

# Black Powder Manufacturing, Testing & Optimizing

Ian von Maltitz

*American Fireworks News*

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*AFN*

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To Yvonne, Debbie and Steven

#### WARNING TO READERS

This publication contains depictions and descriptions of Black Powder (aka gunpowder). The information is based on the experience of the author using specific tools and ingredients under specific conditions not necessarily described in the text. No warranties are made. Readers are cautioned that they must form their own opinion as to the application of anything found in this book.

#### NOTICE TO READERS

Concerning any offer found in this publication to sell or transfer products or information that are subject to governmental regulation, such sales or transfer of the product or information will be made in accord with Federal, State and local laws applicable to the buyer or transferee.

## Preface

It was probably during my meeting with Ken Kosanke in what I affectionately called his "secret mountain laboratory" that I decided to revise what I had written in *Black Powder Manufacture, Methods and Techniques*. When I told Ken that I had grown unhappy with what I had written, in that the material in it was dated and needed correction in parts, he encouraged me with the statement "That's what second editions are for".

Well Ken's laboratory isn't in the mountains. It's not a secret either. And this book is not a second edition of *Black Powder Manufacture, Methods and Techniques*. It is a completely new book, written from a somewhat different perspective. The previous book was written towards the end of the last millennium, and largely reflected the state of amateur pyrotechnic art of the time. This book was written at the beginning of this millennium, and reflects some marked changes in approaches to making Black Powder.

In *Black Powder Manufacture, Methods and Techniques* I included a lot of nostalgia stuff, dating back to early editions of AFN and beyond. Fond memories of times of pyro past are worth recording for posterity - and perhaps are best not mingled too much with the present. This is one reason why this is a new book and not just a rehash of what was written before. Another reason is that many experimenters in the field of pyrotechnics want to move beyond the old mindsets. These mindsets include the many variations on the so-called *CIA method*. They also include the stuck-in-a-rut discussions that cannot get beyond *Pyro Golf* and ball mills cloned to a popular amateur pyrotechnics design.

This book discusses *Pyro Golf* in detail, highlighting both its strengths and weaknesses. It also assures the reader that one can make good Black Powder using different kinds of ball mills, or perhaps even without a ball mill. Along the way this book dispels myths, kills sacred cows, and takes some rather irreverent swipes at the self-appointed amateur Black Powder theocracy. For all these *sins* its author is unrepentant and unapologetic.

Good Black Powder needs good charcoal, so I have dedicated three chapters of this book to making charcoal - good charcoal - the type best suited for making Black Powder. And one doesn't know if one's powder is good until one has tested it; three chapters are dedicated to testing.

This book would not have happened without the encouragement given to me by Ken Kosanke and Jack Drewes. I am especially thankful to Ken for his generous sharing of his material on testing. I am also indebted to Mick Fahringer and Don MacDonald of GOEX, who shared some valuable information and insights on commercial Black Powder manufacture.

Last, but not least, I owe thanks to Yvonne, Debra, and Steven for their sacrifices in giving me the time to write this book.

Ian von Maltitz  
Colorado Springs  
April 2003



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## *Chapter 1 - Safety First*

### **Introduction**

This is the first chapter and it was thought that safety considerations would be a good place to start. So it's **safety first**, safety being first and foremost in our discussions about Black Powder.

This chapter is a "must read" for anyone contemplating the methods and procedures outlined in this book. Even old hands in the Black Powder game should not skip over this part. Black Powder manufacture is dangerous - often more dangerous than the making of certain other more powerful explosives. And yes - Black Powder is an explosive. Here the term "explosive" is not merely a legal description. The stuff can go bang if the conditions are right. This bang can be very powerful and very destructive. Even without the bang, Black Powder can cause very destructive burning very quickly.

Since the first edition of the book, I've realized that there are some facets of safety that were not covered in it. Others were covered, but inadequately. I've tried to adequately address those issues here.

This chapter has been divided into a number of *safety tips*. These safety tips go far beyond the often found one-liners about safety. They are detailed discussions about safety considerations. And they are incomplete! They are not the last word on safety issues. They merely touch upon some general considerations and do not cover every possible circumstance and contingency. Here each individual should develop his or her own heightened awareness of safety.

Some of the terminology in this chapter will be unfamiliar to some of the readers. In placing this chapter before the others, it was unavoidable. The problem should not really impact the safety messages given here, however, and clarity will come by reading the later chapters.

### **Safety Tip # 1 - Small is Beautiful**

Small is beautiful from a safety perspective. Any accident with a small quantity of Black Powder or any other type of pyrotechnic/explosive mix will have less devastating consequences than an accident with larger quantities. This does not necessarily mean that the chances of an accident are reduced with smaller quantities - just that the negative impact is less. Let's look at some of the consequences of an accident involving a larger quantity of Black Powder.

- A larger explosion can result
- A larger fire can result
- The accident is more noticeable to others

A larger explosion and/or fire increases the likelihood and extent of injuries. Many fatal pyrotechnic accidents have been caused by large quantities of materials igniting unexpectedly. Sadly, death has come slowly in some of these where the accident victim has been horribly burned, dying a painful death from the resulting burns.

A high profile accident, even without injuries, can bring with it a multitude of problems. Such an event attracts the attention of neighbors, the police, the fire brigade....., and perhaps others one doesn't want to get the attention of. So small is beautiful but just how small? There are no hard and fast rules here. The basic yardstick to use is small enough to be practically feasible.

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I make my Black Powder in batches of 500 grams. I know of more conservative pyros who only make experimental 100 gram batches. Others make many pounds of Black Powder at a time. It just depends on what one needs the Black Powder for. My Black Powder has been both experimental and for use in small fireworks. Others have made Black Powder purely experimentally and some pyros do require large quantities to lift lots and lots of shells.

Now there is a tradeoff here when one needs to make larger quantities of Black Powder but wants to minimize the dangers found in making such quantities. One can split the required amount of material into several smaller batches but this creates another safety hazard - one is exposed to the same manufacturing hazards for longer periods of time. Thus, statistically speaking, splitting a batch into five separate smaller batches means that one has five times the chance of having an accident. But each accident will be smaller and more easily controllable. There is, however, a way to overcome these conflicting criteria.

One way is to split the manufacturing process into its hazardous and less hazardous components. This is described in tip # 2.

### **Safety Tip # 2 - Splitting the Difference**

Most pyrotechnic processes can be split into hazardous and less hazardous components. This is true for many of the varied processes used in making Black Powder.

When making larger batches of Black Powder it makes sense to make the less hazardous stuff in larger batches and then split these batches into smaller batches during the more hazardous phases. This system can be applied in one way or another to most of the methods used in making Black Powder. Here are some examples.

#### **CIA Method**

Mix the dry potassium nitrate, sulfur and charcoal in larger quantities and then split these quantities into smaller batches before boiling up with water and adding alcohol.

#### **Ball Milling Methods**

Always add the potassium nitrate last. Charcoal and sulfur provide minimal hazards when ground on their own or even together. Once potassium nitrate is added to the sulfur and charcoal the mix becomes dangerous, sometimes extremely dangerous.

The above is especially good advice when making Black Powder by grinding all three of its components in a ball mill. Here one can relatively safely grind large batches of charcoal and sulfur together. The grinding batches can then be reduced when the potassium nitrate is added. And here one does better by grinding the potassium nitrate on its own before mixing with the other components. Ball milling charcoal and sulfur together and then adding potassium nitrate in solution is another way to go.

### **Safety Tip # 3 - Grind Separately**

Some of this advice has already been discussed under Ball Milling Methods in tip # 2. Which ever method one uses for powdering the Black Powder components, it's always safer to grind the potassium nitrate separately. This applies to all types of grinding and milling operations. It is difficult to ignite sulfur or charcoal on their own or even mixed together. Add the potassium nitrate and you have an oxidizer mixed with fuels, creating a potential fire *hazard* or even a bomb. Here are three different *hazard* levels in grinding Black Powder components:

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## **Level 1 - Slightly Hazardous**

Grinding charcoal on its own is slightly hazardous in that a spark could ignite the charcoal dust mixed with air in the container. The same can be said for sulfur. Similar hazards could occur with charcoal and sulfur mixed. Potassium nitrate ground alone does not present any peculiar hazard. Overall the *hazard* level is relatively low here, especially if one takes care to avoid sparks caused by static electricity or other sources such as brushes on an electric motor.

## **Level 2 - Hazardous to Very Hazardous**

While grinding potassium nitrate on its own is relatively safe, mixing it with anything else while grinding can be hazardous. One does not really gain anything by grinding potassium nitrate together with sulfur or charcoal as there are other less hazardous methods whereby the potassium nitrate can be incorporated into the Black Powder mix.

Potassium nitrate is an oxidizer and thus will burn if mixed with a fuel and a source of ignition is applied. Both sulfur and charcoal can be regarded as fuel. Charcoal mixed with potassium nitrate is probably more hazardous than a potassium nitrate/sulfur mix. I haven't seen any definitive studies in this regard but do know that it's possible to make Black Powder with just potassium nitrate and charcoal, without the sulfur.

## **Level 3 - Very Hazardous to Extremely Hazardous**

Mixing potassium nitrate, sulfur and charcoal and grinding them together can very quickly shorten one's pyro career or even one's life. If you have to go this route be very, very careful. If you can, avoid this route entirely. If you can't, at least confine this activity to where it can do minimal damage should an explosion occur and preferably start and stop the mill by remote control.

Unhappily this method has become the method of choice for many, as it gives a quick and simple way of making Black Powder. Part of its attraction is that it does away with the somewhat messy and time-consuming methods involved in separate incorporation of the potassium nitrate.

## **Safety Tip # 4 - Watch the Wet Stuff**

Generally it's safer to manufacture Black Powder wet than dry. Wet reduces friction, reduces heat, reduces dust and generally makes a safer and better manufacturing process. Wet aids the incorporation process and (if done right) results in better and faster Black Powder. Wet is essential for making Black Powder granules. Wet can also be deceptively dangerous.

I learned the dangers of wet Black Powder the hard way.

One day, when cleaning up after making some Black Powder using a combination ball mill/CIA method, I noticed some wet Black Powder had spilled on the ground. I scooped this lot all together and decided I'd put a match to it rather than just throw it in the trash can. After all, it's more fun burning the stuff and Black Powder just lying in the trash could be dangerous. I thus lit a match and held it to the wet stuff. I expected the little pile of Black Powder to just fizzle and sputter - it didn't! Instead I got a loud whoosh which made me fall over backwards in fright, nursing some very sore fingers.

The Black Powder had burned at a frighteningly fast speed and very efficiently. There was very little residue to tell one that Black Powder had been burned on that spot. The realization came over me that wet Black Powder can be just as dangerous as the dry stuff. Over time, I came to

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the conclusion that wet Black Powder can be even more dangerous than dry. Why? Two basic reasons.

1. Wet gives one a very false sense of security.
2. A certain percentage of moisture actually aids the ignition process

This second reason may come as a surprise to many. I grew up believing that the dryer the powder, the more violent and dangerous it was. Not so! A small amount of moisture actually enables a flame to propagate at a higher speed. This is reputedly due to the hydrogen molecules found in water. Yes, there is a break-even point where the speed starts to decrease when more water is added. And too much moisture can result in the potassium nitrate leaching out of the Black Powder, making it weaker. But be warned, even with moisture beyond the break-even point, such powder can still be dangerous.

Another myth is that the effect of static electricity is nullified in a moist environment. Like Black Powder, static electricity actually seems to prefer a slightly moist environment to one which is completely dry.

So be careful when working with wet Black Powder. Remember too that some of the moisture might also be alcohol if one is using the CIA method or similar and that alcohol mixed with water can still ignite.

### **Safety Tip # 5 - Familiarity Breeds Contempt**

I once flew with an airplane pilot who was a staunch believer in flying safety and appeared to practice what he preached. A few months later I was shocked to read of his death in a flying accident. At the time there was quite a lot of speculation about his accident. It was not until my next flight with one of his fellow pilots that I learned the whole story about this sad affair.

He had been a very experienced pilot and had just qualified for a new commercial license before his accident. Apparently he had gone for a flip in a plane - just to celebrate. He then foolishly attempted a celebration barrel roll over the airport - and - forgot to strap himself in his safety harness. Under normal conditions a safety harness is not needed, the simple safety belt will do. But barrel rolls do not constitute normal conditions. His head was thrown against the instrument panel and the rest is history.

Here was an experienced, safety conscious pilot who made only one mistake - his last. He knew the air - he knew his aircraft - and he knew his safety precautions. But maybe he was just too familiar with all of these. Familiarity breeds contempt and is one of the major reasons why experienced people (who should know better) have accidents.

You may follow a certain procedure ninety-nine times without incident. The hundredth time something might go horribly wrong! Being overly familiar with certain things tends to make one lower one's guard and assume that nothing can go wrong. Procedures involved in the manufacture of explosives can be peculiarly prone to go wrong unexpectedly. This is because there are so many variables involved. These variables can change ever so subtly that one is unaware of them. The right combination of changes can be just enough to make something unexpected happen. Stay alert. Stay green. Stay alive.

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## **Safety Tip # 6 - Watch out for Sparks**

Black Powder can be more dangerous than dynamite, more dangerous than TNT, and even more dangerous than a nuclear warhead. How? It only takes one tiny little spark to set it off.

Sparks can come from a number of different sources, the most common in the Black Powder making environment being:

- static electricity
- friction
- electrical contacts

Static electricity can be found just about anywhere. Some areas are more prone to this problem than others. I live in Colorado Springs, which is renowned for its high levels of static. It was this factor which attracted the renowned inventor, Nikolai Tesla to Colorado Springs where he built his famous artificial lightning laboratory. Static is a problem throughout the year here and I usually experience a slight shock when I touch the door handle of my car when getting out.

Coastal areas, with their higher humidity, are generally less prone to static but one cannot ignore the problem there. And, as mentioned elsewhere, moisture can aid the propagation of static electricity. If you doubt that, just think about where the largest known buildups of static electricity occur. Where? In the clouds! Lightning results from a buildup of static charges between the earth and clouds or between the clouds themselves.

Static is a stealthy killer, being the cause of many unexpected and lethal explosions. Recent speculation suggests that certain aircraft disasters can be attributed to static buildup in fuel tanks.

A whole volume could be written on protection against the negative effects of static electricity. So space does not permit us to justly treat the subject here, only to offer a few pointers. Here are some:

- Wear cotton clothing (including underclothing).
- Eliminate static producing materials, such as certain types of carpeting, in your working environment.
- Preferably ground your working surface and yourself.
- Be careful when pouring or shaking materials

Friction causes fire - as the saying goes. Early man learned to make fire by rubbing two sticks together. Today we use matches and lighters, also requiring friction to ignite. Friction can also cause Black Powder to ignite, either through the slow buildup of heat or the sudden production of sparks. This section focuses on the latter.

Sparks can be created by the sudden violent contact of two objects with each other. Examples are:

- steel against steel
- steel against concrete
- stone (such as quartz) against stone

This is the reason why it is best to use copper or brass screens rather than steel and the reason not to use a pestle and mortar made from iron.

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Sparks occur whenever an electrical circuit is made or broken by mechanical type contactors. An example is a simple light switch. Several sources of such sparks are possible with an electrically powered device such as a motor-driven ball mill. Here, the general precaution to take is to keep the Black Powder as far as possible from such electrical sparks. Some practical methods are:

- Use a *brushless* type of motor.
- Mount the on/off switch as far away as possible.
- Ensure that all electrical connections are tight and cannot come loose during operation.

### **Safety Tip # 7 - No Metal, No Metal, No Metal !**

This tip repeats a bit of what has just been said about sparks.

While I was writing the first edition of the book, I worked with someone who had lost his hand during his teenage years. He had commented to some that he had *learned to make gunpowder at an early age*. I later found out that his exploits had expanded to making pipe bombs - and the rest is history.

Making pipe bombs is highly illegal and also highly dangerous. Tragically many have been killed or injured through their own homemade pipe bombs. Tragically, because a high percentage of these have been just kids experimenting, and not planning any acts of terrorism.

Metal containers, including pipes, are an absolute no-no in pyrotechnics. Besides the spark hazard, metal is a good conductor of heat. Thus a rocket, driver or fountain made from a metal tube may suddenly and unexpectedly explode. This is due to heat transferred by the metal to the confined section of the pyrotechnic mix.

### **Safety Tip # 8 - Dress for Success**

Safety Tip # 6 touched on the subject of clothing. This focused on wearing cotton to avoid static. There are other reasons to look at clothing. The right clothing offers protection, should an accident occur.

Cotton has another useful property. It doesn't melt. Unlike synthetic materials, cotton tends to smolder and finally burn when heated. Synthetic materials melt, and molten clothing can often produce greater burn injuries than burning cotton.

Now this discussion can get sidetracked, just like the debate about car seat belts. While many will consider it safer to travel buckled up, there are diehards that consider seat belts more dangerous than they are worth. So some might subscribe to the belief that any kind of clothing worn while making Black Powder is too much of a hazard. Well, that's their choice!

There are all kinds of fire-resistant clothing available that will neither melt nor burn. But these can work out being rather expensive. A viable option is to get a welder's leather apron. Welder's leather gloves are also useful.

Probably the most important form of protection is eye protection. Goggles that are resistant to impact are a good way to go. Another good option is a shield that protects one's entire face. Using both is even better still.

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## **Safety Tip # 9 - Don't Go it Alone**

This tip clashes with some old, well-entrenched traditions. But, in the interests of safety, it is wise to question these cherished practices.

I'm referring, among other things, to the practice of waiting until the little wife is out of the house before embarking on one's latest pyrotechnic exploits. It used to be a grand tradition to hijack the kitchen to cook up a Black Powder brew on the stove. Naturally the wife had to be out of the house for one to do this in *her* kitchen.

Working alone is great. It helps one focus on what one is doing, without distractions. It can also be highly dangerous.

Mountain climbers are cautioned never to climb alone. Scuba divers believe one should not dive alone. But many pyrotechnic enthusiasts work alone, and prefer to do so. And in some ways this is understandable. Pyrotechnics may be a sociable event during fireworks displays, but puttering about in the shed outside is usually not.

Working alone is not a problem — until an accident occurs.

In the unhappy event of an accident, one often needs some help from another person. This is especially true if one is incapacitated in some way. The other person may be able to help you directly or call others for help. Either way, this is preferable to trying to help one's self or trying to call others for help.

Going it alone, and thinking that just a call to 911 will resolve the issue, is not the way to go. One needs to pop a few bubbles here. In the world of make-believe, fueled by Hollywood, a call to 911 brings help within minutes. The emergency responders, from the person who takes the call, to the persons arriving in the ambulance, all know exactly what to do, and do it in the shortest possible time. The harsh realities are quite different!

Accidents involving pyrotechnics or explosives are not the day-to-day type of emergency handled by emergency responders. Any hint that the accident is caused by such activities could result in the whole matter being sidetracked by all the possible overreactions to perceived threats from explosives. So the whole neighborhood might be evacuated and the bomb squad called. No one will enter your premises until they have been *secured* and *swept* for any traces of additional *explosives*.

In the real world not all emergency responders are selfless dedicated individuals like those who were killed and injured while rescuing others at the World Trade Center in New York. Sadly, some are more like those who responded to the massacre at Columbine High School in Colorado. An injured person could bleed to death or die from shock while such engage in other priorities. Sad but true.

## **Safety Tip # 10 - Speed Kills**

Fast burning Black Powder can be dangerous - highly dangerous. It can, under certain circumstances, be lethal. That's not to say that slower burning powder is not dangerous, it's just less dangerous than the faster burning stuff.

This book has a lot to say about how to make fast powder, Black Powder which burns at speeds approaching those found in commercially produced powders. It certainly is an achievement to manufacture powder which burns this fast but such speed might not be necessary or

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desirable. Faster powder ignites more easily and burns more violently; and is thus more dangerous to work with. So if one can do the job with slower powder, this is the way to go.

One problem with slower powder - one usually requires more of it. This is a tradeoff against the safety problems with faster powder which generally requires less.

### **Safety Tip # 11 - Beware of the Great Green Guru**

Yes they are everywhere. They make their presence known at club meetings. They love disseminating their ideas in seminars and in the pages of magazines. And they prowl the Internet, being frequent commentators in chat groups and similar forums. These are the *Great Green Gurus*.

Every endeavor and discipline has its experts or so-called experts. Pyrotechnics is no exception. And it does have its real experts; those who treat pyrotechnics as a discipline, to be properly mastered by the prudent, industrious and wise. And there are the sloppy, careless, indolent loud mouths. They have *been there, done that* in just about everything, and so many times that they lost count. And they are quick on the draw in passing off their own pearls of wisdom. These are the *Great Green Gurus*. Avoid them as if your life depended on it, because in the field of pyrotechnics, including Black Powder manufacture, it probably will!

So just how does one spot them? Well they are often quick to give themselves away. They usually draw on no wisdom but their own. And where they do refer to the wisdom and knowledge of others, they are prone to contradict it. They are *the* authority and might only grudgingly refer to the authority of others when it agrees with their own opinions.

Where it comes to safety, the *Great Green Gurus* can be a real problem. They are quick to lecture the unwashed masses on safety issues while privately ignoring their own advice. And they love to go against the established wisdom on safety. Some will boast about ignoring the well-established wisdom about not mixing chlorates and sulfur or certain other dangerous combinations. After all, they've *done it for years, no problem!* And they've also *made tons of Black Powder*, using methods normally considered to be highly dangerous.

So beware the *Great Green Gurus*. They live charmed lives and often those who get hurt are not themselves but the gullible who follow them.

### **Safety Tip # 12 - Think About the Fallout**

No method used in Black powder manufacture is completely safe but some methods are safer than others. Sometimes one has to choose between different methods and here safety should be the major consideration. And here one should look at the larger picture.

For example, there exists some debate as to which method is safer when the choice is between:

- ball milling all three components together with the danger of the mill exploding
- using the CIA method to incorporate the potassium nitrate but with danger of ignition from a hot stove.

Each method has its own peculiar dangers and, while I personally opt for the second method, there is the bigger picture to consider from safety and other standpoints.

An exploding ball mill can pose a greater danger to life and limb than a pot of wet Black Powder igniting. But a pot of burning powder can cause a fire which can burn down a house. So where

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do we go from here in our choice of the better method? Here one needs to consider practical steps in containing the accident, should it occur. Stated another way: one aims at minimizing the potential fallout.

An exploding ball mill in a suburban neighborhood poses a far greater threat than one in the remote corner of a ranch far away from man and beast. Even if no one is hurt and physical damage is limited in the suburban blast, such an event cannot go unnoticed. And noticed it will be; by the neighbors, the cops, the district attorney.... Yes, a fire will also be noticed but does not create as much excitement as an alleged *terrorist bomb making factory*.

But one can contain the fallout from an exploding ball mill if the milling takes place far away from where it can damage anything and too far away to be noticed.

One can take steps to contain any fire caused by a pot of burning Black Powder by having adequate fire fighting equipment so as to extinguish the flames almost immediately. Better still: heat the mix outside or in a garden shed far away from any living areas.

One needs to look at the bigger picture: what will be the fallout if an accident does happen?

### **Safety Tip # 13 - Tread Carefully in New Territory**

This book covers a lot of new territory not covered in the first edition. Some of this territory is uncharted and could be highly dangerous. This applies particularly to untried and untested milling methods.

I don't generally recommend milling all the Black Powder components together at the same time. While some may treat this as an acceptable risk in ball mills, it can be highly dangerous in other types of mills. Some mills pound and grind their materials in a peculiarly violent manner. I would say it is a sure bet that some of these methods would ignite the Black Powder.

Some of the other new territory covered relates to charcoal making and testing. Both of these have their own unique hazards.

### **Safety Tip #14 - Be Careful when Testing**

This follows on from the previous tip.

Most Black Powder testing is hazardous in one form or another. This is because most testing involves igniting the Black Powder. The ignited Black Powder then either burns at a measurable rate, or explodes. Either scenario could be dangerous if the unexpected happens. In testing, the unexpected often does happen.

Much of the testing described in this edition focuses on Black Powder that is exploded in a confined space, as in eprouvettes, closed bombs, quickness testers and golf ball mortars. All of these devices are dangerous and should be treated with respect. Specific hazards relating to each are described in the chapters on testing.

### **Safety Tip # 15 - *Perfectly Safe* is an Oxymoron**

Some years ago I was working at a gold mine. Gold mines are places where typically tons of explosives are used annually. Very strict procedures are normally adopted when such explosives are used. As a result, accidents from explosives are rare. But on one particular occasion certain procedures were compromised.

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The incident in question involved only a small amount of explosives. The application required only the detonation of booster explosives, not the large quantities normally involved in breaking up thousands of tons of rock. When I first questioned the use of these explosives and some of the procedures involved, I was told it was *perfectly safe*. The rest of the story is irrelevant to these pages, but that short phrase *perfectly safe* is.

When it comes to Black Powder there is nothing that is *perfectly safe*. The term *perfectly safe* is an oxymoron, a contradictory statement. However, it is surprising the number of people who subscribe to the *perfectly safe* myth. While researching this book I came across a patented process developed by the famous Black Powder manufacturer, du Pont. This describes a milling process that uses a type of rod mill. The powder in the mill is dampened with guess what - gasoline! And the patent describes this process as - *perfectly safe*.

Perfectly safe? Don't let this phrase become your famous last words.

### **Safety Tip # 16 - Safer is Not the Right Word**

This tip follows the line of thinking of the last one. Using the word *safer* is a bad choice. A better choice is *less dangerous*. One should not let one's self get out of the mindset that all manufacture of Black Powder is dangerous. Some methods may be less dangerous than others, but should never be considered as safe.

### **Safety Tip # 17 - Be Prepared**

The good old Boy Scout motto says be prepared. Common sense says one should be prepared in case an accident happens.

A simple safety preparation is having a good supply of water on hand in case of accidental ignition. Water is the best stuff to use, as it both smothers the flames and cools whatever is burning. Some fire extinguishers may be bad choices as they merely smother the flames. Pyrotechnic compositions supply their own oxygen so mere smothering does not stop them burning.

Readily available cold water is also very useful in treating one's self if one gets burned. Having a first aid kit close at hand is also very sensible.

### **Safety Tip # 18 - Walk the Walk**

It seems that in all walks of life there are those who do not practice what they preach. Some preach safety but do not practice safety. Some of the staunchest preachers of safety flagrantly ignore the most basic safety precautions. Why?

Well, one can delve deeply into psychology and the study of human nature to describe this phenomenon. I think a simple explanation is that a subtle form of psychological inoculation takes place in some who fervently preach safety to others. One can talk so much about safety that one can be deceived into believing that one is practicing it when one isn't. And ignored safety precautions can be very unforgiving. They also do not take into account one's preaching as evidence in mitigation.

So talk the talk by all means — but more importantly — walk the walk.

## *Chapter 2 - The Right Stuff*

### **Introduction**

This chapter discusses the materials used in Black Powder manufacture.

Like any other pyrotechnic endeavor, the choice of materials for Black Powder manufacture can spell the difference between success and failure. And while I am a firm believer in the statement just made, I have attempted to steer a path between certain extremist views on this subject. There are those who insist that only one particular type of material will work and those who insist that one can make good Black Powder with just about anything. Both extremes are right under certain circumstances and very wrong under others. Here I will attempt to clarify which viewpoint suits a particular application.

My own many and varied attempts at Black Powder manufacture have clearly shown me one important truth: it is worthwhile experimenting with different materials. In my early schoolday Black Powder experiments I found that barbecue charcoal would work just fine and that there was no discernible difference between sulfur and flowers of sulfur. In later years I refined my choice of materials to low acidity sulfur and fast burning charcoal. I learned a lot along the way and the journey was certainly worth it.

I have, in the course of time, been the proud owner of many and varied grades of potassium nitrate. Most of these have ended up in Black Powder in one form or another. My potassium nitrate stock has ranged from analytical reagent grade to industrial grade. No, I never did cross the line to fertilizer grade, my reason being that I could get a good industrial grade at a cheaper price. Analytical reagent grade? Well I never got around to actually using it as I was keeping it for that special application which would require very high purity materials. Very high purity Black Powder was not one of my goals.

This chapter focuses on the three main ingredients of Black Powder: potassium nitrate, sulfur and charcoal. I also thought it would be worthwhile discussing other materials used in the making but not forming part of the final product. These are water and alcohol. There are also certain substitute materials used in the manufacture of Black Powder. Some of these materials can be used effectively in certain circumstances. Others should be avoided as they are either ineffective or downright dangerous!

### **Potassium Nitrate - In General**

Potassium nitrate is the chemical name for a substance commonly known as saltpeter. Its chemical formula is  $\text{KNO}_3$ . It has many uses in industry, agriculture and the home. Thus it is supplied in many different forms and purity specifications. Potassium nitrate has a close cousin, sodium nitrate, also known as Chile saltpeter.

If one follows the history of Black Powder over the centuries, it is clear that many different grades of potassium nitrate have been used successfully. By today's standards, the methods used to obtain and purify this substance were very primitive and crude. This resulted in many tons of potassium nitrate of very dubious purity being produced. Well this stuff worked - in cannons, rockets, and even rifles. And - this kind of stuff might even work today, where needed.

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Yes, you can emulate the methods of our forebears in producing potassium nitrate. There have been several survivalist types of publications describing such methods in great detail. Some have even posted information on the Internet, offering such advice to those finding difficulty in obtaining potassium nitrate. Most of these methods are not for the squeamish and usually involve either urine or excrement from either humans or animals. So you can make your own potassium nitrate from urine or dead cats if you really want to. The real question is: how good is this stuff in pyrotechnics?

For general pyrotechnic use it is best to aim for potassium nitrate of high purity. Usually one can get away with lower purity potassium nitrate in Black Powder manufacture. So for Black Powder use one should not put too much store on some of the pyrotechnic literature which uses terms such as *double refined* and purity quoted in the region of 99.8%. Yes, there is some merit in the age-old saying: "the purer the saltpeter, the better the powder" but some have really gone overboard here.

Perhaps this high purity mindset comes from some of the commercial manufacturers of Black Powder. Here, some will boast of producing better Black Powder because their potassium nitrate is very pure, in the order of 99.9% pure. There is some merit in their claims when one considers that small impurities such as chlorides may increase the powder's tendency to attract moisture. This is an issue if the powder is going to be stored for a long period of time. Otherwise there is no real merit in going to extremes in the purity of the potassium nitrate.

There has been much written about using fertilizer grade potassium nitrate, its main virtue being its cheap price. And it does come cheap, when and where you can get it. It does not seem to be freely available everywhere, so stories of its abundant supply probably come mainly from farming areas. Those who have tried fertilizer grade generally recommend it for Black Powder manufacture. And this recommendation usually says that it is fine to use the fertilizer grade "as is" - without any attempt at further purification. Yes, there are some purists and fanatics out there who feel that one should go to great lengths to purify the material further.

A good technical grade (a.k.a. industrial grade) is usually a good bet. This is in fact what one probably gets anyway from the re-packagers of pyrotechnic materials.

Besides purity, some have emphasized the need for finely ground or pulverized potassium nitrate, preferably with some type of anti-caking agent added. This may be advantageous in certain manufacturing procedures, but is not really needed where the potassium nitrate is dissolved in water. Here the only plus is the potassium nitrate dissolves more quickly.

This discussion has been about potassium nitrate in general terms. It has mentioned different grades that are suitable or possibly suitable for Black Powder use. It is worthwhile knowing more about the different grades available.

### **Potassium Nitrate Grades - In General**

Potassium nitrate comes in different grades. All of them will work in Black Powder. Some will work better than others. All of them cost money. Some of them cost more than others — some much, much more.

What follows are brief descriptions of different grades of potassium nitrate. I was tempted to limit this discussion to just technical and fertilizer grades, but decided to describe the others because some of my readers somewhere are bound to have access to some of these other grades. And like my own experience in purchasing analytical reagent grade, they might be offered some potassium nitrate of a different grade at a price that is difficult to refuse.

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## **Potassium Nitrate - Analytical Reagent Grade**

Analytical Reagent grade (a.k.a. AR grade) is generally considered to be top of the line in terms of purity. This grade is used for laboratory work that requires very accurate results, with minimal interference from chemical impurities.

Analytical Reagent grade is most probably wasted in Black Powder applications. It has purity levels that are unlikely to make any difference in performance. Analytical Reagent grade is prohibitively expensive. If one is given some as a gift or manages to buy some cheaply (like I did), it is best to keep it for other applications.

## **Potassium Nitrate - Laboratory Grade**

Laboratory Grade (a.k.a. Lab Grade or Chemically Pure) is similar to Analytical Reagent Grade but with less exacting purity specifications. It is cheaper than Analytical Reagent grade but costs a lot more than technical or fertilizer grades.

Laboratory Grade may find an odd niche in some laboratory experimentation with Black Powder, but offers no advantages beyond that.

## **Potassium Nitrate - Pharmacy Grade**

Pharmacy grade is not called by this name but rather by the more exotic terms such as USP or BP. USP stands for United States Pharmacopoeia and BP for British Pharmacopoeia. Thus USP grade is sold by pharmacies in the USA, and BP grade in Britain and countries that fell under British influence.

Many aspiring Black Powder makers started out in their quest for powder with stuff got from their local pharmacy. This powder was most probably USP, BP or some equivalent grade.

Pharmacy grades are good for Black Powder making but very expensive. And buying a whole lot of the stuff from a pharmacy may solicit unwanted attention because people who buy potassium nitrate for other purposes generally only buy a little.

## **Potassium Nitrate - Food Grade**

Food grade is a relatively pure grade that is used in food. Its impurity specifications appear to be narrowed to poisonous substances, rather than edible impurities such as salt (sodium chloride). The data in the following table illustrates this:

CRChem Food Grade Potassium Nitrate	
Assay (amount of potassium nitrate)	>99.0%
Loss on drying	<1.0%
Chlorate	Passes test
Lead	<10ppm
Heavy metals	<0.002%

Such a food grade could be used to make Black Powder but may have some impurities that one is not told about, such as salt. For this reason it is possible that a particular food grade is no better than a good technical or agricultural grade.

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## Potassium Nitrate - Technical Grade

Technical Grade is a rather generic term that describes potassium nitrate used in industrial and commercial applications. It is also referred to as *Industrial Grade* or *Commercial Grade*.

Technical Grade is the type that one gets from pyrotechnic suppliers. It could be a very good grade or a relatively poor grade, depending on its specifications. As an illustration, here are some specifications from one manufacturer:

CRChem Technical Grade Potassium Nitrate	
Assay (amount of potassium nitrate)	>98.5%
Insoluble matter	<0.1%
pH (5% solution)	4.5-8.5
Chloride	<0.1%
Sulfate	<0.5%
Sodium	<0.2%
Calcium and Magnesium	<0.1%
Iron	<5ppm
Heavy metals	<10ppm

Now compare the above to the specifications from another manufacturer:

Azot Association Commercial Grade Potassium Nitrate	
Assay (amount of potassium nitrate)	>99.9%
Insoluble matter	<0.01%
Water content	<0.08%
Chlorine salts as NaCl	<0.017%
Carbonates as K <sub>2</sub> CO <sub>3</sub>	<0.01%
Potassium nitrite	<0.01%
Calcium and Magnesium	<0.002%
Iron	<0.005%

The above specification is quite unique, but illustrates the fact that not all technical grades are created equal.

Some manufacturers differentiate between industrial and technical grades as shown in the following grades supplied by SQM.

Grade	KNO <sub>3</sub> Assay
Industrial	96.0%
Refined	99.8%
Technical	99.3%

Other manufacturers offer different types of industrial grades. Such a supplier is Potachem Industrial Co. that offers the following industrial grades:

Industrial Grades	KNO <sub>3</sub> Assay
Fine	99.8%
First	99.4%
Qualified	99.0%

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Now one can see that not all technical/industrial/commercial grades are created equal. Personally, I would hesitate to buy any such grade that was less than 99% pure.

### **Potassium Nitrate - Fertilizer Grade**

I admit to having quite a lot of skepticism about Fertilizer Grade (a.k.a. Agricultural Grade) potassium nitrate. I presumed that it was inherently inferior to other grades, including technical grades.

As a general observation, fertilizer grades are less pure than other grades. However, a good fertilizer grade could be purer than a lower spec technical grade, so it is worthwhile comparing specifications before jumping to conclusions.

Fertilizer grades typically have a purity (KNO<sub>3</sub> assay value) of 98 - 99%. This is pretty good if one thinks about it. A popular brand, KPOWER boasts a purity of 98.8%.

Specifications for fertilizer grades can be a problem. Some of these are quoted in *fertilizer* rather than in *chemical analysis* terms. These specs are rather meaningless when one wants to ascertain purity. Some suppliers give a purity value for the potassium nitrate (which is very useful) but then do not properly identify levels of impurities.

Different types of fertilizer grades are manufactured for different applications. Some are made specifically for certain types of agricultural machinery used to disperse fertilizer. The manufacturing process used for this type of fertilizer can have a negative effect on its use in Black Powder. This is due to certain substances such as waxes being added to assist dispersion and prevent clogging. One should thus avoid using such grades.

The fertilizer grade most recommended by Black Powder makers is known as *Greenhouse Grade*, and is often simply referred to as GG.

### **Sulfur**

Just about any type of sulfur has been found to work in Black Powder. Some pyros have gone to great lengths to ensure that they get the "right stuff. Here the right stuff is perceived to be *sulfur flour*. Again, for general pyrotechnic use, sulfur flour may be regarded as being better. Black Powder, however, seems to have less exacting requirements.

Good powder has been made with agricultural grades of sulfur of suspect purity. And yes, you can use flowers of sulfur. This is the stuff which is considered by some to be an absolute no-no. Here they are right when it comes to mixes which contain chlorates. Black Powder, however, does not pose the dangers found in mixing acidic sulfur with chlorates, unless the maker is mad enough to substitute potassium chlorate for potassium nitrate. A word to the wise: some of those who have tried such substitutions have literally died in the attempt!

A possible drawback with flowers of sulfur is its property of attracting moisture. This can be a disadvantage, particularly if one wants to store the powder for a long length of time.

Most sulfur is supplied in fine powder form and is thus ready for initial incorporation with the other Black Powder ingredients. It is, however, wise to sift out any lumps before mixing. Further milling of the sulfur or sulfur with the other ingredients is usually required for good Black Powder.

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## **Charcoal**

The role of charcoal in Black Powder should not be underestimated.

The choice of charcoal can either make or break the performance of Black Powder. For this reason, charcoal is treated in this book as a subject in its self.

Anyone aspiring to make good Black Powder must give careful consideration to getting charcoal that will perform well. Ordinary briquette charcoal is a particularly bad choice for making Black Powder. Other types of barbecue charcoal may work better than briquettes but are far from the best. So the time, money and effort spent in obtaining good charcoal for making Black Powder is usually well worth it.

## **Water**

Some purists believe that there is merit in using distilled or de-ionized water in Black Powder. There is no proven merit in this. Water of normal purity for household consumption should be just fine. Remember too that Black Powder has been made for centuries using water with purity standards far below what is accepted today.

If one is making sample batches of Black Powder for serious laboratory type testing, using distilled or de-ionized water may be advantageous. Note that there is no proven merit in this. But just for peace of mind that the water is not somehow introducing another unwanted variable, it may be useful to use such high-purity water. It may also help to deflect some criticism of one's testing methods from some of those pedantic pseudo scientists out there.

## **Alcohol**

Alcohol is used both to aid granulation and in the famed CIA method of Black Powder manufacture. It is not used to aid ignition in any way.

Many have opted to use isopropyl alcohol, also known as rubbing alcohol. Isopropyl alcohol has become a favorite for two reasons - it is cheap and easy to obtain (in the USA). Other types of alcohol can be used just as effectively. Black Powder has been made with the two other common types of alcohol: ethyl alcohol (ethanol) and methyl alcohol (methanol).

Ethyl alcohol is the alcohol found in alcoholic beverages. It is good for making Black Powder but very expensive. One can use high alcohol content liquors such as whiskey and vodka to make a somewhat expensive Black Powder. A no less expensive powder can be made from pure ethyl alcohol. In most countries pure ethyl alcohol is expensive — often deliberately so.

Methyl alcohol is similar to ethyl alcohol but far more poisonous. Some rate it better than ethyl alcohol in making Black Powder. It is more difficult to obtain and in some places the purchase of methyl alcohol is restricted by law.

A nearly pure form of ethyl alcohol is what is known as denatured alcohol. Typically this contains 90 - 95% ethyl alcohol. The remaining 5 - 10% contains substances such as methyl alcohol and other ingredients used to give it a foul taste. It may also be colored (usually a violet color) to differentiate it from other liquids and solvents. It can be obtained far more cheaply than ethyl alcohol, depending where and how one buys it. Very expensive options are to buy it from a pharmacy or as fuel for fondue cooking. Far cheaper is to get it as a solvent, usually from a hardware store. Denatured alcohol is known as methylated spirits in some countries.

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## **Substitute Materials**

**Sodium Nitrate** has found its way into Black Powder making. Its use, however, appears to be limited to blasting powder applications. It is not used in making Black Powder used in fireworks.

A very important reason for avoiding sodium nitrate is that it is hygroscopic, i.e. it absorbs moisture. How then does it find practical application in blasting? Typically sodium nitrate blasting powder is supplied in sealed containers which exclude moisture. In theory, one could possibly do something similar in making certain types of fireworks. In practice, it's just not worth the extra trouble.

Does sodium nitrate have any value to the Black Powder experimenter? Well maybe. It can be used to make potassium nitrate. It can also be used to lower the ignition temperature of Black Powder, especially if it is mixed with potassium nitrate.

**Potassium Chlorate** has been tried in Black Powder ever since its discovery in the 18<sup>th</sup> century. The first person who tried this on a large scale was killed by the resulting explosion, along with several others. Yes, it is tempting to try potassium chlorate to create a more powerful Black Powder. This temptation should be avoided. Here there are just too many things that could go wrong, with tragic results.

**Potassium Perchlorate** is used in some substitute sporting powders used in Black Powder firearms. It is also used in some burst charges used in fireworks shells.

**Ascorbic Acid** (commonly known as vitamin C) is used in some substitute powders.

**Phenolphthalein** has been used as a charcoal substitute. Its use hasn't gone much beyond the experimental stage. This is possibly because phenolphthalein is very expensive when compared to charcoal. Phenolphthalein is commonly used to indicate if a chemical solution is alkaline. It also makes a very effective laxative.

**Iron Oxide** has been used in some the more unusual Black Powder formulas. These have been claimed to work but tend to absorb moisture.

**Sugar** has been used by some as a substitute for charcoal. It cannot be regarded as good as charcoal unless it is converted into charcoal first. And charcoal made from sugar does not rate as highly as certain other charcoals.

A good propellant for rockets can, however, be made by melting together a mixture of potassium nitrate and sugar.

Generally the sugar used in Black Powder formulations is ordinary household sugar. Some experimentation has been done with other types of sugars such as lactose (milk sugar).

**Picric acid and picrates** have been used in a few formulations. Picric acid is closely related to TNT and has about the same explosive power. Any mixture which includes picric acid or compounds made with it (picrates) is highly dangerous and should be avoided.

**Urine** was regarded as an essential ingredient in the past where superstition, folklore and bad science abounded. It was actually believed that urine from a wine maker was the best. While it may not harm the Black Powder to damp it with urine, it is not likely to help it either. It might also make the Black Powder and its maker less socially acceptable.

## *Chapter 3 - Entry Level Black Powder*

### **Introduction**

Here we discuss simple ways to make Black Powder. These are ways that compared to modern manufacturing methods are simple and crude, but work nevertheless.

Black Powder has been around for centuries. Just how many centuries is still the subject of debate. Even from the times of its crude beginnings, Black Powder has been made in forms that are effective enough to cause explosions and to propel projectiles. The early methods of manufacture were similar to those described here.

The earliest Black Powder (a.k.a. gunpowder) was made by hand using simple tools. The simple hand methods then evolved into crude machines that became the forerunners of today's manufacturing processes. Along with this evolution in manufacture evolved the perception that *true Black Powder* could only be made by using such manufacturing methods.

This perception is of course a load of nonsense. Yes, it is true that *better* Black Powder can be made using modern methods, but this does not mean that it is the only *true* powder.

Since the first edition of this book was published, a different perception has emerged. This has come about with the increasing number of aspiring Black Powder makers acquiring high efficiency ball mills. This has resulted in a large number of claims of making Black Powder comparable to its commercially manufactured counterparts. So to some at least, a ball mill is essential for making Black Powder. The new perception is that Black Powder made without a ball mill isn't the *real thing*.

So what exactly is Black Powder? And is there a *true* Black Powder?

Black Powder, or gunpowder if one prefers the older terminology, is simply a mechanical mixture of three ingredients: potassium nitrate (a.k.a. saltpeter), sulfur, and charcoal. For the purposes of this discussion, we won't allow ourselves to be sidetracked by variations in the ingredients such as sodium nitrate or powders that don't use sulfur. So Black Powder is a mixture of three ingredients that burn when ignited. This burning can occur in a relatively slow manner or incredibly fast, giving explosive power. Most applications of Black Powder require the latter.

To burn at a fast rate, the ingredients of Black Powder need to be intimately mixed together in the right ratios. This intimate mixing requires one to first reduce the ingredients to very fine powders and then to mix them together thoroughly. This reduction/mixing process is often referred to as *incorporation*. Over the centuries, Black Powder makers have progressively improved their methods of achieving good incorporation. This has resulted in powders that burn much more efficiently than their predecessors.

The whole controversy about *true* Black Powder thus revolves around the efficiencies (or lack thereof) in the incorporation process. Makers of commercial Black Powder have created their own naming system for different degrees of incorporation such as: *green powder*, *pulverin*, or *polverone*. Even these terms may mean different things to different persons.

So what is *true* Black Powder? I don't believe there is an answer that will satisfy everybody. Perhaps another way of asking the question is: "What is *quality* Black Powder?"

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One way of defining quality is: conformance *to* requirements. So if the Black Powder in question is used to lift shells then *quality* Black Powder can be regarded as Black Powder that does the job in a way that approaches normal acceptable performance. The same can be said for Black Powder used in rockets, fuses, drivers, mines, or in firearms. All of these have different performance criteria.

A basic assumption made among many pyrotechnic enthusiasts is that when one uses the term *Black Powder*; one is referring to powder used to propel aerial shells. This powder is commonly called *lift*. This assumption is largely correct, but there are notable exceptions. Black Powder used in drivers can be much made lower in strength than lift. The same can be said for some Black Powder mixes used in rockets.

This chapter starts with simple methods used to make Black Powder. The resulting powders will probably not be anywhere nearly strong enough to lift shells or even propel rockets. But this is just the entry level stage. Later chapters describe ways and means to make fast and super-fast Black Powder.

## **Basic Entry Level Requirements**

At the very basic entry level, one requires the three components for Black Powder:

1. potassium nitrate
2. sulfur
3. charcoal.

A nice-to-have is some kind of scale that can accurately weigh these components, preferably in small quantities. This is nice to have, but not essential at this stage. If a scale is not available, one can measure quantities by volume. This is a crude way of doing things, but we are in crude mode at the moment.

Potassium nitrate normally comes in granulated form and looks similar to salt or sugar. Sulfur is usually in powder form. Charcoal, unless it is supplied as *airfloat* charcoal, needs to be reduced to a powder.

So our starting point at entry level Black Powder is getting the above ingredients together. The next step is to get a container to mix them in and something to mix them with. A china bowl from the kitchen plus a wooden spoon should do the trick.

Black Powder made with this setup is not likely to present much of a danger to its maker, but nevertheless some basic safety precautions need to be taken. The first step is to wear eye protection in the form of goggles or a full face shield. The second step is to make the stuff in an area where should an accidental ignition occur, damages will be minimal.

## **The Next Step**

The next step is to measure out the ingredients in their required ratios. If a scale is available these ratios are (by weight):

potassium nitrate	15 parts
charcoal	3 parts
sulfur	2 parts

The above ratios are the most often used in Black Powder making. The above formula is known

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as the Waltham Abbey formula. Waltham Abbey was the location of the Royal Gunpowder Works in Britain.

If a scale is not available, then the above ratios can be arrived at approximately by volume as follows:

potassium nitrate		3 parts
charcoal		2 parts
sulfur	1/2	part

This last method is less accurate and should only be used if a suitable scale cannot be found.

It has been assumed up until now that the charcoal is in powder form. If it is not, a crude way of crushing it is to put it in a bucket and drop a four-pound hammer on it until most of it is reduced to powder and small pieces. The charcoal is then sieved with a sieve, the finer the sieve mesh the better.

Another way is to wrap the charcoal in several supermarket plastic bags and the hammer it with a hammer until it is reduced to powder and small pieces.

A third way is to use some very fine sandpaper. This method is tedious but worked for me in the past. I used wet-and-dry sandpaper with grit sizes ranging between 300 and 400. I did the sanding of individual pieces of charcoal by hand. At that time I did not have a power tool sander. These in theory would sand faster, but the big problem is how to retrieve the charcoal dust.

The charcoal and sulfur are now mixed together in the bowl with the wooden spoon. One should aim to mix these together as intimately as possible. This ideal holds true for any method of Black Powder manufacture. Fortunately sulfur and charcoal have very distinct colors that enable one to visually ascertain if they are badly mixed. The mix should thus not have any spots or streaks of yellow among the black.

The potassium nitrate is added once the sulfur and charcoal are properly mixed. It is then thoroughly mixed with the other ingredients. Once this is done, the powder is ready for use.

The Black Powder just described is about the most crude and primitive one can get, with matching performance. Such powder will burn very slowly and leave a lot of messy yellowish-white residue. Just about its only redeeming factor is it will give off that fantastic burned Black Powder smell.

My early ventures into Black Powder making were at the beginning of my high school years. Most of us admitted trying to make Black Powder. Most of us did. And the rest were probably liars! Those that made Black Powder, for the most part, were liars too.

The testimonies at the time were just unbelievable. Most of the aspiring Black Powder makers used methods I have just described. Some didn't even make the effort to get the ratios right. But most boasted of incredible explosions with such primitive powder. And most of these stories were highly exaggerated if not downright lies.

Well one can get such a mix to explode if one places enough of it in a sealed container before igniting it. Such an explosion, in comparative terms, is normally very weak. So our primitive Black Powder is just about useless for most things pyrotechnic. It won't make a firecracker. It can't be used as a propellant for shells, comets, or even stars. And it is unlikely to make good rocket fuel. So where do we go from here? The next level is to grind the ingredients.

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## **Grinding Away**

The major deficiency in the above method is the particle size of the ingredients. The larger the size, the slower the burn rate of the powder. So one needs a method to grind each ingredient as finely as possible. Another deficiency is the lack of corning or granulation. This is a process whereby the three ingredients are made into granules.

Granulation will be discussed in the next section. This section focuses on grinding.

One of the oldest methods used to grind Black Powder is the pestle and mortar. Pestles and mortars are commonly used today in laboratories to hand grind chemicals. They are also found in many kitchens where they are used for grinding spices and other food products. So the next step at entry level Black Powder is to obtain a pestle and mortar.

Kitchen pestle and mortars are about the cheapest and easiest to get. A better bet is a small laboratory pestle and mortar made from porcelain or ceramic. These typically cost in the order of between fifteen and thirty US dollars. For the uninitiated, the pestle describes the short rod-like object, the mortar being the bowl the material is placed in. Porcelain and ceramic pestles and mortars have rough grinding surfaces.

To improve the method of making Black Powder described in the previous section, each ingredient is ground separately using the pestle and mortar. This is done by first measuring out the right amount and placing it in the mortar. One then grinds the material by holding the mortar in one hand and the pestle in the other. Grinding is then done in a circular motion by pressing the grinding end of the pestle against the inner wall of the mortar while trapping material between the two. The grinding end of the pestle is the larger, more rounded end.

Note at this stage the method used is to grind each ingredient separately. Later on we will focus on the pros and cons of grinding materials together.

A good place to start is by grinding the charcoal. Once this is ground to a point where one feels one doesn't want to grind it any further, one places the charcoal in the mixing bowl. Next to be ground is the sulfur. Usually sulfur is supplied as a fine powder so possibly little is to be gained by grinding it. Potassium nitrate usually needs to be ground.

The mixing procedure with the ground ingredients is the same as described in the previous section. The resulting Black Powder should be better, maybe a lot better. But it will still be far from what is needed for fast powder applications. We now look at the next step. To do this we need another ingredient - water.

## **Water Works its Wonders**

The earliest Black Powders were made in ways similar to those just described. They suffered from one major deficiency. Their ingredients tended to separate from each other. So powders transported to the front in battles had to be re-mixed prior to their use in cannons. Failure to do so could result in misfires.

Any mixture of powders made from substances having different densities will experience some degree of separation over time. This separation is accelerated if such mixtures are transported or otherwise subjected to movement, especially vibratory movement. There is a simple cure for this problem: make the powder particles stick together. In Black Powder, the way to do this is to mix them with water.

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While some of the pyrotechnic literature recommends small amounts of substances such as dextrin or starch to aid the process of binding the powder particles together, just plain water is usually sufficient. Powders made with binders other than just plain water also have a reputation for burning more slowly. This process of binding the particles together is known as granulation or corning.

## **Granulation**

Granulation (or corning) is essential in making good Black Powder, for the following reasons:

- It promotes faster burning.
- It can be used to control the burn rate
- It reduces the dust hazard.
- It aids incorporation.

These advantages are explored in detail as follows.

**Faster burning** results from more air coming into contact with the Black Powder. This air fills the spaces between the powder grains and thus supplies more oxygen to the ignition process. While a pile of Black Powder that hasn't been granulated will still burn, its burn rate will be a lot slower.

**Control of the burn rate** can be achieved by varying the size of the powder granules. Generally the larger the granules, the slower the burn rate will be. Conversely smaller granules give a faster burn rate.

Why slow down the burn rate? The early makers of Black Powder found out that it was better to use slower burning powders in cannons and faster burning powders in pistols and muskets. The same principle holds true today. Roman candles tend to use faster powders than mortars firing large shells.

Shape is also a factor in controlling the burn rate. More crudely made powders contain granules of different shapes. Better made powders aim for spherically-shaped granules that give more predictable ignition characteristics. Such powders are desirable for sporting Black Powder firearms. Black Powder used in fireworks has less exacting requirements and usually has irregularly-shaped granules.

Round granules of Black Powder burn at a steadily decreasing rate. This is due to the fact that their surface area decreases as the burning progresses. Such powder was usable in cannons but inefficient. More efficient powders aimed for a more constant surface area. This was achieved by manufacturing hollow granules. These granules thus had two distinct surface areas, the one increasing in size during burning, the other decreasing. This in effect created a near constant burning surface area.

Cannons using such powders had a greater range. The more constant burn rate also put less stress on the cannons themselves, making them less prone to bursting when firing.

The hollow powders just described were popularly known as *prismatic powders* because of their prism-like shapes. Many other shapes were later tried, especially during the 19<sup>th</sup> century.

**Reducing the dust** hazard has important safety ramifications both for the manufacture and use of Black Powder.

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Dust in the air can ignite very easily. Dust that has settled can also pose a hazard, especially if it settles on an ignition source such as electrical contacts. Either way, dust is dangerous. Airborne dust can pose a very dangerous explosive hazard. And this hazard is not necessarily restricted to dusts that are themselves explosive or even ignitable under other circumstances.

Ever wonder why they don't use flour in fire extinguishers? Well flour can be very explosive. Put a match into a pile of flour and it will be extinguished immediately. Light a match in a room with sufficient flour dust in it, and it may be just the last match you light. Why? A pile of flour has so little air mixed with it that the burning match quickly dies from lack of oxygen when immersed in it. Flour dust, however, has a much higher air to flour ratio and enough space between the flour particles to ensure ignition. The same principle applies to the raw ingredients used in Black Powder.

This principle of starving or adding air to the ignition process has already been discussed in the previous sections on granulation. Just a word of advice. The trick using a pile of flour to extinguish a match works well with flour. Don't try it with a pile of Black Powder, even if it is un-granulated!

Dust can also create health problems. While the ingredients of Black Powder are not toxic, one should try to avoid breathing in their dust. This dust can act as an irritant to one's lungs and sinuses.

**Aiding incorporation** of the potassium nitrate is a big plus in making Black Powder.

The more intimately the potassium nitrate is incorporated (or mixed) with the other ingredients, the faster and more clean burning the powder.

Potassium nitrate is readily soluble in water. This means that the potassium nitrate particles can be broken down right to the tiniest size possible when dissolved. If such a process is carefully controlled in making Black Powder, a superior powder results.

### **Safer When Wet**

Wet Black Powder is generally safer to work with. This is because wet powder is less prone to accidental ignition.

Accidental ignition of Black Powder is one of the greatest dangers in its manufacture and use. Even though Black Powder is a lot less powerful than most other explosives, it can be considered as being more dangerous to make and work with. This is because Black Powder needs to be ignited to explode and because it is relatively easy to ignite Black Powder.

