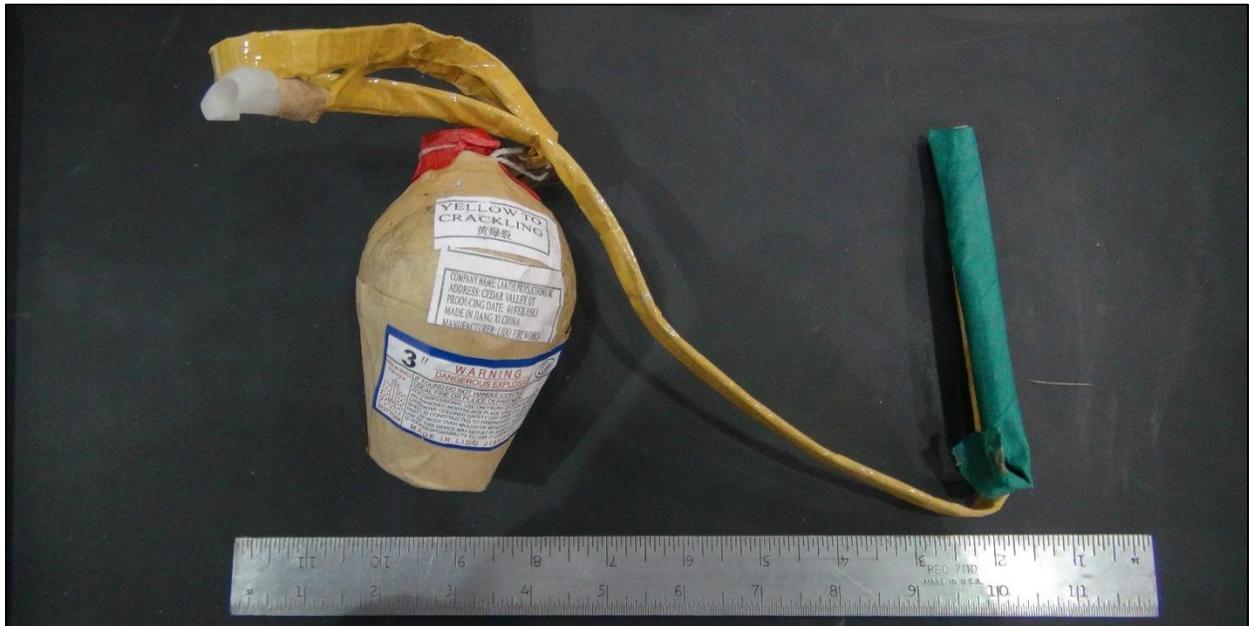


Photo 11: Example Aerial Fireworks Shell – 3-Inch



Photo 12: Example Aerial Fireworks Shell – 3-Inch Shell Case w/ Fuse and Lift Charge Removed



Photo 13: Example Aerial Fireworks Shell – 3-Inch Shell Lift Charge



Photo 14: Example Aerial Fireworks Shell – 3-Inch Shell Case



Photo 15: Example Aerial Fireworks Shell – 3-Inch Shell Burst Charge and Stars



Photo 16: Example Aerial Fireworks Shell – Coated Rice Hulls and Stars



Photo 17: Example Aerial Fireworks Shell – 3-Inch Shell Burst Charge and Stars

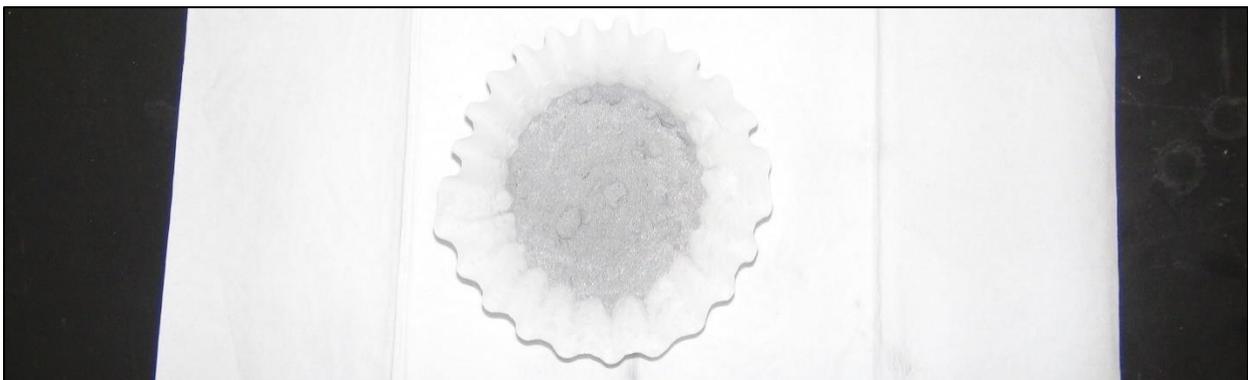


Photo 18: Example Aerial Fireworks Shell – 3-Inch Shell Burst Powder and Stars



Photo 19: Example Aerial Fireworks Cylindrical Shell



Photo 20: Example Aerial Fireworks Cylindrical Shell – Burst Powder

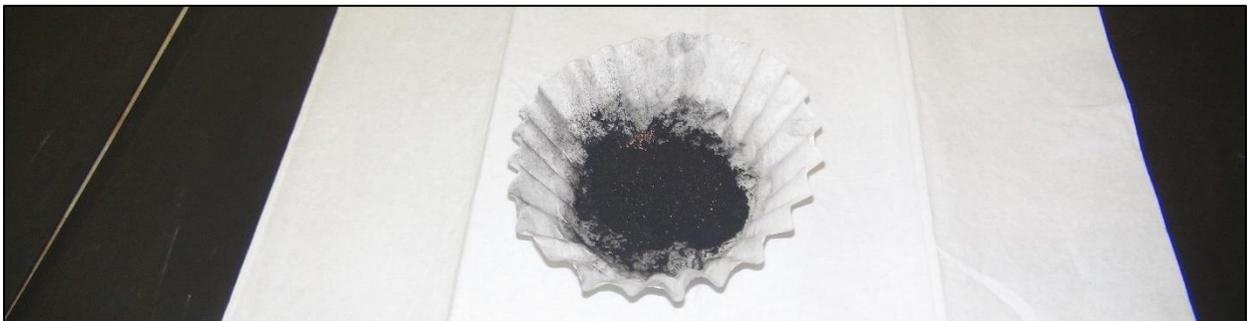


Photo 21: Example Aerial Fireworks Cylindrical Shell – Burst Powder



Photo 22: Example Aerial Fireworks Cylindrical Shell – Burst Powder and Stars



Photo 23: Example Aerial Fireworks Cylindrical Shell – Stars



Photo 24: Example Aerial Fireworks Cylindrical Shell – Burst Powder

C. Burst Charge

“Black powder” is currently used in two parts of aerial shells, the lift charges, and the burst charges. As explained previously, the burst charge, or break charge, is the powder contained within the aerial shell that ignites at its peak height and ruptures case, igniting and propelling the stars out into the designed pattern. The evidence based on the research performed and previous work/dissections of aerial shells, shows that, while the lift charges are most certainly a 1F or 2F black powder charges, commercially available black powders are not being used as the burst charge (or burst powders) in 1.4G aerial fireworks shells. The focus of this research then, was the burst powders being used in these aerial shells and in developing a simple, easy to perform field test to determine if a powder is equivalent or similar to the standard, commercial black powder. This was done to provide a baseline as these commercially available powders are consistent in formulation and output, while break charge powders used in aerial shells appeared to differ from one aerial shell to another.

Current color aerial shells will typically use coated rice hulls (see Photo 8) for their break powder for the 4-inch shells and larger. This is because coated rice hulls will produce a large, symmetrical display but the output is not so large as to shatter the stars or propel them so fast that they do not ignite. This coated rice hull formulation has been equated to a 2FA size black powder. Burst enhancers may be added to the coated shells if addition power is desired. For the smaller shells, granulated black powder or other burst powders are normally used. For many years metal enhancement was being introduced to the formulations creating stronger break charges.

When the desired effect of the aerial shell is a loud noise or bang rather than a visual effect, the shells are referred to as “salutes” and the burst powder is usually a flash powder. These shells will also give off a bright flash/light, but do not have the color, patterns, tails, or other effects of the standard color shells.

Flash powder is a pyrotechnic composition, a mixture of oxidizer and metallic fuel, which when ignited, burns quickly, and produces an intense light. Flash powders are extremely sensitive to a soft flame or spark ignition.

Based on the research performed and the definitions of these types of aerial fireworks shells, the burst powders would range from standard commercially available black powder all the way up to flash powders, which would not be appropriate or very limited use in 1.4G fireworks.

For black powder, during combustion, about half of the powder (~43%) is converted to gas and about half (~56%) remains a solid residue in the bore of the firearm. An additional ~1% remains as water. The residue is called fouling (which results in corroding gun barrels).

The common chemical equation for the combustion of black powder is:

- $6 \text{KNO}_3 + \text{C}_7\text{H}_4\text{O} + 2 \text{S} \rightarrow \text{K}_2\text{CO}_3 + \text{K}_2\text{SO}_4 + \text{K}_2\text{S} + 4 \text{CO}_2 + 2 \text{CO} + 2 \text{H}_2\text{O} + 3 \text{N}_2$

D. APA 87-1 Standard Established Limits

The limits on metal in the burst charge powders are outlined in the AP 87-1 Standard. Metallic powder is limited to 130 mg in 1.4G fireworks when the particle size is less than 100 mesh. Burst charge compositions are limited to “black powder (potassium nitrate, sulfur, and charcoal) or *similar pyrotechnic compositions **without metallic fuel***” for 1.3G fireworks (bold and italicized added for emphasis).

Metallic fuels for burst charges may be used un 1.1G shells. For 1.4G Consumer shells, the bursting charge is limited to 25% of the total chemical composition for the single component with a maximum of 15 grams of burst powder. For 1.3G display shells, the limit would be a maximum of 71 grams total, however, with the enforcement of no metals in the aerial shells, many formulators have resorted to non-metallic formations that are much more sensitive and brisant than the tradition black powders, some of which are approaching flash powders.

Under the UN system, Fireworks are covered under a default system. For spherical or cylindrical shells, 1.4G shells may contain no more than 60 grams of pyrotechnic substance with no more than 2% flash composition. For the 1.3G shells, there is a limit of no more than 25% flash composition. This research will look at both traditional break powders as well as the non-metallic high energy formulations.

E. Potential Existing Tests

The literature was examined for potential tests that would help identify methods or tests that would show explosive output (energy profile) of black powder and alternative formulations that would be used as burst powders. Many tests evaluated require expensive equipment to operate. The tests outlined in the UN Recommendations on the Transport of Dangerous Goods; Manual of Test and Criteria, were evaluated to see if one or more of these could be used or adapted to measure and compare explosive characteristics (energy profile) of burst powders.

1. HLS - Test Description and Potential as Burst Charge Equivalency Test

The first test that was identified as potential test to differentiate aerial shell burst charge powders was the HSL Flash Composition Test of Appendix 7 of the UN MTC. However, previous research efforts had demonstrated that the criteria of this test do not distinguish with enough resolution, the differences between black powder and flash powder. Under this previous research, Health & Safety Laboratory (HSL) of the United Kingdom (UK) via Le Maitre (manufacturer) provided SMS with ten firework articles for testing. The firework articles were broken down to remove the energetic compounds (e.g. burst charge, lift charge, stars, Whistle, 4sec composition, etc.) for testing. On each sample, SMS performed three trials of the US Flash Composition test (US DDT), also known as the Deflagration to Detonation test (25 grams each trial), and three trials of the HSL test procedure with modified firing plug (0.5 grams each trial).

The two different tests yielded the same conclusion for each sample as summarized in Table 3 below. In addition, SMS performed both tests on three different GOEX black powder sizes, Fg, FFFg and FFFFg. These results are also provided in the Table 3 as additional data points.

Table 3: Summary Comparison of Flash Composition Test Methods

Item	Sample	HSL Flash Composition?	US DDT Flash Composition?
1	Flash Report No. 2 (PP247)	Yes	Yes
2	Airburst Large Silver Glitter (P609B)	Yes	Yes
3	Super Gunshot Red (LMA201)	No	No
4	15' Sparkburst (PP483)	Yes	Yes
5	Waterfall 15 x 15 (PP357)	Yes	Yes
6	1.5" 50-60' Mine Red (PP568)	No	No
7	Theatrical Flash Large (1200B)	No	No
8	1.5" 50-60' Mine Silver (PP570)	No	No
9	1.5" 50-60' Mine Yellow (PP575)	No	No
10	Whistle, 4sec (PP334)	Yes	Yes
Black Powder Samples			
11	GOEX Fg Black Powder	Yes	No
12	GOEX FFFg Black Powder	Yes	No
13	GOEX FFFFg Black Powder	Yes	No

Based on the dent criteria established, the US DDT test results indicate that black powder is not a flash powder. Results from the HSL tests indicate that black powder would be considered a flash powder. As such the HSL will not achieve the desired results for this project.

Below is a table summarizing the ability of UN Series tests to differentiate between the explosive response of Black Powder and Flash Powder. The reason this is important is because this is essentially what SMS was tasked to do, find a test that would differentiate powders ranging from standard black powder to a flash composition. As mentioned, the HSL test was the first test evaluated as possible solution. Of the twenty-three tests evaluated, three tests were identified as possibilities for distinguishing differing areal shell burst powders:

- Koenen Test
- Ballistic mortar MK.IIId Test
- Trauzl Test

These last two tests were ruled out as potential solutions based on the sample size required and cost of the testing apparatus (Ballistic mortar MK.IIId +\$217,000) and health hazard concerns and accessibility of the test apparatus (Trauzl Test; lead test blocks). Neither of these last two tests match the DOT criteria of a simple field test. The Koenen test was the only test that had the potential as a solution for the desired test type.

Table 4: Potential Tests for Black Powder Equivalency Tests

UN Series Test	Description	Purpose	Comments on Ability of Test to Distinguish between Black Powder and Flash Powder
Appendix 7 – Flash Composition Test	HSL Flash Composition Test	Determine pressure rate of rise	Flash Powder expected to give a very rapid rate of rise when compared to that of black powder however the HSL test resulted in categorizing some black powder as a flash powder.
Appendix 7 – Flash Composition Test	US Flash Composition Test (Modified US DDT)	Determine if powder with flash powder components behaves as a flash powder	Whether or not the witness plate is holed from modified US DDT test can distinguish between flash powder and black powder. However, handling 25 grams of flash powder is a safety risk.
1(a)/2(a)	UN Gap	Determine if substance propagate a detonation	Flash Powder and Black Powder are expected to give a similar result of propagation with zero cards; however, it’s expected that flash powder would react more violently (and hole the witness plate or fragment the pipe completely) with a larger gap. This test was not chosen due to the hazards of handling a significant quantity of flash powder to complete the testing.
1(b)/2(b)/Test E.2/Test E.3	Koenen/ Dutch or US pressure vessel	Determine if substance generates gas rapidly	Different orifice diameters can be used to differentiate gas generation rate or pressure rate of rise with smaller quantities (<5 grams) of the substance. Preliminary results show that the limiting diameter for black powder is near 8 mm where flash powder is near 20mm for Koenen. The Dutch or US pressure vessel may not be as suitable as the chamber may be damaged when the sample (~5 grams as well) of flash powder ignites. The cost and portability of this test setup may prove to be too much.
1(c)/2(c)/HSL Test	Time-Pressure	Determine pressure rate of rise	Flash Powder expected to give a very rapid rate of rise when compared to that of black powder however the HSL test which is very similar to the time-pressure test resulted in categorizing some black powder as a flash powder.
3(a)	Impact	Determine impact sensitivity	Difference expected in whether the sample is consumed (as both are expected to be sensitive) but identifying differences

UN Series Test	Description	Purpose	Comments on Ability of Test to Distinguish between Black Powder and Flash Powder
			between the responses is expected to be difficult and subjective. This test will not provide a comparison of the output or explosive yield of the burst powders.
3(b)	Friction	Determine friction sensitivity	Difference expected in weather the sample is consumed (as both are expected to be sensitive) but identifying differences between the responses is expected to be difficult and subjective. This test will not provide a comparison of the output or explosive yield of the burst powders.
3(c)	Thermal stability	Determine thermal stability	Difference not expected between black powder and flash powder as both are thermally stable.
3(d)	Small-scale burn	Determine outcome from burning	Difference expected with quantification of burn time with video evidence. Handling of significant amounts (~100 grams) of flash powder is not ideal. Test is simple and easily performed as a field test.
4(a)/5(a)/7(a)	Cap sensitivity	Determine if an explosion occurs from a cap initiation scenario	Both are expected to be cap sensitive.
4(b)	12m drop	Determine drop sensitivity	Drop sensitivity not expected to easily differentiate between the two substances
5(b)	USA DDT	Determine if substance can transition from deflagration to detonation from match ignition	Black powder and flash powder are expected to show differences however performing this test is a safety risk as large quantities of flash powder are required.
5(c)/6(c)/7(e)/7(g)	External fire	Determine effects from the ignition of the material.	Effects expected to be similar to the small-scale burn where difference expected with quantification of burn time with video evidence. Handling of significant amounts (>100 grams) of flash powder is not ideal.

UN Series Test	Description	Purpose	Comments on Ability of Test to Distinguish between Black Powder and Flash Powder
5(d) - Removed from current edition	Princess Incendiary	Determine the likelihood of an ignition event from a fuze laid on top of the material	Whether or not the burning fuze ignites the material is not expected to indicate explosive differences between black powder and flash powder.
6(a)	Single Package	Determine if a mass explosion results from ignition	Mass explosion can result for both depending on the amount of material present and the packaging used. Not expected to reliably indicate explosive differences between black powder and flash powder.
6(b)/7(k)	Stack Test	Determine if mass explosion propagates between packages	Similar to the Single Package test
6(d)	Unconfined package	Determine presence of hazardous effects outside the package	Packaging specific and not expected to reliably indicate explosive differences between black powder and flash powder.
7(b)	EIS gap	Determine susceptibility to shock	Similar to UN Gap test
7(c)/7(d)/7(j)/7(l)	Friability/EIS bullet	Determine effects from impact	Black powder and flash powder susceptibility to impact of a cylinder or bullet not expected to reliably differentiate between the explosive output between the two.
7(f)/7(h)	EIS slow cook-off	Determine slow cook-off result	Confined slow heating of both black powder and flash powder are expected to yield similar results of fragmenting the pipe into 3 or more pieces. Additionally, a significant amount of flash powder is used which is a safety concern. The costs of performing this test is too prohibitive for it to be considered as a candidate.
Test A.1/A.2	BAM 50/60 or TNO 50/70 steel tube test	Determine ability of substance to propagate a detonation	Similar to the Koenen test except with a booster and a much larger tube. Both black powder and flash powder are expected to fail the test.

UN Series Test	Description	Purpose	Comments on Ability of Test to Distinguish between Black Powder and Flash Powder
Test F.1/F.2	Ballistic mortar MK.IIID test	Determine the explosive power of a substance	Differences in the swing distance resulting from the initiation of each substance is expected between the black powder and the flash powder. It is unclear if this would be a good test to easily distinguish between black powder and flash powder.
Test F.3/F.4	Trauzl Test	Used to measure the explosive power of a substance	Volume of the cavity left by cap initiating a small sample of the explosive is evaluated. This test could be used to differentiate between black powder and flash powder however handling lead is undesirable. A copper block could be used as a replacement.

2. Koenen Test - Test Description and Potential as Burst Charge Equivalency Test

After reviewing the HLS results and other potential current test methods, the focus of the best possibility based on the burst powder and configuration was the Koenen Test. This was mainly due to the test setup configuration being very similar to an aerial shell in that the burst powder is initially confined when ignited and then as it ramps up it eventually ruptures the shell casing. The Koenen test behaves in a similar manner in that the material is heated to auto-ignition and, when ignited, the Koenen tube may rupture if the orifice size is not large enough to allow the combustion pressure to relieve.

The Koenen test is used to determine the sensitiveness of solid and liquid substances to intense heat under high confinement. This test utilizes a one-inch diameter by three-inch long, closed-end, metal tube to hold the sample (Photo 25). The tube is deep drawn from DC04, A620, or SPEN sheet steel; 26.5 ± 1.5 grams mass, $75 \text{ }^\circ\text{C}$ 0.5mm length, $0.5 \pm 0.05\text{mm}$ wall, and 30 ± 3 MPa quasi-static bursting pressure. The sample is loaded into the tubes in three equal increments with tamping until the tube is filled to 60mm. Liquids are loaded into the tube to a height of 60mm. The tube is assembled into a reusable closing device (threaded nut and collar) and an orifice plate installed on the open end of the tube (Photo 26).



Photo 25: Koenen Tubes and Orifices



Photo 26: Tube/Orifice Assembled

The tube assembly is placed in the heating and protective device, the test area vacated, burners remotely lit, providing a calibrated heating rate to the sample by propane burners located at four locations around the tube for at least 5 minutes or until the tube ruptures. NOTE: The heating rate is calibrated to $3.3 \pm 0.3^\circ\text{K/sec}$ using the time for 27 cm^3 of dibutyl phthalate to rise from 135°C to 285°C as measured by a 1mm thermocouple placed 43mm below the rim of the tube through a 1.5mm orifice plate (the rise time must be between 41.7 - 50.0 seconds). The sample tube is examined to determine whether an explosion occurred. The limiting diameter of a substance is the largest diameter of the orifice at which the result “explosion” is obtained.

Varying the orifice plate over the top of the sample tube changes the degree of confinement of the sample. The orifice sizes are reduced until an explosion effect occurs, as defined by the rupture pattern, or the substance passes the test with the smallest orifice. Indication that the material is an explosive occurs if the orifice size causing the tube to rupture into multiple fragments is 2 mm or greater. The orifice sizes range from 1 mm up to 22 mm.

After each trial, the fragments of the tube, if any, are collected and weighed. The following effects are differentiated:

- "O": Tube unchanged;
- "A": Bottom of tube bulged out;
- "B": Bottom and wall of the tube bulged out;
- "C": Bottom of tube split;
- "D": Wall of tube split;
- "E": Tube split into two fragments;
- "F": Tube fragmented into three or more mainly large pieces which in some cases may be connected with each other by a narrow strip;
- "G": Tube fragmented into many mainly small pieces, closing device undamaged; and
- "H": Tube fragmented into many very small pieces, closing device bulged out or fragmented.

If a trial results in any of the effects "O" to "E", the result is regarded as "no explosion". If a trial gives the effect "F", "G" or "H", the result is evaluated as "explosion". The test is performed three times at the lowest orifice size that the result "no explosion" is observed. The limiting diameter (LD) is the largest orifice diameter at which the result "explosion" is obtained. The orifice sizes are reduced until an explosion effect occurs or the substance passes the test with the smallest orifice (1.0mm).

The result is considered positive for the UN Series 2 (b) test and the substance to show a violent effect on heating under confinement if the LD is 2.0mm or more. The result is considered negative for the UN Series 2 (b) test and the substance to show no violent effect on heating under confinement if the LD is less than 2.0mm.

The Koenen apparatus is shown in Photo 27 and a Koenen tube with orifice is shown in Photo 28.



Photo 27: Koenen Apparatus

An orifice plate with a (1.0-mm orifice) and cap was placed over each filled tube, as shown in the photo below.



Photo 28: Representative Prepared Sample for Koenen Test

The Koenen Test was thus selected as a candidate for further evaluation as a method for distinguishing aerial shell burst powder strengths.

IV. TEST DESCRIPTIONS & RESULTS

A. Modified UN Test Series 1 (c) Koenen Test

1. Test Description

Initially, a Modified UN Test Series 1 (c) was performed and the Koenen tube was utilized to compare burst powders. The Koenen test utilizes a steel test tube deep drawn from sheet steel conforming to specification DC04 (EN 10027-1), or equivalent A620 (AISI/SAE/ASTM), or equivalent SPCEN (JIS g 3141). The open end of the tube is flanged. The closing plate with an orifice, through which the gases from the decomposition of the burst powders escape, is made from heat-resisting chrome steel (Photos 29 & 30) . A steel threaded collar and nut hold the orifice in place during the test. If the orifice is not of sufficient size, the gases rupture, burst, or fragment the tube. Orifices are available with the following diameter holes: 1.0 – 1.5 – 2.5 – 3.0 – 5.0 – 8.0 – 10.0- 12.0 – 15.0 – 18.0 – 20.0 – 22.0 mm.



Photo 29: Koenen Tubes and Orifices



Photo 30: Tube/Orifice Assembled

2. Determination of Sample Size – Modified Koenen with 4 Burner Ignition Source

a. Test Configuration

The dimensions of the tubes are provided in Figure 7. Normally the sample is placed into the tube up to a depth of 60 mm. This equates to masses from 25-45 grams depending on density of the sample. However, a full tube of burst powders would rupture the tube every time even with the largest orifice size. To adjust for this, different quantities of black powder were evaluated to determine how much powder could be placed in the tube without rupturing the tube but allowing for the full range of orifice sizes (~3 mm to 22 mm orifices) to be utilized so as to get the best resolution between powders that were not achievable in the HSL Test.

The desire of the tests was to find the sample size that would equate the fastest burning powders (flash powders) to a limiting diameter (LD) of 22 mm orifices or greater and equate black powder, the selected baseline powder, to down around a 5-10 mm orifice value for the LD. With this range, it should be relatively easy to identify/discern powders that behaved similar to black powder and those that were approaching, or in and of themselves were, flash compositions.

Substances or mixtures that are more energetic and have burn rates that are relatively fast will require orifices that are much larger than substances that have slower burn rates. If the venting or orifice hole is sufficiently large, the material will burn within the Koenen tube and vent out the specified orifice hole without bursting the tube. If the venting or orifice hole is insufficient, then the Koenen tube will explode and fragment.

The UN criteria for the standard Koenen Test of three or more fragments is the indicator for a failure or an explosion along with a myriad of visual effects of the tube. To allow for a more simplistic/

field test, it was decided with PHMSA input that any rupture of the tube would constitute a failure or be considered a “+” effect, or “explosion”. Tubes the were bulged or visually undamaged would be considered “-”.

In general, substances that require a larger orifice size such that the Koenen tube does not explode, have a much faster burn rate than those substances that do not explode with smaller orifice diameters.

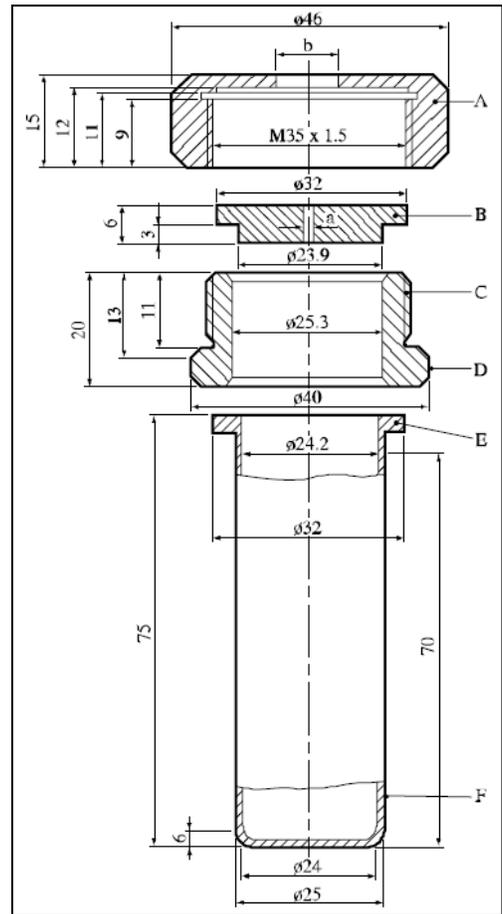


Figure 7: Koenen Tube (mm)

The following table contains the initial screening test results. Based on SMS’s previous experience with performing the Koenen test, 5 grams appeared to be a reasonable amount of powder and a quantity that could be handled safely while placing into a confined test setup and the initial trials were targeted around this sample size. These initial trials were used to help determine the sample size that would be used throughout the project (i.e., 3 grams, 5 grams, or 7 grams per trial). Goex FFFFg (or “4Fg”) Black Powder (BP) was selected as the baseline sample due to its availability and high-precision manufacturing. Consideration for which quantity per trial would be used, was based off (1) the range of orifices available after running the baseline 4Fg black powder (at the various quantities), and (2) material availability. Additional formulations were used at this time including a flash composition, smokeless powder, and the Goex BP with added aluminum powder in the form of German Black to help ensure that these sample sizes were going to provide the desired range of data. German Black is an aluminum powder with a particle size of ~ 5 micron in size. The German Black aluminum (~5micron) (Photo 32) samples were manually mixed with the 4Fg Black Powder. The only changes at this point from the standard Koenen Test were the sample size down to 3 to 7 grams and the test criteria (any rupture of the tube considered a “+” result. Photos 32-41 show the standard setup.

