The WiZard's Great Book of Absoundite and Forbidden Pyrotechnic Knowledge.

I

Black Power
Potassium Nitrate
Charcoal

Black Powder Blasting

FORCE OF GUNPOWDER.

Although theory is not capable of estimating the force, or what is the same thing, the quantity of powder necessary to produce a certain effect, long experience has established the relative quantities of powder which are necessary under different circumstances, with sufficient accuracy for all practical purposes. These different circumstances are always of the same kind with reference to the artillery, where the range is the only thing that varies; whilst in blasting they are very various, loose earth has frequently to be blown up and at other times, solid rock; sometimes, the object is to destroy and hurl the fragments to a distance, and at others to get rid of them in a manner as little dangerous as possible, as for instance, in cases of civil engineering. For the latter purposes, the use of powder has very much increased since the introduction of railroads has given. occasion to so many excavations, and since the use of the galvanic battery in igniting the charge, has rendered the operation so free from all danger and removed all chance of failure from the modes, of blasting under water. A few examples will serve to illustrate the magnitude of such operations, and bear witness to the successful result of* the undertakings.

The line of railroad coming from Folkestone, after passing several viaducts, tunnels, and cuttings, traverses the Abbot's Rock Tunnel. To reach from thence Shakespeare's Cliff (near Dover) in a direct line, the projecting rock at Round Down, an immense mass of chalk, which exactly intercepted the line, had to be removed. The project for removing this rock, which occupied the space of 2400 cubic fathoms, and weighed one million tons, by one single blast, was successfully carried out by Mr. Cubitt. For this purpose, a channel, 361 fathoms long, was made in the direction of the railroad, and perpendicular to this, three shorter side channels: At the end of each side channel, a perpendicular shaft was sunk to the powder chambers, each of which was 13.4 ft. long, 6.1 ft. in height, and 5.5 ft. broad. In the chamber towards the cast, 5510 lbs. of powder (50 barrels) were placed, in the, middle chamber 7714 lbs. (70 barrels), and in the west chamber 6612 lbs. (60 barrels), together, therefore, in the three chambers, 19,836 lbs. of powder. The thickness of the mass of rock from the middle chamber was 85.4 ft., from the two others 67 ft. At the back of the rock, in a perfectly secure situation, a very powerful galvanic battery was placed under a shed, the covered copper wires of which, extending 1219 ft. over the top of the rock to the chambers below, and always resting on the ground, terminated in very fine platina points in the middle of the mass of powder. By making connection with the battery, these points were brought to a red heat, and the enormous charge of powder ignited in the same moment. When all was arranged, care was taken to stop up the entrances to the chambers with dry sand. Besides the charge of powder, a considerable quantity of air was enclosed in the chambers. It would have been quite contrary to the desired result to have actually blasted the rock into the air, or hurled the fragments about with a great loss of powder; the only object was to separate the mass of rock, and allow it to roll into the sea. The accuracy with which the necessary quantity of powder had been estimated, was proved by the wonderful success of the experiment. After firing the powder, neither smoke was evolved, nor report heard; no other noise than that occasioned by the tearing asunder of such an immense mass of chalk was audible to announce the result. The blasted portion of the rock, 500 ft. in breadth, began to sink, and slide gradually into the sea, which was distant 36 fathoms. In four or five minutes all was over. That which was here effected by the force of powder in an instant, would otherwise have taken six months labour, and have cost £7000 sterling. The circumstances attending the explosion, the absence of smoke and report, prove that the charge was just sufficient to overcome the resistance. The gases evolved had sufficient power to sever the mass of rock, without being able to force a passage for themselves at the moment. The game occurred, therefore, here, as in Rumford's experiments, in which neither smoke nor report were perceptible. In both cases, namely, the sulphuret of potassium, and the non-permanent gases, had time to condense, and the other gases to cool, before the walls of the chambers gave way. In most cases, for instance, in shooting with guns or cannon, the sulphuret of potassium is condensed by the cold air, and forms the smoke; the forcible expulsion of the other gases occasions the report by the vibrations of the air. A second combustion also ensues when the hot sulphuret of potassium, and the combustible gases come into contact with the air (p. 382) ; sulphate of potash, carbonic acid, and water, are formed with the flame which is always seen at the mouth of the barrel.

The blasting of the Royal George, a ship of the line, is not less interesting. This vessel was sunk whilst repairing, About sixty years ago, in the harbour of Spithead, through the obstinate ignorance of a lieutenant, in water of 90 fathoms, and as a wreck, rendered the otherwise excellent anchorage unsafe. After some smaller experiments, at first with 198 lbs., And afterwards four successive times with 49 1/2 lbs. of powder, bad been partially crowned with success, Colonel Pasley caused, on the 22nd of September, 1839, a cylinder containing 2552 lbs. of powder, to he fixed to the firmest part of the wreck by the divers. From the well-protected cylinder the conducting wires, covered with a mixture of pitch and tallow, ascended to the surface and from thence to the galvanic battery, situated in a boat at a distance of 500 ft. The protection against the water is so complete, that a charge may be ignited in this manner after having lain under water for ten days. Explosions under water are never accompanied by a report, for reasons already, mentioned; smoke can still less be produced. This was also the case here: three or four seconds after firing, the water was seen to rise in the form of a bee-hive to the height of 30 ft. it then spread itself out in the form of a sheaf, and lastly, sunk together in numerous muddy rings of waves. On the ships in the neighbourhood a shock was felt, as if from an earthquake. The wreck was, in great part, shivered to pieces. The remaining portion was, afterwards, removed in the same manner, May 12th, 1840, by the same engineer. On this occasion, the cylinder with 2328lbs. of powder, was attached to the keel. The result was similar, but the sheaf of water only rose to half the height, although the shock communicated to the water was greater. When the water had settled down, dead fish and fragments of the wreck were seen floating on the surface; even butter and tallow candles, -from the stores of the wreck, were taken up.

Applied To The Arts And To Manufactures.
By Dr. F. Knapp, Professor At The University Of Giessen.
Edited With Numerous Notes And Additions:
By Dr. Edmund Ronalds, Lecturer On Chemistry
At The Middlesex Hospital,
And
Dr. Thomas Richardson, Of Newcastle-On-Tyne.
Vol. 1. London: Hippolyte Bailliere, Publisher, 1848.

I am indebted to the late Louis Woody for supplying me with a photo copy of this.

Brown or cocoa gunpowder.

The introduction of this powder was a great innovation in powder making, as it greatly improved the shooting of big guns and allowed the use of guns of still larger calibre than even the black prismatic powder. It was composed of 79 p.c. nitre, 3 p.c. sulphur, and 18 p.c. charcoal per 100 of dry powder, and contained ordinarily about 2 p.c. moisture. The 'charcoal ' also was a very lightly baked material, the percentage of carbon contained in it being but little higher than that in the (dry) wood or straw from which it was made.

This powder was used for heavy breech-loading guns in the form of hexagonal prisms, having the same dimensions as the black prismatic powder. The rate of ignition and of combustion of the brown prismatic powder was slower than that of the black, and for equal muzzle velocities of the projectile it produced less pressure in the powder-chamber of the gun than black powder, and gave a thinner smoke than the latter.

Brown or cocoa powder gives on explosion a greater quantity of heat and a smaller volume of permanent gases than does an equal weight of black gunpowder; but the larger amount of water vapour in the products of explosion of brown powder have an important influence in lowering temperature. The products of explosion of brown powder undergo considerable dissociation at first, thus lowering the initial pressure in the gun, and subsequent recombination, and so giving sustained pressures as the projectile moves along the bore. The more gradual development of the pressure and the reduction of the maximum pressure increased the life of the gun and rendered the use of lighter guns possible.

Sir Edward Thorpe
A Dictionary of Applied Chemistry
longmans, Green, and Co.
London 1916
Vol. II pg. 410

Brown Powder

"To meet the requirements of the longer and more accurate guns the grains of the [black] powder are gradually increased in size so as to make them burn more slowly. In 1871 Pebble or P powder was made by cutting cubes from pressed slabs and in 1881 Prism powder was made by molding hexagonal prisms and pressing them in a special press. The Germans in 1882 made a brown prism powder, and in spite of attempts to keep the method of manufacture secret, it was being made at Waltham Abbey also two years later. This very large dense powder was required on account of the great increase in the size of naval guns. In 1882 at the bombardment of Alexandria we [the British] had 80-ton guns of 16-bore, and in 1886 110-ton guns of 16 ½ -inch calibre. This powder did not retain its importance long, however, for in the nineties smokeless powder entirely displaced black powder as a propulsive explosive in cannon. With smokeless powder it is now possible to throw a shell weighing a ton a distance of twenty miles.

The charcoal for brown powder or "cocoa" powder was made from rye-straw which was only carbonized very slightly. It was heated only about half an hour, then taken out of the furnace. The carbonization proceeded spontaneously a little further and then the charcoal cooled. The result was a soft charcoal containing a large percentage of oxygen and hydrogen. In the operation of pressing the powder this became a coherent colloid which bound the other constituents together to a dense impervious mass, which burnt comparatively slowly. The cocoa powder gave the best ballistics in heavy ordnance of any "“black" powder ever produced, but it has now been entirely displaced by smokeless powders.

Both black and brown powders have been moulded into prisms which are usually 25 mm high and 40 mm wide, measured across the corners of the hexagons. The value of the brown straw charcoal is that under high pressure it flows and holds the mixture together, making it into an impervious mass, which can only burn at the surface, whereas black powders have slight pores through which the flame can penetrate. This may be seen by examining the powders under the microscope."