Many explosives merely burn when ignited, even if they are confined. Black Powder can explode very violently if ignited while it is confined. And it only takes a small spark from any source to do the trick.

Wetting the Black Powder during manufacture greatly reduces the possibility of accidental ignition. First, any wet substance, including Black Powder, is more difficult to ignite. Second, water reduces possible sources of ignition such as friction and static electricity.

A word of warning! Black Powder can still ignite when wet, and still burn violently once ignited. I found this out the hard way and ended up by literally burning my fingers in the process.

This unhappy event occurred when I decided to get rid of a small pile of newly-made wet powder that had spilled on the ground. I lit a match and applied it to the small pile of wet stuff. It

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ignited almost immediately, causing me to fall over backwards with fright as it burned with a loud woof! It burned very quickly and very well, leaving an almost undetectable amount of residue and some very sore fingers.

### **Making Black Powder Using Granulation**

The first stage of the granulation process involves mixing water with the other three Black Powder ingredients. The trick is to use just enough water. Too little water results in the powder not being wetted enough for the particles to cling to each other. Too much water results in an unworkable *soup* that cannot be properly granulated and may result in some of the potassium nitrate being lost.

So we aim to make a thick slurry by progressively adding small amounts of water to the other ingredients. This is quite a tedious process, as the sulfur and charcoal tend to not mix well until a certain amount of water has been added and the mix stirred sufficiently.

When the ingredients have been thoroughly mixed the actual process of creating granules can begin. Here one has two options:

- Wet granulation
- Dry granulation.

**Wet granulation** is done by taking the wet mixture and forcing it through a sieve. Here a sieve can be made from a piece of screening used on windows or doors. A kitchen sieve can also be used.

The secret to this process is not to have the powder too damp. Powder with the right dampness will form granules or short worm-like pieces when forced through the sieve. Powder that is too damp will recombine into a single wet mass after going through the sieve.

The wet granules are then spread out on a flat surface and left to dry. Some recommend fast drying in the sun for this process. And I emphasize for this process. Other processes that form the powder into pucks before drying should preferably be dried more slowly in the shade.

When the granules are properly dried they are sieved again.

**Dry granulation** is the preferred method of commercial Black Powder manufacturers. Generally this method is also preferred by those who make their own homemade powder.

The dry granulation process is the most dangerous part of commercial Black Powder manufacture. The same cannot be said for homemade powders. Here the milling process is usually the most dangerous part. This is due in part to commercial milling using a wet process, while amateur milling is usually done dry.

Dry granulation is usually done when the wet Black Powder mix has been formed into pucks or pellets and then allowed to dry. It can be done without this process by first spreading the wet Black Powder onto a flat surface and then letting it dry. The resulting solid pieces can then be placed between two sheets of paper and a rolling pin lightly run over them. Only light pressure should be applied to avoid crushing the pieces to fine powder.

The resulting granules are sieved again.

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## **Improving the Process**

The above processes can be improved in a number of ways.

First one can grind all three components together once they have been dampened. This wet process is not entirely safe, but a lot safer than doing this procedure with dry components. This is because the water reduces heating due to friction and also prevents ignition due to static.

Another change worth trying is using a 50/50 mix of alcohol and water instead of plain water. Some have reported using 70% isopropyl alcohol, which is effectively 70% alcohol and 30% water.

Alcohol is reputed to aid the drying process and may also affect the surface tension of the water, which will impact the mixing process. A word of warning! Adding alcohol to the wet mix increases the chances of accidental ignition. Alcohol will burn, even when mixed with water.

Further improvements are described in the following chapters.

## *Chapter 4 — The Charcoalfactor*

### **Introduction**

Some have considered charcoal to be the second most important factor in making Black Powder. It is actually the first.

Good charcoal can give a usable powder with inferior powder making techniques. The converse is not necessarily true. So the first step in making good powder is to get a supply of good charcoal. This goal, in itself, may present quite a challenge. This chapter focuses on what charcoal is and its importance in Black Powder.

On the practical side, different types of charcoal and their relative performances are described. These descriptions are aimed at helping one make intelligent choices in selecting charcoal or wood to make charcoal from. Charcoal making is described in the next chapter.

Some readers may think that I have focused too much on charcoal briquettes and willow charcoal. I have done this for a reason. Briquettes are a bad choice for a number of reasons that need to be highlighted, willow charcoal has centuries of history behind it. This means that fiction has become mixed with fact, and we need to clearly separate the two.

### **What is Charcoal?**

Charcoal is a form of carbon derived from organic matter that has been heated. Other common forms of carbon are graphite and diamonds. Graphite is the main ingredient of the *lead* used in pencils. A diamond is a very pure form of carbon and is the hardest known substance. Diamonds are commonly known for their uses in jewelry. They are also used extensively for cutting other hard substances.

Charcoal, graphite and diamonds differ considerably. Charcoal and graphite are a lot softer than diamonds. Diamonds are typically white in color, while charcoal and graphite are black or dark gray. Charcoal can be converted to graphite by heating it to high temperatures. Diamonds can be made from graphite by subjecting the graphite to high temperatures and pressures.

The principle difference between charcoal and other forms (also referred to as *allotropes*) of carbon is that charcoal is known as *amorphous* carbon while the other forms are called *crystalline* carbon. Thus the carbon atoms in charcoal are arranged in a rather haphazard fashion compared to very ordered alignments found in crystalline carbon. Also charcoal should never be regarded as pure carbon, but rather as an organic hydrocarbon compound. This means that charcoal contains other elements such as hydrogen.

Charcoal, graphite and diamonds are natural occurrences of carbon. Another allotrope of naturally occurring carbon is coal in its various forms. Recently scientists have created a new family of carbon allotropes called *buckyballs*. Each buckyball contains a large number of carbon atoms, giving each buckyball a unique shape. Buckyballs have no proven role in pyrotechnics at the moment, and are beyond the bounds of this discussion.

We can consider organic matter simplistically as the matter that makes up plants or animals. Charcoal can be made from either, but all charcoal used in Black Powder is made from plant

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matter. It's important to draw a distinction here. While plant charcoal comprises mainly carbon, certain types of animal charcoal contain bone that has been heated to high temperatures. Here calcium is the main ingredient. Such charcoal could be considered as being a calcium-rich form of ash. Thus many forms of animal charcoals are nothing like their vegetable counterparts.

The principle element in organic matter is carbon. This is combined with other elements such as hydrogen, oxygen and nitrogen to form complex chemical compounds. When organic matter such as wood is heated, some of the chemicals contained in it are driven off as gases. Other components undergo both physical and chemical changes resulting in a substance comprising mainly carbon plus small quantities of hydrogen and oxygen. Other elements may also be present. Charcoal is thus carbon plus other substances. These other substances are referred to as volatiles.

Just as there is no one true formula for Black Powder's chemical reaction, there is no one true formula for charcoal. Again, some authors of textbooks and technical papers have erred here. The formula for charcoal depends on which charcoal it is meant to represent. Often this representation is approximate and not exact. Unlike chemical compounds that usually can be represented by exact formulas, charcoal should be considered a complex rather than a compound. The difference here is that a chemical compound is formed by different elements bonded together. A complex is a mixture of chemicals without the bonding. For example: potassium nitrate is a compound, Black Powder is a complex.

The situation is slightly more complicated in charcoal than it is in other complexes. Charcoal has the property of being able to adsorb other substances. These substances attach themselves to the surface of the charcoal in a process known as absorption. This is not a true chemical bond but a strong binding together of molecules nevertheless.

The following table shows that even charcoal obtained from the same type of wood may have variations in chemical content.

The data in Table 4-1 is extracted from experiments conducted by Sasse [1] in trying to determine the characteristics of Maple charcoal. These data demonstrate that noticeable differences in chemical composition are to be found in charcoal made with the same type of wood.

Sample	O <sub>2</sub> (%)	C (%)	H <sub>2</sub> (%)	N <sub>2</sub> (%)	S(%)	Ash (%)
1	12.71	78.40	3.26	0.44	0.68	5.11
2	13.10	78.40	3.24	0.35	0.01	4.90
3	14.20	75.83	3.15	0.35	0.02	6.45
4	14.14	75.41	3.24	0.34	0.02	6.85
5	16.03	76.87	3.49	0.32	0.01	3.30

Table 4-1. Chemical Characteristics of Rosewood Maple Charcoal

These values, however, are close to the empirical formula of  $C_{10}H_7O_2$ . Sasse [2] also makes references in a later paper to other empirical formulas for charcoal:

C8.68H4.96O2.00

The above formulas reflect the carbon/volatiles ratio of their respective charcoals. These charcoals can be taken as average for Black Powder applications. Highly carbonized charcoal may be represented by a different formula. Shimizu<sup>6</sup> gives one such example: C<sub>20</sub>H<sub>7</sub>O. This represents a charcoal containing the following percentages:

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Carbon	91%
Hydrogen	3%
Oxygen	6%

Volatiles play a very important role in charcoal. In Black Powder these volatiles assist the charcoal's reaction with potassium nitrate and sulfur. It is thought that the oxygen in the charcoal is the main player here, with hydrogen also having a role.

## **Carbonization - Turning Wood into Charcoal**

Wood is converted into charcoal by a process known as carbonization. The term pyrolysis and destructive distillation also are used to describe the chemical changes that take place when wood is heated. This heat process is a process whereby the wood is heated but not burned. The term *wood* here also applies to other vegetable matter, such as grasses.

Carbonization can be thought of as simply a process where the wood is heated hot enough to burn but does not burn properly because the process is starved of oxygen. Every method used to make charcoal relies on this principle, some methods giving better results than others. The simplest method of making charcoal is to light a fire, let it burn for a while, and then extinguish it. If you had let it burn long enough you would be left with charcoal and ash. If the fire had burned too long you would be left with mainly ash; too short, and the wood would not have charred enough.

The carbonization process begins at about 100 °C when water vapor is driven off.

At about 270 °C volatile gases and liquids are released from the wood. This temperature varies according to the type of wood and other conditions, and is usually in the range of 250 - 290 °C. These gases and liquids are commonly referred to as *volatiles*. These volatiles will ignite in the presence of oxygen. If this ignition occurs with an unrestricted supply of oxygen, the wood will burn in the normal way and be consumed. If the oxygen supply is sufficiently restricted, mainly the volatiles will burn, with very little of the wood burning. This burning usually takes place at temperatures between 400 - 600 °C.

So allowing some oxygen into the process allows the combustion to become self-sustaining. At this point the process is described as being *exothermic*, i.e. giving off heat. Many charcoal manufacturing methods rely on this phenomenon. This exothermic reaction holds a particular attraction to charcoal makers, in that an external heat source is not required to produce charcoal. Indeed, the oldest methods of making charcoal relied on this method, as do many today.

To recap:

- The wood is heated by igniting it in the way one would normally light a fire.
- Volatiles start being released at about 270 °C.
- The volatiles start burning.
- The burn rate of the volatiles is controlled by restricting available oxygen.
- This burning becomes self-sustaining at 400 - 600 °C .

Finally, when most of the volatiles are burned off, the smoke color changes from gray to blue or maybe even becomes transparent. The carbonization is now complete. At this point all oxygen should be cut off from the charred wood to ensure that it does not ignite. The charred wood (charcoal) is then allowed to cool down. Prematurely allowing oxygen to come into contact with the charcoal before it has cooled sufficiently may cause it to ignite.

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The resulting charcoal consists of carbon plus volatiles plus ash. The percentages of these is determined by the carbonization process itself and the characteristics of the wood. An important variable is temperature. The higher the temperature of carbonization, the higher the carbon content, and thus the lower the volatile content. This is shown in the following table of data collected by a French scientist named Violette.

Carbonization Temperature (°C)	Charcoal Color	Yield (%)	Composition of Charcoal (%)		
			C	H	O + N
280-300	brown	34	73.2	4.3	21.9
350-400	black	28-31	77-81		
1000	black	18	82.0	2.3	14.1
1250	black	18	88.1	1.4	9.3

Table 4-2. The Effect of Carbonization Temperature on Charcoal's Chemical Composition.

Violette made an extensive study of charcoals used in Black Powder. His findings show a direct relationship between Black Powder ignition temperatures and the temperatures at which the relative charcoals were carbonized. These findings are important because the burn rate of Black Powder is related to its ignition temperature. Generally, the lower the ignition temperature, the faster the burn rate. Note: I used the term *generally* because there are exceptions to this rule.

Carbonization Temperature (°C)	Ignition Temperature (°C)
260-280	340-360
290-350	360-370
432	400 (approx.)
1000-1500	600-800

Table 4-3. The Relationship between Carbonization Temperature and Ignition Temperature.[1]

Looking at the above data, we notice that very high temperatures (in excess of 1000 °C) raise the ignition temperature quite considerably. Also worth noting, is the higher carbon content of charcoal that has been heated to these higher temperatures.

So higher temperatures raise the carbon content and lower the percentage of volatiles. These percentages are important. Volatiles in charcoal noticeably affect the burn rate of Black Powder made with it. Generally, it is desirable to use such charcoal rather than charcoal where the volatiles have been driven out. Research done by Sassel[1] determined that a 25% volatile content was about the optimal amount for Black Powder use. This figure came from his own research and reference to recent research done by Kirshenbaum, Hintze, Gray, March, Robertson and Rose.

To arrive at the optimal volatile content we see, from the above data, that carbonization should take place between 300 and 400 °C. At 300 °C and below the percentage of volatiles is too high, and there is a possibility of the wood not being properly carbonized. At 400 °C and above the volatiles drop below the optimal 25%. Note: these figures should be taken as approximate values because different woods will have different carbonization characteristics.

Another fact worth noting is self-sustaining methods of charcoal production raise the temperature of the carbonization process to between 400 and 600 °C. This could lower the percentage of volatiles to below optimum. Retort methods that rely on external heat sources don't suffer from this constraint if sufficient attention is given to controlling the heat source.

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If one heats charcoal to very high temperatures it will turn into graphite. I admit that for quite a long period I subscribed to the doctrine that this posed a problem to those making charcoal for Black Powder or other pyrotechnic uses. I am not the only one who got sucked into this doctrine. There are others who still reckon that graphite is an issue that charcoal makers should take note of. My position now is quite different.

Contrary to some opinions, amorphous carbon changes into graphite at temperatures much higher than those encountered in normal charcoal production. Typically these temperatures are in excess of 2000 °C. Graphite is often manufactured at temperatures of 2800 °C and above.

Myths about graphite haven't only infected the pyrotechnic community. New developments in carbon fibers are often mistakenly referred to as graphites. So we have *graphite-based* golf clubs and other modern marvels using the unique properties of carbon fibers. In fact, none of these new developments use graphite. The carbon in question is amorphous carbon that has undergone carefully controlled heat processes. Some of this carbon comes from wood but usually comes from man-made hydrocarbon compounds and complexes. This carbon has been carefully heated to temperatures well above charcoal carbonization temperatures but below temperatures where graphite is formed.

Now graphite may form at lower than normal temperatures in the presence of catalysts. Different metals are known to have this catalytic property. So the walls of a retort may possibly introduce a small amount of graphite into the charcoal. Chances are it won't, at least not to an extent where it becomes an issue.

Personally, I think the whole graphite issue has become an unnecessary distraction, being fostered by those who resist the idea that volatiles play a major role in charcoal's performance. And while the research into charcoal can still be regarded as young, the role of volatiles has been confirmed by scientists in Britain, Europe and the USA. Other scientific data shows that graphite is formed at much higher temperatures than those suggested by some. And until someone can demonstrate with sufficient credible experimental data that graphite is an issue, I believe we can safely ignore it.

## **Good Charcoal vs. Bad Charcoal**

Just as some will argue that there is no such thing as an ugly woman, some will contend that there is no such thing as bad charcoal. In one sense they are right. All charcoal (or most of it) is good for one or other purpose. Some is good for barbecues. Other charcoal is less suitable. Some works well in gas masks. Others are better suited to filtering liquids. Some charcoals are good in Black Powder. Others are just plain bad.

The two most important criteria for the choice of charcoal are:

- what it is made from
- how it is made.

**What it is made from** determines whether a charcoal will burn fast, slow, hotter or cooler. It may also determine other properties, but in Black Powder we are mainly concerned with the speed at which it burns. Certain woods yield faster burning charcoal than others. Typically these are softer woods, such as willow. Different woods are examined later in this chapter.

**How it is made** can be a deciding factor in Black Powder performance. Charcoal that is heated to too high a temperature loses volatiles that are vital to its performance in Black Powder. And

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while there are many ways to make charcoal, the method known as the *retort method* has a track record of being the best for Black Powder charcoal. So the retort method is the method of choice and is focused on extensively in the next chapter.

### **Charcoal Briquettes**

In times past, many pyrotechnic amateurs enthused about making Black Powder from charcoal briquettes. These briquettes were cheap and easy to come by. The fact that briquette charcoal has largely superseded plain wood charcoal as barbecue charcoal may also have something to do with it.

While it is wrong to totally dismiss claims of useable Black Powder being made from briquette charcoal, it is undesirable to enthuse about them. Briquettes are quite a horrible choice where Black Powder charcoal is needed. Briquettes are a bad choice for the following reasons:

- Briquettes are made from hardwood charcoal
- Briquettes contain a binder
- Briquettes leave a high ash content
- Some briquettes contain chemicals (other than binders)
- Some briquettes contain coal
- Some briquettes contain wood that has not been charred.

You just don't know what you are getting with briquettes. But be assured, most of the stuff you get with them is just plain bad for good Black Powder. Working our way up the list from the bottom, let's examine the reasons why briquettes may not be a good choice.

**Wood** is found in certain briquettes. Here the issue is not just wood that has not been charred. While it is possible that some briquettes contain wood that has escaped charring in the charcoal making process, wood is introduced deliberately in some briquettes to improve the cooking process. These briquettes are normally sold as such, boasting an improved smoked taste being imparted to the meat being barbecued.

**Coal** is added to the charcoal in some briquettes. A very popular brand of briquette is alleged to contain both anthracite and *mineral* charcoal. Mineral charcoal is a type of coal and is not a charcoal in the normal sense.

**Chemicals** (other than binders) are alleged to be found in a very popular brand of briquette. These include sodium nitrate, lime and borax. Other briquettes are alleged to contain petroleum products. It is true that some briquettes do contain petroleum products because they are sold as such. These products are similar to the fire starter liquids used to start charcoal fires.

**High ash content** is very common in briquette charcoal. Anyone who has cleaned out a barbecue that used charcoal briquettes knows this. The ash issue, however, goes beyond the issue of normal ash produced by either wood or charcoal. Briquette "ash" contains residues of other substances added to the charcoal. Some have suggested that such substances could be either clay or lime.

**Binders** used in charcoal briquettes may inhibit the Black Powder's burning characteristics. In most instances, binders are avoided in Black Powder manufacture. Another problems with briquette binders is that one usually does not know what one is getting. Some briquettes use only water as a binder. More commonly, chemicals of one type or another are used. Starch is a popular binder for briquettes. Examples of other organic binders are: bituminous material, waste pulp mill liquor, and citrus cannery waste. Presently the state of the art is changing with

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new non-organic chemical binders being introduced.

**Hardwood charcoal** generally burns more slowly than its softer counterparts. Briquettes use hardwood charcoal because of this property. This feature is desirable in barbecue charcoal but not in making Black Powder.

Some of the allegations concerning the contents of briquettes are quite easy to substantiate. Others are not. I was tempted to mention the name of a popular briquette that is alleged to contain all kinds of additives, but decided not to when my queries addressed to the manufacturer were ignored. Others have claimed that this manufacturer came clean with them, divulging the contents of their briquettes. These claims seemed quite credible, as the content descriptions were augmented by descriptions of their functions. The manufacturer also described his manufacturing process in a credible manner. So while there is quite compelling evidence to substantiate the claims of additives to this popular brand of briquette, this evidence is not confirmed satisfactorily enough to reveal its name.

With all these negatives concerning charcoal briquettes, are they useful for Black Powder making? Directly, no. Indirectly, yes. Charcoal briquettes can be used as a fuel to cook up one's own homemade charcoal!

## **Willow Charcoal - Myths and Facts**

For centuries many have regarded willow charcoal to be superior to other types when making Black Powder. Older Black Powder literature abounds with references to willow charcoal, making few references to charcoal from other trees or plants. While some advocate using vine charcoal, even these references tend to lump vine charcoal together with willow. Thus willow charcoal appears to be a clear winner. But is it the best?

Comparison tests made on willow charcoals, together with charcoals from other woods, have indicated that although willow charcoals perform well in Black Powder, certain other charcoals perform better. So to claim that willow is the best is to subscribe to a centuries-old myth.

Another consideration is that not all willow charcoal is created equal. Manufacturing methods aside, in North America alone there are an estimated 75 to 90 different species of willow. This difference in estimates may have something to do with the fact that certain species of willow are hybrids, being both difficult to properly classify and identify. Worldwide an estimated 300 species of willow exist, willows come in different shapes and sizes, ranging from plants a few inches tall to large trees.

With so many varieties of willow, it is a sure bet that not all of them have been tested for their charcoal-producing properties. To date, only a few have been tested with any reasonable degree of scientific accuracy. So there is still a whole new world out there for the keen experimenter.

However, tests that have been done on willow charcoal have revealed an undisputed fact - willow charcoal does make a good fast-burning Black Powder. And it has done so for centuries all over the world. So one can't go wrong with properly-made willow charcoal. And the facts so far indicate that any type of willow will yield acceptable charcoal.

Having extolled the virtues of willow charcoal, we go to the next question - where does one get it?

There are two routes to obtaining willow charcoal:

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- buying it
- making it one's self.

Buying willow charcoal is easier said than done. Most charcoal suppliers sell hardwood charcoal. Even charcoal bought from suppliers of pyrotechnic chemicals is usually of a hardwood variety. There are, however, suppliers of charcoal that specifically sell willow charcoal and identify it as such. These include:

- medicinal suppliers of organic medicinal substances, including charcoal
- artist materials suppliers
- pyrotechnic suppliers.

**Medicinal suppliers** is probably the most expensive choice. Their charcoal demands a certain purity for human consumption and being *medicinal* in itself suggests justification for high prices. Some of this charcoal may also be activated to the extent that its useful volatiles have been driven out. Another problem area is the charcoal's *medicinal value* may have been enhanced with other substances. Some of the medicinal suppliers are really into fringe medicine. Others go even beyond that into alchemy, sorcery and witchcraft. (No, I'm not kidding!)

**Artist materials suppliers** sell willow charcoal sticks that are used for charcoal sketches. Other woods are also used, so it is best to specify willow when ordering.

**Pyrotechnic suppliers** should be considered the best choice, if and when they can be found. Willow charcoal is not normally available from the more prominent suppliers of pyrotechnic chemicals. Rather it is usually sold by individuals who have made the stuff themselves. Quality will naturally vary, as will the type of willow wood used. A big advantage in ordering from such persons is that they can describe the method used to make their charcoal. This should give one some indication of the charcoal's performance.

Making one's own charcoal is often the better choice for a number of reasons. These, together with practical ways of making charcoal are described in the next chapter and will not be dealt with here. But, before one can make charcoal, one must ensure a supply of the right type of wood - in this case, willow wood. The following section describes a few of the more common willow trees, giving both their common and botanical names.

### **Weeping Willow** (*Salix babylonica*)

I do admit that I once subscribed to the myth that most willow trees were a type of *weeping willow*. It took me quite a while to realize that not all willow trees had long *weeping* types of branches. Many don't look at all like Weeping Willows so far as their branches go. However, close examination of the leaves, bark and flowers of the different varieties of willow reveal similarities that make them relatively easy to identify.

My experiments with willow charcoal were done exclusively with weeping willow (*Salix babylonica*) wood. The reason for this was that weeping willow trees grew abundantly alongside dams and streams near where I lived. Finding dead branches from these that were useable for making charcoal was relatively easy. So I had a plentiful supply of willow charcoal that worked well and thus looked no further. Other pyros who have tested weeping willow charcoal (on both sides of the Atlantic), report good results.

The weeping willow is reported to have originally come from China. It is now grown in many parts of the world, including the USA.

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## **Black Willow** (*Salix nigra*)

Black willow was a top performer in the tests done by Wilson. Black willow is mainly found in the eastern parts of the USA. Here it occurs over a very wide area. It is also found in some portions of the mid-west and southwest of the USA. The black willow is reputed to be the only willow wood used for lumber in the USA.

Black willow features quite frequently in references to Black Powder made in the USA. This is not surprising as most of the Black Powder manufacture occurred in regions where black willow grows.

## **Rocky Mountain Willow** (*Salix monticola* Bebb)

Rocky Mountain willow performed well in tests done by Wilson<sup>3</sup> but not nearly as well as Black willow.

## **Crack Willow** (*Salix fragilis*)

Crack Willow (a.k.a. brittle willow) originates in Eurasia and was introduced to North America during the Colonial period. It is reputed to give a very good Black Powder charcoal.

## **White willow** (*Salix alba*)

White willow is found in the eastern USA and was introduced from Europe. Charcoal made from the white willow is one of the charcoals specified for Black Powder used by the British Military.

## **Wisconsin Weeping Willow** (*Salix blanda*)

The Wisconsin weeping willow is a hybrid between *Salix babylonica* and *Salix fragilis*. Both of these trees have a good track record concerning Black Powder charcoal, so a hybrid of the two could have potential.

## **Pacific willow** (*Salix lasiandra*)

Pacific willow is described as an excellent performer in the tests done by Wilson<sup>3</sup>, although there is some uncertainty concerning the identification of this particular source of willow wood. The uncertainty relates to this particular tree being thought to be black willow. Indeed black willow and Pacific willow do resemble each other. Pacific willow, however, is more common in the area where this sample was got from.

Pacific willow is found along the Pacific coast of Canada and the USA and elsewhere in the western parts of these two countries. Other charcoal types are discussed in the following text. These are arranged in alphabetical order.

## **Acacia Charcoal**

Acacia trees grow prolifically in Africa and are found in other parts of the world, including North America and Asia. They are often used as sources for charcoal. The only reference I could find for Black Powder charcoal was that from the *Acacia jacquemontii* tree that is found in India.

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## **Activated Charcoal**

Most charcoal can be considered as having been *activated* to a degree. The activation process is principally one whereby the charcoal is heated to a temperature high enough to drive out substances that have been adsorbed by it. This frees the charcoal up to adsorb other substances. Note the word *adsorb* is not a wrong spelling of *absorb*. It does have a different meaning. Adsorption is similar to absorption but also quite different.

If one spills some water, one might use a cloth or a paper towel to remove it. The cloth or paper towel absorbs the water. In this process the water soaks into the other substance and becomes part of it. Adsorption works differently by attracting the other substance to its surface where it sticks in a way similar to steel filings sticking to a magnet. In activated charcoal, this adsorption is incredible, with charcoal being able to adsorb very large amounts of other substances.

Activated charcoal is thus the charcoal of choice in filters used to filter liquids or gases. There is a school of thought that suggests that charcoal made for Black Powder use should be activated to give better performance.

For Black Powder use, activation may come as a mixed blessing. Some activation typically results in the charcoal adsorbing oxygen from the air. This gives a faster reacting charcoal. Too much activation would tend to drive out the volatiles that are essential for better performance.

Not all activated charcoals are created equal. They are made from different woods, some with better activation properties than others. Charcoal made from coconut shells is considered to be superior for gas adsorption and certain other chemical adsorption processes. Some activated charcoal on the market is not charcoal at all, but anthracite, a hard form of coal. The activation process itself may be different for different applications.

As stated earlier, the activation process is principally one of heating the charcoal to drive out substances that have been adsorbed by it. In new, unused charcoal, these substances are mainly volatiles. Some heat activation methods use superheated steam to heat the charcoal and rinse out the volatiles.

Another important component of activation is grinding the charcoal to expose more of its surface area. A third component, used in specific applications, is to add chemicals to the charcoal. These chemicals, in turn, react with other chemicals in application-specific reactions. Sometimes these chemicals are added before the activation process in order to assist it. In other applications the chemicals are added after activation.

Typically commercially available activated charcoal is made from hard wood. This alone makes it a bad choice for Black Powder. The volatiles in charcoal are essential for fast-burning Black Powder. Removing these seriously degrades the potential usefulness of the charcoals. Chemical additives are another no-no.

Overall, activated charcoal is a bad choice for the Black Powder maker.

## **Ailanthus Charcoal**

Ailanthus (*Ailanthus altissima*), a.k.a. Tree of Heaven, performed very well in Wilson's<sup>3</sup> tests. This tree is native to Asia, but has been introduced to the USA where it has thrived. It has thrived to the extent that it is now regarded as a weed. This *weed* status may be good news, at least for a while. People like to get rid of weeds by digging them out or chopping them down, and will welcome anyone helping to clear away the wood.

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## **Air Float Charcoal**

Many have been attracted to air-float charcoal because it comes as a finely ground charcoal, ready for incorporation with other chemicals. It seemed the natural choice to those that thought that finely dividing the ingredients of Black Powder was more important than other considerations.

The term *air-float* merely defines that the charcoal is finely ground. It does not specify the type of wood the charcoal is made from or how the charcoal is made. Typically air-float charcoal has been made from hard woods. Tests with air-float charcoal in Black Powder mixes have yielded disappointing results.

So much for air-float charcoal the way it used to be. Recently makers of pyrotechnic charcoals have been advertising air-float charcoal made from lighter woods such as willow and Paulownia. These should work very well in Black Powder.

## **Alder Charcoal**

Red alder (*Alnus rubra*) charcoal gave some of the fastest Black Powders in the tests done by Wilson<sup>3</sup>. Thinleaf alder (*Alnus tenuifolia* Nutt.) was somewhat slower. These two varieties of Alder are found in the USA. The French are reputed to have used another variety, *Alnus glutinosa*. This variety is also one of the charcoals specified for Black Powder used by the British military.

Italian alder (*Alnus cordata*) is also reputed to yield a very good Black Powder charcoal.

## **Animal Charcoal**

As stated elsewhere, all Black Powder on record has been made from *vegetable* rather than *animal* charcoal. I could leave the matter to rest at this last statement, but I just know that there is someone out there that may be tempted to investigate the properties of animal charcoal in making Black Powder. Hence some discussion on this matter is in order.

Unlike vegetable matter that is comprised mainly of carbon, animal matter is comprised mainly of carbon and calcium. The calcium is found in the bone matter and most of the carbon in the rest. Some charcoal that is sold as *animal charcoal* is actually bone charcoal that has a high percentage of calcium. Another type of commercially made animal charcoal is made mainly from blood and is used as a blackening agent.

Yes, it is possible to make your own animal charcoal by just letting your steak cook too long. Wasteful and expensive! A cheaper option perhaps is to experiment with road kill. But be careful. Such activities may produce some unpleasant aromas and attract unwelcome attention from neighbors.

Personally, I don't think there are any scientific breakthroughs to be experienced by experimenting with animal charcoal.

## **Apple Charcoal**

When talking about *apple* charcoal we are referring to charcoal made from the wood of an apple tree (just in case there is any confusion).

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According to O'Neill<sup>4</sup>, apple charcoal ranks among the fastest Black Powder charcoals. The apple he used was *Pyrus mains*. Wilson's<sup>3</sup> tests on Oregon crabapple (*Malus fusca*) yielded very different results, being on the lower end of the performance scale.

There are many other species of Apple that may yield different results and are probably worth experimenting with.

## **Artist's Charcoal**

Some artists use charcoal to sketch with. This practice has been around for thousands of years. Early cave dwellers used charcoal to draw on cave walls.

Modern artist charcoal usually comes from willow or grape vines, both good choices for Black Powder charcoal. Artist's charcoal is available from artist suppliers. It is expensive, and would not be my first choice if I wanted willow or grape vine charcoal.

Some artist's charcoal is made from lime wood, also reputed to make a good Black Powder charcoal.

## **Aspen Charcoal**

Aspen trees grow profusely in my neck of the woods. In fact, during fall they become quite a tourist attraction. Many people come from all over for a type of pilgrimage to see the aspen trees near a small town called Cripple Creek. Aspen is related to willow and is a member of the poplar family. It makes good Black Powder charcoal.

Trembling aspen (*Populus tremuloides* Michx.) ranked among the top performers in Wilson's<sup>3</sup> tests.

## **Balsa Charcoal**

Nobody has come forward yet to substantiate claims of the effectiveness of balsa charcoal or to dispel these claims as a myth. Perhaps the prohibitive cost of balsa wood may have something to do with it. So the jury is still out. Balsa is either the best source of Black Powder charcoal around or just another pyrotechnic urban legend.

## **Bamboo Charcoal**

The use of bamboo (*Bambusa* sp.) charcoal can be traced back to some of the earliest Chinese developments of Black Powder. Bamboo was one of their preferred charcoals along with willow. O'Neill's<sup>4</sup> experiments showed acceptable results for bamboo, burning at a rate slightly faster than hemp. Bamboo charcoal has a high ash content.

## **Beech Charcoal**

The European beech (*Fagus sylvatica*) is found in Britain and Europe and has been introduced to North America where it is widely planted as an ornamental tree. It resembles the American beech (*Fagus grandifolia*) but is smaller, has darker bark, and smaller, more elliptical leaves.

Beech wood has been used to make Black Powder charcoal in Britain.

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## **Buckthorn Charcoal**

Both alder buckthorn (*Rhamnus frangula L.*) and Carolina buckthorn (*Rhamnus coroliniana*) were tested by Wilson<sup>3</sup>. These ranked among the top performers.

Alder buckthorn (*Frangus Alnus*) charcoal is specified for Black Powder by the British military.

## **Cherry Charcoal**

George Washington probably did not have charcoal in mind when he cut down the cherry tree. However, some Black Powder makers might.

Cherry (*Prunus virginiana*), a.k.a. chokecherry, performed adequately in Wilson's<sup>3</sup> tests.

## **Coconut Shell Charcoal**

Activated charcoal used for industrial and laboratory uses is often made from coconut shells. These give a superior activated charcoal in many laboratory and industrial applications, especially in gas adsorption applications. This is a hardwood charcoal and is probably not good for Black Powder making. The activation process itself may have driven off too many volatiles, making this charcoal even less viable.

Many, because of its popularity, have tried coconut charcoal. This has often been more by accident than design - they did not know beforehand that the stuff they were getting was made from Coconuts. To date, I have not seen any credible claims of coconut charcoal producing good Black Powder.

## **Cotton Charcoal**

Cotton fabric charcoal was tested by Wilson<sup>3</sup> and found to yield results on the lower end of the performance scale. Right near the bottom end was charcoal made from cotton balls. These tests conflict with the idea that cotton charcoal ranks among the best. The method of making the charcoal may have something to do with this disparity. Or, the stories about cotton charcoal being among the best may be yet another pyrotechnic urban legend.

## **Cottonwood Charcoal**

Cottonwood is type of poplar. Poplar features quite frequently in the Black Powder literature. Poplars are actually related to willows.

Cottonwood is alleged to have replaced willow during the American Civil war when willow wood was in short supply. Narrow leaf cottonwood (*Populus angustifolia* James) in Wilson's<sup>3</sup> experiments ranked among the top performers.

## **Dogwood Charcoal**

Flowering dogwood (*Cornus florida*) yielded Black Powder that burned at about 80% of the rate of the fastest powders in the tests done by O'Neill<sup>4</sup>.

Red dogwood (*Cornus sanguined*) has been used for Black Powder charcoal in Britain.

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## **Grape Vine Charcoal**

Charcoal made from grape vines (*Vitis* sp.) has been used for centuries. Grape vine charcoal burned at about 75% of the rate of the fastest powders in O'Neill's<sup>4</sup> experiments, so grape vine charcoal could be a very good choice. Similar tests performed by Wilson<sup>3</sup> gave less impressive results. There may be any number of reasons for this disparity, including the fact that there are different varieties of grape.

However, vine charcoal does have a high ash content, so one might consider adjusting the formula to optimize speed. As mentioned, there are many varieties of grape vines, with some probably giving better results than others.

Like willow charcoal, vine charcoal is also used by artists to make charcoal sketches. So one can buy vine charcoal from artist suppliers. This stuff works out very expensive and may not necessarily be made in a way that is best for use in making Black Powder.

## **Grass Charcoal**

No, this does not refer to hemp under another name.

Grass, in its various forms, has given disappointing results. My worst Black Powder charcoal came from a type of African pampas grass. Others have reported unsatisfactory results with lawn clippings.

An exception is bamboo, which is also a member of the grass family.

## **Hazel Charcoal**

Hazel (*Corylus avellana*) is native to Britain and Europe. Its wood has been used to make Black Powder charcoal in Britain.

Other varieties of hazel grow in the USA.

## **Hemp Charcoal**

The mere mention of the word *hemp* elicits a chuckle or raised eyebrows. This is because hemp describes a plant that is part of the *cannabis* family. This plant family yields a familiar drug known as *marijuana*, popularly known by many names such as *pot*, *grass*, *weed*, *hash*, *boom* and *dagga*.

The botanical name for the hemp plant is *Cannabis sativa*. This botanical designation includes plants that are high (pun intended) in drug content and those that have minimal amounts. The latter are still legally cultivated in some parts of the world to yield fibers used to make rope or twine. So the laws in some countries allow the cultivation of this variety, while forbidding the cultivation of the drug-yielding types. In the USA the cultivation of hemp is illegal, while imported hemp products in the form of rope or twine are not.

Tests done by O'Neill<sup>4</sup> indicate that hemp gave a Black Powder that performed approximately as well as mesquite but slower than bamboo. So hemp may be overrated. Perhaps a lot more testing needs to be done here to determine whether hemp can actually live up to its reputation as giving a superior charcoal.

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Shimizu<sup>6</sup> indicates that hemp charcoal has a tendency to absorb moisture and its ash content may tend toward the high side. The ash also has been shown to contain a number of different compounds of silicon, copper, potassium, aluminum, and phosphorus.

### **Hornbeam Charcoal**

Varieties of Hornbeam (*Carpinus*) are found in the Northern Hemisphere in North and Central America, Europe, Asia, and Japan. Hornbeams produce an extremely hard wood. In spite of this fact, Black Powder charcoal has been made from this wood in the past.

### **Horse Chestnut Charcoal**

The Horse chestnut (*Aesculus hippocastanum*) is a member of the Buckeye family. It is native to Asia and southeastern Europe and has been introduced to North America. Its wood is reputed to be used for Black Powder charcoal in Europe.

### **Jute Charcoal**

Jute is a plant that is used to produce fibers, just like hemp and sisal. Jute is grown in Asia and Africa. Jute sticks are reputed to make good Black Powder charcoal. Jute twine may be worth experimenting with.

### **Lime Charcoal**

Charcoal made from the wood Lime trees is mentioned in J.R. Partington's *A History of Greek Fire and Gunpowder*. Lime wood may be worth experimenting with. Other citrus trees such as orange, lemon, grapefruit, and tangerine could also merit investigation.

### **Madrone Charcoal**

The Pacific madrone (*Arbutus menziesii*) is reputed to have been used for making Black Powder charcoal.

### **Maple Charcoal**

Maple charcoal has been one of the charcoals of choice in commercial Black Powder manufactured in the USA. The last remaining commercial Black Powder manufacturer in the USA, GOEX, is reputed to use maple charcoal. So did their predecessor, Du Pont. Experiments done by Wilson<sup>3</sup> and O'Neill<sup>4</sup> have demonstrated that maple charcoal ranks among the fastest.

There are approximately a dozen different types of maple growing in North America, and many more worldwide. O'Neill<sup>4</sup> doesn't identify the particular type of maple in his experiments. Wilson<sup>3</sup> identifies his maple as silver maple (*Acer saccharinum*). This maple has a relatively soft wood when compared with other maples, and may account in part for its good performance.

There are nearly 150 species of maple worldwide. Most are found in eastern Asia. Some of these have been introduced into the USA. One such maple is the plane tree maple (*Acer pseudoplatanus*). This tree, a.k.a. sycamore, was regarded as a good source of charcoal by British powder makers.

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Does GOEX use maple charcoal? They won't say. They claim to use a blend of charcoals. This blend is mainly hard woods with a small percentage of soft woods [7].

Sasse [1] focused on maple charcoal. Even within the narrower confines of Sasse's research, notable variations in properties were found between charcoal samples. One example is the variation in volatile content of between 21 and 29%. Sasse concluded that the properties could vary even between different samples in the same lot obtained from a single supplier. He suggests that these variations are due to variations in the wood used and differences in temperature in different parts of the kiln. His proposed solution to this problem is to pre-blend such charcoal to obtain a more predictable Black Powder

### **Mesquite Charcoal**

Mesquite (*Prosopis glandulosa*) wood imparts a unique flavor to food. Some charcoal briquettes contain mesquite sawdust to enhance the flavor of food cooked over it. Another common practice is to add mesquite wood chips to a charcoal barbecue fire. Yet another option is to use charcoal made from mesquite wood.

Mesquite charcoal is relatively easy to get. Some supermarkets stock it. The better type is the straight wood charcoal rather than briquettes. O'Neill's<sup>4</sup> experiments showed that mesquite charcoal gave Black Powder with about half the power of the fastest powders. It also has a high ash content. Mesquite charcoal may be suitable for some, but don't use it if you want really fast powder.

### **Oak Charcoal**

Oak has been regarded as a hard wood by many, and thus avoided. However, experiments done by O'Neill<sup>4</sup> on one species of oak, *Quercus chrysolepis*, gave very good results. Charcoal from this oak ranked with some of the fastest tested. There are many species of oak throughout the world and different oak woods are bound to exhibit different results.

Charcoal used in Black Powder is reported to be made from varieties of oak in Britain and Australia.

### **Paulownia Charcoal**

Paulownia (*Paulownia tomentosa*) charcoal features quite prominently in the pyrotechnic literature and is frequently discussed among pyrotechnic enthusiasts. Some have rated it even better than willow charcoal in Black Powder. Shimizu<sup>6</sup> rates paulownia charcoal very highly as a Black Powder charcoal.

Paulownia charcoal ranked among the fastest Black Powder charcoals in O'Neill's<sup>4</sup> tests. Some pyrotechnic suppliers sell paulownia charcoal.

### **Pine Charcoal**

Pine charcoal has often been the focus of certain pyrotechnic spark effects, rather than a viable component of Black Powder. However, experiments by O'Neill<sup>4</sup> with a certain species of pine, *Pinus radiata*, yielded Black Powder that burned at about 80% of the rate of the fastest powders. This comes as a bit of a surprise, because traditionally pine has been shunned by aspiring Black Powder makers. Another surprise is that the species tested by O'Neill is classed as a *hardwood* pine.

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There are many species of pine throughout the world. North America alone has about 35 indigenous species plus a few that have been introduced from other countries. Pine wood is very popular as a cheap source of timber, and thus is easy to obtain. Pine should not be ignored.

Some pyrotechnic suppliers sell pine charcoal.

### **Plum Charcoal**

Plum (*Prunus domestica*) topped the list in O'Neill's<sup>4</sup> experiments. It gave the fastest powder and also had one of the lowest ash contents.

### **Poplar Charcoal**

Poplar is mentioned in some of the literature as being a good source of Black Powder charcoal. It is indeed, if one remembers that aspen and cottonwood are a type of poplar. Unfortunately the names aspen and cottonwood tend to obscure the fact that these are poplar trees under a different name. Poplars are related to willows, excellent sources of Black Powder charcoal.

### **Serviceberry Charcoal**

Serviceberry (*Amelanchier* Medic. Sp.) performed adequately in Wilson's<sup>3</sup> tests. Serviceberries are widely distributed across the temperate regions of the USA.

### **Sesban Charcoal**

Sesban (*sesbania sesban*) (a.k.a. Egyptian Rattle Pod, Suriminta, Soriminta) is found in Africa and Asia. It is reputed to give an excellent Black Powder charcoal.

### **Tamarind Charcoal**

The Tamarind Tree (*Tamarindus indica* LINN.) is found in India, Africa, and as a cultivated tree in the West Indies. Its wood is reputed to be an excellent source of Black Powder charcoal.

### **Teak Charcoal**

Teak (*Tectonia grandis*) performed adequately in Wilson's<sup>3</sup> tests.

### **Turmeric Charcoal**

I have found only one reference to turmeric charcoal. This related to a patented process for making a Black Powder substitute. What its alleged merits are, I don't know. Turmeric is a common household spice but making charcoal from it could be hideously expensive. Personally, I'd pass on this one.

### **Umbauba Charcoal**

The umbauba Tree (*Cecropiapalmata, peltata*) (a.k.a. embauba, imbauba, trumpet tree) is native to Brazil and is found in other parts of South America. Umbauba charcoal is the charcoal

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of choice in making Elephant Brand Black Powder[8]at their factory in Brazil. Again, here is an example of Black Powder makers finding a local source of charcoal that is both plentiful and cheap.

### **Charcoal Substitutes**

In my early days of pyrotechnic experimentation the schoolboy folklore of the day reckoned that ordinary sugar made a good substitute for charcoal. Some rather embellished stories went around about gunpowder that was actually white instead of black but still possessed remarkable explosive properties.

Personally, I don't think that any of those extravagant claims had any factual basis, being nothing more than wishful thinking. Closer to reality is the fact that sugar is a hydrocarbon that has proved successful in rocket fuel mixes. Typically these mixes use sugar and potassium nitrate melted and fused together. Another approach is to make a saturated solution of sugar and potassium nitrate.

However, sugar is not a good substitute for fast-burning Black Powder. This holds true for not only ordinary household sugar but other sugars such as lactose (milk sugar) and fructose (fruit sugar).

Why look for a substitute for charcoal? For some the answer may be that certain substitutes may be more easily obtainable than a specialty charcoal such as willow. However, a more important issue is repeatability. Charcoal, by its varying nature, suffers from a repeatability problem. So the real attraction is to find a chemical compound that works as well as charcoal but can be produced to more exacting repeatable specifications.

Charcoal substitutes have been investigated by researchers seeking Black Powder with characteristics that are more predictable. Notable is the research conducted by Wise, Sasse and Holmes who tested many different crystalline organic compounds. They concluded that some of these compounds showed promise as viable alternatives for charcoal. Their research, however, did not exhaustively test the properties of the powders produced and they concluded that such tests were necessary before claims of a viable charcoal substitute could be considered conclusive.

Additional research has been done by Weber. His tests focused on a process that used phenolphthalein as a charcoal substitute, with promising results. However, his findings were not subjected to all the test criteria outlined by Wise, Sasse and Holmes.

To date, no one has found a truly viable substitute for charcoal. Maybe they never will. The problem with charcoal is that it has both physical and chemical properties that influence its role in Black Powder. The searchers for substitutes have focused primarily on the chemical characteristics of charcoal.

### **The Quest for the Ultimate Charcoal**

If a suitable substitute cannot be found, the next best choice is a charcoal that is ideal, or close to ideal. Can such a charcoal be found? And what exactly is ideal?

With the many thousands of varieties of plants throughout the earth, it is indeed possible to find at least one with better properties than those in common use. But what is ideal?

Ideal may mean different things to different persons. Some may regard the fastest burning

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charcoal in existence as ideal. Others may opt for consistency in performance from one batch to another. Those looking for speed would often be quite happy to have a charcoal with good consistency. The converse may not necessarily be true.

Because charcoal is an organic substance made from plants, and because it possesses both chemical and physical properties the variables out there affecting these are quite extensive in scope. For a plant (be it a tree, shrub, grass or vine) to yield a charcoal with consistent performance, the following factors may come into play:

- The age of the plant
- Soil conditions
- Environmental conditions
- Seasonal changes.

Those variables pertain to the living plant. Once the plant has been harvested for charcoal making, the following considerations come into play:

- The age of the harvested wood.
- The method used to carbonize the wood.
- The temperature(s) at which the wood is carbonized.
- Storage and treatment of the resulting charcoal.

**The age of the harvested wood** could influence the resulting charcoal. Chemical changes do take place in wood during its drying process. Barbecue and smoker enthusiasts will tell one that the wood used to give *that special flavor* should be dried but not dried too long. Wood that is too green may also compromise the carbonization process, resulting in improperly or unevenly charred wood.

**The method used to carbonize the wood** is very critical. And herein lies the greatest problem. While some may postulate grandiose schemes that would ensure consistency in heating the wood and ensuring proper destructive distillation, such postulations rarely translate into the viable real world.

Recently I worked on a kiln that was used to reactivate charcoal. The goal here was to bring charcoal back to its activated state after it had been used to extract gold from cyanide solution. The process was basically a simple one. The charcoal (minus its precious gold) was heated in a rotating gas-fired kiln.

The feed into the kiln was carefully controlled, as was the rotation speed of the kiln. The temperature of the kiln was controlled and monitored in two separate heating zones. The pressure difference between the kiln stack and atmospheric pressure was monitored by very sensitive pressure transducers that connected to an electronic controller. The kiln itself was carefully sealed so that air from outside would not compromise the process.

The kiln's parameters were recorded, studied, and analyzed by a metallurgist for several months. Control parameters were tweaked, analyzed, and tweaked again, many times. The performance of the kiln did improve. But it was a constant challenge to make it perform in a consistent manner. Similar challenges await others.

The point that I am making is that any kiln/retort system that aims for a high degree of consistency in finished product is quite a challenge. Even under laboratory conditions, the challenges are still there.

**The temperature(s) at which the wood is carbonized** plays a very critical role in the charcoal's performance.

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Temperature is the most commonly controlled parameter in the process industries, and also in the home. It can be very easy to control or very challenging. Temperature controllers can range from the humble thermostat, invented more than a century ago, to sophisticated microcomputer-based electronic controllers.

One of the greatest challenges in charcoal production is to ensure consistent heating throughout the retort. Assuming the retort is the type where the vessel is heated from outside, the biggest challenge is to ensure that the wood in the center of the retort reaches the correct temperature while the wood closest to the sides of the vessel are not overheated. Heat must thus be applied and regulated in a carefully controlled manner.

To apply heat without overheating the sides of the vessel requires that the vessel be either rotated if the heat source is in one spot or the heat be applied evenly around the outer perimeter of the vessel. To ensure that temperature does not overshoot its desired value, a sensor (such as a thermocouple) needs to be mounted inside the vessel right next to its wall. Another sensor needs to be mounted in the center of the vessel to determine when the center is hot enough.

**Storage and treatment of the resulting charcoal** is important to ensure that the performance differences between freshly made charcoal and stored charcoal are kept to a minimum. Charcoal can adsorb gases from the atmosphere. This means that it can (among other things) attract oxygen and water vapor. Both of these influence its performance.

Personally, I doubt that even those equipped with proper laboratory equipment could do all that is necessary to control the above variables. Amateurs have even less chance. And controlling the above is just the beginning. The next challenge is to accurately measure both the chemical and physical properties of the charcoal. And even if that could be done, arriving at a common consensus relating to what should be measured and how could take forever.

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## Chapter 5 -- Charcoal factor II

### Introduction

This chapter is about making charcoal. Here we explore how charcoal has been made over the centuries, culminating in the highly efficient processes that have been developed over the last century. And, most important of all, we explore ways and means of making one's own charcoal.

I was tempted to leave out information about traditional methods and focus just on small retort processes. I changed my mind while perusing much of the literature on charcoal, including the pyrotechnic literature. Most of this literature focuses, to a fault, on the older traditional methods while virtually ignoring modern charcoal-making processes. So, in going with the flow, I have described the older processes. But I do have different agenda - to promote retort processes and to discourage would-be charcoal makers from trying variations on the older methods.

To some my focus on the retort method may seem a little narrow-minded. But I have done this for two basic reasons:

- The retort process has a track record of yielding better Black Powder charcoal.
- Retorts are easy to operate with minimal skills.

The discussions in this chapter begin with the basic traditional methods of making charcoal. More advanced methods are then dealt with. Finally both simple and more sophisticated ways of making one's own charcoal are discussed. And just a word of encouragement to those who may find some of these discussions rather tedious: charcoal making is easy - and it can be a lot of fun!

### Charcoal Pits

Charcoal, like compost, has been made either in pits or in heaps. This section describes pit charcoal. A charcoal pit is also described as an *earth kiln* because earth is used to seal out air. Pits may be dug in flat ground or on a slope.

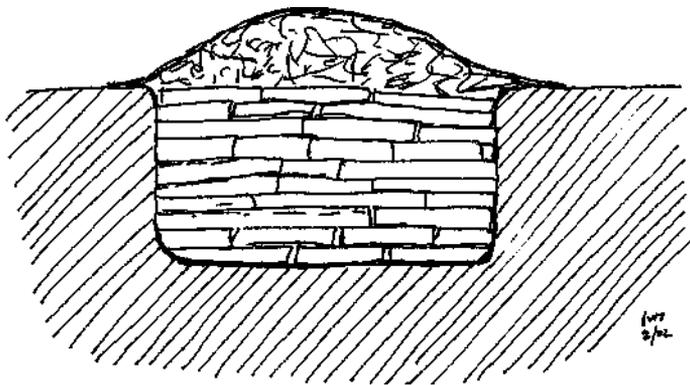


Figure 5-1. Charcoal Earth kiln on level ground

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Basically a charcoal pit is a hole dug in the ground that has been filled with burning wood and covered with earth to keep the air out. Pits may range in capacity from less than 30 cubic feet to six hundred to a thousand cubic feet.

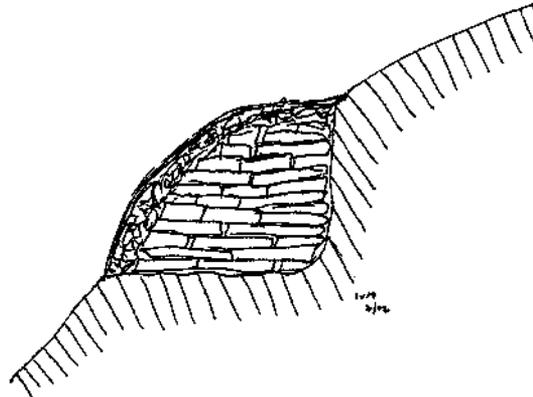


Figure 5-2. Charcoal Earth Kiln on Sloping Ground

In the smallest pits charcoal is made by starting a fire at the bottom of the pit and then adding wood until the pit is full. This wood is then covered with leaves, grass and finally with earth. The carbonization takes about two days.

Larger pits are first filled with wood and then fired in the center, where an open space going right to the bottom of the pit is filled with combustible material. The wood is then allowed to burn well before it is covered with vegetation and earth. Air vents are made in the covering to control carbonization. Adjustments to the carbonization rate are made by opening or closing these air vents. The whole cycle, including cooling can take up to a week.

The largest pits have an air inlet on one side and a smoke outlet on the other. The bottom layer of wood is loosely piled lengthwise to allow air flow from one end to the other. Alternate layers of crosswise and lengthwise wood are piled on top of this first layer. Unlike the first layer, these layers are densely packed. The wood pile is then sealed with vegetation and earth. The fire is started at the inlet side, and slowly burns its way to the outlet.

Carbonization may take up to a whole month in the largest pits and cooling a month or longer.

Charcoal pits can be very dangerous as persons may step on them, not realizing that there is hot material under their feet. The most dangerous time is during the cooling cycle when no smoke or vapors are given off, thus giving no warning.

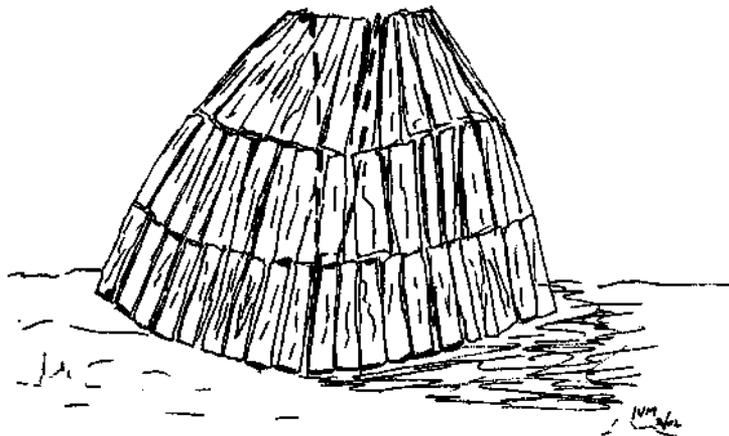
While charcoal pits are easy to construct they suffer from the following disadvantages:

- Carbonization is difficult to control.
- The charcoal yield is low.
- Pits are not suited for very small scale production.
- Pits cannot be used during the rainy season.
- Charcoal may end up being contaminated with other matter such as earth.

## **Charcoal Mounds**

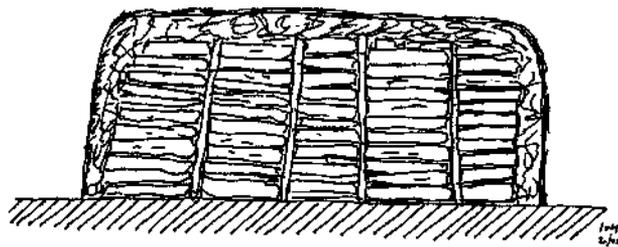
Mound charcoal is made in a similar way to pit charcoal, but instead of burying the wood, the wood is formed above ground into a heap or mound. It has the advantage over pit charcoal in that it does not require the extra labor of digging a pit. Its downside is its requirement for more sealing material. Charcoal mounds, like their pit counterparts, are described as *earth kilns*.