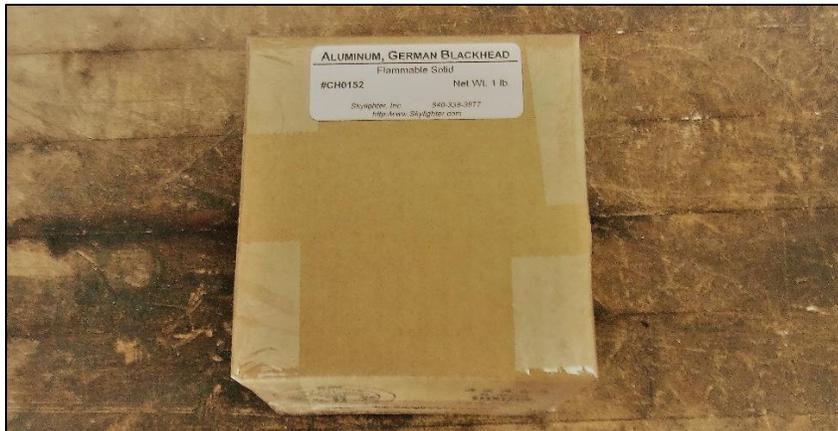


Photo 31: German Black Aluminum Powder (~5micron)

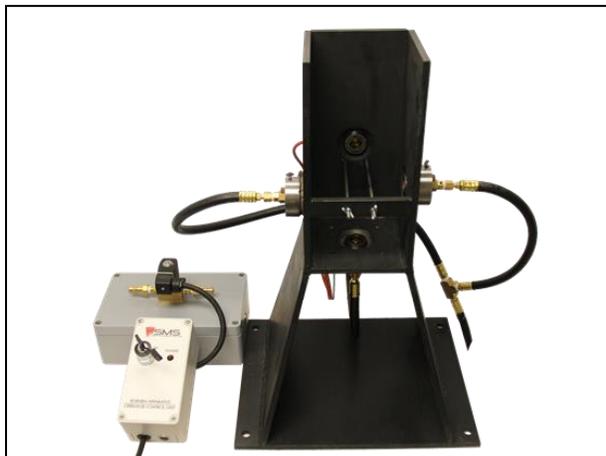


Photo 32: Standard 4 Burner Ignition Source Setup

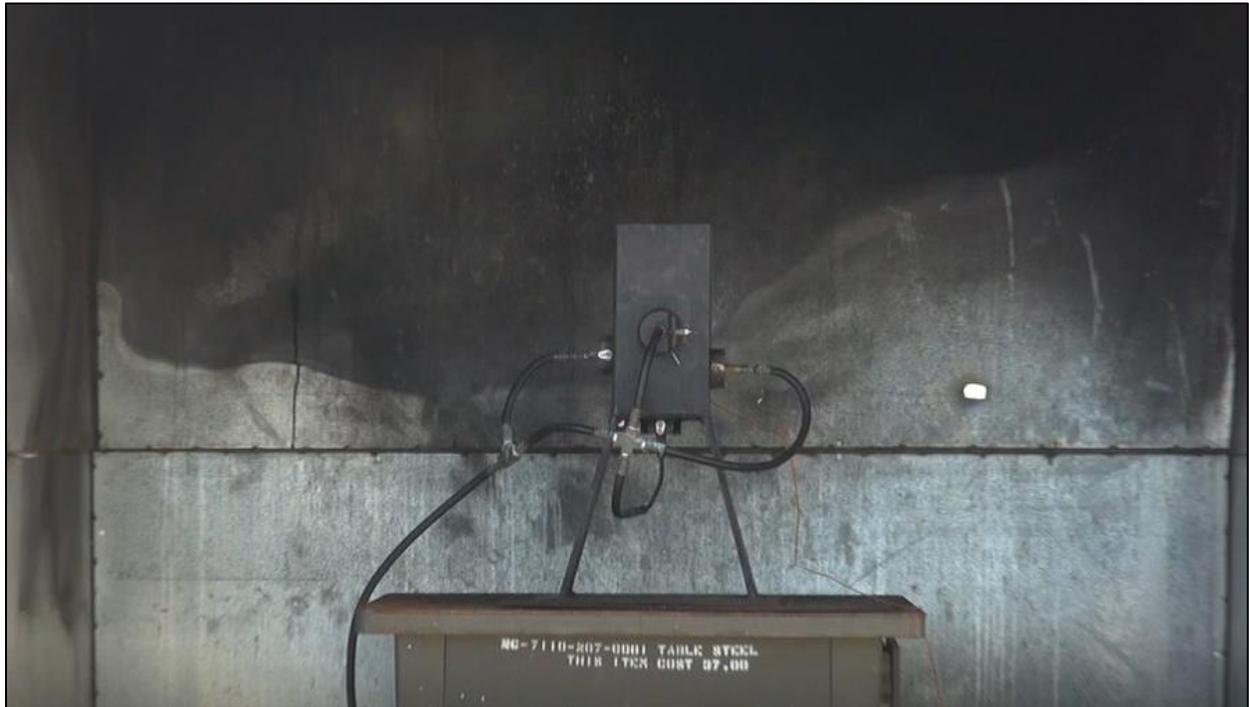


Photo 33: Standard 4 Burner Ignition Source Setup

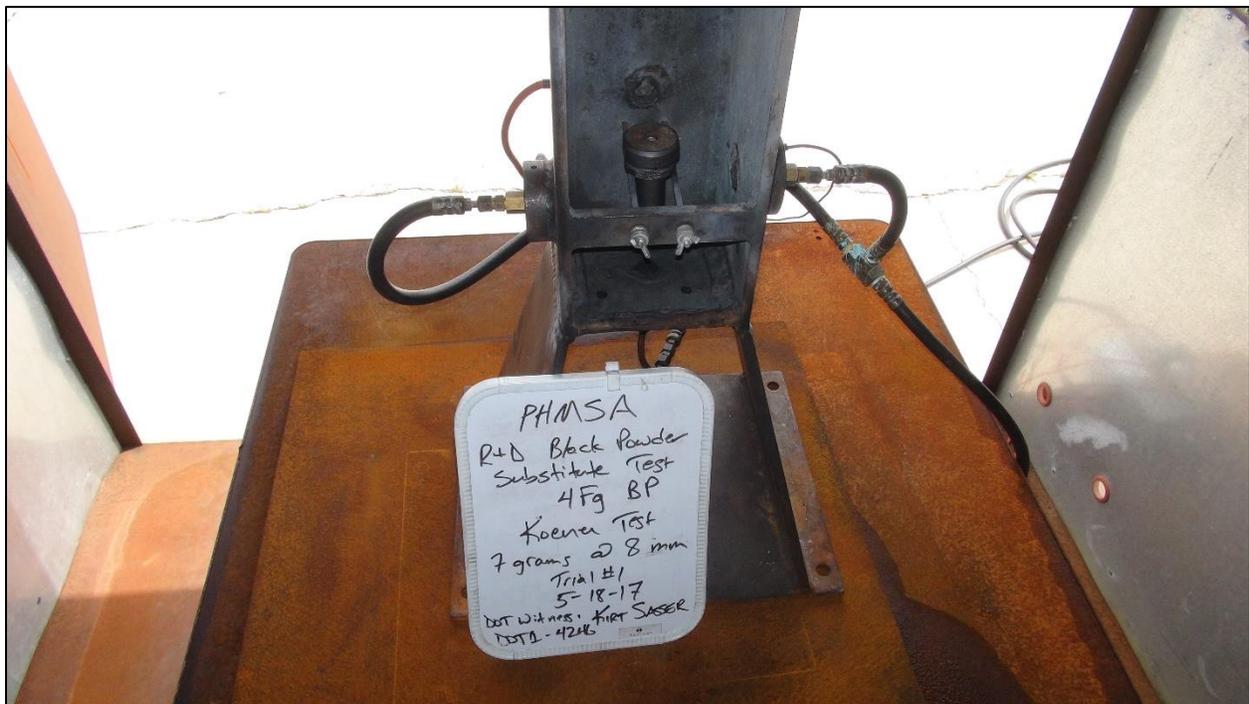


Photo 34: Standard 4 Burner Ignition Source Setup

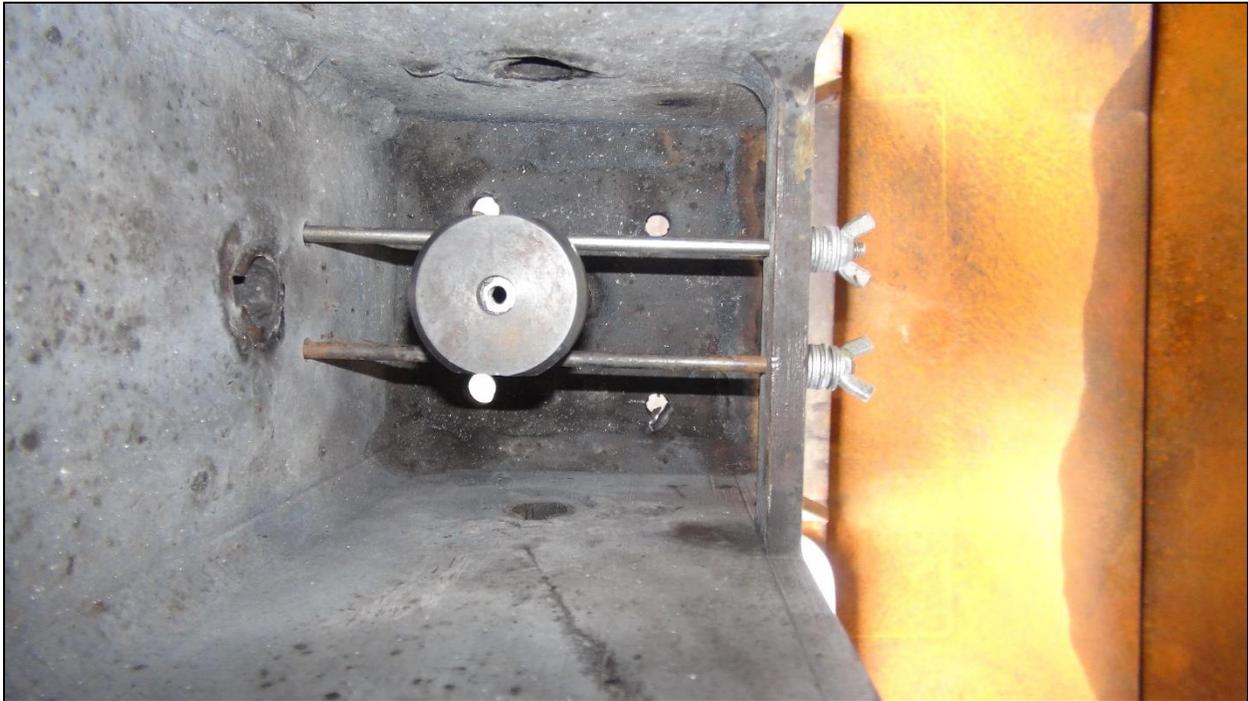


Photo 35: Standard 4 Burner Ignition Source Setup

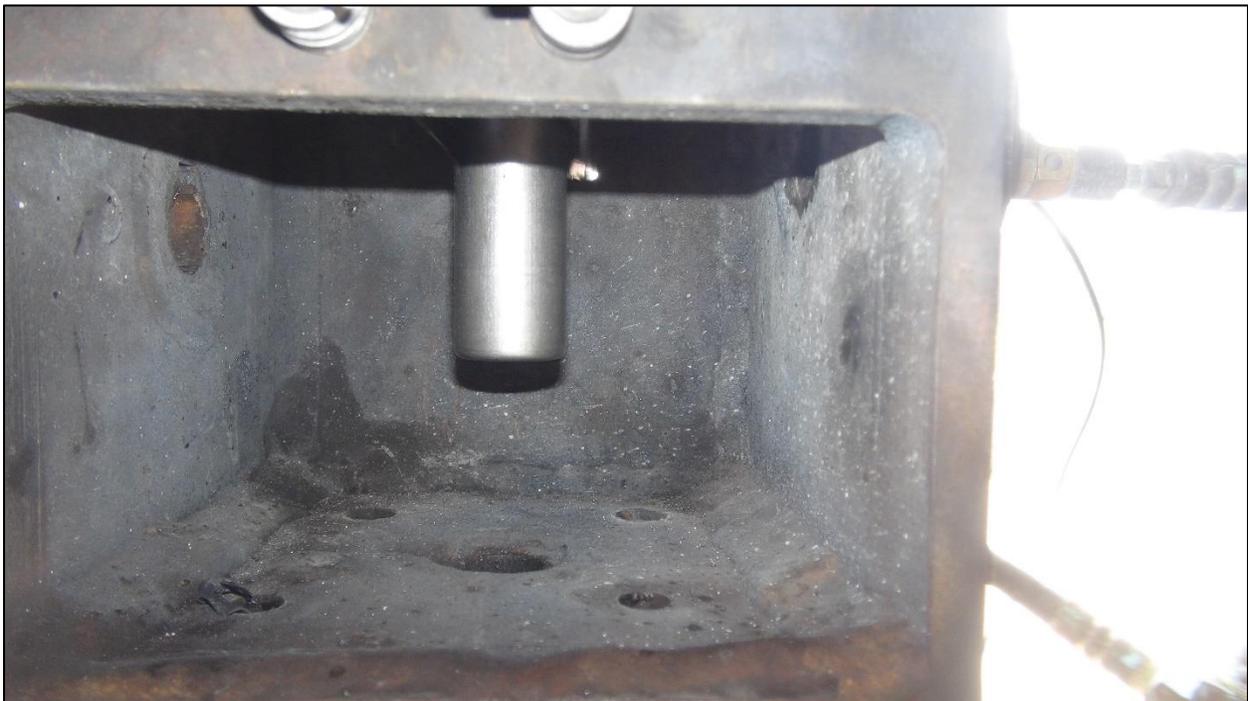


Photo 36: Standard 4 Burner Ignition Source Setup



Photo 37: Standard 4 Burner Ignition Source Setup



Photo 38: GOEX 4Fg + German Black (94%/6%)



Photo 39: Canister Shell #1 (Burst Powder)



Photo 40: Canister Shell #2 (Burst Powder)



Photo 41: Canister Shell #3 (Burst Powder)

b. Test Results

It was quickly evident that samples sizes of less than 5 grams would put results of the established baseline powder too low (failures at 3mm) and 7 grams would push the baseline powder too high. As such, a sample size of 5 grams was selected to be used for the remaining test. Table 5 shows the results of this initial modified Koenen test with the ignition source of the standard 4 gas burner system which would increase the sample to its autoignition temperature, at which time the sample would ignite and burn.

Table 5: Koenen Preliminary Screening Test Results – Standard 4 Burner Ignition Source

<u>Material</u>	<u>Orifice Size</u>	<u>Results</u>	<u>Pass/Fail</u>
3 g - FFFFg Goex Black Powder	3mm	“+”	Fail
	5mm	“-“	Pass
5 g - FFFFg Goex Black Powder	5mm	“+”	Fail
	8mm	“-“	Pass
	10mm	“-“	Pass
7 g –FFFFg Goex Black Powder	8mm	“+”	Fail
	10mm	“-“	Pass
5 g – Pure Flash Comp	10mm	“+”	Fail
	20mm	“+”	Fail
5 g – AR2210 (flaked smokeless powder)	5mm	“+”	Fail
	10mm	“-“	Pass
5 g – 94% FFFFg/ 6% 5μ Al. Powder (German Black)	8mm	“+”	Fail
	10mm	“+”	Fail
	15mm	“-“	Pass
	20mm	“-“	Pass
7 g – 94% FFFFg/ 6% 5μ Al. Powder (German Black)	18mm	“-“	Pass
5 g – 97% FFFFg/ 3% 5μ Al. Powder (German Black)	8mm	“-“	Pass
	12mm	“-“	Pass
5 g – Canister Shell #1 (Burst Powder)	20mm	“+”	Fail
5 g – Canister Shell #2 (Burst Powder)	20mm	“-“	Pass
5 g – Canister Shell #3 (Burst Powder)	20mm	“-“	Pass

Photos 42-50 show examples of the test results.



Photo 42: GOEX 4Fg @ 8 mm Vent (5 Grams) – PASS

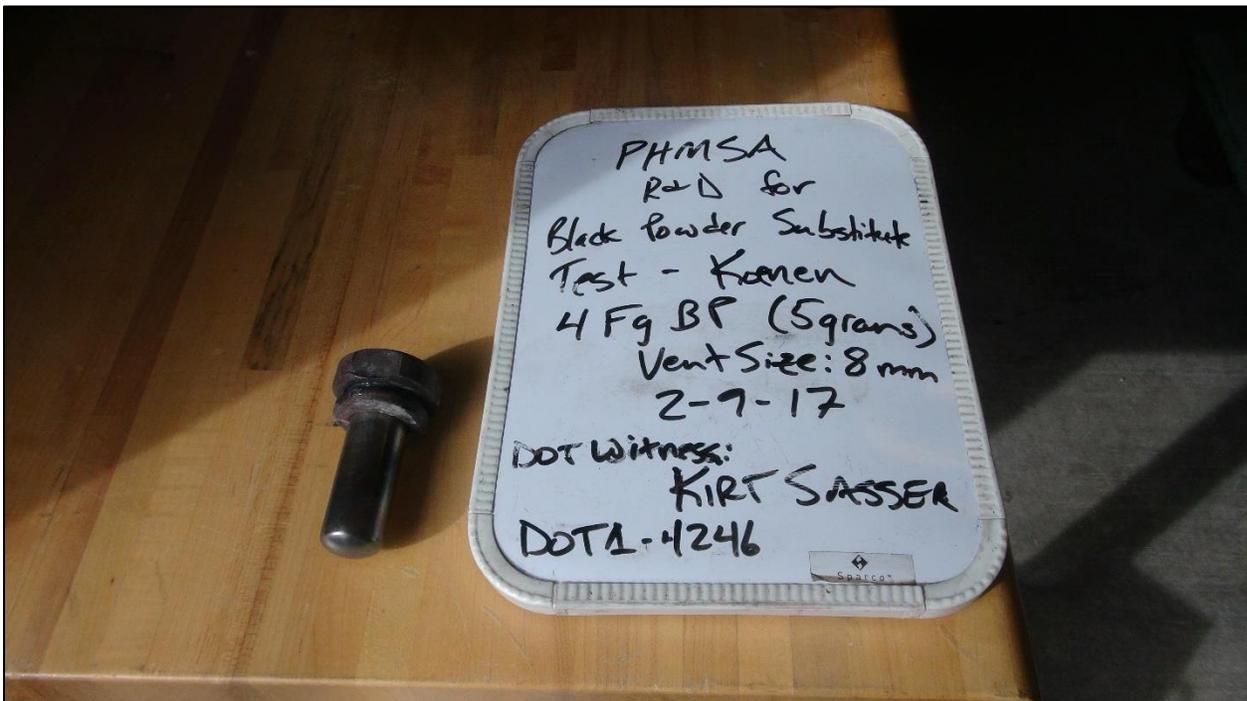


Photo 43: GOEX 4Fg @ 8 mm Vent (5 Grams)

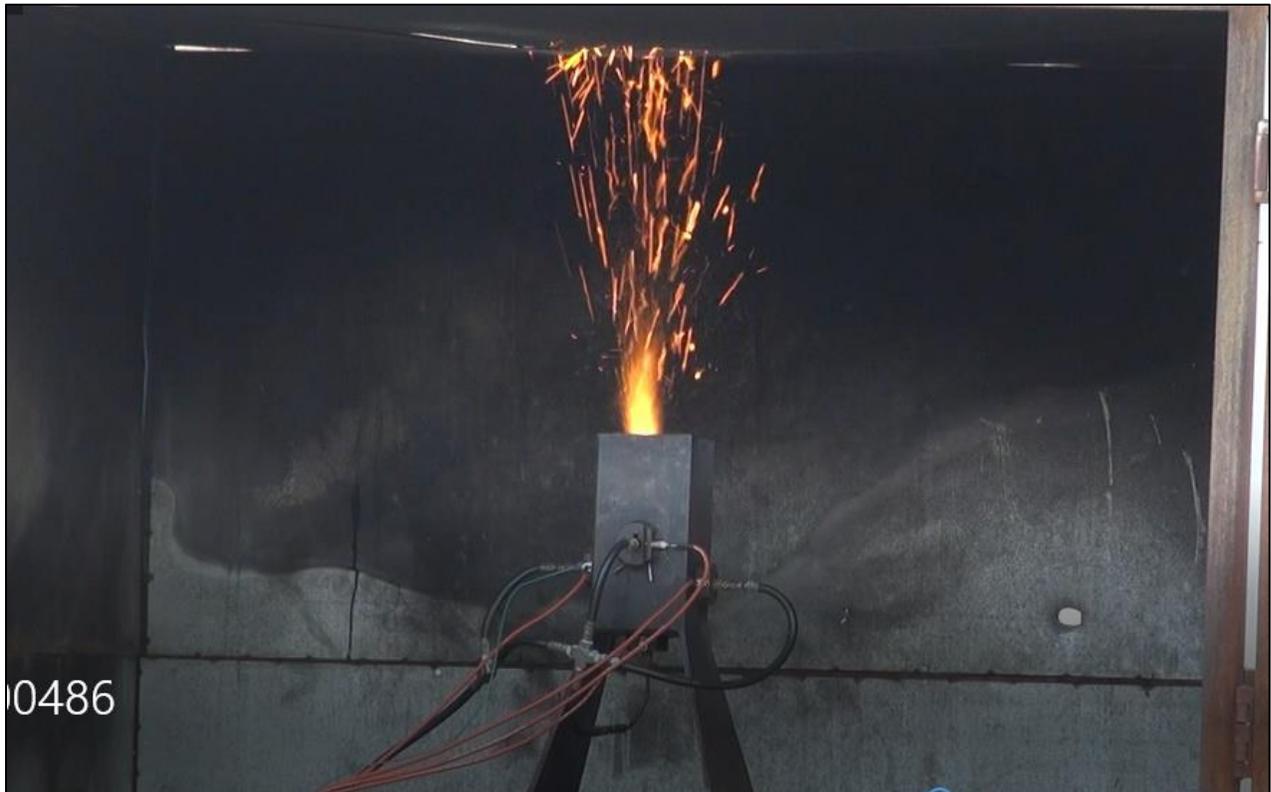


Photo 44: AR2210 @ 8 mm Vent (5 Grams)

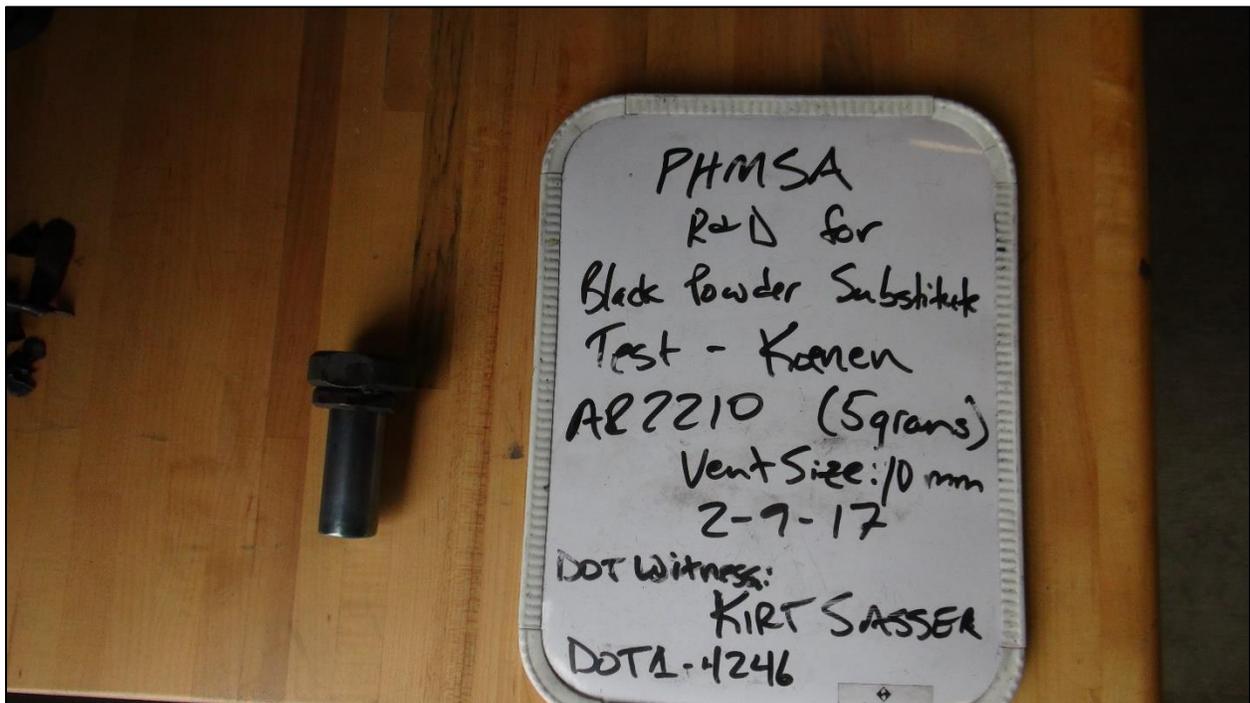


Photo 45: AR2210 @ 10 mm Vent (5 Grams)

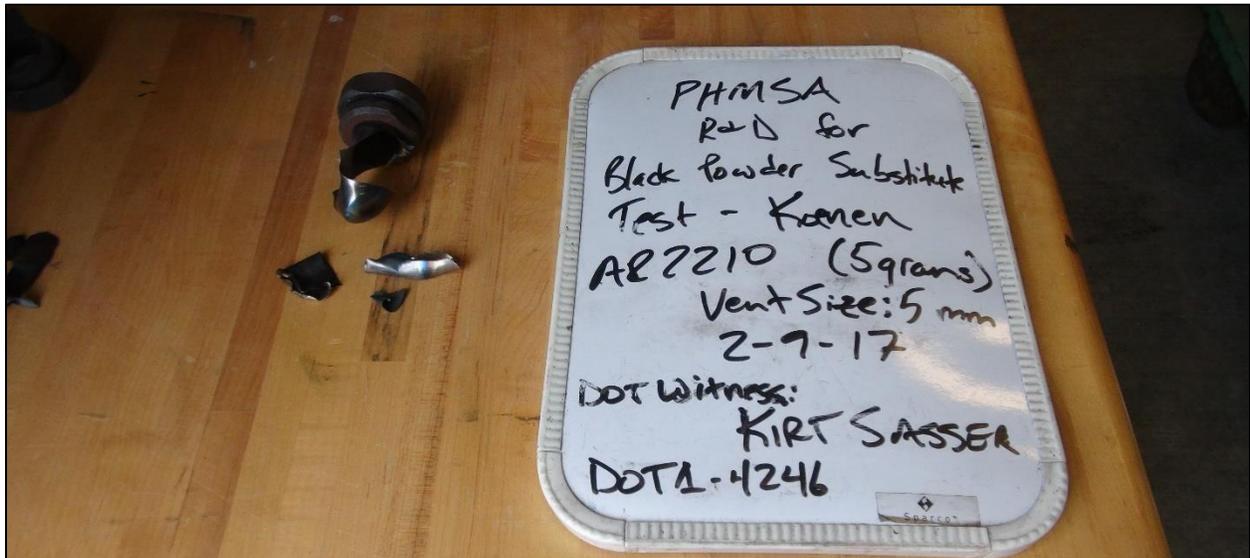


Photo 46: AR2210 @ 5 mm Vent (5 Grams)



Photo 47: GOEX 4Fg + German Black (94/6) @ 15 mm Vent (5 Grams)



Photo 48: GOEX 4Fg @ 8 mm Vent (7 Grams)



Photo 49: Flash Comp @ 20 mm Vent (5 Grams)

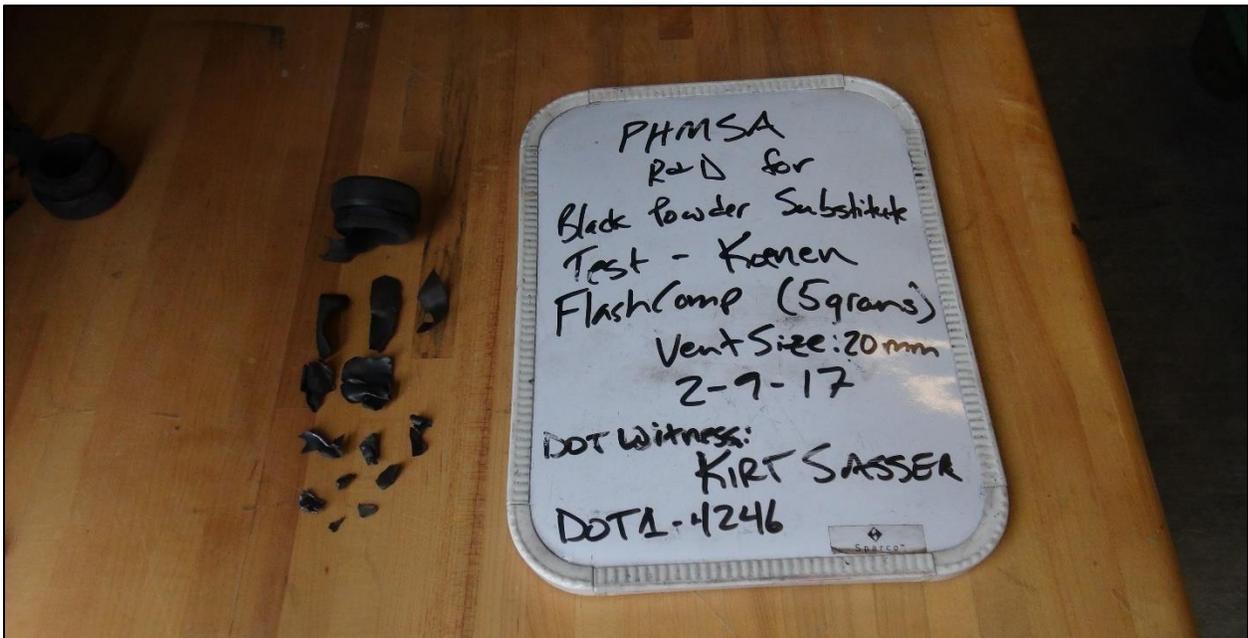


Photo 50: Flash Comp @ 20 mm Vent (5 Grams)

c. Sample Size Determination

After the initial screening tests were completed, 5 grams of material per trial was selected as the sample size for testing of burst powders to meet the aforementioned considerations.

d. Initial Conclusions

Initial results indicated that this method is promising prospect as a test to differentiate between burst powders. The sample sizes for each trial will be 5 grams. Black Powder appeared to have a limiting diameter of around 5mm. One item that had yet to be addressed, was that the standard Koenen apparatus was not exactly a field test and might not be practical or inexpensive to perform. Additional modification would be required to make this a simple, field test.

Table 6 provides the estimated limiting diameter based on the testing performed using the 4 Burner Ignition Source. This test method allows a very easy comparison of the powders to be made. The powders arranged in ascending order based on the limiting diameter.