Arthur Marshall
Explosives Volume I History and Manufacture
P Blackiston's and Son 1917

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"At one time prismatic powder with a very high density, burning approximately parallel layers, acquired an importance, but now it is only of historical interest.

It was invented in the United States by [T.J.] Rodman and improved during the period 1868-1882. It had the form of a hexagonal prism with a central channel and was manufactured in the usual way with the distinction that the grains from the corning mill were finally compressed into prisms by the Vyshnegradskii hydraulic press. It was used for long-range gun fire. Since the powder for this purpose must burn as slowly as possible the following measures were taken to reduce the rate of burning:

(1) Brown charcoal was used.
(2)
(3)
(4)

Tadeusz Urbanski
Chemistry and Technology of Explosives
Vol. III (?)
Pergamon Press 1964

Cleaning the barrel of a 16 ½-inch black powder gun! Not me!!

Black Powder — *The final torture*

The ultimate torment, slow death by burning at the stake, was practised in England and Scotland for several centuries, not only for heresy or witchcraft, but for the 'petty treason' of murdering a husband. Sometimes a degree of mercy was allowed: the victim was strangled as the flames took hold. ....

Other victims were allowed to have small bags of gunpowder hung round their necks and waists, but even this was not always effective. Henry Moore's *Complete Protestant Martyrology* (1809) gives a horribly detailed account of the death of Dr John Hooper, Lord Bishop of Gloucester, who was burned for heresy during the reign of the Catholic Mary I in 1555:

Being now in his shirt, he trussed it between his legs, where he had a pound of gunpowder in a bladder, and under each arm the same quantity. He now went up to the stake, where three iron hoops were brought ... The iron hoop was then put round his waist, which being made too short, he shrank and put in his belly with his hand; but when they offered to bind his neck and legs he refused them, saying, 'I am well assured I shall not trouble you' ... Then the reeds were thrown up, and he received two bundles of them in his own hands, and put one under each arm.

Command was now given that the fire should be kindled; but, owing to the number of green faggots, it was some time before the flames set fire to the reeds. The wind being adverse, and the morning very cold, the flames blew from him, so that he was scarcely touched by the fire. Another fire was soon kindled of a more vehement nature: it was now the bladders of gunpowder exploded, but they proved of no service to the suffering prelate. He now prayed with a loud voice, 'Lord Jesus, have mercy upon me; Lord Jesus, have mercy upon me; Lord Jesus, receive my spirit': and these were the last words he was heard to utter.

But even when his face was completely black with the flames, and his tongue swelled so that he could not speak, yet his lips went till they were shrunk to the gums; and he knocked his breast with his hands until one of his arms fell off, and then continued knocking with the other while the fat, water, and blood dripped out at his finger ends. At length, by renewing of the fire, his strength was gone, and his hand fastened in the iron which was put round him. Soon after, the whole lower part of his body being consumed, he fell over the iron that bound him, into the fire ... This holy martyr was more than *three quarters of an hour* consuming, the inexpressible anguish of which he endured ... moving neither forwards, backwards, nor to any side: his nether parts were consumed, and his bowels fell out some time before he expired.

Brian Innes
The History of Torture
St. Martins Press 1998

Chemical Theory of Black Powder

Journal of the Society of Chemical Industry.
November 30, 1891
Volume X Page 947 & ff.

XXIL-EXPLOSIVES, MATCHES, Etc.

Chemical Theory of Gunpowder. H. Debus.
Annalen, 1891, **265**,257-315.

THE author discusses briefly the earlier views which were entertained in regard to the products of the explosive decomposition of gunpowder, and refers to a former paper of his (Annalen, 212, 295-298) for a fuller statement of his views on this part of the subject. He notes how Noble and Abel confirmed the conclusions of Karolyi that the quality of the products of decomposition when the powder is fired in a closed vessel is only dependent on the composition of the powder and not on the pressure developed. The quantities of the products, however, as found in different experiments and by, different observers, vary greatly, but the author points out several circumstances which would account for these discrepancies, and states that both the quality and quantity of the products of decomposition depend solely on the composition of the powder. As the composition of powders is very varied, so must also be that of the products of their decomposition, and the author proceeds to develop a general equation which can be adapted to each special case, and by means of which the quantities of the different products of decomposition, the amount of heat and gas produced, and also the relative energy can be calculated, and shows that theory and facts agree, and that it is possible to determine by purely theoretical means the composition of a powder which is required to fulfil specific conditions . He assumes that the combustion is complete, and that the powder is composed only of saltpetre, sulphur, and pure carbon, the other constituents of the carbon being neglected. He also takes the potassium sulphide formed to be K_2S_2. The quantity of e carbonic oxide formed most be determined by experiment , and this he represents by *a,* then the general equation for the decomposition is " follows :—

$$
\begin{aligned}
&x\,(KN03) + y\,C + z\,S \\
&= 1/28\,[4\,x + 8\,y - 16\,z — 4\,a]\,K_2CO_3 \\
&+ 1/28\,[20\,z - 16\,y + 4\,z + 8\,a]\,K_2SO_4 \\
&+ 1/28\,[—10\,x + 8\,y + 12\,z — 4\,a]\,K_2S_2 \\
&+ 1/28\,[— 4\,x + 20\,y, + 16, z — 24\,a]\,CO_2 \\
&+ \quad a\,CO \\
&+ \quad 1/2\,x\,N2
\end{aligned}
$$

The author gives an example of the calculation of the relative potential energy of a powder by means of this formula, and adopts Berthelot's suggestion to take the product of the volume of gas evolved and the umber of calories given off as a measure of the potential energy and then deduces the general principle ; *that in every kind of powder the quantity of gas increases and the quantity of heat decreases with the carbon and sulphur*. Next follows a comparison of theory with the results obtained by Noble and Abel, and Roux: and Sarrau (Jahresbericht, 1873, 1029—1030), when it is shown that al the latter experimenters found in all cases more gas calories than the former, the order in which the relative potential energy of the powders follow is the same according to both observers, and agrees, with theory. The author then goes on to describe bio representation of the quantities of saltpetre, sulphur, and, carbon, which are converted into potassium carbonate, sulphate, disulphate, carbonic acid, and nitrogen ; but the original article as well as another (Annalen, **213**, 40), must be cousulted [sic] for explanations of the authors views and method of calculation. He shows that " when the amount of saltpetre and sulphur is constant the energy increases with the carbon, and when the saltpetre and carbon are constant it, decreases as the quantity of sulphur increases." As an evidence of the insufficiency of the purely empirical methods which have been hitherto employed in determining the composition of gunpowders, he points to the great variations in the proportions which have been used from time to time in different countries and in different works. In Prussia military powder had the following composition in the undermentioned years :—

Year	Saltpetere	Charcoal	Sulphur
1774	74.4	13.3	12.3
1800	75.0	15.0	10.0
1843	75.0	13.5	11.5
1874	74.0	16.0	10.0

The composition of the powder of the principal countries of the world may be referred to the three following types :—

----------	I.	II.	III.
Saltpetere	74.5	75.0	75.5
Charcoal	15.5	12.5	14.5
Sulphur	10.0	12.5	10.0

The author states that in the same manner as the sulphur on a match carries the combustion to the wood, so in gunpowder the burning sulphur bents the saltpetre and charcoal to the temperature of reaction. A mixture of saitpetre and charcoal is less easily ignited than one containing sulphur in addition, and he concludes also that a certain proportion of sulphur is necessary. in reference to cocoa powder it is shown that theoretically it is the strongest powder, and this agrees with experience.—W. M.

TNT Equivalency of Black Powder

Extracted from:
Hazards from Salute/Flash/Star Compositions A brief literature survey. By donald j haarmann aka The WiZ

Scanned in from: The PGII Bulletin No. 65. May 1989.

Parts that gagged the scanner and a few others have been deleted.

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TNT Equivalencies of Black Powder. Volume 1: Management Summary and Technical Discussion, H.S. Napadensk and J.J. Swatosh Jr., lTIRJ6265-3, Sept. 1972, NTIS ADA-044444. 69 pages + vii.

"Black powder charges ranging in weight from 8 to 150 pounds were evaluated under different levels of confinement. The TNT equivalence for the final product were found to range between zero to 43% for impulse and zero to 24% for pressure, depending upon the level of confinement, the weight of explosive and booster, and the distance form the explosion."

The generally quoted figure for the detonation velocity of BP is 400 meters/sec. However A.F. Belyaev and RKh. Kurbangalina; Russ. J. Phys.Chem. 38:309-310,1964, as quoted in the LLNL Explosives Handbook, URCL-52997, provide the following figures Density g/cm3 appx. 0.7, det velocity appx. 1.35 km/sec.

digesteth, fermenteth, and ripeneth

The old method of obtaining saltpetre was to collect vegetable and animal refuse containing nitrogen, the sweepings of slaughter- houses, weeds, etc., into heaps and to mix this with limestone, old mortar, earth and ashes. These heaps were sheltered from the rain, and kept moist from time to time with runnings from stables and other urine.