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*Figure 5-3. Traditional circular mound kiln*

Charcoal mounds are less constrained by rainy weather than charcoal pits. Airflow is easier to control in mounds, thus making it easier to regulate the carbonization. Over the centuries charcoal mounds have been preferred to pits. Mounds are made either in rectangular shapes or round shapes, the latter being more prevalent.



*Figure 5-4. Rectangular Mound Kiln*

Circular mounds typically have capacities ranging from five hundred to two thousand cubic feet.

Construction of the mound begins with one or more stakes being planted vertically in the ground. Wood is then stacked vertically layer by layer against these stakes. When the mound is completely stacked, the center stake is removed and the space left by it forms a chimney. The mound is then covered with vegetation and earth and fired in the center by hot charcoal and kindling material.

Carbonization is typically controlled by opening air holes around the mound and smoke holes on the top. Mounds covered with other substances such as sawdust or rice husks instead of earth may not need air and smoke holes. The whole charcoal-making cycle usually varies from about one week to a month, depending on the size of the mound. However, this time can be as short as one day with very small mounds containing about 15 cubic feet of wood.

The above describes a simple charcoal mound kiln. More advanced mound kilns have a more sophisticated ventilation system, giving better results.

While charcoal mounds are better than pits, they still suffer from the following shortcomings:

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- Mounds are not suited for very small scale production.
- Mound charcoal may end up being contaminated with other matter such as earth.

## **Charcoal Hard-Walled Kilns**

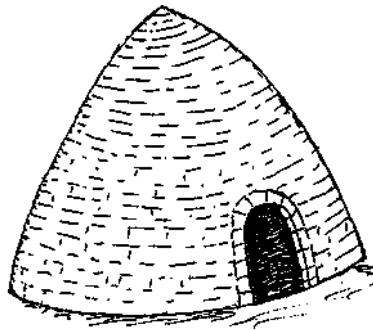
Although charcoal pits and mounds are referred to as kilns, one normally thinks of a kiln as being a more permanent structure. Indeed, some have been permanent enough to have lasted for centuries. Charcoal pits and mounds earn the title of kiln from their temporary covering of earth and vegetation. Permanent kilns replace this with a permanent hard shell of stone, clay, brick, concrete or metal.

Although most of these kilns are truly permanent, some could more accurately be described as semi-permanent. These are brick kilns that are dismantled and re-erected elsewhere. Here the kiln is constructed without mortar to bind the bricks together, and erection, dismantling and re-erecting the kiln can be done in a short space of time by skilled workers.

There is some debate as to the difference between a kiln and a retort. This subject is discussed in detail in the retorts section of this chapter.

Hard-walled kilns have varied tremendously in size. Some small clay kilns look like small clay domestic ovens, both in shape and in size. Other, more modern kilns are like large storage sheds. These are loaded by logging trucks driving right into them.

Hard-walled kilns offered major advantages over their predecessors. They could be loaded and fired up more quickly. They also were less inclined to contaminate the charcoal with other substances such as earth and clay. Hard walls were also less likely to break and let unwanted air into the kiln.



*Figure 5-5. Beehive kiln*

## **Charcoal Brick Kilns**

Kilns made of bricks have achieved world-wide popularity. A popular design for such kilns has been in the shape of a beehive. Such kilns are found worldwide. In the USA many examples of beehive kilns still exist, reminders of past thriving charcoal industries. Typically, such kilns were made of bricks. Some of these were made with such fine craftsmanship that no mortar was used in their construction.

## BLACK POWDER MANUFACTURE, TESTING & OPTIMIZING

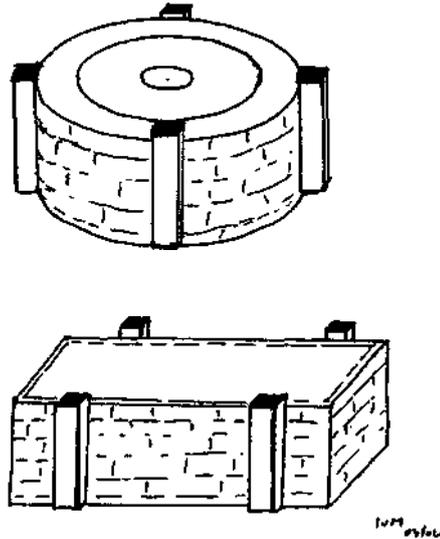


Figure 5-6. Brick kilns

To work successfully, a brick kiln needs to meet the following requirements:

- easy to construct
- withstand thermal stresses of heating and cooling
- withstand mechanical stresses of loading and unloading
- be weather resistant, especially to rain.

Brick kilns have been around for centuries, but have undergone certain improvements in the last century. Of note are:

- Brazilian beehive kilns
- Argentine half-orange kilns
- European Schwartz kilns
- USA Missouri kilns.

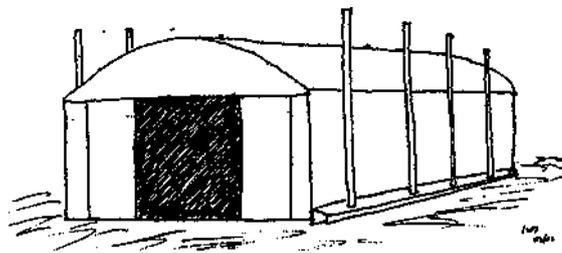


Figure 5-7. Large modern Missouri type kiln

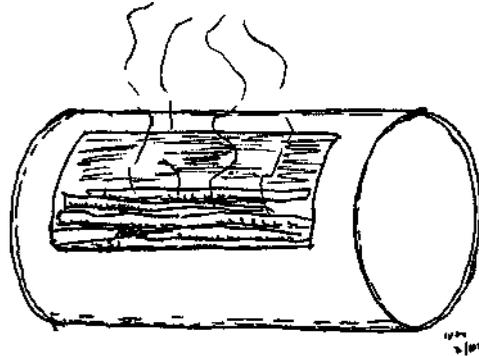
The European Schwartz kiln is unique in that it uses hot flue gases from an external fire grate, so it is similar to a retort in its operation. The other three types work on the traditional principle of lighting a fire within the kiln and then starving the fire of oxygen.

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## **Drum Charcoal**

In many developing countries small-scale production of charcoal is done in drums. Typically, these are standard 33, 44, or 55 gallon drums that have been modified for charcoal making.

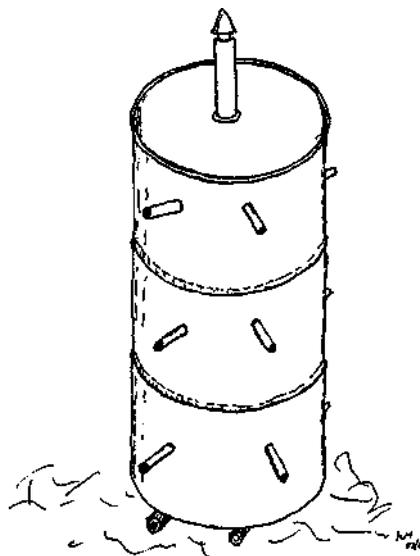
Charcoal made in drums can either follow the traditional kiln method whereby a fire is started in the drum and then starved of oxygen or the retort method where a sealed drum containing the wood is heated by an external heat source. This section discusses the former, leaving the latter to the discussion under retorts.



*Figure 5-8. Horizontal drum*

Drums can be used either horizontally or vertically. One way of using a drum is to cut a door in the side of the drum and fill the drum with wood. The wood is then ignited and the door replaced. To ensure proper sealing, the drum is then rolled until the door is underneath. The sealing can be improved by packing earth around the drum.

When drums are used vertically, a fire is typically started in the bottom and wood is fed from the top until the drum is filled. A lid is then placed on top. This lid normally has some kind of chimney to let the gases escape. Air vents on the side of the drum are progressively opened and shut until all the wood is ignited. At this point, most of the vents are shut, allowing only a minimal amount of air into the drum.



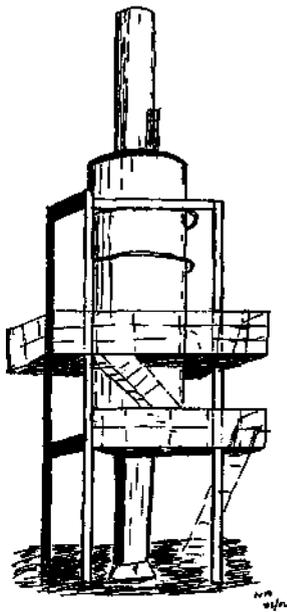
*Figure 5-9. Vertical Drum*

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Drum methods are finding increasing favor with do-it-yourself enthusiasts who are tired of commercially made charcoal and want to make their own. In some developing countries whole charcoal industries exist that make charcoal exclusively in drums. A large amount of coconut charcoal is produced this way.

## **Modern Charcoal Retorts**

Modern charcoal retorts come in many shapes and sizes. The 20<sup>th</sup> century brought us a whole lot of innovative charcoal retort designs that sought to both optimize charcoal production and its by-products. A whole book would be inadequate to describe these modern innovations in detail. The treatment given to them here could be best described as superficial.



*Figure 5-10. Industrial Retort*

Charcoal retort processes can be categorized into two distinct categories:

- batch processes
- continuous processes.

**Batch processes** describe charcoal that is made in distinct batches. Here sufficient wood for a single batch is loaded into the retort, which is then heated until the wood is carbonized. The carbonized wood is then cooled and removed from the retort. This process is then repeated, each process yielding a finite amount of charcoal.

**Continuous processes** continuously feed wood into one side of the retort and remove charcoal from the other. Continuous processing is the preferred method for large-scale production. This is true not only for charcoal production, but for many other industrial processes.

The larger manufacturers of charcoal normally opt for a continuous process, while smaller suppliers may prefer a batch system. Batch systems are usually a lot cheaper. Home-made charcoal is always made in batches. It makes no sense to try and make a small-scale continuous system. Practically speaking, it may not be possible to make a small retort that has a continuous throughput.

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## **Making Good Charcoal**

This section focuses on making good charcoal. Here *good* may also be interpreted as *good enough*. Good enough implies not necessarily the best. It also does not relate to charcoal that has been optimized for Black Powder making.

Making good charcoal is easy and cheap. Optimized charcoal is more difficult and maybe a lot more expensive to make. Good charcoal is within the reach of anyone. Optimized charcoal is not. For the few purists and fanatics, optimized charcoal is described elsewhere in this chapter

Charcoal has been made for centuries, using many and varied methods. Some of the pyrotechnic literature gets rather sidetracked here, describing large-scale production methods that are basically meaningless to the small-scale user. Such methods are, more often than not, unsuited for good Black Powder charcoal. Some of these methods are, however, described here. The reason for this is to demonstrate some of their drawbacks and to point potential charcoal makers to more suitable methods. Although such methods may yield acceptable charcoal, recent history has shown that there is only one good method to make good Black Powder charcoal. This method is known as *the retort method*.

## **The Retort Method - An Overview**

Right until the end of the 18<sup>th</sup> century charcoal used in Black Powder was made using old traditional methods that resulted in charcoal that was far from optimal. That is to say, far from optimal so far as Black Powder manufacture concerned. Charcoal making then took a big step forward with an invention by an Englishman, Richard Watson (a.k.a. Bishop Watson)<sup>1</sup>. His method used metal cylinders that were filled with wood and sealed prior to heating. Ballistic tests on Black Powder made with this charcoal showed an increase in range of about 60%. Watson's invention is today known as *the retort method*.

Just what is the retort method? The retort method is a method whereby wood or other charcoal-yielding substances are placed in a sealed container and heated. The retort method is thus different than traditional methods that have built piles of charcoal-yielding materials or placed such materials in pits or kilns. These latter two methods involved actually igniting the charcoal-producing materials. The retort method usually does not. And I say *usually* because some retort methods work in a similar fashion to the traditional pits, piles and kilns.

Another source of confusion is that some retorts heat the wood directly, rather than by heating the outside of the retort. This is accomplished by passing hot gases through the wood. So not all retorts are created equal and one may justifiably argue that some retorts should be called *kilns*, rather than retorts. However, from a Black Powder maker's perspective, the retort method needs to be clearly and narrowly defined, having the following characteristics:

- The heat used is generated from an external source.
- The amount of oxygen that comes into contact with the wood is reduced to an absolute minimum.

The above characteristics are essential to our understanding of where the retort method differs from other charcoal making methods. Any method of charcoal manufacture that relies on igniting the wood that yields the charcoal cannot be considered as a *retort method* according to the above definition. Such methods use an *internal* rather than an *external* heat source. An internal heat source requires a certain amount of oxygen to keep it going.

Speaking in more scientific terminology, the retort method relies on an *endothermic* (heat ab-

## BLACK POWDER MANUFACTURE, TESTING & OPTIMIZING

sorbing) reaction, while other methods use an *exothermic* (heat generating) reaction. The latter method has been the method of choice for charcoal makers over the centuries for one major reason: it is more economically viable to produce charcoal this way. Unfortunately centuries of tradition have created a bias in the way that many look at charcoal manufacture. Thus any method that uses a sealed container is wrongly presented as a retort method.

So a true retort method (from a Black Powder perspective) relies on an external source of heat and a method whereby oxygen in contact with the wood is minimized. This can be achieved by tightly packing the wood in a sealed container and heating it from the outside. Another more difficult-to-achieve method is to pass hot oxygen-depleted gases through a container packed with wood. This method is better suited to large industrial-scale charcoal manufacture.

The retort method offers some major advantages over other methods because such a method:

- produces charcoal free from contaminants such as mud
- chars the material more consistently
- carbonizes the wood faster
- tends to create less ash
- gives better control over volatile content
- is more environmentally friendly.

Another advantage of the retort method is that it can be used in both small-scale and large-scale production. So one can use this method to make batches weighing just a few ounces to batches weighing many pounds. This is really good news to the person who only needs a little charcoal or who only has a small quantity of the required wood from which to make the charcoal. Another piece of good news is that simple retorts don't require the skills and supervision that the other methods do.

The retort method also has a spin-off in terms of other substances that can be extracted during the charcoal-making process. The process of heating the wood causes certain liquid substances contained in the wood to be boiled off in a process known as destructive distillation. Acetic acid, acetone and wood alcohol (methyl alcohol or methanol) used to be obtained this way.

In practice, the modern charcoal maker often has no use for these by-products and makes no effort to collect them. Also markets have changed over the years, making it less economically viable to retrieve certain by-products. Methyl alcohol, for example, is now made more efficiently using other means.

Does the retort method have any disadvantages? It may in terms of fuel efficiency by requiring more fuel to produce a given amount of charcoal. This could pose a setback to those programs in developing countries that try to promote charcoal as an economic source of fuel. It also creates some financial challenges to those making charcoal for domestic or industrial purposes. Charcoal for Black Powder use, however, is generally not inhibited by these constraints.

A retort can be made from just about anything that can be sealed and withstand high temperatures. Choices can range from a small coffee can to a large drum with a capacity of hundreds of gallons. These larger retort systems often have complex control systems to ensure good temperature and feed control. Some of these systems attempt to optimize energy use by burning the gases given off by the heated wood.

While larger charcoal retorts offer a fascinating study in themselves, and are discussed in this chapter, there is little to be gained by trying to emulate them on a smaller scale. So small is beautiful here. How small depends on the needs and resources of the person needing the charcoal.

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Popular choices for small-scale charcoal production have been 33, 44 or 55 gallon drums. Here *small-scale* is a relative term. Typical small-scale Black Powder production requires far less charcoal than the amount held in such drums. Here the upper limit is usually in the order of about 5 gallons, with the lower limits being set by the smallest size can that has a replaceable heat-resistant lid.

In choosing the container size that best suits one's needs, it is important to remember that the container must be filled with wood. Half-filled containers will just not work properly because they allow too much space for air. Another constraint in container size relates to what can be heated properly with the system available. Larger containers also typically take longer to reach operating temperature. So it's best to start off small and then go to bigger containers if one has to.

### **Finding the Right Wood**

Finding the right wood may present an even bigger challenge than converting the wood into charcoal. The previous chapter described woods from different trees that are suitable for Black Powder charcoal. Those of us who live in areas where one or another of such species grows could quite easily obtain at least a few small branches to experiment with. The rest of us need to look to other resources.

Looking back to the time I was still living with my parents, I wish I knew then what I know now. Then I didn't realize the importance of charcoal in Black Powder. If I had, I would have been able to experiment with a whole world of woods. On the edge of our suburb was a stream where weeping willow abounded. And although our own backyard did not have weeping willows, it had a pussy willow. This was more of a shrub than a tree, but a member of the willow family nevertheless. We also had plenty of other trees.

We lived on a half-acre plot that used to be part of a peach orchard. In our garden, besides different varieties of peach, we had plum, pomegranate, quince, almond, mulberry, blackberry, apricot, nectarine, crab apple, lemon, and gooseberry. My aunt next door had some grape vines, fig, avocado, and different types of mulberry. Other neighbors had cherry trees. And these were just the fruit trees.

We also had different varieties of acacia, cabbage tree, white stinkwood (a type of elm), syringa, and honeysuckle. (The latter two, I've been told, make excellent Black Powder charcoal). Added to that were a whole variety of rose bushes, some very large, almost trees. And our sidewalk had a whole bunch of jacaranda trees.

My dad had a passion for pruning. He even got into trouble with my grandmother when he pruned her beloved almond trees. She believed that one should never prune an almond tree, but he reckoned that it had gotten too untidy and did. So I would have had plenty of stuff to play with, if only I knew.

Another issue I learned about at that time was that fruit trees are difficult-to-define kind of beasts. One could grow two or three different types of peaches on the same tree by using such techniques as grafting and budding. One could also do this with other fruits. I learned later that some grape vines were habitually grafted onto roots from another type of vine that was more disease-resistant.

Most of us are not nearly so fortunate to live right in the middle of a whole lot of trees that may yield a great charcoal. And, I myself, am now a member of this less fortunate majority. I only have a few small trees in my garden. So to get wood suitable for making charcoal I, and most others, have the following choices:

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- Venture outdoors in search of the right kind of trees and then make a plan to obtain wood from them.
- Purchase the desired wood from a number of sources.

Before venturing outdoors in search of trees, it's a good idea to research the type of trees one can expect to find in one's own neck of the woods. One should then get some kind of field reference to enable one to identify the trees in question. Generally trees and shrubs are more easy to identify in summer than in winter. The spring and summer seasons put leaves, flowers, and fruit on the trees, making identification that much easier.

Just some words of caution. Generally forget about chopping down trees on land that does not belong to you. One usually has to settle for branches, and normally dead ones at that. On many properties one is forbidden to collect firewood, and collecting wood for any reason may be taken the wrong way. But one may be lucky enough to get permission to take just a few dead branches. It is worthwhile to ask, as the *firewood* regulations are often designed to discourage campers from denuding areas around their campsites.

Some woods such as pine, come ready cut as lumber and are quite easy to obtain. These may not be the best, but could offer a start to the charcoal maker. However, just a word of caution. Lumber is often treated chemically to make it more resistant to the weather or insects. This treatment could be bad news, so be sure you have untreated wood.

While most barbecue enthusiasts use charcoal, some prefer wood or a mixture of wood and charcoal. So there is a market out there for those who prefer the flavors imparted by particular woods and there are suppliers who are willing to meet their needs. At the moment, these suppliers supply mainly restaurants but that is already changing. One such supplier in the USA is the Lazzari Fuel Company. They offer the following woods:

- Almond
- Apple
- Alder
- Cherry
- Hickory
- Mesquite
- Oak
- Walnut

Their hickory and mesquite are the most popular and can be found on supermarket shelves. The others one may have order. No, they don't supply willow, but some of the fruit tree charcoal may be worth experimenting with. Lazzari also supply oak, hickory and mesquite lump charcoal.

While *Lazzari* is a prominent supplier, there are many other vendors of smoking and cooking woods. The best way to find these is to search for them on the Internet. There are plenty of them out there, and their numbers appear to be increasing. Some may only offer two or three types of wood, others a lot more.

The barbecue/smoker community have tested a number of different woods and have decided upon those that are desirable as follows:

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- Acacia
- Alder
- Almond
- Apple
- Apricot
- Ash
- Birch
- Cherry
- Cottonwood
- Crab Apple
- Grape Vine
- Grapefruit
- Hickory
- Lemon
- Lilac
- Maple
- Mesquite
- Mulberry
- Nectarine
- Oak (Red Oak, White Oak)
- Olive
- Orange
- Peach
- Pear
- Pecan
- Plum
- Walnut (English Walnut, Black Walnut)
- Willow.

Not all of the above are suitable for Black Powder of course. And wood from one supplier is bound to have different characteristics than the same wood from a different supplier. It's a matter of pot luck here, but could still be worth pursuing.

## **Preparing the Wood**

The two most hotly debated topics about preparing wood for charcoal are:

- wet or dry
- bark or de-barked.

Wet wood here refers to wood that has been freshly removed from the tree, while dry wood refers to wood that has been left to dry for a period of time. Naturally, wet wood requires more energy to produce charcoal as the wood has to be dried before it can be carbonized. This is possibly the major reason why wood is often left to dry before it is carbonized. Another factor is the chemical make-up of wood and charcoal. Undoubtedly the chemical make-up of dried wood is somewhat different to that of wet. Whether there are significant differences in the resulting charcoal is debatable. The bottom line is that charcoal made from either wet or dry wood works in Black Powder.

Charcoal made from wood with the bark removed works in Black Powder. So does charcoal made from wood with its bark. But to get the best results from a particular wood, it is wise to remove the bark for a number of reasons:

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- The bark is on the outside of the tree and is not shielded from the environment. Thus it can collect dust, sand, bird droppings, pesticides, and maybe a whole lot of other things.
- The physical characteristics of the bark are different to those of the rest of the wood.
- Bark contains chemicals not found in the rest of the wood. Some of these, such as silicon, may be detrimental to the charcoal's performance.

### **Inner or Outer?**

Another subject of debate is whether one should use wood from the outer part of the tree (nearest the bark) or the wood from the inner part (a.k.a. the heart wood).

The general consensus of opinion is that wood from the outer parts is softer and burns faster. Conversely the heart wood is harder and slower burning. Thus the outer wood is preferred by some. In practice this issue relates more to wood obtained from the trunk of the tree than its branches. And often wood from the trunk is not available anyway.

Wood used in the past to make Black Powder was often obtained from young branches of trees that were cut off the tree, or coppiced, specifically for charcoal manufacture. This practice of coppicing was done in such a way as to promote the re-growth of the young branches, ensuring a renewal source of wood. Often trees that were amenable to coppicing were grown in preference to others.

### **Optimized Charcoal**

This discussion is about charcoal that is optimized for use in Black Powder. The term optimized may mean something quite different if one requires charcoal for other purposes, for example, activated charcoal used for adsorption of chemical substances.

Optimizing charcoal for Black Powder is largely concerned with controlling the amount of volatiles left in the charcoal after charring. An optimal amount of volatiles produces an optimal-burning rate. How does one control this volatile content? One does this by carefully controlling temperature.

Temperature is measured and controlled more frequently than any other parameter such as pressure, flow or level. Temperature can be measured accurately in many and varied ways, often quite cheaply. Temperature is often easier to measure than other parameters.

The chapter on testing describes thermocouples and resistance temperature detectors (RTDs) used to accurately measure temperature. These devices do a very good job but do require additional electronic devices to display their temperature readings. To get very accurate readings, thermocouples or RTDs are recommended. Less exacting requirements can use simple mechanical dial thermometers. These are less accurate than thermocouples and RTDs and usually take longer to respond to temperature changes. Another disadvantage is they cannot be read remotely.

Dial thermometers often come with an integral probe attached. Others are made with a bulb (which acts as the sensor) attached to a capillary tube.

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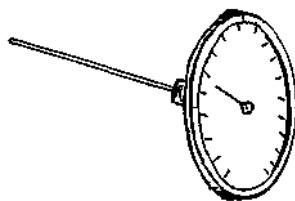


Figure 5-11. Dial Thermometer

Dial thermometers vary in price from about 10 US dollars to a few hundred US dollars. It doesn't make sense buying an expensive dial thermometer to make charcoal. Cheaper types should perform adequately. I do not recommend the use of an ordinary mercury-in-glass thermometer. Most of those sold will not be able to read high enough temperatures anyway. Those that do should be considered too fragile for the particular application.

Now it's not enough to just measure the temperature to ensure optimal charcoal. One needs to control it. In a typical industrial plant, temperature is controlled by one of two methods:

- manual control
- automatic control.

**Manual control** is performed by a person manually adjusting the heat source. This can be done by making just one adjustment and then leaving the heat source alone, or by constantly monitoring the temperature and either increasing or decreasing the heat accordingly. The first method is known as *open loop* control, the second method as *closed loop* control. Some refer to manual control as being open loop control and automatic control as being closed loop. This is not strictly correct. Human intervention that compensates for differences in actual and desired temperatures is in fact *closing the loop*.

Manual control is less expensive to implement than automatic control, but requires frequent or constant human supervision to get acceptable results. Generally, it is also less accurate than automatic control.

**Automatic control** is performed by either a mechanical or electronic device that measures the temperature and compares it with the desired value, also known as the set point value. If the temperature is less than the set point, heat is applied or increased; if it is greater, heat is removed or decreased. A simple thermostat is a type of automatic temperature controller.

Increasing or decreasing the heat requires the automatic controller to either turn the heat source on and off or proportionally regulate it. The former method is often preferred as it is cheaper and easier to implement. Thermostats are usually on-off devices.

Whether one's choice is manual or automatic control, the system used must be controllable. That means that the heat source needs to be able to be varied. Here a wood or charcoal fire is unsuited as a heat source. Viable alternatives are electricity or gas. A camping gas stove or a portable electric hot plate are good choices for small retorts.

## Optimization Strategies

In the great big real world temperature control can require nothing more than a simple thermostat in certain applications. Other applications may need nothing less than a sophisticated computerized control system. Optimized charcoal making needs a temperature control strategy. This strategy can be relatively simple in small-scale charcoal making. The same may not be true for large-scale industrial retorts, but these are not our concern.

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The biggest challenge is to heat all of the wood to within the required temperature range. This means that one should ensure that the wood closest to the heat source is not overheated, while the wood furthest away is heated to a high enough temperature. Generally this requires a slow heating process. Here low to moderate heat is applied to the walls of the container holding the wood. This heat is monitored just inside the walls of the container and also in the center of the stack of wood.

Two temperature probes are essential to this heating strategy. The outer probe is needed to ensure the applied heat is within the correct range. The inner probe is needed to ensure that the wood in the center comes up to temperature properly. Note that proper placement of the probes is important in any temperature control application. This application is no exception.

The tips of both probes should be at points close to a plane running through the center of the container. The outer probe should be placed close to the wall of the container but should not touch the wall itself.

Small containers envisaged thus far have generally been thin walled containers such as metal cans used for coffee, food or paint. These have the advantage of being easy to obtain. They also cost nothing or close to nothing. However, they do suffer from one disadvantage - the thickness (or lack of it) of their walls.

Thin-walled containers distribute heat less evenly than their thick-walled counterparts. This is one reason why thick-walled cooking utensils are preferred in certain cooking applications. So thick-walled containers in charcoal making are better where consistency is aimed at in each batch. This wall thickness is even more critical where heat can only be applied at one point such as the bottom of the container.

## **References**

- 1) J.R. Partington, A History of Greek Fire and Gunpowder, The Johns Hopkins University Press,(1999)

## *Chapter 6 - Tuning for Speed - More Bang for Your*

### **Introduction**

In chapter three we looked at crude methods of making Black Powder. These were basically entry level methods. These gave one an introduction to making Black Powder, but hardly yielded a Black Powder that was usable in the normal sense.

Crudely made Black Powder burns very slowly. This slow burning rate makes it unsuitable for use as an explosive or a propellant. It also leaves a horrible residue of unburned materials. This chapter describes ways of improving Black Powder production, giving powders that burn faster and cleaner. Later chapters will describe even better methods.

### **Focusing on Charcoal**

My early attempts at making Black Powder used whatever charcoal I had available. Usually this was barbecue charcoal. I didn't realize at the time how critical charcoal was.

Of the three basic ingredients in Black Powder, charcoal is the most variable. The choice of charcoal can make or break a powder's performance. So the first thing one must look at when one wants to improve the speed of one's Black Powder is the choice of charcoal. Other considerations are secondary.

The last two chapters discussed charcoal in depth, describing its importance, its desirable properties, and how to make it. If making one's own charcoal is the only option in obtaining good charcoal, then one should focus on this before going on to improved methods and techniques of making Black Powder.

My own tests, and tests done by others have shown that speed increases of between 100% to 500% are possible when better choices of charcoal are made. These improvements are significant, and can well make the difference between an unusable and a usable Black Powder.

### **Focusing on the Other Ingredients**

While charcoal can have the largest influence on a Black Powder's efficiency, the other two ingredients play their part as well. It pays to use potassium nitrate of acceptable purity. The same is true of sulfur, although to a lesser extent.

Personally, I don't think it is wise to start out making Black Powder with agricultural grade materials. These are better left till later, when one has gotten to the stage of producing good burning powders. When one has reached this point, it could be a worthwhile exercise to start experimenting with agricultural grade materials. Why do I say this? Simply because the less pure agricultural grade materials could introduce factors that could inhibit the burn properties. These could sound a note of discouragement when it is least needed.

# BLACK POWDER MANUFACTURE, TESTING & OPTIMIZING

## **Fiddling with the Formula**

In my early attempts to make Black Powder I had some very disappointing failures. But through it all I consoled myself with the thought: "At least I've got the right formula!"

I felt quite smug about it. I had read about Black Powder in encyclopedias and other books. I knew most of my friends were wrong, and I was right. They were messing around with equal quantities of the three ingredients. And they were doing it by volume! I weighed my ingredients according to the right proportions, which were:

- potassium nitrate 15 parts (or 75%)
- sulfur 2 parts (or 10%)
- charcoal 3 parts (or 15%)

So I was sure that I had got this part right. I didn't know it at the time -I was wrong.

The formula I used was the most well known formula for Black Powder. It is commonly referred to as the Waltham Abbey formula. Waltham Abbey was the place where the Royal Gunpowder Works in Britain was situated. Today the old powder works have been turned into a museum.

Waltham Abbey became a center of influence in the Black Powder world. At one point in history they decided on a standard formula for Black Powder, the one shown above. Since this time, this formula has become the most well known and most popular formula for Black Powder.

The Waltham Abbey formula is pretty close to what many manufacturers were using at the time anyway. Over the centuries, improved manufacturing methods and plenty of experimentation had shown that ratios approximating the 15:2:3 were optimal for powders used in propellants. My wrong assumption came about by not properly understanding this particular part of history.

I had assumed that the 15:2:3 formula had evolved mainly from better science based on better experimentation. In fact, better manufacturing methods played a very important part. So the Waltham Abbey formula is very good for good manufacturing methods. It may not be as good for methods that are crude. Also it may not necessarily be optimal for all types of charcoal.

Some of the earliest formulas (14<sup>th</sup> century) used the following ratios:

- potassium nitrate 6 parts (or 66.6%)
- sulfur 1 part (or 11.1%)
- charcoal 2 parts (or 22.2%)

A similar ratio to the one above has been used right until the end of the 20<sup>th</sup> century. Its formula is:

- potassium nitrate 6 parts (or 75%)
- sulfur 1 parts (or 12.5%)
- charcoal 1 parts (or 12.5%)

Another popular ratio during the 14<sup>th</sup> to 17<sup>th</sup> centuries was:

- potassium nitrate 4 parts (or 66.6%)
- sulfur 1 parts (or 16.7%)
- charcoal 1 parts (or 16.7%)

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A formula attributed to Whitehorn in the 16<sup>th</sup> century was:

- potassium nitrate 5 parts (or 62.5%)
- sulfur 1 parts (or 12.5%)
- charcoal 2 parts (or 25%)

The Spanish Conquistadors are reputed to have used a similar ratio of:

- potassium nitrate 5 parts (or 71.4%)
- sulfur 1 parts (or 14.3%)
- charcoal 1 parts (or 14.3%)

These older formulas are worth trying when one is using crude grinding and mixing methods. Another issue is the charcoal used. Typically a higher percentage of charcoal should be used where the charcoal has a high ash content.

So much for variations in the formula where the manufacturing methods are crude. But is it worthwhile to vary the formula when more efficient methods are used? Yes it is. Black Powder makers have been doing this for a long time and are still doing this today. Most of the modern deviations from the 15:2:3 ratio are slight. For example, one formula for *sporting powder* used in firearms is as follows:

- potassium nitrate 76 parts (or 76%)
- sulfur 9.5 parts (or 9.5%)
- charcoal 14.5 parts (or 14.5%)

Other sporting powder formulas, deviating slightly more from the 15:2:3 ratio are:

- potassium nitrate 78 parts (or 78%)
- sulfur 10 parts (or 10%)
- charcoal 12 parts (or 12%)

and

- potassium nitrate 78 parts (or 78%)
- sulfur 9 parts (or 9%)
- charcoal 13 parts (or 13%).

Two examples of military powders close to the 15:2:3 ratio are:

- potassium nitrate 74 parts (or 74%)
- sulfur 10 parts (or 10%)
- charcoal 16 parts (or 16%)

and

- potassium nitrate 76 parts (or 76%)
- sulfur 9 parts (or 9%)
- charcoal 14 parts (or 14%).

Another modern formula, generally referred to as the PGII*mix* is as follows:

- potassium nitrate 25 parts (or 73.5%)
- sulfur 4 parts (or 11.8%)
- charcoal 5 parts (or 14.7%)

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There are quite a few other formulas around that use lower proportions of potassium nitrate and higher proportions of sulfur and charcoal. These are probably best left alone, unless one wants a really slow-burning powder. A good rule of thumb when experimenting with different formulas is to keep the ratios within the following limits:

- Potassium nitrate to sulfur: 4:1 to 8:1
- Potassium nitrate to charcoal: 4:1 to 6:1
- Charcoal to sulfur: 1:1 to 2:1.

The next issue we should look at relates to better ways of grinding or milling the materials.

### **Grinding Away Better**

Using a pestle and mortar to grind the ingredients is a very inefficient way of doing things. It can also be very hard on the hands.

Many pyrotechnic enthusiasts start out with small pestles and mortars. This is partly due to these costing less. Small can be beautiful in pyrotechnic experiments. It can also be painful and less efficient. Small pestles and mortars are painful on the hands. Larger is more comfortable and more efficient.

Even more efficient hand grinding has severe limitations. These limitations can only be met with some form of mechanized method of reducing the particle sizes of the ingredients. The next two chapters examine such methods, with a particular focus on ball milling.

### **The "in solution" Solution**

Why grind when you can dissolve? Dissolving a substance in water or some other solution effectively reduces that substance to microscopically tiny sizes right down to the size of individual molecules. This is something that no grinding method can do. In Black Powder, the most promising candidate for this treatment is the potassium nitrate.

Potassium nitrate dissolves fairly easily in water. Once dissolved, it can be readily absorbed into charcoal by mixing the charcoal with the potassium nitrate solution. Note I used the word *absorbed* here, rather than *adsorbed*. This is not a mistake. Although charcoal is especially noted for its adsorption properties, the process here is one of absorption. The potassium nitrate is absorbed into the porous structure of the charcoal, but does not get adsorbed by its surface.

What about sulfur? Sulfur can also be dissolved in certain liquids. Water is not one of them. Neither is alcohol. However, sulfur can be dissolved in carbon disulfide. Is this a viable way to go? No, it doesn't appear to offer any notable advantages. This particular issue is discussed in a later chapter.

Returning to potassium nitrate, it is important to make a saturated solution before mixing it with the charcoal. This is very important, as a saturated solution is vital for the success of this process. Somewhere in the process the potassium nitrate solution must be saturated. The best place in the process is before the mixing with the charcoal. Trying to achieve this after this stage is more difficult.

The next step is to get the potassium nitrate out of solution.

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## **The "out of solution" Solution**

Black Powder needs to be dry, or reasonable dry to burn properly. The potassium nitrate in solution thus needs to be gotten out of the solution.

There are two issues worth noting:

- Potassium nitrate can be lost through a leaching out process.
- Potassium nitrate crystals can end up being too large.

**Losses through leaching out** occur when some of the potassium nitrate in solution is separated from the other Black Powder components. This problem usually occurs when the potassium nitrate solution is not properly saturated, but can occur under other conditions. Sometimes the leaching out does not fully separate the potassium nitrate from the other components, but rather leaves a surface residue of potassium nitrate crystals. Either way, leaching out is bad news.

What exactly is this leaching out process? Leaching out is the removal of substances by dissolving them in a solvent and then removing this solvent. This removed solvent carries the dissolved substance with it. Sometimes leaching out is used deliberately in separating different substances.

When a leaching out process is done deliberately a large amount of solvent is typically used, with the solvent being removed from the mixture. Unintentional leaching typically uses less solvent, with the solvent ending up somewhere on the surface of the mix. Here it evaporates, leaving behind a crystalline residue. Leaching out is bad news for Black Powder. It makes it weaker, or in extreme cases, totally unusable.

**Potassium nitrate crystals end up being too large** when the moist Black Powder takes too long to dry. Here one can lose any advantage gained by dissolving the potassium nitrate in water. Any solution of a crystalline substance will yield small crystals if allowed to cool or dry out rapidly. If the solution dries out or cools slowly the opposite occurs; large crystals are formed.

So here we have two problem phenomena, leaching out and large crystals. The further bad news is that in trying to solve the one problem, one can end up creating the other! Leaching out can occur if the moist Black Powder is dried too quickly. Unacceptably large potassium nitrate crystals can form if the Black Powder is dried too slowly. How do we overcome these conflicts?

We overcome these conflicts by using the following strategy:

- 1) Start off with a saturated hot solution of potassium nitrate.
- 2) Remove the bulk of the water from the mix as quickly as possible.
- 3) Complete the drying process in a controlled way.

These three points are now described in detail.

**A saturated hot solution of potassium nitrate** aids the process by promoting rapid recrystallization. A saturated solution will yield crystals very soon after it starts to cool. A non-saturated solution will not. The longer the solution takes to crystallize, the more chance it has to leach out some of the potassium nitrate.

**Removing the bulk of the water from the mix as quickly as possible** can be achieved in a number of different ways, some of them practical, others not.

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The first way is to rapidly heat the mix, causing the water to evaporate. This is hardly practical and can be very dangerous. Heating the mix to too high a temperature may cause it to ignite. This possibility is relatively remote. A more likely scenario is a small amount of mix coming into contact with a hotter surface and then igniting. This can happen if the heating process causes some sputtering in the mix from escaping steam. Another issue that needs to be considered is the fact that the higher the temperature of the mix, the easier it will ignite from another source such as a spark.

So rapidly heating the mix may not be a good idea. What about slowly heating it?

A second possibility is to slowly heat the mix until all the water boils off. This sounds reasonable as the temperature of the solution will remain at slightly above 100 °C until all the water has boiled off. This temperature is still way below that needed to ignite the mix. In theory perhaps, this could be done. In practice it could be very dangerous. Practically speaking, it is very difficult to control the temperature of the container once all the water has left it. A hot spot could develop very quickly, causing the whole batch to ignite.

A third approach is to spread the mix over a large surface. A large surface area promotes evaporation. It also causes the mix to cool faster, promoting the rapid formation of crystals.

A fourth way is to drop the atmospheric pressure rapidly. This will cause rapid evaporation. The only problem is how to do this in a practical way. Some type of vacuum producing device is needed. A simple solution such as a vacuum cleaner could be very dangerous if some Black Powder got into the wrong place at the wrong time.

Yet another way is to add something to the mix that robs it of its water. Alcohol does this well.

Using alcohol is the basis of what is known as the *alcohol displacement method*. This method is perhaps better known as the *CIA method*. This so-called CIA method is described in a later chapter.

**Completing the drying process in a controlled way** may seem to conflict directly with what has just been said. However, there is no conflict if we consider this as a step completely separate from the former step. Here we are talking about the final stages of the drying process. The first stages focus on getting a large portion of the water out of the mix and cooling the mix rapidly. The last stages focus on the small amount of water left in the Black Powder mix.

Just a word of clarification about the previous steps. These are best done together with rapid mixing to ensure the process happens uniformly throughout.

The final drying stages need to be done in such a way that any gains made in the previous stages are not lost. This means that heating should be avoided or kept to a minimum. The reasons for this are because heating:

- can cause slower (and thus larger) crystal growth
- promote leaching out
- cause uneven drying with the surface dry but with the core still wet.

So heating should be avoided until right at the end of the process. Here I have successfully used heating to bake solid compressed pucks of Black Powder mix to increase hardness of the grains. The way I did this was to dry the pucks in the shade for most of the time and then baked them in direct sunlight when they were almost dry.

Pucks and pressure are discussed next.

# BLACK POWDER MANUFACTURE, TESTING & OPTIMIZING

## **Putting on the Pressure**

Here we tackle a rather controversial issue: the role of pressing in making Black Powder.

Commercial manufacturers of Black Powder press the damp milled product under high pressure before granulation. This process is controlled carefully to ensure that the right amount of pressure is applied. The right pressure ensures that the Black Powder will conform to desired density specifications. Controlling the density gives some control over consistency in performance.

About ten years ago I corresponded with several pyrotechnic enthusiasts living in the USA and Europe. Much of the discussion centered on producing fast Black Powder that met or possibly exceeded the speeds of commercial powders. The prevailing doctrine at the time was that pressing the powder before corning increased its speed. Some believed that an increase of up to 100% was possible if the right pressing process and pressures were used. Some even spoke about *plastic flow* and other such scientific-sounding matters.

I admit I was sucked into this doctrine, and remained that way when I wrote the first edition of this book.

One of the reasons I believed that pressing increased the speed of the powder was my own experimental data. I had made a few batches of Black Powder and found that moderate pressing in a pasta maker gave an increase in speed of about 10%. The literature on the CIA method also presented data showing speed increases in compressed powder. So I was hooked on the idea *of pressed is best*, and planned to make a better powder press.

## **Pressed is Best?**

I never got around to making my improved powder press. And in some ways, I am glad that I didn't.

Is pressed best? It could be if one wants hard grains of Black Powder. Hard is better for some applications such as Roman Candles. Does pressing improve speed? No, usually it does not.

A lot of experimental data, gathered by scientists and by amateurs shows that less dense powders generally burn faster. This goes against what was once the accepted doctrine. But accepted doctrines, based on suppositions, need to bow before factual data gained from numerous well planned and controlled experiments.

So in general, less dense powders burn faster. This means that going to the trouble of pressing Black Powder at a high pressure to improve speed could be a waste of time and money. So are there any advantages to pressing? Yes, there are.

Pressing offers the following advantages:

- 1) Black Powder that is suitable for applications such as Roman candles.
- 2) Consistency in performance is more easily achieved.
- 3) The powder is less likely to crumble in storage and during transportation.
- 4) Moisture is less easily absorbed by denser powders.

These points are now explored in detail.

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**Roman candles** need a powder that is hard enough to resist crumbling during the manufacturing process. This is very important to ensure that the propellant properties of the Black Powder are not compromised. When powder granules are crushed, the powder generally burns at a slower rate. This is because the important spaces between the granules gets plugged up with the crushed powder.

**Consistency in performance** is more easily achieved with powders made to a consistent density specification. This is very hard to achieve without some form of pressing.

**Pressed powder** is less likely to crumble in storage and during transportation. The issue of powder crumbling was discussed under Roman candles. However, in any application where Black Powder is used as an explosive or propellant, crumbling adversely affects performance.

**Moisture is less easily absorbed** by denser powders. When Black Powder absorbs moisture its performance changes. Very small amounts of moisture can actually make Black Powder burn faster. However, any amount of moisture above this very small amount causes a degradation in performance.

Having said all that, how much pressing is actually needed. The good news is: not much. My simple pasta maker press was adequate for Roman candles. It should be fine for many other applications. Others have also found moderate pressing to be adequate for their needs. So does one need a high pressure press? The answer is: generally speaking, no.

## **Pressing Methods and Techniques**

So far we have established that some form of pressing is desirable, but not necessarily essential. This section explores different pressing methods and techniques.

Before examining different methods it is a good idea to discuss some techniques that apply to all of the methods used. These are summarized as:

1. Black Powder should be pressed moist but preferably not wet.
2. Slow, even pressure should be applied.
3. Consistent pressure should be applied for each batch.
4. Pressure should be applied long enough to ensure consistency.

These are discussed in detail.

**Black Powder should be pressed moist** to ensure that the particles bind together. However, the amount of water should be minimized. Thus it is best to press moist, rather than wet Black Powder. If the powder contains too much water when pressed, some leaching out may occur.

**Slow, even pressure should be applied** to ensure that the Black Powder is pressed in a consistent manner. If this is not done, the pressed powder may have some spots that are more compressed than others.

**Consistent pressure should be applied** with each batch to ensure that it has the same characteristics as previous batches. Of course, if one deliberately wants to vary characteristics from one batch to another, this rule doesn't apply.

**Pressure should be applied long enough** to ensure that the powder has been consistently pressed. This consistency relates to previous batches, and also to consistency within the batch itself. The way to achieve this is to apply the desired pressure and maintain it for an acceptable period of time. Some refer to this time as the *dwelling time*. Times I have seen quoted for high

## BLACK POWDER MANUFACTURE, TESTING & OPTIMIZING

pressure compression have varied between 30 and 60 seconds. Low pressure compression would probably require less time.

Having described some techniques applicable to all the different methods, we now move on to the methods themselves. There are many different ways of doing this, but most rely on variations of the following two methods:

- 1) Sandwiching the Black Powder between one or more plates (usually made of metal such as aluminum) and squeezing the plates together.
- 2) Placing the Black Powder in an enclosed chamber or cylinder and then applying pressure.

All these methods produce solid pieces of material, typically in the form of pucks or pellets. These pucks or pellets are then dried. Different methods are now discussed. These are:

- 1) Powder pizzas using sandwiched plates.
- 2) Powder pucks using a pasta maker.
- 3) Powder pellets using a pump and press.
- 4) Paper tube powder pucks.

These are now described in detail:

**Powder pizzas using sandwiched plates** is a method that was described to me by an Italian pyrotechnic enthusiast. He referred to the resulting product as a type of *pizza*.

This method sandwiches a layer of moist Black Powder mix between two aluminum plates. Typically the layer of Black Powder is 1/4" thick. More powder and plates can then be successively stacked on the first sandwich, giving a multi-layered sandwich. The sandwich is finally compressed using a hydraulic press.

A simpler method is to use a bench vise to squeeze the plates together. The system can be improved by placing sheets of waxed paper between the aluminum plates and the Black Powder. This helps when one wants to remove the *pizza* from the plates.

**Powder pucks made with a pasta maker** was a method I discovered by accident when I was trying to extrude Black Powder *spaghetti* from a pasta maker. All I got was some of the moisture that exited via the spaghetti former holes. I was left with a solidly formed puck and decided this could be a better way to go.

Pasta makers are similar to meat grinders and sausage makers. These use a type of screw feed mechanism to compress material and feed it out in either ground or extruded form. Compared to other methods, the pressure is rather low, but good enough for a good Black Powder puck.

**Powder pellets using a pump and press** can be made with a pump used to make pumped stars and a simple press, such as an arbor press.

Not everyone is the proud owner of a star pump. This isn't a problem. A simple pump can be made from a medical syringe. All one needs to do is to cleanly cut the needle end off so the plunger can be pushed all the way through.

The Black Powder pellets are made by placing a measured amount (by volume) of Black Powder in the tube of the pump. A couple of layers of paper toweling are then placed on a solid flat surface. The end of the pump tube is placed on the toweling and pressure is applied to the plunger. This pressure can be applied by hand but is better done by using a press. An arbor press can be used if the required pressures are not that great. An ordinary drill press could be used instead of an arbor press. Greater pressures would need something like a hydraulic press.

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An important point is worth noting: air should be excluded from the pump as much as possible. This applies both to the open end and the plunger end. This is important to ensure consistency in the pressing process.

**Paper tube powder pucks** are made by compressing Black Powder in a paper tube and then cutting the tube open.

## **Granulation**

The last phase in making Black Powder is granulation. In commercial Black Powder plants this is often regarded as the most dangerous phase. Granulation of homemade powders is not without its dangers, but tends to be less dangerous (relatively speaking) than milling.

**Wet granulation** is the simplest form of granulation, and was described briefly in the last chapter. This is achieved by pressing a dampened Black Powder mix through a sieve. The preferred way of doing this is to use a wooden spoon or spatula rather than a metal one. Metal against metal can cause sparks, although this is far less likely to happen with dampened material. Personally I prefer to play safe and not use metal against metal.

It is quite an art to achieve the right moisture content with this method. Too much water causes the Black Powder to stick together after passing through the sieve, while too little makes excessively crumbly grains. Fortunately this method allows one to easily recycle the Black Powder mix. If the mix is too dry one can add a small amount of additional water and push it through the sieve again. If it is too wet one can allow it to dry out for a while, or one can add some dry powder. The latter method is still employed in modern powder mills to adjust moisture content during the milling process. Adding dry powder is also the preferred method in our granulation process.

Drying out wet powder by letting it stand for a while can result in weaker powder due to the potassium nitrate leaching out of the mix. Keeping aside some dry powder to mix with over-wetted powder overcomes this problem. Thus the two rules to be followed for better wet granulation moisture adjustment are:

- Add water if the mix is too dry.
- Add dry powder if the mix is too wet.

After the powder has been granulated it needs to be properly dried and then sieved again. A good place to start is to use the same sieve that was used to produce the wet granules. This will get rid of any larger lumps that may have formed. A finer sieve should then be used to sift out the finer particles. These fines can then be recycled in the next batch.

Wet granulation is adequate if low density crumbly granules of Black Powder are acceptable for a particular application. The issue of low density versus high density powders was discussed earlier in this chapter with somewhat of a bias towards high density powders. However, many pyrotechnic enthusiasts from all parts of the world have found low density granules adequate for lifting shells. This applies in particular to larger shells, which can be lifted with slower powders than those used for smaller shells and comets. Part of this attraction for low density powder is the granulation process is easier. Harder granules require a *harder* process. Harder granules require a dry granulation process.

**Dry granulation** is used to create harder, more dense, Black Powder. This process involves the granulation of Black Powder than has been formed into pucks or pellets. Granulation is carried out when the pucks or pellets are completely dry, a process that often takes a few days or even longer.

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Some may be tempted to start granulation before the drying process is complete. I don't recommend this because it reduces the potential hardness of the granules. Incomplete drying usually means a difference in moisture content between the outside and inside of the puck or pellet. This means that the resulting granules will be inconsistent in hardness and density.

Dry granulation is potentially more dangerous than wet granulation. The reasons for this are:

- Dry granulation requires an extra step in the process, this being the creation of pucks or pellets.
- Dry Black Powder is easier to ignite than wet.
- More energy in the form of shock or friction is applied in this process.

Before moving on, some discussion of the above three points is in order.

Generally, any extra step in an inherently dangerous process has the potential to increase the danger in the process. This is especially true if the extra step is dangerous in itself. Otherwise the danger comes from the added handling time and the fact that more complexity increases the chance of mistakes being made. The dangers presented in the extra step of making pucks or pellets have more to do with these last-mentioned reasons than dangers in the process itself.

It probably goes without saying that dry powder is easier to ignite than wet, and dangers from static, friction, and shock are generally much greater when the powder is dry.

Crushing the puck or pellet to yield the required granules can impart energy in the form of shock or friction. Excessive energy can cause the powder to ignite.

The crushing process can be done in a number of ways. Some of the tried and tested methods are:

- Applying pressure with a rolling pin
- Striking with a small non-metallic mallet
- Squeezing in a vise
- Using a jaw crusher.

These are now described in detail.

**Rolling pins** can be used to crush pucks or pellets that have been pressed under a relatively low pressure. They may not work too well with harder stuff. Using a rolling pin is my preferred way of doing this process.

Traditionally rolling pins have been made of wood. Recently I have seen quite a number made from plastic. I have always stuck to wood, but plastic may work if the plastic is hard enough to withstand pressure against hard Black Powder granules.

I prefer to sandwich the puck or pellet between some sheets of paper before crushing. In some of my kitchen capers I have used a glass bottle in place of a rolling pin when I didn't have a rolling pin handy. However, when working with Black Powder I have always used a proper rolling pin. Glass and Black Powder do not mix well under any circumstances. Broken glass propelled by any form of explosion can be peculiarly lethal!

**Non-metallic mallets** can effectively crush a puck or pellet by striking them with a light blow. For safety's sake, it is best to use as light a blow as possible. The right amount of force can be found by experimentation. This is done by starting off with very light taps and then progressing to harder taps until the puck or pellet shatters. Wrapping the puck or pellet in a plastic bag

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before crushing is a good way to stop the resulting pieces from flying away and getting lost.

A **vise** works as a good crusher by placing the puck or pellet in a plastic bag and slowly squeezing it between the jaws a the vise.

**Jaw crushers** are machines having two or more wheels or *jaws* containing grooves or other types of serrated edges. Such crushers are used in laboratories to crush hard materials. Some pyrotechnic enthusiasts have built their own jaw crushers. These have typically used brass or some other non-sparking material for their jaws. Jaw crushers can crush a lot of pucks or pellets quickly but do carry with them a certain amount of danger that they may cause accidental ignition.

Dry granulation tends to produce a wide range of granule sizes. This can be an advantage or disadvantage, depending on one's applications. The resulting granules need to be sifted into different sizes, using a variety of sieve sizes. Very large granules need to be re-crushed. Inevitably the process of crushing and re-crushing leaves some residue of very fine powder. This powder has its uses as *meal powder* in a variety of pyrotechnic applications. It can also be re-worked into future batches of Black Powder.

## *Chapter 7 - The CIA Connection*

### **Introduction**

There is a certain popular mythology surrounding the contents of this chapter. The myths revolve around "secret" methods developed for CIA and other clandestine field operators. These methods would enable such persons to quickly formulate concoctions to blow up various assortments of baddies, all in the name of freedom, democracy and making the world a better and safer place to live in.

Such heroes, armed only with their wits and a Swiss army knife, would thus be able to do what others considered to be nothing short of the miraculous. And, wonder of wonders, their secrets have finally been released into the public domain. The truth is somewhat less romantic.

This chapter describes one of a series of studies initiated by the Frankford arsenal way back in the sixties. These studies investigated methods whereby US armed forces would be able to manufacture and use improvised weaponry in the field. It really had nothing to do with the CIA. This chapter has emulated the way that the original data was presented and thus its layout and format differs somewhat from the rest of the book.

This so-called "CIA method" attracted many pyrotechnic enthusiasts (pyros) who had wanted to try their hands at making their own Black Powder but had often been disappointed and frustrated in their efforts. While this method gave improved results to some, it was not quite what many pyros hoped it would be.

Results of my own personal attempts were also disappointing. Although I achieved a jump in performance over simple hand-ground Black Powder, the CIA method powder did not even come close to powder needed to eject the stars in my Roman candles. Yes, it did burn faster. In fact it burned at a linear burn rate of five times that of the hand-ground powder! This was no mean feat in itself, but it was still not fast enough.

I discussed my findings and frustrations with others and learned that their experiences were similar to mine. My burning speed had hit a ceiling of about 15 centimeters per second (cm/sec.). Theirs had too. Were we all doing something wrong? Yes we were. We were all wrong to buy into the notion that acceptable Black Powder could be made without proper milling and incorporation.

Reading carefully between the lines in the official descriptions of the CIA method reveals that proper milling and incorporation is recommended. Of course, under field expedient conditions this may not always be possible. Maybe for this reason the milling and incorporation stage has been downplayed. Unfortunately, it has been downplayed to the extent that many have tried the CIA method using ingredients that have been poorly milled and incorporated. Their results have naturally been disappointing.

So the CIA method has gotten a bad press. And some have gone on their own personal jihads to try and discredit it in any way they can. Some of these same individuals may even find this book hopelessly flawed because it devotes a whole chapter (and a long one at that) to the CIA method.

The CIA method does have its drawbacks. These are discussed both in this chapter and in

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other chapters of this book. It does, however, offer some advantages that other methods do not. The CIA method:

- Offers a viable entry-level method to those starting out in making Black Powder.
- Does not require special machines or equipment (for slower powders).
- Does not have the explosion hazard of some other methods.

The CIA method, when it is combined with ball milling, can produce a Black Powder that is more than adequate for use as a propellant. This has been proven by both myself and experimenters all over the world with whom I have been corresponding over the years.

What follows in the rest of this chapter is the traditional CIA method text, gleaned from the original government reports. After this I have added my own tested and proven methods for improving the CIA method, together with ball milling to yield a very fast powder. An honest attempt is also made to point out deficiencies and drawbacks in the CIA method.