Table 6: U.S. Based Break Powder Compositions Limiting Diameters

<u>Material</u>	<u>Limiting Diameter (mm)</u>
3 g - FFFFg Goex Black Powder	≥3
5 g - FFFFg Goex Black Powder	≥5
5 g – 97% FFFFg/ 3% 5μ Al. Powder (German Black)	<8
7 g –FFFFg Goex Black Powder	8
5 g – AR2210 (flaked smokeless powder)	<10
5 g – 94% FFFFg/ 6% 5μ Al. Powder (German Black)	<15
7 g – 94% FFFFg/ 6% 5μ Al. Powder (German Black)	<18
5 g – Canister Shell #2 (Burst Powder)	<20
5 g – Canister Shell #3 (Burst Powder)	<20
5 g – Pure Flash Comp	>20
5 g – Canister Shell #1 (Burst Powder)	>20

3. Additional Trials to Establish 4Fg Black Powder Baseline

Additional testing was required to establish the baseline criteria for 4Fg Goex BP as well as continue gathering data on other potential burst powder formulations.

a. Test Configuration

The standard four burner ignition source setup was used and the Threshold Initiation Levels (TIL) for the limiting diameters were determined for the black powder samples. Threshold initiation levels (TILs) is a statistical method to provide a simple and direct comparison of

materials for easy ranking. This additional testing also helps in determining how the final test sequence will be performed (i.e., how many trials will be required to be performed). The TIL utilizes a series of test dependent, standard levels for testing the material. The test is performed at a predetermined level (orifice sizes). If the event results in a “go” reaction, the test is redone at the next lower level. If the event produces a “no-go” reaction, the test is redone at the same level. The level which produces 20 consecutive “no-go” results with at least one “go” at the next higher level is the TIL. The TIL provides statistical confidence in the established limiting diameter levels. TILs were produced for the 3-gram and 5-gram, 4Fg black powder sample sizes.

In this set of tests, various aluminum powder samples were manually mixed with the FFFFg Black Powder until the sample appeared homogenous (~5micron) (Photos 51 and 52). This excise was done to try to answer the question of what happens if metal powder is added to the burst charge powders as analytical data seemed to show that small particle size metals, particularly aluminum, were showing up in the burst powders of aerial shells. The following powders were tested in this phase of the project:

- FFFFg Goex Black Powder
- 95% FFFFg, 5% (5 μ Al. Powder)
- 95% FFFFg, 5% (0.88-0.105mm Al. Powder)
- 95% FFFFg, 5% (0.125-0.149mm Al. Powder)
- 90% FFFFg, 10% (5 μ Al. Powder)
- 90% FFFFg, 10% (0.88-0.105mm Al. Powder)
- 90% FFFFg, 10% (0.125-0.149mm Al. Powder)
- 85% FFFFg, 15% (5 μ Al. Powder)
- 85% FFFFg, 15% (0.88-0.105mm Al. Powder)
- 85% FFFFg, 15% (0.125-0.149mm Al. Powder)

Photos 53 and 54 show a few of the samples used in this phase of testing.



Photo 51: German Black Aluminum Powder (~5micron)



Photo 52: German Black Aluminum Powder (~5micron)



Photo 53: 90% 4Fg / 10% Aluminum Powder (~5micron)

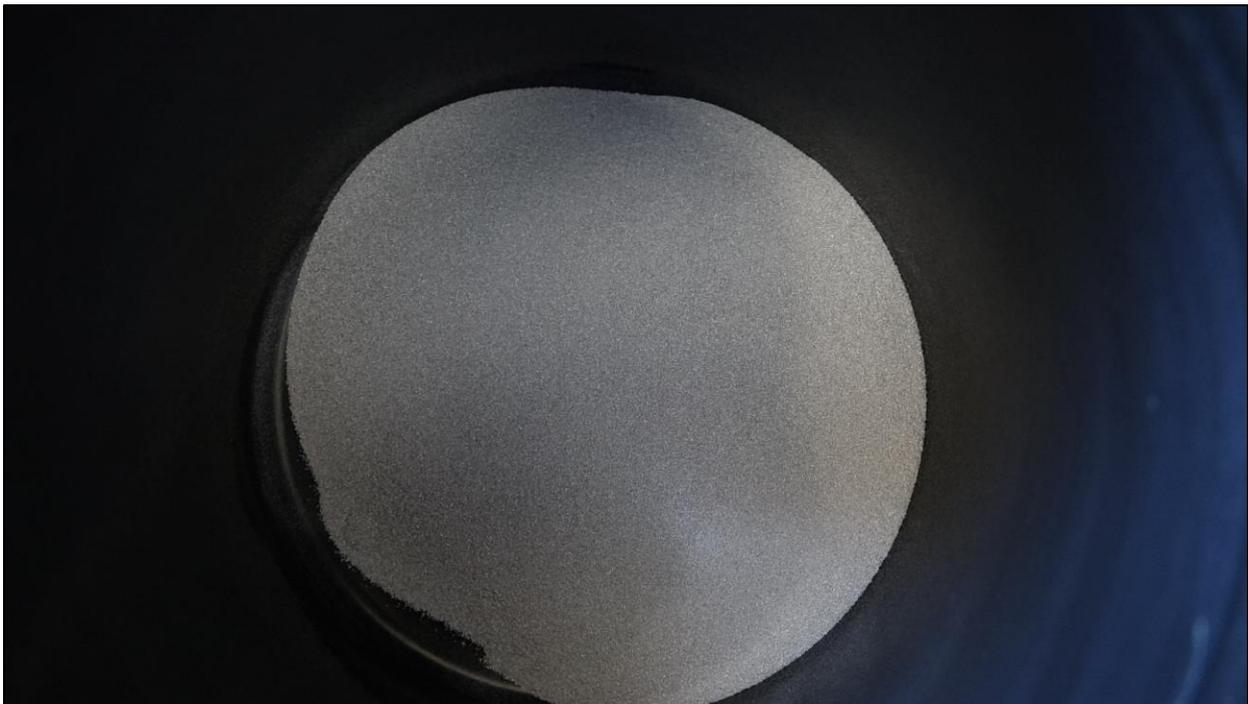


Photo 54: 90% 4Fg / 10% Aluminum Powder (0.88-0.105 um)

b. Test Results

The following table includes the test results using the Koenen Apparatus and the standard four burner ignition source. For each trial, the time from ignition to the detonation was recorded. Additionally, the Koenen tubes were weighed pre-test and post-test. It was initial thought that this data might assist in determining the pass/fail criteria. However, it should be noted that tube weight cannot be a criterion as the tubes were almost always contaminated with test material residue after each trial that could not be easily removed. In general, the Koenen tubes weighed more after each test trial due to this residue.

Table 7: Koenen Test Results – Ignition Mode: Auto-Ignition – Black Powder Compositions

<u>Material</u>	<u>Metal Content (%)</u>	<u>Orifice Size</u>	<u>Trial</u>	<u>Results</u>	<u>Pass/Fail</u>
3 g - FFFFg Goex Black Powder	0	3mm	1	"+"	Fail
	0	5mm	1	"-"	Pass
			2	"-"	Pass
			3	"-"	Pass
			4	"-"	Pass
			5	"-"	Pass
			6	"-"	Pass
			7	"-"	Pass
			8	"-"	Pass
			9	"-"	Pass
			10	"-"	Pass
			11	"-"	Pass
			12	"-"	Pass
			13	"-"	Pass
			14	"-"	Pass
			15	"-"	Pass
			16	"-"	Pass
			17	"-"	Pass
			18	"-"	Pass
			19	"-"	Pass
			20	"-"	Pass

<u>Material</u>	<u>Metal Content (%)</u>	<u>Orifice Size</u>	<u>Trial</u>	<u>Results</u>	<u>Pass/ Fail</u>
5 g - FFFFg Goex Black Powder	0	5mm	1	"+"	Fail
			2	"+"	Fail
	0	8mm	1	"+"	Fail
			2	"_"	Pass
	0	10mm	1	"_"	Pass
			2	"_"	Pass
			3	"_"	Pass
			4	"_"	Pass
			5	"_"	Pass
			6	"_"	Pass
			7	"_"	Pass
			8	"_"	Pass
			9	"_"	Pass
			10	"_"	Pass
			11	"_"	Pass
			12	"_"	Pass
			13	"_"	Pass
			14	"_"	Pass
			15	"_"	Pass
			16	"_"	Pass
17			"_"	Pass	
18			"_"	Pass	
19	"_"	Pass			
20	"_"	Pass			
0	0	12mm	1	"_"	Pass
5 g – 95% FFFFg	5% (5μ Al. Powder)	8mm	1	"+"	Fail
			2	"_"	Pass
	5% (5μ Al. Powder)	10mm	1	"_"	Pass
			2	"_"	Pass
			3	"_"	Pass

Material	Metal Content (%)	Orifice Size	Trial	Results	Pass/ Fail
5 g – 95% FFFFg	5% (0.88-0.105mm Al. Powder)	5mm	1	“+”	Fail
			2	“-”	Pass
	5% (0.88-0.105mm Al. Powder)	8mm	1	“-”	Pass
			2	“-”	Pass
			3	“-”	Pass
	5 g – 95% FFFFg	5% (0.125-0.149mm Al. Powder)	5mm	1	“+”
2				“-”	Pass
5% (0.125-0.149mm Al. Powder)		8mm	1	“-”	Pass
			3	“-”	Pass
5 g – 90% FFFFg	10% (5 μ Al. Powder)	8mm	1	“+”	Fail
			2	“-”	Pass
	10% (5 μ Al. Powder)	10mm	1	“-”	Pass
			3	“-”	Pass
	10% (5 μ Al. Powder)	12mm	1	“-”	Pass
5 g – 90% FFFFg	10% (0.88-0.105mm Al. Powder)	5mm	1	“+”	Fail
			2	“-”	Pass
	10% (0.88-0.105mm Al. Powder)	8mm	1	“-”	Pass
			3	“-”	Pass
	10% (0.88-0.105mm Al. Powder)	10mm	1	“-”	Pass
5 g – 90% FFFFg	10% (0.125-0.149mm Al. Powder)	5mm	1	“+”	Fail
			2	“-”	Pass
	10% (0.125-0.149mm Al. Powder)	8mm	1	“-”	Pass
			3	“-”	Pass
5 g – 85% FFFFg	15% (5 μ Al. Powder)	5mm	1	“+”	Fail
			2	“-”	Pass
	15% (5 μ Al. Powder)	8mm	1	“-”	Pass
			3	“-”	Pass

<u>Material</u>	<u>Metal Content (%)</u>	<u>Orifice Size</u>	<u>Trial</u>	<u>Results</u>	<u>Pass/Fail</u>
5 g – 85% FFFg	15% (0.88-0.105mm Al. Powder)	5mm	1	“+”	Fail
	15% (0.88-0.105mm Al. Powder)	8mm	1	“-”	Pass
			2	“-”	Pass
			3	“-”	Pass
5 g – 85% FFFg	15% (0.125-0.149mm Al. Powder)	5mm	1	“+”	Fail
	15% (0.125-0.149mm Al. Powder)	8mm	1	“-”	Pass
			2	“-”	Pass
			3	“-”	Pass

Photos 55 and 56 show examples of the test results.

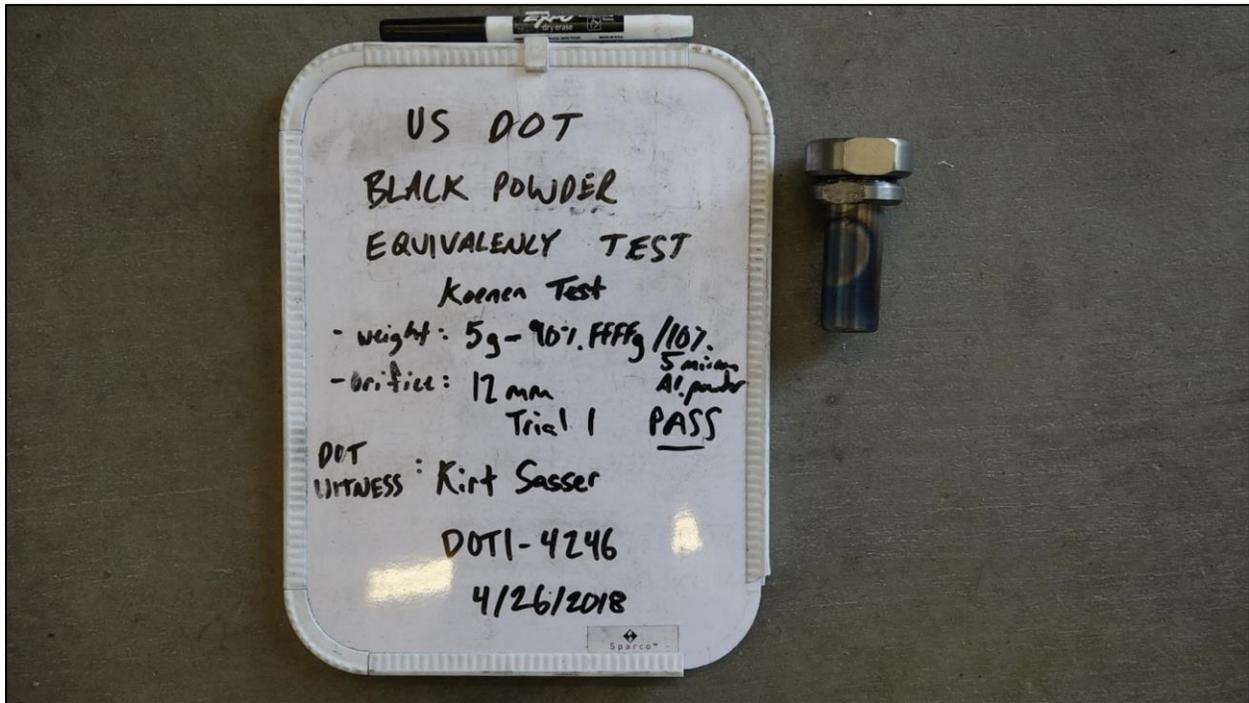


Photo 55: 90% 4Fg / 10% Aluminum Powder (~5micron) @ 12 mm Vent (5 Grams) – PASS

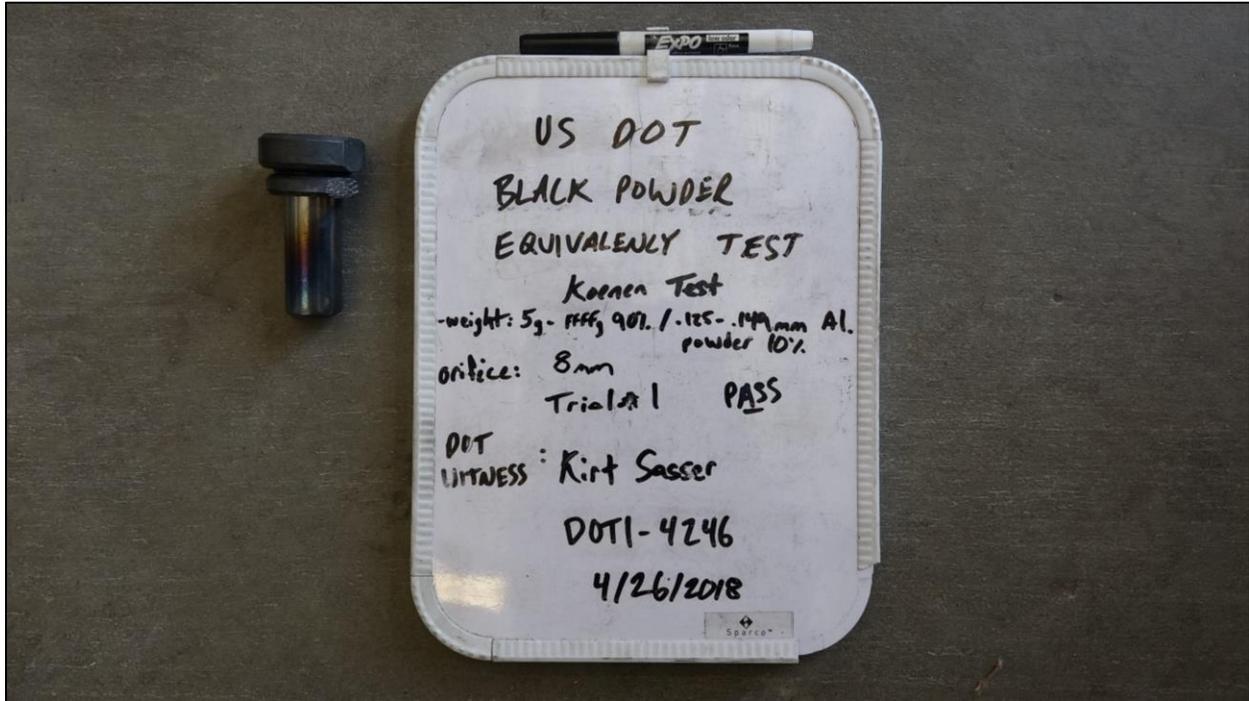


Photo 56: 90% 4Fg /10% Aluminum Powder (0.125-0.149 um) @ 8 mm Vent (5 Grams) – PASS

c. Conclusions

Table 2 results indicate that the mixture of 4Fg with various particle size aluminum powders, do not enhance the 4Fg black powder. In fact, the opposite occurs where it appears that the higher percentages of aluminum powder desensitizes the 4Fg Black Powder (refer to 4Fg TIL of 10mm needed for Pass Criteria vs. only 8mm needed for Pass Criteria for aluminum enhanced 4FG powder). This is when aluminum powder is simply mixed in with black powder. If the aluminum powder is considered as part of the formulation design, there may be a different result.

In addition, the results of these tests seem to suggest that the TIL level of number of trials to determine a level is an adequate termination point. The TIL for a sample of size of 5 grams for the baseline 4Fg black powder was 10 mm orifice size, corresponding to a limiting diameter of 8 mm. For the 3-gram sample size the TIL was 5 mm, corresponding to a limiting diameter for 3 mm. The purpose of the performing the TILs was to determine if the standard 3 trials with no tube rupture would be an acceptable indicator for these tests. Based on the results of this test, three trials are adequately predicting the limiting diameter or equivalency level.

Table 8 provides the estimated limiting diameter based on the testing performed using the 4 Burner Ignition Source. This test method allows a very easy comparison of the powders to be made. The powders arranged in ascending order based on the limiting diameter.

Table 8: U.S. Based Break Powder Compositions Limiting Diameters

<u>Material</u>	<u>Limiting Diameter (mm)</u>
3 g - FFFFg Goex Black Powder	3
5 g – 95% FFFFg 5% (0.88-0.105mm Al. Powder)	5
5 g – 95% FFFFg 5% (0.125-0.149mm Al. Powder)	5
5 g – 90% FFFFg 10% (0.088-0.105mm Al. Powder)	5
5 g – 90% FFFFg 10% (0.125-0.149mm Al. Powder)	5
5 g – 85% FFFFg 15% (5 μ Al. Powder)	5
5 g – 90% FFFFg 10% (0.088-0.105mm Al. Powder)	5
5 g – 85% FFFFg 15% (0.125-0.149mm Al. Powder)	5
5 g - FFFFg Goex Black Powder	8
5 g – 95% FFFFg, 5% (5 μ Al. Powder)	8
5 g – 90% FFFFg 10% (5 μ Al. Powder)	8

4. Whistle Composition Testing

After establishing baselines with the commercial black powder and establishing that the simple addition of metal powder by weight percent did not cause the powders to burn more violently, SMS conducted Koenen testing on a couple of “whistle compositions”, which are known to burn at faster speeds with the 4-Burner Ignition System setup.

a. Test Configuration – Whistle Comps

The standard four burner ignition source setup was used to test a few whistle composition. The composition of the whistle powders tested are provided in Table 9.

Table 9: Whistle Composition Constituents

<u>Name</u>	<u>Chemical Constituent</u>	<u>Weight Percentage (%)</u>
Whistle Composition #1	Potassium Perchlorate	70
	Sodium Benzoate	30
Whistle Composition #3	Potassium Perchlorate	70
	Sodium Benzoate	10
	Corn Starch	20

Photos 57 and 58 show the Whistle Compositions #1 and #3, respectively.

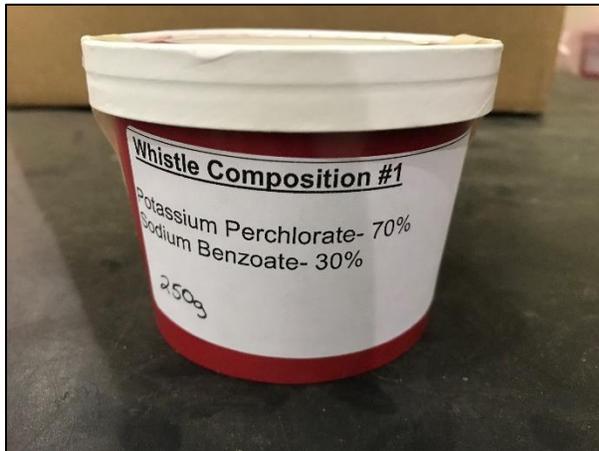


Photo 57: Whistle Composition #1 (provided by RES Specialty Pyrotechnics)

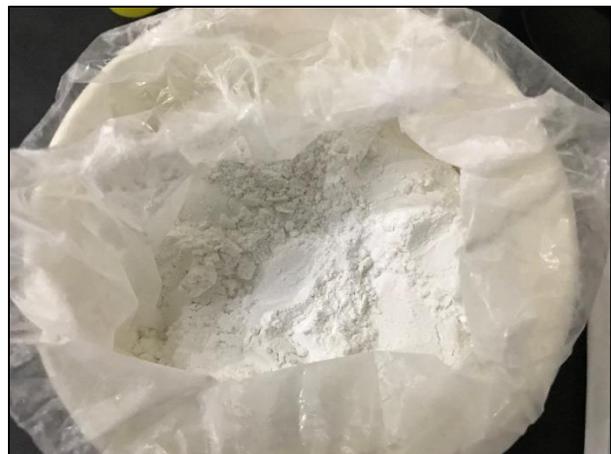
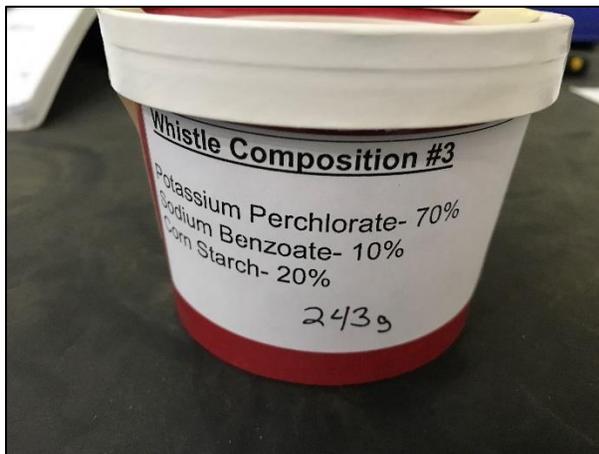


Photo 58: Whistle Composition #3 (provided by RES Specialty Pyrotechnics)

The sodium benzoate in these formulations would indicate that these compositions would most likely be closer to flash compositions and would be on the other end of the spectrum from the traditional black powder burst charge powders.

This concluded the testing using the 4-burner propane ignition (auto-ignition) source.

b. Test Results – Whistle Comps

The following table includes the test results for the 5 whistle compositions using the Koenen Apparatus and the standard four burner ignition source.

Table 10: Koenen Test Results - Ignition Mode: Auto-Ignition – Whistle Compositions

<u>Material</u>	<u>Orifice Size</u>	<u>Trial</u>	<u>Results</u>	<u>Pass/ Fail</u>
3 g – Whistle Composition #1	18mm	1	“+”	Fail
	20mm	1	“+”	Fail
	22mm	1	“-”	Pass
		2	“+”	Fail
5 g – Whistle Composition #1	18mm	1	“+”	Fail
	22mm	1	“+”	Fail
3 g – Whistle Composition #3	22mm	1	“+”	Fail
5 g – Whistle Composition #3	22 mm	1	“+”	Fail
		2	“+”	Fail

Photos 59 and 60 are representative photos of the test results for the Whistle Compositions using the Koenen Apparatus with the standard four burner ignition source.



Photo 59: Whistle Composition #1 Typical Post-Test Results

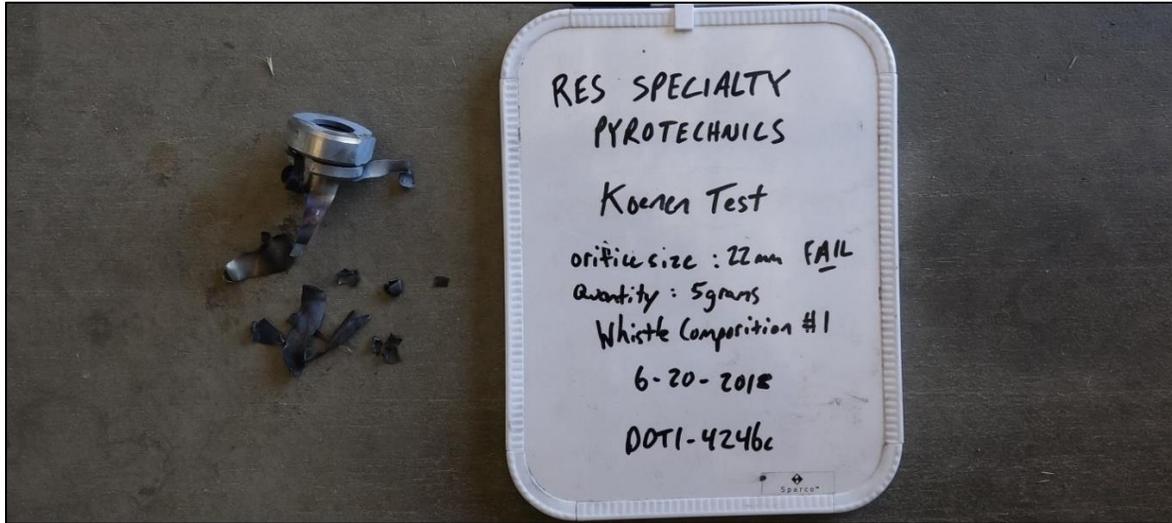


Photo 60: Whistle Composition #1 Typical Post-Test Results

c. Conclusions

The whistle comps are all exhibit limiting diameters that are in excess of those available through the Koenen Apparatus setup. The results of these tests demonstrate these types of compositions are more like flash compositions when ignited under semi-confinement and would not be considered equivalent to black powder.

Table 11 provides the estimated limiting diameter based on the testing performed using the 4 Burner Ignition Source.

Table 11: U.S. Based Break Powder Compositions Limiting Diameters

<u>Material</u>	<u>Limiting Diameter (mm)</u>
3 g – Whistle Composition #1	> 22
5 g – Whistle Composition #1	> 22
3 g – Whistle Composition #3	> 22
5 g – Whistle Composition #3	> 22

5. E-Match Ignition Source - Koenen Apparatus – Additional Modifications

For industry to easily evaluate black powder equivalency of their lift and burst charges, it is necessary to have an apparatus that is simpler to use than the standard Koenen test, which requires a propane fuel source, sparker modules, burner nozzles, an ignition system, etc. In an effort to simplify the ignition source, the entire ignition system was replaced with an electric match (Photo 61). This change of ignition source also changes the ignition mode from autoignition to spark/flame ignition. The match best simulates the initiation source found in pyrotechnic articles vs. the standard four burner Koenen ignition source that is creating an auto-ignition scenario.

a. Test Configuration – Electric Match Ignition Source

Initial tests were performed simply using the standard Koenen Apparatus as it afforded protection from the potentially fragmenting tubes.



Photo 61: Standard Electric Match



Photo 62: Modified Koenen Test with Electric Match Ignition Source

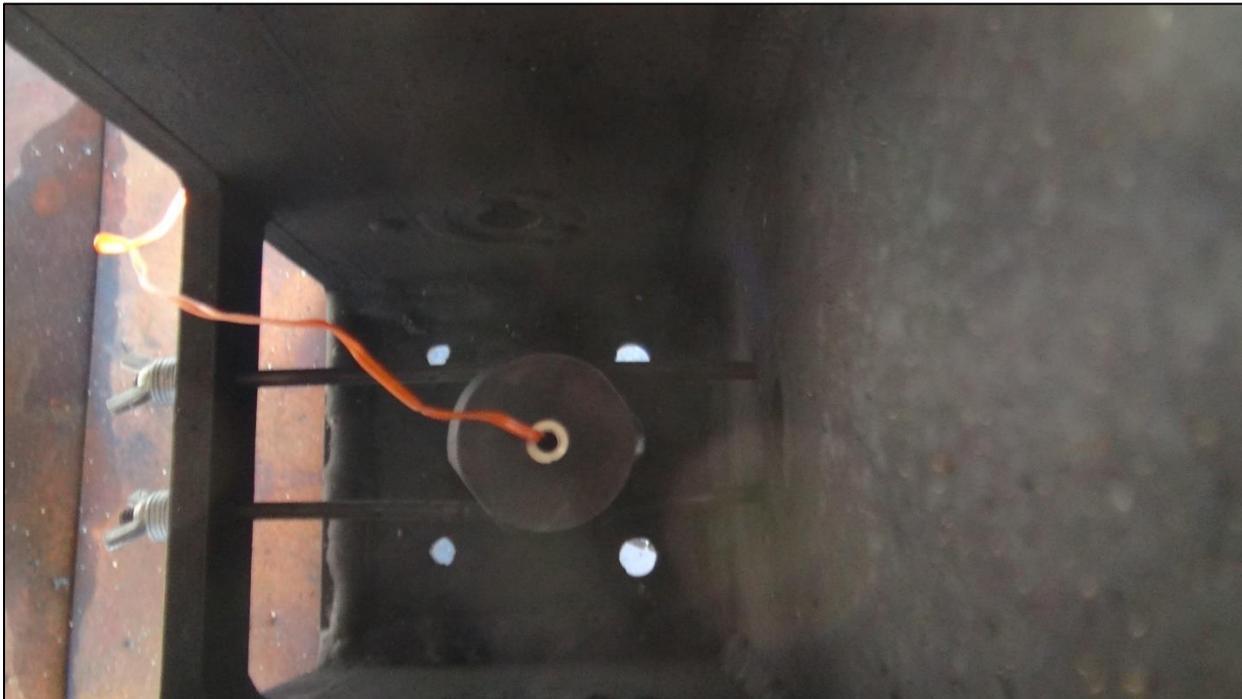


Photo 63: Modified Koenen Test with Electric Match Ignition Source



Photo 64: Modified Koenen Test with Electric Match Ignition Source

Later SMS designed a test fixture that could be used in combination with the Koenen tube and collar/orifice assembly but with an electric match introduced through the orifice diameter. Figure 9 shows the initial design which was designated as the Black Powder Equivalency Test Apparatus. Per the procedure, once the operator loads the Koenen tube and attaches the collar/orifice assembly, the operator can approach the test chamber from the protected side. This allows the match to be inserted from the top after the tube has been positioned. The match is simply placed through the orifice opening until a slight resistance is felt, signaling that the match head is at the bottom of the tube. At this point the operator may secure the lead line with tape or simply bend the match to ensure that it stays at the bottom of the tube. In the event that the powder does ignite during match insertion, the majority of the fragments would be captured in the lower section of the test fixture (Photo 72). A removal Lexan shield was also designed into the unit. Drawings for construction will be made available for public use. Figure 8 explains the electric match ignition source.