As late as in the reign of James I (1624), we find in an indenture between the King and Thomas Warricke, Peter Sparke, Michael Townshend and John Fells, the statement that " for making of the saltpetre which hath been formerly and now is made it has been found a matter of mere necessity to dig houses, cellars, vaults, stables, dovehouses and such like places, wherewith divers of his Majesty's subjects have found themselves grieved. " We are also informed that the conveyance of the liquors, vessels, tubs, ashes, etc, from place to place in carts had been a frequent source of nuisance and litigation.

The above persons purporting to have invented a new process for making saltpetre undertake to make it " as good and perfect as any hath formerly been, and shall be vented at cheaper and easier rates than formerly his Majesty or his loving subjects have paid for-the same, which said saltpetre as His Majesty is informed is to be or may be made of an artificial mixture or composition of chalk, all sorts of limestone and lime, marl, divers minerals, and other nitrous mines and other kind of ordinary earth, street dirt, or rubbish, stable dung, emptying of vaults, the excrements of all living creatures, their bodies putrified, all vegetables putrified or rotted, or the ashes, of them, and these or any of these mixed together in proportion as they may be most conveniently had, and shall be found most useful in such places where the said works shall be thought fit to be erected, which said artificial mixture or composition of any or all the foresaid ingredients is often times moistened with urine of men and beasts, petre, or nitrous wells, and springs, and all other concrete juices and blood of all sorts as can be gotten, and shall be fit and convenient for it, and divers times turned and removed, by which means the mixture in time digesteth, fermenteth, and ripeneth, from whence there is engendered the seed or mine of saltpetre which afterwards is to be extracted with common water, urine, the water of petre or nitrous wells, and springs, and then either breathed away in the sun or air, or stoved with gentle heat or boiled with a stronger fire with his proper additament of ashes, lime, and such like for separating the common salt and other mixtures naturally growing in the liquor and afterwards refined into perfect saltpetre. "

The King then granted the patentees licence to exercise their invention for a term of twenty-one years and to set up houses for preparing the artificial earth, etc.

On 26th December of the same year " was issued a proclamation, commanding that no dovehouses or cellars be paved, except that part of the cellars where the wine and beer is laid, in order that the growth of saltpetre might not be obstructed." (Patent Roll, 22 James I, part 4, No. 9 dorso.)

The Rise and Progress of the British Explosives Industry
Published under the auspices of the:—
VIIth International Congress of Applied Chemistry
E A Brayley Hodgetts editor
Whittaker and Co. London 1909

Black Powder — Ebonite

HYDRAULIC CAKE-PRESSES

Ebonite plates are decidedly preferable to those of copper, they are not so easily bent out of shape and always retain a plain smooth surface, and also because they have sufficient elasticity to transmit the pressure evenly all over the layer of powder, even should they get out of the horizontal. On the other hand , they have the disadvantage of becoming easily electrified; in fact, alternate layers of ebonite and powder really form an electric pile. It is quite possible that by excessive friction, and still more so in the case of a thunderstorm, the whole pile may become charged with electricity. A case came to the author's knowledge where a workman, just as he had finished charging the press and had opened the water-pressure valve, saw a thunderstorm approaching. According to his instructions, he left the building, leaving in the meantime the powder under pressure. After the thunderstorm had passed over he returned to the house, and was about to discharge the press when it exploded. Before his death the man stated that when he was about to empty the press a spark about four inches long passed from it to his finger. This points to the advisability of providing presses with an earth connection in order to prevent accumulation of electricity.

Oscar Guttmann
The Manufacture of Explosives
New York McMillan and Co. 1895

Lesmok Powder

Contrary to the general idea, black and Lesmok powders both are dangerous to make and to handle. The handloader should bear all this in mind and treat them with considerable care. In the days of black powder manufacture, explosions were very frequent, and occasionally serious damage and loss of life was incurred. An example of this was revealed by a former official of the Oriental Powder Company of South Windham, Maine. Although the factory was located in this small town, the executive offices were for many years located in the City of Portland, some fifteen miles away. Explosions occurred at regular intervals, and usually with sufficient ferocity to be beard and felt in Portland. After each explosion, there was a general rush for South Windham and a long line of applicants for jobs of cleaning up debris, rebuilding damaged buildings, *and* operating the rebuilt plants.

Lesmok powder is even more dangerous to make and handle than black powder, and despite the fact that it was formerly *the* propellant in match .22 rifle cartridges, it is now in complete discard. The makers of this powder and factories loading it would be glad to eliminate it entirely, and look forward with keen anticipation toward its obsolescence and discontinuance.

Philip B. Sharpe
Complete Guide to Handloading 3rd ed 2nd revision
Funk & Wagnalls 1953

The History of the first Establishment of Gunpowder Works in England.

The History of the first Establishment of Gunpowder Works in England.

Before quitting the subject of the establishment of powder works, I have deemed that it will be of considerable interest to give such information as I have been able to obtain on this subject through the kindness of Mr. Hart, of the Public Record Office, which will in a considerable degree afford information as to whence we derived our supplies both of gunpowder and cannon.

The exact period when this important article of warfare, gunpowder, was first made use of by our ancestors cannot now be determined; but from the testimony of various records, as shown by the Rev. Joseph Hunter, in a paper printed in the " Archæologia," vol. 23, it is evident that it was used at the battle of Cressy, for in an account rendered by John Cook, the clerk of the king's great wardrobe, of the monies received and expended by him from the 22nd Dec., 19 Edward III. (1349), it is stated that 912 pounds of saltpetre, and 886 pounds of quick sulphur, were supplied to the king for his guns.

On the 25th November, 1346, the king issued a writ, commanding that all the saltpetre and sulphur that was anywhere to be sold should be bought. The total amount obtained was 750 pounds of saltpetre and 310 pounds of quick sulphur.

In the time of Henry VI. an enterprising merchant of London, John Judde, who was skilled in devising warlike instruments, made at his own expense sixty guns, called serpentines, and also "stuff for *gunnepowdre of saltpietre and suphur,* to the weight of xx tonne," which lie offered to deliver to the treasurer for the king's use under certain conditions, in consideration of which good service the king by letters patent, dated 21st Dec., in the thirty-fifth year of his reign, constituted him Master-General of the Ordnance for life.

It was not, however, till the latter part of the reign of Elizabeth that public attention was drawn to the necessity of establishing at home the manufacture of gunpowder, which before had been chiefly supplied by importing from abroad. It had been up to that time an open trade; but the Government being compelled, by the menacing attitude which Spain assumed, to provide more efficient means of defence, commenced the granting of patents for the manufacture of gunpowder, which constituted it a monopoly in the hands of those whom the Government thought proper to trust with the privilege.

The first establishment of gunpowder mills of any importance appears to have been at Long Ditton, near Kingston, in Surrey, by George Evelyn, grandfather of the celebrated Sir John Evelyn. He had mills also at Leigh Place, near Godstone, in the same county. The Evelyn family is said to have brought the art over from Flanders. The mills at Faversham, in Kent, were in operation as far back as the time of Elizabeth ; but those of the Evelyns, at Godstone, were at this time of the greatest importance.

It appears, also, that on the 28th January, 1589, the thirty-first of queen Elizabeth, was granted to George Evelyn, Esq., Richard Hills, and John Evelyn, gentlemen, licence and authority for the term of eleven years to dig, open, and work for saltpetre within the realms of England and Ireland, and all other dominions where the same should be found, as well as within the queen's own lands and grounds and those of her subjects, except in the city of London and two miles distant from the walls of the same, and the counties of York, Northumberland, Westmoreland, Cumberland, and the Bishopric of Durham, and all the saltpetre so found was to be made into powder for the queen's service.

And on the 26th April, 31 Elizabeth, George Constable, Esq., had similar licence to dig for saltpetre within the counties of' York, Nottingham, Lancaster, Northumberland, Westmoreland, Cumberland, and the Bishopric of Durham, for the term of eleven years.

8th of January, 32 Elizabeth (1590), Thomas Robinson and Robert Robinson had a similar licence to dig for saltpetre within the cities of London and Westminster, and within two miles of the city of London, or from the old palace of Westminster, for the term of ten years.

By letters patent, dated 7th September, 41 Elizabeth, after reciting that John Evelyn, John Wrenham, gentlemen, Richard Hardinge, Esq., and Simeon Furner, gentleman, had undertaken to deliver yearly into the store of the Tower of London a greater quantity of good, perfect, and serviceable corn gunpowder, meet and serviceable for cannon and caliver shot, at a lower rate than was before paid, whereby the queen would not be driven to seek the said proportion of gunpowder out of any foreign countries, and that they had devised means of making saltpetre, whereby the excessive waste and spoil of woods and other inconveniences to the queen's subjects will be avoided, licence was granted them for the term of ten years to make and work for all and all manner of saltpetre and gunpowder within the realms of England and Ireland, and all other the queen's dominions, and to have the sole making of all manner of saltpetre and gunpowder within the realms of England and Ireland, except in the county of York, the city of York, the counties of Nottingham, Lancaster, Northumberland, Westmoreland, Cumberland, and the Bishopric of Durham ; and they had from the last day of April similar licences for those excepted places for the same term of ten years.

These parties were bound, it appears, to deliver during the term 100 lasts of powder [A "last" of gunpowder equals; 2 400 lbs i.e., 24 barrels /djh/] ; good, serviceable corn powder, eight lasts ; and eight hundred pounds weight every month, half of which was to be *cannon corn gunpowder,* and half to be caliver [rifle /djh/] corn powder, at the price of sevenpence per pound ; and they had permission to sell to the public.