## **Approach**

The following sequence was pursued in the Black Powder study:

- Literature survey.
- Evaluation of techniques for the preparation of Black Powder.
- Performance evaluation of various preparations.
- Preparation of a field manual outlining preparation procedure(s).

## **Discussion**

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

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

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

Of necessity, a method meeting the design criteria of the current program must employ available utensils, and require a very limited degree of practice or instruction. This would dictate that principles of preparation be unaffected by a broad range of variation in the procedure, or

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be a procedure of ultimate simplicity. This goal was achieved through the investigation and development of a unique precipitation procedure. Essentially, this method consists of preparing a hot slurry of charcoal and sulfur suspended in concentrated aqueous potassium nitrate with the subsequent precipitation of Black Powder by rapid drowning in a common organic solvent such as isopropanol. From this point, filtration, granulation, and drying were facile rapid processes. The resulting products were generally consistent from lot to lot, and approached commercial powders in performance. The method and powder performance test results are considered adequate fulfillment of the requirements of the program.

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

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

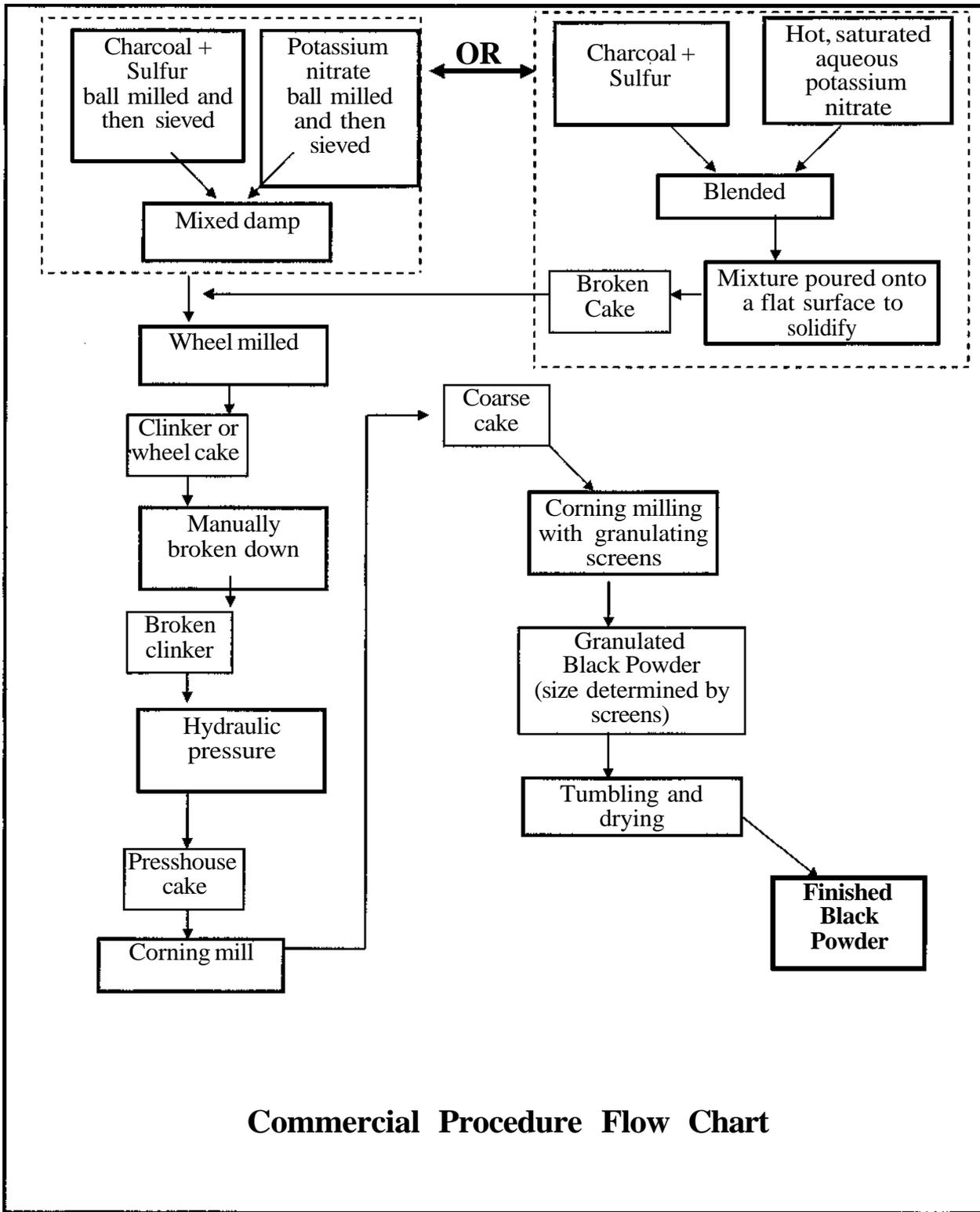
## **Experimental**

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

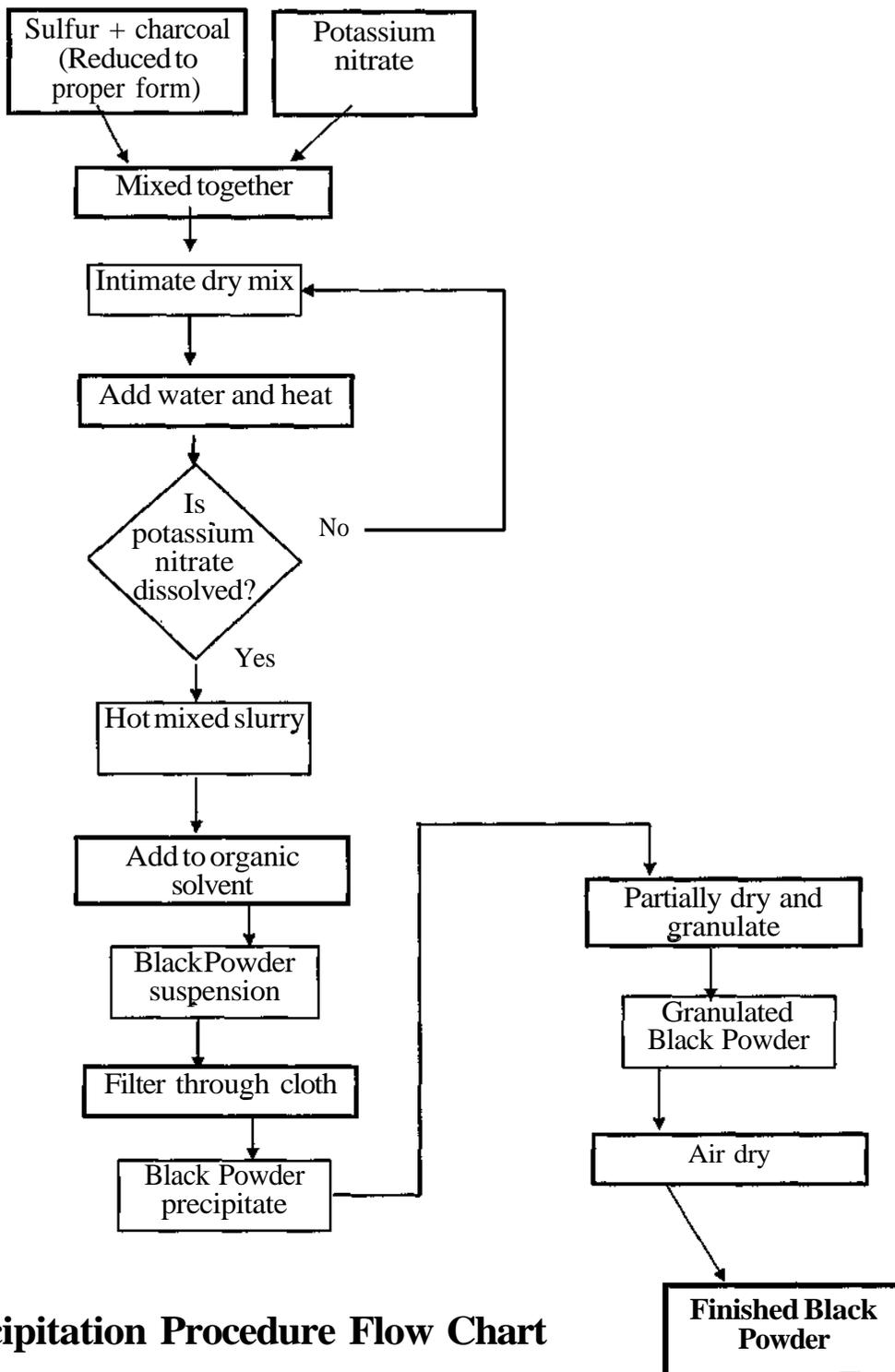
Simplicity and safety are inherent in the method since no extensive premixing operations are required and the mixture can be handled in a moist form throughout the preparation. It is noteworthy that the method is independent of the prior particle form of the potassium nitrate since total solution of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

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

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Lot No.	Granulation	Comments	Burn Rate (cm/sec.)
1	10-16 mesh		12.6
2	10 -16 mesh		7.7
3	Similar in size to a grain of sand	Sodium nitrate base	0.96
4	Dry mixed powder with no granular structure		0.22
5	3.5 - 16 mesh		6.1
5	16-80 mesh		2.0
5	Through 80 mesh		0.95
6	10 - 16 mesh	Cake pressed manually	2.2
6	10 - 16 mesh	Cake pressed at 4 tons	7.7
6	10-16 mesh	Cake pressed at 5 tons	8.6
6	10 - 16 mesh	Cake pressed at 6 tons	10.7
7	10 - 16 mesh	100% IPA (Note 2) precipitated	16.2
8	10 - 16 mesh	70% IPA precipitated	18.4
9A	10-16 mesh	70% IPA precipitated	10.5
9A	Riced grains	70% IPA precipitated	Instantaneous
9B	10 - 16 mesh	NaCl saturated 70% IPA precipitated	10.5
9B	Riced grains	NaCl saturated 70% IPA precipitated	10.5
9C	10 - 16 mesh	KNO <sub>3</sub> saturated 70% IPA precipitated	Instantaneous
9C	Riced grains	KNO <sub>3</sub> saturated 70% IPA precipitated	Instantaneous
10		KNO <sub>3</sub> saturated 70% IPA precipitated	14.0
10	10 - 16 mesh	KNO <sub>3</sub> saturated 70% IPA precipitated	8.4
10	16-25 mesh	KNO <sub>3</sub> saturated 70% IPA precipitated	6.5
10	On 10 mesh	KNO <sub>3</sub> saturated 70% IPA precipitated	1.4
10	Rewet riced grains	KNO <sub>3</sub> saturated 70% IPA precipitated	21.0
11	10 - 16 mesh	70% IPA precipitated	15.0
12A	10-16 mesh	80 proof vodka precipitated	12.8
12A	16-25 mesh	80 proof vodka precipitated	14.0
12A	Rewet riced grains	80 proof vodka precipitated	24.8
12B	10 - 16 mesh	KNO <sub>3</sub> saturated 80 proof vodka precipitated	13.2
12B	16 - 25 mesh	KNO <sub>3</sub> saturated 80 proof vodka precipitated	13.1
12B	Rewet riced grains	KNO <sub>3</sub> saturated 80 proof vodka precipitated	12.1
13A	Riced grains	70% IPA precipitated	8.3
13B	Riced grains	Absolute methanol precipitated	11.1
14	Riced grains	70% IPA precipitated	15.0
14	Rewet riced grains	70% IPA precipitated	14.0

Table 7-1. Burning Rates of the Various Black Powder Preparations (Note 1)

Note 1: All preparations were KNO<sub>3</sub> based unless designated otherwise

Note 2: IPA - isopropyl alcohol

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## **Black Powder Preparations -- Initial Phase**

The preliminary Black Powder formulations were made using both potassium and sodium nitrates and employed simple laboratory procedures. In the initial three lots the weighed or volume measured ingredients (column 3, Table 7-2) were moistened with water, thoroughly blended in a mortar and pressed between two metal plates using moderate hydraulic pressure. The resulting cake, which was approximately one-sixteenth of an inch thick, was oven-dried at 60 degrees C. and granulated by gentle crushing on a hard, flat surface with a length of pipe. A fourth lot of Black Powder containing sodium nitrate was prepared by a dry blending method. The volume of ingredients given in column 3, Table 7-2, were placed in a three pound coffee can, fitted with a polyethylene cover, and the can rotated in all directions until an apparently uniform mixture was obtained. The four powders were evaluated for burning rate and impact sensitivity; results are given in Table 7-2. The burning rates of the four compositions varied considerably; the sodium nitrate composition was the slowest burning, which is in accord with studies made by previous workers.

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

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

In Lot Number 5, fifteen grams of finely ground carbon were mixed intimately with 10 grams of ground sulfur flour and placed in a heavy iron skillet on low heat. The mixture was heated and stirred until the constituents were uniform in appearance. The carbon/sulfur mixture was removed from the skillet and stirred until cool. This mixture was added to a previously heated solution of 75 grams of potassium nitrate in 40 milliliters of water. The skillet and contents were removed from the heat and stirred until a uniformly wet paste was obtained. The slightly moist powder was poured onto a hard, flat surface and pressed manually into a thin cake with a rolling pin. The powder was dried at 60 degrees C. The dried lumps were broken into uniform granules and sieved through cheesecloth onto a nylon scarf. The portion on the scarf was shaken until all the fine powder was sifted out. The three portions were evaluated for the burning rate as described in the previous section with the following results:

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

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

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Powder	Ingredients	Percentages	Impact Sensitivity	Burn Rate cm/sec	Particle Size for Burn Rate
Lot 1	KNO <sub>3</sub> C S	75 by weight 15 by weight 10 by weight	No fire at 103 cm	12.6	Through # 10 sieve but not through # 16
Lot 2	KNO <sub>3</sub> C S	58.2 by volume 32.4 by volume 9.4 by volume	No fire at 103 cm	7.7	Same as Lot 1
Lot 3	NaNO <sub>3</sub> C S	72 by weight 17 by weight 11 by weight	No fire at 103 cm	0.96	Similar in size to grains of sand
Lot 4	NaNO <sub>3</sub> C S	42.4 by volume 46.9 by volume 10.9 by volume	10% fire at 23 cm and no fire at 22 cm	0.22	Dry mixed powder with no granular structure

Table 7-2. Properties of Various Black Powder Formulations

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

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

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

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## **Black Powder Preparation by the Precipitation Method**

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

### Lot No. 7

Potassium nitrate (technical grade)	150 g
Charcoal	30 g
Sulfur	20 g

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

### Lot No. 8

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

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

# BLACK POWDER MANUFACTURE, TESTING & OPTIMIZING

Lot No. 9	
Potassium nitrate	340 g.
Charcoal	68 g.
Sulfur	45 g.
Water	325 ml
Isopropanol (70%)	500 ml
Isopropanol (70%, saturated with NaCl)	250 ml
Isopropanol (70%, saturated with KNO <sub>3</sub> )	250 ml

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

- 1) 9A - One half of lot precipitated in 500 ml. 70% isopropanol
- 2) 9B - One quarter of lot precipitated in 250 ml. 70% isopropanol, saturated with NaCl
- 3) 9C - One quarter of lot precipitated in 250 ml. 70% isopropanol, saturated with KNO<sub>3</sub>

Each sub-lot was ultimately divided into quarters to accomplish air and oven drying and compare granulation methods as shown in Table 7-3.

Lot No. 10	
Potassium nitrate	1020 g.
Charcoal	204 g.
Sulfur	136 g.
Water	900 ml
Isopropanol (saturated with KNO <sub>3</sub> )	3000 ml

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

Sub-Batch	Granulation (mesh)	Burn Rate (cm/sec)	
		Air Dried	Oven Dried (2 hrs. @ 90 deg C)
9A	10- 16	10.5	11.0
9B	10- 16	10.5	8.0
9C	10- 16	Instantaneous	11.0
9A	Riced grains *	10.5	11.1
9B	Riced grains *	Instantaneous	8.1
9C	Riced grains *	Instantaneous	11.0

Table 7-3. *Drying and Granulation Comparisons*

\* A potato ricer was used to granulate the powder. Such a granulation produces a typical particle range of 75: 10 - 16 mesh and 25% 16 - 25 mesh admixed with a small quantity of fines.

## BLACK POWDER MANUFACTURE, TESTING & OPTIMIZING

Granulation (mesh)	Burning Rate (cm/sec)
10	14.0
10- 16	8.4
16-25	6.5
251	1.4
Riced, Rewet*	21.0

*Table 7-4. Granulation and Burn Rate Comparisons*

\*Accomplished by moistening the original powder with 70% isopropanol, granulating with a ricer and air drying.

Lot No. 1	
Potassium nitrate	340 g.
Charcoal	68 g.
Sulfur	44 g.
Water	300 ml
Isopropanol	1000ml

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

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

Lot No. 12	
Potassium nitrate	340 g.
Charcoal	68 g.
Sulfur	44 g.
Water	300 ml
Vodka (80 proof)	500 ml
Vodka (80 proof, saturated with KNO <sub>3</sub> )	500 ml

Universal availability dictated that vodka be given consideration as a precipitation medium in the field preparation of Black Powder. Initial preparation was as previously outlined; the lot divided into two equal parts and precipitated in vodka with (12A) and without nitrate saturation (12B). One half of each sub-lot was rolled, air dried, crushed and screened into definite granulation ranges; and the second half forced through a potato ricer and allowed to air dry. Burning rates of the resulting powders are given in Table 7-5.

## BLACK POWDER MANUFACTURE, TESTING & OPTIMIZING

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

Table 7-5. *Drying, Granulation and Rewetting Comparisons*

\* See footnote in Table 7-4.

A question of nitrate loss through solubility in the precipitation method prompted the retention of solvent after removal of solids in a lot similar in composition to Lot No. 9. Evaporation of the isopropanol yielded 61.7g. of KNO<sub>3</sub> representing a weight loss in the powder of 18.1%. Compensation for this loss results in a 78/13.2/8.8 composition.

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

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

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

As indicated, volume measurements were made of the ingredients. Blending was performed in the normal fashion with the exception that the fuels were added to previously dampened nitrate to eliminate working with the oxidizer-fuel system in the dry state. The filtration was accomplished using a linen towel. After air drying to a slightly moist consistency, the material was granulated using a potato ricer and sun dried for one hour. The burn rate was determined to be 15 cm/sec. Re-wetting and re-ricing did not appreciably alter this particular product (14 cm/sec.). Weight-volume relationships for the various ingredients are given in Table 7-6.

# BLACK POWDER MANUFACTURE, TESTING & OPTIMIZING

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

Table 7-6. *Weight-Volume Relationship of Ingredients of Black Powder*

## **Application Tests**

One pound of Black Powder, as produced for Lot No. 5, contained in a 12" x 2" plastic tube was placed in a 36" x 2" hole, fused and covered with soil. On ignition, a hole approximately 20 inches in diameter was produced.

Three pounds of Black Powder, as produced for Lot No. I3A was contained in a coffee can and buried 42" deep. Ignition produced a crater six feet in diameter.

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

Several propellant grains were prepared from powders similar to Lot No. 11 and Lot No. I3A, both loosely packed and pressed to several tons. In each case the powder exploded when ignited in a nozzled chamber indicating the need for a modifier to control the burning rate.

Three pounds of powder were prepared in a manner similar to that described in I3A except that 270 grams of soluble starch were intimately mixed with the wet powder prior to ricing. Subsequent to air drying, the composition was re-wet with 10% water, and two 1 1/4" cylinders having 1/8" center holes were pressed at five tons. The grains weighed 50 grams each and after air drying were incorporated into rocket configuration. After addition of fuse and ignition material, the unit, which weighed 580 grams, functioned as anticipated and attained an altitude of 400 feet.

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

For instance, one small batch of 75%  $KClO_3$ , 12.5% S, 12.5% C, prepared by wet blending in isopropanol exhibited an impact sensitivity of 15 cm (10% fire level). RDX gave a value of 32 cm. Other compositions prepared include 75%  $KClO_3$ , 25% sugar, 20 cm; 60%  $KClO_3$ , 35% sugar, 50% C, 10 cm. Also, these mixtures were prepared using solvent blending. In addition, a promising "White powder", namely potassium nitrate-sugar, was briefly investigated. Such a composition was prepared and evaluated in the following manner: Sixty-five grams of potassium nitrate and 35 grams of granulated cane sugar were placed in an iron skillet and sufficient water added in increments with heating and stirring until solution was affected. Shortly after the mixture began to boil, a precipitate formed resulting in a white slurry. Stirring was continued until the mass changed with additional heating from a crystalline slurry to a smooth homogeneous mass. At this point, the material was poured onto a flat surface and worked by rubbing with the wooden stirring rod into small lumps. If the mixture is allowed to cool without stirring, a single mass is formed which is very difficult to granulate. The burning rate of the 10-16 mesh product was 9.3 cm/sec.

# BLACK POWDER MANUFACTURE, TESTING & OPTIMIZING

This approach to indigenous pyrotechnics should warrant further study from a standpoint of fuel selection, oxidizer selection and fabrication techniques. Previous investigations have shown KNO<sub>3</sub>-sugar compositions to be satisfactory propellants.

## **Conclusions**

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

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

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

## **The CIA Method**

### **Preparation of Black Powder:**

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

### **Material Required:**

Heat source such as a kitchen stove (or an open fire, if it is the only available source)

Two-gallon bucket (metal or plastic)

Cooking pan or skillet; 4 quart capacity

Flat window screen, at least 1-foot square

Large wooden spoon or stick

Plain weave cloth sheet (at least 2 feet square)

Measuring cup (8 ounces)

Potassium nitrate (granulated)

Powdered wood charcoal

Powdered sulfur

Rubbing alcohol (70% isopropyl alcohol) or wood (methyl) alcohol

Water

# BLACK POWDER MANUFACTURE, TESTING & OPTIMIZING

## **Procedure:**

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

Add 2 more cups of water to the mixture and place the pan on the heating source. Allow the liquid to come to a simmer with sufficient stirring to obtain an evenly mixed blend. With vigorous stirring, rapidly pour this mixture into 5 pints of alcohol contained in the 2-gallon bucket.

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

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

Dry the granulated Black Powder by spreading on a flat surface in about a 1/2-inch layer. Sun drying is preferred for this step.

## **Improving the CIA Method**

Most aspiring Black Powder makers who have tried the CIA method have come away disappointed. Some of these have gone to great lengths to try to *get it right*, without success. Is there a right way or a better way, and does this way (if it exists) make powder fast enough to lift shells or Roman candle stars?

Yes, there are right and wrong ways to the CIA method. There are also better ways.

The introduction to this chapter recommended ball milling. Alas, for really fast powder, one needs to ball mill. If ball milling is not an option then some other milling method needs to be found. Other methods that attempt good incorporation without milling have been tried numerous times. None of these have been found to give acceptable fast powder. These include:

- mixing the charcoal with molten sulfur
- dissolving the sulfur in a solvent
- heating the Black Powder solution to above the melting point of sulfur.

All of the above aimed at incorporating the sulfur in a better way. While it can be argued that some of the above methods may make marginal improvements to the process, none have been reported to substantially increase the speed of the powder.

So milling appears to be the only viable way of getting the CIA method to yield good results. But why use the CIA method if one is going to mill anyway? Good question. There is little use for the CIA method if one's motivation to use it is to avoid milling entirely. Personally, my feelings are that if one is comfortable with three-component ball milling of Black Powder, then one should not waste time messing with the CIA method. If one has doubts (especially in the area of safety) concerning three-component milling, the combined milling/CIA method is a good alternative.

## BLACK POWDER MANUFACTURE, TESTING & OPTIMIZING

Personally, I have opted for this last method. My main motivation was the safety aspect. My other motivation was sheer curiosity. Here I was curious to investigate whether one could make a viable Black Powder using such a method.

Along with this method I aimed at optimizing the CIA method by focusing on the basic principles involved and improving on them.

### **Optimizing the CIA Method**

There are a number of things one should focus on if one wishes to optimize the CIA method. These are:

- ball milling
- mixing and heating
- alcohol quantity
- cooling
- pressing
- granulating

**Ball milling** is essential for very fast powder. If one requires slower powder, one may get away without ball milling.

Ball milling here relates to milling the charcoal and sulfur and then using the CIA method to incorporate the potassium nitrate. Here one is not only restricted to ball milling. If one is not milling the potassium nitrate together with the sulfur and charcoal, then high-energy milling methods can be used. These may mill the ingredients to a finer powder than is possible using a ball mill. Finer powder could potentially give a faster Black Powder.

**Mixing and heating** needs to be done properly to ensure thorough incorporation. There is very little meaningful data available to show exactly how much mixing is required for how long to give optimal results. However, common sense suggests that the solution of potassium nitrate should be properly saturated. It may also be helpful to let the mix stand for a while before heating.

Usually one can only mix the solution of potassium nitrate with the other ingredients when the solution is hot. What I have done is to heat the solution and stir in the milled charcoal and sulfur until everything is properly mixed. I then let the mix stand and cool for about half an hour, and then I reheat it until it boils. This cooling and reheating actually may not be necessary.

**Alcohol quantity** is one area where it pays to be generous. Often this is where people try to cut corners. My philosophy is that more alcohol is needed to ensure that as much water as possible is absorbed by the alcohol. Another reason for using large amounts of alcohol is one can cool the hot Black Powder mix faster if more alcohol is used. This point is discussed under the next heading.

**Cooling** should take place as fast as possible. The faster the cooling, the smaller the resulting potassium nitrate crystals. Faster cooling can be achieved by cooling the alcohol in a refrigerator freezer and by using lots of it.

The method that I have followed is to leave the alcohol in the freezer for a long time and also to freeze the metal bowl that I put the alcohol in. These procedures aim at making the alcohol as cold as possible prior to adding the hot Black Powder mix.

## BLACK POWDER MANUFACTURE, TESTING & OPTIMIZING

**Pressing** has recently created a fair amount of controversy.

In times past the general Black Powder folklore stated that pressing always increased the speed of the powder, unless too much pressure was exerted. Some of the data given in other parts of this chapter shows that pressing actually increased the speed of the powder. My personal experiments showed a 10% increase in speed between the pressed and unpressed powder.

Some of the Black Powder literature gives experimental data showing that the less denser powders actually worked better in propellant applications than their denser pressed counterparts. Recent tests using Pyro Golf and similar methods have agreed with these findings, so not pressing the powder does appear to have some merit.

Why the results in another part of this chapter conflict with this other experimental data is not clear. Perhaps the manufacturing processes themselves have something to do with it.

My system uses a pasta maker to press the powder into pucks. When I first tried this device, I was hoping to extrude the powder into strands like spaghetti. I reckoned that this would be a good way to try and corn the powder. It didn't work as planned. The powder itself stayed inside the pasta maker, while the liquid was squeezed through the holes. But it did make good solid Black Powder pucks. So I made pucks instead of *Black Powder spaghetti*.

Black Powder pucks need to be properly dried before they are granulated. I found that drying them out of direct sunlight was best for most of the time, with drying in direct sunlight good for the final stage of drying. There are some good reasons for doing it this way.

Drying the pucks in direct sunlight when they are still very wet causes the water to leech out some of the potassium nitrate. This leaves the inside of the puck with a depleted percentage of potassium nitrate and the outside with an excess. Both situations are bad. So it is best to only dry the puck in direct sunlight once most of the moisture has gone through drying in the shade.

The final direct sunlight drying helps bake the puck, making it harder.

When my pucks were thoroughly dry, I then proceeded with the corning process. I placed the pucks on a flat surface between two pieces of paper and crushed them with a rolling pin. I then sieved the resulting granules into different sizes. Chunks left on the largest sieve were crushed again.

## *Chapter 8--Milling Methods*

### **Introduction**

While a simple pestle and mortar can be used to make serviceable Black Powder, the process is a long and tedious one. It is also very hard on the hands. Over the centuries many improvements have been made in milling Black Powder. These methods yielded not only better quality powder but allowed powder makers to manufacture larger quantities.

They did this by making larger milling devices and by using more powerful sources of power in place of human strength. Thus powder mills were simple machines that used (originally) power sources such as horses or water. Later machines used steam and electricity. This chapter describes both medium to large scale manufacture and small scale homemade techniques.

Medium to large scale Black Powder production has used four methods of note. These are:

- stamp mills
- ball mills
- wheel mills
- jet mills

### **Stamp Mills**

Stamp mills have a very bad track record. While they make useable Black Powder, they are very prone to accidents. So stamp mills are no longer in use in Black Powder manufacture. In fact, in some countries they were outlawed long ago.

What are stamp mills? Stated simply they are a type of mechanized pestle and mortar, with the pestle being continuously raised and dropped into the mortar. This is a stamping action, rather than a circular grinding action.

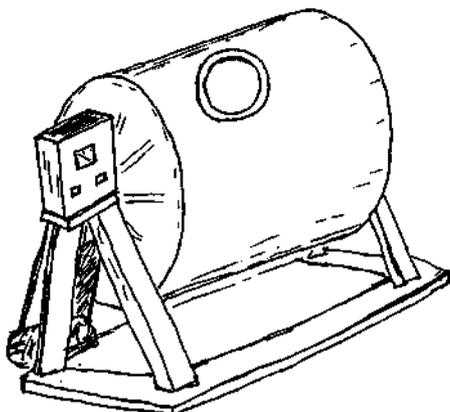
Most of us think of a pestle and mortar as something that works similar to the hand devices that rely on a circular grinding motion to mill the materials. However, many pestles and mortars throughout history have used a stamping action rather than a circular grinding action. In Africa, rural subsistence farmers still grind corn using a stamping type of pestle and mortar.

The stamping or hammering action in stamp mills is quite violent and has triggered many explosions in its day. Stamp mills are always run wet, to reduce the possibility of ignition. Even so, an unacceptable number of accidents occurred, leading to stamp mills being phased out of service.

### **Ball Mills**

The mining and manufacturing industries use ball milling as one of their milling methods. Manufacturing industries use a wide variety of ball mill sizes, ranging from small laboratory size units to large continuously fed ball mills. Mining typically uses very large ball mills.

## BLACK POWDER MANUFACTURE, TESTING & OPTIMIZING



*Figure 8-1. A small industrial ball mill*

Ball mills are not only used to grind materials, they are also used to mix inks, paints and certain powders. Some high energy ball mills are employed in making metal alloys through high impact milling. Some ball mills are used to polish manufactured goods.

Ball mills are used extensively by amateur pyrotechnic enthusiasts. They are not the usual method of choice for large scale manufacture of Black Powder. This honor goes to wheel mills.

However, ball mills are used for mixing and pre-grinding of materials prior to wheel milling. They are also used in the final stages of the manufacture of sporting powders. Here they are used without the balls, and are best described as tumblers, rather than mills. This tumbling action is used to round off the powder grains. It is also used in the final stage of manufacture to glaze the powder grains with a thin coat of graphite.

While ball mills are not the preferred milling method in large-scale manufacturing operations, they seem to have found favor in smaller operations. The many smaller Black Powder manufacturers in China are reputed to use ball mills. Some of these even have the advantage of being able to unload their charge remotely, something that cannot currently be done with wheel mills.

### **Wheel Mills**

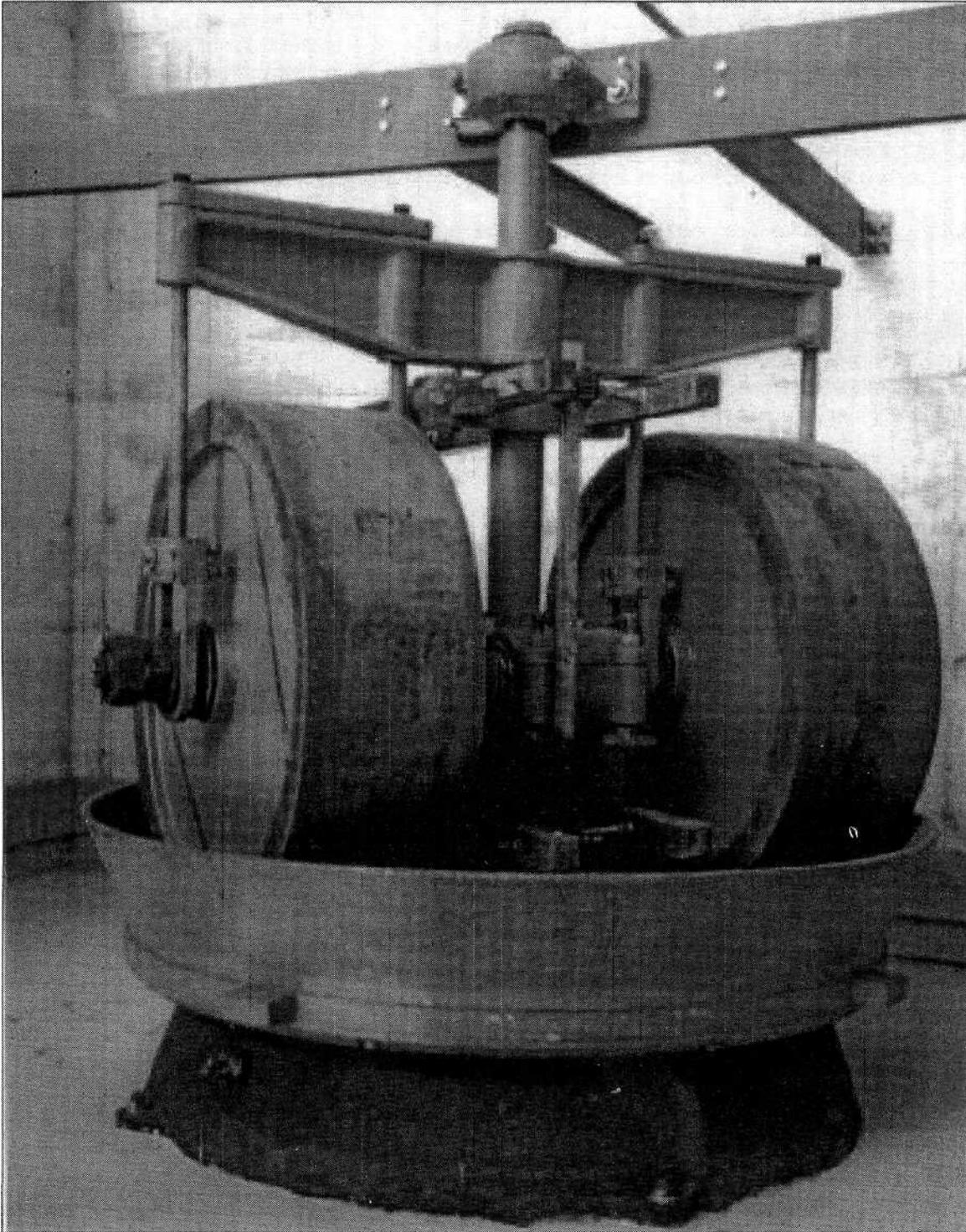
Wheel mills, also known as edge runners, are used by modern larger-scale Black Powder manufacturers. Wheel milling is their method of choice.

Wheel mills have been around for centuries and have a good track record of producing good Black Powder, while generally being less hazardous to operate than other types of mills. Early wheel mills used horses or water wheels for power. Modern mills use modern power sources such as electricity.

What is a wheel mill? A wheel mill is a device that mills substances by pressing them between wheels and a flat surface. A common use of wheel mills is to extract oil from oil-producing plants such as olives and sesame seeds.

A typical Black Powder wheel mill is a large device, ranging in size from half a ton to twenty tons. Generally the wheels of such mills are made of steel. Some of the older wheel mills used only one wheel. The norm today is to use a pair of wheels.

## BLACK POWDER MANUFACTURE, TESTING & OPTIMIZING



*Figure 8-2. A wheel mill used in a modern Black Powder plant.*

Although wheel mills are typically very heavy, the full weight of the wheel is not applied to the Black Powder mix. There is usually a small gap between the wheel and the bottom of the container holding the mix. The resulting incorporation is thus a process of squeezing and kneading the mix. A small amount of water is always added to the mix. This helps the incorporation process and also inhibits accidental ignition.

# BLACK POWDER MANUFACTURE, TESTING & OPTIMIZING

Wheel mills are normally equipped with scrapers that act as a type of spatula. These are used to ensure that the mix is constantly re-positioned under the wheels. This re-positioning is necessary because the wheels tend to push a certain amount of the mix out of their path.

Wheel mills can be operated at low speeds of 0.25 - 0.5 rpm or high speeds of 10 - 15 rpm. Water is added periodically to the mix to ensure that it stays damp. This is necessary because the milling action creates a buildup of heat, which evaporates the water.

Although wheel mills are considered to be less dangerous than other milling methods, they have been the cause of accidents over the centuries they have been in use. For this reason, wheel mills are always operated remotely.

One of the known hazards is the mix getting too dry, making it prone to accidental ignition. Wheel mill operators thus need to constantly add water to the mix. Adding water keeps the mix moist and also reduces heat that builds up during the milling process. This heat buildup makes the water evaporate more quickly, requiring frequent replenishment.

## **Jet Mills**

Jet mills are typically used in laboratories to finely pulverize materials. They can also be used on a larger scale. One example is the Lovold<sup>1</sup> jet mill used to manufacture Black Powder.

The Lovold jet mill theoretically offers some advantages over traditional milling methods but hasn't yet found favor with commercial manufacturers. To date they seem to have been used only for making military Black Powder in small quantities. It is even debatable whether one could describe these operations as manufacturing rather than just small-scale feasibility investigations. But who knows? Even in an open society, not everybody knows what the military is up to.

## **Small Scale Techniques**

We have just looked at the methods used in making Black Powder in medium to large quantities. We now look at small-scale methods. These methods include both tried and tested methods and some that may be worth experimenting with.

All milling methods are dangerous, and any method decided upon must be approached cautiously. This applies particularly to methods that are not yet tried and tested.

Small scale methods discussed here are:

- motorized pestles and mortars
- coffee grinders
- blenders
- tumblers
- ring and puck pulverizers
- small ball mills
- small wheel mills

## **Motorized Pestles and Mortars**

I came across a description of one of these while researching laboratory milling methods. This does not appear to be a common or popular milling method, but it does exist nevertheless.

## BLACK POWDER MANUFACTURE, TESTING & OPTIMIZING

Basically this device uses a motor to drive the pestle in a rotary type motion. This is certainly better than hand grinding and appears to give reasonable results. It is also simple enough for one to make one's own. One way of doing it is to mount the mortar on some kind of turntable and keep the pestle stationary. Here one would mount the mortar slightly off center.

### **Coffee Grinders**

Coffee grinders are designed to grind coffee beans to the size normally used in making coffee, and not any smaller. Coffee grinders are thus useful for pre-grinding charcoal prior to its being ground finer by some other milling method. They aren't much use for grinding potassium nitrate or sulfur, unless these contain large chunks of material.

### **Blenders**

Aspiring Black Powder makers have often asked about blenders.

To date I have only heard of Black Powder made in blenders that was used in comets, rockets and quick match. These all used a three-component slurry mixed with water.

There is possibly some potential here in making fast Black Powder, but one must blend it as a wet slurry! Dry blending could be very dangerous.

### **Tumblers**

Tumblers are used by lapidary enthusiasts (rock hounds) for polishing semi-precious stones. They are also used by gun enthusiasts who reload their own ammunition. The latter use tumblers to polish cartridges and cartridge cases.

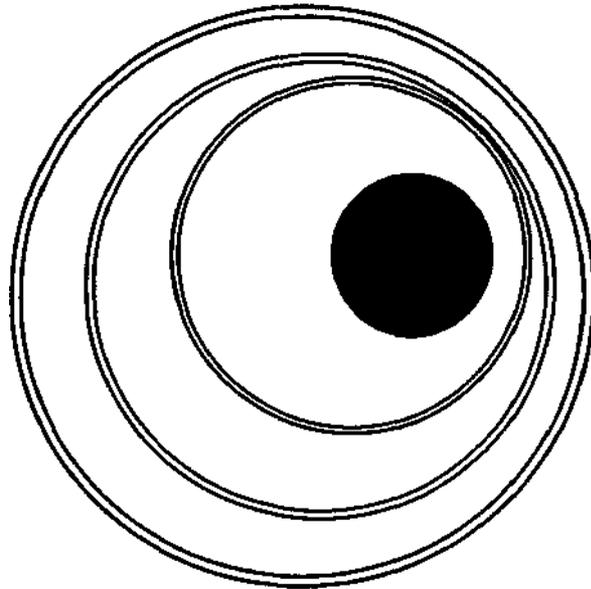
Tumblers have been used successfully as ball mills. They may require some modification to their speed for optimal use. Some of the smaller tumblers on the market may be a bit too weak for good ball milling.

### **Ring and Puck Pulverizers**

I was quite amazed the first time I saw one of these in operation. It reduced a small sample of rock to talc-like powder in the matter of seconds!

Ring and puck pulverizers are typically used to grind mineral samples. They do this by pulverizing the material in a very violent manner. Such grinding is totally unsuited for three-component Black Powder as an explosion can be regarded as inevitable. However, it does offer possibilities in grinding the individual components.

The principle on which ring and puck pulverizers operate is a hardened steel puck surrounded by two concentric steel rings. These are placed in a sealed container, together with the material to be ground, and then vibrated violently. This vibration action causes the puck and rings to collide while sandwiching the material between them.



*Figure 8-3. Ring and puck pulverizer rings*

Ring and puck pulverizers are very expensive and it is probably quite a challenge to try and make one's own. If one does have access to one, it may be worth experimenting with in separate grinding of the components. Under no circumstances should one try grinding more than one component at a time.

## **Small Ball Mills**

High quality Black Powder can be made in small laboratory-sized ball mills. This is presently the milling method of choice for amateur pyrotechnic enthusiasts. Small ball mills are covered in the next chapter.

## **Small Wheel Mills**

I do admit I was very skeptical when I first heard about small wheel mills. I pictured them as not being able to hold a candle to their big brothers that weighed several tons. I now have to admit that I was probably wrong.

While size (in terms of heaviness) does count in manufacturing operations, it may be far less important in small-scale enterprises. Small laboratory-scale wheel mills have been used successfully to make small batches (about ten pounds) of Black Powder. This is probably because the secret of wheel milling does not lie in crushing the mix, but rather in squeezing and kneading it.

However, there is presently so little data available on the effectiveness (or lack thereof) of small wheel mills. I would venture to say that the jury is still out on this one.

## **Notes**

- 1) Kjell Lovold, Process for the Preparation of Black Powder, U.S. Patent 3,660,546 (1972)

# BLACK POWDER MANUFACTURE, TESTING & OPTIMIZING

## **Introduction**

This chapter examines ball milling.

Ball milling is one of the most popular methods used for making homemade Black Powder. In fact, it is difficult to make good Black Powder without some method of milling that goes beyond hand grinding. Ball milling is a good method to use, and has proven to be the most viable up till now.

## **Ball Mill Safety**

One thing that bugs me about ball milling is the very small number of reported accidents. Please don't get me wrong. I don't wish such an accident on anyone. I'm just wondering what the true situation is.

With many pyrotechnic enthusiasts acquiring or making their own ball mills these days there is a lot of pyrotechnic ball milling going on. Some of these enthusiasts are self-confessed *serial Black Powder* makers, meaning they have made lots of the stuff. With more cars on the road the greater the chance of traffic accidents. With more and more people ball milling Black Powder, the greater the chance of ball mills exploding. So why haven't more done just that? There are a number of explanations, such as:

- Everyone doing it follows all the proper safety precautions all the time.
- The perceived dangers are lower than most thought them to be.
- The accidents are under reported.

What is the true situation? It is probably a combination of all the above.

My concern is that too many will gravitate to the second reason: *the perceived dangers are lower than most thought them to be*. Even if this were true, and the other two reasons were to be discounted, the danger of complacency is still a reality. And it's easy to be complacent and perhaps gain the dubious distinction of being the first recorded amateur ball mill induced fatality.

Safety is discussed in the first chapter of this book, but it is profitable to recap on some of the safety concerns specific to ball milling. These are:

- Avoid three component milling where possible.
- Mill in small quantities.
- Watch out for sparks.
- Watch out for static.
- Use blast shields.
- Use remote control.
- Be aware of mechanical hazards.
- Keep it clean

The above points are now discussed in detail.

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**Avoid three component milling** where possible. Here the term *three component* means the three components of Black Powder, namely potassium nitrate, sulfur, and charcoal.

Three component milling is the most dangerous type of Black Powder milling. Unhappily, this has become the method of choice for many. Arguably it is the quickest and easiest way to make Black Powder and it is very tempting to go this route. Personally, I would avoid it. There are ways of making serviceable Black Powder without resorting to three component milling.

One such method is to mill each component separately. Another is to mill the charcoal and sulfur together or the potassium nitrate and sulfur together. The latter is more hazardous, as one is grinding an oxidizer with another component. However, a worse choice is to mill the potassium nitrate and charcoal together. This mix is far more likely to ignite than a mix of potassium nitrate and sulfur.

Having said that, I cannot leave this subject without mentioning some of the different opinions and controversies. Some consider milling charcoal and sulfur on their own to be more hazardous than milling them together. There have been known cases of sulfur igniting when milled on its own. Some have blamed this on sulfur's tendency to build up electrostatic charges. Some have attempted to get around this problem by adding a small amount of potassium nitrate to the sulfur to increase its electric conductivity.

Another hazard associated with milling sulfur is the milling process causes the milled material to heat up. Hot sulfur can thus ignite if air is allowed into the milling jar while grinding. The sulfur could also ignite if the jar were opened while still hot.

Charcoal dust can also ignite if mixed with oxygen and enough heat is supplied. For this reason, it is wise to let a milling jar containing charcoal cool down before opening it.

Having given a warning about avoiding milling potassium nitrate and charcoal together, I need to elaborate on this point. Generally a potassium nitrate/charcoal mix is more likely to ignite than a potassium nitrate/sulfur mix. However, mixing a small amount of charcoal with the potassium nitrate is regarded by some as safer.

The safety issue here is that adding pure potassium nitrate to a finely milled mix of sulfur and charcoal is considered to be more hazardous than adding a blend of potassium nitrate and charcoal. The trick here is to use only a small amount of charcoal, the ratio of which should not exceed 1:15. (Note: the normal ratio of charcoal to potassium nitrate is 1:5)

**Mill in small quantities.** Small is beautiful. The larger the mill, the bigger the bang when an accident happens. The bigger the bang, the greater the danger to life and limb.

**Watch out for sparks** that can come from a number of sources.

First, one must ensure that the milling media do not spark when knocked or rubbed together.

Second, one should eliminate all sources of electric sparks. Here it is better to use a brushless motor. One must also ensure that there are no loose electrical contacts near the mill. The motor should also be mounted where the likelihood of materials falling into it is minimized.

Third, one should avoid materials that create static electricity. This applies both to the mill and the clothes one is wearing. Another course worth pursuing is to provide an electrical ground to the mill.

**Watch out for static** electricity from a number of sources.

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This point was touched on under the last heading.

Static electricity must be taken seriously. It has a track record of being a *stealth killer*, having caused the deaths of thousands without leaving any compelling evidence that it was the cause of the tragedy.

Static electricity is different to the normal electricity that we are familiar with, in that no electric current flows until the instant that two opposite electrically charged bodies are brought close enough together for current to flow from the one to the other. This current flow is of a very short duration and often produces a spark. Lightning is caused by static electricity.

Static electricity is created when friction occurs between two different substances. These substances can be solids, liquids, or gases.

The static dangers peculiar to ball milling are the materials the mill is made from and also the charging and emptying of the jar. Another danger that applies to all pyrotechnic manufacture comes from clothes, including shoes.

A common material used to make ball mill jars is PVC. This has been known to build up static on the outside of the jar. This in itself is usually not a hazard while the mill is running but could be when emptying the mill. Here is a good idea to electrically ground the mill, while at the same time grounding one's self. Directly grounding one's self can be done by using what is known as an *anti-static wrist strap*, obtainable from suppliers of electronic components and tools. An added advantage of using such a device is that it gives your body a high resistance path to ground, thus reducing the dangers of electric shock, should you touch any electric circuit.

**Use blast shields.** Blast shields are used to protect one from the effects of an explosion. In some circumstances they are used close to the explosive device while the person behind the shield handles the device using robotic type arms. This is possibly the ideal way to charge and empty a ball mill. In practice, this is rarely possible.

The next best choice is to have a shield between one's self and the mill, with one's arms and hands extended beyond the shield to handle the mill. Here one's body is shielded but one's arms and hands are exposed. This is hardly ideal, but better than having no protection at all.

A very good idea is to use a full face shield to shield one's face, rather than just a pair of safety glasses.

**Use remote control** to turn the mill on and off. One way of doing this is to run a long extension cord.

If one uses such an extension cord, one should not overlook the possibility of someone else switching power to the cord while one is handling the mill. One should ensure this does not happen.

**Be aware of mechanical hazards** when operating the mill. It is easy to focus on the dangers of fire or explosion and neglect simple mechanical dangers.

The larger and more powerful the mill, the greater the danger from its mechanics. A motor with sufficient power can easily mangle a finger or even sever it. Loose clothing can get caught in the mill while it is running, dragging the wearer into it.

**Keep it clean.** This means keeping the area around the mill free from any Black Powder dust

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or residue. This is one of the golden rules in pyrotechnics, but is often neglected. Another issue is other substances. Small pieces of iron or iron particles can cause sparking if allowed to contaminate a ball mill. Another source of sparks is titanium.

Both iron and titanium are used in pyrotechnics. Both of these should not come anywhere near a ball mill that contains substances or mixes that can ignite. While the sparking properties of iron are known to many, the properties of titanium are not. Believe me, I have seen titanium spark with the slightest amount of friction. Personally, I believe it to be more hazardous as a source of sparks than any other metallic substance I have experimented with.

### **Beyond Simple Ball Milling**

This chapter goes beyond the simple jar type ball mills that many of us have come to know and love. It ventures into the area of *high energy* ball milling. This is uncharted territory, and should be trodden cautiously. Ordinary jar ball mills are tumbler type mills. Although very effective, their grinding action is relatively gentle. This is gentle enough to grind even three component Black Powder without frequent incidents of an explosive nature.

The high energy methods discussed should under no circumstances be used to grind potassium nitrate mixed with anything else. And extra caution is advised when grinding sulfur and charcoal together.

### **Ball Mill Types**

This chapter looks at three different types of ball mills:

- simple jar mills
- planetary ball mills
- vibratory ball mills

Simple jar mills are the most popular type of ball mill and the discussions at the end of this chapter concerning mill charge and speed apply mainly to these.

### **Simple Jar Mills**

Most of us are familiar with the simple jar mills, also known as tumbler mills. These work on the principle of a jar being rotated on its axis, this axis being in a horizontal plane or close to a horizontal plane.

Jar mills rely on the force of gravity to act on the balls. So forces acting on the balls can never be greater than the force of gravity. This limitation effectively places a limit on the grinding action that takes place. Thus jar mills grind more slowly and less efficiently than other types of mills that can produce forces greater than the force of gravity.

Jar mills are thus regarded as *low energy* ball mills. This low energy characteristic is actually an advantage in Black Powder milling, as low energy milling is less dangerous than high energy milling. So one can get away with milling Black Powder in a low energy mill, while high energy mills can virtually guarantee an explosion. Here I am talking about three-component milling.

Jar mills typically use jars made from metal, plastic or ceramic materials. Some jars have removable liners.

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A typical jar mill is driven by an electric motor connected to a horizontal roller that comes into contact with the jar. The jar rests on this roller and on a second roller that rotates freely and is usually not driven. Some jar mills are equipped with electronic speed controls, while others use a belt and different size pulleys to vary the speed.

One can construct one's own jar mill or purchase one from either a laboratory supplier, a supplier of lapidary equipment, or a supplier of reloading equipment. Laboratory equipment is usually very expensive. Lapidary or reloading suppliers are better bets. They offer tumblers that are used to polish stones or cartridges and cartridge cases, and may describe their equipment as *tumblers* rather than ball mills. Tumblers and ball mills basically are the same. They are just used in different ways.

Tumblers used for lapidary and reloading may not run at speeds optimal for efficient ball milling. One may need to modify these to run at higher speeds. Any such equipment should be purchased with ease of modification in mind. Another issue is that the smaller sizes of tumbler may be a little weak for efficient ball milling.

Another source of jar ball mills is a pyrotechnic supplier. Some suppliers sell pyrotechnic machinery such as star rolling machines and ball mills. They may be even better to deal with. At least one can discuss with them what one is planning to use it for and maybe get some good advice in the process.

It is fairly easy to make one's own ball mill. Simple, low cost, methods are described in the chapter on budget Black Powder. These should be regarded as *entry-level* ball mills. They work but are relatively inefficient. Those wishing to invest time, money and energy in making a high efficiency jar ball mill should invest in a copy of Lloyd Sponenburgh's *Ball Milling Theory and Practice for the Amateur Pyrotechnician* or get back issues of AFN that contained Lloyd's articles on ball milling.

### **Planetary Ball Mills**

Planetary ball mills are high energy devices and are unsuited for three-component milling of Black Powder. They can, however, be used to mill the individual components separately.

Planetary ball mills are not constrained by the force of gravity. In a sense they create their own gravitational force, which is greater than the Earth's force of gravity. This is done by producing centrifugal forces by spinning a set of milling jars at high speed around a central axis. The jars can thus be likened to planets in the solar system. And like planets, each jar rotates on its own axis.

Unlike jar ball mills, planetary ball mills have their jars mounted horizontally. This is because the greatest force on them is the centrifugal force generated by the spinning action. In essence, the milling action in planetary mills is similar to that of jar mills. The main difference is the force exerted on the balls in a planetary mill is higher than that exerted in a jar mill. This force can be dangerously high if the mill contains a Black Powder mix.

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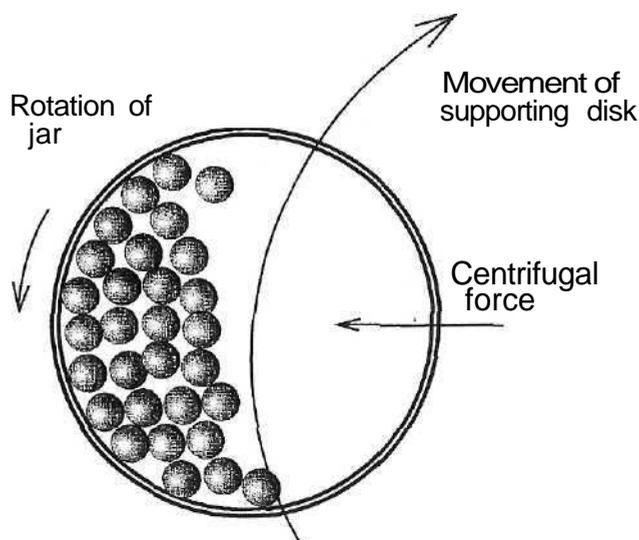


Figure 9-1. Motion of planetary ball mill

Planetary mills are used in both laboratory and lapidary applications. They are very expensive. Possibly the only real advantage they offer is shorter milling times. If this is critical in a production-type environment a planetary mill may be a good investment.

### **Vibratory Ball Mills**

Vibratory ball mills use vibration energy to perform the milling action. Such mills are *high energy* mills and one is not advised to use them in three-component Black Powder milling.

Ball mills using the vibration principle can be bought from suppliers of laboratory, lapidary or reloading equipment. At the time of writing this, I am not aware of any suppliers of pyrotechnic equipment offering these.

There has been a limited amount of experimentation with vibratory ball mills in pyrotechnic applications. Indications are that they do a good job of grinding Black Powder materials. Again it should be stressed that these are *high energy* devices and should not be used for three-component milling. And not all vibratory ball mills are created equal. Some are much more violent than others, so due caution is advised.

Vibratory mills are usually electric powered although it is possible to construct such a device that is pneumatic (compressed air) powered. Vibration is usually accomplished by one of two methods:

- eccentric cams
- vibrating solenoids.

**Eccentric cams** are cams basically unbalanced weights attached to motor shafts. This causes the motor, and whatever it is attached to, to vibrate. This principle is used in vibrators in pocket pagers and in vibrating sanders. Large industrial vibrating screens use this principle as well.

**Vibrating solenoids** use an electric solenoid that is powered with an alternating current (AC). The frequency of this current is typically the same as that supplied by electric utilities; this is 50 or 60 Hz, depending on which part of the world one lives in.

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Any AC solenoid will vibrate slightly. Vibrating solenoids increase the vibration effect by introducing an opposing magnetic field, either in the form of a permanent magnet or an electromagnet.