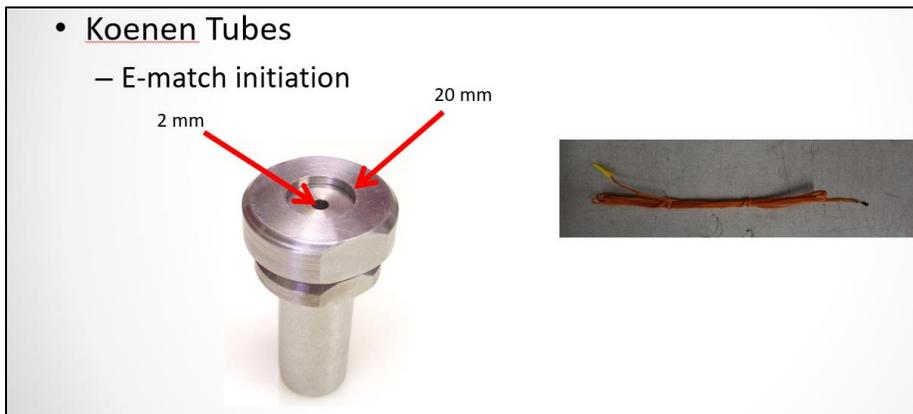


Figure 8: Electric Match Ignition Source

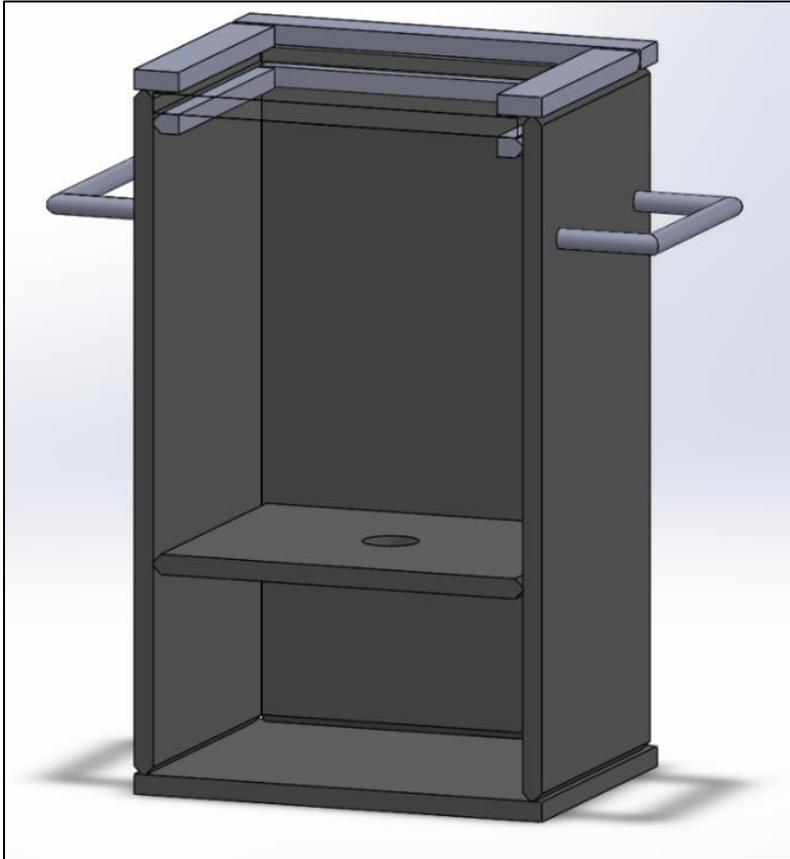


Figure 9: Black Powder Equivalency Test Apparatus



Photo 65: Black Powder Equivalency Test Apparatus



Photo 66: Black Powder Equivalency Test Apparatus

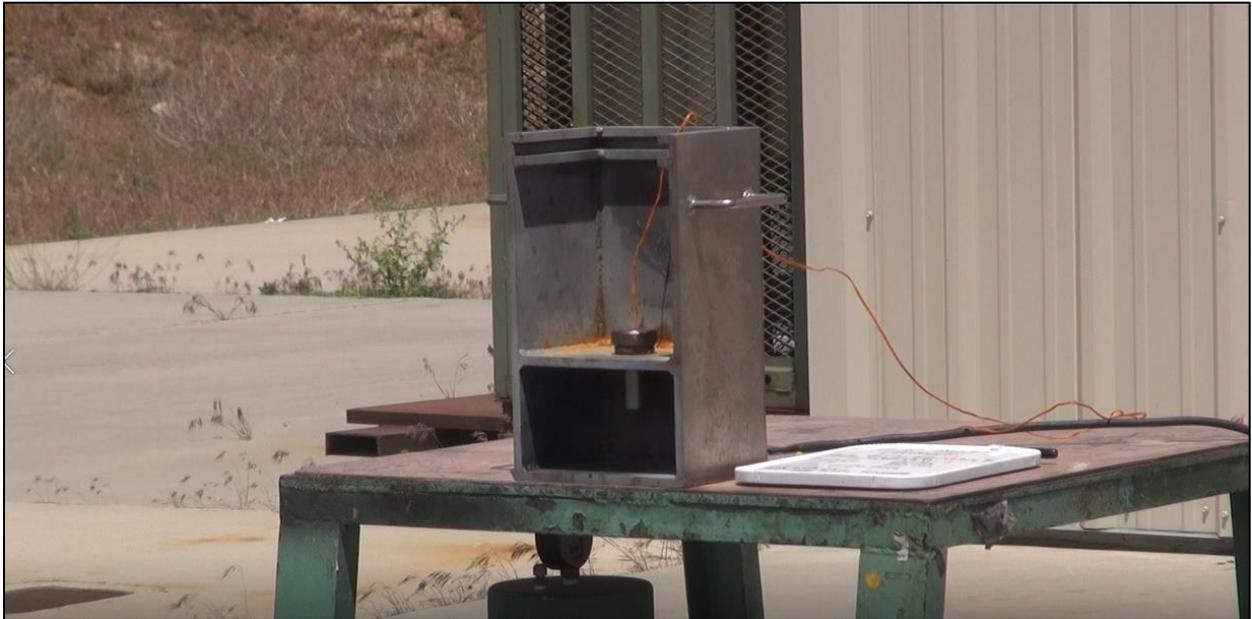


Photo 67: Black Powder Equivalency Test Fixture



Photo 68: Black Powder Equivalency Test Fixture



Photo 69: Black Powder Equivalency Test Fixture



Photo 70: Black Powder Equivalency Test Fixture



Photo 71: Black Powder Equivalency Test Fixture



Photo 72: Black Powder Equivalency Test Apparatus

Table 12 contains the formulations of powders tested in the initial phase of testing using an electric match ignition source.

Table 12: Initial Break Powder Compositions Tested with Electric Match

<u>Name</u>	<u>Chemical Constituent</u>	<u>Weight Percentage (%)</u>
FFFFg Goex Black Powder	Potassium Nitrate	75*
	Charcoal	15*
	Sulfur	10*
Whistle Composition #1	Potassium Perchlorate	70
	Sodium Benzoate	30
Whistle Composition #2	Potassium Perchlorate	70
	Sodium Benzoate	20
	Corn Starch	10
Whistle Composition #3	Potassium Perchlorate	70
	Sodium Benzoate	10
	Corn Starch	20
Whistle composition #4	Potassium Perchlorate	63.6
	Sodium Benzoate	27.3
	Titanium (60-100 mesh)	9.1
Whistle composition #5	Potassium Perchlorate	61.95
	Sodium Salicylate	26.55
	Red Iron Oxide	0.9
	Titanium (60-100 mesh)	8.9
	Mineral Oil	1.7
Canister Shell #2 (Burst Powder)	Potassium perchlorate	57
	Sulfur	19
	Aluminum	13.5
	Charcoal	10.5
6" Shell Burst Charge – Rice Hulls	Unknown	-

<u>Name</u>	<u>Chemical Constituent</u>	<u>Weight Percentage (%)</u>
6" Shell Lift Charge	~2FG Black Powder	-
Burst Charge Containing Metal	Potassium Perchlorate	68.5
	Aluminum, Indian Blackhead (< 53µm)	12.5
	Charcoal	11.4
	Sulfur	7.6
Non-Metallic Burst Charge Composition	Potassium Perchlorate	74
	Potassium Benzoate	22
	Charcoal	4

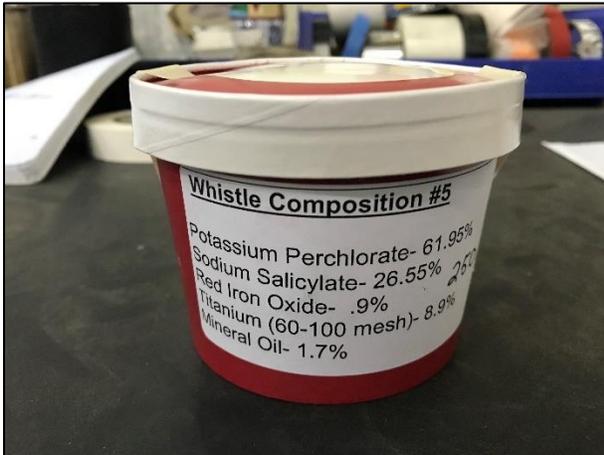


Photo 73: Whistle Composition #5 (provided by RES Specialty Pyrotechnics)



Photo 74: 6" Shell Burst Charge – Rice Hulls



Photo 75: 6" Shell Burst Charge – Rice Hulls



Photo 76: 6" Shell Lift Charge - ~ 2Fg Black Powder

b. Test Results – Electric Match Ignition Source

The following table includes the test results for compositions using the Koenen Assembly and an electric match ignition source.

Table 13: Initial Break Powder Compositions Tested with Electric Match

<u>Material</u>	<u>Orifice Size</u>	<u>Trial</u>	<u>Results</u>	<u>Pass/ Fail</u>
FFFFg Goex Black Powder	5mm	1	“+”	Fail
	8mm	1	“-”	Pass
		2	“-”	Pass
		3	“-”	Pass
		4	“-”	Pass
		5	“-”	Pass
		6	“-”	Pass
		7	“-”	Pass
		8	“-”	Pass
		9	“-”	Pass
		10	“-”	Pass
		11	“-”	Pass
		12	“-”	Pass
		13	“-”	Pass
		14	“-”	Pass
		15	“-”	Pass
		16	“-”	Pass
		17	“-”	Pass
		18	“-”	Pass
		19	“-”	Pass
		20	“-”	Pass
	10mm	1	“-”	Pass
Whistle Composition #1	22mm	1	“+”	Fail
Whistle Composition #2	22mm	1	“+”	Fail
Whistle Composition #3	12mm	1	“+”	Fail
	15mm	1	“+”	Fail
	18mm	1	“-”	Pass
		2	“-”	Pass
		3	“-”	Pass
	20mm	1	“-”	Pass
22mm	1	“-”	Pass	

<u>Material</u>	<u>Orifice Size</u>	<u>Trial</u>	<u>Results</u>	<u>Pass/ Fail</u>
Whistle Composition #4	22mm	1	“+”	Fail
Whistle Composition #5	15mm	1	“+”	Fail
	18mm	1	“-”	Pass
		2	“-”	Pass
		3	“-”	Pass
	20mm	1	“-”	Pass
	22mm	1	“-”	Pass
6” Shell Burst Charge – Rice Hulls	3mm	1	“+”	Fail
	5mm	1	“-”	Pass
		2	“-”	Pass
		3	“-”	Pass
	8mm	1	“-”	Pass
6” Shell Lift Charge	3mm	1	“+”	Fail
	5mm	1	“-”	Pass
		2	“-”	Pass
		3	“-”	Pass
	8mm	1	“-”	Pass
Canister Shell #2 (Burst Powder)	10mm	1	“+”	Fail
	15mm	1	“+”	Fail
	20mm	1	“-”	Pass
Burst Charge Containing Metal	8mm	1	“+”	Fail
	10mm	1	“-”	Pass
		2	“+”	Fail
	12mm	1	“+”	Fail
	15mm	1	“-”	Pass
		2	“-”	Pass
		3	“-”	Pass
Non-Metallic Burst Charge Composition (Hybrid Burst Charge)	8mm	1	“+”	Fail
	10mm	1	“-”	Pass
		2	“-”	Pass
		3	“-”	Pass
	15mm	1	“-”	Pass
	18mm	1	“-”	Pass

Photos 78-86 show examples of the test results.



Photo 78: Whistle Composition #1 @ 22 mm Vent (5 Grams) – FAIL

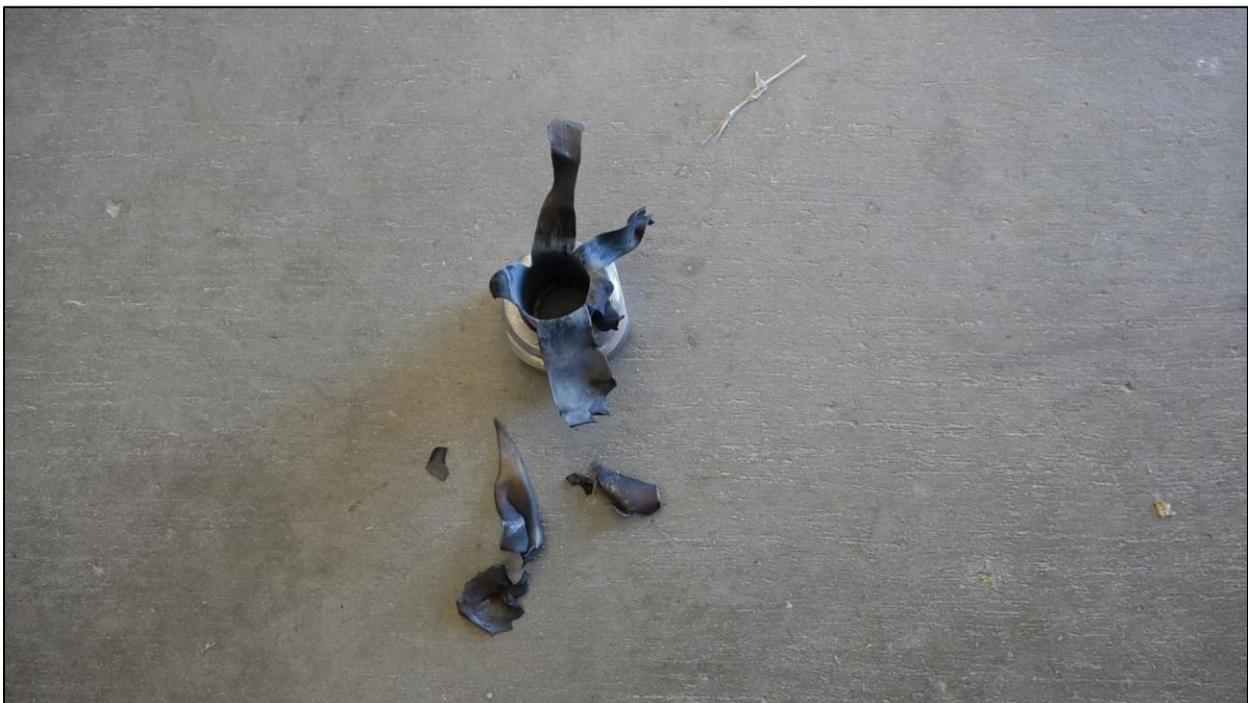


Photo 79: Whistle Composition #1 @ 20 mm Vent (5 Grams) – FAIL

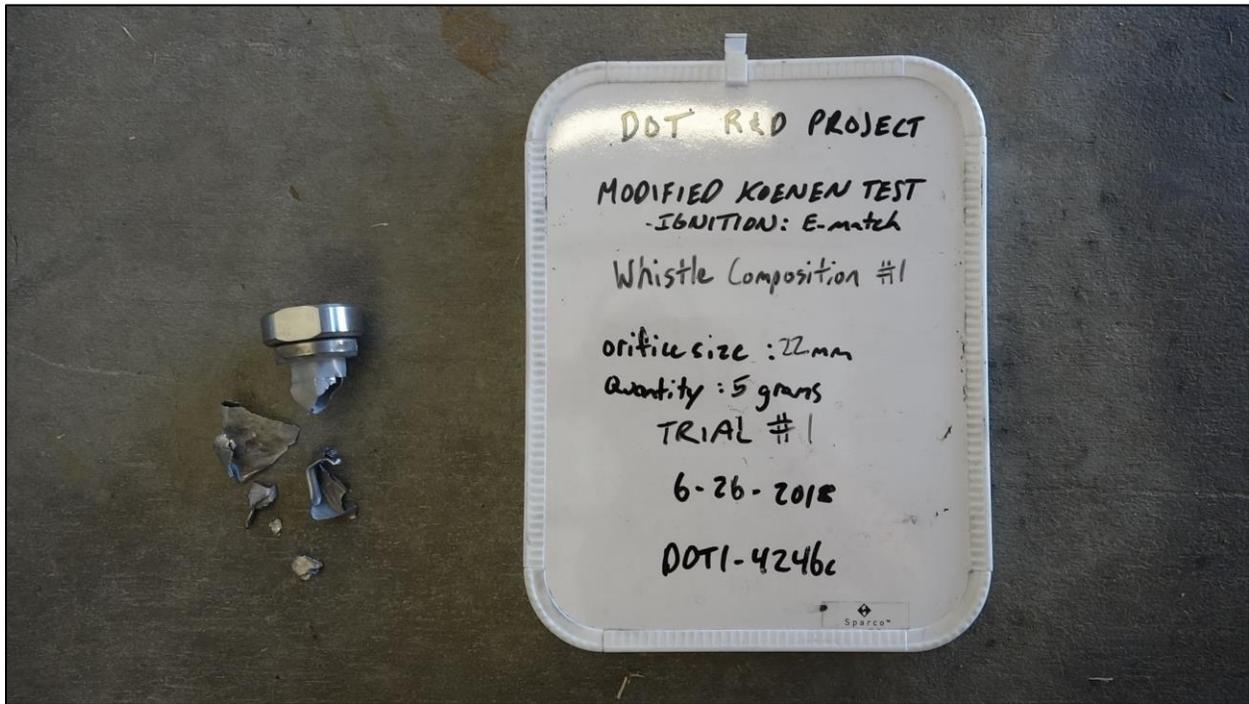


Photo 80: Whistle Composition #1 @ 22 mm Vent (5 Grams) – FAIL



Photo 81: Whistle Composition #3 @ 22 mm Vent (5 Grams) – PASS



Photo 82: Whistle Composition #3 @ 15 mm Vent (5 Grams) – FAIL

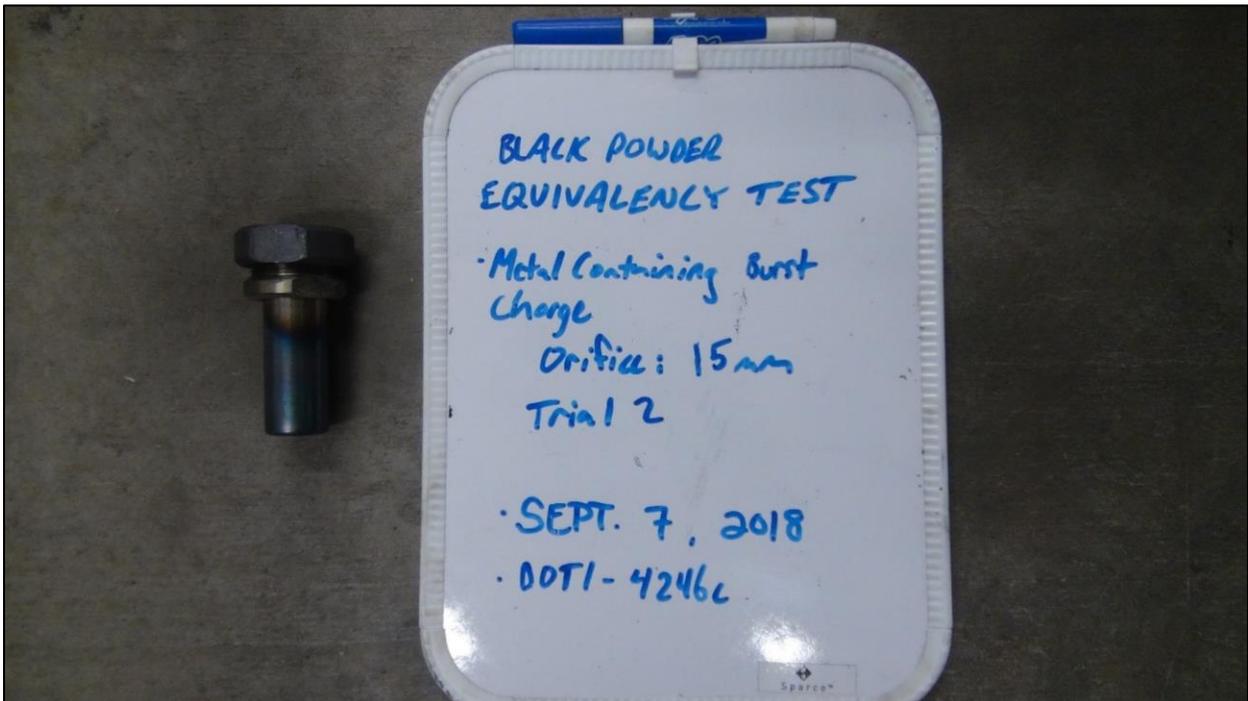


Photo 83: Burst Charge Containing Metal @ 15 mm Vent (5 Grams) – PASS



Photo 84: Burst Charge Containing Metal @ 10 mm Vent (5 Grams) – FAIL

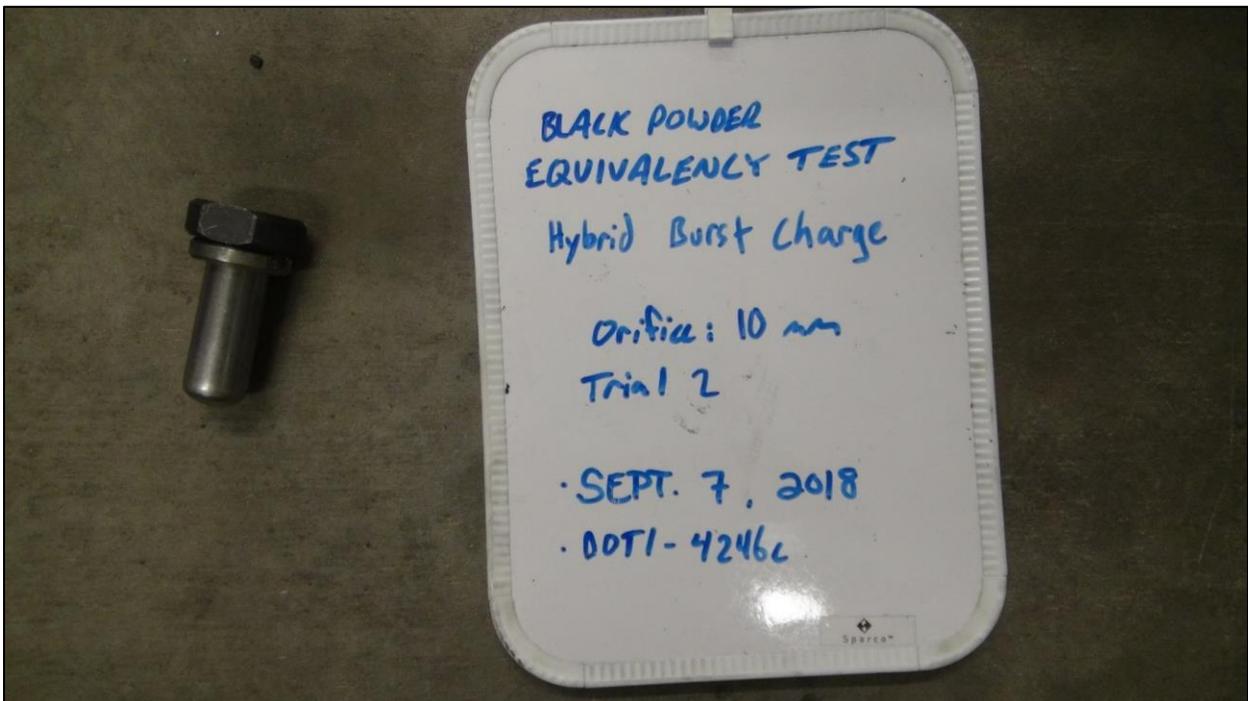


Photo 85: Hybrid Burst Charge @ 10 mm Vent (5 Grams) – PASS



Photo 86: Hybrid Burst Charge @ 8 mm Vent (5 Grams) – FAIL

c. Conclusions

The results of the set of test data indicate that the limiting diameter for commercial black powder 4Fg is 5 mm. The tests also indicate that the new Burst Charge Equivalency Test appears to provide the data desired of such a test and is demonstrating to be easy to operate and easy to interpret. Compositions like the coated rice hulls would be expected to have limiting diameters near that of black powder. The testing confirms this, showing a limiting diameter of 3 mm for the coated rice hulls.

It has hypothesized that this test method would have the range to predict compositions containing metal all the way up to flash compositions. This initial phase of testing indicates that this is true, with the whistle compositions having limiting diameters above 22 mm for the faster acting compositions (Whistle Compositions #1, #2, and #4), and the slightly slower burning compositions (Whistle Compositions #3, and #5) each having a limited diameter of 15 mm.

The 6" lift powder, which was identified as being approximately a 2Fg black powder in size, tested out with a limiting diameter of 5 mm, the same as the commercial black powder.

The Canister Shell burst powder, which was known to be a faster burning composition, appeared to have a limiting diameter nearing a flash composition, as expected (limited quantities at the time of testing resulted in limited trials).

In addition, SMS was able to prepare a Burst Charge powder with metal configured into the combustion formulation (“Burst Charge Containing Metal”). This was to see if there would be a difference from the previous testing with small particle aluminum powder simply added to commercial black powder. The test results indicate that this is the case, and that when the metal is factored into the exothermic redox chemical reaction equation, metal enhancement of the burn rate does occur, to a point, as will be discussed later. This testing showed that the Burst Charge Containing Metal had a limiting diameter of 15 mm as compared to the 5 mm for 4Fg black powder.

And finally, SMS prepared a Non-Metallic Burst Charge Composition, to show how compositions can be prepared without metals and still achieve compositions that are burning much faster than the standard black powder. This was the case for the Non-Metallic Burst Charge Composition which had a limiting diameter of 10 mm while 4Fg black powder has a limiting diameter of 5 mm.

The additional importance of this data set is that it shows that this method can differentiate powders from the slow burning rice husks all the way up to compositions approaching flash comps.

Table 14 provides the estimated limiting diameter based on the testing performed. It is very apparent the potential power his test method can have as it can readily identify those powders that are similar to the standard commercial black powder and which ones are closer to flash powder in strength under confinement. The powders arranged in ascending order based on the limiting diameter.

Table 14: U.S. Based Break Powder Compositions Limiting Diameters

<u>Material</u>	<u>Limiting Diameter (mm)</u>
6” Shell Lift Charge	3
6” Shell Burst Charge – Rice Hulls	3
FFFg Goex Black Powder	5
Non-Metallic Burst Charge Composition	8
Burst Charge Containing Metal	12
Whistle Composition #3	15
Whistle Composition #5	15
Canister Shell #2 (Burst Powder)	≥ 15
Whistle Composition #1	> 22
Whistle Composition #2	> 22
Whistle Composition #4	> 22

6. Burst Charge Equivalency Test – China Formulations

During the duration of this project, SMS had the opportunity to travel to China. To take advantage of this opportunity, SMS shipped over the Burst Charge Equivalency Test Fixture along a few Koenen tube assemblies and performed testing on enhanced non-metallic powders. The test results are presented in this section.

a. Test Configuration – China Compositions - Electric Match Ignition Source

The newly developed Burst Charge Equivalency Test Fixture with an electric match ignition source was used as the test configuration of this data set. Tests were performed at the Hailong Factory, Liuyang, China. A special thanks to Jake’s Fireworks for assisting in making this happen.

Table 15 contains the formulations of powders tested in this phase of testing using an electric match ignition source.

Table 15: Chinese Break Powder Compositions (Hailong Factory, Liuyang, China)

<u>Name</u>	<u>Chemical Constituent</u>	<u>Weight Percentage (%)</u>
Benzoate Break Charge	Potassium Perchlorate	80
	Potassium Benzoate	20
Whistle Composition	Potassium Perchlorate	70
	Potassium Benzoate	30
Standard Break Charge with 5% aluminum powder	Potassium Perchlorate	65
	Sulfur	16
	Charcoal	14
	Aluminum (81 microns)	5
Standard Break Charge with 10% aluminum powder	Potassium Perchlorate	62
	Sulfur	15
	Charcoal	13
	Aluminum (81 microns)	10
Standard Break Charge with 15% aluminum powder	Potassium Perchlorate	59
	Sulfur	14
	Charcoal	12
	Aluminum (81 microns)	15
Standard Break Charge	Potassium Perchlorate	69
	Sulfur	17
	Charcoal	14

The following photos show the powders under review.