Thus we have established on undisputed testimony that gunpowder of different sized grains, or corned—an art probably obtained from Flanders was generally used at this time ; and that before this date the greater quantity of gunpowder used in Great Britain had been imported from abroad.

It may be a wrong supposition, but with all this digging for saltpetre, to the great distress and worrying of the inhabitants of houses in the town and country, gardens, Orchards, &c., which led to much discontent, probably our great Shakespeare took the expression—Act 1, Henry IV. :—

"*And that it was great pity, so it was,*
That villanous saltpetre should be digg'd
Out of the bowels of this harmless earth."

Since the general practice in countries where it abounds is to obtain it by lixiviation of the upper soils.

It appears by letters patent, dated 24th January, 18 James I. (1621), that in consequence of the abuses and inconveniences which the inhabitants of this kingdom complained of as sustained from the servants of the above patentees, that the patent was revoked on the 17th December, and after reciting that there was in the kingdom a great quantity of the m*ine of saltpetre,* it stated that the King had once determined again to furnish the store of gunpowder by importation, but still as there were inconveniences in this mode of obtaining the necessary supplies of gunpowder, the King thought it expedient to continue the manufacture in the kingdom, and to establish certain vigilance and care to press all abuses complained of by his loving subjects.

The King then granted to George, Marquis of Buckingham, High Admiral of England; Lord George Carew, Master of the Ordnance ; and Sir Lionel Cranfield, Knight, Master of the Court of Wards and Liveries, licence to make and work, for saltpetre and gunpowder.

On the 16th of January, 20 James, a proclamation was issued which, after stating the great inconvenience of the sale of weak and defective gunpowder, ordered that no persons should make gunpowder in England and Wales, or any saltpetre, but by warrant of His Majesty's Commission, and that no saltpetre could be sold or bought but to and from the King's powder maker and all gunpowder was to be proved and allowed by the sworn proof-master, and marked by him, for which he was to have a fee of sixpence the barrel. The marks of the proof-master were three crowns for the best, two crowns if new and strong, but O W and one crown for old powder now worked, but good and strong, and fit for ordnance for one year's service at least.

By an indenture, dated 26th April, 2 Charles I. (1626), Made between the King on the one part and Sir John Brooke, Knight, and Thomas Russell, Esq., after reciting that there was never yet made, since the first making of saltpetre in the kingdom, being about the beginning of the reign of Queen Elizabeth, a third part of the saltpetre required for the service of the kingdom, but the King, as well as his subjects, were forced to procure the same from Barbary, France, Poland, Hamburgh, and other places in Germany; and that Brooke and Russell bad discovered a new mode of making saltpetre, whereby the King should have whatever quantity was required ; the King' granted them a licence to exercise this invention for twenty-one years, and they were to be paid £3 3s. 4d. for every hundred-weight of saltpetre delivered into the store in the Tower.

The East India Company by this time had begun importing great quantities of saltpetre, and had erected gunpowder mills in the county of Surrey, but being in an inconvenient situation they were pulled down by the King's direction. The East India Company then petitioned for leave to erect mills in the counties of Surrey, Kent, and Sussex, or any or either of them, and accordingly by letters patent, dated 17th August, 2 Charles 1. (1626), they were empowered to do so, and also to convert into powder all such saltpetre as should be imported by them from foreign parts, and to employ the same powder for their own use, or to the use of any of the King's subjects.

28th April, 5 Charles I. (1629), the King granted Richard Lord Weston, High Treasurer of England, and others, commissions to work for saltpetre ; and on the

18th April, 10 Charles I. (1634), a similar commission was granted to Richard Earl of' Portland and others.

No doubt the manufacture of gunpowder at this time was a very profitable investment of money, and we find by a commission dated 8th March, 12 Charles I. (1637), directed to the Bishop of London, and others, a contract was made with Samuel Cordwell and John Collins for the solo working and making into gunpowder all saltpetre made in England or imported.

A commission dated 26th April, Charles I. (1637), after reciting that grievances had arisen from the indiscriminate sale of gunpowder— Mountjoy, Earl of Newport, and others were ordered and authorised to make choice of and license persons who were desirous of buying and receiving gunpowder from any of the Royal magazines, and selling the same by retail.

7th June (1637), another commission was granted to the Bishop of London and others, giving the licence to dig for saltpetre, and to make gunpowder.

17th March, 16 Charles II (1663), was issued a proclamation prohibiting the exportation of saltpetre for three months.

June 5th, I8 Charles II. (1666), a commission was granted to John Lord Berkeley, Baron of Stratton, and Sir John Dunscombe, Knight, Thomas Chichely, Esq., commissioners for the execution of the office of Ordnance, William Legg, Lieutenant of the Ordnance, John Evelyn, of Deptford, E. Strong, Esq., Edward Sherborne, Esq., Clerk of the Ordnance, and Jonas Moore, Esq., to dig and work for saltpetre, and make the same into gunpowder for the King's service.

22nd July (1689), was issued another proclamation prohibiting the exportation of saltpetre.

> Letters patent, dated 29th October, 1692, were granted to Our trusty and well-beloved subjects—Richard Earl of Belmont, in our kingdom of Ireland ; Peregrine Bertie and Phillip Bertie, Esqs., sons of our trusty and right well-beloved cousin and councillor, Robert Earl of Lindsey, Sir John Huband, Bart., Sir Nicolas Pelham, and Sir John Bucknall, Knights ; William Gulston, William Tindal, Thomas Cox, Rupert Brown, Richard Dayrell, William Barnesby, John Hoskyns, Esqrs. ; John Seger Widenfelt, Charles Cox, Thomas Malyn, John Sherman, Patrick Gordon, Samuel Antrim, Jonathan Smith, gentlemen; Thomas Dawson, and James West, merchants; and all such others as shall hereafter be admitted and made free of the Company by the name of the Governor and Company, for making and refining of saltpetre within the kingdoms of England and Ireland, and to have continuance for ever."

They were to sell and deliver into the office of the Ordnance two hundred tons of the best white saltpetre, duly refined, within one year from the date of the patent, and every year afterwards such quantities, not exceeding one thousand tons in any one year, "is should be required by the Ordnance, at the price of £70 the ton, in case it bore that price in the market; or if not, then at the market price.

The were also to pay, yearly, during the continuance of their grant, to the Treasurer of the Navy, 1000l. towards the relief and maintenance Of maimed, aged, and decayed seamen, until a hospital should be built for them ; after the erection of which, the money would go towards the support of the hospital."

There is no record, Mr. Hart states, that lie has met with, of this remarkable charter of incorporation, in any works on the subject of gunpowder, nor is it known when the company was dissolved, or the charter surrendered.

There can be little doubt, however, that, as by the East India Company's Charter, the Company was bound to import a certain quantity of saltpetre annually, for the use of the Ordnance, probably quite sufficient for the Government purposes, that the supply from the Governor and Company was quite unnecessary, and that the discovery of William Tindal and Thomas Cox, Esqs., of a " new way of making saltpetre in great quantities," on which the company was formed, was of no commercial value, and thus the supply of Indian saltpetre led to the discontinuance of their project. [According to the charter of 1693, A. D., the East India Company was bound to furnish the Government with 500 tons of saltpetre annually, at from £38 to £45 per ton.]

I have thus, through the kindness of Mr. W. Hart, of the Public Record Office, been able to place before the readers of this volume sonic interesting facts which will establish the certainty that although the manufacture of gunpowder commenced in England in the time of Edward III. (1345), it was not until the reign of queen Elizabeth, when the improved art was imported from Flanders by the Evelyns, that it was fairly established also that until the reign of Charles II. the quantity required for the King's service, and of saltpetre also, was not sufficient, and that large supplies were imported from various foreign countries. It will also account for the supply to the East Indian armies after the East India Company had established their manufactories in England, in aid of the quantity furnished to the Bengal Government by the native manufacturers, until the time of Mr. John Farquhar, in 1794.—EDITOR.

[Col. Samuel Parlby, Retired Bengal Artillery — Editor]

Col. William Anderson, C.B., Late Agent At Ishapore.
Sketch Of The Mode Of Manufacturing Gunpowder At The Ishapore Mills In Bengal. With A Record Of The Experiments Carried On To Ascertain The Value Of Charge, Windage, Vent And Weight, Etc; In Mortars And Muskets; Also Reports Of The Various Proofs Of Powder.
London: John Weale, 59, High Holborn.
1862.

Horse Dung

601. II. Murtineddu's Powders consist of mixtures of nitrate of soda (with or without saltpeter), with sulphur and various substances as tan, coal, sawdust, &c.

The mixture, patented in England consists of: —

Saltpeter	100 parts.
Sulphur	100
Sawdust	50
Horse dung	50
Sea salt	10
Treacle	4

The object of adding the treacle is to give cohesion to the composition. It is claimed that "this composition does not cause explosion upwards as with gunpowder."
(D., p 608, and Spec. No. 2,403, 14.10.56.)

From:— JP Cundill *A Dictionary of Explosives* 2nd ed 1895

- - - - -

Horse dung Explosive.
W. Eberle
USP 910 365 (1909)

Potassium nitrate	12 parts
Sulphur	3
Charcoal	1
Pulverized horse dung	1

Fülöp & Lackovic Explosive (Hungarian):

Fresh horse dung	60%
Potassium nitrate	26
Sulphur	10
Dye	4

- - - - - -

Edhos of Escho. According to Molina, Explosvio Echos —

Ammonium nitrate	75%
Silicon	16
Aluminium powder	2
Dried horse dung ("Ipposino")	7

was used by the Italians for military purposes.