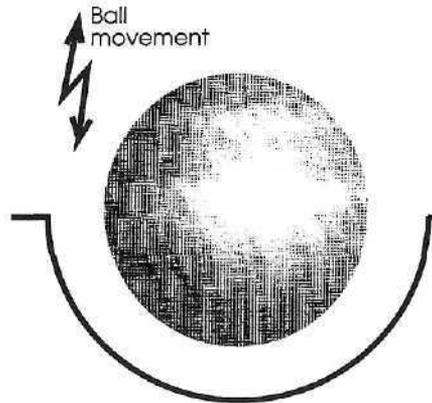


Figure 9-2. Vibratory ball mill mechanism

Vibratory ball mills either use a large number of balls like other ball mills or just one single large ball. With a large number of balls, vibration energy is applied to the whole mill, causing both the balls and the material being ground to vibrate. With a single ball, vibration energy is imparted to the ball, causing it to bounce up and down at a fast rate, crushing the material underneath it. Single ball vibratory mills are typically used to mill small amounts of material.

## **Milling Media**

The term *milling media* refers to the balls themselves. There are many different types of milling media on the market today. Many of these are designed for exacting laboratory applications, with prices to suit. Here we are looking at some proven, cost effective, solutions.

There are several criteria involved in media selection, such as:

- size
- weight
- hardness
- sparking characteristics
- corrosion resistance
- cost

**Size** influences two important criteria in ball milling. The first is the weight of each ball. The second is the critical speed of the ball mill. This second criterion is tied up with the ratio of the diameter of the mill to the diameter of each ball. If this ratio is high, then changes made to the size of the ball need to be significant to have any impact on critical speed. This ratio should be fairly high anyway to get a good tumbling action while milling.

It is not a good idea to mix balls of different sizes.

**Weight** is a function of the size and density of the ball. A general rule of thumb is the heavier the ball the better.

**Hardness** is less critical in Black Powder applications than in others that use harder materials. Probably the only time that hardness is an issue here is if one uses soft materials such as lead.

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Even lead that is not hardened works in Black Powder milling but tends to get out of shape and wear quicker.

**Sparking characteristics** could literally spell the difference between life and death in making Black Powder. Some materials such as iron, steel, and some ceramics are proven generators of sparks when knocked or rubbed together. It goes without saying that these should never be used in three-component Black Powder applications. It is also not a good idea to mill potassium nitrate with such media if it is mixed with anything else.

**Corrosion resistance** specifies how resistant the medium is to corrosion from other chemicals. Potassium nitrate corrodes iron and steel very rapidly and may also react with other materials. Corrosion creates contamination in the material being milled and may adversely affect the grinding efficiency of the balls themselves.

**Cost** can be an important consideration, but should be factored in carefully. For example, some media may cost less but wear more quickly.

Some popular choices of milling media are:

- lead
- steel
- brass
- glass
- ceramic

**Lead** balls are used for such things as fishing sinkers and as shot in Black Powder firearms. Both of these can be used in ball mills. I have personally used lead fishing sinkers.

Lead is very dense, making even small balls heavy. This is an advantage. Lead, however, has some disadvantages. Lead is soft. Thus lead balls can be easily bent out of shape. This softness also contributes to small particles being impregnated in the surface of the lead ball. Antimony-hardened lead goes some of the way to resolve these problems but does not offer the same hardness that other materials do.

Another issue with lead is that a small amount of lead contamination in the material that one is grinding is inevitable.

Lead sinkers have holes or slits in them that end up being filled with the material being ground. So these are not one's first choice. Lead shot used in Black Powder firearms is a better choice. These don't have slits or holes in them and generally are more perfectly round. Some are made with antimony-hardened lead. Some manufacturers offer lead shot that is brass plated.

When ordering lead shot be sure that it is the balls you are ordering and not shot that comes in bullet shapes.

Some are attracted to casting their own balls from scrap lead, some of this scrap coming from worn ball mill balls that are being recycled. For those who do a lot of ball milling, this practice may be worthwhile. However, there are additional hazards that come with casting lead. One hazard is hot melted lead can cause very painful burns if accidentally spilled or splashed around. The other danger is breathing in the fumes from the molten lead. This is a more insidious danger, because lead can accumulate in one's system over a period of time and give one lead poisoning.

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**Steel** balls are readily available from a number of sources as ball bearings.

Steel has the disadvantage of corroding (rusting) rapidly when it comes into contact with potassium nitrate. This problem may be overcome by using stainless steel balls.

Another problem steel poses is sparks. Steel hitting against steel can give off sparks. It is very unwise to use steel in three-component Black Powder milling.

**Brass** balls are less readily available than steel balls and are more expensive. They do offer the advantage of being non-sparking. Brass does tend to corrode, but not as rapidly as iron and steel.

**Glass** balls are readily available in the form of toy marbles. Other glass *marbles* are supplied specifically for milling applications. Glass is rumored to give off sparks so it is best to err on the side of caution when using glass.

**Ceramic** media is available in various shapes, sizes, and varieties of materials. Many have made ceramic the material of choice in pyrotechnic applications. Just a word of caution - not all ceramic materials are created equal. Some ceramics give off sparks when rubbed or knocked together. One way of testing ceramic materials for this property is to rub and knock two pieces together in a darkened room. This test is not foolproof. If it does spark then one knows that the ceramic in question poses a definite danger; if it doesn't one cannot be a hundred percent sure.

Ceramics generally are hard and dense. They are not prone to corrosion or contamination from other chemicals. They are thus an excellent choice, especially types that are proven to be non-sparking.

## **Wooden Balls?**

Believe it or not, wooden balls have been used in ball mills to make Black Powder. But not in the same way that other balls are used.

Some manufacturing processes mill the three ingredients of Black Powder separately, and then mix them in a drum that contains wooden humps along its inner circumference. This method is used in some factories to mix the ingredients prior to wheel milling. Others use this method as part of the final process prior to pressing and corning.

Other processes use a similar method, but use wooden balls rather than wooden humps. But, unlike the just-described process, this method uses ingredients that have been dampened with about 8-10% of water.

The balls in this process work by a kneading action, rather than the collision/grinding action found in normal ball mills. This action can be likened to the action of a wheel mill.

## **Charging the Mill**

For optimal grinding, a ball mill needs to be charged with the right amount of material and milling media. This may vary in the different types of ball mills discussed above. However, we will only concern ourselves here with jar type mills as these are the most popular.

Before we look at suggested amounts, it is worthwhile to get a good picture of what happens inside a ball mill. This picture will also help us in the next section that discusses speed.

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A ball mill is normally charged with both balls and material. Sometimes the balls are left out when only mixing and no grinding is required. Usually we consider a ball mill balls and all.

If we place just a few balls in the mill with a little bit of material, the balls will end up just rolling over the material, doing very little grinding. Most of the grinding action here will be due to the weights of the individual balls resting on the material. Some grinding may take place from the balls colliding with one another, provided that there is some material trapped between them. This whole scenario makes for very weak and inefficient milling.

Now if we add a whole lot of material so as to effectively drown the balls, we also end up with an inefficient system. Here the movement of the balls is dampened by the material. Even when they do move, the dampening effect of the material prevents any real grinding from taking place.

Going to the other extreme, we can virtually fill the mill with balls and then add a little bit of material. Here the balls will hardly be able to move because there are so many of them. Very few balls will actually come into contact with the material, so their presence is wasted. If we add more material we may end up with a virtually static system, with very little grinding taking place.

We have just looked at some extreme situations, all giving very poor performance. Optimal performance is achieved somewhere in between. This *somewhere* is what we will look at.

If we place balls in a ball mill, and rotate it very slowly, the balls will form a pile, the top of which will be approximately horizontal. If we increase the speed of rotation the top of this pile will shift from a horizontal position. We thus end up with one side of the pile being lower than the other. This is due to frictional forces.

Now if we increase the rotation speed even more, the angle of the top of the pile increases. This increases until balls at the higher end break free and start rolling down towards the lower end. This is shown in the figure below.

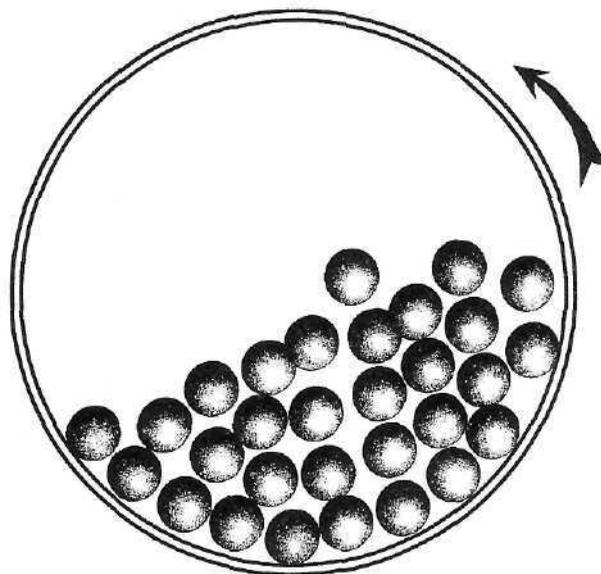


Figure 9-3. Start of cascading action

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Increasing our speed further still, causes the balls at the top end to break away faster and to roll down quicker. This rolling action results in collisions with other balls, causing them to break free.

The collisions of the cascading balls results in a grinding action if there is material between these balls.

What is the optimal amount for ball charging? This is simply the amount that will give the longest cascading distance. This amount is 50% of the jar capacity.

What amount of material is optimal? This amount is typically 25%.

## Mill Speed

From the above discussion, it is evident that speed plays an important role.

If the speed is too low, the cascading action does not take place. If the speed is too high, then centrifugal forces will press the balls against the walls of the mill. This will prevent them from breaking free, and thus no grinding will take place.

There is a critical point where the speed is high enough to project the ball so that it does not roll over the others in a cascading fashion. At this point the ball flies through the air and impacts the balls on the lower end of the pile. This end of the pile is referred to by some as the *Davis Zone*. This action is illustrated in the figure below.

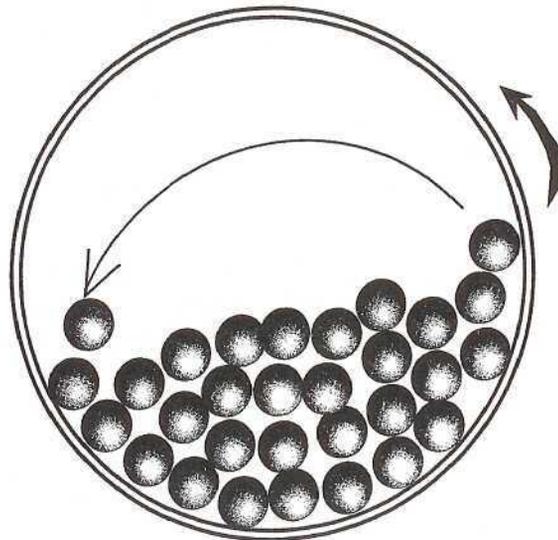


Figure 9-4, Action at critical point

Impact energy in the *Davis Zone* is a lot higher than could be achieved otherwise. Unfortunately, the number of impacts is limited, giving a rather inefficient grinding action. Another downside is the mill jar wears out faster. The speed at which the ball flies through the air without cascading is called the *critical speed*.

As a general rule of thumb for dry milling, the ball mill should be run at 65% of the critical speed. The critical speed is dependent on the diameter of the jar and the diameter of the balls. Here we assume that all the balls are the same size. There is no advantage in using different sizes of balls. The formula for the critical speed is as follows:

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$$\frac{265.45}{\sqrt{\text{jar i.d.} - \text{ball o.d.}}}$$

Note that the above formula for critical speed is for dimensions in inches. The critical speed is given in revolutions per minute.

So much for the math and the theoretical considerations. How does one know in practice if the mill is running at an acceptable speed? One method is to count the number of revolutions it makes in a minute. This should give an indication if it is running in the theoretical acceptable speed range. A better method is to monitor what it is actually doing.

Most ball mills are sealed, so it is not possible to actually see what is going on inside them. Even if one were able to make a ball mill from transparent materials, its inside would probably be coated with dust, making observation difficult. Fortunately one can get a good idea of what a ball mill is doing by listening to it.

Ball mills are noisy. Even inefficient ball mills can be noisy. I was once running a very inefficient ball mill in my garage and my neighbor came and asked me what I was doing. (Note that I was milling sulfur and charcoal so the danger from accidental ignition was minimal.)

The sounds that a ball mill makes gives one a good idea what it is doing. If the ball mill makes intermittent thumping sounds, it probably means that it is overcharged or running too fast. A swishing sound could indicate that the balls are slipping. A continuous cascading sound indicates that the mill is running properly with an acceptable charge and speed.

## **Wet or Dry?**

Some milling of Black Powder is done wet, other milling is done dry. What is the best way if one is ball milling?

Stamp mills are always run wet. The same applies to wheel mills. In both these instances wet milling is considered mandatory. It is just too dangerous to run a stamp mill or wheel mill dry. Safety issues aside, wet milling in both these processes is regarded as being of help to the milling process. Ball mills are different.

Ball milling can be done wet with some materials, but not with others. Black Powder does not mill well in a ball mill if it is wet. The biggest problem is the materials stick to the balls, hindering their movement and making the grinding action very inefficient. Another phenomenon is the materials sticking to the walls of the ball mill.

While some have reported acceptable results with Black Powder that has been slightly dampened prior to milling, most haven't. The issue of the material sticking to the walls is seen by some as being beneficial in the last stage of milling. Here a small bit of water is added after the milling process itself is considered to be almost complete. This small bit of water then causes to materials that have been thoroughly ground and mixed to cake on the walls of the mill.

Very little additional grinding now takes place, the main action here being the caking. Once the *cake* has reached an acceptable hardness, the mill is stopped and the balls removed. The cake is then scraped off with a spatula and dried. The dried cake is then crushed and sieved in a simple corning process.

## *Chapter 10 — Turbocharged Black Powder*

### **Introduction**

This chapter focuses on optimization methods aimed at producing very fast Black Powder.

Before exploring the various methodologies used to produce very fast powder, it is worthwhile examining reasons why one might wish to do so. It is also worth looking at reasons why one may wish to stay with slower-burning Black Powder. Faster powders offer better performance in certain applications. Slower powders work better in others.

And then there is the issue of safety. Faster powders are generally more hazardous than slower powders. This applies both in their manufacture and in their use.

### **Fast Powders and Their Uses**

Fast powders are used in many different applications, including:

- Black Powder firearms
- Lift (propellant) for fireworks shells
- Propellant for roman candles
- Propellant for comets
- Burst charges for fireworks shells
- Fireworks mines
- Fuses
- Rockets
- Firecrackers

One notices from the above list that some of the more important fireworks applications require fast Black Powder. So generally, for fireworks use, fast Black Powder is more desirable. Just how fast depends on the application.

Roman candles typically need a faster powder than shells. Comets and small shells need a faster powder than large shells. Rockets may or may not need a fast powder, depending on a number of different factors. Fuses, depending on the type and application, could require a fast powder.

Slow powders have less going for them. Blasting powder generally is a slower powder. Some rockets are better made with slow powder. Slow powder also finds its uses in fireworks such as gerbs, fountains, and drivers. Some fuses use slow powder.

### **How Fast is Fast?**

The term fast means different things to different people. To some fast means Black Powder that is fast enough to do the job. To others fast means faster than anything else in existence.

Black Powder that is fast enough to do the job could be a lot slower than the fastest powders around. Before the modern proliferation of very fast powders, many pyrotechnic enthusiasts successfully launched shells with powders that burned a lot slower than their commercial

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counterparts. These required more powder, but still worked. The problem of requiring extra powder was offset by differences in cost. Commercial powders, although requiring less material still cost a lot more.

Such slower burning powders worked well for large shells and maybe not so well for smaller shells. They were not recommended for Roman candles. This last-mentioned constraint was one of the main reasons that I aspired to making faster powder - I needed the stuff for Roman candles.

But is there any objective measure of fast? Is there any baseline? One objective measure of fast is to make comparisons with commercial powders such as Elephant and GOEX. This is quite a good objective measure as one can assume that such commercial brands will maintain reasonable consistencies from one batch to the next.

### **How Safe is Fast?**

Fast is not safe!

Fast is highly dangerous. Faster is even more dangerous, both in manufacture and in use. All Black Powder manufacture and use is dangerous to some extent. Making and using faster powders increases one's exposure to danger.

At one time it was considered prudent to make powders that were just fast enough to do the job in hand. Today this wisdom does not get the attention it deserves. One reason for this is the phenomenon discussed in the next section.

### **Competition Grade Black Powder**

In the last few years a new type of Black Powder has gained prominence in amateur pyrotechnic circles. This is the so-called *competition grade* Black Powder.

What is competition grade and how did it come about? Competition grade Black Powder is a powder that is considered to be faster than its commercial counterparts, sometimes quite a lot faster. Another way of defining competition grade is a powder that gives top readings in *Pyro Golf* competitions. What is *Pyro Golf*?

*Pyro Golf* is a test mortar that fires golf balls. It is used to test the strength of Black Powder both in laboratory type tests, and in competitions between amateur Black Powder makers. *Pyro Golf* is described in detail in the chapter on testing.

Before *Pyro Golf* came on the scene there was no such thing as competition grade Black Powder. Now there is — inspired in part by *Pyro Golf*. Other factors did come into play such as more and more pyrotechnic enthusiasts acquiring high efficiency ball mills.

*Pyro Golf*, with its spin-off of competition grade powders, has had a very positive impact on amateur Black Powder making. It has inspired an interest in making Black Powder that did not exist before. This resulted in many making powders with speeds that at one time would not have been thought possible. It also acted as a catalyst in challenging some cherished myths about making Black Powder, including the value of high pressure pressing and certain charcoals. Sadly it has also had a negative impact.

Because *Pyro Golf* competitions focus on speed to the exclusion of other properties, speed has been given a status that perhaps it shouldn't have. Thus some now always equate fastest with

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best and measure so-called improvements solely in terms of improvements in speed. This doctrine has led to the rise of a new generation of the *Great Green Gurus* I described in the first chapter. Their powders are always the fastest on the planet — with promises of faster yet to come — when they have discovered the ultimate fast charcoal somewhere, somehow!

### **Moving On**

More will be said about competition grade powders later in this chapter and in the chapter on testing. We now move on to methods and techniques for making really fast powders.

To make high speed powder one must come to terms with the following:

- Milling is mandatory
- Corning is recommended and is usually necessary

Some may disagree with the above statements. However, centuries of Black Powder manufacture have shown these to be true - again and again and again.

### **Mandatory Milling**

About ten years ago I corresponded with a fellow enthusiast who was experimenting with making Black Powder. His one memorable comment to me was: "You must ball mill. The difference is as night and day." I ball milled — and the difference was just as he had described!

Some form of milling is mandatory if one wants to make fast Black Powder. There is just no way around this fact. Believe me, I have searched diligently for other methods that would obviate the need for milling. I have yet to find any. I have yet to find anyone who has.

I have tried the CIA method without milling. I have investigated heating up the sulfur and melting it into the charcoal. I have perused experiments done for the US military in exploring solvents that would dissolve sulfur. None of these have yielded any meaningful gains when compared with milling. So milling lives, like it or not.

Beyond hand milling with a pestle and mortar, ball milling is the method of choice for most. Thus when milling is described from now on in this chapter, ball milling is assumed unless stated otherwise.

### **Consider Corning**

Corning should be considered as advisable rather than an option. Besides creating Black Powder grains that can be used in regulating burn speed, corning also keeps the fine particles of potassium nitrate, sulfur, and charcoal from separating from one another. Thus corned powder is on the whole better powder. Even very fine meal powders are corned powders, as opposed to just dry mixtures of finely ground materials.

### **Milling Options**

So far we have established that milling itself is not an option if one wants fast Black Powder. But milling is dangerous no matter what method of milling is used. This is an unfortunate fact, but the dangers themselves can be reduced by choosing different options in the milling process itself. These options have been described in previous chapters. Here they are explored in more detail.

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The most dangerous type of milling operation is the milling of all three components of Black Powder together. Fortunately one does not need to do this to get a very fast powder. There are ways of avoiding three-component milling without compromising performance. These are:

- Single component milling
- Double + single component milling
- Double + double component milling
- Double component milling + dissolved potassium nitrate

### **Single Component Milling**

Single component milling involves milling the charcoal, sulfur, and potassium nitrate separately and then mixing them together.

Before describing this process, dispelling a common myth is in order here. This concerns three component milling and what actually happens during the milling process.

A common misconception is that milling sulfur and potassium nitrate together with charcoal has the effect of pressing the other components into the charcoal. This doesn't happen because the tiny holes (or pores) in the charcoal are too small to accommodate the sulfur and potassium nitrate particles. This pressing process thus happens neither during milling nor during subsequent pressing.

So what is there to be gained by milling all three components together if this effect does not happen? Plenty, because the milling process is also a mixing process. It is actually this mixing process that is the critical factor in ensuring that the Black Powder is properly incorporated. Understanding this concept is the key to understanding how good powder can still be made without three-component milling.

Milling typically takes longer than mixing. In fact proper mixing mostly takes place when each component has been milled fine enough to ensure good intimate mixing. This is one reason why those who favor three-component milling often opt for a pre-milling process of milling the individual components before milling them together.

It makes a lot of sense to mill the components individually for a long length of time and then mix them for a shorter time period. For example one could mill each component for three hours and then mix them together by milling them together for about an hour. This process still involves three-component milling, but for a shorter time period. The shorter this time period is the less chance there is of the mill exploding.

The just-mentioned method uses the ball mill in the final mixing stage, but unfortunately creates the situation where one has had to revert to three-component milling. This three-component milling is for a shorter time period but it is still three-component milling. Are there any alternatives? Yes, there are.

Mixing can be done by sieving the three components together. The more one does this, the more intimate the mix. Alternately, one can sieve the charcoal and sulfur until they are thoroughly mixed and then sieve them together with the potassium nitrate. The mixing process can also be varied by stirring the components together with a wooden spoon.

But just as ball milling beats hand grinding, doing the mixing in *a* ball mill beats doing it by hand.

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One may opt for another solution by doing the mixing in the ball mill but without the balls. This reduces some of the dangers created by the milling media but also reduces the mixing efficiency. Reducing the efficiency means having to increase the mixing time. Increasing the mixing time increases the danger of an accident.

Other problems can occur with single component milling. Sulfur for example, can build up a static charge if milled on its own. This charge is dissipated if charcoal is added to the sulfur and both are milled together. Potassium nitrate milled on its own presents another problem. The finely milled potassium nitrate particles have a tendency of clumping together if not mixed with another substance. These problems are addressed in the following sections.

## **Double + Single Component Milling**

This section discusses double plus single component milling. The double part is a mixture of charcoal and sulfur, while the single part is potassium nitrate. The charcoal and sulfur are milled together and then mixed with the potassium nitrate that has been milled on its own.

Note that this process only considers mixtures of charcoal and sulfur and not the other possibilities such as potassium nitrate and sulfur or potassium nitrate and charcoal. The reason for this is safety.

Before milling the charcoal with the sulfur, it is a good idea to reduce its particle sizes. A good way of doing this is to sieve the charcoal through a 50 mesh or finer sieve before mixing it with the sulfur.

This process reduces the chances of a static charge being built up on the sulfur but does not address two other important issues.

The first issue is the problem of the tendency of finely ground potassium nitrate to agglomerate. This is the process whereby the particles tend to clump together. This can be quite a serious issue, but it can be solved by mixing some charcoal with the potassium nitrate.

The second issue relates to the danger of spontaneous ignition when the finely ground potassium nitrate is added to the other components, also finely ground. I am really not sure how prevalent this danger really is. French powder makers seemed to think so. This caused them to opt for the solution described in the next section.

## **Double + Double Component Milling**

The perceived dangers in the last section can be got around by creating two double component mixes: potassium nitrate + charcoal, and charcoal + sulfur.

Potassium nitrate with charcoal is potentially nearly as dangerous as a three-component mix if the ratios of potassium nitrate to charcoal are in critical or near critical proportions. Typically these ratios vary between 4:1 and 6:1. Mixes in this range of ratios have the potential of igniting easily and burning very efficiently.

We get around this problem by increasing the ratio of potassium nitrate to charcoal to a ratio of 15:1. Thus if we are working with Waltham Abbey proportions we take one third of the charcoal and mix this with the potassium nitrate. The remaining two thirds are mixed with the sulfur.

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## **Double Component Milling + Dissolved KNO<sub>3</sub>**

Another option is to combine milling with the so-called CIA method. This method differs from the others in that the potassium nitrate is not milled at all; rather it is completely dissolved in water.

This method completely eliminates the need to mill potassium nitrate and also eliminates any dry mixing of the potassium nitrate with the other components. From a safety standpoint these are two big plusses. However, there are some downsides with this method.

The first downside is that one is trading one danger for another. So it's a case of picking one's poison. There have been some rather fierce and somewhat meaningless debates on this issue, with each side accusing the other of promoting dangerous practices. The bottom line is: all methods used to make Black Powder are dangerous, period!

Cooking up a Black Powder mixture at a temperature slightly higher than the boiling point of water can result in some painful scalding if some of the mix splashes onto one's bare skin. A worst case scenario of the mix igniting could result in a horrible fire, with horrible burns and damage to property. An explosion using this method is highly unlikely. An explosion from dry milling and mixing is a distinct possibility. So pick your poison - fire or explosion.

The second downside is one of accuracy. Some potassium nitrate is usually lost. This means that its ratio to the other components is reduced. This reduction will usually result in some loss of speed.

The third important downside is the issue of cost. Alcohol (even cheap alcohol), is expensive. And to do a proper job, lots of alcohol is needed.

Other negatives are that alcohol precipitation is tedious, time consuming, and messy.

The so-called CIA method is described in the chapter entitled *The CIA Connection*, together with my suggestions for optimizing this technique.

## **Pressing and Corning**

With the exception of the last-mentioned method that involves dissolving the potassium nitrate in water, all the powders are pressed with a minimal amount of dampening. Some dampening is needed to ensure proper pressing into pucks or pellets.

Some have suggested using just alcohol for this process instead of water or an alcohol/water mix. I don't recommend this practice because potassium nitrate is insoluble in alcohol. A small amount of water dissolves some of the fine potassium nitrate powder, causing it to bind together with the other ingredients. This is very important for the formation of viable Black Powder grains. If this water is kept to an absolute minimum then one doesn't have to worry about problems such as leaching out or the formation of large crystals.

To practically implement this dampening process one should add water in very small increments and mix the powder with a wooden spoon until it just starts clumping together. A good way of doing this is to place the water in a hand-held spray bottle like those used to hold window cleaner, and lightly spray the surface of the mix while mixing.

The dampened mix is then pressed into pucks or pellets using any of the techniques described in previous chapters. Some techniques will probably yield better results than others.

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The resulting pucks or pellets are then dried. I have dried mine for just over a day during the hot summer months. Others have reported drying times of a week or longer. There are no hard and fast rules here. Each person needs to find out what is best for them, in their particular circumstances.

The thoroughly dried pucks or pellets are corned using a variety of techniques. Some have gone to the trouble of acquiring or making machines similar to those used by commercial manufacturers, but most haven't. So crude and simple still rules the day here.

My preferred method is to place individual pucks between sheets of paper on a hard flat surface and run a pastry rolling pin over them, the same way that one rolls out pastry. I apply enough pressure to initially crack the pucks, causing them to crumble. I then repeat the process until the whole puck is reduced to small grains.

Others have placed their pucks or pellets on a hard surface under layers of plastic sheeting and struck them with a mallet. Another method is to place the puck or pellet in a press and slowly apply pressure until it crumbles. Note that this pressure should be applied slowly in a controlled manner. Too fast an application of pressure could result in the press acting in a way similar to an impact tester, igniting the powder in the process.

The resulting grains are then passed through sieves to give the required sizes.

### **Beyond Corning**

Black Powder that has gone through the corning process is ready to be used as is. No further processing is necessary. However, some improvements in performance can be gained by a process that is often referred to as *polishing*.

Polishing the powder grains rounds off their rough edges and yields a better consistency in grain shape. It also helps to pre-empt some breaking up of individual grains during transport and handling. This in turn may relate to better performance, depending on the application. Sporting powders are usually polished.

The polishing process can be carried out by simply tumbling the grains in a ball mill without the balls. The grains are tumbled until the desired finish is reached and then sieved to remove the fines.

Sporting powders usually have a small amount of graphite added during the polishing process. The graphite assists in making the powder flow better when loading and offers some protection against moisture. Such powders are known as *glazed* powders and are denoted with a *g* suffix, e.g. 2Fg.

Glazed powders are more difficult to ignite than their unglazed counterparts, and thus effectively have a slower burning rate. Glazed powder grain sizes are different to those used in unglazed powders. Thus a 2Fg powder is much smaller in size than a 2Fa powder. The same holds true for the other sizes such as 3Fg, 4Fg, etc.

### **Competition Grade Black Powder Revisited**

Having explored ways and means of making very fast powders that could comfortably perform as competition grade Black Powder, it is worth paying attention to the competition itself.

The Pyro Golf competition originated with a few Black Powder enthusiasts comparing different

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powders made with different charcoals. This stimulated more interest in homemade Black Powders. A catalyst was the discovery that it was possible to make Black Powders that performed as well as (or even better than) commercial powders such as GOEX. So Pyro Golf inspired a renewed interest in homemade powders.

The original Pyro Golf tests used 4 grams of powder per test. As the new generation of amateur powder makers improved their powders, 4 grams was found to be too much. This amount was reduced to 3.5 grams and finally to 2. From a competition standpoint, 2 grams of Black Powder is now considered the rule. Flight times are thus related to 2 grams, rather than the larger amounts previously used. This is important when comparing older results with their more recent counterparts.

Other rules that appear to be unchanged at the time of this writing relate to density and grain size. Both of these can have a significant impact on performance.

As a general rule, the lower the density the faster the powder. This conflicts with the once-cherished belief that high density powders burned faster. The inverse relationship between density and speed has been confirmed by many different tests done by different persons, at different times, under different conditions. The following tables give snapshots of some of these tests:

<b>Powder Type</b>	<b>Average Flight Times (in seconds)</b>	
	<b>High Density</b>	<b>Low Density</b>
<b>JF</b>	<b>11.90</b>	<b>13.91</b>
<b>TD</b>	<b>11.47</b>	<b>13.29</b>
<b>DM</b>	<b>8.19</b>	<b>8.86</b>

The above data shows a flight time variation of between 8 and 17 percent, the variation being defined as the difference between low density and high density powders.

<b>Powder Type</b>	<b>Average Muzzle Velocity (feet/second)</b>	
	<b>High Density</b>	<b>Low Density</b>
<b>Silver Maple</b>	<b>330</b>	<b>360</b>
<b>Aspen</b>	<b>360</b>	<b>380</b>

The above data shows a muzzle velocity variation of between 5 and 9 percent, the variation being defined as the difference between low density and high density powders.

<b>Powder Type</b>	<b>Average Peak Pressure (psi)</b>	
	<b>High Density</b>	<b>Low Density</b>
<b>Silver Maple</b>	<b>390</b>	<b>560</b>
<b>Aspen</b>	<b>460</b>	<b>600</b>

The above data shows a peak pressure variation of between 30 and 44 percent, the variation being defined as the difference between low density and high density powders.

<b>Powder Type</b>	<b>Average Muzzle Velocity (feet/second)</b>	
	<b>2F</b>	<b>4F</b>
<b>GOEX</b>	<b>200</b>	<b>250</b>
<b>NLC</b>	<b>220</b>	<b>420</b>

The above data shows a muzzle velocity variation of between 25 and 91 percent, the variation being defined as the difference between large grain and small grain powders.

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Powder Type	Average Peak Pressure (psi)	
	2F	4F
GOEX	84	130
NLC	170	730

The above data shows a peak pressure variation of between 55 and 429 percent, the variation being defined as the difference between large grain and small grain powders.

Powder Type	Average Flight Times (in seconds)			
	2Fa	2Fg	3Fg	4Fg
GOEX	10.09	12.59	12.66	12.87

The above set of data is interesting in that there is a 25 percent variation between the 2Fa and 2Fg powders but only a 27.5 percent difference between the 2Fa and 4Fg powders. This suggests that the influence of grain size drops dramatically after a certain point.

The above test snapshots demonstrate that significant differences in muzzle velocity, peak pressure, and flight time can occur with changes in density and grain size. This means that these variables need to be taken into account when comparing different powders. So where does this leave so-called competition grade powders? At the time of writing this it leaves them wanting.

Comparing two powders with significant differences in density is like comparing apples and oranges. The same can be said about comparing powders with different average grain sizes. And here I am not merely referring to different grades such as 2Fa and 4Fa. Differences can occur even within the grade size itself. The following data should help to illustrate this point:

Grade	Largest Grains (inches)	Smallest Grains (inches)	Ratio (largest/smallest)
Fa	0.3125	0.157	1.99:1
2Fa	0.187	0.0661	2.83:1
3Fa	0.0787	0.0469	1.68:1
4Fa	0.0661	0.0331	2.00:1
Pyro Golf	0.0937	0.0661	1.42:1

The above data show ratios varying from 2.83:1 to 1.42:1. In fairness to the Pyro Golf competition ratios, these yield a closer grain size than any of the others shown. However, a ratio of 1.42:1 can be improved upon to yield more meaningful results.

An improved ratio of about 1.19:1 can be realized by using two adjacent standard sieve sizes in the ranges of No.8 (0.0937 inches) to No.20 (0.0331 inches).

## **GOEX Black Powder Revisited**

It has become common practice to compare homemade powders to GOEX, with oft-repeated claims of being *faster than GOEX*. At one time such claims were met with a certain amount of skepticism. This is no longer the case. Many of such claims are valid.

Even laying *aside fudge factors* such as tweaking densities and grain sizes to give faster speeds than GOEX, many claims of faster powders are still legitimate. Why is GOEX slower? And for that matter, why are other commercial powders slower?

Part of the answer to these questions is that commercial manufacturers don't aim for the fast-

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est powders on the planet. They have other important objectives such as consistency from batch to batch and powders that have good ballistic properties.

I recently spoke to a representative from GOEX who has been in the Black Powder making business for close on forty years. His father before him was also a Black Powder man with long years of service in the industry. He commented on the fact that faster is not necessarily better. He illustrated this with examples from sporting grade powders that compromised accuracy when made to perform slightly faster than normal. And no, one cannot always just reduce the amount of powder if the powder burns faster. The science of ballistics is a bit more complex than that.

Could GOEX make faster powders? They certainly could by perhaps using different charcoals and reducing the densities of their finished product. They could also be creative in changing their grain sizing. But to what end?

Such an end result could be faster powders that compromise other characteristics such as consistency from batch to batch, resistance to moisture absorption, and resistance to crumbling when handled and transported. Such considerations may not be important to an amateur experimenter, but are very important to commercial manufacturers and many of their customers.

So no, there is no conspiracy among commercial manufacturers to keep the speed of their powders down, thus forcing helpless consumers into buying more of the stuff.

### **Conclusion**

This chapter has described ways and means of making fast Black Powder. It has also shown that such powders can meet or exceed the speeds of those found in commercial powders. And it has shown that making fast Black Powder is a relatively simple process.

This last point may have a peculiar significance to those who have been hoodwinked into thinking that the secrets of fast Black Powder belong to those who have spent many years in perfecting the art. Black Powder making is not a black art. Conversely it is not rocket science. Its secrets are not closely guarded by a small group of luminaries. It can be made by anyone who is willing to apply themselves in a disciplined and common sense way.

## *Chapter 11 — Black Powder on a Shoestring*

### **Introduction**

This chapter focuses on ways and means of reducing the cost of making Black Powder. We can make cuts both in the price of materials and equipment. We begin by looking at cheaper materials and then focus on different types of equipment.

The first edition of this book highlighted some very cheap and rather nasty pieces of equipment. I have deliberately omitted these for a number of reasons. One reason was that some of these were only suitable for very small batches of Black Powder. Another reason was the fact that Black Powder making among amateur pyrotechnic enthusiasts has matured somewhat since then, leaving some of these methods more in the realm of pyrotechnic nostalgia than in modern-day usefulness.

A chapter that focuses on cheaper ways and means would be incomplete without a mention of the more expensive ways of doing things. These more expensive ways are mentioned throughout the chapter, but I've focused on specific places to avoid right after this introduction.

### **Expensive - A Path That is Best Less Traveled**

Certain places are by their very nature more expensive. Their goods are more expensive. Their services are more expensive. Their quality may or may not be any better. And even if their quality is better, it still may not justify the extra cost. When it comes to pyrotechnic equipment and materials, certain places are best avoided if one wants to save money. The first of such places is anything that claims to be a *laboratory* supplier.

The word *laboratory* can be regarded as code for the word *expensive*. So can other words such as *professional*, *scientific*, *aircraft*, *medicinal*, *organic*, *pharmaceutical*, and *medical*. This list is by no means exhaustive, but covers most of those that threaten to separate the aspiring Black Powder maker from money that could be spent more productively.

Back to the word *laboratory*. Laboratory suppliers deal in a whole lot of useful equipment including scales, ball mills, and sieves. Some supply a wide variety of chemicals, including those used to make Black Powder. Laboratory equipment is never cheap, and laboratory grade chemicals are too expensive for most Black Powder uses.

The only piece of lab equipment I have invested any large amount of money in is a good scale for weighing chemicals. I have gone to cheaper suppliers for other equipment, including sieves. One exception to this rule is a 40 mesh sieve that I bought from one lab supplier's stock clearance sale. I got this at a very good price, and I would never have even considered paying full list for it.

Laboratory ball mills are often outrageously expensive, and one may even end up paying more for just a milling jar than a complete mill with jar(s) elsewhere. The same can be said for some of their milling media.

I learned in my early days in electronics that the word *professional* often meant high prices for components that could be bought a lot cheaper elsewhere without the professional label. Some-

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times *professional* can mean better value for money, but often it is not. As a general principle, take time to do some comparison shopping with anything that is sold under the *professional* label.

The word *scientific* is often just another word for *laboratory* when it comes to equipment and chemical supplies.

Nearly everything sold to the aircraft industry is expensive. Even food sold within close proximity of aircraft is expensive! So don't be lured into anything aircraft *going cheap* that maybe someday will be useful pyrotechnically. Chances are good that one could buy the same equipment much cheaper elsewhere. To date I haven't come across any aircraft equipment that may be useful in pyrotechnics, but who knows?

Anything related to the well being of the human body somehow finds its way into the hands of those who both ask (and get) exorbitant prices for it. So anything *medicinal*, *organic*, or *medical* should be given a miss. Related to this is *pharmaceutical*. Pharmaceutical grade chemicals (e.g. BP and USP) are an overkill in Black Powder and are very expensive when compared to technical and agricultural grades.

In the days of my youth, my friendly neighborhood pharmacist was the source of most of my pyrotechnic equipment and chemicals. (At this point I won't describe the not-so-friendly neighborhood pharmacists.) I found out later that a lot of the stuff I was getting from him came from the laboratory suppliers, with his own no-too-modest markup. So bear in mind that anything one's local pharmacist needs to order specially will come at a price even higher than those asked by the lab suppliers.

Having discussed expensive sources of equipment and chemicals, we move on to a rather gray area: suppliers who are both cheap and expensive at the same time. Perhaps the most prominent of these, from a Black Powder perspective, are pottery suppliers and pyrotechnic suppliers.

Pottery suppliers offer equipment such as ball mills and sieves, and also some chemicals that have pyrotechnic applications. The chemicals they supply are not usually in the Black Powder category, so these will be ignored here. Ball mills from pottery suppliers are generally laboratory types. Sometimes they offer tumblers used by lapidary enthusiasts to polish stones.

Ball mills from pottery suppliers generally will cost as much as those obtained elsewhere, and maybe even more. The same can be said for some pottery suppliers who sell scales. Sieves are a different story. Generally I have found their sieves to be a lot cheaper than their laboratory counterparts.

The world of those supplying pyrotechnic supplies to professionals and amateurs is filled with contradictions. Some are cheap on chemicals, but expensive on equipment. Others are the opposite. At one time some drew a distinction between *fringe* suppliers who advertised exclusively in popular magazines such as *Popular Mechanics* and *Popular Science*. These were expensive when compared to those who advertised in pyrotechnic publications such as *AFN* and the *PGI Bulletin*. Today this distinction has become blurred, and those who were demonized in the past as being expensive are not necessarily so any more.

### **Cheaper Potassium Nitrate**

Potassium nitrate is the most expensive component in Black Powder; typically it costs more than sulfur and charcoal. More potassium nitrate is used than both sulfur and charcoal combined. Cutting the cost of potassium nitrate is significant in cutting the cost of Black Powder.

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The second chapter of this book looked at different grades of potassium nitrate. Part of its focus was on grades that were substantially cheaper, these being technical and fertilizer grades. Both technical (a.k.a. industrial or commercial) grades and fertilizer grades have been used successfully in making Black Powder. Some have had better success than others.

I used to think that most fertilizer grades were inferior to technical grades. However, after comparing the specifications of a number of different technical and fertilizer grades, I have come to a different conclusion. While some fertilizer grades are inferior (in terms of purity), others are not. It just depends on which brand one chooses to compare with another.

For example, KPOWER, a popular fertilizer grade in the USA boasts a purity of 98.9%. Other suppliers of fertilizer grades boast a purity of between 98 and 99%. Technical grades usually claim purity levels of greater than 99%, with some purity levels at 99.8% and even 99.9%. But some technical grades only claim a purity level of 95%, as do some fertilizer grades. So while in general technical grades are purer, in certain instances a technical grade may be less pure than a fertilizer grade with which it is being compared.

Where does one get the stuff? Technical grade pyrotechnic chemicals are supplied by companies that cater specifically for those requiring pyrotechnic materials. Most of their chemicals are technical grade. If one wants to save money, one must buy in bulk. The best places to do this are the companies that sell in bulk to the pyrotechnic suppliers. These are either the manufacturers themselves or their agents.

Fertilizer grade potassium nitrate can be found in stores that supply fertilizer to the agricultural community, i.e. farmers. The average city hardware store or supermarket rarely stocks potassium nitrate in its fertilizer section. They typically sell fertilizers that are mixtures of different chemicals.

### **Cheaper Sulfur**

Sulfur comprises only about 10% of a typical Black Powder mix. Compare this with about 75% potassium nitrate, and one sees that the influence of sulfur's cost on the total materials cost is minimal. So while one may achieve substantial savings by buying cheap potassium nitrate, one cannot do the same with sulfur. Still, one may not wish to pay too much for the product. This may be especially true if the cost of 10% sulfur approaches that of 75% potassium nitrate! Sulfur is found in abundance, both in nature and as a by-product of various chemical processes. And it does come cheap if one does not require it to be very pure.

At one time I shunned cheaper sources of sulfur. In those days I was an idealist, looking for sulfur with minimal acid content. I first tried to get what is described in pyrotechnic literature as *sulfur flour*. When I could not lay my hands on this, I purchased some rather expensive *precipitated sulfur* from a pharmacy. Later I experimented with other less pure forms of sulfur, including dusting sulfur bought cheaply from a local nursery. This was 98% pure. I then crossed the line to the much maligned *flowers of sulfur*, notorious for its acidity.

Dusting sulfur worked and so did flowers of sulfur. I decided then that the type of sulfur was not that important in Black Powder. My findings seem to be confirmed by others. Some reported using sulfur with purities as low as 90% and even 86%. I tend to get a bit uncomfortable with impurities in the range of 10 - 14%, especially if these impurities are not identified. But 10 - 14% impurities in sulfur translates to roughly 1 - 1.4% overall in the Black Powder mix.

Black Powder made with flowers of sulfur will tend to attract more moisture due to its acid content. This could be a disadvantage if one plans to store the powder for any length of time. Other impurities in sulfur could have a similar detrimental effect or may have some other, perhaps unnoticeable, effects. One can usually get away with using cheap sulfur of dubious purity.

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## **Cheaper Charcoal**

As a general rule, I do not advise anyone to look around for the cheapest available charcoal. Good Black Powder requires a good charcoal, and good charcoal that is suitable for Black Powder does not come cheap. I prefer to think of charcoal as being expensive and less expensive, rather than cheap and cheapest.

Commercial manufacturers of charcoal do not supply charcoal that is optimal for Black Powder use, so one is either forced to make one's own or buy from a supplier of specialty charcoals. From a cost point of view, one's only option if one wishes to buy it is to buy it from a specialty supplier of pyrotechnic charcoals. Other suppliers of specialty charcoals are prohibitively expensive. A few of pyrotechnic charcoals have sprung up in the USA in recent years. To date, I am not aware of any elsewhere in the world.

If one decides to make one's own charcoal, then one can look around for cheap sources of fuel to get the job done. One of these sources may be charcoal, the type one buys in the supermarket.

And one can make one's own charcoal very cheaply. The chapters that described charcoal earlier on may have obscured this fact. If one is not too ambitious in the size of the charcoal making operation, one can make charcoal in something like a coffee can or paint can. And if one does not aspire to optimizing the process, all the expensive apparatus needed for good temperature control can be ignored.

When it comes to making charcoal cheaply, small is often beautiful. Small means smaller and cheaper containers used for the retort, less fuel to heat the material, and a minimal amount of material itself.

Expensive temperature control equipment? Ill let you into a little secret. Most Black Powder makers who make their own charcoal don't bother to control its temperature. They may hint that they do, but in reality all they do is attempt to supply heat that is not excessive. This can be done by simply surrounding the retort with hot charcoal coals. I have successfully made charcoal by building a small wood fire around a coffee can retort.

Naturally there are those who do aspire to optimize their charcoal by controlling its distillation temperature. Less expensive methods of temperature measurement and control are explored later in this chapter.

## **Cheaper Alcohol**

Yes, one can write an article under this heading for any number of publications and be guaranteed an audience. Cheaper alcohol is attractive to many, and is usually gained by one of two time-honored routes:

- Buying alcohol free from government-imposed tax restraints.
- Brewing and distilling one's own.

Both of the above could land one in trouble with the law, so I'm not suggesting ways and means of getting alcohol in any illegal fashion.

The second choice is a definite no-no in this discussion. Legal considerations aside, it is just too much trouble to brew and distill one's own alcohol. Concerning the first choice, there are ways to buy alcohol free from the high taxes that are normally imposed on ethyl alcohol. Ethyl

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alcohol is the type of alcohol found in alcohol beverages such as beer, wine, and spirits. This subject is dealt with later on in this section.

The most popular type of alcohol used in Black Powder making is isopropyl alcohol. Popular here (as in many other instances) does not necessarily relate to better, rather it relates to cheap and being easily available. And isopropyl alcohol is cheap and easily available if one lives in the USA. It can work out expensive and harder to get in other places.

The cheapest type of isopropyl alcohol contains 70% alcohol. This means that it is less effective for alcohol displacement methods such as the so-called CIA method, but probably good enough. Higher purity isopropyl alcohol, such as 90%, 95% or 100%, costs a lot more. The lower purity 70% isopropyl alcohol is often subject to sales pricing in both pharmacies and supermarkets, making it even cheaper if one waits for such opportunities.

Isopropyl alcohol is often referred to as rubbing alcohol, and usually comes with a 70% alcohol content. Here one needs to be a bit careful when buying *rubbing alcohol*. Some rubbing alcohol is not isopropyl alcohol at all, but rather ethyl alcohol with added ingredients aimed at having a soothing action on the skin. And some isopropyl alcohol based rubbing alcohols contain similar ingredients. Such ingredients may add some unwanted variables to the Black Powder mix, and it is best to avoid them. Rubbing alcohols with such enhancements are usually more expensive anyway.

Other common forms of alcohol are methyl alcohol, a.k.a. methanol or wood alcohol, and ethyl alcohol. Ethyl alcohol is the alcohol found in alcoholic beverages and is also referred to as ethanol.

Methyl alcohol is usually expensive and its purchase is often restricted. I have yet to find a cheap methyl alcohol that is freely available. But that is just my experience. It may be different in some parts of the world. Just a word of warning about methyl alcohol: it is poisonous, far more poisonous than ethyl alcohol. Its best to minimize one's exposure to methyl alcohol, both on one's skin and through breathing in its fumes.

I promised to describe cheap sources of ethyl alcohol, and what follows is a fulfillment of that promise. I did have in mind cheap ethyl alcohol for pyrotechnic purposes. Sorry if any of my readers thought differently!

The cheapest and most easily available form of ethyl alcohol comes in a form known as denatured alcohol, a.k.a. methylated spirits. Denatured alcohol is ethyl alcohol that has a small percentage of other substances mixed with it. This percentage is usually somewhere in the order of 5%. These substances are added to the alcohol to discourage people from drinking it. With most people this strategy works pretty well. With most that is. Some sectors of society, politically correctly known as *the homeless*, still regard denatured alcohol as their beverage of choice.

Denatured alcohol can be obtained very expensively or quite cheaply, depending on how one buys it. The stuff sold in pharmacies should be avoided, so should denatured alcohol used as fuel for fondues. The type to look for is usually found in hardware stores in their paint section, where it is sold as a solvent.

### **Cheaper Water**

No, this is not a joke! And I never thought that this topic would make the pages of this chapter. That is...not until recently.

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Water can be had very cheaply or very expensively. The difference is between being sensible or being gullible.

Years ago one of my cousins told me about a place she had visited in Europe where drinking water cost more than wine. I found the story rather difficult to believe at first, having been raised in an environment where tap water was the only water we drank. Bottled water was not available. And even if it were, it would have been a non-seller.

I reasoned at the time that the issue of bottled water related more to undeveloped countries where good drinking water was hard to come by, or to rural backwaters in developed countries. I was wrong. I just did not understand the fetish that millions had for bottled drinking water, or the clever marketing that had created it. If the truth be told, much of the bottled water that is drunk by the millions of gallons every day is no better than ordinary tap water.

A similar fetish exists in pyrotechnic circles where distilled or de-ionized water is regarded as better than tap water. Certain pyrotechnic applications such as color compositions require very high levels of purity. This purity can be compromised by water that has a high mineral content. Here one could be justified in using distilled or de-ionized water.

In my judgment, one should only use distilled or de-ionized water in making Black Powder in the following situations:

- 1) The local water supply is highly suspect. This situation is often true with local well water. It is usually less of an issue with potable water from municipal utility suppliers.
- 2) One is making experimental Black Powder under laboratory type conditions, and desires high experimental accuracy.

The second point is worth noting. I have heard of some experimenters using dusting sulfur and fertilizer grade potassium nitrate mixed with distilled or de-ionized water. What a waste! Purer water mixed with less-than-pure essential ingredients achieves nothing. The thinking behind such actions probably lies more in the realm of superstition and alchemy than in true science.

So the bottom line is - use tap water unless you have good reasons to justify using the much more expensive alternatives.

### **Weighing Cheaply**

An essential piece of equipment for anyone wanting to make Black Powder is an accurate scale. There is no way to get around this requirement. Measuring quantities by volume is just too inaccurate and unpredictable. So one needs some kind of scale that will give reasonably dependable accuracy.

A general rule in any type of measuring device is: the better the accuracy the higher the price. Scales (a.k.a. mass measuring devices) are no exception. High accuracy scales come with high price tags. And anyone serious about experimenting with pyrotechnics should regard investing in an accurate scale as a rite of passage. But one has to start somewhere, and there are cheaper entry level alternatives.

Today I own a very accurate type of scale known as a *triple beam balance*. Mine is actually slightly better than the normal triple beam in that its third beam is replaced with an accurate vernier dial. This allows me to weigh substances to within one hundredth of a gram. This is quite an overkill for Black Powder work, but could be justified with other pyrotechnic mixtures made in small quantities. To tell the truth, measuring to within one tenth of a gram is good enough for most pyrotechnic use.

## BLACK POWDER MANUFACTURE, TESTING & OPTIMIZING

I did not start out with a triple beam balance. I started my pyrotechnic exploits with something a lot simpler.

My first scale was a simple balance used to weigh letters, i.e. a postal scale. This was a true balance where one placed the object to be weighed on one side and weights on the other. I did not have weights. I used small tacks instead. This system, although primitive, actually worked quite well.

My next scale was a photographer's chemical scale. This could weigh substances to the nearest gram. It was certainly good enough for small batches of Black Powder in the order of about 500 grams per batch. Why not smaller batches, like 100 grams?

It could have been used for smaller batches but with a loss of accuracy. In a 100 gram batch, for example, I could have weighed everything to the nearest gram. Unfortunately my 10 grams of sulfur could have ended up as either 9 or 11 grams. If I had worked carefully I could have maybe reduced the gap to between 9.5 and 10.5 grams. The same kind of problem would have been encountered with the charcoal. And, naturally my potassium nitrate could have been out between half a gram and one gram, except here the relative inaccuracy would be lower.

Weighing to the nearest gram or half gram restricts one somewhat in pyrotechnics, even if one's pursuits don't go beyond Black Powder. So I recommend a scale that weighs to within one tenth of a gram or better for serious Black Powder experimentation. These constraints, however, may not apply to the *entry level* experimenter.

Finding a cheap scale that weighs to within one gram is a challenge in itself. Finding one that weighs to within a fraction of a gram is even harder. The good news is that there are affordable solutions out there. One just has to find them. Here are several options:

- Digital electronic scales
- Reloader scales
- Kitchen diet scales
- Toy scales

An observant reader may have noted that I have omitted photographer's scales discussed earlier. This is because these are no longer available. Photographic chemicals have moved from being supplied in powders to being supplied in liquids. Another omission is postal scales. Most of those found today are not the sensitive balance type that I started out with. A typical resolution on these scales is 2.5 or 5 grams. Prior to writing the first edition of this book I did manage to buy a really cheap postal scale that measured in one gram increments. I haven't seen any since then.

**Digital electronic scales** can work out cheaper than a triple beam balance and still give comparable accuracy. One needs to shop around a bit here. Many sell for \$100 or more and only measure to within one gram.

I recently saw a good buy at a *rock shop*, which sold a variety of scales for weighing gemstones. One of these was a small digital scale. It weighed in tenth gram increments and cost \$70.00, the best price I have seen so far for a small digital scale with this accuracy.

**Reloader scales** (a.k.a. powder scales) are used by firearms enthusiasts for reloading ammunition. These usually do not weigh in either ounces or grams, but in a unit of weight called grains. One grain is equal to 1/480th of an ounce. Such scales can be very useful for weighing small batches very accurately but could be a real pain for larger amounts.

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Such a scale has a typical range of 500 grains, just over one ounce. Some have a maximum range of double this amount.

Powder scales come in both mechanical and electronic versions. The mechanical types cost upwards of \$30.00 and the electronic types upward of \$100.00. Some are extremely accurate and can weigh to within a tenth of a grain.

**Kitchen diet scales** typically read to the nearest five grams. This is not good enough for small batches of ingredients, so it is best to try and modify a diet scale to read in lower increments. One can do this by changing its spring for one with a lower tension.

**Toy scales** are just that, toys. But they can still be used if one is prepared to suffer some loss of accuracy.

I admit that I was skeptical about any scale designed for kids. Those I was familiar with were the crude scales used to teach arithmetic, and crude toy kitchen scales. I changed my mind when I tested a small scale that came with my daughter's chemistry set. This weighed quite accurately. Maybe it was a bit unfair to label this as a mere toy. Other toy scales? Some may work. Those used for teaching arithmetic are probably the best bet. These scales usually come without scale pans so one may need to be a bit creative here.

### **Ball Milling Cheaply**

Owning a ball mill could make a sizable dent in one's pocket. This dent could range in size from a hundred dollars to several thousand. Even a hundred dollars is quite a hurdle to many. Fortunately the entry level costs to ball milling can be very low. One can make a simple ball mill from, scrap components and end up laying out only a few dollars or even nothing at all!

When it comes to high costs in ball milling, the bad news does not end at the ball mill itself. Milling jars can be expensive, some very expensive. And then there is the cost of the milling media. This can work out very expensive if one uses the wrong type. The *right* type can also be costly if bought from an expensive source.

The cheapest route for most is to make one's own ball mill, including the milling jars. Some types of milling media, such as lead balls, can also be homemade. At the time of writing this, the best source for such information is *Ball Milling Theory and Practice for the Amateur Pyrotechnician*, by Lloyd Sponenburgh. Lloyd gives information on how to make an inexpensive ball mill, with inexpensive milling jars. He also tells one how to cast one's own media, plus a whole lot of other useful information.

His ball mill is inexpensive when compared to its laboratory counterparts. It is, however, a bit costly in terms of both time and money for many entry level users of ball mills. And for those who are looking for cheaper alternatives, read on.

### **A Cheap Junk Box Ball Mill**

I had hankered after building a ball mill for a long time. And I knew that sooner or later such a device would become indispensable. I took the plunge one day when I realized that I had reached a ceiling in my experiments with the so-called CIA method. I couldn't make a Black Powder that burned faster than 15 centimeters per second in linear burn rate tests. I knew I just had to ball mill, and I just had to own a ball mill. I got together some junk lying around, and soon had one.

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My ball mill was very inefficient, but it was cheap! In fact it cost me nothing in terms of money, just in time. And it took next to no time to build it.

I had a small motor that had once served to drive a turntable in the days when music was recorded on vinyl disks known as records. Today these have been superseded by CDs. This motor turned at a rate of about one revolution per second. This was, I figured, about the right speed that the mill should rotate at, and decided on a direct drive system.