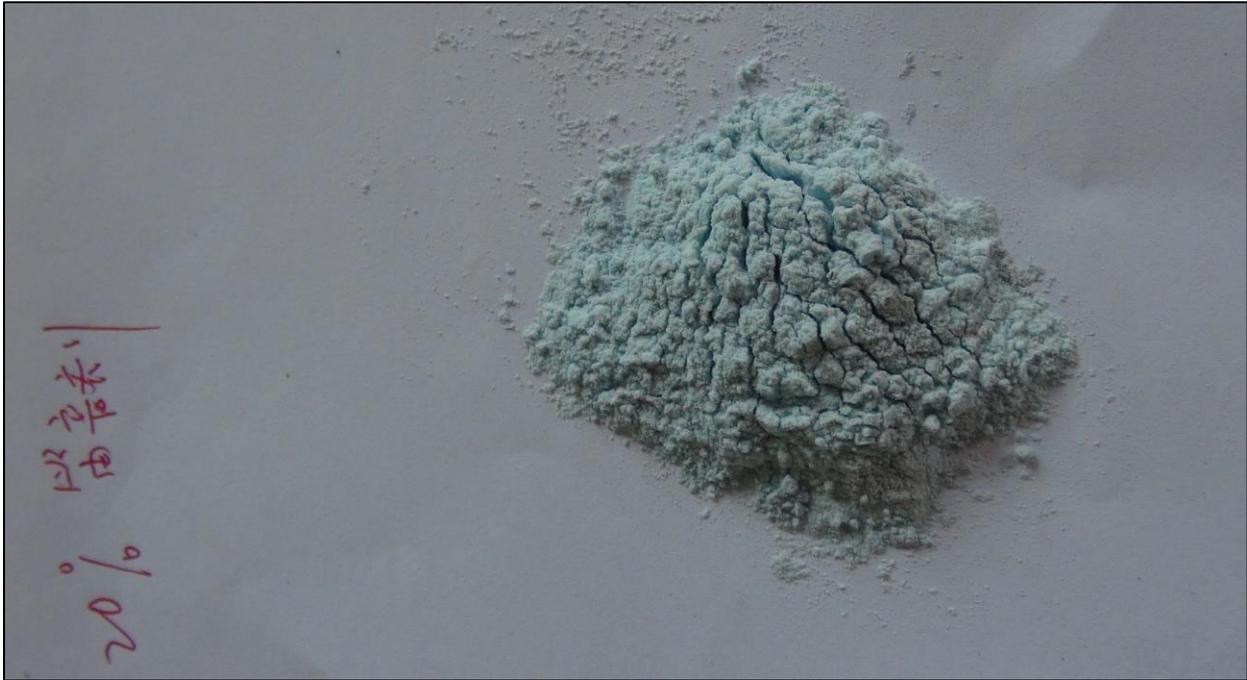


Photo 87: Benzoate Break Charge



Photo 88: Whistle Composition

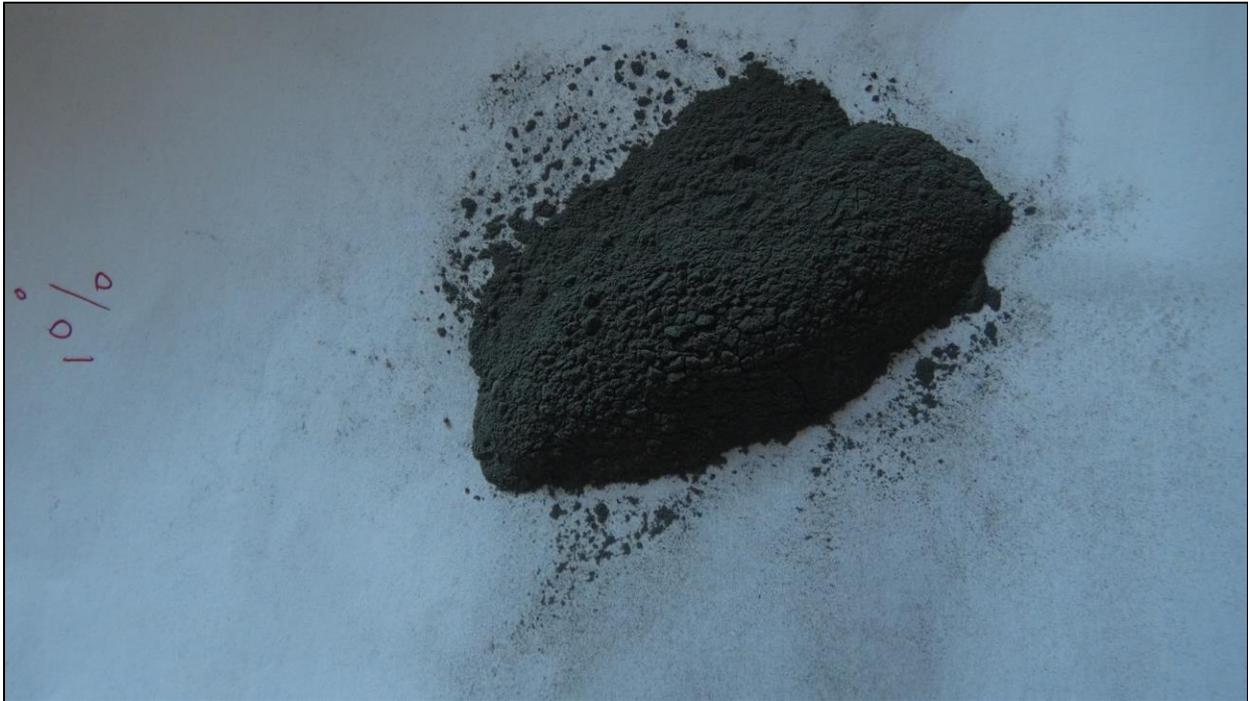


Photo 89: Standard Break Charge with 10% aluminum powder



Photo 90: Standard Break Charge

b. Test Results – China Compositions - Electric Match Ignition Source

The following table includes the test results for the Chinese compositions using the Koenen assembly and an electric match ignition source.

Table 16: Chinese Break Powder Compositions Tested with Electric Match

<u>Material</u>	<u>Orifice Size</u>	<u>Trial</u>	<u>Results</u>	<u>Pass/ Fail</u>
Benzoate Break Charge	8mm	1	“+”	Fail
	10mm	1	“-”	Pass
		2	“-”	Pass
		3	“-”	Pass
	18mm	1	“-”	Pass
Whistle Composition	10mm	1	“+”	Fail
	18mm	1	“-”	Pass
Standard Break Charge with 5% aluminum powder	8mm	1	“+”	Fail
	18mm	1	“-”	Pass
Standard Break Charge with 10% aluminum powder	8mm	1	“+”	Fail
	18mm	1	“-”	Pass
		2	“+”	Fail
	22mm	1	“-”	Pass
Standard Break Charge with 15% aluminum powder	10mm	1	“+”	Fail
	18mm	1	“-”	Pass
		2	“-”	Pass
Standard Break Charge	8mm	1	“+”	Fail
		2	“+”	Fail
	18mm	1	“-”	Pass
Control sample: Standard Break Charge with 5% aluminum powder (newly prepared from Standard Break Charge sample)	18mm	1	“-”	Pass (not as reactive as original prepared sample)

Photos 91-94 show examples of the test results.



Photo 91: Standard Break Charge @ 8 mm Vent (5 Grams) – FAIL

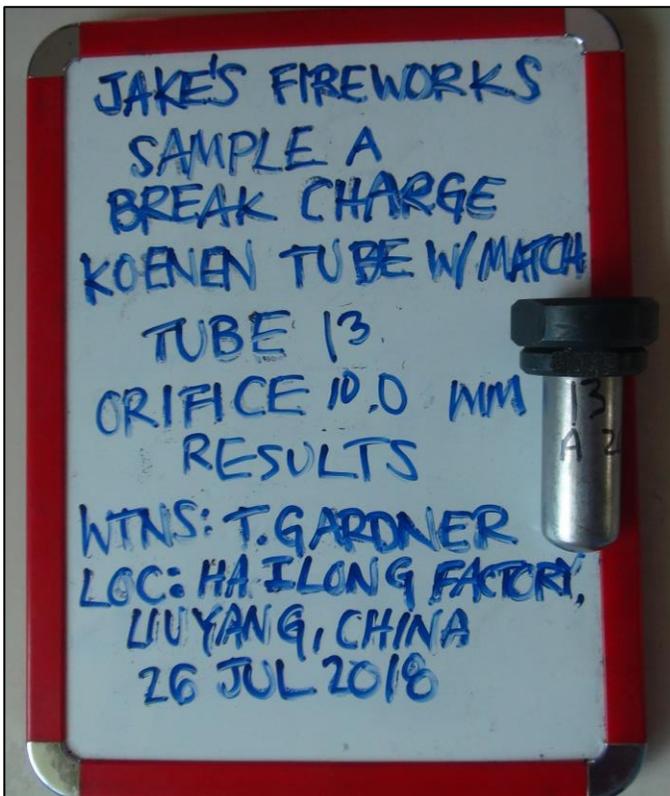


Photo 92: Standard Break Charge @ 10 mm Vent (5 Grams) – PASS

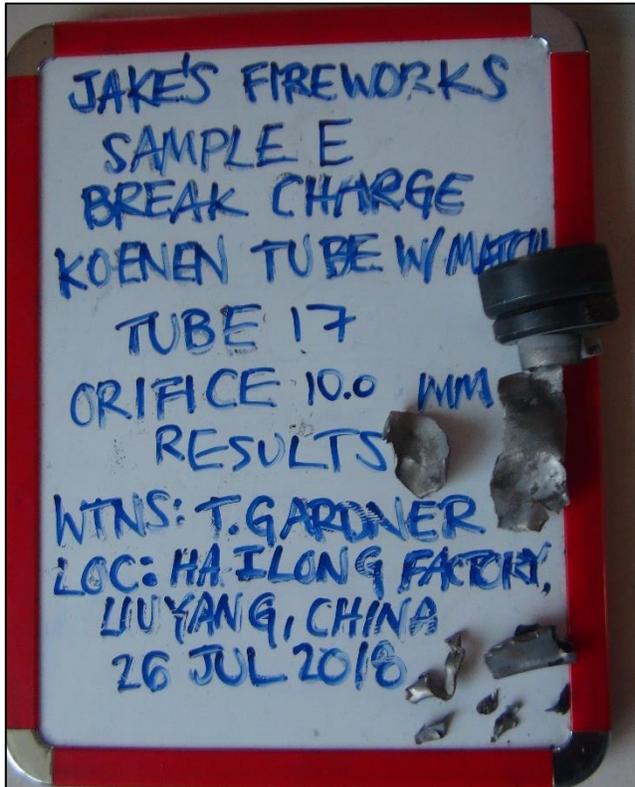


Photo 93: Standard Break Charge w/ 15% Al Powder @ 10 mm Vent (5 Grams) – FAIL

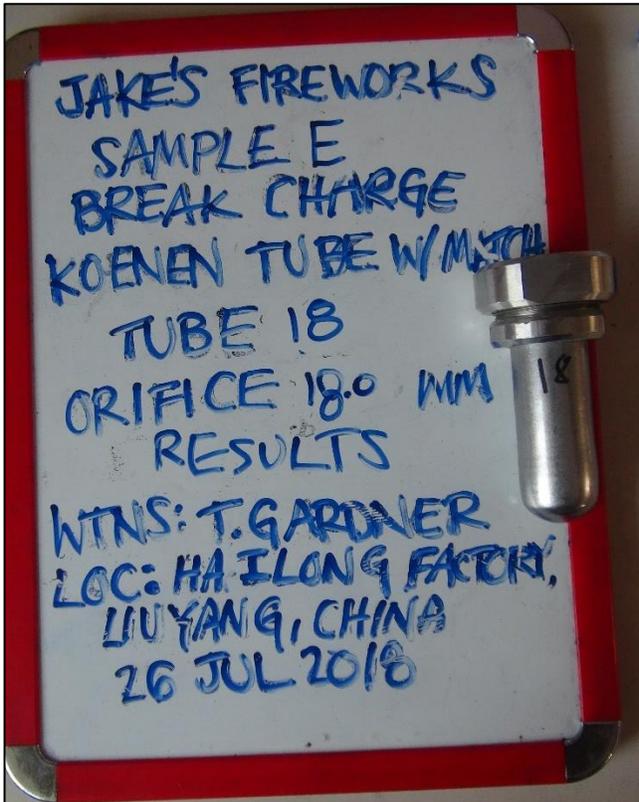


Photo 94: Standard Break Charge w/ 15% Al Powder @ 18 mm Vent (5 Grams) – PASS

c. Conclusions

While testing was limited, the data from this test set provides significant insight into the break charges coming out of China. Standard break charges are not using potassium nitrate but potassium perchlorate in their compositions. The standard break charge has a limiting diameter greater than standard black powder but less than flash compositions. In fact, all the formulations are testing at or below 18 mm, meaning that even when enhanced with metal, they are not approaching flash compositions in general.

It should be noted that the same trend was observed in this data set in that the addition of metal percentage did not increase the burn rate somewhere between 5 and 15% metal.

Table 17 provides the estimated limiting diameter based on the testing performed. The powders arranged in ascending order based on the limiting diameter.

Table 17: U.S. Based Break Powder Compositions Limiting Diameters

<u>Material</u>	<u>Limiting Diameter (mm)</u>
Benzoate Break Charge	8
Whistle Composition	> 10
Standard Break Charge with 5% aluminum powder	> 8

<u>Material</u>	<u>Limiting Diameter (mm)</u>
Standard Break Charge with 10% aluminum powder	> 18
Standard Break Charge with 15% aluminum powder	> 10 but < 18
Standard Break Charge	> 8

7. U.S. Based Break Charge Formulations

Additional testing was performed on selected break charge formulations. The test results are presented in this section.

a. Test Configuration – U.S. Compositions - Electric Match Ignition Source

The newly developed Burst Charge Equivalency Test Fixture with an electric match ignition source was used as the test configuration of this data set. The Test Fixture was modified slightly during this phase. A small channel was cut from the front edge of the plate holding the orifice assembly (Photos 95 – 99). This change allowed for the remove of the post-test tube much easier reducing the sample turn-around time by approximately 30% due to the difficulty in removing partially fragmented tubes from the previous fixture design (Photos 100 – 101). Tests were performed at the MP Associates, in Lone, CA. A special thanks to MP Associates for assisting in making this happen.



Photo 95: Burst Charge Equivalency Test Fixture Design Change

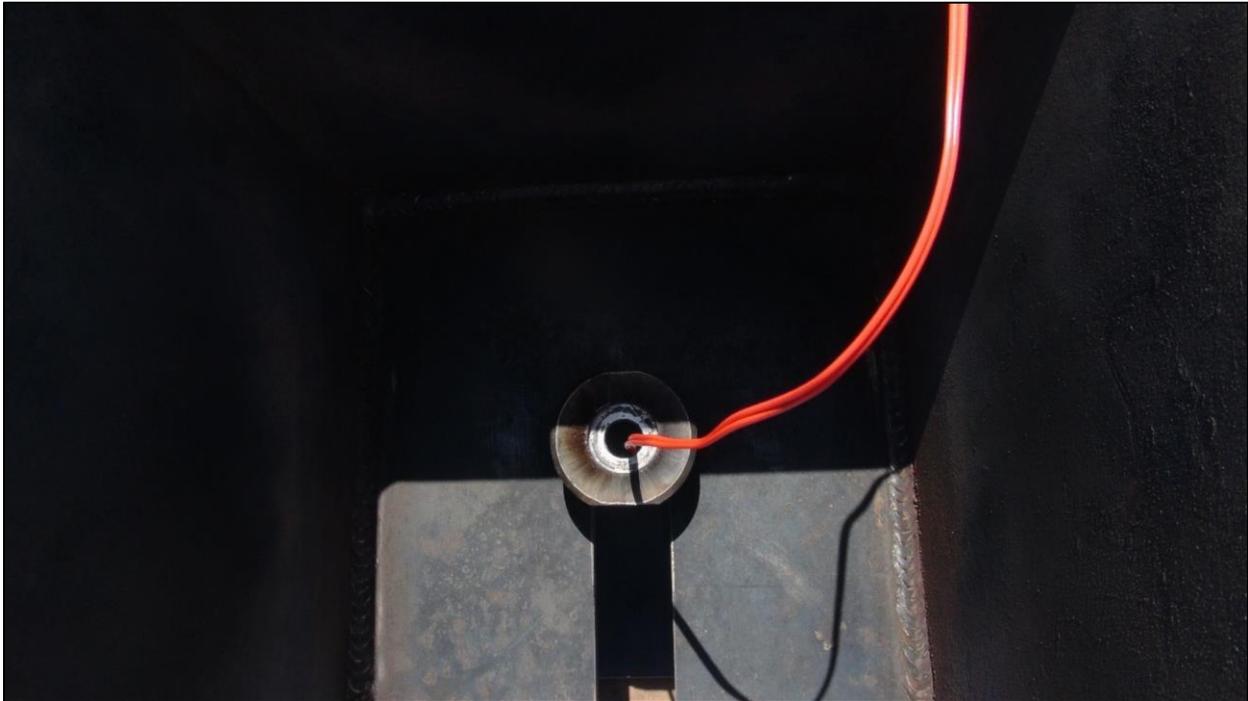


Photo 96: Burst Charge Equivalency Test Fixture Design Change



Photo 97: Burst Charge Equivalency Test Fixture Design Change



Photo 98: Burst Charge Equivalency Test Fixture Design Change



Photo 99: Burst Charge Equivalency Test Fixture Design Change



Photo 100: Burst Charge Equivalency Test Fixture Fragmented Tube Removal



Photo 101: Burst Charge Equivalency Test Fixture Fragmented Tube Removal

Table 18 contains the formulations of powders tested in this phase of testing using an electric match ignition source.

Table 18: U.S. Based Break Powder Compositions

<u>Name</u>	<u>Chemical Constituent</u>	<u>Weight Percentage (%)</u>
60% Potassium Perchlorate 40% German Dark Pyro Aluminum	Potassium Perchlorate	60
	German Dark Pyro Aluminum	40
70% Potassium Perchlorate 30% Potassium Benzoate	Potassium Perchlorate	70
	Potassium Benzoate	30
65% Potassium Perchlorate 35% German Dark Pyro Aluminum	Potassium Perchlorate	70
	Potassium Benzoate	30
GOEX 5FBP (unglazed)	Potassium Perchlorate	75
	Sulfur	10
	Charcoal	15
Black Powder, 5% Al Powder (GB) optimized	Potassium Nitrate	-
	Sulfur	-
	Charcoal	-
	Aluminum (5 microns)	5
90% GOEX Meal Powder, 10% Al Powder (GB) optimized	Potassium Nitrate	-
	Sulfur	-
	Charcoal	-
	Aluminum (81 microns)	10
35% Potassium Nitrate, 23% Potassium Perchlorate, 13% Potassium Benzoate, 10% Charcoal	Potassium Nitrate	35
	Potassium Perchlorate	23
	Potassium Benzoate	13
	Charcoal	10
70% Potassium Perchlorate, 20% Potassium Benzoate, 10% Air Float Charcoal	Potassium Perchlorate	70
	Potassium Benzoate	20
	Air Float Charcoal	10
Spanish Salute Powder (#7)	Potassium Nitrate	35
	Potassium Perchlorate	23
	Potassium Benzoate	13
	Charcoal	10
Black Powder with 10% Al Optimized	Potassium Nitrate	-
	Sulfur	-
	Charcoal	-
	Aluminum (5 microns)	10
68% Barium Nitrate,	Barium Nitrate	38

<u>Name</u>	<u>Chemical Constituent</u>	<u>Weight Percentage (%)</u>
9% Sulfur, 23% Aluminum (GB)	Sulfur	9
	Aluminum (5 microns)	23
50% Potassium Perchlorate, 50% Magnesium (44 microns or smaller)	Potassium Perchlorate	50
	Magnesium (44 microns or smaller)	50
70% Potassium Perchlorate, 30% German Dark Pyro Aluminum	Potassium Perchlorate	70
	Alum GD Pyro Aluminum (5 microns)	30
Meal Powder	Potassium Nitrate	75
	Sulfur	10
	Charcoal	15

The following photos show the powders under review.



Photo 102: 5Fg GOEX Black Powder

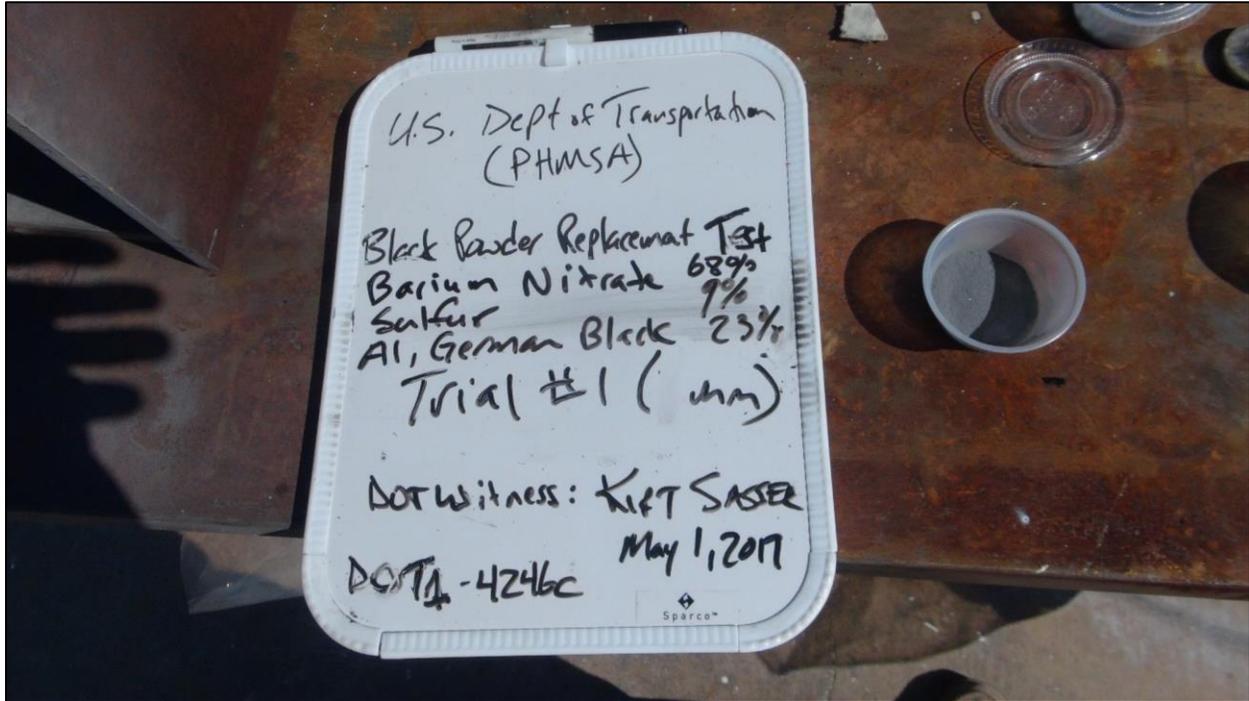


Photo 105: 68% Barium Nitrate, 9% Sulfur, 23% Aluminum (German Black)

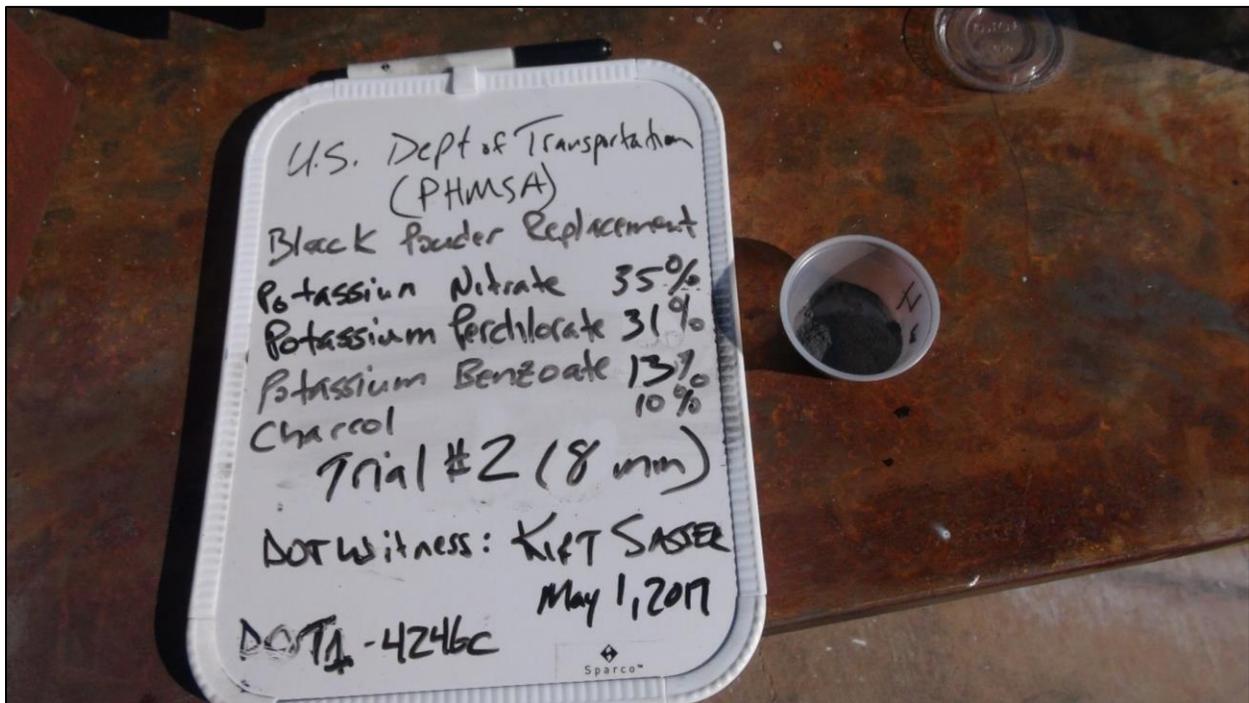


Photo 106: 35% KNO₃, 31% KClO₄, 13% Potassium Benzoate, 10% Charcoal

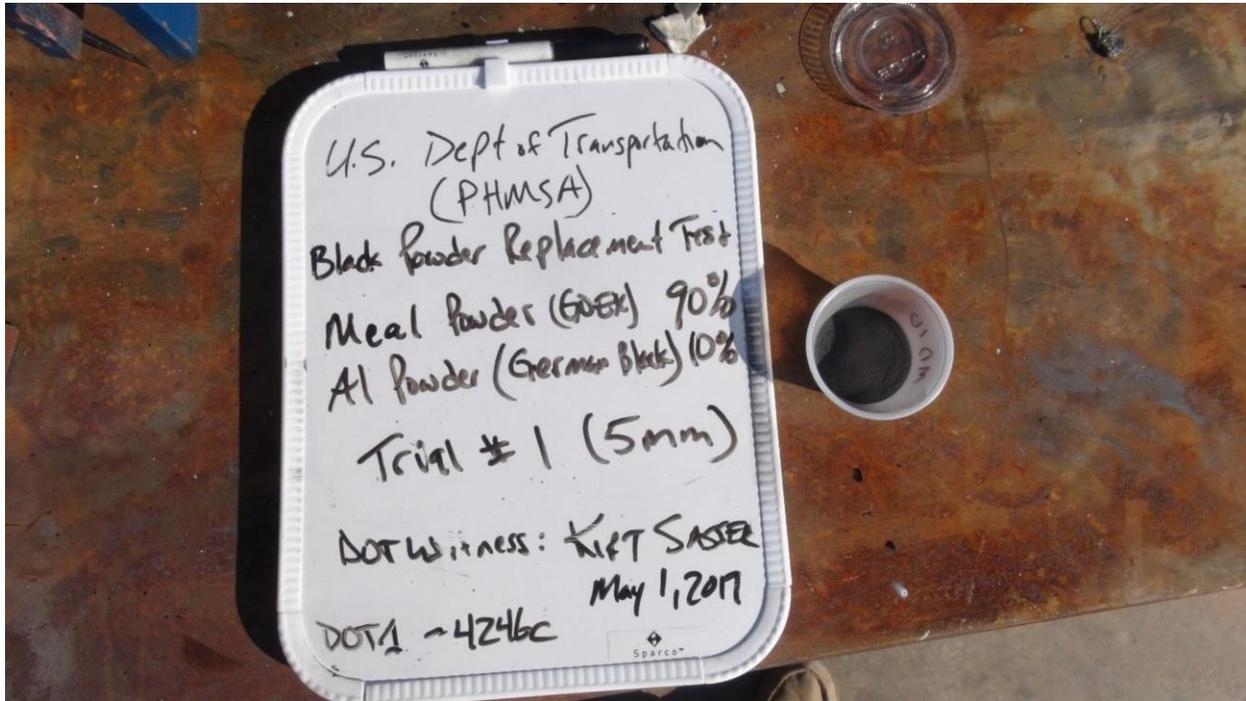


Photo 107: 90% GOEX Meal Powder, 10% Aluminum Powder (German Black)



Photo 108: Burst Charge Equivalency Test Setup



Photo 109: Burst Charge Equivalency Test Setup

b. Test Results – U.S. Compositions - Electric Match Ignition Source

The following table includes the test results for compositions using the Koenen Assembly and an electric match ignition source.