Praepositer (or Präposit). An explosive similar to black powder manufactured in the 1870's by the International Praeposite Co Powder Works, Millville, NJ, until the plant exploded. The composition was potassium nitrate, sulphur, charcoal, and "Hipposine", the latter ingredient being finely pulverized dried horse dung. The same explosive was manufactured in Germany by the Deutsch Präpositwerke G. m. b. H., Karlsruhe in Bavaria.

[Hippo is Greek for horse.]

From:— PATR 2700

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Test results showed that five of the systems tested (1, 3, 5, 8, and 9) exhibited high order reaction as indicated by their capability of fragmenting the test vehicles into large pieces.

No. 3

Potassium nitrate	70%
Sulphur	18
Charcoal	6
Animal dung (chicken)	6

B Jackson, Jr. & SM Kaye
Improvised Pyrotechnic Mixtures for Guerrilla Warfare Applications
Picatinny Arsenal 1964

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An Improved Blasting Powder
S. Fülöp and M.J. Lackovic, both of Buda-Pest.
English Patent 13,822, June 4th, 1897

This powder consists of a mixture in the proportions of horse dung, 28 parts ; saltpeter, 39 parts ; fine gunpowder, 23 parts ; sulphur, 10 parts.

In:— *The Journal of the Society of Chemical Industry*. May 31, 1896.
cf — above from PATR-2700.

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Blasting Powder
J. Tollner, Assignor to F.G. Dokken-andle, and H.M. Grant, New York
USP 757,693, April 19, 1904

Potassium nitrate	15%
Sodium nitrate	30

Sulphur	15
Spent tan bark	20
Horse manure	20

Horse Dung II

USP 757, 693
April 19, 1904
Jocob Tollner. of New York, N.Y.,
Assignor to F.G. Dokken Wadel and H.M. Grant
of New York, N.Y.

Be it known that I, Jacob Tollner, a citizen of Austria-Hungary, residing at New York, in the county of New York and State of New York, have invented certain new and useful Improvements in Blasting compounds, of which the following is a specification.

This invention relates to blasting compounds.

The object of the invention is to produce a blasting compound which shall be practically non-explosive except under pressure which shall not readily ignite, and which when burned in confinement produces large volumes of gas which are developed slowly and which act with great pressure to rupture masses of rock, coal, &c., when properly placed and ignited in a blast.

My invention consists in the compound which I shall now describe.

I take nitrate of potash, approximately fifteen per centum by weight, nitrate of soda, say, thirty per centum; sulfur; fifteen per centum; spent tanbark, twenty per centum. These ingredients are pulverized while in a dry state, I take of horse-manure, preferably fresh, (or if dry then moistened to a pasty consistency,) twenty per cent., and thoroughly incorporate, mix, or grind together all these ingredients. The moisture of the horse-manure produces with the other ingredients a pasty compound which will not explode under ordinary conditions while mixing, and the whole may be safely ground in a mill or mortar. When the compound is thoroughly mixed, it should be dried, when it may be broken into lumps or may be crumbled or pulverized. It is generally inadvisable to granulate the compound, as the granulation would ad to the expense without increasing the efficiency.

The compound may be stored in barrel or other receptacles. It is not very combustible and requires a strong steady fuse for its ignition.

The tanbark in the above compound is a woody substance which is thought to be more slowly combustible than charcoal as commonly used in gunpowder. The mixture of nitrates of soda and potash is believed to give a better result than would either nitrate separately. The admixture of the various ingredients without dissolution of soluble material, but in a slightly-moistened condition avoids danger in compounding. Horse-manure in its natural state generally contains a considerable volume of gas, as may be fond by chemical analysis, and when the ingredients are united as above described a blasting compound is produce which burns slowly. but with great and relatively long-continued pressure, so that by actual trial in quarries the effective work of this blasting powder is found to be much greater than that of dynamite as commonly used.

What I claim is —

1. The blasting compound described, consisting of nitrate of potash, nitrate of soda, sulfur, tanbark, and horse-manure combined in about the proportions specified.

2. A composition of matter consisting of nitrate of potash, approximately fifteen per centum, nitrate or soda, approximately thirty per cent., sulfur, approximately twenty per cent., and horse-manure, approximately twenty per cent., mixed and incorporated substantially as described.

In testimony whereof I affix my signature in response of two witnesses.

Jacob Tollner

a contrivance for the preservation of horses

Curtis's and Harvey, Limited.

Farversham works.

The output of the factory [ca. 1760] is stated to have been about eighty barrels of service powder per week. The mills were worked both by water and by horses, and Jacob in his " History of Faversham " gives an account of " a contrivance for the preservation of the horses that grind the powder " in the shape of a sort of suit of leather amour to protect them from the frequent explosions that occurred.

The Rise and Progress of the British Explosives Industry
Published under the auspices of the:—
VIIth International Congress of Applied Chemistry
E A Brayley Hodgetts editor
Whittaker and Co. London 1909

GUNPOWDER IN BRAZIL.

U.S. Cons. Reps., Dec. 1898, 602.
In:— Journal of the Society of Chemical Industry
No. 1.—XVIII January, 31, 1899

In reply to inquiries from a New York export association, Consul Furniss sends the following from Bahia, under date of August 24 :—

There is one factory in this consular district devoted to the manufacture of powder. This is situated in Cachocira, a town of about 5,000 inhabitants, some 20 miles distant from here, and reached by daily boats. The town is on the Paraguacu River, a few miles above the head of the bay on which Bahia City is situated.

The annual output amounts to about 4,000 kegs of 25 lb. each, and it retails at from 30 to 40 milreis per keg. As the milreis varies each day in value, a definite price cannot be given j but to-day a milreis is worth 14.2 cents in United States currency.

The greater part of the manufacture is common black sporting powder; a very little of a better grade is made. Much of the powder is used to manufacture fireworks.

The powder produced here, does not suit the requirements of the market, but, on account of the State restrictions, and more particularly the municipal restrictions of Bahia, and in view of the fact that this is the only port of entrance into this consular district, the people have to be content with that of local manufacture, and the Cachoeira powder is much used in the surrounding country.

There are no Government regulations prohibiting the importation of powder, but after it arrives in the harbour it is loaded on a Government boat and conveyed to the quay, whence it is removed to the Government magazine, about four miles distant ; all this at the expense of the importer. At this magazine the Government keeps a guard, and an importer making a sale is required to petition the chief of police of this city, who, at his discretion, gives licence for the removal of part or all of the quantity petitioned for. For this service the fee amounts to about 50 milreis (7.10 dols.), and the petitioner is compelled to tell the destination of the powder to be withdrawn. The licence for removal is presented to the Custom house, the duty paid, a permit received, and, upon presentation of this to the officer in charge of the magazine, the powder is delivered. The party purchasing from the importer must, sign the receipt attached to the permit issued by the police, swear to it before a notary, and return it to the police department, under penalty of a heavy fine for non-compliance.

Powder, according to present regulations, may stay in the Government magazine until wanted; a charge of I per cent. being exacted for storage, which amount is payable upon withdrawal. Ample storage is provided for any amount that may be imported.

In the city of Bahia, no powder is allowed to be kept, except in cartridges and shells for sportsmen's use ; and, on account of the restrictions, there is only one store where these are handled. There are several places where powder can be purchased clandestinely, at prices sufficiently high to re-imburse for risk run. This practice is indulged in by the smaller stores, where it is sold under the name “ farinha prata," or black flour.

Log Splitting

When logs are split up to be burned quickly, the same method is used as when splitting stumps : but if they are to be split for fence rails, cordwood, charcoal, or other purposes where comparatively even and regular sections are required, Du Pont Blasting Powder, in granulation FF, FFF or FFFF, should be used.

This explosive is so much slower in action than dynamite that a series of properly gauged and property placed charges will split a log along the grain just as evenly as if a number of wedges were used.

This method of splitting logs is so much quicker, cheaper and easier than any other, that those who have once become proficient at it never give it up. Augur holes one inch or more in diameter are bored along the line of the grain, about one-quarter to one-half the way through the log, the depth of the holes and the distance between them depending oil the kind of wood, the grain and the diameter of the log. A few ounces of FF Blasting Powder are put into the bottom of each hole, care being taken to see that the hole is dry, then wooden plugs are driven firmly into the tops of the holes to tamp or confine the charge.

In some kinds of wood it is best to leave a considerable air space between bottom of the plug and the powder. The plug must have a groove large enough to admit the electric squib wire or the fuse. As blasting powder is exploded by a spark or flame it is not necessary to use a detonator with it. Electric squibs are similar in appearance to electric blasting caps, except that they have a paper capsule instead of a copper cap. They do not explode when the electric current passes through them, but ignite the blasting powder by a flash. If electric squibs and a blasting machine are used for exploding the charges, they can all be fired simultaneously. This usually is the best and cheapest way as little less powder is required than when the charges are exploded separately with fuse. When using electric squibs, it is only necessary to have the grove or channel in the sides of the wooden pugs large enough for two small wires to run through it, if the cap of the electric squib is put in placed before the plug is driven in. When driving the plug care must be taken that the wires are kept free, and that the insulation on them is not damaged. If it is not covenient to provide wooden plugs in this work, damp clay tamping may be used on top of a wad of newspaper. A log two feet in diameter and four or five feet long, can usually be split in two with one two-ounce change of FF blasting powder. Longer logs require two or more holes and logs of greater diameter require heavier changes. The holes should be from one to two inches in diameter.