For a milling jar, I decided on a round freezer container. This had a screw-type lid. Such a lid would have presented a hazard had I opted for milling all three components at once. I reckoned that I would only need to mill the sulfur and charcoal and kept the jar with the screw-type lid. I found that the jar sat snugly in a one kilogram (two pound) coffee can. The other scrap components were a few pieces of wood, and two wheels from a discarded walker for toddlers.

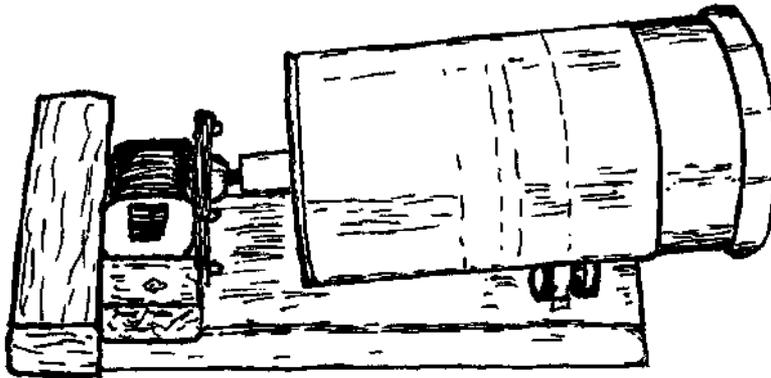


Figure 11-1. Ball mill - side view

Coupling the motor to the coffee can presented the biggest challenge. I had a round piece of plastic about an inch in diameter with a hole running through its center. This hole fit snugly on the shaft of the motor. I was lucky to have two snugly fitting components! This in itself saved me quite a bit of extra effort.

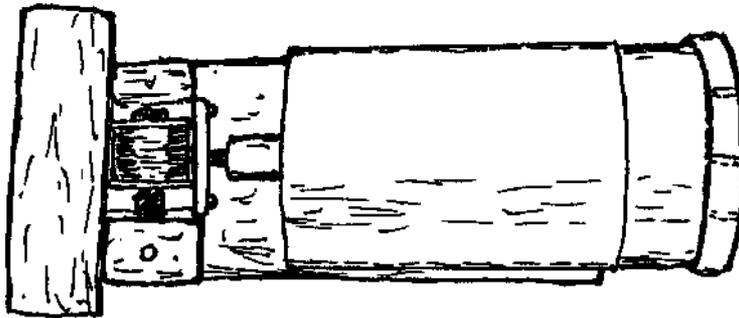
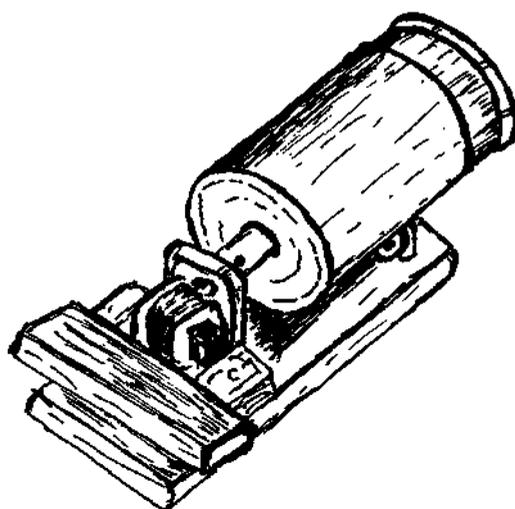


Figure 11-2. Ball mill - top view

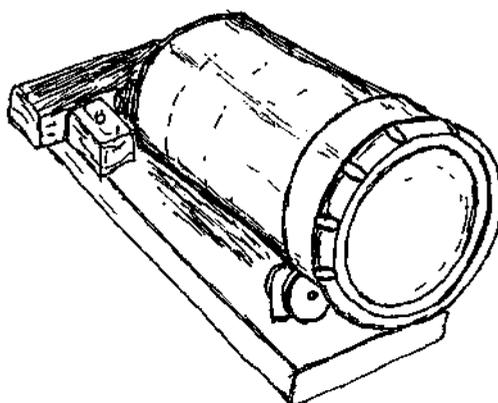
I glued the piece of plastic to the center of the bottom of the coffee can with a strong epoxy glue. This ended up being an unsatisfactory arrangement, as the piece of plastic kept separating from the can, no matter what gluing strategy I tried. Eventually I drilled some small holes in the bottom of the can and screwed the plastic onto it. This arrangement held up for the many hours that I ran the mill.

Although small, the motor was heavy enough to anchor the mill on one end. In fact, I mounted it loosely between three blocks of wood connected together in a U shape. The other end of the mill rested on the two wheels.



*Figure 11-3. Ball mill- back view*

I used some small round lead fishing sinkers for media. The motor tended to overheat, so I ran it on a timer using 15 minute on/off intervals. This mill was inefficient and took forever to mill a good powder. But it cost me nothing, and was knocked together in no time. That made its price versus performance ratio better than anything else around.



*Figure 11-4. Ball mill - front view*

Some may shudder at such an inefficient ball mill. My philosophy was: "Why wait until one has more money, time, or both before indulging in the joys of ball-milled powder!"

### **Cheaper Sieves**

Before looking at cheaper sources for sieves, let us look at the most expensive source so we can immediately cross it off our list. The most sieves around are those sold for laboratory purposes. Admittedly these are made to very accurate specifications, which in some way justifies their high cost.

Laboratory sieves often come in sets that can be stacked on top of each other. They can also be bought singly. I have bought one such sieve in my life. It was a small 40 mesh sieve that was being sold on a lab clearance sale. For me, buying this was the exception rather than the rule.

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I have bought both 80 mesh and 100 mesh sieves from pottery suppliers. Such sieves are typically used to sift glazes used in making pottery. One pottery supplier listed a 200 mesh sieve in one of her catalogs, but was out of stock. I would have happily bought this as well, had it been available.

Some of the pyrotechnic suppliers sell sieves at reasonable prices.

Another route is to use ordinary kitchen sieves. Typically these come in the larger mesh sizes, making them suitable for granulating Black Powder. From a safety perspective, they may pose a hazard, especially if one uses them for sieving dry powder. This is because they are made of plated steel, which may spark if rubbed against certain other hard substances. Here it is best to use a wooded spoon or spatula if one needs to use in implement in conjunction with the sieve.

I have personally had no problem with kitchen sieves. I have been careful to sieve only small quantities at a time, and then at a safe distance from any other containers of Black Powder. But I have still stuck to the belief the operation was more hazardous than with other types of materials.

The kitchen sieves I have just described were made of steel. Some kitchen sieves are made from plastic. Plastic could pose a *hazard* in the form of static electricity. Here I would err on the side of caution and not pass dry materials through them.

There are many amateur accounts of using window or door screens for granulating Black Powder. Some of these are very easily removed from windows and doors, allowing them to be used for both their original purpose and for making Black Powder. At the time of writing this, a disease called the West Nile Virus has swept across the United States and has just reached the west coast. This disease is carried by mosquitoes, and such screens are designed to keep both flies and mosquitoes out. So maybe one should take into account this additional hazard as well.

Making one's own sieves is certainly a viable option. All one needs is some sieve material and something to stretch it over. Such material is obtainable in various sizes and types such as copper, brass, bronze, and stainless steel. A well known supplier in the USA is McMaster Carr.

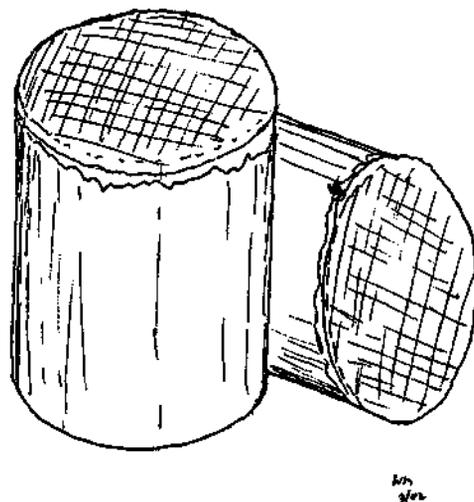


Figure 11-5. Coffee can sieves

## BLACK POWDER MANUFACTURE, TESTING & OPTIMIZING

One method of making a sieve is to cut the bottom off a tin can and stretch the material across it. The material is then held in place with a piece of wire wrapped around it, with its ends twisted together.

Another way to make sieves is to stretch the material across an embroidery hoop. Such hoops can be bought for a couple of dollars or less, and are typically made of wood, metal, or plastic. A disadvantage of such a method is the shallowness of the sieve. Embroidery hoops could also be combined with the tin can method, with the outer hoop being used as a clamp rather than using a piece of wire.

### **Cheaper Temperature Control**

To optimize the performance of homemade charcoal, one needs to control its temperature in a meaningful way. Unfortunately temperature control can be expensive. Just a simple temperature probe or gauge to measure the temperature can cost one the same amount of money as a scale or ball mill. And that is just the cost of the measuring device. An electronic controller can cost even more. Cost is probably the major reason why very few attempt to control temperature when making charcoal. At best, control is limited to reducing gross overheating.

But there are cheaper ways of measuring temperature, much cheaper. And if one has time to spare controlling temperature manually, one can do without an expensive electronic controller. Here's how.

There are two inexpensive options in measuring temperature in the range needed for charcoal making. One is an inexpensive dial gauge. The other is an inexpensive thermocouple. Both of these options can be very expensive if the wrong choice is made, but quite inexpensive if one follows the right strategy.

Very cheap dial gauges are available as kitchen thermometers. Unfortunately none of these read a high enough temperature for charcoal making. Ideally such a gauge should read up to 400 °C (750 °F). These are more difficult to find. I found one such gauge at a hardware store, which was used to measure temperature in gas-fired barbecues. Unfortunately its probe was less than an inch long, too short for practical use in making charcoal. Some instrument suppliers stock gauges in the required range. With luck one can find a cheap low-end gauge with the right range. Some gauges I have looked at have an upper limit of 700 °F. They should work for most charcoal making experiments.

Thermocouples can be had in prices ranging from a few dollars to many hundreds of dollars. The very high price thermocouples are typically used to measure very high temperatures, and are made from very expensive metals such as platinum and rhodium. Thermocouples used at lower temperatures are typically made from less expensive metals such as iron, copper, and nickel. Fortunately charcoal is made in this lower temperature range.

The two most popular (and also least expensive) types of thermocouple are:

- **Type J** - also known as iron-Constantan or iron - cupro - nickel. Some use chemical symbols to refer to a type J thermocouple as a type Fe - CuNi.
- **Type K** - also known as nickel-chrome nickel, or NiCr - Ni.

Type J thermocouples are typically used to measure lower temperatures than type K. Both will work in charcoal making. In the past type J seemed to dominate the market more than type K. However, in recent years I have noticed more and more type K thermocouples being supplied as generic temperature probes. So it is probably easier to find an inexpensive type K than a type J. I have found some type K thermocouples selling for as little as about twelve US dollars.

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It is worth shopping around for thermocouples. One doesn't need an expensive thermocouple for charcoal applications. Some have got by with an ultra cheap approach by just twisting a pair of thermocouple wires together to make their own thermocouple. This can even be done by twisting together the ends of a thermocouple extension lead. The wires in these leads (for type J and K thermocouples) are made from the same materials that are used in the thermocouple itself. This is the ultimate cheap thermocouple, but comes at the expense of being less accurate than one made from actual thermocouple wire.

Unfortunately a thermocouple requires an electronic instrument to display the temperature it is reading. These can work out to be quite expensive if one is looking for an instrument that measures temperature directly. A much cheaper solution is to use an instrument that measures milli-volts. The milli-volt value is then converted to a temperature value. Fortunately many of the cheaper digital multimeters that are now available will do this.

To measure temperature very accurately with a thermocouple one needs to measure milli-volts to at least the second decimal place, i.e. to within one hundredth of a milli-volt. Most cheap digital multimeters cannot do this. The best they can do is to measure within one tenth of a milli-volt. This gives a less accurate temperature reading but is probably good enough for less-than-fanatical charcoal making. Another issue affecting measurement accuracy is that of temperature compensation.

A thermocouple's output voltage is relative to a differential temperature rather than absolute temperature. In practice a thermocouple usually measures the difference between the temperature it is measuring and ambient temperature. Ambient temperature is normally considered to be in the order of 20 °C or 68 °F. Tables that give values of thermocouple voltages relative to temperature are often referenced to these ambient temperature values. This means that if the ambient temperature is at this value, then the output measured at the ends of the thermocouple cable corresponds directly to the value in the table. If the ambient temperature is lower than the reference value, then the measured temperature is correspondingly lower. If it is higher, then the measured temperature is correspondingly higher.

For accurate measurements one thus needs an independent method of measuring ambient temperature. Fortunately this can be done with any inexpensive thermometer used to measure room temperature.

Recently I priced an inexpensive multimeter from Harbor Freight. This sells for under forty US dollars and comes with a type K thermocouple. It reads temperature directly. This is about the cheapest of such devices currently on the market, and is possibly the best solution for many.

I wish I could offer a cheap automatic control solution, but I can't. Temperature controllers tend to be expensive, even for the cheaper types. An alternate solution is to control the temperature manually. This method is discussed in the second chapter on charcoal.

Before leaving this discussion on thermocouples, I need to put in a word of caution about some of the so-called thermocouples that are sold in hardware stores and similar outlets. These are typically used as safety devices for domestic and industrial heating equipment that uses gas. The *thermocouple* used here is merely a type of temperature switch that cuts off the gas supply if the pilot flame goes out. Some of these are sold as *thermocouplers* rather than *thermocouples*. Unfortunately most are sold as *thermocouples*. How does one tell the difference?

A true thermocouple always has two wires made from dissimilar metals, usually coded with different colors. In type J thermocouples these colors are red and white, and in type K yellow and white. The white wire is the positive wire.

## *12 — The Chemistry of Black Powder*

### **Introduction**

This chapter is based on an article I wrote for the *Journal of Pyrotechnics*. I have scaled the original article down somewhat to pitch it at a level more consistent with the rest of this book. Nevertheless, the materials in this chapter are more for the *heavies* who aim to know everything there is to know about Black Powder. One can make good Black Powder by skipping this chapter entirely. If you are one of the *heavies* or a *wanna-be heavy*, or just curious, then read on. Otherwise go to the next chapter.

### **Early History**

Before we had chemists we had alchemists. These mixed their rather basic and crude knowledge of chemistry with mysticism, magic, and the occult. The advent of the invention of Black Powder naturally attracted their attention and spawned many mystical theories as to how Black Powder got its explosive power. Some even speculated that this power came straight from Satan himself.

Over the ensuing centuries alchemy became the more reputable science of chemistry. This gave rise to more respectable theories, but still produced some offbeat speculation such as the phlogiston theory of combustion. Scientists abandoned this theory when more and more scientific data was discovered that contradicted it. Thus the speculation of bygone ages has been largely replaced with solid knowledge backed by solid data.

But there remain, to this day, areas of our knowledge about Black Powder that are sparse and even virtually non-existent. Having been around for so long, and studied so extensively, many have concluded that we know all there is to know about the chemistry of Black Powder. This is not true.

This chapter presents a summary of the present scientific knowledge of the chemistry of Black Powder, highlighting areas where this knowledge is far from complete.

### **The Problems with Black Powder Research**

Black Powder research has been hampered by several factors. Perhaps the most important common factor in most of these can be summarized in one word - money.

Interest in Black Powder has waned dramatically over the years. In many applications Black Powder has been replaced with more modern explosives and propellants, so there is very little motivation to spend any meaningful amount of money in further research on Black Powder. Even the big spenders like the military have shifted their focus of research to other avenues. There are rumors floating around that interest in Black Powder may be revived in the military for certain specialized applications. But these are little more than rumors. Even if there is no smoke without fire - and no fire without Black Powder - such military research could take a while to be declassified and released into the public domain.

Another factor inhibiting Black Powder research relates strictly to the laws of physics. Black

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Powder is often used as an explosive or a propellant. This means that the products of its chemical reactions are dispersed in a way that makes collecting them extremely difficult. Some have tried to get around this problem by burning black Powder under more controlled and less violent conditions. Unfortunately the chemical reactions in Black Powder under these conditions can be significantly different from their more violent counterparts, making some of the observations and data gathered irrelevant.

Research is also hampered by lack of standardization. There are no official *standards* as such that can be traced to national or international standards organizations. Different researchers have used different methods of measurement and testing. Recent research has also been sporadic, making it difficult or impossible for later researchers to contact those involved in prior research.

### **The Black Powder in Question**

Most of the discussion in this chapter concerns Black Powder made with standard ingredients. These ingredients are also assumed to be: potassium nitrate (KNO<sub>3</sub>), sulfur (S) and charcoal (C) in the approximate percentages of 75, 10 and 15 respectively, i.e. the Waltham Abbey formula.

Here and elsewhere in this chapter charcoal is represented merely as carbon (C). This convention is used to both simplify some of the discussions and to accurately represent the works of the various authors quoted. Many of these authors treated charcoal as pure carbon, ignoring its smaller percentages of other elements such as hydrogen and oxygen. So charcoal is represented just as carbon where deemed appropriate and as a complex of carbon and other substances where it is helpful to examine charcoal in greater detail.

While most of the discussion in this chapter concerns *standard* Black Powder, we also explore the properties of sulfurless Black Powder and Black Powder that substitutes sodium nitrate (NaNO<sub>3</sub>) for potassium nitrate.

### **Chemical Reactions**

When Black Powder is ignited certain chemical reactions occur. These result in the ingredients in the Black Powder being converted into other chemical substances, both solids and gases. The rapid formation of gases gives Black Powder its explosive and propellant properties.

In the early 19<sup>th</sup> century Guy-Lussac [1] proposed that the gases formed by exploding Black Powder comprised:

Carbon Dioxide (CO <sub>2</sub> )	52.6%
Carbon Monoxide (CO)	5%
Nitrogen (N <sub>2</sub> )	42.4%

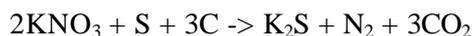
These results were contested by Piobert [1] but the main disagreement appears to relate to gas volumes rather than content. Later research conducted by numerous other researchers shows that these conclusions concerning the types of gases produced were overly simplistic and that many other gaseous products could be formed. Notable is the extensive research done by Noble and Abel.[2] Nevertheless; these later experiments showed that the principle gases produced from exploding Black Powder are carbon dioxide and nitrogen.

Further experiments were conducted by Chevreuil [1] who exploded Black Powder in a gun barrel and also burned it in the open air. These experiments showed that quite different results are obtained when Black Powder is ignited under different conditions. Later experiments by Noble and Abel [2] re-affirmed these results.

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We need to pause at this point and consider the implications of the findings of Chevreuil, Noble and Abel. Their findings showed that in effect there is no definitive chemical equation that can represent Black Powder ignition under all conditions. This single fact is critical to anyone's understanding of Black Powder chemistry.

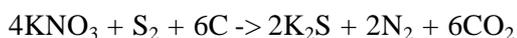
Chevreuil concluded that Black Powder exploded in the barrel of a gun reacted according to the following equation:



Part of Chevreuil's reasoning points to the fact that this formula represents almost exactly the proportions found in Black Powder made with the 75:10:15 ratios. Substituting the atomic masses of KNO<sub>3</sub>, S and C into the above formula gives:

KNO <sub>3</sub>	74.8%
S	11.9%
C	13.3%

This explanation seems to have gained enough credibility in certain quarters that even more than a century later it was still accepted by some. I have a chemistry textbook[3] dated 1936, which accepts the above theoretical explanation with the above formula modified only as follows:



Graham [1] accepted Chevreuil's view, and expanded on it by proposing that potassium sulfide (K<sub>2</sub>S) is converted to the sulfate (K<sub>2</sub>SO<sub>4</sub>) when it comes into contact with air.

Slower burning Black Powder, according to Chevreuil, yielded carbon and the following potassium compounds: sulfide, sulfate, carbonate (K<sub>2</sub>CO<sub>3</sub>), cyanide (KCN), nitrate and nitrite (KNO<sub>2</sub>).

In 1857 Bunsen and Schischkoff published a classic paper on Black Powder research.[1,2] This research investigated the nature and proportions of the permanent gases generated when Black Powder explodes and the amount of heat generated by this transformation. From these experimental data they deduced theoretically the temperature of explosion, the maximum pressure in a closed chamber, and the total theoretical work done on projecting a projectile. It is worth noting that these findings were theoretical in nature as their experiments did not properly emulate the type of conditions typically found when Black Powder is exploded in a confined space. Their experiments were rather performed on Black Powder that was deflagrated by being allowed to fall into a heated bulb. [2]

From these observations they concluded that the permanent gases represented only about 31% by weight of the powder and occupied a volume of 193 times that of the original unexploded Black Powder. A representative table (Table 7-1)[2] of their results is shown as follows:

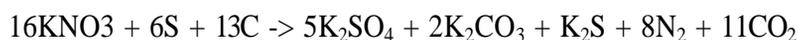
## BLACK POWDER MANUFACTURE, TESTING & OPTIMIZING

Sample Size	Components Before Ignition (in grams)	Components after Ignition (in grams)		
1 gram of Black Powder	KNO <sub>3</sub> 0.7899	Solids		
	S 0.0984	0.6806	K <sub>2</sub> CO <sub>3</sub>	0.1264
	Charcoal:		K <sub>2</sub> S <sub>2</sub> O <sub>3</sub>	0.0327
	C 0.0769		K <sub>2</sub> SO <sub>4</sub>	0.4227
	H 0.0041		K <sub>2</sub> S	0.0213
	O 0.0307		KCNS	0.0030
			KNO <sub>3</sub>	0.0372
			(NH <sub>4</sub> ) <sub>2</sub> CO <sub>3</sub>	0.0286
			S	0.0014
			C	0.0073
			Gases	
		0.3138	H <sub>2</sub> S	0.0018
			O	0.0014
			CO	0.0094
			CO <sub>2</sub>	0.2012
			H	0.0002
			N	0.0998

Table 12-1. Results of the Bunsen and Schischkoff Experiments

It can be seen from the above table that the Black Powder used in this experiment was comprised of a slightly different formula than the commonly used ratio of 75:10:15. Here the approximate ratio is: potassium nitrate 79%, sulfur 9% and charcoal 12%. Also worth noting is their representation of charcoal as a substance comprising not only carbon, but also oxygen and hydrogen.

Berthelot [1] derived the following equation on the basis of Bunsen and Schischkoff's investigations:

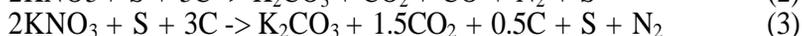
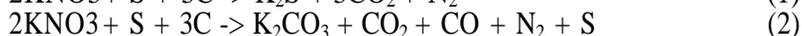
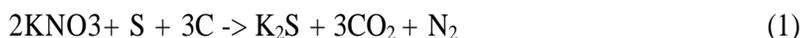


He then developed the first theory about the explosion of Black Powder. Here he drew extensively on the experimental work of Bunsen and Schischkoff. Berthelot's theory assumes two limiting cases for the decomposition of Black Powder.

In Berthelot's first case, K<sub>2</sub>CO<sub>3</sub> forms the chief product of decomposition and K<sub>2</sub>SO<sub>4</sub> a by-product.

In his second case, K<sub>2</sub>SO<sub>4</sub> forms the chief product of decomposition and K<sub>2</sub>CO<sub>3</sub> by-product.

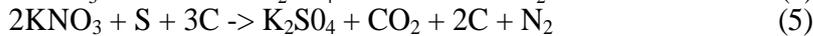
In the first case, the decomposition proceeds according to the following three equations:



Berthelot further proposed that the above occurred in the ratios of 1/3 for equation (1), 1/2 for equation (2), and the remaining 1/6 equation (3).

In the second case, the decomposition proceeds according to equations (1) and (3) above plus the following two equations:

## BLACK POWDER MANUFACTURE, TESTING & OPTIMIZING



And the above are supposed to occur in the proposed ratios of 1/3 for equation (1), 1/2 for equation (3), 1/8 for equation (4), and the remaining 1/24 for equation (5).

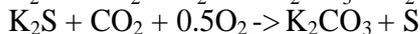
A different conclusion was reached by Debus who concluded that Black Powder burns in a two-stage process. In the first stage, oxidation occurs according to the following exothermic reaction:



The resulting products are then reduced according to the following endothermic (heat absorbing) reactions:

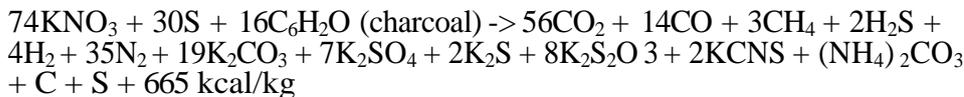


The resulting potassium sulfide may undergo the following further reactions:



A part of the unburned potassium sulfide and sulfur gives  $\text{K}_2\text{S}_2$ .

Much later Kast [1] derived the following equation:



The above picture is rather confusing, isn't it? It seems that no one can really agree as to what exactly happens when Black Powder is ignited. It is tempting to accept the more modern explanations and discard the old, a common practice in scientific research. Unfortunately the issue is not that simple.

A point that possibly got lost in the above jungle of information and theories is that Black Powder can burn under a whole lot of different conditions, and burn *differently* under each unique condition. (Remember the discoveries of Chevreuil, Noble and Abel.) This means that changes in the chemical reactions can range from the very subtle to the radically different!

Ignition conditions can vary widely in practice from high-pressure ignition, which occurs in guns (of both large and small caliber) to lower pressures found in fireworks applications such as mortars, Roman candles and mines. Environmental factors such as temperature and relative humidity might also come into play. Noble and Abel[2] found so many variations in their experiments that they concluded that no value could be attached to a general chemical expression relating to the burning of Black Powder. So there is no "one true formula" for the chemical reaction that occurs when Black Powder is ignited. Any formula used to show what happens when Black Powder burns needs to be qualified with the conditions under which the burning occurred.

Another consideration is some variation in the formula used in Black Powder manufacture. While the traditional Waltham Abbey ratio of 75:10:15 can be regarded as a standard, some

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variations do occur in practice. Propellant powders used by the military and in fireworks usually stick quite closely to the 75:10:15 ratio. This is illustrated in the following two tables (Table 12-2[2] and Table 12-3 [4]).

Description	Potassium Nitrate (%)	Sulfur (%)	Charcoal (%)	Water (%)
Pebble Powder	74.76	10.07	14.22	0.95
Rifle Large-grain	75.1	10.27	13.52	1.11
Rifle Fine-grain	75.18	9.93	14.09	0.80
Fine-grain	73.91	10.02	14.59	1.48
Spanish Spherical Pebble Powder	75.59	12.42	11.34	0.65
Sporting Powder	77.99	9.84	11.17	—
Austrian Cannon Powder	73.78	12.80	13.39	—
Austrian Small Arms Powder	77.15	8.63	14.27	—
Cannon Powder	74.66	12.49	12.85	—
Russian Powder	74.18	9.89	14.83	1.10

Table 12-2. Analysis of Black Powders (circa 1875)[2]

Description	Potassium Nitrate (%)	Sulfur (%)	Charcoal (%)	Water (%)	Ash (%)
Du Pont 3814	73.88	9.97	15.71	0.30	0.14
Du Pont 7625	73.59	10.61	14.84	0.82	0.14
CIL 1-Keg-A	73.13	10.83	14.61	0.64	0.79
CIL 1-Keg-B	73.13	10.83	14.61	0.64	0.79
GOE 76-3	74.34	10.25	14.66	0.48	0.27
Du Pont 7846	74.01	9.92	15.01	0.79	0.27
GOE 78-1	74.43	9.95	14.54	0.49	0.58
GOE 78-2	74.45	9.88	14.88	0.20	0.59
CIL 8-2-73	72.92	10.83	14.78	0.65	0.82
CIL 4-23	73.93	10.63	14.05	0.63	0.48

Table 12-3. Analysis of Black Powders (circa 1975)[4]

Note that in the above table the percentage value of potassium nitrate includes tiny percentages of impurities such as potassium sulfate and potassium chloride.

The above data, gathered approximately a century apart, indicate that the 75:10:15 ratio has been fairly closely adhered to, especially with the more modern powders. This is not only true for Black Powder produced in Britain and the USA, but for Black Powder manufactured elsewhere as well. For example, Shimizu[5] gives an analysis of Japanese Black Powder containing 74.20% potassium nitrate, 9.62% sulfur, and 16.18% charcoal.

However, much variation exists in powders used for blasting. Here, not only the ratios of the three principal ingredients differ, but it is common to find additional ingredients in such powders. Blasting powders also tend to substitute sodium nitrate for potassium nitrate and some use both oxidizers. Substitutes for charcoal are also found in some blasting powder formulas. Table 12-4 [1] and Table 12-5 [1] show some of these variations.

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Description	KNO <sub>3</sub> (%)	Sulfur (%)	Charcoal (%)	Ammonium sulfate and copper sulfate(%)
Strong blasting (French)	75	10	15	
Slow blasting (French)	40	30	30	
No. 1 blasting (German and Polish)	73-77	8-15	10-15	
No. 1 Bobbinite (w/2.5-3.5% paraffin) (British)	62-65	1.5-2.5	17-19.5	13-17
No. 2 Bobbinite (w/7-9% starch) (British)	63-66	1.5-2.5	18.5-20.5	

Table 12-4. Blasting Powder Compositions (Potassium Nitrate Based)[1]

Description	NaNO <sub>3</sub> (%)	KNO <sub>3</sub> instead of NaNO <sub>3</sub> (%)	Sulfur (%)	Charcoal or substitutes (%)
No. 1 black blasting (German)	70-75	up to 25	9-15	10-16
Blasting (American)	70-74	-	11-13	15-17
No. 3 black blasting (Petrolastite or Haloclastite)	71-76	up to 5	9-11	15-19 of coal-tar pitch
No. 2 black blasting	70-75	up to 5	9-15	10-6 of lignite

Table 12-5. Blasting Powder Compositions (Sodium Nitrate Based)[1]

Further consideration should be given to other variations in manufacture such as the degree of incorporation and the resulting density of the powder. A major factor, which often is not given the consideration it deserves, is the type of charcoal used.

## The Significance of Charcoal

While little variation is found in potassium nitrate of high purity and minimal variation in sulfur, significant differences can be found in the different charcoals used in Black Powder. These differences have been described in depth in the earlier chapters on charcoal. These chapters also described the very important role that charcoal plays in Black Powder.

The foregoing discussion focused on the resulting products produced when Black Powder is ignited and its ignition allowed to go to completion. While some of this discussion has involved intermediate reactions in the combustion process, it has not attempted to explain the ignition process itself, i.e. what happens when heat of sufficient intensity is applied to the powder, causing it to ignite.

## The Ignition Process

We now look at the ignition process itself.

The chief player in Black Powder ignition is its oxidizer, potassium nitrate. Here, as with other oxidizers, potassium nitrate supplies oxygen to the reaction. This oxygen, if supplied with enough heat, causes the two other components to burn. Given the right combination of the ratios of the ingredients and an efficient method of manufacture, Black Powder burns very rapidly. This rapid burning makes it useful as an explosive or propellant.

While the potassium nitrate supplies most of the oxygen to the reaction, a small percentage of oxygen is contributed by the charcoal and also possibly by the atmosphere. Charcoal itself con-

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tains oxygen atoms in its chemical composition and may contain absorbed oxygen and other atmospheric gases.

## **Ignition phases**

Although Black Powder ignites very rapidly, its ignition can be separated into several distinct phases. The most important phase is the decomposition of the potassium nitrate, which is preceded by a pre-ignition phase. This decomposition of the potassium nitrate is essential for it to yield its important oxygen component to the process. The decomposition starts with the melting of the potassium nitrate.

Potassium nitrate melts at 334 °C. Its counterpart, sodium nitrate, used in various blasting powders, melts at 307 °C. This suggests that sodium nitrate powders ignite at lower temperatures, which indeed they do. Interestingly, an eutectic mixture of potassium nitrate and sodium nitrate melts at 220 °C. [1] A *eutectic mixture* is a mixture where the mixed substances are mixed in such proportions as to give the lowest possible melting point.

Experiments have shown, however, that the Black Powder ignition process begins at a much lower temperature. This is due to the influences of the charcoal and sulfur. An important factor here is the melting point of sulfur, which is about 115 °C. Thus at about 150 °C, molten sulfur reacts with hydrogen to form hydrogen sulfide (H<sub>2</sub>S). This H<sub>2</sub>S then reacts with the KNO<sub>3</sub> to form potassium sulfate (K<sub>2</sub>SO<sub>4</sub>). This reaction emits heat, causing the KNO<sub>3</sub> to melt. This process is often referred to as the pre-ignition process.

## **Sulfur's importance**

The importance of sulfur has been demonstrated in experiments performed by Hoffmann. [1] These experiments showed that sulfur did the following:

- Facilitated an increase in the quantity of gases evolved on explosion.
- Reduced initial decomposition temperature and temperature at which explosion occurred.
- Intensified the sensitiveness of mixtures to impact.
- Counteracted the formation of carbon monoxide.

The above conclusions were drawn in part from some of the following experimental data:

Potassium nitrate ignited with carbon gives only K<sub>2</sub>CO<sub>3</sub>, but in the presence of sulfur gives CO<sub>2</sub>, K<sub>2</sub>SO<sub>4</sub> and K<sub>2</sub>S. Thus more gases are created by exploding Black Powder that contains sulfur than Black Powder that does not.

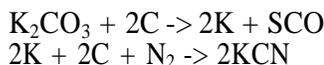
A mixture of two moles of KNO<sub>3</sub> and three moles of carbon (charcoal with a 71% carbon content) begins to decompose at 320 °C and explodes at 357 °C, while a mixture of two moles of KNO<sub>3</sub> and one mole of sulfur begins to decompose at 310 °C and explodes at 450 °C. A mixture of KNO<sub>3</sub> with both sulfur and charcoal yields both lower decomposition and explosive temperatures as shown in an experiment where a mixture of two moles of KNO<sub>3</sub>, one mole of sulfur and three moles of carbon begins to decompose at 290 °C and explodes at 311 °C. This third experiment is very significant in that it shows that sulfur does not only reduce decomposition and explosion temperatures, but it greatly narrows the gap between initial decomposition and explosion temperatures. Sulfur increases the sensitiveness of Black Powder mixes to impact while carbon (charcoal) reduces it. This is shown by the following experimental data:

A 2 kg mass dropped from 45-50 cm caused a mixture of KNO<sub>3</sub> and sulfur to explode while a mixture of KNO<sub>3</sub> and charcoal was unaffected. A mixture of KNO<sub>3</sub>

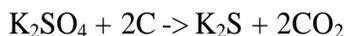
## BLACK POWDER MANUFACTURE, TESTING & OPTIMIZING

with both sulfur and charcoal exploded when a 2 kg weight was dropped on it from a height of 70-85 cm.

Hoffman[1] also concluded that sulfur counteracts the formation of carbon monoxide when Black Powder explodes and also has an impact on the amount of potassium cyanide (KCN) gas produced. This is because the addition of sulfur causes  $K_2SO_4$  to be formed in addition to  $K_2CO_3$ . Thus the amount of potential  $K_2CO_3$  is reduced. The formation of  $K_2CO_3$  causes both carbon monoxide and potassium cyanide to be formed as follows:



The decomposition of  $K_2SO_4$  does not result in either carbon monoxide or potassium cyanide gases forming, as shown in the following equation:



The importance of sulfur in Black Powder is further emphasized by experiments in trying to find a substitute for charcoal. Such experiments were conducted by Wise, et al [6] at the US Army Ballistic Research Laboratory at the Aberdeen Proving Ground in Maryland. Their research demonstrated that sulfur has a profound effect on combustion when phenolic materials were used as charcoal substitutes. Their experiments did, however, reveal opposing trends with different types of phenolic materials. For example, quinizarin and anthraflavic acid both produced more rapid burning powders with the absence of sulfur. Other polyphenols exhibited the opposite trend, but at a lower magnitude. These data challenged the perceived importance of the sulfur being reduced by organic compounds and strengthened the hypothesis that the influence of sulfur is more marked in its role in the flame-spread rate after ignition occurs. Wise, Sasse and Holmes concluded that this hypothesis needs to be explored further using both charcoal and charcoal-substitute mixes.

### **Sulfurless Black Powder**

No discussion about the role of sulfur in Black Powder would be complete without examining useable Black Powder that does not contain any sulfur. Here the term "useable" denotes Black Powder that performs adequately as an explosive, propellant or igniter. Probably the most famous type of sulfurless Black Powder was the so-called "Cocoa" powder that used incompletely carbonized charcoal. [1] This charcoal, known as "Cocoa" or "Red" charcoal, was typically manufactured at a temperature of 140 - 175 °C. It had a carbon content of 52 - 54%, which is much lower than other charcoals used in Black Powder. Its major drawback was its sensitivity to friction, which easily ignited it.

A stoichiometric mixture of Black Powder comprises 87.1% potassium nitrate and 12.9% charcoal. The decomposition occurring after ignition can be represented theoretically as follows:



In practice, sulfurless Black Powder mixtures are generally not used for propellants, but rather as igniters. These have a potassium nitrate content of between 70 - 80% and a charcoal content of between 20 - 30%. Some "sulfurless" powders actually do contain a small percentage (about 2%) of sulfur, which is far below the normal percentage.

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## **Binary mixtures**

Blackwood and Bowden[1]made extensive studies on the ignition of Black Powder and also on the following binary mixes:

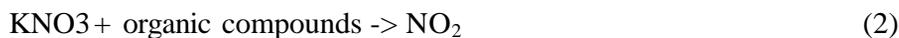
- potassium nitrate + sulfur
- sulfur + charcoal
- potassium nitrate and charcoal.

Among their findings they concluded that ignition could take place as low as 130° C, depending on the pressure that the Black Powder was subjected to. They also confirmed the importance of having charcoal with the right constituents. In their opinion, it was advantageous to remove the constituents, which could be dissolved with organic solvents. This, they said, made ignition easier and gave a faster burn rate.

Blackwood and Bowden formulated the mechanism for Black Powder's ignition and subsequent burning reactions. Accordingly, sulfur reacts first with the organic substances in the charcoal:



Potassium nitrate reacts almost simultaneously with these organic compounds:



The following reactions may also occur:



This last reaction proceeds until all the H<sub>2</sub>S is consumed. The remaining NO<sub>2</sub> then reacts with the unconsumed sulfur according to the following reaction:



The SO<sub>2</sub> formed in the above reaction may then immediately react with the KNO<sub>3</sub> as follows:



Reactions (5) and (6) are endothermic (absorbing heat), while reaction (7) is strongly exothermic (giving off heat). The above reactions (1-7) constitute the ignition process.

Blackwood and Bowden concluded that the chief reaction is the oxidation of charcoal by the potassium nitrate. This is when the Black Powder starts to burn.

## **Flame spread rates**

The flame spread rate of Black Powder is firstly dependent on the solid salts produced after ignition has commenced. These tiny hot pieces of solid matter are driven into the surrounding Black Powder, causing it to ignite and the flame to spread until all the powder is consumed. While the production of solid hot particles produced by different chemical reactions is an important factor in Black Powder's flame spread characteristics, other physical attributes are also important.

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Many processes have been tried over the centuries to improve and control the flame spread attributes of Black Powder. Essential to these is the process of granulation or corning where the Black Powder is formed into solid grains. Recent research on the influence of physical properties on the burn rate has been done by Sasse [7] and also by White and Horst.[8] Sasse's research showed flame spread to be dependent on density, surface area and free volume. White and Horst found that grain position and the ability of grains to move was important.

Thus the flame spread rate of any sample of Black Powder is dependent both on the chemical reactions that take place and on the physical attributes of the powder grains.

## **The Influence of Moisture**

Most Black Powder contains some moisture, and this property does have an effect on the powder's ignition and explosive properties. Nearly every Black Powder manufacturing process uses water, some of which remains in the powder. Black Powder may also absorb moisture from the atmosphere. There remains a certain amount of controversy about whether a certain small percentage of moisture aids ignition or not. The author's own observations indicate that it might. Some have made similar claims, which have been refuted by other authorities. Shimizu [5] refers to an optimal moisture content of about 1%, but this statement in itself appears based more on hearsay rather than empirical evidence from experimentation.

Where there is agreement is the fact that moisture does have an effect and variations in moisture do produce variations in ignition. So where uniformity in performance is critical, the challenge is to find a range of moisture content where performance can be regarded as sufficiently uniform and to control this moisture range.

One suggested range is between 0.3 - 0.5%. [9] Here the challenge is to keep the moisture level above 0.3% while not allowing it to exceed 0.5%. This is far more difficult to achieve than merely aiming at a specified upper moisture limit.

## **The Effect of Aging**

Another area of controversy is the effect of aging on Black Powder. Black Powder has shown itself to be far less susceptible to aging than many other explosives, but the question is - does it actually (like a good wine) improve with age? And if it does so, under what conditions does it age - and why? One possibility is that the charcoal in the Black Powder absorbs oxygen from the atmosphere over a period of time.

The question of aging is a difficult one to answer as the aging process itself, by its very nature, takes a long time. A properly objective test would be to determine the properties of a batch (or batches) of Black Powder and then perform the same tests after an aging period. Practically speaking, this would be difficult to achieve.

## **Conclusion**

Over several centuries a considerable amount of knowledge has been gained concerning the chemistry of Black Powder. Some of this knowledge comes from extensive research done under tightly controlled laboratory conditions and supplemented with field research in practical applications. But there is still a lot we don't know. And there is still a lot to be gained from further research and experimentation.

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## *Chapter 13--Other Methods*

### **Introduction**

This chapter discusses other methods of making Black Powder. Much of the material contained in this chapter has limited practical use to the average experimenter. Nevertheless it is interesting to know what others have tried in the past. It is also interesting to find out what the current state of the art is with respect to alternate methods of Black Powder manufacture.

### **Cool Marble**

An early Chinese method (circa 600 AD) used a large marble slab to cool a hot mix of Black Powder. They did this by first blending together the sulfur and charcoal in a bowl. A saturated solution of potassium nitrate was then brought to the boil and the sulfur/charcoal mix added to it, with constant stirring.

This hot mix was then rapidly cooled by pouring it onto a cold marble slab, and still stirred while cooling. This stirring aided the cooling process and ensured uniformity in the mix. One method they used to stir the mix was to pass a large stone roller back and forth over the cooling mass of Black Powder.

In some respects this cool marble method is similar to the CIA method in that it works on the principle of rapidly cooling a saturated mix of Black Powder. I don't know of anyone who has tried to emulate this rather unique Chinese method of manufacture. Naturally it comes with the normal dangers associated with a hot mix of Black Powder. It also suffers from some of the deficiencies found in the CIA method, the most important of these being the need to mill the sulfur and charcoal and thoroughly blend these before mixing with the potassium nitrate. However, it does have an advantage over the CIA method in that there is less chance of losing potassium nitrate through leaching out.

### **Ideas that Suck**

Another way of getting potassium nitrate to crystallize out very quickly from a hot (preferably close to boiling) solution is to rapidly drop the surrounding pressure. This sudden drop in pressure will cause the water to boil off very quickly, leaving behind rapidly crystallized potassium nitrate. Another way of achieving this is to heat the solution very quickly with a heat source, which has a temperature much higher than the boiling point. This form of *flash heating* is very dangerous as ignition of the Black Powder is virtually assured under these circumstances.

One way of achieving a rapid drop in pressure is to apply a vacuum to the container of hot Black Powder mix. Naturally this container needs to be sealed with some form of outlet to the vacuum. This idea was patented under US Patent number 160,053 in 1875 by Edward Greene.

It is not clear if Mr. Greene actually built the apparatus he describes. There are no drawings or sketches in his patent, and no experimental data. He describes a method whereby a saturated solution of potassium or sodium nitrate is mixed with sulfur and charcoal and heated in a closed vessel to somewhere close to the boiling point of water. The mix is stirred with some

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form of mechanical stirrer throughout this process. After sufficient heating and stirring, a vacuum pump is connected to the vessel. The vacuum pressure applied causes the water in the mix to rapidly evaporate, leaving behind a mix with very small crystals of potassium nitrate.

I don't know of anyone who has actually tried this idea. At one time I toyed with the thought of doing something similar with a wet/dry vacuum cleaner such as a *Shop Vac*. I never got that far. Maybe it is just as well. Some of the resulting powder could have ended up in a wrong part of the vacuum cleaner - the part that has electric sparks generated by the motor brushes. However, there may be a better and less hazardous way of attempting this method.

One such method is to evacuate (remove air so as to create a vacuum) another vessel to an acceptable vacuum pressure. The vacuum pump is then turned off and removed. This vessel is then connected to the vessel containing the hot Black Powder mix, effectively reducing the pressure in the mix vessel. This method removes the hazard of using an electrical pump and has the added advantage of being able to apply vacuum pressure very quickly.

### **Du Pont's Crazy Idea**

The invention described in this section could fit very well under the heading in the last section, in the figurative rather than the literal sense, of course.

The du Pont family developed many processes over the nearly two centuries they were in the Black Powder manufacturing business. One of these was an invention by Ernest du Pont, patented July 1, 1919 under U.S. patent number 1,308,342.

The basic idea comprised a rod mill that was used to mill Black Powder. Instead of using water in this mill to dampen the mix, du Pont used gasoline! His reasoning was that a liquid solvent (such as water) was undesirable. Potassium nitrate is insoluble in gasoline, as are sulfur and charcoal.

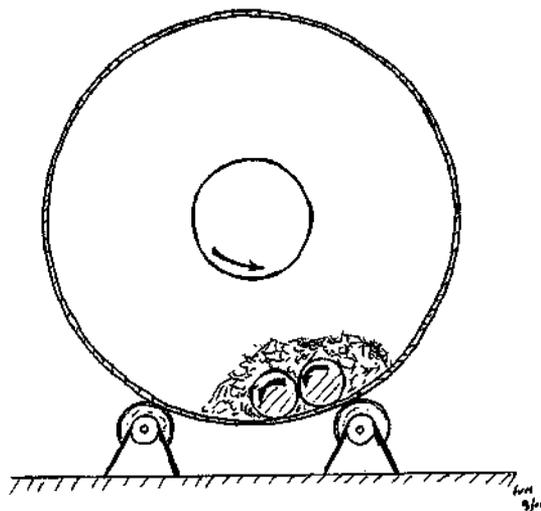


Figure 13-1. Du Pont's rod mill

Du Pont claimed that this method did not require the high pressure used in other manufacturing processes. For this reason, so he claimed, this new process was "perfectly safe". Maybe it was, if one was extra careful not to ignite the gasoline! And think about it: millions dispense gasoline into vehicle gas tanks every day without incident. They then proceed to ignite this gasoline in closed containers with explosive force.

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So just maybe gasoline isn't so dangerous after all. Having noted all that, this particular du Pont process doesn't seem to have taken off as a viable method in the du Pont powder mills. Maybe just as well.

### **Enriched with Vitamin C**

My parents were great believers in vitamin C. Every winter the whole family was heavily dosed with vitamin C to increase our resistance to the common cold. To this day I'm still not sure how effective it was. However, what I did learn along the way was that vitamin C had another name - ascorbic acid.

Years later I was to find another use for ascorbic acid. It was used in pyrotechnics. My first introduction to its pyro uses came from someone who had worked in film and theater pyrotechnics. His particular application of ascorbic acid was in the manufacture of smoke devices that could be used indoors. Later I learned that ascorbic acid had also been used in substitute Black Powders.

Ascorbic acid powders generally use potassium nitrate as an oxidizer and ascorbic acid as the fuel. Other inorganic oxidizers can also be used, although the preference seems to be for potassium nitrate. The proportions of the oxidizer vary between 50% and 75% and the ascorbic acid between 25% and 50%. Some powder use erythorbic acid in place of ascorbic acid, or a mixture of the two.

The manufacturing process is usually quite simple with the potassium nitrate and ascorbic acid mixed together with water to form a slurry. Cellulose fibers are also added to some mixes. Intimate mixing, as with other Black Powder processes is essential. Some processes heat the slurry, being careful to control the temperature within certain limits. If this is not done, certain undesirable chemical reactions can occur. The mix is then dried, compressed, and granulated.

Some formulas use potassium or ammonium perchlorate in addition to the potassium nitrate. Generally the percentage of these other oxidizers is small, in the order of 15%.

These substitute powders that use ascorbic/erythorbic acid mixes are generally aimed at the sporting firearms market. Thus these powders seem better suited for high-pressure applications and may not be suitable for fireworks mortars.

The above methods of making substitute powders are described in detail in the following U.S. patents:

4,497,676	4,728,376	4,881,993
5,449,423	5,569,875	5,633,476

## **Introduction**

In the first edition of this book, I wrote a chapter that was titled *Just Testing*. It was a short chapter that did not say much. It described some simple ways to test Black Powder and for many these simple tests were good enough. Quite a lot has changed since then.

Thanks partly to this book, but thanks largely to the many pyrotechnic experimenters who are willing to share their knowledge, making serviceable Black Powder is no longer a mystery. However, testing Black Powder is. Today we see a shift from asking how to make Black Powder to "How do I test it?" My curiosity in this regard resulted in the original short chapter on testing becoming a long chapter. I changed its title from *Just Testing* to *Testing, Testing, Testing*. The chapter then became horribly long and I ended up splitting it into three.

Very little information on testing has appeared in amateur pyrotechnic literature and discussion in different amateur forums often gets bogged down in needless controversy when it comes to testing. Technical papers on testing written by those who are either involved in the commercial manufacture of Black Powder or in military applications offer little appeal to the average experimenter. And historical methods found in books on the history of Black Powder often appear outdated and irrelevant.

So where do we go from here? What I have attempted in this chapter, and the two following it, is to sort the wheat from the chaff. I have drawn on many resources: historical, amateur, professional and military. Among this pile of information I have attempted to focus on both the interesting and the relevant. Some readers will undoubtedly not like what is written here because some of the subjects dealt with require one to be skeptical, critical, controversial and irreverent. Some of the subject matter discussed might thus offend the bigots who claim the superiority of their favorite testing method. They are not alone. A careful reading of the history of Black Powder reveals many heated controversies relating to testing methods.

And critical we should be. At the same time we should be neither too narrow minded nor too broad minded. Good testing methods have been rejected, only to gain acceptance later when reason prevailed. Conversely some supposedly great testers were found to have flaws after many years of use.

I had originally planned this as one large chapter entitled *Testing, Testing, Testing...* The original chapter ended up being too large, so I split it into two sections, and then finally three. We still have *Testing, Testing, Testing*, but now with a separate chapter describing different aspects of Black Powder test processes.

This chapter discusses basic principles involved in testing. The next chapter describes older historical methods. The third chapter on testing explores modern methods with possible future improvements using modern technology.

## **Why Test?**

Why test Black Powder?

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There are many answers to this question, depending on the particular circumstances of the maker or user. Manufacturers of just about anything will usually test their products to ensure that they meet certain expectations of those who will buy them. Users test the stuff they bought to be sure that it meets their expectations. Both might take these tests a step further. Manufacturers might separate tested material into different performance categories. This is typical of a manufacturer who makes products such as transistors. Each transistor after testing could be allocated an A, B, or C suffix depending on its tested characteristics. A user of transistors could want transistors more closely matched than the manufacturer is willing to supply and thus do his own testing to achieve this goal.

Testing often tries to achieve high standards of consistency and accuracy. Unfortunately this has often sent a message that testing is the domain of the pedantic, fanatical, picky, and those who just love to "rain on parades" and spoil everybody's fun. Not so. Testing can be useful, maybe essential, but often great fun in itself!

From earliest times after the invention of Black Powder, it has been put to military uses. Yes, I know that some can argue that military potential took a while to develop in certain cultures, but develop it did. Military use thus dictated that one's very life could depend on the performance of the Black Powder used. One just doesn't welcome the surprise that the powder won't perform as expected when the enemy is just yards away. So testing of one form or another has become a way of life when it comes to military arms and ammunition.

Early Black Powder was very crude by today's standards. Its ingredients were merely finely ground and mixed together. There was no granulation process that caused the particles to stick together. This resulted in the ingredients separating during transport to the battle lines, requiring re-mixing on site. Early testing thus focused on whether the mix had been compromised in transit.

Later testing focused on how much power each batch of Black Powder gave. This was important to gunners who wanted to know the expected range of their guns. Many variables were still found in Black Powder manufacture, including ingredients which had been adulterated with other substances. This adulteration not only resulted in poorer performance but often made the challenge of "keeping one's powder dry" even more difficult. This was because potassium nitrate was often mixed with salt. Such adulteration not only reduced performance but added an ingredient that made the Black Powder absorb moisture faster.

Modern testing of Black Powder has less to do with life threatening scenarios than before but still has its place. Manufacturers will naturally test their products. Consistency and predictability is important to those who use Black Powder in competitive shooting. Those who make their own powder may want to get a feel for how well it will work before using it to lift shells. Others might just want the ego trip of knowing that their powder is faster than others.

All of the above are valid reasons for wanting to test Black Powder. And all have their own criteria that determine which method (or methods) are the best to use.

### **Accuracy, Precision and Repeatability**

Accuracy, precision and repeatability are three terms which are often rightly and wrongly used interchangeably. Precision can determine the accuracy of a measurement and repeatability may give one a degree of confidence in the accuracy of a series of measurements. Accuracy may be defined in terms of repeatability but also in terms of a whole lot of other criteria such as linearity, zero drift, and resolution. So what do these terms actually mean?

**Accuracy** is a generic term which describes the nature and extent of the difference between the

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measurement of a value and its actual value. For example, one can check the accuracy of one's wristwatch by comparing its time with the time signal of a local radio station. The accuracy here would be measured in terms of the difference in time - either in minutes or in seconds. A more precise measurement of accuracy could be obtained from a standard with greater accuracy such as an atomic clock. Here one could (theoretically) measure the inaccuracy of the wristwatch to the tiniest fraction of a second. Practically one is limited by the resolution of the wristwatch.

**Precision** describes how accurately a measurement can be made. For example, measuring a distance of a mile is done with greater precision if one can measure it to the closest inch rather than the closest yard. Precision is often confused with repeatability and resolution.

**Repeatability** describes (as its name implies) how repeatable measurements of the same quantity are. For example, if the temperature of an object is measured three different times with two different thermometers, the thermometer which reads more consistently has a higher measure of repeatability.

**Resolution** defines the smallest increment to which a device or system can measure. For example, a tape measure that measures to the nearest millimeter has a resolution of half a millimeter. Some will argue that the actual resolution is half a millimeter because one's eye can judge the space between two one millimeter increments. So the definition of a particular system's resolution might be strictly determined by how the system is used and how measurement data is interpreted.

Figure 14-1 below shows three targets that have been shot at by different marksmen.

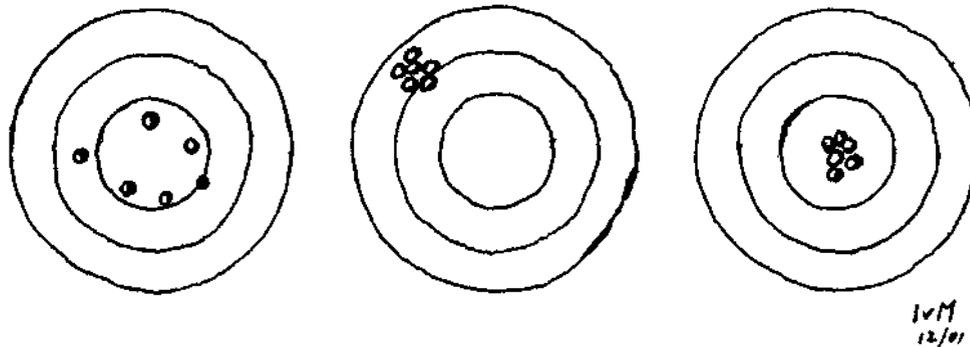


Figure 14-1. Targets

The first target shows the shots scattered in the area of the bull's eye. This marksman has scored higher than the one who shot at the second target. His shots were very closely grouped together but quite far from the bull's eye. The third marksman also had a tight grouping of shots, all in the bull's eye. The first target showed shots fired with a good level of *precision* but a bad level of *repeatability*. The second target shows shots fired with a high level of *repeatability* but lacked *precision*. The third marksman shot with both *precision and repeatability*.

In Black Powder testing, precision is often illusive. This is because there is no set standard that tests can be judged by. An individual or a radio station can set or check their times by an atomic clock that is regarded as a very accurate standard. Common measurements such as weight and length are measured against very accurate national and international standards. Electrical measurements such as voltage, current and resistance can be checked against standards housed in national laboratories. These standards are known as primary standards. In practice, most electrical test equipment is not tested directly against these standards but against what are known as secondary standards. Secondary standards are measured against primary standards. They need not be as accurate but their accuracy is not allowed to deviate

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from the primary standard by more than a specified amount.