Table 19: U.S. Based Break Powder Compositions Tested with Electric Match

<u>Material</u>	<u>Orifice Size</u>	<u>Trial</u>	<u>Results</u>	<u>Pass/ Fail</u>
60% Potassium Perchlorate 40% German Dark Pyro Aluminum	12mm	1	“+”	Fail
	15mm	1	“-”	Pass
		2	“+”	Fail
	18mm	1	“-”	Pass
		2	“-”	Pass
		3	“-”	Pass
	20mm	1	“-”	Pass
22mm	1	“-”	Pass	
70% Potassium Perchlorate 30% Potassium Benzoate	10mm	1	“+”	Fail
	12mm	1	“-”	Pass
		2	“+”	Fail
	15mm	1	“-”	Pass

<u>Material</u>	<u>Orifice Size</u>	<u>Trial</u>	<u>Results</u>	<u>Pass/ Fail</u>
		2	"_"	Pass
		3	"_"	Pass
	22mm	1	"_"	Pass
65% Potassium Perchlorate 35% German Dark Pyro Aluminum	22mm	1	"+"	Fail
GOEX 5Fg Black Powder (unglazed)	3mm	1	"+"	Fail
	5mm	1	"_"	Pass
		2	"_"	Pass
		3	"_"	Pass
Black Powder, 5% Al Powder (GB) optimized	5mm	1	"_"	Pass
90% GOEX Meal Powder, 10% Al Powder (GB)	5mm	1	"+"	Fail
	8mm	1	"_"	Pass
		2	"_"	Pass
		3	"+"	Fail
	10mm	1	"_"	Pass
		1	"_"	Pass
35% Potassium Nitrate, 23% Potassium Perchlorate, 13% Potassium Benzoate, 10% Charcoal	5mm	1	"+"	Fail
	8mm	1	"_"	Pass
		2	"_"	Pass
		3	"_"	Pass
	10mm	1	"_"	Pass
70% Potassium Perchlorate, 20% Potassium Benzoate, 10% Air Float Charcoal	5mm	1	"+"	Fail
	8mm	1	"_"	Pass
		2	"+"	Fail
	10mm	1	"_"	Pass
		2	"+"	Fail
	12mm	1	"_"	Pass
		2	"_"	Pass
		3	"_"	Pass
Spanish Salute Powder (#7)	22mm	1	"+"	Fail
Black Powder with 10% Al Optimized	5mm	1	"+"	Fail

<u>Material</u>	<u>Orifice Size</u>	<u>Trial</u>	<u>Results</u>	<u>Pass/ Fail</u>
	8mm	1	"_"	Pass
		2	"_"	Pass
		3	"_"	Pass
	10mm	1	"_"	Pass
68% Barium Nitrate, 9% Sulfur, 23% Aluminum (GB)	5mm	1	"+"	Fail
	8mm	1	"+"	Fail
	10mm	1	"_"	Pass
		2	"_"	Pass
		3	"_"	Pass
50% Potassium Perchlorate, 50 % Magnesium (44 microns or smaller)	18mm	1	"_"	Pass
	20mm	1	"_"	Pass
	22mm	1	"_"	Pass
70% Potassium Perchlorate, 30% German Dark Pyro Aluminum	22mm	1	"+"	Fail
Meal Powder	3mm	1	"+"	Fail
	5mm	1	"_"	Pass
		2	"_"	Pass
		3	"_"	Pass

Photos 110 – 115 show examples of the test results.

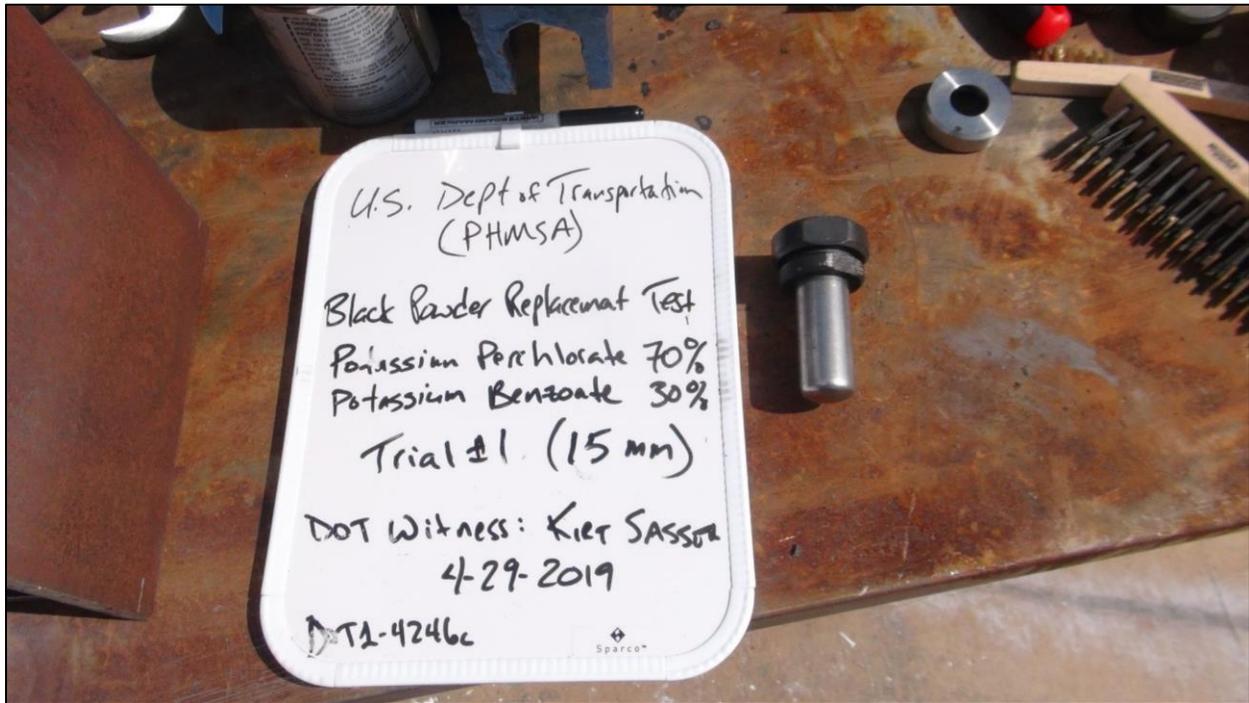


Photo 110: 70% Potassium Perchlorate, 30% Potassium Benzoate @ 15 mm (5 Grams) – PASS

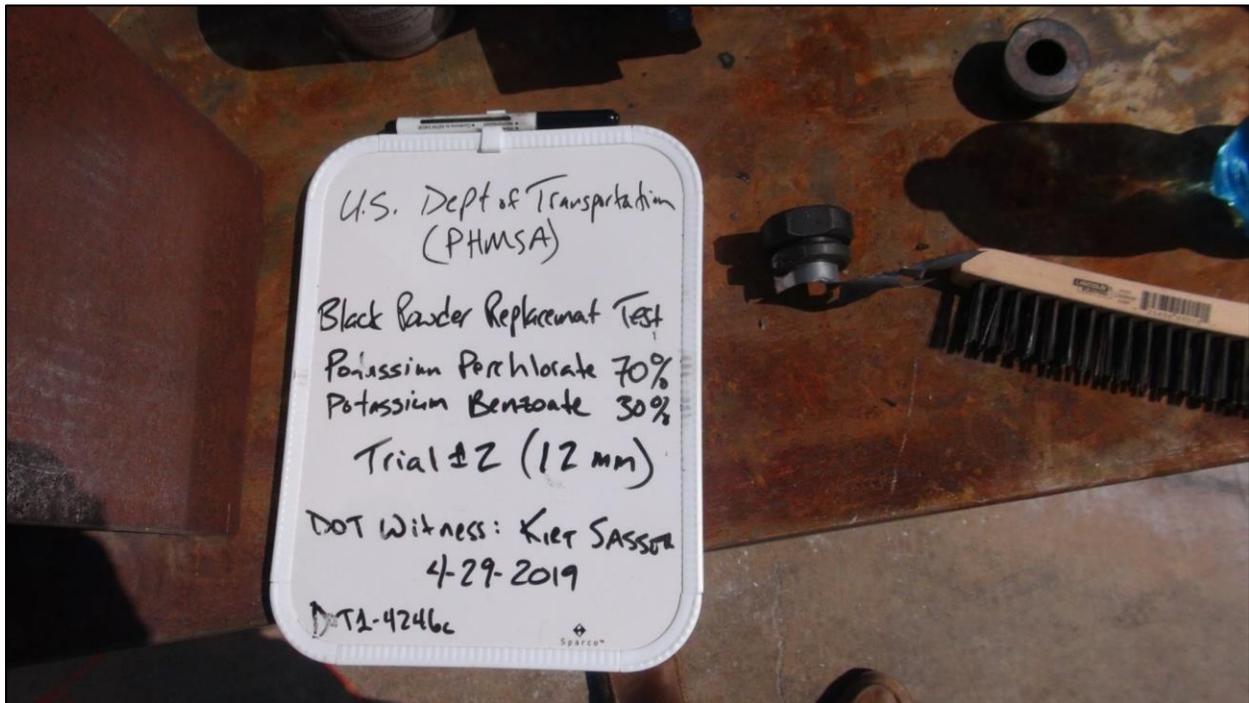


Photo 111: 70% Potassium Perchlorate, 30% Potassium Benzoate @ 12 mm (5 Grams) – FAIL

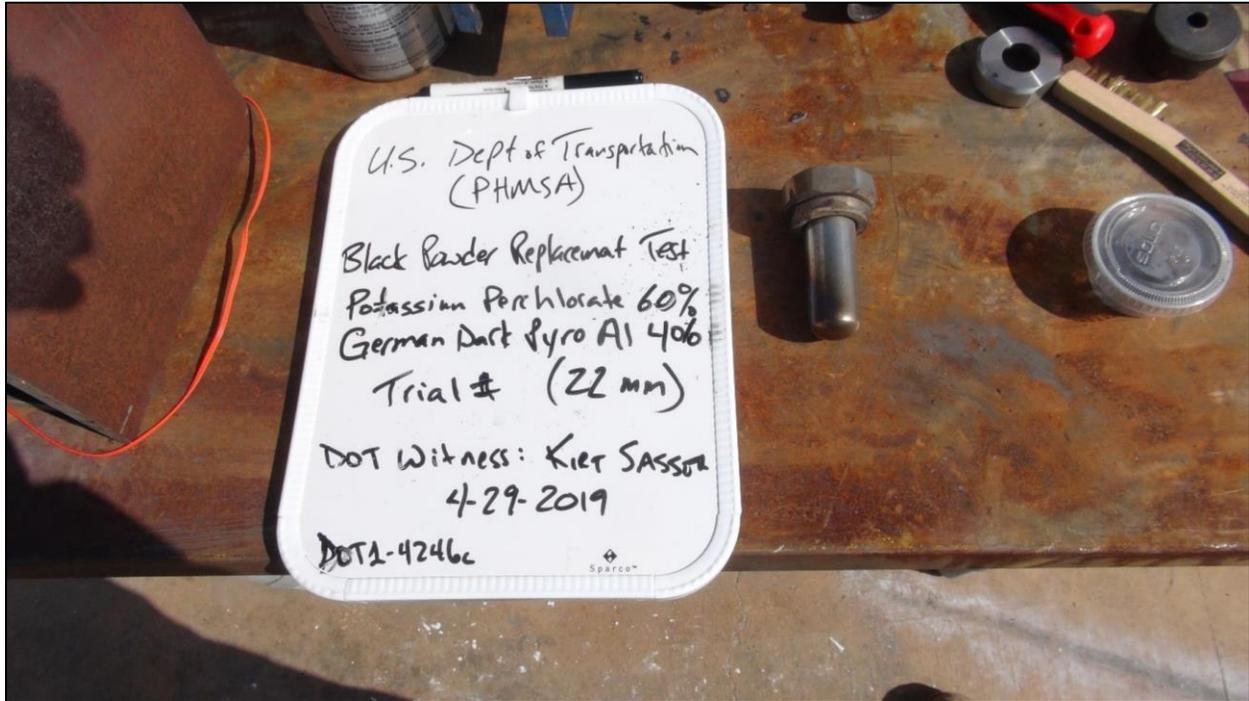


Photo 112: 60% Potassium Perchlorate, 40% German Dark Pyro Al @ 22mm (5 Grams) – PASS



Photo 113: 60% Potassium Perchlorate, 40% German Dark Pyro Al @ 20mm (5 Grams) – PASS

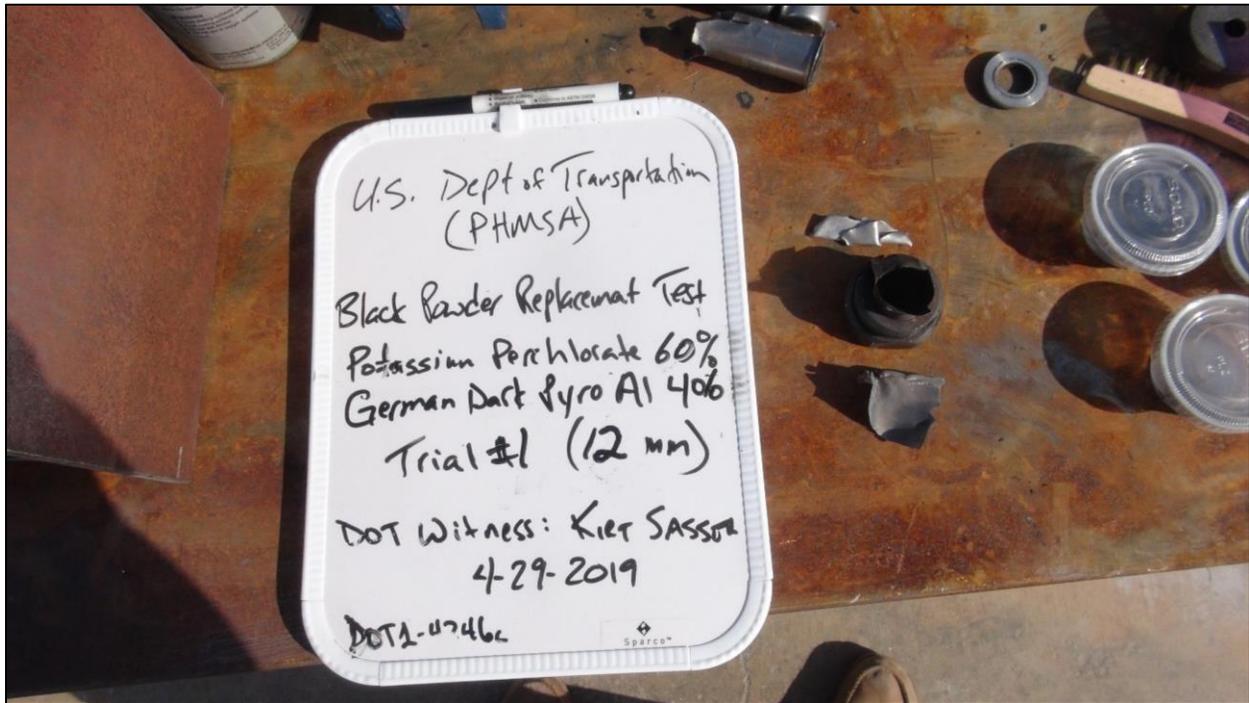


Photo 114: 60% Potassium Perchlorate, 40% German Dark Pyro Al @ 12 mm (5 Grams) – FAIL



Photo 115: Spanish Salute @ 22 mm (5 Grams) – FAIL

c. Conclusions

The results of the set of test data indicate that the limiting diameter for commercial black powder 5Fg is 3 mm. This would be expected based on previous test data showing 4Fg having a limited diameter of 5 mm. Again, the tests also indicate that the new Burst Charge Equivalency Test appears to provide the data desired of such a test and is demonstrating to be easy to operate and easy to interpret. Compositions like the meal powder would be expected to have limiting diameters near that of black powder. The testing confirms this, showing a limiting diameter of 3 mm for the meal powder.

It has hypothesized that this test method would have the range to predict compositions containing metal all the way up to flash compositions. This phase of testing indicates that this is true, with a current standard break charge composition like 60% Potassium Perchlorate/ 40% German Dark Pyro Aluminum having a limiting diameter of 15 mm and a non-metallic 70% Potassium Perchlorate/ 30% Potassium Benzoate having a limiting diameter of 12 mm.

Table 20 provides the estimated limiting diameter based on the testing performed. The powders arranged in ascending order based on the limiting diameter.

Table 20: U.S. Based Break Powder Compositions Limiting Diameters

<u>Material</u>	<u>Limiting Diameter (mm)</u>
Meal Powder	3
GOEX 5FBP (unglazed)	3
Black Powder, 5% Al Powder (GB) Optimized	< 5
Black Powder with 10% Al Optimized	5
35% Potassium Nitrate, 23% Potassium Perchlorate, 13% Potassium Benzoate, 10% Charcoal	5
GOEX Meal Powder 90%, Al Powder (GB) 10%	8
68% Barium Nitrate, 9% Sulfur, 23% Aluminum (GB)	8
70% Potassium Perchlorate, 20% Potassium Benzoate, 10% Air Float Charcoal	10
70% Potassium Perchlorate, 30% Potassium Benzoate	12
60% Potassium Perchlorate, 40% German Dark Pyro Aluminum	15
50% Potassium Perchlorate, 50 % Magnesium (44 microns or smaller)	< 18
Potassium Perchlorate 65%, German Dark Pyro Aluminum 35%	> 22
Potassium Perchlorate 70%, German Dark Pyro Aluminum 30%	> 22
Spanish Salute Powder (#7)	> 22

8. HLS Flash Testing Compositions (SMS-3524-R1)

Additional testing was performed on selected break charge formulations. The test results are presented in this section.

a. Test Configuration – Flash Composition Testing - Electric Match Ignition Source

The newly developed Burst Charge Equivalency Test Fixture with an electric match ignition source was used as the test configuration of this data set. Tests were performed at SMS’s test facility at the Tooele Army Depot. Testing was performed on compositions previously used in flash composition testing

The following powders were tested in this phase of testing using an electric match ignition source. The formulations were unknown. The samples correspond to samples tested under SMS-3524-R1. The samples were extracted from assembled firework shells. The samples were as follows:

- Flash Report
- Theatrical Flash
- 15’ Sparkblast
- Waterfall
- 50’ – 60’ Yellow Mine
- Le Maitre Whistle Comp
- Super Gunshot Red

The following photos show the powders under review.



Photo 116: Flash Report No. 2 (PP247)



Photo 117: Theatrical Flash Large (1200B)



Photo 118: Theatrical Flash Large (1200B)



Photo 119: 15' Sparkburst (PP483)



Photo 120: Waterfall 15 x 15 (PP357)



Photo 121: Waterfall 15 x 15 (PP357)



Photo 122: Waterfall 15 x 15 (PP357)



Photo 123: 1.5" 50-60' Mine Yellow (PP575)



Photo 124: 1.5" 50-60' Mine Yellow (PP575)



Photo 125: Whistle Comp, 4sec (PP334)



Photo 126: Whistle Comp



Photo 127: Whistle Comp, 4sec (PP334)



Photo 128: Super Gunshot Red

b. Test Results – U.S. Compositions - Electric Match Ignition Source

The following table includes the test results for compositions previously tested in the HSL research using the Koenen Assembly and an electric match ignition source.

Table 21: U.S. Based Break Powder Compositions Tested with Electric Match

<u>Material</u>	<u>Orifice Size</u>	<u>Trial</u>	<u>Results</u>	<u>Pass/ Fail</u>
Flash Report	22mm	1	“+”	Fail
Theatrical Flash	3mm	1	“-”	Pass
		2	“-”	Pass
		3	“-”	Pass
	5mm	1	“-”	Pass
15’ Sparkblast	3mm	1	“+”	Fail
	5mm	1	“-”	Pass
		2	“-”	Pass
		3	“-”	Pass
	8mm	1	“-”	Pass
	12mm	1	“-”	Pass
	20mm	1	“-”	Pass
Waterfall	10mm	1	“+”	Fail
	12mm	1	“-”	Pass
		2	“-”	Pass
		3	“-”	Pass
	15mm	1	“-”	Pass
	22mm	1	“-”	Pass
50’ – 60’ Yellow Mine	10mm	1	“+”	Fail
	12mm	1	“-”	Pass
		2	“-”	Pass
		3	“-”	Pass
Le Maitre Whistle Comp	20mm	1	“+”	Fail
	22mm	1	“-”	Pass
		2	“-”	Pass
		3	“-”	Pass
Super Gunshot Red	20mm	1	“+”	Fail
	22mm	1	“-”	Pass
		2	“-”	Pass

<u>Material</u>	<u>Orifice Size</u>	<u>Trial</u>	<u>Results</u>	<u>Pass/ Fail</u>
		3	“-“	Pass

Photos 129 – 133 show examples of the test results.

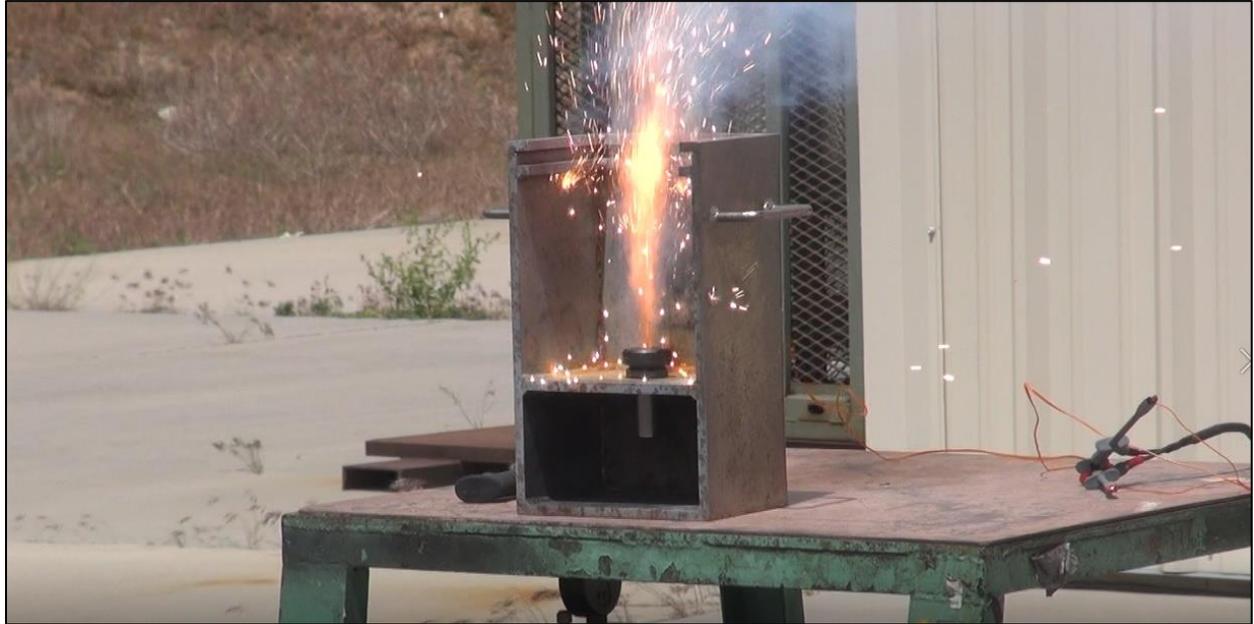


Photo 129: Waterfall @ 15 mm Vent (5 Grams) – PASS



Photo 130: Waterfall @ 10 mm Vent (5 Grams) – FAIL



Photo 131: Theatrical Flash Large @ 3 mm Vent (5 Grams) – PASS

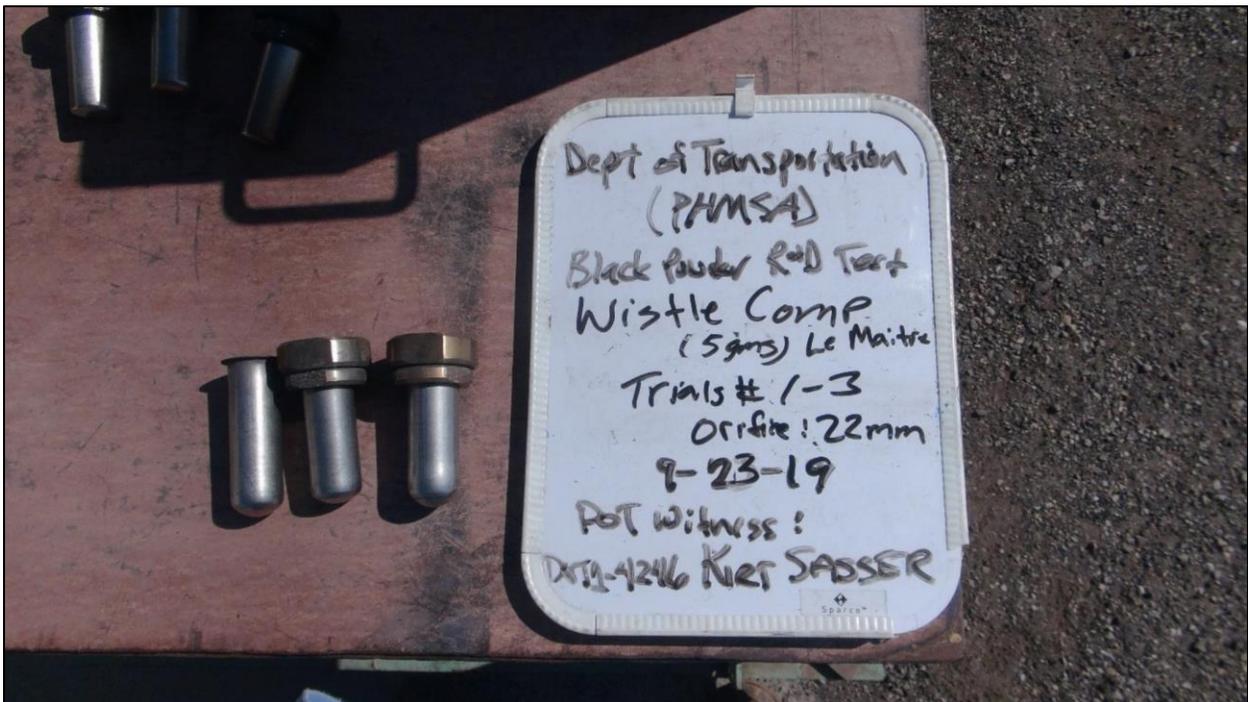


Photo 132: Whistle Comp @ 22 mm Vent (5 Grams) – PASS

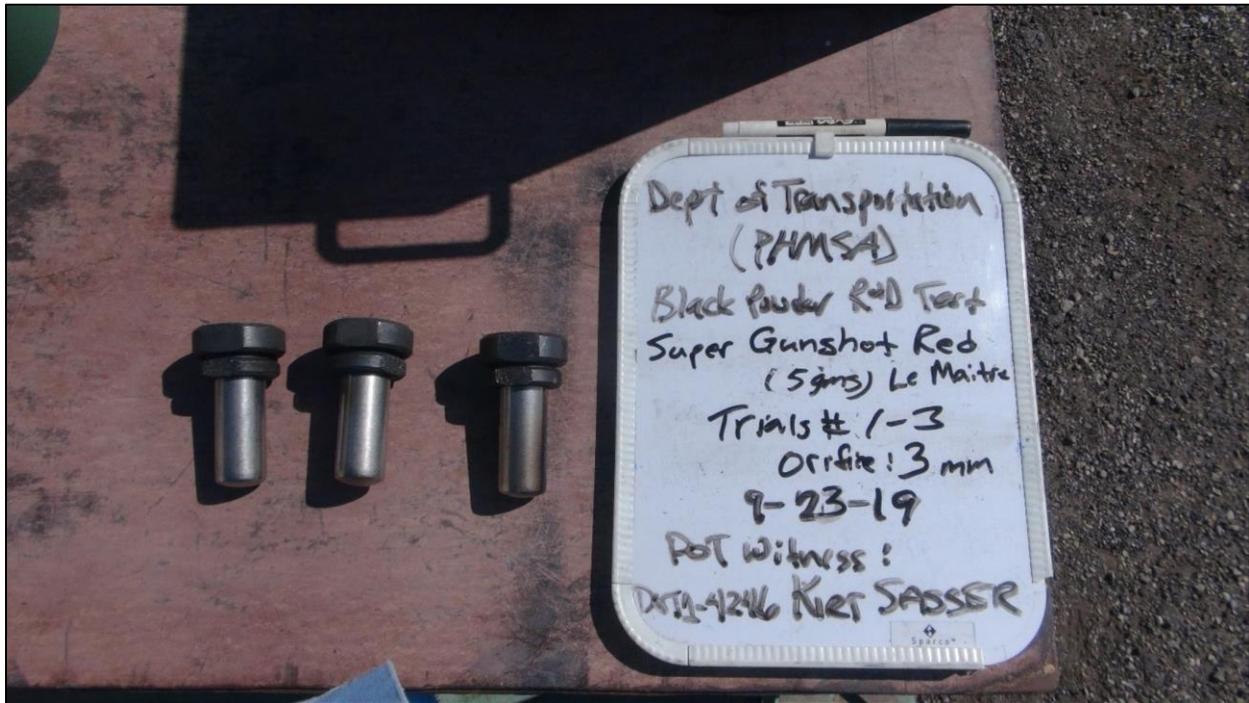


Photo 133: Super Gunshot Red @ 3 mm Vent (5 Grams) – PASS

c. Conclusions

The testing showed that the Flash Report and Whistle Comp were on the higher end of the spectrum as expected, however, no correlation could be made between this data and the HSL data.

Table 22 provides the limiting diameter based on the testing performed. This test method allows a very easy comparison of the powders to be made. The powders arranged in ascending order based on the limiting diameter.

Table 22: U.S. Based Break Powder Compositions Limiting Diameters

<u>Material</u>	<u>Limiting Diameter (mm)</u>
Theatrical Flash	< 3
15' Sparkblast	3
Waterfall	10
50' – 60' Yellow Mine	10
Le Maître Whistle Comp	20
Super Gunshot Red	20
Flash Report	> 22

9. *Vent reduction by e-match lead wires*

As previously mentioned, this test method was developed to help compare the relative energy outputs of burst charge powders for aerial fireworks shells. The use of an electric match and the consequences to the desired objective were evaluated in the development of the test method. Two obvious details with the use of the e-match are the reduction of vent area from the e-match leads and the energy released by the match head. A short discussion of these details is provided in the following:

As the objective of this test method is to give a relative comparison of the energy output of the burst powders, the reduction of vent area due to the electric match leads is inconsequential as the reduction is constant. The area taken up by the leads is small enough such that the effective vent area is always larger as the orifice diameters increase. The cross-sectional area of the electric match lead wires occupies a lesser percentage of the vent hole as the diameter of the vent hole increases.

For example, the insulation on the lead wires of the Daveyfire SA 2001 A/N 28Br electric matches used in this study each have a diameter of 1.25 mm, or a cross-sectional area of 1.23 mm². The paired, or “zipped” wires have a combined area of 2.46 mm². The cross-sectional area of the 22 mm orifice is 380 mm². The percentage of the vent area occupied by the lead wires is then,

$$(2.46 \text{ mm}^2 / 380 \text{ mm}^2) \times 100\% = 0.65\%$$

The area percentage occupied by the lead wires for the 18 mm and 15 mm orifices is 0.97 and 1.4 %, respectively. These reductions in area are considered insignificant as the system is the same for all powders being evaluated.