DuPont Framers Handbook: Instructions in the use of Dynamite for Clearing Land Planting and Cultivating Trees, Drainage, Ditching and Subsoiling 1913

Hazardville

*The History of American Manufacturers, pub*lished in Philadelphia in 1868, says: "At Hazardville, near Hartford, Conn., are the extensive gun powder mills of the famous Hazard Powder Company who have mills also in the towns of East Hartford and Canton. This company has 18 sets of rolling mills with 36 iron manufacturing wheels each weighing 8 tons, 7 granulating mills, 5 screw press buildings and 3 hydraulic presses Of 500 tons each. All are in different and separate buildings. In addition, about 5o buildings are used for dusting, assorting, drying, mixing, pulverizing, glazing and packing of powder. Extensive saltpeter refineries and magazines, cooper shops, iron, woodworking and machine plants are also maintained in all, about 125 buildings are located at their main works at Hazardville and Scitio extending over a mile in length and a half-mile in width.

"To propel this vast amount of machinery, 25 waterwheels and 3 steam engines are employed. ... This company manufactures annually over a million dollars' worth of powder of various kinds known as Government, Sporting, Shipping and Mining powder, of which large quantities were at one time exported to Europe."

Colonel Hazard died May 7, 1868, but his business continued for nearly a half-century longer. The census of 186o gives the value of the company's output as $991,500. The company was too powerful and turned out too high-grade a product to be overlooked by Du Pont. About 1876 the majority of stock control fell into the hands of Du Pont interests, and the concern passed out of the picture with the formation of the Du Pont Corporation in 1902.

Philip B. Sharpe
Complete Guide to Handloading 3rd ed 2nd revision
Funk & Wagnalls 1953

Chinese Discovery Black Powder

Some discoveries that may have been Sun Ssu-Mo's are embodied in short extracts quoted in other collections. For example, the *Chu Chia Shen Phin Tan Fa* (see pp. 159, 197) appears to quote him as follows:

Take of sulphur and saltpetre *(hsiao shih) 2* oz. each and grind them together, then put them in a silver-melting crucible or a refractory pot *(sha kuan)*. Dig a pit in the ground and put the vessel inside it so that its top is level with the ground, and cover it all round with earth. Take three perfect pods of the soap-bean tree, [Gleditschia sinensis] uneaten by insects, and char them so that they keep their shape, then put them into the pot (with the sulphur and saltpetre). After the flames have subsided close the mouth and place three catties (lb) of glowing charcoal (on the lid); when this has been about one third consumed remove all of it. The substance need not be cool before it is taken out-it has been 'subdued by fire' *(fu huo*) (i.e chemical changes have taken place giving a new and stable product).

Someone seems to have been engaged here about +650 in an operation designed, as it were, to produce potassium sulphate, and therefore not very exciting, but on the way he stumbled upon the first preparation of a deflagrating (and later explosive) mixture in the history of all civilisation. b Exciting must have been the word for that.

THE OLDEST DOCUMENT IN THE HISTORY OF GUNPOWDER.

Journal of the Society of Chemical Industry June 15, 1904
Meeting held at Burlington House, on Monday, May 2nd, 1904.
MR. WALTER F. REID IN THE Chair.

THE OLDEST DOCUMENT IN THE HISTORY OF GUNPOWDER.

By OSCAR GUTTMANN, M. INST., C.E., F.C.S.

The so-called ancient records concerning the invention of gunpowder should be approached with great caution, since manuscripts of doubtful date and origin which had been ,inadequately translated were used to serve various nations and individuals as proofs of their claim to this invention. Only such documents present a special interest, which furnish information about the time preceding, 1354, and as there is no doubt that even the Arabs did not shoot up to 1313, although they knew gunpowder-like mixtures since 1280, we are limited in our investigation to within a period of 40 years.

I have shown in another place, that the Arabian manuscript in the St. Petersburg Library, which was published by Reinaud and Favé, is of no importance, since, apart from other objections, its date is very doubtful. The oft cited passage in the Indian " Gentoo Laws " also become, valueless when correctly translated, and the description of rifles and of the composition and manufacture of gunpowder, as published by Prof. Gustav Oppert from the " Sukraniti," (On the Weapons, Army Organisation, and Political Maxims of the Ancient Hindus. Madras,1880.) is doubtless of more recent origin than he supposes. Oppert had before him only copies 200 years old of a lost original, and the learned Indian, Praphulla Chandra Ray, peremptorily denies (Praphulla Chandra Ray, History of Hindu Chemistry. London, 1903.) that the Indians knew gunpowder in the 14th century.

The following are the only existing unimpeachable documents :—

1. The accounts of King Edward III's private wardrobe from 1344 to 1347, and the accounts of the same King's great wardrobe from 1345 to 1349, in both of which there are entries of payments for gunpowder, and also sulphur and saltpetre for the King's guns.
2. The accounts of the town of Aix-la-Chapelle of 1346, showing some expenditure for an iron gun and saltpetre for same.
3. A document at the town library of Tournay, giving an account of experiments With a gun in 1346 by Pierre de Bruges.
4. The accounts, published in Muratori, Vol. 24, of Aimone di Challant, Sire di Fenis, Guardian or Lanzo in Northern Italy from 1347 to 1348, according to which Master Hugonino di Chatillon in 1347 made 4 bronze gun,; for the Marquess of Monferrato, in the Aosta valley, each of which weighed about 45 lbs., was fired by means of gunpowder, and threw leaden balls with large, iron-bound arrows.
5. The accounts dating 1342 for the artillery of Rihoult Castle, published in the "Mémoires de la Société des Antiquaries de la Morinie," tome 5. The guns threw

wooden arrows, bound with iron, and centred by means of copper discs. The price of the powder was about 30s. per pound.

6. A document in the Paris National Library, according to which 5 iron and 5 bronze cannon were bought in 1339 for the defence of the town of Cambray, for which Estienne Marel supplied saltpetre and sulphur, but in such small quantities only (for the mum of 11 livres 4 sous) that at the existing prices; of that time barely 3 pounds of powder would work out per cannon.

7. A document in the same library, according to which Guillaume du Moulin from Boulogne acknowledges the receipt on the 11th July 1338 from Thomas Fouques, guardian of the galley-house at Rouen, of " one iron pot for shooting, fire-arrows, 4S iron-bound and feathered arrows, one pound or saltpetre, and half-a-pound of live sulphur for making powder for shooting the said arrow. This would hardly give 20 grms. of powder to each arrow. Hitherto there was a general disinclination to regard these arrow-shooting guns favourably, but I am now in a position to give a picture of one.

In Prof. Oppert's above-mentioned book I found a footnote, which drew attention to a manuscript in Oxford dating from 1336, and I therefore endeavoured to get particulars and later on to inspect it myself. To my surprise the manuscript proved to bear the date 1326. It is written by Walter de Millemete, is entitled " *De Oflíciis Regum*," and is kept in Christchurch library. It is beautifully illuminated. I have only been able to obtain the right of reproduction for the new edition of my book on Explosives, the authorities of Christchurch jealously guarding any further publication. The manuscript begins as follows :—

" *Hic incipiunt rubrice capitulorlim huius libri de nobilitatibus sapienciis et prudenciis regum editi ad honorem illustris domiiii Edwardi dei gfacia Regis anglie incipientis reguare Anno dominiab incarnacione Milesimo Tricentesimo Vicesimo Sexto*."

Translation.—" Here begin the lists of the chapters of this book about the noble origin and the prudence of kings, edited in honour of the illustrious Lord Edward, by God's grape King of England, who began to reign in the year of our Lord, 1326. " (This is old reckoning; King Edward entered his reign in the year 1327 of the new.)

The contents of the book in no way refer to the history or the invention of gunpowder, but only deal with the duties and qualities of kings. It must have been begun in the reign of Edward II., because it contains pictures of him as king, and no doubt it was originally intended as a present to him. The very elaborate and rich illuminations must have taken t long time, probably more than a year, since such fine work could only be done in the summer months, so that it was very likely already begun in 1325.

There is on the last page of the manuscript a richly adorned frame surrounding the text, and on its lower part the reproduction of a bottle-shaped gun resting on a wooden trestle. The shape of the bottle resembles an antique urn (see sketch). It is closed by means of an arrow, which bag a ball on its lower extremity, and a warrior in full armour is in the act of firing this gun by means of an incandescent rod, intending to burst the lock of a castle-gate. This gives us on the one hand an, authentic and the oldest date for the use of gunpowder, and on the other hand, it is a most interesting illustration of the earliest guns, and the manner in which they were used. The gradual progress towards the use of the ball from the end of the arrow, only, as is known, in the way of ball-shaped common stones, is so far only conjecture.

I am indebted for the photograph of this interesting page to the kindness of the trustees of the Wake Trust, and for the information concerning it to the late Prof. York Powell and Prof. Webb, to whom I herewith tender my sincere thanks.

Now only we can believe a passage in John Barbour's life of Robert Bruce, King of Scotland, which was written in 1375, and has, hitherto been considered a fable. Barbour wrote of the year 1327 :—

"Twa noweltys that dai thai saw,
That forouth in Scotland had bene nade,
Tymmris for helmys war the tane,
That thaim thoucht than off grete bewte,
And alsua woudre for to se ;
The tothyr *crakys* war off wer,
That thai befor herd nevir er."