Black Powder has no such standards. There isn't even agreement among manufacturers relating to testing methods and their accuracy. The same could be said for scientists. So how is such test data to be used? In Black Powder testing, the most important characteristic of a test instrument or system is its repeatability.

## **Principles of Accurate Measurement**

Using accurate equipment is no guarantee that one's results will be accurate. Good techniques and practices should be followed to ensure maximum accuracy. This section offers a few guidelines.

One of the first principles of testing and experimentation is that any test should preferably be performed more than once. Three times is usually considered a minimum, with twice being the barest minimum. Data from a single test is always questionable. A second test that yields data close enough to the first test may be considered enough to validate the first test. If a significant difference exists between the two tests a third test is highly desirable.

Consistency is needed to get repeatable results. Thus each test should be done in a way that is consistent with previous tests. This means that the apparatus used should be in the same state for each test. In Black Powder testing this is often summarized in one short statement: keep it clean. Black Powder (even of high quality) leaves solid residue which can skew test results if not removed. Consistency also means not changing any of the variables such as weight, volume, ambient temperature and particle size. This also means that such variables should be measured and controlled accurately.

Sometimes it is desirable to change one of the variables to expand the scope of the test. Here it is advisable to change only one variable at a time.

It goes without saying that safety should be a top priority. Most Black Powder testing methods have the potential to create a nasty accident if safety is ignored.

## **Apples and Oranges**

In the first edition of this book, I wrote about comparing apples with pears. This related to inconsistencies that I experienced with my own linear burn rate tests. In many ways, apples and pears are similar as fruits. Apples and oranges are very different. The expression *comparing apples with oranges* is also more popular.

Consistency is very important to ensure good experimental results. Most of our focus has been consistency within the bounds of one's own experiments, but there are other considerations.

Many comparison tests use a commercially made Black Powder to create some kind of testing baseline. This is good practice but has unwittingly introduced misinterpreted data that has evolved into Black Powder mythology. Thus popular misconceptions have arisen from tests that have compared certain commercial powders to home-brewed powders. It is common to hear the phrase *faster than GOEX* and infer that a powder superior to that particular commercial product has been created. The *faster than GOEX* claim may boost certain egos but economizes with the truth.

The truth of the matter is that it is not that difficult to create a powder that burns faster than its commercial counterpart, simply because commercial powders do not always aim for the

## BLACK POWDER MANUFACTURE, TESTING & OPTIMIZING

fastest possible burn rate. Other considerations such as consistency and the potential to store the powder for long periods of time are important objectives to commercial powder makers.

Unfortunately the perception pendulum has swung too far yet again, creating a new mythology. The perception here pertains to what Black Powder actually is. In days gone by, it was common to refer to Black Powder made in factories that used wheel mills as *true* Black Powder. All other powders made by hand, in ball mills, or by precipitation/displacement methods were considered to be *not true* Black Powder. The pendulum has swung the other way. Now it is common to refer to factory-made Black Powder as inferior, being bettered by the stuff created in amateur ball mills. Both these perceptions, in their extreme expressions, are wrong.

Today, Black Powder factories use wheel mills because wheel mills make a very good powder. Like other methods such as stamp mills, wheel mills have been around for centuries. They have ousted stamp mills because so many accidents occurred with stamp mills. They are also used in preference to ball mills and not without reason. Wheel mills are safer to operate than ball mills and produce a more consistent product.

If ball milling produced an overall superior product to wheel mills, it is a sure bet that Black Powder manufacturers would consider ball-milled powder as a viable option. Ball mills are so much cheaper to make than wheel mills. So how do many justify their claims to superior ball-milled powder? Most such claims are based on only one factor, speed. And speed here relates to how fast the powder can project a projectile. Here the projectile is a certain type of projectile such as a golf ball or a baseball, so chosen to approximate fireworks projectiles such as shells.

It is worth noting that powders (particularly substitute powders such as Pyrodex) optimized for sporting firearms appear to perform woefully inadequately when tested in a mortar-type apparatus. This is because these powders are designed for high pressure applications. Pyrotechnic mortars are low pressure devices. This is why gun barrels are made of steel while pyrotechnic mortars are often made of cardboard or plastic.

Homemade ball-milled powders are often a lot less dense than commercial powders. Low-density powders do tend to burn quicker than high-density powders. So comparing such powders could be an apples versus oranges situation.

While some modern testing techniques have had their results skewed through apples versus oranges comparisons, this problem is by no means a uniquely modern one. Right from the earliest times of Black Powder manufacture and testing, tests have been devised that have fallen prey to this problem. And some of these tests were popular for centuries before being questioned and discredited!

The following two chapters discuss a variety of different tests, both from historical and practical perspectives. Apples and oranges, strengths and weaknesses, practical and impractical, myths and facts, await one in the next two chapters.

# BLACK POWDER MANUFACTURE, TESTING & OPTIMIZING

## **Introduction**

This chapter looks at test methods used from earliest times in the history of Black Powder, up to the beginning of the 20<sup>th</sup> century. The next chapter describes modern test methods used in the 20<sup>th</sup> and 21<sup>st</sup> centuries.

I have used both the terms *Black Powder* and *gunpowder* in this chapter. Basically they can be considered as one and the same. The term *gunpowder* is the older historic name for Black Powder. I felt it more fitting to use the term *gunpowder* when describing certain historic devices and events. This is the word that was used in the original descriptions and is still in use in some of the literature today.

## **Ye Olde Ancient and Crude Ways to Test Thy Gunpowder**

Older historical ways of testing Black Powder have both the power to fascinate and amuse. While these attributes apply to their historical context it is also fascinating and amusing observing modern day claims of having "invented" a new method of testing Black Powder. Alas, there is nothing new under the sun. Most modern testing methods have their roots in practices which were developed centuries ago.

A clear example of this is the much beloved Pyro Golf. To claim that this is a modern invention is inaccurate. Rather it is a refinement on the mortar eprouvette testers which have been around for centuries. The same can be said for linear burn rate testers, quickness testers and any number of other methods. Most modern tests are just older tests which have been refined, expanded or repackaged.

Some older tests are amusing. Consider the taste test. Here one actually tasted the gunpowder to check if its ingredients passed muster. Experts supposedly could tell the difference between powder that had reasonably pure potassium nitrate and that which contained common salt. Some might also have discerned impurities in the other ingredients. Potassium nitrate is not poisonous if taken in small quantities and one may even take larger quantities of common salt without any ill effects. So is there any danger in actually tasting Black Powder? With modern powder, probably not, but with powders of bygone days it may have required a special kind of courage to perform a taste test. Potassium nitrate was in times past derived from urine. So impure potassium nitrate might contain, well it's anybody's guess!

Modern powders usually strictly contain three ingredients plus water. All these ingredients, potassium nitrate, sulfur and charcoal are not poisonous. Older powders may have, beside adulterated ingredients, contained certain other substances which supposedly improved the powder's properties. These could be anything, ranging from mercury to manure.

Another test involved placing a sample of Black Powder in the palm of one's hand and igniting it. If the powder burned without burning one's hand it passed - if it burned the hand it failed. Surprisingly this test is not based, as some might suppose, on superstition and folklore. It does have a scientific basis. Good Black Powder burns virtually instantaneously without noticeable residue. Inferior powder is slower burning with a residue. Both of these properties can give one's hand a warm, but not necessarily fuzzy, feeling.

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Gradually more sophisticated methods of testing were developed. Because Black Powder was used to force an object such as a cannon ball to move in a certain desired direction, it made sense to build a device to measure this force in such a way that it represented a corresponding force exerted on the projectile used in battle. Hence the early eprouvettes were born.

### **Early Eprouvettes**

The earliest recorded eprouvette reminds one of a trash can with a hinged lid. It also brings back fond memories of my boyhood.

At that time we had a mailbox with a hinged lid on top. One of my favorite tricks was to place a large firecracker in the mailbox and time the lighting of the fuse so that it exploded just as someone was passing by. I later refined the system to light a very short fuse with some steel wool that ignited when an electric current from a battery passed through it. My electric firing system worked well, much to my delight, and much to the consternation of the local pedestrian traffic.

The blast from the firecrackers usually raised the lid of the mailbox, the larger crackers causing the lid to jam itself open. I didn't know it at the time - that I had inadvertently rediscovered a way of testing explosive power. A similar discovery was made centuries earlier and resulted in a device to test gunpowder. Such a device was described by William Bourne in 1578.

The principle of operation of Bourne's device is to ignite Black Powder in a confined space with the resulting force raising the lid of the device. The greater the amount of force, the higher the lid is raised. A ratchet stops the lid returning and keeps it in its highest-reached position. William Bourne wrote the following description of his device (spelling kept as in the original text):

And as touching this, how to make an Instrument or Engine for to knowe the goodnesse or badnesse of powder, (that is to say) to knowe the strength or weaknesse thereof, they may doo in this manner first, make in mettall or Yron a round boxe, of an inch and a half in breadth more or lesse at your discretion, and of two ynches deepe more or lesse, at your discretion, and let it be placed so, that it may stand vpright, and haue a little tuchhole at the lower part therof, and the couer or lid be of reasonable weight, & the otherside of the couer or lid right against the ioynt to haue a hole fitted of purpose, and then vpon that side that the ioynt of the couer or lid is of, there must bee raised a thing that must haue of yron or other mettall a part of a circle, and the ende of that must goe through the square hole in the lid of the couer, and the other ende to goe with a pinne or ioynt right ouer the ioynt of the couer or lid, and the said crooked thing or part of a circle, to haue teeth or notches, like vnto a Sawe, and the teeth to stand vpwards, and then it is finished, and then, whensoever that you list to prooue the strength of powder, and you hauing of diuers sortes of powder, then wey some small quantities of the powder, and then put that into the Boxe, and then let downe the couer or lidde, and then giue fire vnto it at the touchhole, and then the powder will blowe vp the couer or lid, and then the teeth or notches being filed or trimmed of purpose, will holde up the lid at the highest, and yet not staying in the blowing of it vpwards, for that the teeth standeth so, to stay it that it shal not come downwards, and then trying or prouing diuers sortes of powder, you shall knowe which is the best or strongest powder, or weaker powder, by the blowing vp of the lid or couer, you putting putting in the powder by weight. &c. And this is the forme of the engine or instrument.

The rather confusing picture given in the above description is greatly clarified in the sketch below.

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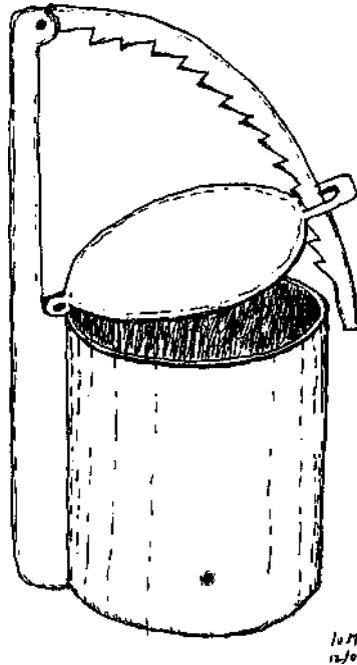


Figure 15-1. Bourne's eprouvette

Yes, a picture is worth a thousand words (or even considerably less if the words are in old English where spelling, grammar and usage are somewhat different).

## **Later Refinements**

Later eprouvettes contained many refinements. Some worked on the principal of gravitational resistance like Bourne's did. Others relied on resistance from a spring mechanism or from friction. All relied on moving an object (or objects) a certain distance, this distance relating to the strength of the powder. This distance was either a linear distance or an angular distance, depending on the principles involved.

In most instances the moving object remained integral to the eprouvette, or somehow stayed attached to it with a retaining device such as a rope or chain.

## **Vertical Ratchet Eprouvettes**

Many of the earliest eprouvettes relied on gravitational resistance. A noticeable feature of these was a departure from Bourne's method of moving a hinged object. These eprouvettes moved an object linearly in a vertical direction, the object in question being propelled by a small measured charge of Black Powder. Most, however, retained Bourne's ratchet technique. Some of these testers used a single ratchet, while others used two.

An exception to the popular ratchet technique was a tester developed by Furtenbach.

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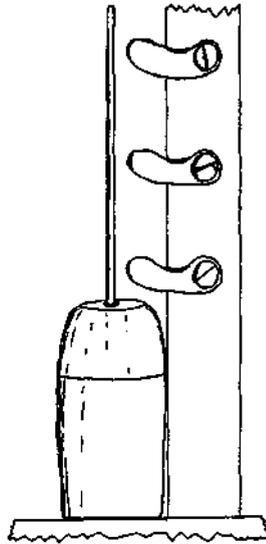


Figure 15-2. Mechanism of Furtenbach's tester

Instead of using a ratchet to maintain the final position of the moved object, Furtenbach's tester used a number of swiveling arms, each an inch apart.

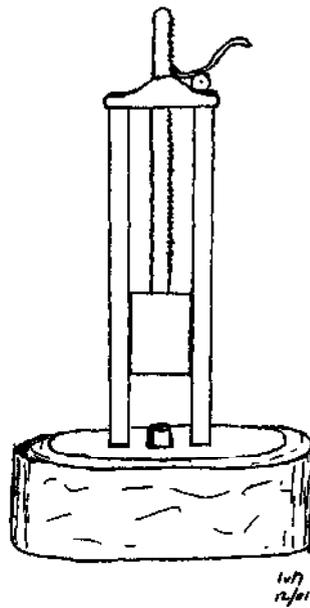


Figure 15-3. Single ratchet vertical ratchet tester

The above illustration shows a single ratchet eprovette. The following illustration shows an eprovette using a dual ratchet mechanism.

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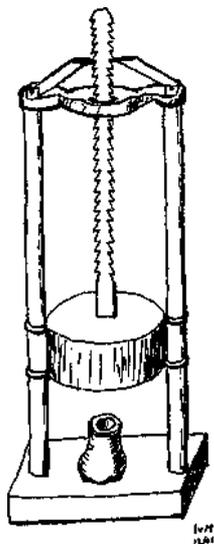


Figure 15-4. Dual ratchet eprouvette

Vertical ratchet eprouvettes have been used right from the 17<sup>th</sup> to the 20<sup>th</sup> centuries. However, their mechanism was regarded as awkwardly large by some, even in hand-held units. This resulted in many designs reverting to a hinged mechanism like Bourne's. Although these looked very different to Bourne's device, they had the following similar features:

- A small cylinder in which the powder was ignited
- A hinged lid covering the cylinder
- A ratchet to record the final position of the hinged lid.

Two major differences, however, are worth noting:

- The cylinder was a lot smaller
- The hinged lid rarely relied on gravitational resistance, but rather on a spring.

These early eprouvettes typically consisted of a mechanism mounted on a metal bar, and are referred to as *straight eprouvettes*.

## **Straight Eprouvettes**

Perhaps one of the biggest attractions of straight eprouvettes was they were light, portable hand-held devices. Such attractive features influenced other devices such as V-spring eprouvettes and pistol eprouvettes.

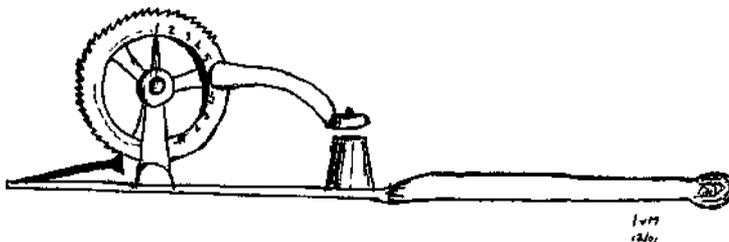


Figure 15-5. Basic straight eprouvette

The above illustration shows a basic straight eprouvette. Another simple straight eprouvette is shown in the next illustration

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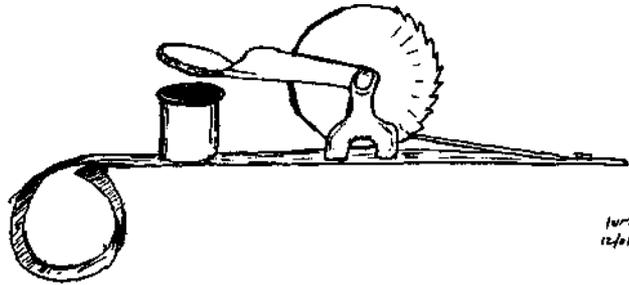


Figure 15-6. Simple straight eprouvette

The following illustration shows another variation.

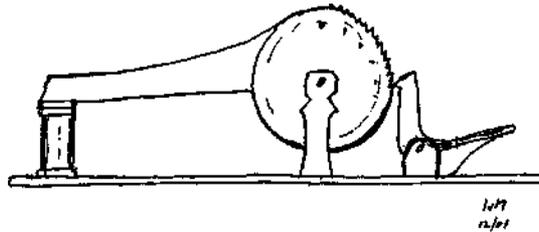


Figure 15-7. Straight eprouvette variation

The straight eprouvette became the forerunner of the pistol eprouvette. The following illustration shows a straight eprouvette with a pistol-type handle. Note, that unlike a pistol, this eprouvette has no firing mechanism, and still relies on the user to light the Black Powder with some external form of ignition.

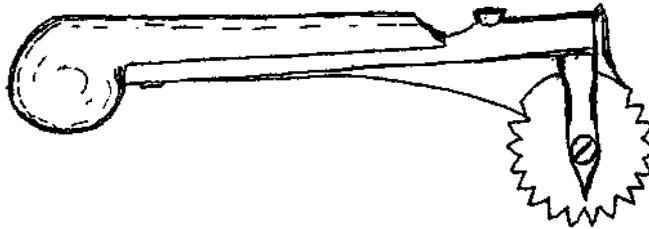


Figure 15-8. Straight eprouvette with pistol-type handle

Although most straight eprouvettes relied on a spring resistance mechanism, some relied on gravitational force to provide resistance, as shown in the eprouvette depicted in the illustration below.

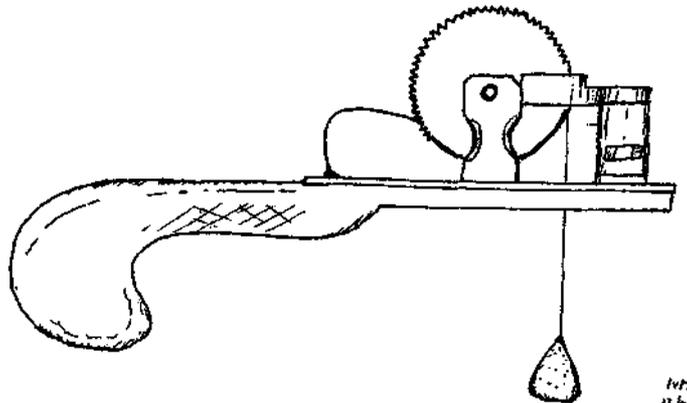
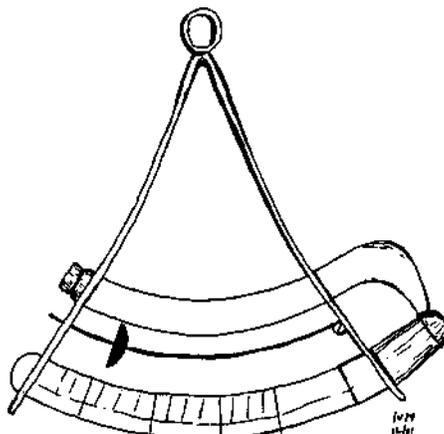


Figure 15-9. Gravity resistance eprouvette

## **V-Spring Epreuves**

V-spring epreuves evolved from V-spring scales that were used to measure weight. Some were made with the dual functionality of being both a scale and a gunpowder tester.



*Figure 15-10. V-spring epreuve*

V-springs are springs made in the shape of a V and look similar to a wishbone. All use a protractor type device to measure the change in the V-spring's angle when force is applied. The scale of this protractor was often in units other than degrees.

Naturally such a V-spring would spring back to its original position after a test firing and thus required a means of retaining a record of the angular change. A ratchet mechanism could have been used here, but the designers favored a pointer that was held in place by friction. This was probably a good move on their part, as ratchet mechanisms would have been more cumbersome and more expensive.

Some manufacturers produced pistol epreuves with V-springs, with some being able to be used as both a pistol and an epreuve.

## **Pistol Epreuves**

Pistol epreuves very popular with artillery officers but found favor among civilian users as well. As their name implies, pistol epreuves are made in a similar fashion to pistols. Some could actually work as pistols by removing the epreuve mechanism and replacing it with a screw-on pistol barrel.

Some of the earliest pistol epreuves relied on a vertical ratchet mechanism for determining a powder's strength. They were thus variations on the original vertical ratchet tester. Later developments used a wheel with spring resistance like the early straight epreuves. Most pistol epreuves made since then have used this principle in one form or another. Pistol epreuves ignited the Black Powder in the same way as their normal pistol counterparts did. Thus their development followed the evolution from matchlock to flintlock to percussion ignition. It was also common to find very ornately made epreuves in the same way that some pistols were very ornately made.

## BLACK POWDER MANUFACTURE, TESTING & OPTIMIZING

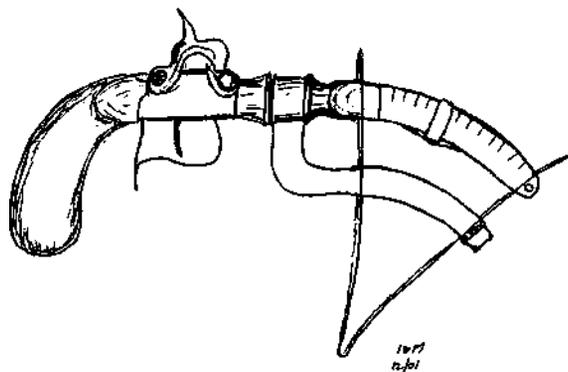


Figure 15-11. V-spring pistol eprouvette

Most pistol eprouvettes used some form of ratchet wheel mechanism, but there are some notable exceptions. V-spring eprouvettes were fitted to pistols in various configurations. Instead of using a vertical ratchet bar, some pistol eprouvettes used a horizontal friction bar.

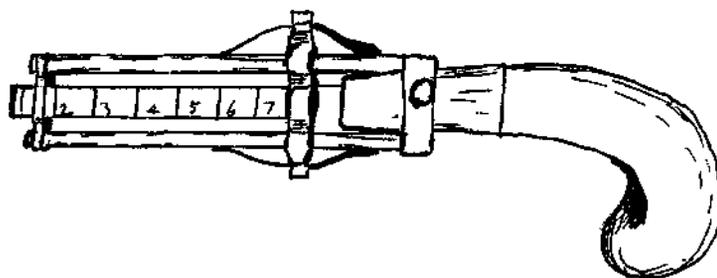


Figure 15-12. Friction bar pistol eprouvette

A few pistol eprouvettes relied on gravity to give the required resistance, but rather than using Bourne's hinged lid principle, these rotated a wheel from which a weight was suspended.

### **Mortar Eprouvettes**

Mortar eprouvettes differed from other eprouvette types in that they expelled a projectile. As their name implies, mortar eprouvettes are a type of mortar.

Many variations exist in mortar eprouvettes pertaining to overall size, barrel length and type of projectile. All, however, rely on either one of two different techniques. The first technique is to fire the projectile horizontally, the second is to fire it vertically. Both these techniques needed an accurate way of measuring this distance.

It is easy to measure the horizontal distance an object has moved but it can be quite a challenge to measure the vertical distance. To meet this challenge, long poles known as *gunpowder test poles* were used. Such a pole would be mounted next to the mortar. It had easy-to-read height marks that could be read from a distance. A *designated volunteer* would fire the mortar and four accredited observers would observe the maximum height reached by the test projectile. This feat was not as hard as it sounds. The projectile was naturally stationary at its maximum height and it was fairly easy to measure this height visually. This vertical test had the added advantage that it could be done in a small amount of space and was safer than horizontal tests. It did have one problem, however. The test projectile could possibly fall on the mortar and damage it. To get around this, the mortar was tilted at a slight angle from vertical.

## BLACK POWDER MANUFACTURE, TESTING & OPTIMIZING

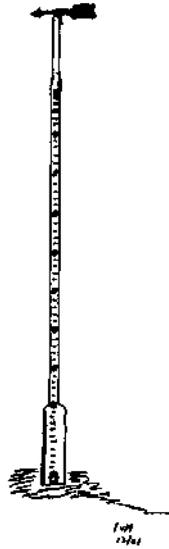


Figure 15-13. Gunpowder test pole

Mortar eprouvettes were first described in the 17<sup>th</sup> century. Towards the end of the 17<sup>th</sup> century they were officially adopted by Royal Edicts in France to be the favored powder proving method. Many regarded these devices as the most accurate means to test Black Powder. It wasn't until the 19<sup>th</sup> century that it was discovered that these devices favored faster-burning fine powders due to their short barrels.

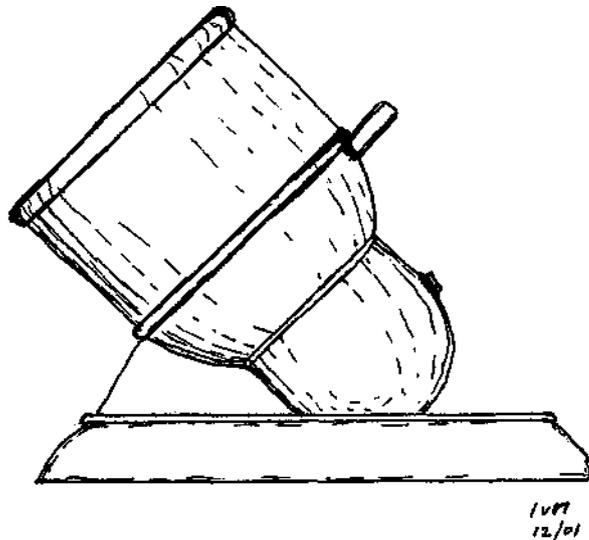


Figure 15-14. Mortareprouvette

Mortar eprouvettes came in many shapes and sizes. One of the first recorded devices was only about twice the size of the tiny eprouvette described by Bourne. Conversely some military eprouvettes fired 8 inch diameter balls weighing just under 70 pounds.

# BLACK POWDER MANUFACTURE, TESTING & OPTIMIZING

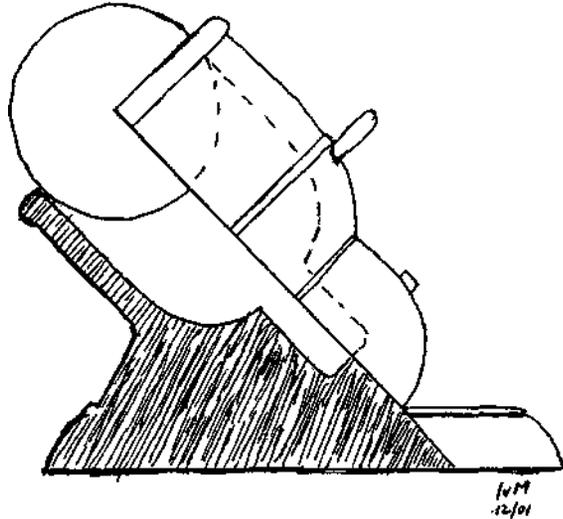


Figure 15-15. Mortar eprouvette cutaway view

A popular angle for mortar eprouvettes was 45 degrees, giving a maximum horizontal range. Typically this range was between 200 and 300 meters. Other mortars used an angle of 75 degrees which gave a much shorter range in the order of 40 to 60 meters. A near-vertical mortar was developed in the 19<sup>th</sup> century that was inclined at an angle of 84 degrees. An 18 yard silk tape was attached to this ball that measured its height of travel to the nearest inch. The mortar was placed close to a wooden box filled with straw and fired so that the ball fell into the box.

Possibly the greatest shortcoming of the mortar eprouvette was its short barrel. This barrel was a lot shorter than the cannons it was supposed to test powder for. In fact, some mortar eprouvettes had barrels that allowed only about half of the ball to be enclosed. An important lesson is to be gained here. A good test requires close simulation of actual conditions under which the product is used.

## **The Eprouvette of du Me**

In 1702 a French artillery officer, M du Me, proposed a quite different means of measuring powder strength. His system used a square bent metal tube containing water. A small charge of Black Powder was ignited in an enclosed section on one end of the tube, the resulting pressure expelling a certain volume of water on the other. The power of the powder was reckoned to be proportional to the volume of water expelled. No one knows for sure if such a device was ever constructed even though its principle was approved by the French Royal Scientific Academy.

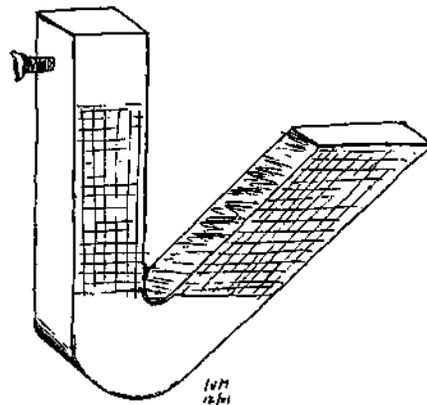


Figure 15-16. Eprouvette of du Me

# BLACK POWDER MANUFACTURE. TESTING & OPTIMIZING

## Wagner's Lever Gauge

Just as Bourne's eprouvette reminded me of bygone pyrotechnic days, Wagner's lever gauge (a.k.a. a recoil eprouvette) reminded me of one of the first scales that I owned. It also had a counterweight and a quarter-circle scale with a pointer arm.

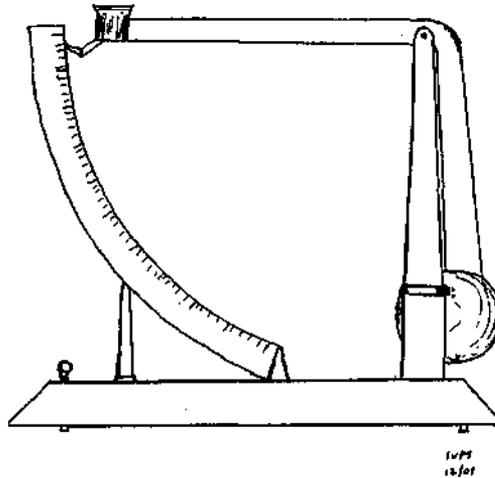


Figure 15-17. Wagner's lever gauge

Just like my scale, Wagner's gauge works on a gravitational principle. It comprises a lever arm, bent at right angles in the center. On one end is a counterweight and on the other a small container. Black Powder is placed in this container and ignited. Its reaction force moves the lever a certain distance, which is measured on the scale. In a sense, Wagner's device can be considered to be similar to a ballistic pendulum, described later in this chapter. This is because the motion of the counterweight is a pendulum-like motion

## Du Pont's Recoil Gauge

Another type of recoil eprouvette was proposed by Alfred V du Pont. No one knows for sure if he actually built this device.

This gauge works on a similar principle to the Wagner's lever gauge, except that the lever here is straight, rather than bent in the middle. A container of Black Powder is ignited on one side and its reactive force is countered by a weight suspended on the other. The amount of movement, which translates to the amount of force, is measured by a measuring tape connected to the counterweight arm.

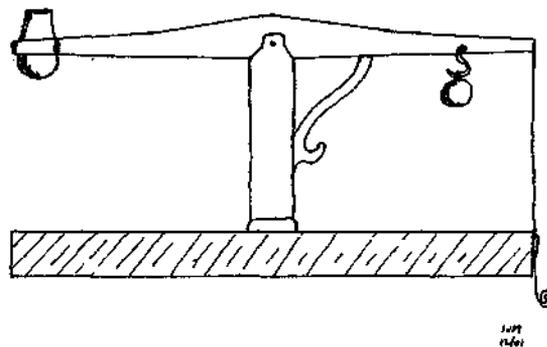


Figure 15-18. Du Pont's recoil gauge

# BLACK POWDER MANUFACTURE, TESTING & OPTIMIZING

## **Ballistic Pendulums**

Ballistic pendulums measure ballistic force that can be translated to a powder's power. They work on the principle of a force being applied to a pendulum which causes it to swing a certain number of degrees. This swing is proportional to the force exerted on the pendulum.

There are basically two different types of ballistic pendulum:

- Pendulums that fire a projectile
- Pendulums that are struck by a projectile.

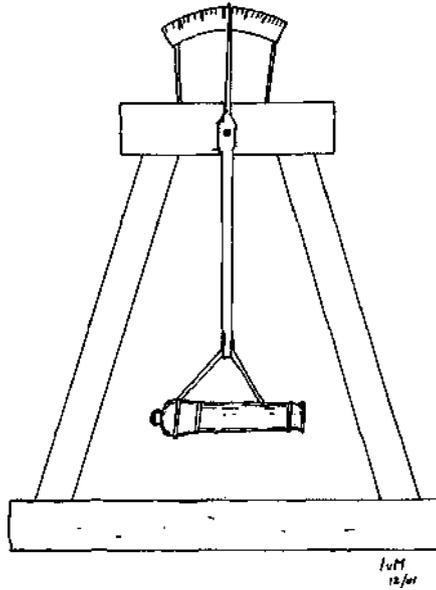


Figure 15-19. Basic ballistic pendulum

The above illustration depicts a basic ballistic pendulum. Ideally the part that fires the projectile should be as similar as possible to the device that uses the Black Powder in the field. Ballistic pendulums that work on the principle of being struck by a projectile are known as *ballistic target pendulums*. An example of one is shown in the illustration below.

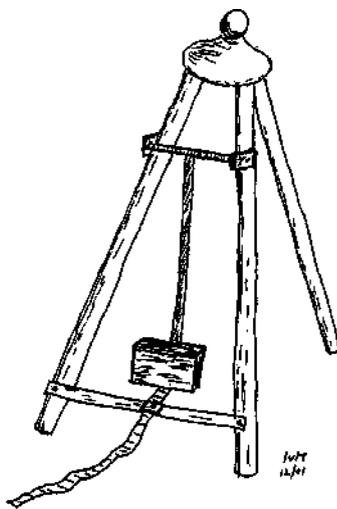


Figure 15-20. Ballistic target pendulum

## BLACK POWDER MANUFACTURE, TESTING & OPTIMIZING

Some ballistic pendulums combine both of the above.

Ballistic pendulums have varied considerably in size. Some have used a cannon or mortar to fire the projectile while others have used smaller arms such as rifles. Three different methods have been used to determine their amount of swing:

- A pointer at the top of the pendulum pointing to the number of degrees the pendulum has moved.
- A pointer at the bottom of the pendulum pointing to the number of degrees the pendulum has moved.
- A measuring tape that is pulled by the moving pendulum, measuring the distance the pendulum has moved.

The last two methods offer a distinct advantage over the first in that they can measure with better resolution.

### **Eprouvettes under Fire**

Throughout the centuries eprouvettes have come under fire from those who have questioned their accuracy. And not without reason; some eprouvettes have shown themselves to be notoriously inaccurate.

The reasons for these inaccuracies are many. Some of the more common reasons are:

- Some of the explosive force escapes without doing any work. Typically this happens either through the touchhole where the powder is ignited or where the lid or projectile comes into contact with the enclosed cylinder that holds the powder.
- Resistance devices such as springs weaken over time.
- Large powder grains cannot be properly tested in small eprouvettes.
- The eprouvette does not properly emulate the conditions under which the powder is used, e.g. a short-barreled mortar cannot properly emulate a long-barreled cannon.

Anything affecting the effectiveness of military weaponry has been subject to scathing criticism by those who have relied on such weaponry in the field of combat. So in studying eprouvettes we are indeed fortunate to have straight, no-nonsense, critical comments from military users.

In 1845 Captain Alfred Mordecai made these comments about mortar eprouvettes:

By comparing the results of the proofs by the eprouvettes with those furnished by the cannon pendulum, it will appear that the eprouvettes are entirely useless as instruments for testing the relative projectile force of different kinds of powder, when employed in large charges in cannon. Powders of little density or of fine grain, which burn most rapidly, give the highest proof with the eprouvettes, whilst the reverse is nearly true with the cannon.

The only real use of these eprouvettes is to check and verify the uniformity of a current manufacture of powder...

...The only reliable mode of proving the strength of gunpowder is to test it, with service charges, in the arms for which it is designed; for which purpose the ballistic pendulum is perfectly adapted...

But for the proof of powder for small arms, the small ballistic pendulum is a simple, convenient and accurate instrument.

# BLACK POWDER MANUFACTURE, TESTING & OPTIMIZING

In 1878 Major WC Hojel gave these comments:

The shooting ranges obtained with the mortar eprouvettes are sometimes erroneously called a measure of the relative force of the gunpowder, However, owing to the shortness of the bore, it is understandable that the said instrument is better suited to the rate of burn of the powder than of the gas pressure the powder can develop. And so it may happen that a very coarse type of powder, giving a very short range with the mortar, can be very satisfactory in a gun with a longer bore.

In 1906 Sir Andrew Noble also criticized the mortar eprouvette, as follows:

The variations in energy of new powders were chiefly due to the method of proof then in use, the Eprouvette mortar, - nothing can be conceived better adapted for passing into service powders unsuitable for guns of that time.

Not everyone agreed with the above observations and comments. Some commentators claimed to get very good results with mortar eprouvettes, while others claimed poor results from ballistic pendulum tests. However, most agreed that mortars with short barrels were useless in determining the properties of coarse powders used in cannons.

Vertical ratchet eprouvettes have also come under fire. Here is a comment made by C von Decker in 1826:

The most unsatisfactory test is the one with a small eprouvette with ratchet bar, in which a few *Gran* [a German medical unit of weight equal to 0.062 grams] have to lift the bar, plus an additional weight, to a certain height. The springs on the bar get loose, the bar will rust; in short the test is very inadequate.

This criticism is somewhat unfair in that it refers to problems that could easily be overcome with proper maintenance of the eprouvette. Others have stated preferences for vertical ratchet eprouvettes, such as Robins (circa 1805) who stated:

On particular occasions, other contrivances are made use of; all of which bear some analogy to the common powder-tries, sold at the shops; only they are more artfully fabricated, and instead of a spring they move a weight, which is a more certain and more equable power. But these machines are yet liable to great irregularities; for as they are moved by the instantaneous stroke of the flame, and not by its continuous pressure, they do not determine the force of the fired powder with that certainty and uniformity, which were to be desired in these kinds of trial.

Robins makes a very revealing comment when he says as they are moved by the instantaneous stroke of the flame, and not by its continuous pressure. Ken Kosanke made a similar comment to me when I asked him about his observations concerning a replica eprouvette that he had used. His comment was that such a device was a peak pressure device, giving readings relating to a peak pressure rather than the average of a longer continuous pressure.

The device used by Ken Kosanke was not a vertical ratchet tester, but a hand-held pistol eprouvette that used a spring, rather than gravity, to provide resistance. However, this device, like its vertical ratchet counterpart, had a small powder chamber that did not allow pressure to build up over time. Thus a longer and larger powder chamber is preferred, but usually not realized in practice in most eprouvettes that have been made.

Before leaving vertical ratchet testers it is worth taking note of some of the tests done by Furtentbach. His tester didn't actually use a ratchet but something similar. For the purposes of this

## BLACK POWDER MANUFACTURE, TESTING & OPTIMIZING

discussion we can consider his tester to be of the vertical ratchet type.

Furtenbach tested different powders, including coarse cannon, finer gun powder, sporting powder and an extraordinary fine powder. His results are shown in the following table:

<u>Powder Type</u>	<u>Height block Moved (inches)</u>
Coarse cannon powder	4
Finer gun powder	5
Sporting powder	9
Extraordinary fine powder	12

These results show a wide difference between coarse and fine powders using the same tester.

While many consider vertical ratchet mechanisms superior to mechanisms using springs, the latter type of mechanism cannot be discounted. Possibly one of the best comments on these is given by Lt. Col. Peter Hawker in his *Instructions to Young Sportsmen*, first published in 1814. Here is what he wrote (in the third edition of 1824):

With regard to the strength and other good qualities of gunpowder, I shall recommend the *eprouvette* (or *powder proof*), whereby we can always be certain of finding out the best; provided, that this machine is properly made, properly used, and nicely cleaned after every fire. I should observe, however, that the *little* trifling things called *powder-proofs*, or *powder-tryers*, which sell for three or four shillings, are as likely to mislead as to inform the person using them.

The proper 'eprouvette' is very correctly made; the wheel on which the graduations are marked is large, and the spring strong; consequently the resistance of the force to the powder is considerable. *The stronger is the better*, for, without the *resistance is strong*, a *correct* proof cannot be obtained; because, if *not sufficiently strong to detain the powder in the chamber* long enough for all the particles to ignite, many of them (especially in powder of good firm grain) will *fly off unburned*, and, of course, a *part only* of the charge would be proved.

The part, attached to the wheel of the eprouvette, which shuts the mouth of the chamber, should be nicely adjusted, that on looking closely at the parts, when in contact, no light can be seen between them; - because, in proportion to the windage, the proof will be lower; and, therefore, incorrect.

Three fires, at least, should always be made in proving, and the *average* taken as the mean amount; - care should be taken, after every fire, to clean the chamber nicely --.

Here Hawker touches on some important attributes of well made eprouvettes, and highlights potential inaccuracies caused by deficiencies both in manufacture and use. He notes that the powder charge in the eprouvette needs to be completely burned for proper operation.

Comments dating back one or two centuries do not give us a fully objective picture of how well eprouvettes performed. And it should be noted that not all the commentators were in agreement. A better picture can be obtained by actually testing eprouvettes. Fortunately such tests have been done. Notable among these are the tests performed by H.L Visser and R.T.W. Kempers. These are well documented in Kempers' Eprouvettes. In addition to documenting these tests, the book has some unique photos of eprouvettes being fired.

The tests documented by Kempers have shown a very wide variation in performance. The best eprouvette yielded fairly consistent results, with a 4.4% mean deviation from the average reading. The worst gave a 35.8% mean deviation from the average reading. Such tests demonstrated the repeatability (or lack thereof of each device tested).

## BLACK POWDER MANUFACTURE, TESTING & OPTIMIZING

Other tests done by Kempers using a single eprouvette with different grades of Black Powder gave different readings for each grade. The highest readings were obtained with 4Fg powder and the lowest with 1Fg. He also tested 5Fg and cannon powder. The cannon powder did not ignite at all while the 5Fg gave very inconsistent results. The most consistent results came from 3Fg. These tests confirm yet again that some testers can give meaningful test results with some grades of Black Powder, while giving non-existent or suspect results with others.

### **Linear Burn Rate Tests**

Linear burn rate tests rank among the oldest tests performed on Black Powder. The earliest and crudest recorded linear burn rate tests were those done by artillery men in the field. Their test usually consisted of pouring powder on the ground over a measured distance, igniting it, and timing how long it burned. The efficiency of the powder was inversely proportional to the burn time.

Certain refinements to the original crude methods have been introduced over the years, one of these being du Pont's quickness tester, which is described in the next section.

### **Du Pont's Quickness Tester**

The term quickness tester was used by the du Pont family of powder makers to describe a comparison type of linear burn rate tester. It should not be confused with modern quickness testers that use an entirely different mechanism for testing Black Powder.

Unlike other linear burn rate tests that required some method of timing the burn rate, du Pont's tester compared the burn rate of one powder with another. This gave the user a ratio value of the one powder's speed to another. Further tests, using two powders at a time, would let one compare any number of powders that one cared to test.

### **Other Miscellaneous Test Methods**

This last section briefly described other methods used over the centuries to test Black Powder. Some of these tests, in various modified forms, are still used today for testing the effectiveness of certain types of ammunition.

Penetration tests of various types and effectiveness have been used both to test the explosive power of powders and the penetration effectiveness of certain types of bullets.

Some of the earliest tests involved simply firing the projectiles used (usually metal balls) into a bank of clay. The strength of the powder was determined by measuring how far the projectile had penetrated the clay. This rather crude test is still used by some to this day. More sophisticated penetration tests used a pile of thin wooden boards stacked together. The effectiveness of the powder was measured in terms of the number of the boards that the projectile penetrated.

Other penetration tests used a specially designed cutting tool, which was attached to a gun barrel. The cutting edge of the tool was pressed against a copper plate and the gun was then fired. The resulting explosive force drove the cutter into the copper plate. The cutting edge of the tool was V-shaped. This resulted in the length of the cut being proportional to the pressure exerted. This length was used to measure the pressure, rather than the depth of the cut, because this method was easier and more accurate.

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Snow and sand were used by some of the American settlers well into the 19th century to test the effectiveness of new batches of powder. They did this by firing their rifles close to the snow or sand and observing the patterns produced. Snow was their first choice, sand the second.

Sink, don't swim is probably the best way to describe a test that involved placing grains of Black Powder in a container of water. If the powder was "good" it sank, if not, it floated. Common sense says that this tests only the density of the powder. Perhaps at that time differences in density defined differences in performance more than other properties did.

Glass, brass and copper plates have been used to test how well Black Powder has been incorporated. A small pile of Black Powder ignited on a glass, brass, or copper sheet should leave a minimal amount of residue if it has been properly incorporated. Conversely, badly incorporated powder leaves large amounts of residue, mainly in the form of melted potassium nitrate and sulfur.

### **Further Reading**

For extensive descriptions of eprouvettes and many of the test methods described in this chapter, the following book is recommended:

RTW Kempers, *Eprouvettes, A Comprehensive Study of Early Devices for the Testing of Gunpowder*, Royal Armouries, (1998)

Much of the material in this chapter has been gleaned from the above book, and is mostly a very brief summary of what Kempers describes.

Another classic book on the history of Black Powder that touches on historical test methods is:

J.R. Partington, *A History of Greek Fire and Gunpowder*, The Johns Hopkins University Press, (1999).

## *16 -- Modern Test Methods*

### **Introduction**

This is the final chapter on testing, and describes modern methods. The term *modern methods* is a relative one in the sense that some of the methods described have been around for a long time, but are still in use today.

This chapter is not limited to test methods, but also describes equipment that can be used in certain tests. Included is a somewhat long discussion on temperature measurement, something that may be relevant to some tests but not to others. I had to include temperature measurement somewhere. It relates both to testing and to charcoal making, and had to find a home in either one of these sections of the book. It ended up here.

### **Linear Burn Rate Tests Revisited**

Linear burn rate tests (a.k.a. flame spread rate tests) are among the oldest and easiest methods of testing Black Powder. They also have the dubious rating of being a fairly accurate to horribly inaccurate measures of a powder's potency. Part of this inaccuracy problem is due to not enough care being taken to ensure that each test is performed in identical fashion to previous tests. If tests are not done in a repeatable way, then one can expect repeatability problems with the results. Linear burn rate tests measure a powder's flame spread characteristics. These may or may not correspond to the powder's ability to act as a propellant. This is the greatest weakness in this type of test.

As described in the previous chapter, the earliest and crudest recorded linear burn rate tests were done by artillerymen in the field. Their test consisted of pouring powder on the ground over a measured distance, igniting it, and timing how long it burned. Similar tests have been carried out by many others who have placed their powder on sheets of metal or wood.

I have personally used such a method by creating a string of powder approximately one meter in length on a metal sheet. It can be used as a ballpark comparison test if one is careful to maintain consistency in one's method of testing. This can be quite a challenge. Essentially one should try and maintain consistency in:

- Volume or weight of Black Powder used
- Even distribution of powder along the entire length
- Start and stop time measurement
- Ambient temperature.

Perhaps the most difficult of the above objectives is to ensure an even distribution of the powder. A good way of achieving this is to accurately cut grooves in the wood or metal and to fill these grooves with powder. Excess powder is scraped off with a non-sparking spatula. An easier method is to use small-channeled tubing made from materials such as aluminum. The grooves found in tongue-and-grooved wooden planks may be another ready-made option.

Another method is to use a V-block. This is usually made from a length of right-angled metal such as *angle iron*. Angle aluminum is also worth experimenting with. Such a method is shown in the illustration below.

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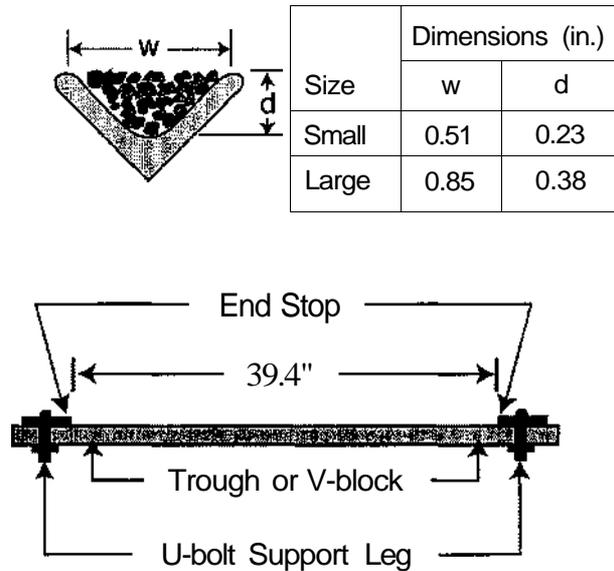


Figure 16-1. V-block made from a piece of angle iron.

In a V-block the powder is filled to the top of the V, as shown in the illustration above.

If one doesn't opt for the just-mentioned methods of controlling powder distribution one should not be tempted to skimp on the amount of powder used. Although there is no "ideal" weight or volume, enough Black Powder should be available to ensure that there is no discernible space between powder grains. Failure to do this may result in longer and less consistent burning times.

Linear burn rate tests require accurate time measurement. Here are three ways to do this:

- Record the burning powder on video
- Use a hand stopwatch
- Use an electronic sensor-actuated stopwatch.

The first method requires a video camera plus some method of accurately counting video frames. This feature might be found in the camera itself or the VCR used to play back the tapes. A hand stopwatch is the simplest and easiest method to use and also the cheapest. Its downside is its relative inaccuracy compared with the other two methods. An electronic sensor-actuated stop watch can give very accurate results if used properly. These are quite easy to make. Such a stopwatch is described elsewhere in this chapter.

Linear burn rate tests have their limitations. Such tests measure the powder's flame spread ability that may not necessarily translate to its characteristics as a propellant. They are useful in that they can be done with a limited amount of equipment and can give good comparison tests with different batches of powder using the same manufacturing method. Where flame spread characteristics are more important than propellant properties, linear burn rate tests may yield more meaningful results.

## **Strand Burn Rate Tests**

Strand burn rate tests are in some ways similar to linear burn rate tests, but different in other ways. The major difference here is that strands are comprised of compressed powder, while linear burn rate tests use powder in its normal granular form. Strands thus may not accurately

# BLACK POWDER MANUFACTURE, TESTING & OPTIMIZING

represent the behavior of the granulated powder. However, this particular attribute may be irrelevant, depending on the aims of the tests.

Strands used in strand burn rate tests are classified as:

- uncovered strands
- covered strands.

**Uncovered strands** suffer from the disadvantage of having two potentially different burn rates. This is because burning can occur on any exposed surface. Ideally burning takes place only at the end of the strand. If it does not, then the measurement data is compromised.

**Covered strands** burn only at the end where they are ignited, like a cigarette. Covered strands are thus preferred to uncovered strands. This covering is achieved by using some form of surface inhibitor. One way of doing this is to paint the surface of the strand with a type of fire resistant material. Another way is to compress the powder into a thick-walled cardboard tube.

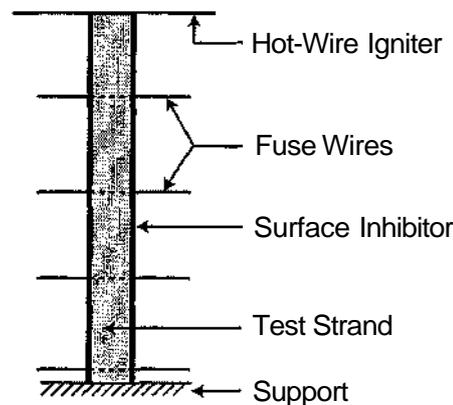


Figure 16-2. A strand used for testing

A basic setup for strand testing is shown in the above illustration. This drawing is not to scale, as often the diameter of a test strand is much smaller than its length. Some of the earlier strand test methods (developed during World War II) used strands 1/8 inch in diameter. More modern tests have typically used 3/16 inch diameter strands. The cross section of a strand can be either round or square.

In the setup shown in the illustration the strand is stood vertically on a horizontal support. Its top end is ignited with a hot-wire igniter. Fuse wires are spaced at measured distances apart and are connected to an electronic timer that measures the burn rate.

Another method is to compress the powder into a thick-walled cardboard tube to a measured depth. This should be done by adding the powder to the tube in small increments and compressing it with a consistent pressure. The tube is then suspended horizontally and ignited on one end with a blowtorch. The burning is timed until it has burned all the way to the other end. A good way of timing this is to film the whole burning sequence with a video camera.

## Quickness Tests

The term *quickness test* can mean different things to different people. Historically certain linear burn rate tests were described by some as quickness tests. Today the term is generally understood to mean an electronic testing device that measures pressure changes in a closed container where a sample of Black Powder has been ignited.

## BLACK POWDER MANUFACTURE, TESTING & OPTIMIZING

Modern quickness testers are thus a type of *closed bomb* tester. Some, in fact, use the term *closed bomb* tester when referring to quickness testers.

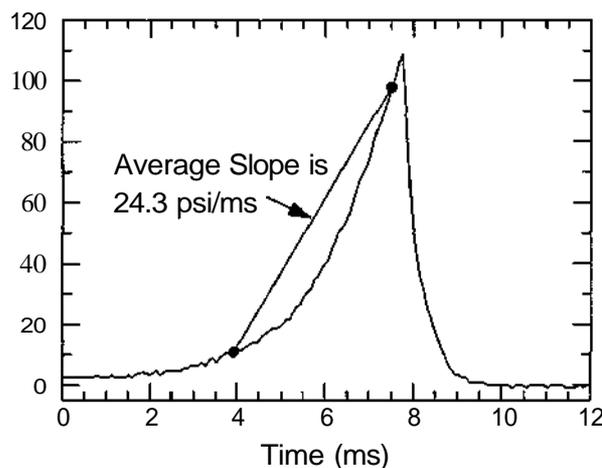


Figure 16-3. Graph of a quickness test

The above graph is taken from an actual quickness test performed on Black Powder used in the American Civil War. This graph shows the output of a pressure transducer over a very short period of time.

### **Closed Bomb Tests**

A closed bomb is a closed container in which an explosive is ignited or detonated. In the case of Black Powder, the powder is ignited, resulting in an explosion. In its crudest form, such an enclosed bomb is buried. The resulting explosion is made large enough to burst the container and create a sizable crater in the ground. The size of this crater determines the power of the powder.

More refined methods rely on exploding a lot less powder in much smaller, but usually stronger, containers. Thus the explosive power is confined to the container where its properties such as changes in pressure and temperature are measured. Modern quickness testers use this method.

From the literature, it appears that the terms *closed bomb* and *quickness tester* are used interchangeably. Further discussions on closed bomb testing can thus be found under the section on quickness tests in this chapter.

### **Pyro Golf**

Pyro Golf is a modern form of mortar eprouvette that has become very popular among amateur pyrotechnic enthusiasts. A more refined version of this device is used in professional pyrotechnic laboratories. Guy Lichtenwalter is generally credited with creating Pyro Golf, with Ken Kosanke identifying needed improvements to turn Pyro Golf into a viable scientific instrument.

The principle on which Pyro Golf operates is to project a standard golf ball into the air and to measure the golf ball's time of flight. Older mortar eprouvettes relied on accurately estimating the height a projectile reached or the distance it traveled. Pyro Golf is thus easier to use than its predecessors, and theoretically more accurate. Laboratory versions measure the muzzle ve-

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locity of the projectile and usually stop the projectile's flight shortly after it has left the mortar tube.

Modern technology gives Pyro Golf a number of advantages over its forebears for the following reasons:

- Its parts can be more accurately machined
- A "standard" golf ball is better than many past standards
- Electronic timing can be done very accurately
- Temperature and pressure variations can be accurately measured and thus can be controlled or compensated for.

Pyro Golf also has the advantage of a longer barrel than its short mortar predecessors. This makes it a better simulator of actual use, assuming that most of such applications are for lifting fireworks shells. Like many other testing methods, Pyro Golf has its detractors. Many of the criticisms aimed at Pyro Golf have been of a theoretical nature, based on little or no empirical evidence to substantiate the various claims. Some have become totally sidetracked into discussions on the ballistic properties of golf balls - *hanging* golf balls and *dimpled* golf balls!

Just how accurate is Pyro Golf? By most amateur/hobbyist standards Pyro Golf is probably the most accurate method around, assuming the application is typical of amateur/hobbyist pyrotechnic pursuits. Those interested in sporting Black Powders would probably do better to get their test data from tests done with actual firearms. Flame spread rate applications would be better tested with linear burn rate tests or some other method that focused on flame spread, such as strand burn rate tests.

Does Pyro Golf have any disadvantages? Yes it does. First, a properly built Pyro Golf apparatus is beyond the reach of the average hobbyist. This means that most who wish to use such a device need to wait for the odd window of opportunity that might present itself at gatherings such as the annual PGI Convention. It's not a method that most can use to test their batches of Black Powder on a regular basis.