10. *Energetic contribution of the electric match to the energy released by the burst charge sample*

The pyrotechnic composition in a Daveyfire A/N 28Br electric match head has a mass of 80 mg and a heat of explosion comparable to those of the three burst charges tested. Thus, the energy contribution from the electric match head to the burning of 5g (5,000 mg) of a burst charge sample equates to approximately,

$$(80 \text{ mg} / 5,000 \text{ mg}) \times 100\% = 1.6\%$$

This is also judged to be insignificant, having little effect on the test results as once again, this value is constant, and the results are comparative in nature from sample to sample.

B. Standard Procedure for Burst Charge Equivalency Test (Electric Match) Ignition System

The standard test procedure for Burst Charge Equivalency Test with the electric match ignition system is outlined in the following steps. An operator:

1. Weighs out five (5) grams of the test substance and place in the bottom of the test tube.
2. Selects a vent size and assemble the orifice, collar, and nut
3. Positions test assembly in protective fixture. The fixture is positioned such that the operator approaches from the back side for safety purposes.
4. Inserts an electric match through the vent hole and down into the powder.
5. Functions the electric match.
6. Examines the test tube for damage:
 - a. If the tube does not rupture, the vent size is decreased, and the process repeated until a rupture occurs.
 - b. If the tube ruptures, the vent size is increased, and the process repeated until a rupture does not occur.
 - c. The test is then repeated until 3 trials are performed at the smallest vent size without rupture occurring.
 - d. The limiting diameter (LD) is the orifice diameter at which the result "explosion" is obtained just below the smallest vent size where 3 trials are performed at without rupture occurring.
 - e. The limiting diameter (LD) can be compared to other powders performed using this same method.

C. Cost Analysis

Based on the data presented, this test method provides a valuable resource to understanding burst powder comparisons for relatively an inexpensive cost. A cost analysis was performed to determine the costs for performing this test. These are as follows:

• Initial Setup:	1 set of collars (3), nuts (3), orifices (9) (reusable)	\$1,725
• Protective Device:	1 device (reusable)	\$500-\$1,500
• Koenen Tubes:	1 tube (non-reusable)	\$40
• Electric Match:	1 match	\$5
• Total Initial Setup Costs:		<u>\$3,225</u>
• Price per Sample (internal)		\$315

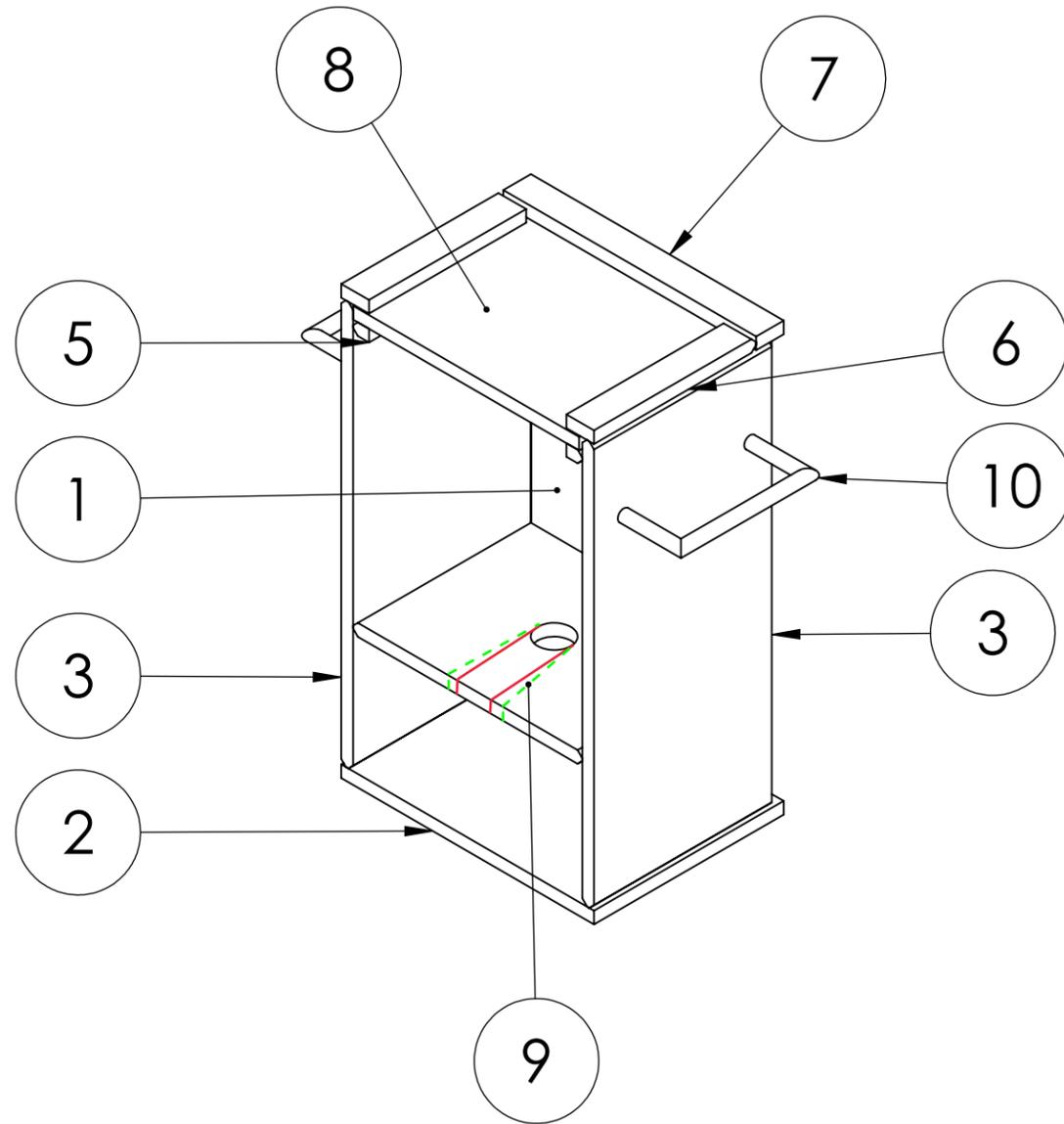
The initial setup and protective device will range from \$1,500 to \$3,225. Internal manufacturing would reduce this cost significantly. Based on the testing performed, the limiting diameter was established between approximately 5-7 trials. This would equate to \$225 to \$315 of consumable costs to establish the limiting diameter per composition. It is estimated

that one trial could be performed every 30 minutes (conservatively). Internally then, the cost for establish the limiting diameter would be no more than \$315 per sample. Commercial costs (outside lab) would be much higher and are estimated as follows assuming samples are provided test ready:

- 1 Sample: \$1,300
- 5 Samples: \$5,320
- 15 Samples: \$10,100
- 15 Samples: \$14,800
- 20 Samples: \$19,450

V. APPENDIX – PROTECTIVE DEVICE DRAWINGS

ITEM NO.	PART NUMBER	DESCRIPTION	QTY.
1	KNNSHLD-005	BACK	1
2	KNNSHLD-004	BOTTOM	1
3	KNNSHLD-003	SIDES	2
4	KNNSHLD-007	LEXAN SUPPORT RAIL	1
5	KNNSHLD-008	LEXAN SUPPORT RAIL	2
6	KNNSHLD-009	LEXAN SUPPORT RAIL	2
7	KNNSHLD-011	LEXAN SUPPORT RAIL	1
8	KNNSHLD-012	TOP (3/8" LEXAN)	1
9	KNNSHLD-006	TUBE SUPPORT	1
10	KNNSHLD-013	HANDLES	2



NOTES:

1. UNLESS OTHERWISE SPECIFIED, INTERPRET DIMENSIONS, SYMBOLS, ETC. IN ACCORDANCE WITH APPLICABLE NOTES AND DOCUMENTS LISTED:

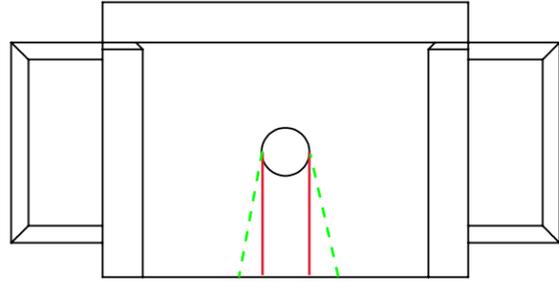
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 FILLET RADII TO BE 1/32 MAX
 DIMENSIONS AND TOLERANCES USASI Y14.5-66
 ABBREVIATIONSMIL-STD-12

SURFACE TEXTURE TO BE $\sqrt{125}$ USASI B46.1-62
 THREAD DIM. & TOL NBS HANDBOOK H-28
 WELD SYMBOLS AWS A2.0-58
 DRILLED HOLE TOLERANCES AND 10387

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		UNLESS OTHERWISE SPECIFIED:				SMS Inc.	
		DIMENSIONS ARE IN INCHES		DRAWN			
		TOLERANCES:		CHECKED		BURST CHARGE EQUIVALENCY TEST FIXTURE	
		FRACTIONAL ±		ENG APPR.			
		ANGULAR: MACH ± BEND ±		MFG APPR.			
		TWO PLACE DECIMAL ±		Q.A.		SIZE DWG. NO. REV	
		THREE PLACE DECIMAL ±		COMMENTS:		B KNNSHLD-000-01 NR	
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APPLICATION				DO NOT SCALE DRAWING			

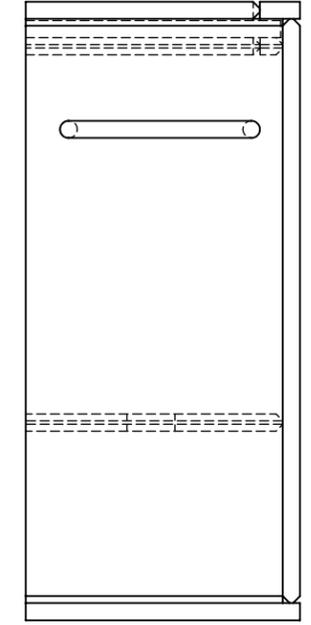
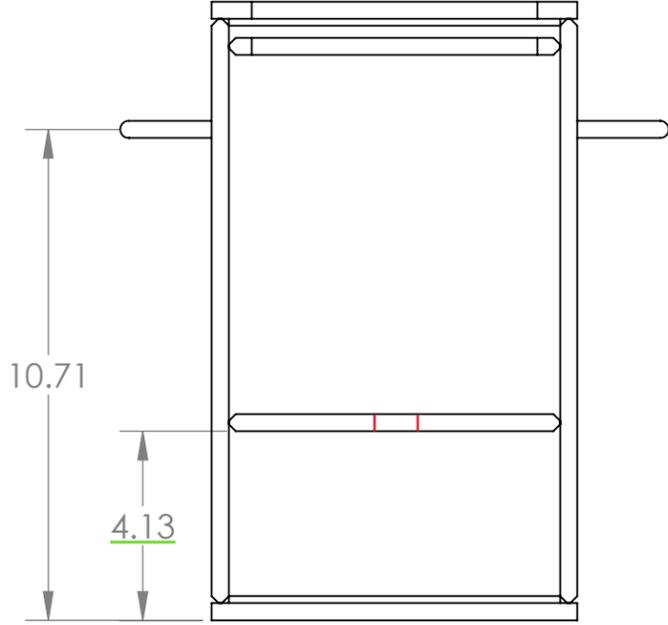
8 7 6 5 4 3 2 1



— TUBE SUPPORT: OPTION 1 - SLOT
 - - - TUBE SUPPORT: OPTION 2 - TAPERED SLOT

REVISIONS				
ZONE	REV.	DESCRIPTION	DATE	APPROVED
	NR	NEW RELEASE	4/19/2017	RKC

NOTE: FULL PENETRATING WELDS ON ALL EDGES.



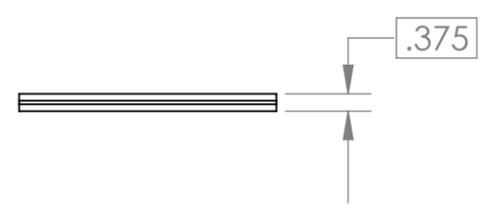
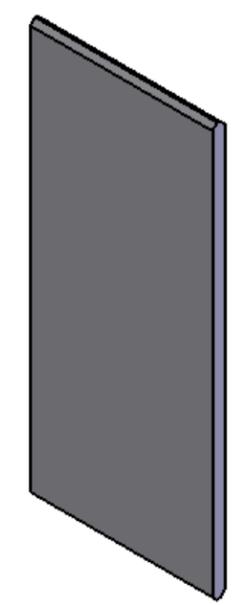
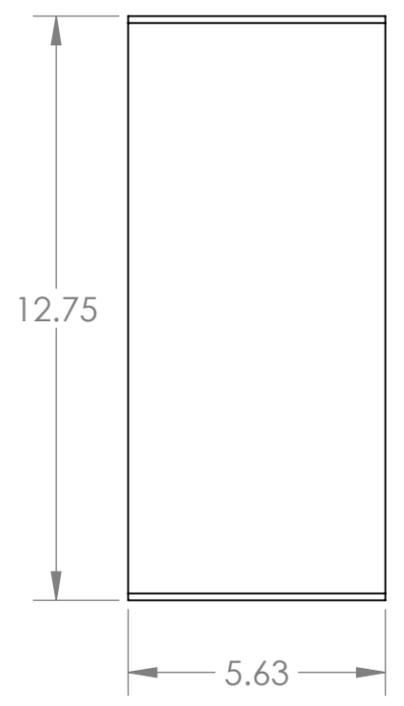
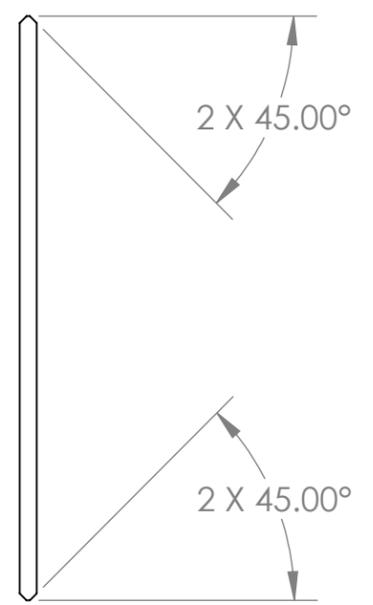
NOTES:
 1. UNLESS OTHERWISE SPECIFIED, INTERPRET DIMENSIONS, SYMBOLS, ETC. IN ACCORDANCE WITH APPLICABLE NOTES AND DOCUMENTS LISTED:
 BREAK SHARP EDGES .003 TO .015 R
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 ABBREVIATIONSMIL-STD-12
 SURFACE TEXTURE TO BE $\sqrt{125}$ USASI B46.1-62
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FRACTIONAL ± .125		ENG APPR.			
ANGULAR: MACH ± BEND ±		MFG APPR.			
TWO PLACE DECIMAL ± .05		Q.A.			
THREE PLACE DECIMAL ± .01		COMMENTS:			
INTERPRET GEOMETRIC TOLERANCING PER:					
MATERIAL					
FINISH					
BLACK ZINC CHROMATE					
NEXT ASSY	USED ON				
APPLICATION		DO NOT SCALE DRAWING			
SIZE	DWG. NO.	REV			
B	KNNSHLD-000	NR			
SCALE: 1:4	WEIGHT:	SHEET 2 OF 2			

8 7 6 5 4 3 2 1

REVISIONS				
ZONE	REV.	DESCRIPTION	DATE	APPROVED
	NR	NEW RELEASE	4/19/2017	RKC



NOTES:

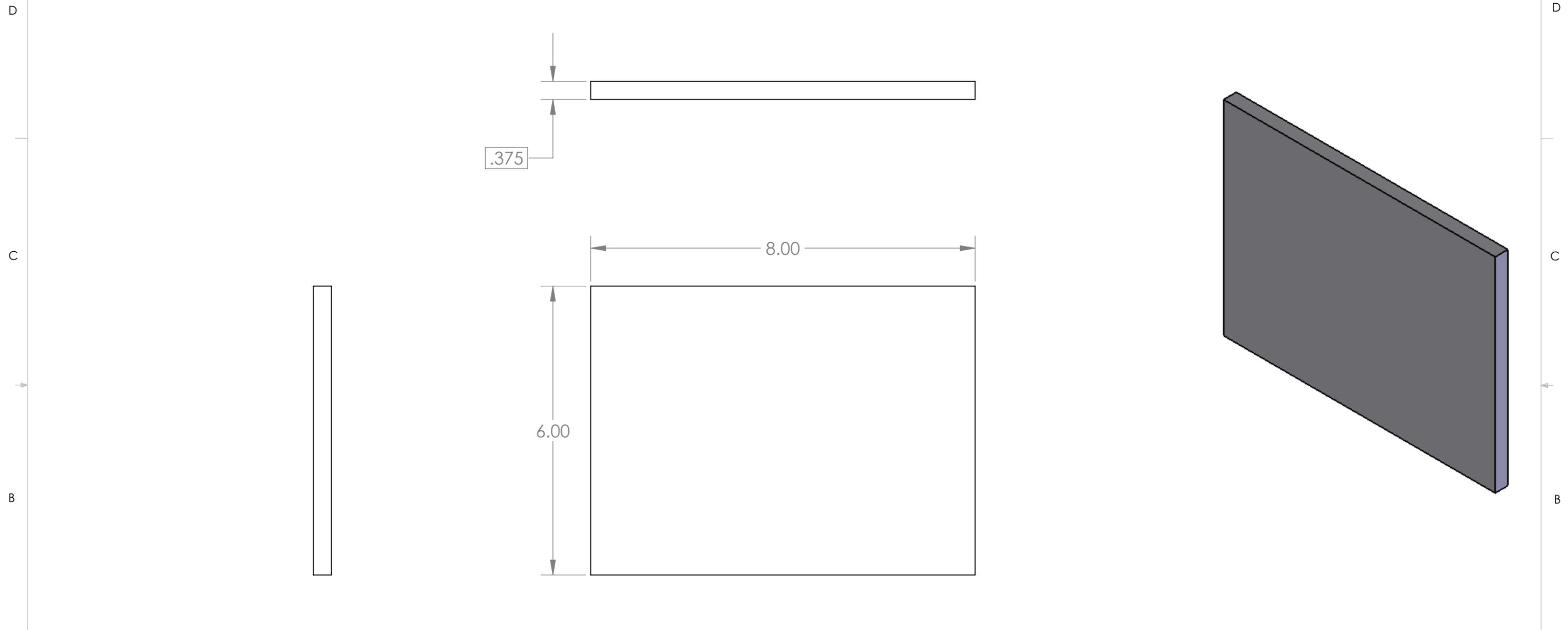
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 BREAK SHARP EDGES .003 TO .015 R
 FILLET RADII TO BE 1/32 MAX
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SURFACE TEXTURE TO BE $\sqrt{125}$ USASI B46.1-62
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		TOLERANCES:		CHECKED			
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REVISIONS				
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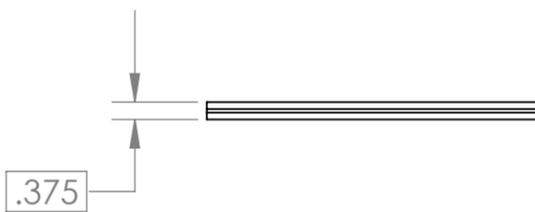
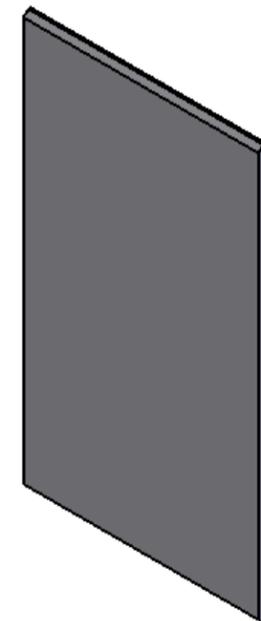
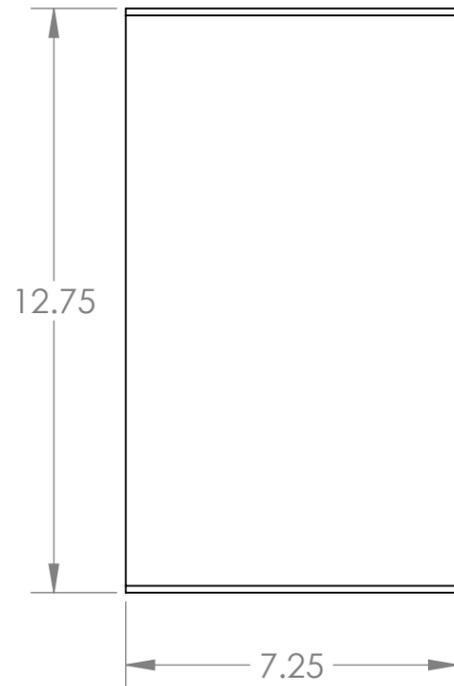
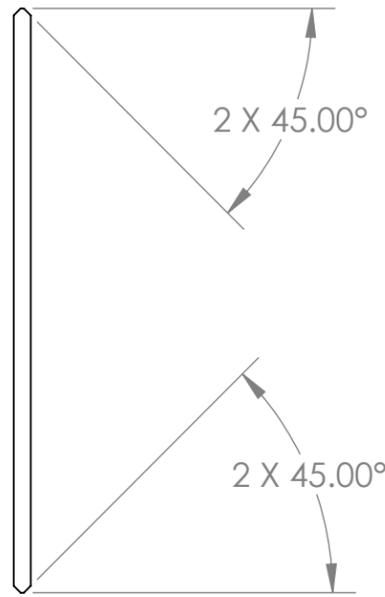
NOTES:

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 FILLET RADII TO BE 1/32 MAX
 DIMNESIONS AND TOLERANCESUSASI Y14.5-66
 ABBREVIATIONSMIL-STD-12
 SURFACE TEXTURE TO BE $\sqrt{125}$ USASI B46.1-62
 THREAD DIM. & TOL NBS HANDBOOK H-28
 WELD SYMBOLS AWS A2.0-58
 DRILLED HOLE TOLERANCES AND 10387

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FRACTIONAL ± .125		ENG APPR.				
ANGULAR: MACH ± BEND ±		MFG APPR.			SIZE B DWG. NO. KNNSHLD-004 REV NR	
TWO PLACE DECIMAL ± .05		Q.A.				
THREE PLACE DECIMAL ± .01		COMMENTS:			SCALE: 1:2 WEIGHT: SHEET 1 OF 1	
INTERPRET GEOMETRIC TOLERANCING PER:						
MATERIAL		A36				
FINISH		BLACK OXIDE				
APPLICATION	DO NOT SCALE DRAWING					

REVISIONS				
ZONE	REV.	DESCRIPTION	DATE	APPROVED
	NR	NEW RELEASE	4/19/2017	RKC



NOTES:

1. UNLESS OTHERWISE SPECIFIED, INTERPRET DIMENSIONS, SYMBOLS, ETC. IN ACCORDANCE WITH APPLICABLE NOTES AND DOCUMENTS LISTED:

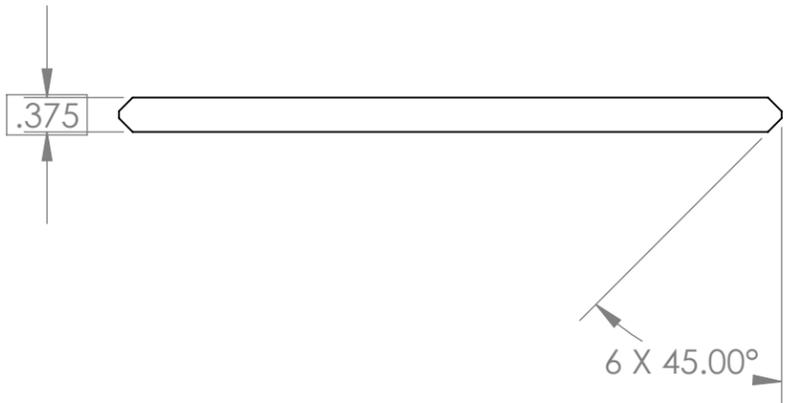
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 ABBREVIATIONSMIL-STD-12

SURFACE TEXTURE TO BE $\sqrt{125}$ USASI B46.1-62
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 DRILLED HOLE TOLERANCES AND 10387

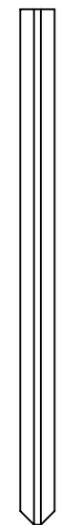
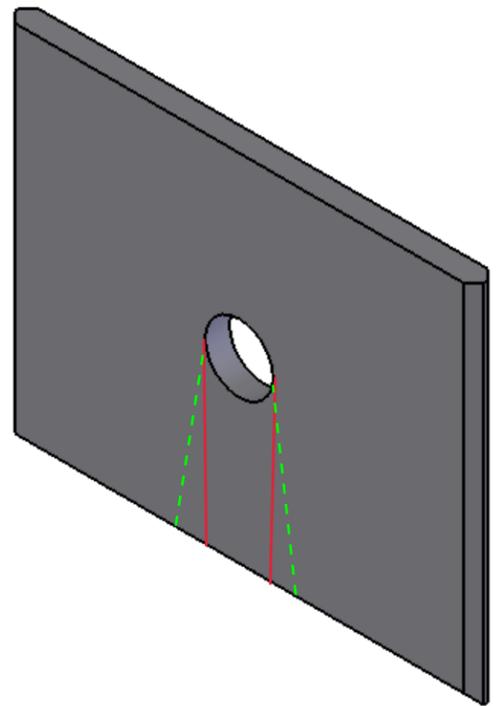
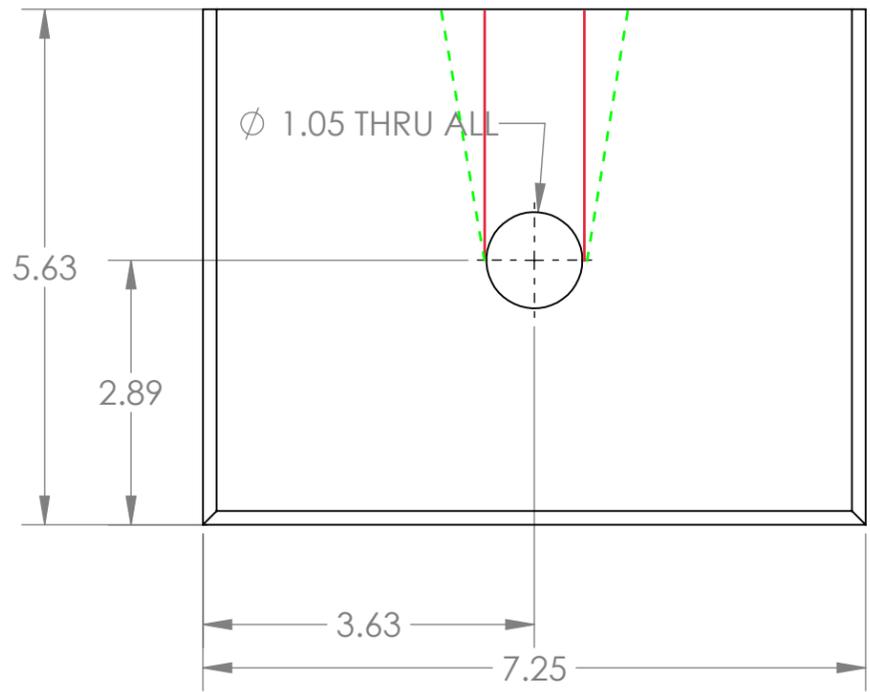
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		UNLESS OTHERWISE SPECIFIED:		NAME	DATE	SMS Inc. TITLE: BACK SIZE DWG. NO. REV B KNNSHLD-005 NR	
		DIMENSIONS ARE IN INCHES		DRAWN	RKC		4/19/2017
		TOLERANCES:		CHECKED			
		FRACTIONAL ± .125		ENG APPR.			
		ANGULAR: MACH ± BEND ±		MFG APPR.			
		TWO PLACE DECIMAL ± .05		Q.A.			
		THREE PLACE DECIMAL ± .01		COMMENTS:			
		INTERPRET GEOMETRIC TOLERANCING PER:					
		MATERIAL					
		A36					
		FINISH					
		BLACK OXIDE					
NEXT ASSY	USED ON	APPLICATION		DO NOT SCALE DRAWING			

REVISIONS				
ZONE	REV.	DESCRIPTION	DATE	APPROVED
	NR	NEW RELEASE	4/19/2017	RKC



— TUBE SUPPORT: OPTION 1 - SLOT
 - - - TUBE SUPPORT: OPTION 1 - TAPERED SLOT



NOTES:
 1. UNLESS OTHERWISE SPECIFIED, INTERPRET DIMENSIONS, SYMBOLS, ETC. IN ACCORDANCE WITH APPLICABLE NOTES AND DOCUMENTS LISTED:
 BREAK SHARP EDGES .003 TO .015 R
 FILLET RADII TO BE 1/32 MAX
 DIMENSIONS AND TOLERANCESUSASI Y14.5-66
 ABBREVIATIONSMIL-STD-12
 SURFACE TEXTURE TO BE $\sqrt{125}$ USASI B46.1-62
 THREAD DIM. & TOLNBS HANDBOOK H-28
 WELD SYMBOLSAWS A2.0-58
 DRILLED HOLE TOLERANCESAND 10387

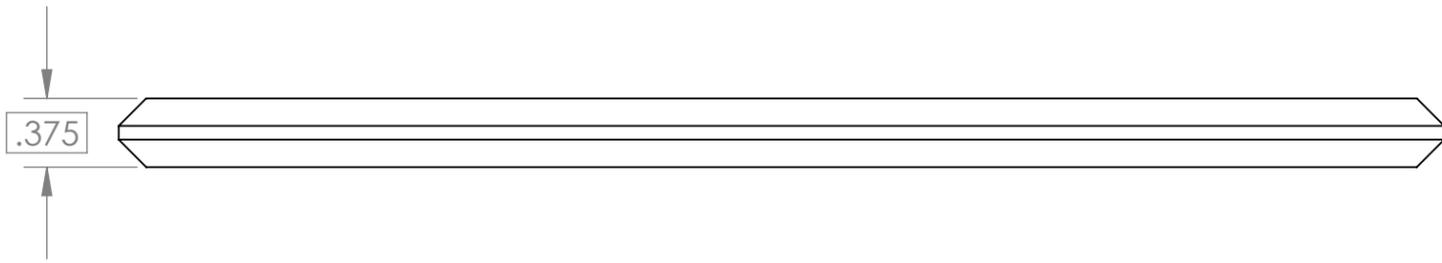
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UNLESS OTHERWISE SPECIFIED:		NAME	DATE	SMS Inc. TITLE: TUBE SUPPORT SIZE DWG. NO. REV B KNNSHLD-006-01 NR SCALE: 1:1 WEIGHT: SHEET 1 OF 1
DIMENSIONS ARE IN INCHES		DRAWN	RKC	
TOLERANCES:		CHECKED		
FRACTIONAL ± .125		ENG APPR.		
ANGULAR: MACH ± BEND ±		MFG APPR.		
TWO PLACE DECIMAL ± .05		Q.A.		
THREE PLACE DECIMAL ± .01		COMMENTS:		
INTERPRET GEOMETRIC TOLERANCING PER:				
MATERIAL				
A36				
FINISH				
BLACK OXIDE				
NEXT ASSY	USED ON			
APPLICATION	DO NOT SCALE DRAWING			