So much appears now certain, that the knowledge of the propelling force of gunpowder-like mixtures must have come about between 1313 and 1325. I am of opinion that Berthold Schwarz invented the guns, only the date of their invention must be put back much further than 1353, as written on his monument at Freiburg.

Carbonized Peas!

Another black powder which will be pleasantly recalled by old-timers was "American Rifle" and "Creedmore Rifle." This was developed by Chauncey J. Olds, plant superintendent of the Schaghticoke Powder Company, incorporated March 10, 1858, which operated a plant at Schaghticoke, New York, Olds designed a new powder and was granted a United States patent, #387507, April 17, 1888. His formula specified 75% saltpeter, 9% sulphur, 11.5% willow charcoal, and 4.5% charcoal manufactured from carbonized peas. This powder was popular with expert shooters, both in rifle and shotgun, and was manufactured and widely sold until 1903- Olds was paid $500 a year for his invention and his widow received that sum until her death in 1919..

Philip B. Sharpe
Complete Guide to handloading 3rd ed 2nd revision
Funk & Wagnalls 1953

Poudre Kusique

Prep. Nitre [potassium nitrate] and sulphur, of each 50 parts; powdered charcoal and antimony, of each 1 part; mix and divide into doses of 2 grammes, and put three doses in each packet. Given to dogs in a ball of butter, to prevent the disorders to which they are liable. [worms?] A popular French nostrum.

Arnold James Cooley
The Book of Useful Knowledge
A Cyclopedia of Six Thousand Practical Receipts, and Collateral Information..
D Appleton & Co. 1857

[I wonder. Which end of the dog do you put the fuse in? /djh/]

General Principles of Gunpowder

General Principles of Gunpowder.

The first object in the manufacture of gunpowder is, to obtain, in small space and weight, a material which produces, when excited by chemical action, a high propellent force, possessing an expansive power which shall be gradual, progressive and under good control.

Such is gunpowder. Thus the weight of 2 oz. will, in its expansion when fired, propel 1088 oz., the weight of an iron ball placed before it in a mortar, the distance of 100 yards. We are thus supplied with a power the artillerist requires, infinitely superior to the mechanical contrivances of ancient times.

The expansion of gunpowder, though amazingly rapid, is by no means instantaneous, as, if so, it would be totally unfit for the purposes we apply it to, for the following reasons:—we can only apply it to artillery projectiles or fire-arms generally, by using chambers of metal to confine its expanding power, except on the side of the shot or projectile placed before it, in which we allow it to expand. Now all metals consist of particles held together by what is termed the power of cohesion, and that this is only a limited power of resistance, differing in different metals, we know by experiment. Thus fulminating powders, laid upon an open plate of metal, though confined only by the atmosphere, reduce a perforation in the plate of metal from the momentary impulse of the force ; and when applied to cannon or shot placed before it in a confined chamber, shatter both probably, without producing any intended *projectile mo*tion.

Hence the advantages of gunpowder ; its expansion is progressive, and there is time given to overcome the inertia, of a weight of matter placed before it, and to impart this force to the projectile, without the evil consequences of fulminating powders.

Gunpowder has also the advantage of being easily transported, and, under proper precautions, with perfect safety ; and it is a singular circumstance, that not with standing the advance of science, and the wonderful chemical progress of moderns, there has been no substance yet produced that possesses all Its advantages, and that the three material, used in it's composition from the earliest times, viz., saltpetre, charcoal, and sulphur, have not been superseded by others. [Gun cotton, and other proposed substitutes for gunpowder, will be noticed in the Appendix. -EDITOR]

Chemical Principles of Gunpowder

Chemistry teaches us that there exist certain properties in matter which, when different atoms of various kinds are brought into contact, will, under the influence of beat, produce the wonderful phenomena called sudden decomposition and consequent explosion, changing their condition from solid particles to an expansive gaseous or aëriform state. We cannot explain *the nature of the power,* but we can, by experiment with it, produce effects, and from these calculate the power we can produce, with other results.

There can be little doubt that, in the first formation of gunpowder, when the science of chemistry was comparatively unknown, accidental circumstances led to these properties being discovered in the mixture of saltpetre, charcoal and sulphur ; probably, in the first instance, only saltpetre and charcoal were used.

But at the present day, from the science of chemistry, we find that in these ingredients the following properties exist, which render them so essential for the purpose of forming gunpowder.

In saltpetre, or nitrate of potash, we have a compound consisting of nitric acid and a base of potassium.

Chemists of the greatest celebrity have not given to their analysis the exactness of the proportions, but if we take the fair medium, we may consider saltpetre to consist of,

Nitric acid 54
Potassium 46 [Celebrated chemists differ in the proportions. See Appendix.]

The charcoal and the sulphur may be considered as simple substances if pure ; and though this is seldom the case, we must so consider them at present.

In the three components of gunpowder, we have, therefore, a compound and two simple combustibles.

The compound (the saltpetre) consists of nitric acid combined with a base of potash. The nitric acid consists of oxygen and nitrogen, six parts of oxygen to one of nitrogen ; when saltpetre is exposed to a red heat, or above 800°, it decomposes gradually if there is no combustible present, and a portion of the oxygen and all the nitrogen will pass away into the atmosphere, the other portion of the oxygen will unite with the potash, and form oxide of potassium ; but when we bring a combustible, as charcoal, into contact with the saltpetre at this heat, a violent and sudden decomposition takes place, and consequent explosion, from its striking the surrounding air so suddenly. The oxygen combines with the carbon, forming carbonic acid gas, and the nitrogen is set free. It is found from experiment that the volume of gaseous or aëriform fluid thus formed, occupies a space as a permanently elastic fluid, about 240 to 290 times that of the bulk of the gunpowder used, when cooled down to the state of the atmosphere ; but at the time of explosion the heat generated is so great, that the expansion of this volume of gas is increased from four to eight times in bulk, varying according to quantity and quality of the gunpowder and the circumstances of the explosion. Such is the cause of the amazing power of fired gunpowder.

We have yet taken no notice of the third material used in the composition of gunpowder, viz., sulphur; nor of the base of' the saltpetre, potash, For neither the sulphur nor the potash arc elements from which the expanding gas is formed; that proceeds alone from the combination of the oxygen of the nitre with the carbon, for gunpowder of equal strength can be formed with saltpetre and charcoal only ; but the sulphur has many valuable properties, which render its mixture necessary and advantageous in the manufacture of good gunpowder ; it is highly combustible at a lower temperature, about 550o, and in the combustion, no doubt, assists in the ignition of the charcoal and combines with the potash forming sulphuret of potassium. It has the valuable property, being unalterable itself in moisture, of closing the absorbent pores of the charcoal, and from its hardness and tenacity assists in adding firmness to the grain of gunpowder, qualities that are invaluable when powder is to be stored or transported. Good gunpowder cannot, therefore, be manufactured without a due portion of sulphur.

The question, therefore, now is, what are the best proportions of the three ingredients ? Chemists have decided, in general terms, that the proportion of charcoal should be just sufficient to absorb the oxygen of the saltpetre, and the sulphur to saturate the potash. Then, according to the theory of chemical equivalents, the weight of the compound will be the sum of the weights of the equivalents, thus :—

Dr. Shaughnessy remarks on the above :—" There are, at least, seven definite compounds of sulphur and potassium, and there are, at least, two of carbon and oxygen always formed on the explosion of gunpowder. The carbonic acid in part combines with potassa ; the sulphur is partly converted into sulphuric acid ; compounds of nitrogen and oxygen, especially nitric oxyde (N 1, 0 2), are produced, and in some analyses cyanogen and its compounds have been detected ; all this is cited to show that the results cannot be enunciated in the above simple tern-is, and admit not of estimation in these simple formula."

There is a simplicity in the above calculations ; but as we find that the most celebrated chemists offer different results, and that it is easy to combine under given proportions atomic weights into other and different forms, we must pause ere we accept the above theory as complete.

As regards gunpowder, we may observe, and it is a curious circumstance, that the resulting proportions of chemistry are nearly those universally made use of by manufacturers of gunpowder in early times.

The quantity of gas, the temperature of the combustion, and the expansion under this temperature, are uncertain and varied quantities ; but the average may be taken, that one measure of gunpowder will yield from 240 to 290 equal measures of permanent elastic gas ; [Gay Lussac estimated this quantity of permanently gaseous volume at 450. See Appendix on this subject. -EDITOR] that if the temperature during combustion is as high as 2196o Fahr., this will create an expansion or a propulsive force of about 1592 atmospheres; taking the atmosphere at only 14 1/2 lbs., there results the astonishing pressure of' 1592 x 14 1/2 = 22,074 lbs. on the square inch of surface at the moment of combustion.

NOTE.—I am well aware that many writers on the subject have estimated these measures, temperature and expansive forces, both in excess and below the average statement here given. Great variety will arise from the nature of the charcoal used in the composition, supposing the other ingredients pure, and also from the circumstances under which such experiments are tried, from the quality of the manufactured material and the proportions used. The subject will be further alluded to in the Appendix.—EDITOR.

Col. William Anderson, C.B., Late Agent At Ishapore.

Sketch Of The Mode Of Manufacturing Gunpowder At The Ishapore Mills In Bengal. With A Record Of The Experiments Carried On To Ascertain The Value Of Charge, Windage, Vent And Weight, Etc; In Mortars And Muskets; Also Reports Of The Various Proofs Of Powder.
London: John Weale, 59, High Holborn.
1862.