Another disadvantage of Pyro Golf is the size of a standard golf ball itself. While a golf ball is comparable in size to small shells, comets, and even Roman candle stars, it is a lot smaller than the larger fireworks shells. Larger projectiles typically use slower powders. Such powders may display mediocre performance in Pyro Golf tests, but perform adequately when propelling larger projectiles. Yes, one can still test such powders with Pyro Golf. Results from such tests, however, should be interpreted with caution.

What follows is a general description of the Pyro Golf apparatus itself. Here I have included details about both the apparatus used by hobbyists to compare powders and the laboratory apparatus used by Ken Kosanke of PyroLabs, Inc. I have had the pleasure of visiting PyroLabs and examining the laboratory version of Pyro Golf.

The Pyro Golf Black Powder tester is basically a test mortar, like its predecessors. However, unlike the mortar eprouvettes of days gone by, Pyro Golf has a number of significant refinements. These refinements give Pyro Golf a distinctive edge when it comes to repeatability of measurements. The two most notable differences between Pyro Golf and other test mortars are that Pyro Golf has a long barrel (relative to the size of its projectile) and a carefully designed breech plug.

At this point we need to pause and qualify the statements just made. In the strictest sense, Pyro Golf shouldn't be compared to mortar eprouvettes which are used for testing powders to be used in cannons because the applications of these devices are different. However, virtually

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all mortar eprouvettes suffered from problems relating to short barrels. And none had (to the best of my knowledge) breech plugs comparable to Pyro Golf.

Pyro Golf also owes its present accuracy to the fact that it was critically examined during its infancy to determine any flaws in its design and operation. Conversely some mortar eprouvettes were only critically examined after centuries of use!

The projectile used in Pyro Golf is usually a standard golf ball. Some tests have also been done using a short length of 1.75 inch diameter nylon rod. Both of these have cross sectional areas close to that of the mortar tube, an important consideration for accurate testing. The laboratory version comprises:

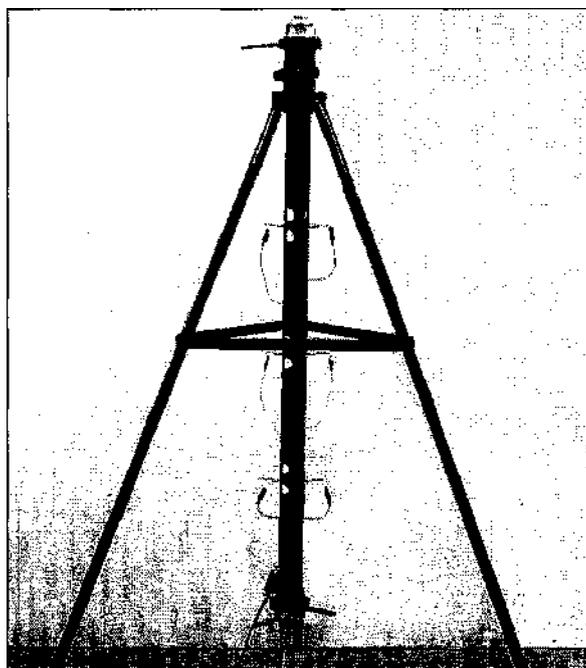
- 1) A mortar consisting of a breech plug and barrel.
- 2) An electronic time-of-flight measurement system.
- 3) A projectile arrester.

The hobbyist version is similar with some notable differences:

- 1) No projectile arrester
- 2) No built-in time-of-flight measurement.

The hobbyist version is designed to work in an open outdoor area, whereas the laboratory version is designed to work in a laboratory building. The laboratory version thus needs to constrain the projectile after it has left the barrel. The hobbyist version sends the projectile into the air and measures the flight time from the time it leaves the mouth of the barrel to the time it takes to hit the ground. This is typically in the order of seconds, this time being measured with stopwatches. The laboratory model measures the velocity of the projectile while it is still in the barrel, this time being in the order of milliseconds. This timing is done by a high speed electronic counter, triggered by trip wires that are broken by the flight of the projectile.

The following figure shows the laboratory model, supported by a tripod.



*Figure 16-4 . Laboratory Pyro Golf instrument and tripod*

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The breech plug of the laboratory model is a bit more complex than the hobbyist version. This is because it is fitted with temperature and pressure sensors, and is ignited with an electric match. The hobbyist model is typically ignited with fuse rather than a match. A cross sectional drawing of the laboratory model breech plug is shown in the following figure.

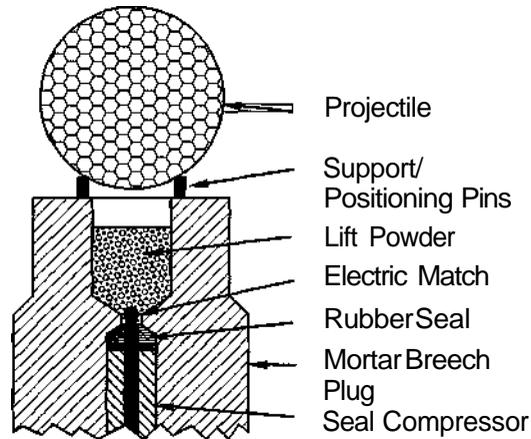


Figure 16-5. Cross sectional drawing of breech plug

Another enhancement found on the laboratory model is a pair of small cartridge heaters used to keep the breech plug at a constant temperature.

The next figure shows the breech plug with the rubber seal, seal compressor, and electric match removed.

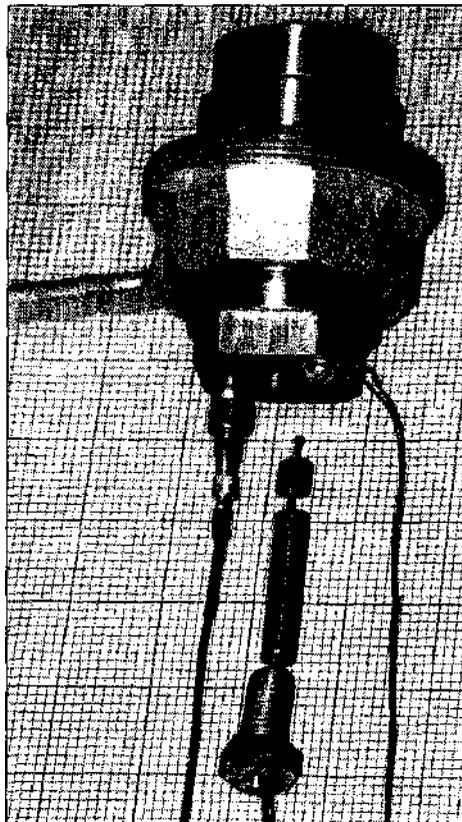


Figure 16-6. External view of breech plug

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Some readers may have noticed three timing trip-wires in the illustration showing the complete laboratory instrument. One could use only two, but three are better for more accurate measurements. Using three trip-wires lets one measure the velocity of each firing twice instead of only once. Each trip-wire is spaced one foot from its neighbor. A close-up view of a trip-wire is shown in the following figure.

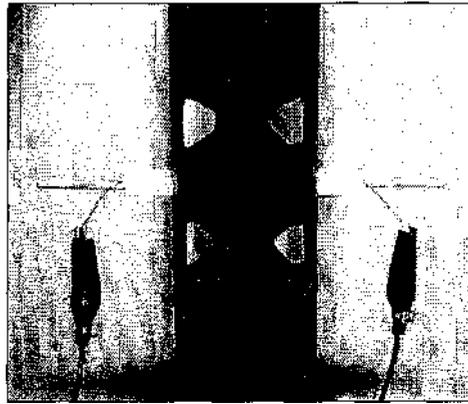


Figure 16-7. Close-up view of timing trip-wire.

Above and below each trip-wire is a set of exhaust holes, allowing gases to exhaust from the mortar tube. The arrester portion is positioned just beyond the third set of exhaust holes. This is basically an enclosed pipe with a hard rubber disk mounted at its end.

Several variables have been shown to influence the accuracy of Pyro Golf. Some of the more significant variables are:

- Point of ignition
- Temperature
- Cleaning
- Particle size.

**Point of ignition** defines the point where the Black Powder being tested is ignited. Varying this point of ignition has given wide discrepancies in results, ranging from 10% to 50%. Thus it is important to ignite the Black Powder at the same point in each test. The best point of ignition is right at the bottom of the test chamber.

**Temperature** can have a marked influence on test results. Some tests have shown an approximate increase of 50% in muzzle velocity over a range of 16 to 130 °F ( -9 to 54 °C). The temperature of the Pyro Golf apparatus is increased slightly with each firing, and this increase needs to be taken into account when interpreting data. Using the block heaters described earlier does a lot to get around this problem. The instrument is heated to a temperature of 80 °F (27 °C), somewhat above average room temperature. That seems to be a good temperature for reasonably accurate temperature control under test conditions.

What I personally like about this approach is that the Black Powder itself can be heated to the same stable temperature. This eliminates another possible variable that could influence the results. My personal experience with other types of testing has shown that the temperature of the Black Powder can influence the temperature of ignition and speed of burning. My experiences have been confirmed by others, and are consistent with the theory relating to ignition and propagation of Black Powder.

**Cleaning** is important, irrespective of what apparatus is being used for testing. Pyro Golf is no exception. Black Powder has a rather nasty habit of leaving solid residues in any container that

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it is fired in. These residues collect in the barrels of mortars and guns, promoting rust or other types of corrosion, and also affecting performance from one firing to the next. In Pyro Golf the affect on performance is demonstrated with increased muzzle velocity.

A simple way around this problem is to clean the barrel of the mortar after each firing. This is far too time consuming. Even cleaning it after three firings it too time consuming. An acceptable method is to run three tests with three firings in each test, a total of nine firings. In each test the order of firing is changed. This system effectively cancels out differences brought about by residues accumulating in the barrel. After each set of nine firings, the barrel is cleaned.

**Particle size** is possibly the least understood and most controversial variable affecting accuracy. And maybe the reason for most of the controversy is that very few understand this issue properly.

Particle sizing is discussed in detail in the section dealing with sieves. This discussion won't be repeated here. However, it is important to appreciate that accurate and tight sizing of Black Powder granules is very important for consistency and repeatability of results.

### **Pyro Baseball**

Pyro Baseball is a relative newcomer that is modeled on Pyro Golf. Here a baseball is used instead of a golf ball. An arguable advantage here is that a baseball more accurately represents the size of a fireworks shell than a golf ball does. A baseball can also be fired from a fireworks mortar, so no special mortar is required.

Pyro Baseball, when compared to Pyro Golf, also has some perceived disadvantages:

- Baseballs are not as aerodynamically well-designed as golf balls
- A standard fireworks mortar tends to be a less well-made device than a Pyro Golf mortar
- Baseballs need more powder for each test.

Will Pyro Baseball inspire future variations such as Pyro Football, Pyro Soccer or Pyro Bowling? Who knows? Maybe some nut will try, and undoubtedly some would welcome the diversions that such activities offer. Practically speaking, such variations will tend to offer more disadvantages than advantages.

### **Photography**

High speed cinematic photography has been an important tool in the study of explosives. Unfortunately high speed equipment comes at a high price tag, way beyond the pockets of most.

High speed is precisely what it claims to be, filming an object at the rate of hundreds or even thousands of frames per second. Standard cinematic or video cameras (with typical frame rates of 25 or 30 frames per second) are very slow in comparison. But such cameras can be used in testing Black Powder, depending on the method used.

A video camera cannot meaningfully record an explosion fast enough to allow a serious study of it. It can, however, be used to record slower phenomenon such as the burning of unconfined Black Powder. Time stamping is a useful feature to have. A *must have* is the ability to view the results frame by frame. Many VCRs now offer this feature.

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## **Electronic Sensors**

Electronic sensors are electronic devices that sense or measure properties such as temperature or pressure by converting these properties into electronic signals. Another name for a sensor is a transducer. And yes, there are the pedantic crowd who spend many hours writing to editors of instrument and control magazines spelling out the "differences" between a transducer and a sensor. We won't pursue this peculiar distraction here.

Perhaps the cheapest and crudest sensor used in Black Powder tests is simply a thin piece of wire. Two such pieces of wire can be used to determine the velocity of a projectile. Here the wires are placed in the projectile's path a set distance from each other. The projectile breaks the wires and the time measured between these breaks determines the velocity of the projectile and the power of the powder. The wires themselves are connected to an electronic stopwatch which starts and stops when the electronic circuit senses that the wires are no longer passing current. Another variation on this theme is to use very thin wire that can be quickly burned through by some burning Black Powder.

Measuring the actual temperature of burning Black Powder is quite a challenge in itself, and apart from pure scientific research, probably has little practical value. Also one has little hope of getting any meaningful temperature with conventional temperature sensors such as thermocouples and resistance thermometers. This is because conventional sensors cannot respond fast enough. One can, however, use sophisticated optical sensors to measure the radiant heat given off by the burning Black Powder. These can be very expensive.

Temperature measurements are useful in ensuring the repeatability of tests. Test results do vary with ambient temperature, so it may be important to either control or record this variable. The same criterion applies to test equipment. Black Powder burned in testers such as mortar or quickness testers does raise the temperature of the tester. This increase in temperature needs to be taken into account and may in itself yield some meaningful test data.

The three most popular electronic sensor types used to measure temperature in industrial processes are:

- Thermocouples
- Resistance thermometers (RTDs)
- Thermistors.

**Thermocouples** generate a small electrical voltage in proportion to temperature. Typically this voltage is in the thousandths of a volt (milli-volt) range. This tiny voltage is amplified by an electronic instrument and scaled appropriately to give a temperature reading. An important characteristic to note is that thermocouples measure differences in temperatures, not actual temperature values. Usually this temperature difference is the difference between the temperature detected at the tip of the thermocouple probe and the ambient temperature. Thus an instrument used to measure temperature with a thermocouple needs to also be able to measure its own ambient temperature and to compensate its reading accordingly.

**Resistance thermometers** are precision made resistors that change their resistance in proportion to temperature. They are also called *resistance temperature detectors* or *RTDs*. The term RTD is the most popular term for these devices. Most are made of platinum and are thus very expensive. RTDs are the sensor of choice for very accurate laboratory work. The most common RTD has a resistance of 100 ohms at 0 degrees Celsius and changes about 0.38 degrees for every one degree change of temperature. Electronic instruments measure these resistance changes by passing a very small current through the RTD that converts its resistance value to a voltage.

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**Thermistors** are similar to RTDs in that their resistance changes with temperature. They have the advantage of being a lot cheaper than either thermocouples or RTDs. Their big disadvantage is that their resistance change is not linearly proportional to temperature. Thermistors are generally avoided where accurate temperature measurement is required and tend to be used more in protecting electronic devices from overheating.

## Using Thermocouples

Thermocouples provide a relatively inexpensive but acceptably accurate way of measuring temperature. They are very versatile in that they can be used in a number of different ways in a whole lot of different applications. Thermocouples have an added advantage over RTDs in that they are generally far more robust. Thermocouples do, however, have traps for the unwary.

The first trap is that some devices going under the name *thermocouple* are not thermocouples in the true sense. A thermocouple is a device that produces a voltage proportional to the temperature it is sensing. Some so-called thermocouples are mechanical switching devices used as protection mechanisms in gas fired systems. These are often referred to as *thermocouplers* rather than *thermocouples* but some still use the word *thermocouple* to describe these.

The second thing to be aware of is that there are different types of thermocouple designed to measure different temperature ranges. In the USA these are given letters such as E, J, K, R, S, T and W. The first three (E,J,K) are the types we are most likely to use in Black Powder testing. Type J is the most popular thermocouple type, followed by type K. Thermocouples made in Europe use a different naming convention such as Iron-Constantan (or Fe-CuNi) for type J and Nickel-Chrome-Nickel (or NiCr-Ni) for type K. Such thermocouple also differ slightly in their electrical characteristics to their American counterparts.

Third, one should always remember that a thermocouple measures temperature differences, this difference usually being the difference between the temperature measured and ambient (room) temperature. This requires special consideration in the type of cables used to connect thermocouples to measuring instruments. The correct way is to use thermocouple extension cable. This is cable made from the same material as the thermocouple. In more expensive thermocouples, such as type R and S that are made from platinum and rhodium, such cables are made from different, cheaper materials. These cables are more accurately described as *compensating cables*. Care must be taken with both extension and compensating cables to wire them up with the correct polarity. If this is not done, the thermocouple will still work, but large errors can result.

Correct polarity is another trap for the unwary. For some reason, the most popular thermocouple types (J and K) use red colored cable for the negative side of the extension cable. This is opposite to the normal electrical convention that uses red for positive. Many have been caught out by this one because most persons with an electrical background assume that red designates positive polarity.

Some thermocouple cables get discolored with age and one cannot tell their colors apart. There are two simple tricks to get around this problem. In both type J and K thermocouples the positive side of the cable is more easily attracted by a magnet than the negative side. Another way of determining polarity is to twist two bared ends of the cable together and heat them with a match or lighter while measuring their output voltage on the other ends. The actual voltage value is irrelevant but one should be able to tell polarity by noting which test lead is connected to which side of the cable and whether the meter reads positive or negative. If the meter reads positive the positive test lead is connected to its corresponding positive cable side - the same with the negative. If the meter reads negative then the positive test lead is connected to the negative side - vice versa with the negative test lead.

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## Using RTDs

RTDs tend to cost a lot more than thermocouples and tend to be more fragile. For this reason they are generally used in place of thermocouples only when high precision is required.

Unlike thermocouples, one doesn't have to worry about polarity with RTDs; they are resistors and their polarity is irrelevant. They also do not require special cables. Ordinary electrical cables using copper wires are used. This simplicity ends here. RTDs need to be connected in special ways to ensure maximum accuracy. These ways are specified as:

- two-wire connected
- three-wire connected
- four-wire connected.

**Two-wire connected** RTDs are to be found in older implementations or RTD-based equipment. This system is cumbersome and less accurate than the more modern two- and three-wire systems. Nevertheless it is described here because older systems tend to be sold off cheaply and one may still be able to get one's hands on such equipment.

RTDs are resistors that are fed with a constant current that is converted to a voltage. Electronic instruments do not measure resistance directly but measure the voltage across a resistance. All electronic cables have a certain amount of resistance and this resistance is effectively added to the RTDs resistance, introducing a certain margin of error. To complicate matters, the resistance of this cable also changes with changes in temperature. Two wire systems attempt to accommodate this problem by ensuring that the cables connected to the RTD have a standard resistance, which is typically 10 ohms.

To ensure this standard value, a two-wire instrument is supplied with a small bobbin of resistance wire. The total resistance value of this bobbin of wire plus the cable resistance should equal the standard value (e.g., 10 ohms). To achieve this, the user needs to very accurately measure the resistance of the cable and subtract this amount of resistance from the bobbin.

If the RTD is used as a bench-top instrument with a very short length of cable, then one can get away without the above exercise. But there are still things one needs to watch out for. Typically an older used system will have some of the wire wound off the bobbin already, so the resistance of the bobbin needs to be accurately checked before use.

Two-wire connected RTDs still suffer from accuracy problems even if the standard resistance is set to its proper value. This is because of resistance differences in the cable due to temperature changes. These differences are partly overcome in three-wire systems and completely overcome in four-wire systems.

**Three-wire connected** RTDs eliminate half of the cable resistance temperature change problem and do not need a standard resistor. This reduces the effort involved in connecting such a system.

**Four-wire connected** RTDs eliminate the cable resistance change problem. Such systems offer the highest degree of accuracy. With stable and accurate electronic instrumentation accurate temperature measurements are possible to within a tiny fraction of a degree.

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## **Electronic Test Equipment**

Electronic test equipment is connected to electronic sensors in order to convert their signals to meaningful data. Typically these data are time, temperature and pressure. Some tests require both the measurement of time and some other component such as pressure in order to record the profile of a measurement over a time period. An example of this is a quickness tester that measures the rate at which pressure rises.

## **General Purpose Electronic Timers**

A cheap \$6.95 stopwatch may be more than adequate for certain tests where accurate timing is a must. Such a timepiece, however, may prove inadequate for certain timing requirements.

Timing of very short intervals is not the job for a hand-held stopwatch, but rather for a fast, sensor-actuated electronic timer. Yes, you can tear open your stopwatch and wire in some extra circuitry to trigger it with triggers other than a fast finger or thumb. But the hassle may not be worth it. Fortunately, electronic timers are easy and cheap to build.

An accurate electronic timer needs the following:

- A stable and accurate clock pulse generator
- Dividers to divide the pulses into second pulses and fraction of a second pulses
- A foolproof method to stop and start the counter
- Display to show the count value.

Such a timer can be built with inexpensive components that are easy to obtain. While it has not been my intention to include electronic designs and schematics in this book, I can at least point aspiring timer builders in the right direction.

Many applications circuits can be obtained from the manufacturers of electronic components. Most of these manufacturers have web sites where one can download application circuits. My approach usually is to get on an electronic retailer web site such as *DigiKey* or *Jameco*. I then identify a component that could possibly meet my needs and then use their links to obtain a specification sheet or application note. Application notes often contain complete circuits that frequently can be used as is, or with some modification.

Manufacturers such as *Intersil* offer a variety of chips that contain complete counters. These require only a few extra components such as a display and switches to create a complete solution. Other manufacturers offer inexpensive building blocks that one can put together to build a counter. To clarify our picture of what is required, it is helpful to look at these building blocks in detail.

**Quartz crystals** are used to provide very accurate timing pulses. Today one can buy quartz crystals packaged together with other components to give a complete oscillator. This is the easiest route to follow. Typically one would use a quartz crystal oscillator having a frequency of 100 kHz or 1 MHz. If one were wanting to measure time to within one hundredth of a millisecond or one thousandth of a millisecond one would use direct outputs from either one or the other of the just-mentioned oscillators. If one wanted less accuracy, one would use a divider chip.

**Divider** chips divide the number of pulses by a specified number, such as 10. For example, if one wants to measure to the nearest tenth of a millisecond, one would divide the pulses from a 100 kHz counter by 10 or a 1 MHz counter by 100. Typical divide by ten counter circuits are the 7490 series in TTL logic or 4017 series in CMOS logic.

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Similar to divider chips are counter chips that both divide and count the number of pulses. These chips are called BCD (binary coded decimal) counters. Many of these can now directly drive displays known as 7 segment displays. Some chips contain two or three BCD counters with display drivers.

**Foolproof methods** to start and stop the counter are needed to ensure that the counter starts and stops at the right time. There are many ways to do this in electronics. Most involve a circuit known as a *flip flop*. Such a circuit uses a start switch to tell the flip flop to change its state to where it allows the counter to start counting and a stop switch to change its state to where it tells the counter to stop counting.

**Displays** are most cost effectively implemented by using 7 segment LED displays.

### **Epreuves Revisited**

The last chapter described epreuves in depth. Are any of these devices useful in doing Black Powder tests today? Given their somewhat bad track record, one would be tempted to say no. Personally, I would not go out of my way to get hold of an epreuve to do testing. But if somebody gave me an epreuve, I would be tempted to test how well it performed.

Apart from some replicas that are still being made today, most epreuves in circulation are collector's pieces, and their chief value should be viewed as such. It would be a great pity (some would say a crime) to damage or destroy a valuable collector's piece just to find out how it performed as a tester.

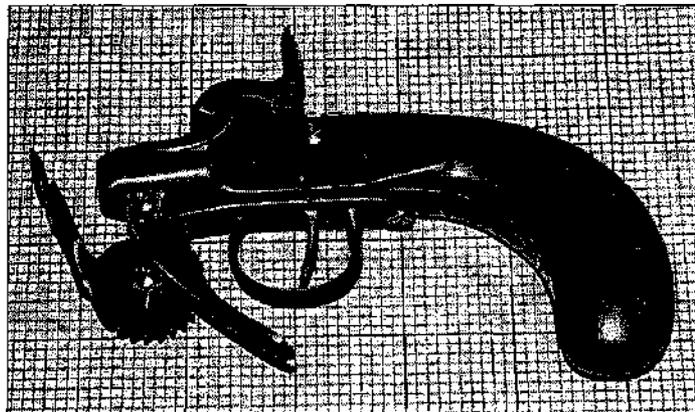


Figure 16-8. A working replica of a pistol epreuve

Collectible epreuves should be treated the same way as antique collectible firearms. It is best not to fire them as deterioration over the years could lead to a bad malfunction that could cause a nasty accident. Admittedly some antique firearms are still fired today, but usually only after undergoing careful inspection by an expert in the field. Antique epreuves should be treated in the same way.

### **Palm-of-Hand Tests Revisited**

Some have tried the *palm-of-hand* test in recent times and found it wanting. In theory at least, good powder ignited in the palm of one's hand should burn without causing discomfort. In practice - yes, no, and maybe.

Years ago, while on a Boy Scout hike, the subject of thistles came up. This perhaps had to do

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with the fact that there were plenty of thistles in the area we were hiking through. One of our group started talking about the perception that if one grasped a thistle firmly it would not sting one. Several others challenged him on this point, and to prove his assertion he got hold of a thistle and proceeded with a real live demonstration.

He placed the thistle carefully on the palm of one hand and then hit it squarely with the other. "See, what did I tell you!" he proclaimed triumphantly. Later he was observed to be wringing his hands together, obviously in pain. In theory, he was probably right. In practice, his technique was wrong. He should have just grasped the thistle firmly in the palm of one hand, and not tried his own variation.

The above anecdote may have something to do with the fact that palm-of-hand tests work for some and not for others. There are many variables involved in tests of this nature such as the amount of powder and the method of ignition. Other more subjective variables are the toughness of each individual's hand and individual pain thresholds. However, there is no clearly defined *right way* of conducting such tests.

Kemper has performed two such tests with different powders, igniting the powders with a red-hot nail. Both experiments resulted in slight burns on the palm of his hand. These experiments were done with good quality powders, and not followed (for understandable reasons) with tests on inferior quality powders.

The theory behind palm-of-hand tests rests on the fact that a good powder will burn with minimal residue. Thus a good powder will leave a minimal amount of hot residue on one's hand. Actual palm-of-hand tests have verified this fact, so how then can one's hands end up getting burned? I think I may have the answer to that question.

Some of my own experiments in pyrotechnics got me some very sore fingers. Here I literally burned my fingers. The first occasion was when I lit some stars with a match. These stars were made with aluminum and dampened with alcohol. The alcohol had not fully evaporated when I lit the stars, and started burning with its characteristic blue flame. Impatiently I rubbed one of the stars with the end of the match, trying to encourage it to ignite properly. It did, with a very bright hot flame, burning my fingers in the process.

On another occasion I ignited some residue from some magnesium stars. I decided to flick a burning match on the residue, rather than hold the match to it. After all, I had learned my lesson from the just-described experience. The star residue ignited quite violently, leaving me nursing sore fingers yet again.

In both these unhappy experiences none of the burning came from residue stuck to my fingers. The burns came purely from having my fingers too close to a brief-burning flame with a lot of heat. While Black Powder does not burn as hot as aluminum or magnesium, it still burns hot enough to cause discomfort to any part of one's anatomy held close enough to it.

### **Lead Tubes**

The lead tube method of testing Black Powder is still used today. In my recent discussions with representatives from GOEX, I was told that they still use this test in comparing burn rates of powder used in Black Powder firearms.

What is the lead tube method? It is simply a lead tube that is stuffed with Black Powder and then flattened. The tube is cut to a specified length and ignited on one end. The burning time from the one end to the other is measured with a stopwatch. The time taken to burn the whole length is a measure of the strength of the powder, the shorter the time the stronger the powder.

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In real life testing situations the lead tube method is used to determine if the strength of the powder is within a certain range. Powder that burns too quickly or too slowly fails the test.

### **Chemical Analysis**

Commercial Black Powder makers such as GOEX perform chemical analysis tests to determine consistency in manufacture. In addition to quality control, such tests give an indirect indication of a particular powder's expected performance.

Such tests are usually beyond the reach of the average amateur, and are probably quite unnecessary in most amateur situations. Where consistency in performance needs to be measured, any number of other test methods could be used.

### **Density Measurements**

The density of Black Powder was originally measured by the mercury displacement method. This involved using mercury, an element that is both very expensive and very hazardous to use. Modern laboratory density measurements use a helium displacement method. This avoids the hazards associated with mercury but is still beyond the budget of most.

A rather crude way of measuring density is to place a measured weight of Black Powder in a laboratory measuring cylinder and measure its volume. Density is given by the mass divided by the volume.

The just-mentioned method is crude and is probably useful only in doing rough density comparisons with powders made the same way and which have the same size granules.

### **Sieve Sizing**

Sieve sizing, like density measurements, does not in itself measure the performance of a powder. Rather it is a measure of one of the physical properties of a powder.

Sieve sizing can be crucial to getting accurate results from most tests, and is often overlooked. Most tests that test granulated Black Powder (as opposed to very fine meal powder or powder compressed into a tube) give different results with different granule sizes, so it is important to sieve test samples to within a specified range of granule sizes. For example if one wants make comparison tests with 3F commercially made powder, one needs to sieve the powder to be comparable in size with typical 3F powders.

Unfortunately, even sieving powders so that they are comparable with commercial sizes can introduce an appreciable margin of error. This is because even within commercial powder sizing specifications there is quite a large degree of size variation. Some powder sizes have a ratio between largest and smallest granules of 2:1, others even larger. Different powders also tend to have different percentages of intermediate sizes between the largest and smallest granules. So to avoid yet another *apples versus oranges* scenario it is prudent to sieve the powder to an even narrower range to reduce the margin of error.

### **Firearms**

The best way to test Black Powder used in firearms is with an actual firearm. Other tests may yield inaccurate and misleading results.

## BLACK POWDER MANUFACTURE. TESTING & OPTIMIZING

Black Powder burns very differently in firearms than it does in fireworks applications such as fireworks mortars. This is because pressures developed in the barrel of a gun are very much higher than those found in the barrel of a mortar or the tube of a Roman candle. So while other tests may give an indication of how well (or how badly) a particular powder will perform in a firearm, nothing beats using the firearm itself.

### **Apples and Oranges Revisited**

Apples and oranges situations can occur in just about any testing method if proper discipline to ensure consistency in data is not maintained. Ignoring any significant variable such as temperature or granule size can result in one's apples being compared to oranges. But there are some contradictions in the laws relating to our apples and oranges. Such contradictions can be used to advantage, hence our previous discussion on apples and oranges is worth revisiting.

This section looks at ways and means of doing comparison tests that both avoid apples and oranges situations and make testing easier, and in some instances, cheaper.

A classic apples and oranges scenario is performing linear burn rate tests on different powders made by different persons using different methods. Such tests can give a false idea on how well a powder performs as a propellant. However, linear burn rate tests performed on different batches of powder made by the same method can give meaningful results. How? To understand how we need to separate reality from mythology.

A common myth surrounding Black Powder testing is that there are absolute standards of measurement. This myth has created an untold number of headaches, heartaches, and frustrations. It has also exacted a high cost in time, money, and human life. There are no absolute measurement standards when it comes to Black Powder's performance. All standards are relative.

Just to clear up any confusion, we are talking *performance* standards here. We are not discussing properties that can be measured according to absolute standards such as density, grain size, or chemical purity. All these can be measured against standards set by national and international bodies.

The ultimate way of measuring the performance of Black Powder in any given application is to test it in that particular application. Nothing can beat such a test, provided, of course, that the test is done according to proper scientific procedures. The next best type of testing is a test that closely simulates the application, e.g. a test mortar where the application uses the Black Powder shells fired by a mortar. Both these tests allow one to compare different powders made by different persons, using different methods. The test in an application will give the best comparison, while one can have reasonable confidence in the simulation method.

Now test the different powders used to propel shells in a test that does not simulate or duplicate the application, such as a linear burn rate test. One may find differences in performance that don't relate to the application or simulation tests. Why? The answer is, we have shifted the testing to a different set of criteria. We have moved from apples compared with apples to apples compared with oranges.

Now take just one of the powders tested in our shell propellant application and run a linear burn rate test on it. Record these results and repeat both tests with a different batch of powder made the same way. Compare the results, and one will have a high probability of finding a definite relationship between the linear burn rate tests and the application or simulation tests. Why? The answer is that application or simulation tests create individual baselines for the different powders tested.

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These baselines can be used to compare the relative propellant strengths of different powders. This is the most common use they are put to. But they can also be used to create other accurate methods of testing. One does this by running a flame spread rate test on the same powder that was used in the baseline test and recording these results. Now any powder made the same way with similar properties can be tested with a flame spread rate test and its results compared with the baseline flame spread rate test. If the latest flame spread rate is faster than the first, the powder should have relatively stronger propellant properties. Likewise if the flame spread rate is slower, the powder should have relatively slower propellant properties.

What have we done here? We have inferred propellant properties of a powder from its flame spread rate. But isn't this comparing apples with oranges? No it isn't because we are doing flame spread rate tests on the same type of powder. This can be done if we limit the flame spread rate comparisons to different batches of powder made the same way. But this method cannot directly be used to compare propellant properties of different powders made in different ways.

We have just challenged another myth in the above discussion. That myth relates to the value of indirect testing methods such as using flame spread tests to determine propellant properties. Indirect testing methods are indeed valuable if they are restricted to tests that compare apples with apples and oranges with oranges.

Want to know a secret? Such indirect testing is commonly used in laboratory and manufacturing situations. GOEX uses lead tube tests to determine the propellant properties of sporting powders. Significant scientific research has been done by using strand burn rate tests related to somewhat different applications. So before investing in a somewhat complex and expensive testing apparatus, seriously consider indirect testing. It may cost you pennies instead of tens or hundreds of dollars.

### **Acknowledgements**

I am indebted to Ken Kosanke from Pyro Labs for much of the material in this chapter including all the graphics. Some of this material has been drawn from articles published by Kosanke et al. Other material comes from correspondence I have had with Ken Kosanke in writing, by telephone, and in person. I also owe a vote of thanks to Don MacDonald from GOEX who described some of their test procedures to me.

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# BLACKPOWDERMANUFACTURE, TESTING & OPTIMIZING

## **Last of the Breed - A Visit to GOEX**

by John M. Drewes

A 2-inch article in the *Wall Street Journal* of September 12, 1972, was enough to send a shiver of apprehension up the spine of any fireworks guy. The headline was simple:

### **Du Pont Co. to Close Out Black Powder Operations**

The text said that the Belin Works plant in Moosic, Pennsylvania, would be closed after March, 1973, when their government contract for Black Powder expires, because that contract takes the full output of the plant. It said that Du Pont hoped to sell the plant.

There were a couple of things incorrect in that little article, and the one that bothered fireworks people most was the fact that the government military contract did NOT take the full plant output. In fact, Black Powder for fireworks and for fuse manufacturing consumed a substantial amount of that output. Well, what was the big deal? Couldn't fireworks manufacturers who used Black Powder simply switch to another supplier? Nope. The fact was then, and still is today, that the Belin Works is the sole remaining commercial manufacturer of Black Powder in all of North America. If production ceased at that plant, we would have to find our source overseas, and that meant unreliable and expensive delivery, ever-increasing prices, and spotty quality control. The fireworks trade faced a real dilemma.

The U.S. fireworks trade had a perpetual problem with Black Powder. There were always periodic droughts when it would be unavailable for various lengths of time. If you had the magazine capacity and enough money (rare in the fireworks trade), you would stock up. The problem is that the manufacture of Black Powder is a rather hazardous undertaking, and plants would suffer incidents from time to time. Each blow up would shut down the plant for anywhere from a few days to a few months, usually, it seemed, just when you most needed the product. Now the trade was faced with the plant closing forever, and reliance on European Black Powder was not a happy thought for struggling manufacturers.

The story does have a happy ending. The plant was purchased by the Gearhard Owens Company, and later spun off to the GOEX we know today. The 100+ year old Belin Works is still in operation, still offering the only source of U.S.-made Black Powder in North America.

There's an incredible story connected with the Belin Works plant. It's all tangled up with the American Dream, and immigrants finding success in the New World, and just the right new technology at just the right time. Now here it was 170 years later, and the du Pont empire that started with Black Powder at Brandywine Creek in 1802 was about to abandon its last remaining plant. Had Black Powder come to the end of the road?

Not by a long shot! The military still used a substantial quantity each year, and the fireworks trade - well, that could go on forever! There definitely was enough market for a reduced operation, and Gearhard Owens decided to address that need. So the forty-five workers remained, the plant stayed in operation, and the only thing that changed was the name.

Now it is 20+ years later and I find myself living about an hour east of this historic plant. Black Powder is still used by the military, and the fireworks trade still needs a substantial quantity each year. Wouldn't it be great, I ask myself, if I could tour the plant, interview some of the

## BLACK POWDER MANUFACTURE, TESTING & OPTIMIZING

people, and find out what it's like to make Black Powder today? I've talked to plant manager (and then GOEX vice president; today president) Frank Fahringer on the phone a few times, so another quick phone call got the invitation I needed.

It was just a few days later when The WiZ (Don Haarmann) and I were heading down 1-81, looking for the airport exit. That's right, the Black Powder plant that was built in the heart of the anthracite mining district when King Coal meant wealth and power, now found itself surrounded by civilization, with an international airport as a neighbor. And finding the exit didn't help all that much, because the road leading to the main gate turned out to be an unobtrusive side road leading behind a trucking company terminal. You'd never know that these fenced-in, heavily wooded acres hid the last Black Powder operation in the United States and Canada.

It's been said that fireworks people can sense magazines and fireworks operations, and although the gate was open and there were no signs visible, we *knew* this was it, so we drove up the road and into the compound.

The place seemed deserted. We saw no workers bustling around, no vehicles plying the roads, but we did see something that really looked out of place - it appeared to be a large cast iron bathtub, painted red, with a wooden top, sitting all by itself at the side of the road. Why is a bath tub here? We would soon learn the reason.

The road made a turn to the right and suddenly we were in front of the administration building. It was just as we expected, an older wooden building, painted white, and a few steps took us inside where we were greeted by a woman administrator. Before we had much of a chance to interview her, we were ushered into a large office, where we found Frank Fahringer sitting behind his desk.

Frank's background is engineering, and probably he is the best man for this job today. He immediately puts us at ease. The hard questions begin. Frank will tell us anything we want to know except how many pounds the plant produces yearly, and what percentage is military vs. civilian.

The company is very proud of its workforce. These experienced powder makers bring their sons into the company, and there are many two-generation families represented, with even a few three-generations. Are the workers fully aware of the danger? Yes. After an accidental explosion, do many workers quit. No. How do the workers feel about working around Black Powder. They take great pride in being part of this unique team.

It isn't long before Frank puts on his coat and we head out for a walking tour of the manufacturing facilities. The first thing we note is that everything is made of wood, painted white. And there are more of those curious bathtubs! There seems to be some kind of narrow-gauge railway running through the manufacturing stations, but the rails are wooden. Frank explains that product is moved between stations in wooden cars on those wooden rails, and gravity plays a big role in the process.

What is the overall view of that process? Frank explains that the steps to making Black Powder are simple: the raw materials are manipulated to get them to the manufacturing stage, then they are put into a big wheel mill where a 7 or 8 ton wheel squeezes them for hours, "incorporating" the potassium nitrate, sulfur and charcoal. Then the "wheel cake" is really squeezed hard in a hydraulic press to make "press cake", which is then broken up and screened for size. Finally, the material is tumbled and dried, and most of it is glazed at the same time with graphite. A final removal of dust brings it to the packing stage, and then removal to the magazine area. It's not a complicated process, but just a case of squeezing the chemicals hard and long enough.

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As one researcher said, the sulfur is intimately mixed into the pores or spaces in the charcoal, then the potassium nitrate is added, with water. Some of the nitrate goes into solution and during processing, minute crystals of nitrate surround the sulfur/charcoal matrix. Wow, what a mouthful!

First step in this fascinating process is the bulk storage buildings. The potassium nitrate and sulfur are delivered to the plant in railroad hopper cars which unload atop the buildings! The material is scooped into holes in the roof and falls down into massive piles. Anyone who has ever bought a one-pound container of potassium nitrate would appreciate seeing 80,000 lbs. Enormous doors front this building, and when Frank opened them, indeed, we were confronted with the largest pile of potassium nitrate you could imagine! Frank explained that the nitrate was further processed before incorporation, including screening, magnetic inspection for tramp metal, and milling.

The next stop was the sulfur building, and when Frank opened the doors we found a very large, pitch dark room. As we entered and our eyes became accustomed to the light level, we could discern another enormous

pile - sulfur. Now sulfur is noted for its static electric propensity, and I could swear that this incredible pile of sulfur had a blue glow spread over it. There was no doubt in my mind that in that very dim light I could actually see a charge of electricity in the sulfur. Frank wouldn't confirm or deny my observation.

Prior to incorporation, the charcoal is moved around the plant in ridiculously large canvas bags. We didn't get close enough to measure, but they seemed to be almost 6x6-feet. That's a lot of charcoal!

The real action begins at the wheel house, where those tremendous wheels grind away for hours, churning and kneading the ingredients into the Black Powder. But before reaching this point, the potassium nitrate was processed as mentioned above, and the sulfur and charcoal were mixed together and ball milled to make a really intimate mix. These processes are done in some of the original buildings: brick, concrete and field stone buildings erected during the peak of the Industrial Revolution.

Finally the ingredients arrive at the wheel house. You can see in the picture that this is an open arrangement, and the single worker does everything with wooden tools, from wooden wheelbarrows, to wooden shovels, to wooden rakes. This skilled powder maker *charges* the mill by using a wooden shovel to spread the ingredients evenly around the trough. After satisfying himself about the even distribution, he walks to the remote START switch and sets the wheels in motion. As the wheels slowly revolve, a plough-like device follows, turning the squeezed material back into the track. The mill is actually two wheels, and they turn at something like 10



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rpm. They don't actually contact the trough, and the secret of how much pressure is applied to the powder is determined by the thickness of the mix.

Originally the wheels were designed to be operated by water power, but today, of course, they are run by electric motors. They use an enormous amount of energy and the plant would be poorly managed if the wheels were allowed to operate longer than is necessary. The run could be anywhere from 3 to 8 hours, depending on the charge and type of powder being made.



## BLACK POWDER MANUFACTURE, TESTING & OPTIMIZING

Perhaps the biggest secret of all to making successful Black Powder is the skill of the wheel mill operator. He watches the milling process closely and his experience tells him when it is necessary to add more water, which is an extremely vital step.

Eventually the mill process is complete and the material is raked and shoveled out of the trough and carried to the press house. It is Black Powder to be sure, but it is very soft at this stage, and not yet a commercial product. In the press house the material is placed in a large hydraulic press, with a layer of material, then a metal separator plate, then more material, another plate, etc. until the press is full. Then the press is operated and allowed to squeeze the material. Frank wouldn't tell us the time and pressure, but some Black Powder references say 6,000 psi, with time varying to over two hours.

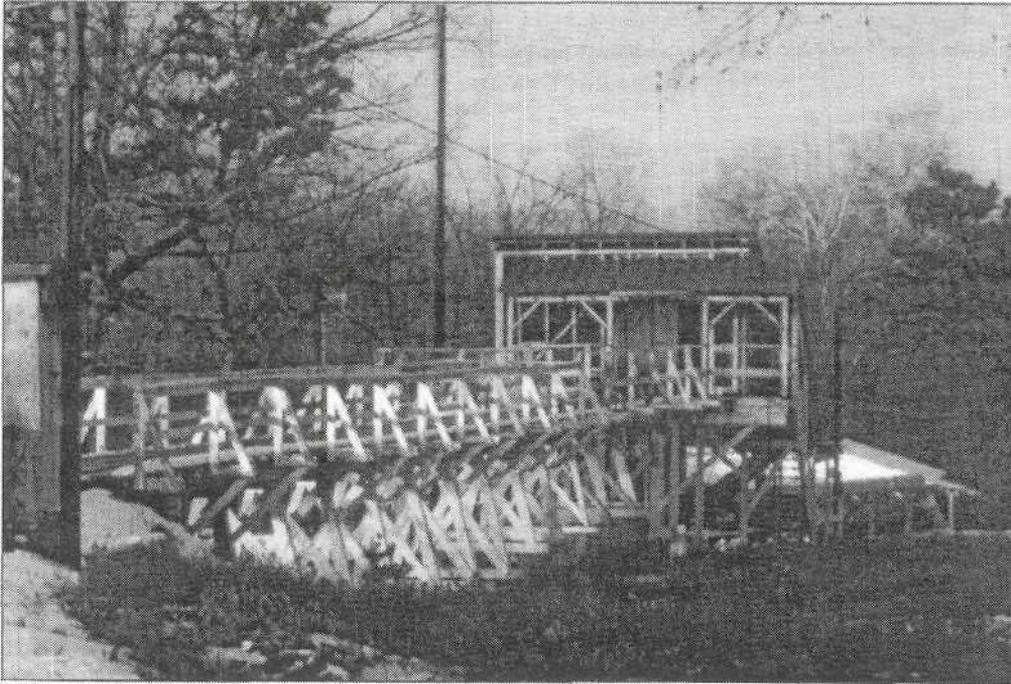


The *press cake* that comes out of the press is Black Powder, and there's no doubt about that, although we were told it is still sometimes referred to as *green powder*, not to be confused with the *green powder* that is sometimes used in fireworks.

The more dangerous aspects of Black Powder manufacturing occur now. The *press cake* is large chunks of very hard Black Powder, and it is useless in this condition. It comes out of the press in lots of about 4,000 lbs., and this material is carefully transported to the next step, the corning building, where everything is done remotely.

Here the Black Powder is conveyed to the top of elaborate machinery which breaks up the cakes and further crushes the material into usable sized pieces, all the while removing the dust and fines. (It is very important to remove the dust during the various processes, and all this material is returned to the wheel mill for processing into the next batch.) The corning process is considered to be the most hazardous in the operation, and great care is taken to minimize employee exposure to that danger. Eventually the powder has passed through the corning process, the dust has been removed, the cakes have been crushed to particles, and the particles have been screened to like sizes. Now another vital step takes place. The finished material from several batches is screen-blended in a unique device. This process tends to even out any anomalies in the various batches, and produces powder that is within specs.

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Now the powder must be dried and glazed, a process that can take up to 8 hours. A substantial charge is put in a large barrel and is tumbled. The original DuPont process required that the charge be heated with steam so as to hasten the drying, but the process today requires that merely the heat of tumbling is sufficient to dry the product to specifications. During this process the powder is given a glaze by adding a quantity of graphite powder.

The finished, glazed, sized Black Powder is now transported to the packing building where it is loaded into appropriate packaging. This too is a rather dangerous operation, usually performed by one, sometimes by two employees. No electricity enters the building, and interior illumination is provided by lights shining through the windows.

All the while Frank was explaining the process to us, we were walking around the plant, mostly following the railway. We could see workers busy at their various chores; they ignored us most of the time, but we noticed that when we entered a building, one or more of them would leave. Finally we couldn't stand it any longer so we asked Frank why they were doing that. He merely pointed to a sign next to the door. It seems that each building permits a certain maximum number of people in it when Black Powder is being processed, and by our entering a building, one or more workers were *required* to leave. Needless to say, we didn't spend much time in any building before Frank had us on our way to the next.

We asked Frank what happened to the material when it left the packing building. He pointed to a series of low structures some distance away. We noticed what appeared to be an awfully large number of telephone poles sticking up among them. Ah yes, this was the magazine area, and those poles were lightning protection. It was a very curious arrangement indeed, but who wants to work around a large quantity of explosives magazines without lightning protection?!

Several features of our visit stuck in our minds. First, the plant occupies a large piece of ground, is entirely fenced in, and is heavily wooded. Most of the buildings are of original construction, but everything seems to be maintained at a high level of cleanliness and utility. The most prominent feature of the plant is the white-painted wooden railway that workers use to trundle carloads of material through the processes.

## BLACK POWDER MANUFACTURE, TESTING & OPTIMIZING

While originally built in a very rural area, it is today completely surrounded by built-up areas, but the locals don't seem to mind. In fact, much of the local population has worked in the plant, or has kin who have or are working there, and they are proud of their tradition of making Black Powder.

The visit was a wonderful way to spend an afternoon. Frank Fahringer was friendly and knowledgeable, and he made the tour really memorable. We did note that of the six or so wheel mills, only one was being used, seeming to indicate that the plant is working at less than 20% of capacity. Yet that 20% is enough to provide all the Black Powder that the military, the fuse makers and the fireworks makers can use. It's sobering to realize that this historic plant is the last remaining representative of the days when Black Powder use was at its peak, and when every city had its own Black Powder manufacturing plant.

Oh yes, those bathtubs that were scattered all over the plant? They were indeed filled with water and were for workers to jump into in case of an accident. It's always handy to have a pool of water to jump into when you make Black Powder!



*All GOEX plant visit photos by Donald J. Haarmann*

They say all good things must eventually come to an end, and so it was with the GOEX plant in Moosic, Pa. Rail delivery of raw materials was vital to the operation; in the late 1990s the railroad line serving the plant was discontinued. The plant had to close. M. J. (Mick) Fahringer, son of Frank Fahringer and now president of GOEX, scoured the country for a new site. He discovered a military plant in Louisiana that was closing. It seemed perfect for Black Powder. Thus the sole remaining Black Powder producer in the U.S. now resides in Doyline, La.

[www.goexpowder.com](http://www.goexpowder.com)

# BLACK POWDER MANUFACTURE, TESTING & OPTIMIZING

## *Appendix 2*

### COMMERCIAL SPECIFICATIONS\* FOR FIREWORKS POWDERS

#### GOEX

NAME	GRAIN SIZES			
	Through		On	
	Sieve	Opening (inches)	Sieve	Opening (inches)
FA	20/64	0.3125	No.5	0.157
2FA	No.4	0.187	No.12	0.0661
3FA	No.10	0.0787	No.16	0.0469
4FA	No.12	0.0661	No.20	0.0331
5FA	No.20	0.0331	No.50	0.0117
6FA	No.30	0.0232	No.50	0.0117
7FA	No.40	0.0165	No.100	0.0059
Meal D	No.40	0.0165	-	-
Fine Meal	No.100	0.0059	-	-
Extra Fine Meal	No.140	0.0041	-	-

#### ELEPHANT BRAND

NAME	SIZE RANGE	
	Largest	Smallest
1 Fa(sic)	8.0mm	4.0 mm
2Fa	4.76	1.68
4Fa	1.68	0.84
5Fa	0.84	0.297
7Fa	0.42	0.149
Meal-D	0.42	-
Fine Meal	0.149	-
Extra Fine Meal	0.105	-

\* From company supplied data.

# BLACK POWDER MANUFACTURE, TESTING & OPTIMIZING

## Appendix 3

<b>RECOMMENDED LIFT CHARGE FOR SHELLS</b>		
<b>SIZE</b>	<b>TYPE</b>	<b>LIFT</b> (2FA Blasting or Cannon size)
<b>CYLINDRICAL SHELLS</b>		
3"	One-break	1 oz.
3"	Color & Report	1¼-1½ oz.
4"	Color	2 oz.
4"	Color & Report, Serpents. Whistle. Siatine, etc.	2½ oz.
5"	Color	3 oz.
5"	Color & Report, Color & Pupadella, Color Artillery, etc.	4-5 oz.
6"	Color	4 oz.
6"	Color & Report	~6 oz.
6"	Pillbox Shells, etc. with longer time (big shells)	6 oz.
<b>ROUND SHELLS</b>		
3"	See note below	¾ oz.
4"	"	1½ oz.
5"	"	2½ oz.
6"	"	3½ oz.
8"	"	6 oz.
<b>RECOMMENDED MORTAR LENGTH</b>		
3"	24"	
4"	24-30"	
5"	30"	
6"	30"	
NOTE: These will cover many situations. However, each shell must be judged by its particular circumstances.		

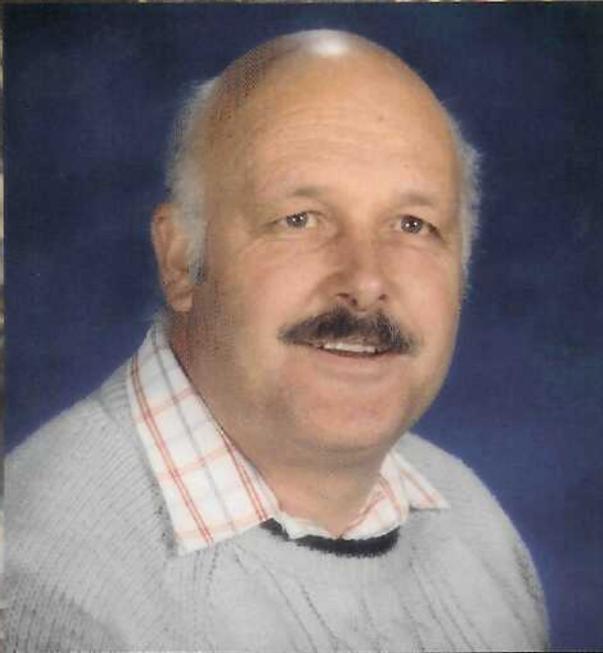
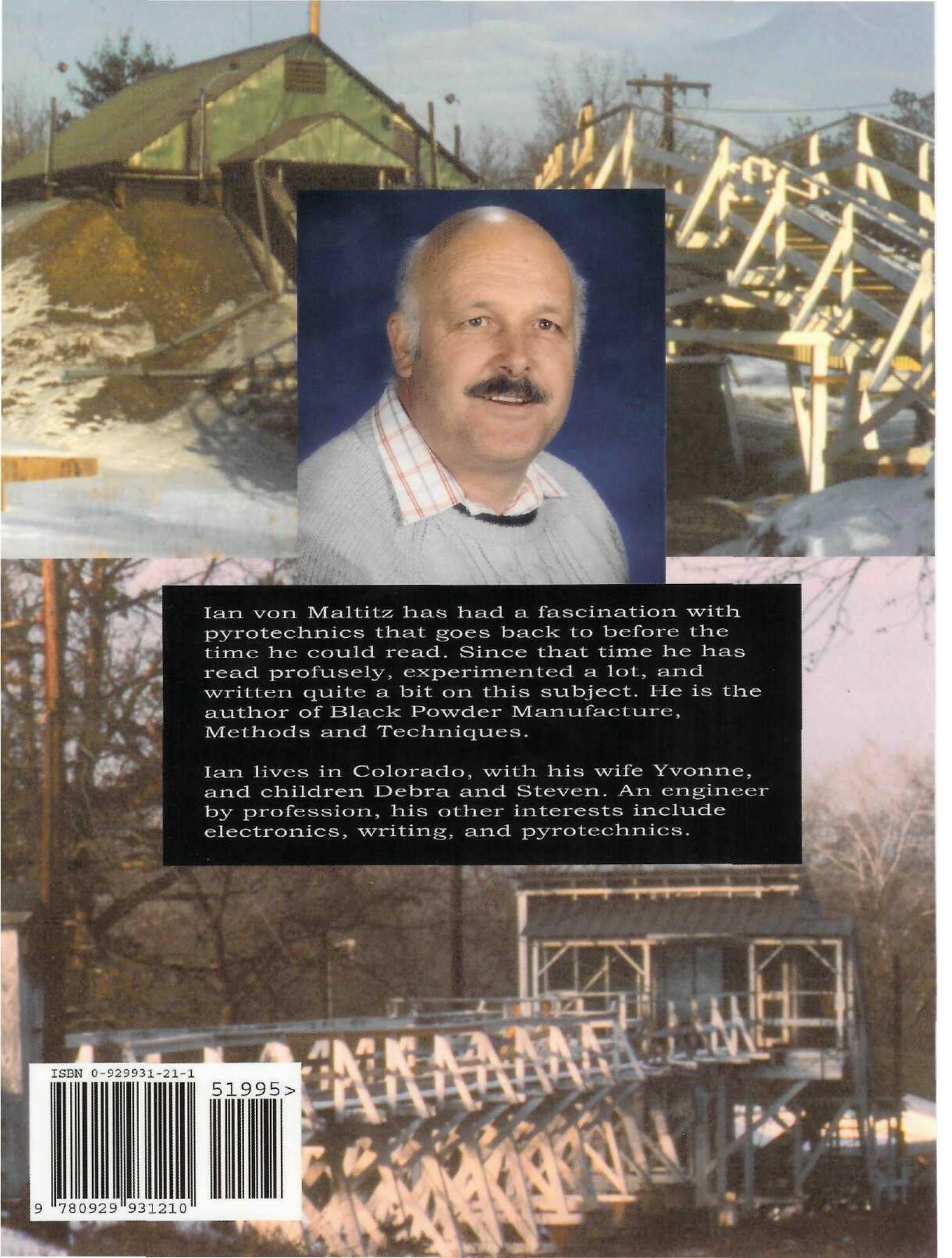
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Ian von Maltitz has had a fascination with pyrotechnics that goes back to before the time he could read. Since that time he has read profusely, experimented a lot, and written quite a bit on this subject. He is the author of *Black Powder Manufacture, Methods and Techniques*.

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