8 7 6 5 4 3 2 1

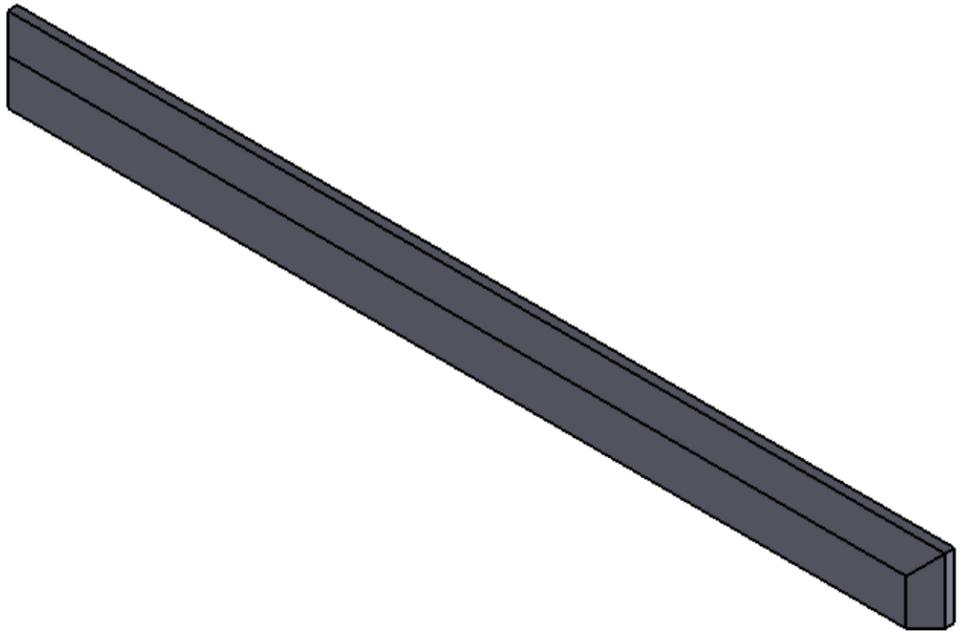
REVISIONS				
ZONE	REV.	DESCRIPTION	DATE	APPROVED
	NR	NEW RELEASE	4/19/2017	RKC

D



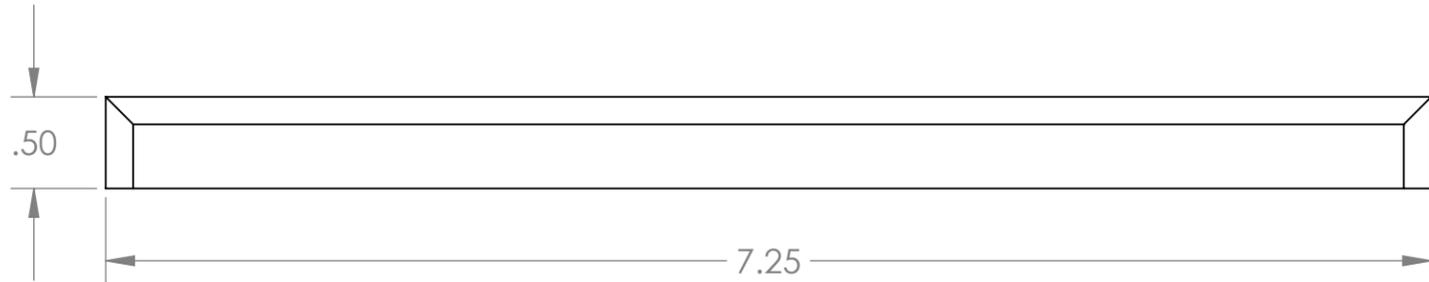
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C

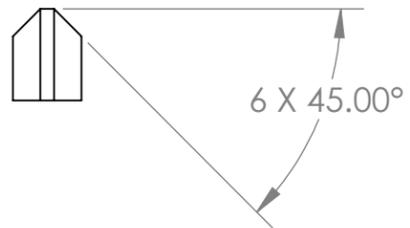


C

B



B



NOTES:

1. UNLESS OTHERWISE SPECIFIED, INTERPRET DIMENSIONS, SYMBOLS, ETC. IN ACCORDANCE WITH APPLICABLE NOTES AND DOCUMENTS LISTED:

BREAK SHARP EDGES .003 TO .015 R
 FILLET RADII TO BE 1/32 MAX
 DIMNESIONS AND TOLERANCESUSASI Y14.5-66
 ABBREVIATIONSMIL-STD-12

SURFACE TEXTURE TO BE $\sqrt{125}$ USASI B46.1-62
 THREAD DIM. & TOLNBS HANDBOOK H-28
 WELD SYMBOLSAWS A2.0-58
 DRILLED HOLE TOLERANCESAND 10387

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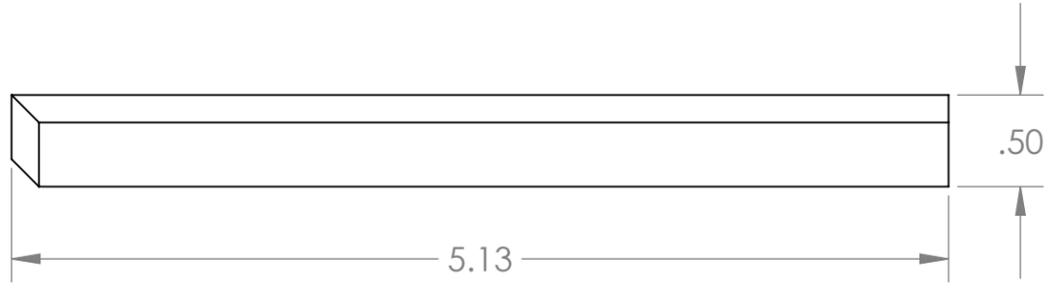
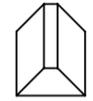
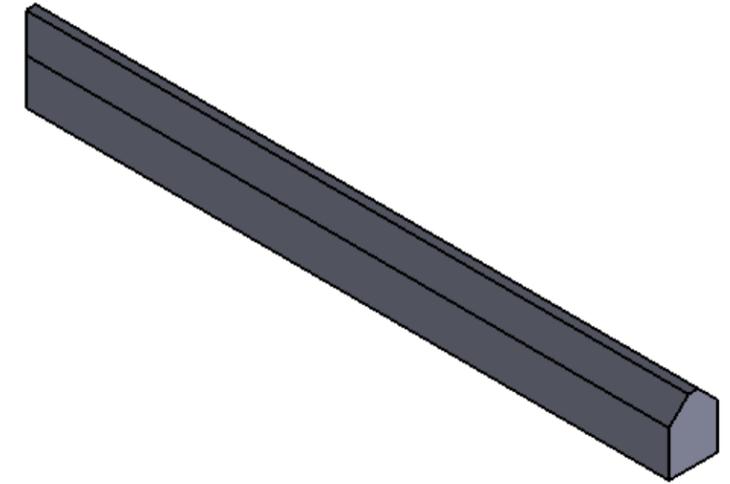
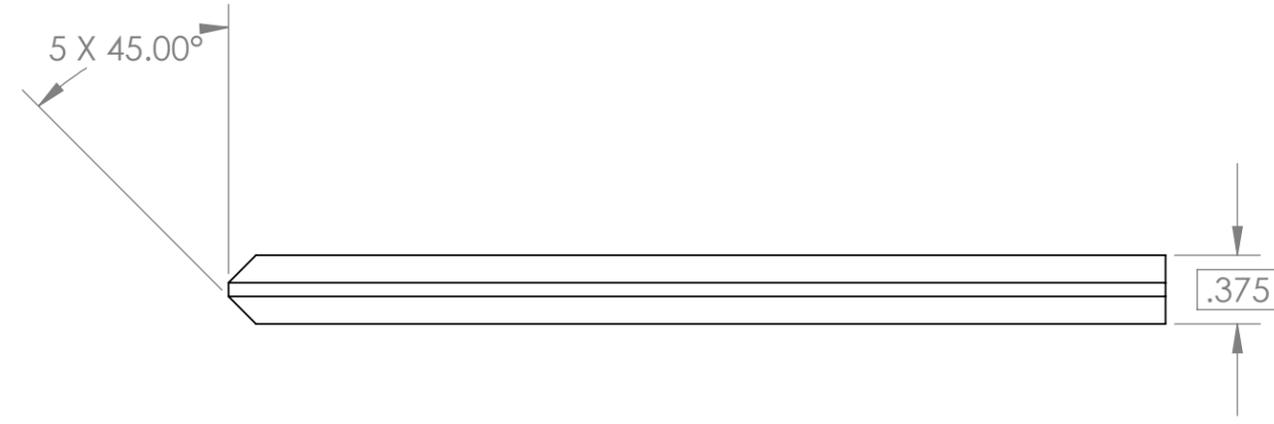
		UNLESS OTHERWISE SPECIFIED:				NAME		DATE		<h1>SMS Inc.</h1>			
		DIMENSIONS ARE IN INCHES		DRAWN		RKC		4/19/2017				TITLE:	
		TOLERANCES:		CHECKED									
		FRACTIONAL ± .125		ENG APPR.									
		ANGULAR: MACH ± BEND ±		MFG APPR.						SIZE DWG. NO. REV B KNNSHLD-007 NR			
		TWO PLACE DECIMAL ± .05		Q.A.									
		THREE PLACE DECIMAL ± .01		COMMENTS:						SCALE: 1:1 WEIGHT: SHEET 1 OF 1			
		INTERPRET GEOMETRIC TOLERANCING PER:		MATERIAL		A36							
		MATERIAL		FINISH		BLACK OXIDE							
NEXT ASSY		USED ON		APPLICATION		DO NOT SCALE DRAWING							

A

A

8 7 6 5 4 3 2 1

REVISIONS				
ZONE	REV.	DESCRIPTION	DATE	APPROVED
	NR	NEW RELEASE	4/19/2017	RKC



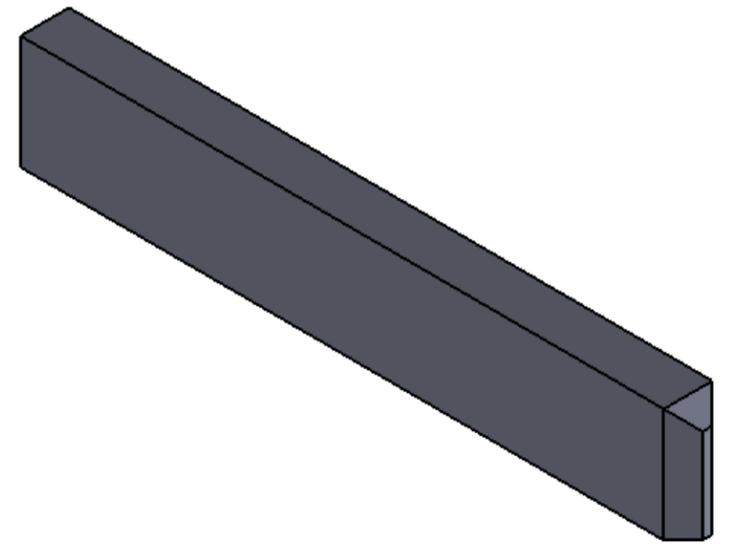
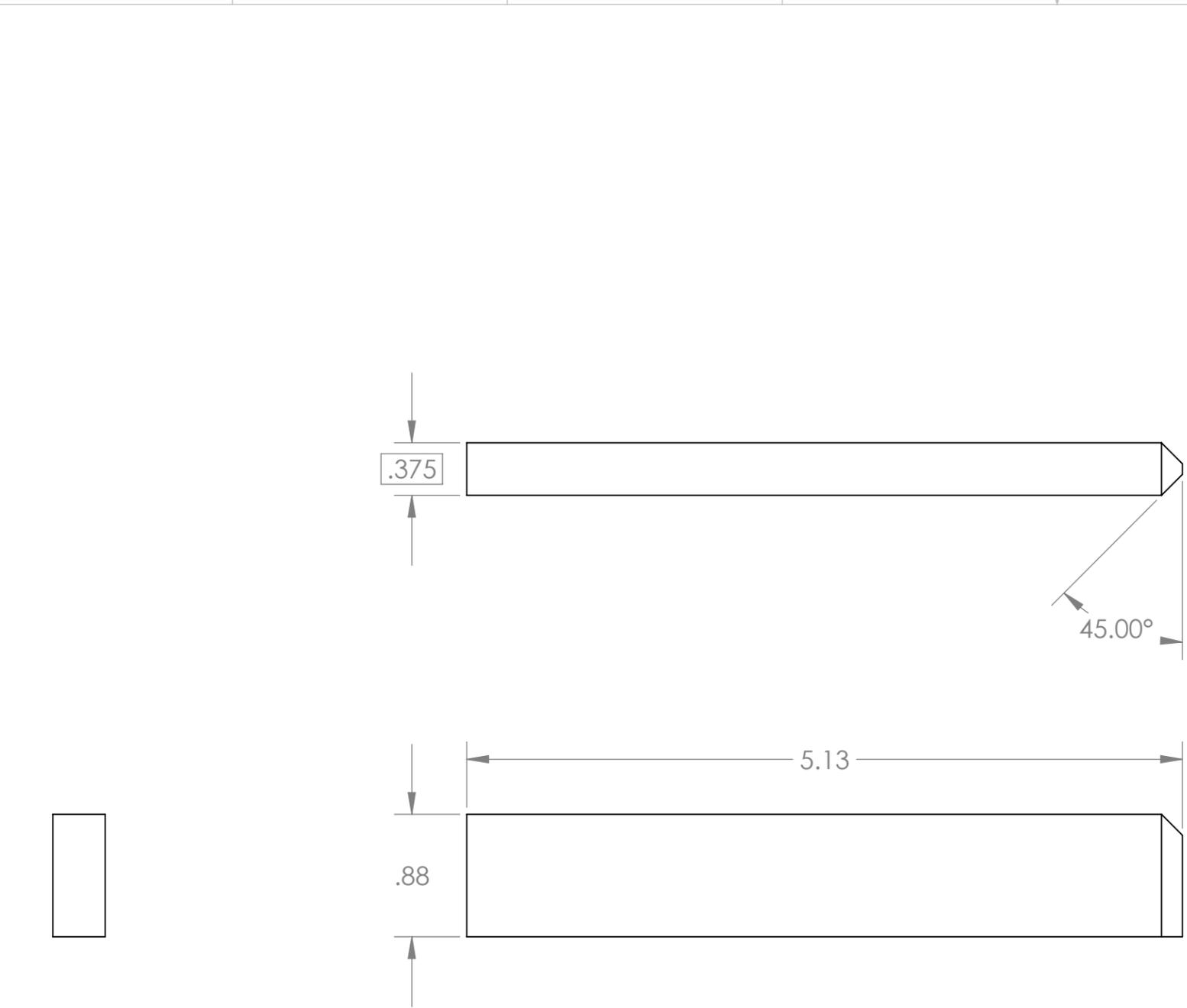
NOTES:

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 BREAK SHARP EDGES .003 TO .015 R
 FILLET RADII TO BE 1/32 MAX
 DIMNESIONS AND TOLERANCESUSASI Y14.5-66
 ABBREVIATIONSMIL-STD-12

SURFACE TEXTURE TO BE $\sqrt{125}$ USASI B46.1-62
 THREAD DIM. & TOL NBS HANDBOOK H-28
 WELD SYMBOLS AWS A2.0-58
 DRILLED HOLE TOLERANCES AND 10387

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UNLESS OTHERWISE SPECIFIED:		NAME	DATE	SMS Inc.		
DIMENSIONS ARE IN INCHES		DRAWN	RKC		TITLE:	
TOLERANCES:		CHECKED				
FRACTIONAL ± .125		ENG APPR.				
ANGULAR: MACH ± BEND ±		MFG APPR.				
TWO PLACE DECIMAL ± .05		Q.A.		QTY: 2		
THREE PLACE DECIMAL ± .01		COMMENTS:				
INTERPRET GEOMETRIC TOLERANCING PER:		SCALE: 1:1 WEIGHT: SHEET 1 OF 1				
MATERIAL				SIZE	DWG. NO.	REV
A36				B	KNNSHLD-008	NR
FINISH		DO NOT SCALE DRAWING				
BLACK OXIDE						
NEXT ASSY	USED ON					
APPLICATION						



REVISIONS				
ZONE	REV.	DESCRIPTION	DATE	APPROVED
	NR	NEW RELEASE	4/19/2017	RKC

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 DIMNESIONS AND TOLERANCES USASI Y14.5-66
 ABBREVIATIONSMIL-STD-12

SURFACE TEXTURE TO BE $\sqrt{125}$ USASI B46.1-62
 THREAD DIM. & TOLNBS HANDBOOK H-28
 WELD SYMBOLS AWS A2.0-58
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DIMENSIONS ARE IN INCHES		DRAWN	RKC			4/19/2017
TOLERANCES:		CHECKED				
FRACTIONAL ± .125		ENG APPR.				
ANGULAR: MACH ± BEND ±		MFG APPR.			TITLE:	
TWO PLACE DECIMAL ± .05		Q.A.			SIZE	
THREE PLACE DECIMAL ± .01		COMMENTS:			DWG. NO.	
INTERPRET GEOMETRIC TOLERANCING PER:		QTY: 2			REV	
MATERIAL					NR	
A36						
FINISH						
BLACK OXIDE						
NEXT ASSY	USED ON			SCALE: 1:1	WEIGHT:	
APPLICATION	DO NOT SCALE DRAWING			SHEET 1 OF 1		



REVISIONS				
ZONE	REV.	DESCRIPTION	DATE	APPROVED
	NR	NEW RELEASE	4/19/2017	RKC

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 DIMNESIONS AND TOLERANCESUSASI Y14.5-66
 ABBREVIATIONSMIL-STD-12

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 THREAD DIM. & TOL NBS HANDBOOK H-28
 WELD SYMBOLS AWS A2.0-58
 DRILLED HOLE TOLERANCES AND 10387

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		UNLESS OTHERWISE SPECIFIED:	NAME	DATE	SMS Inc.	
		DIMENSIONS ARE IN INCHES	DRAWN	RKC		TITLE:
		TOLERANCES:	CHECKED			
		FRACTIONAL \pm .125	ENG APPR.			
		ANGULAR: MACH \pm BEND \pm	MFG APPR.			
		TWO PLACE DECIMAL \pm .05	Q.A.			
		THREE PLACE DECIMAL \pm .01	COMMENTS:			
		INTERPRET GEOMETRIC TOLERANCING PER:				
		MATERIAL				
		A36				
		FINISH				
		BLACK OXIDE				
NEXT ASSY	USED ON					
APPLICATION		DO NOT SCALE DRAWING				
SIZE	DWG. NO.	REV				
B	KNNSHLD-011	NR				
SCALE: 1:1	WEIGHT:	SHEET 1 OF 1				

8 7 6 5 4 3 2 1

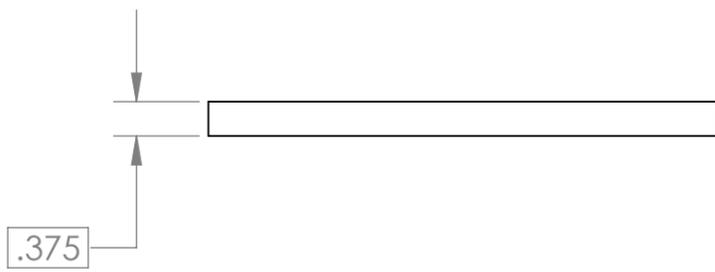
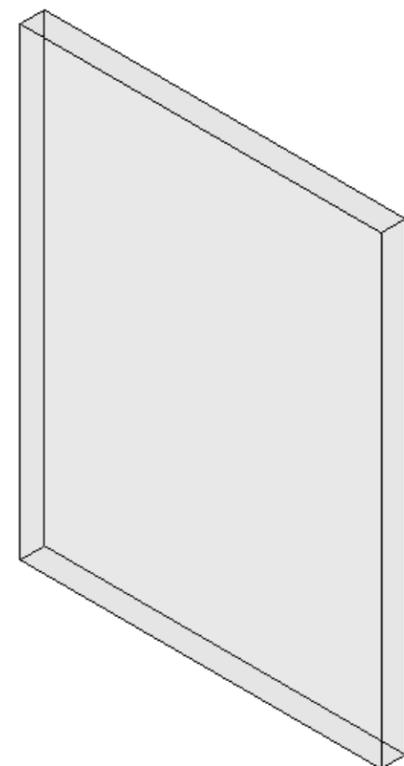
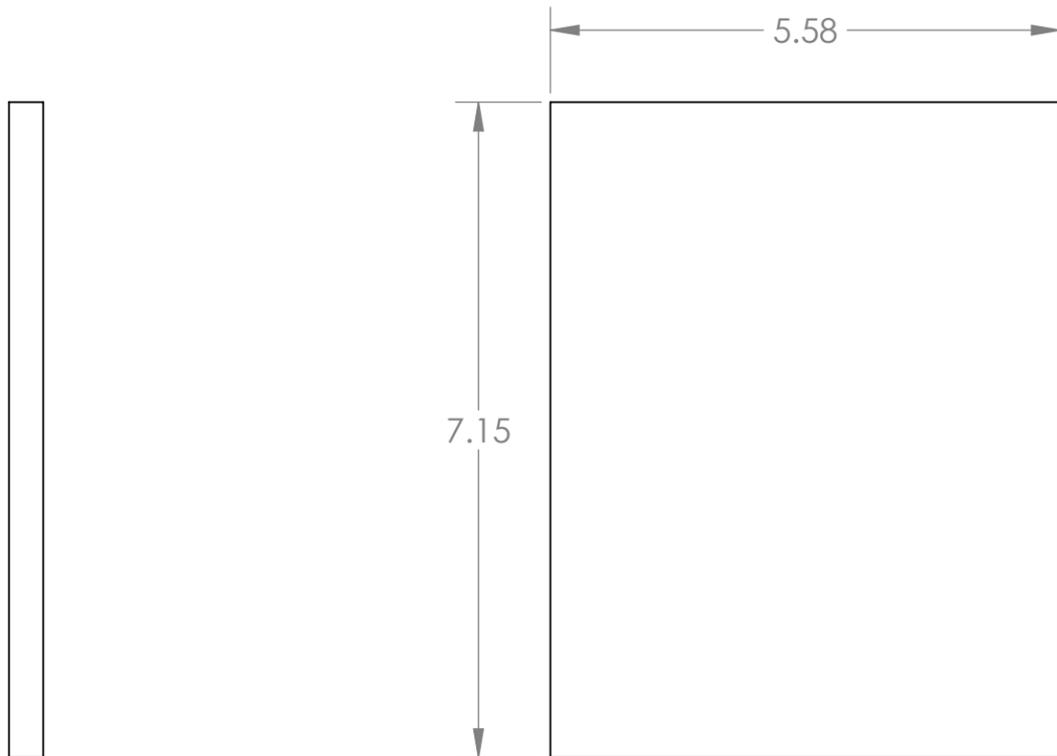
REVISIONS				
ZONE	REV.	DESCRIPTION	DATE	APPROVED
	NR	NEW RELEASE	4/19/2017	RKC

D

C

B

A



NOTES:

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 BREAK SHARP EDGES .003 TO .015 R
 FILLET RADII TO BE 1/32 MAX
 DIMENSIONS AND TOLERANCESUSASI Y14.5-66
 ABBREVIATIONSMIL-STD-12

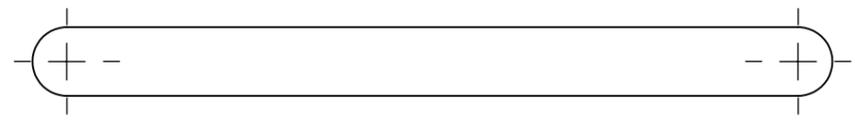
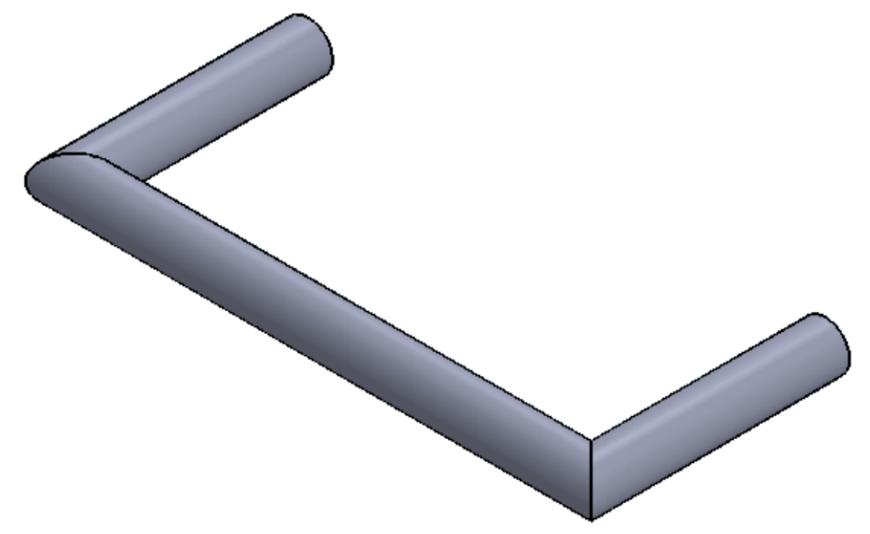
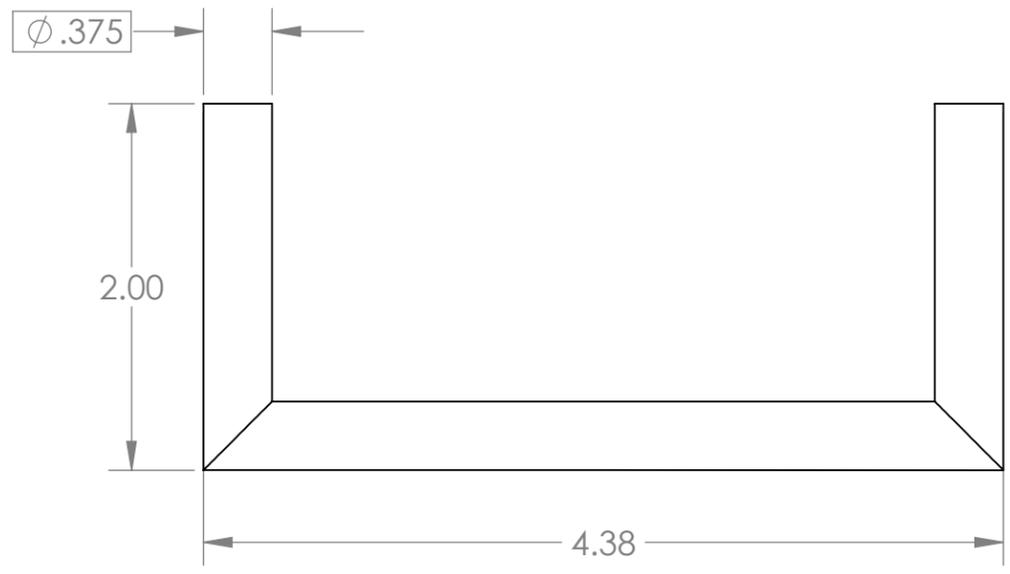
SURFACE TEXTURE TO BE $\sqrt{125}$ USASI B46.1-62
 THREAD DIM. & TOL NBS HANDBOOK H-28
 WELD SYMBOLS AWS A2.0-58
 DRILLED HOLE TOLERANCES AND 10387

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		UNLESS OTHERWISE SPECIFIED:		NAME	DATE	<h1>SMS Inc.</h1>			
		DIMENSIONS ARE IN INCHES		DRAWN	RKC		<p>TITLE:</p> <h2>TOP</h2>		
		TOLERANCES:		CHECKED					
		FRACTIONAL ± .125		ENG APPR.					
		ANGULAR: MACH ± BEND ±		MFG APPR.					
		TWO PLACE DECIMAL ± .05		Q.A.					
		THREE PLACE DECIMAL ± .01		COMMENTS:					
		INTERPRET GEOMETRIC TOLERANCING PER:				SIZE	DWG. NO.	REV	
		MATERIAL				B	KNNSHLD-012	NR	
		FINISH				SCALE: 1:2		WEIGHT:	SHEET 1 OF 1
NEXT ASSY		USED ON							
APPLICATION		DO NOT SCALE DRAWING							

8 7 6 5 4 3 2 1

REVISIONS				
ZONE	REV.	DESCRIPTION	DATE	APPROVED
	NR	NEW RELEASE	4/19/2017	RKC



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 THREAD DIM. & TOL NBS HANDBOOK H-28
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		DIMENSIONS ARE IN INCHES	DRAWN	RKC		4/19/2017
		TOLERANCES:	CHECKED			
		FRACTIONAL $\pm .125$	ENG APPR.			
		ANGULAR: MACH \pm BEND \pm	MFG APPR.			
		TWO PLACE DECIMAL $\pm .05$	Q.A.			
		THREE PLACE DECIMAL $\pm .01$	COMMENTS:			
		INTERPRET GEOMETRIC TOLERANCING PER:	QTY: 2		TITLE:	
		MATERIAL			HANDLES	
		0.375" MILD STEEL			SIZE DWG. NO. REV	
NEXT ASSY	USED ON	FINISH			B KNNSHLD-013 NR	
APPLICATION		DO NOT SCALE DRAWING			SCALE: 1:1 WEIGHT: SHEET 1 OF 1	