Re-Shaking Black Powder

In France [black] powder was formerly examined once a year for moisture. For this purpose it was re-shaken in the magazine, which was done as follows:-The barrels were rolled on the floor of the magazine, which was covered with hair rugs. If the sound was uniform, then the powder was good. Any powder found to be moist was dried in the air, if its moisture were not more than 6 to 7 per cent. The barrels were also dried, and after dusting it the powder was again packed. If it bad clogged into lumps, they were broken by hand; and if the barrel was moist, the powder was put into a dry one and well shaken in order to divide the lumps. Powder so re-worked was not put into its original place again, but what had been lying below was put on the top, and *vice versd. If* the moisture of the powder were more than 7 per cent., or if the saltpetre had begun to effloresce, then the powder was again stamped, after it had been determined by a quantitative analysis that the proportions had not altered.

In order to determine the moisture three samples of the powder to be examined are taken-one from the bottom, one from the centre, and one from the surface of the barrel; then the samples are carefully mixed and 5 grammes are weighed out, dried, and again weighed.

In Prussia, powder was formerly exposed to the sun every two years, but it is now only done every eight or ten years in all cases where the magazines are dry and well adapted for storage.

Oscar Guttmann
The Manufacture of Explosives
New York McMillan and Co. 1895

The Saltpeter Men

1630, 14th February. Sir Francis Seymour to Secretary Coke. The saltpetre men care not in whose houses they dig, threatening men that by their commission they may dig in any man's house, in any room, and at any time, which will prove a great grievance to the country. In the town where the writer lives they have digged up some malting rooms, and threaten to dig more. They dig up the entries and halls of divers men. If any oppose them they break up men's houses and dig by force. They make men carry their saltpetre at a groat a mile, and take their carriages in sowing time and harvest, with many other oppressions. Hopes that these men may not be allowed to strain their commission. The saltpetre man's name for Wilts is Hellyer. (S. P. Dom. Charles 1, vol. clxi, No. i.)

1630, 20th February. Petition of Hugh Grove, Deputy for making saltpetre to the Lords of the Admiralty. Complains of Thomas Stallam and others of Thetford for refusing to carry saltpetre liquors. Prays that they may be sent for by warrant. (S. P. Dom. Charles 1, vol. clxi,,No. 35)

1630, 6th March. Gabriel Dowse and others to the Lords of the Admiralty. The complaints of wrongs committed by Stevens the saltpetre man are so great that they had not been able to reduce them into method. Pray a respite of their certificate for a fortnight or three weeks. (S. P. Dom. Charles 1, vol. clxii, No. 40'

1630, 23rd March. Thos. Bond to Nicholas. Understands Lords of the Admiralty have referred the collection of the proofs against the saltpetre men to two knights. . . . saltpetre men make their vaunts that they will get their Iiberty and carry themselves in the country as formerly. . . . If the saltpetre men go down without redress of wrongs it will despair into the heart of the country.... (S. P. Dom. Charles vol. clxiii, No. 40)

1630 30th April. Sir William Russell, Sir John Wolsterholme, and Sir Kenelm Digby to the Lords of the Admiralty. Report on consideration of the complaints and examinations sent in against Mr. Hilliard and Mr. Stephens, saltpetre men and their servants. According to the proofs there is no part of their commission which they have not extremely abused. As in digging in all places without distinction, as in parlours, bedchambers, threshing and malting floors yea, God's own house they have not forborne; so they respect not times, digging in the breeding time in dovehouses, and working sometimes a month together, whereby the flights of doves are destroyed; and without respect to harvest time in barns and in malting houses, when green malt is upon the floor; and bedchambers, placing their tubs by the bedside of the old and sick, even of women in childbed, and persons on their death-beds. They have undermined walls, and seldom fill up the places they have digged. In taking up, cart they observe no seasons, and charge more carts than are needful, discharging some again for bribes, and overload the carts they employ. They do not pay the prices for carriage required by the commission. They take up coals not only where they a sold but from those that have fetched them 20 or 30 miles by land for their own winter's provision. They recommend that the offenders should be punished, and that the commission be taken in, and a new one made out, with restrictions designed to put an end to the abuses complained of (S. P. Dom. Charles 11 vol. clxv, No. 38.)

1630, 26th June. Petition of Nicholas Stephens, Deputy saltpetre men to the Lords of the Admiralty. The Lords having directed Attorney General to proceed against him in the especially in the charge of digging in the Norton, he begs them to consider the declaration annexed, to withdraw the order for proceeding in the Star Chamber.

Annexing the declaration above alluded to. At a time great want of saltpetre he removed only some waste and unnecessary part of the soil of the church of Chipping Norton, as with the concurrence of the parishioners and ministers he had done in the churches of Coventry, Warwick, and Oxford. Other digging was done in his absence by his servant, whom he cast into Oxford gaol, and made satisfaction to the parishioners. (S. Dom. Charles I, vol. clx, No. 46.)

1630, July. Petition of Thomas Hilliard, one of the saltpetre men, on behalf of himself and his servants to the Lords of the Admiralty. By commission dated April 28, 5 Charles I, they were authorized to work for petre in the houses of any of His Majesty's subjects, and within privileged places. About January last, petitioner's workmen endeavoured to dig in the pigeon house of Thomas Bond, who disobeyed the commission, and complained against petitioner, and in February last procured him and his workmen to be sent for by warrant. They have ever since remained prisoners. Pray to be dismissed. (S. P. Dom. Charles I, vol. clxxi, No. 79.)

1631, 16th March. Thomas Thornhill to the Lords of the Admiralty. He complains of endeavours made to prevent the search for saltpetre, by laying soap ashes on the earth, paving cellars with stone, or filling them with gravel. (S. P. Dom Charles I, vol. clxxxvi, No. 102.)

1631, April. Requests of Stephen Barrett, John Vincent, Thomas Hilliard, and five others, the Deputies of the Lords of the Admiralty for making saltpetre, to the same Lords. It being the pleasure of the Lords to renew or alter the Commission under which the Deputies act, they set forth certain provisions which they desire to have inserted in the new Commission for their defence. Among other things, if forbidden to dig in bedrooms, they desire not to be debarred from digging in other rooms in dwelling houses; also that owners of dove houses and stables should be prohibited from adopting measures which, prevent the growth of saltpetre; that owners of carriages may still be compellable to carry the saltpetre at 4d. a mile; that the Deputies may take- wood ashes wherever found at a certain reasonable price; with other provisions framed in the same spirit. (S. P. Dom. Charles 1, vol. clxxxix, No. 89.)

1631, 14th June. Matthew Goad, Deputy Clerk of the Star Chamber, to the judges of the same Court. Certificate that in the cause of John Morley and others against Thos. Hilliard and others, it is confessed in the answers of the defendants that some of them dug for saltpetre under the beds of persons who were sick therein, that compositions were taken for discharge of carts commanded to carry saltpetre, that Hilliard hired horses to draw his wife's coach up and down the country at the King's price, and caused the country to carry coals for the work of saltpetre, and sold the same again to his own advantage. (S. P. Dom. Charles I, vol. cxciii, No. 83.)

1634, 14th March. A proclamation for the preservation of the mines of saltpetre. No dovehouse or dovecot or cellar to be paved, and no stables pitched paved or gravelled, where horse feet stand, but planked only. (Rymer's " Foedera," xix, p. 601.)

18th March. The Lords of the Admiralty to the Governor and Company of Soapboilers. Give orders that the saltpetre men are to have the pre-emption of wood ashes, on the ground that saltpetre is a commodity of such necessary use for the King and Public that it ~ought to be preferred before the making of soap. (S. P. Dom. Charles I, vol. cclxiii, No. i.)

1634, 15th November. Richard Bagnall, slatpeter man to Nicholas. Sends enclosed list of names of those who have lately carried forth their earth in their pigeon houses. If some course be not taken others will do the same, and it will be impossible for the saltpetre men to supply their great proportions, besides destroying the mine. (S. P. Dom. Charles I, Vol. cclxxvii, No. 52.)

Annexed list (52. i) above mentioned. It contains names of persons in cos. Oxford and Warwick.

1634, 2nd December. Petition of John Giffard, saltpetre man to the Lords of the Admiralty. His hindrances by refusal of people in Gloucester to carry coal from the adjacent pits to his boiling-house in Thornburg; also because they carry off the earth from their pigeon-houses to manure their lands. (S. P. Dom. Charles I, Vol. cclxxviii, No. 4-)

1634, 26th November. The Lords of the Admiralty to Montjoy Earl of Newport. His Majesty is resolved to take into his hands and disposition all the gunpowder made of the saltpetre of the kingdom, for better furnishing his occasions and those of his subjects. (S. P. Dom. Charles I, vol. cclxxvii, No. 96.)

1634, 2nd December. Petition of John Giffard, saltpetre man to the Lords of the Admiralty. His hindrances by refusal of people in Gloucester to carry coal from the adjacent pits to his boilinghouse in Thornburg; also because they carry off the earth from their pigeon-houses to manure their lands. (S. P. Dom. Charles I, vol. cclxxviii, No- 4.)

1635, 18th April. Admiralty order to enquire concerning complaints of Thomas Thornhill that divers persons in Somerset, contrary to proclamations, have carried forth the earth out of their dovehouses, and divers inn-keepers have paved their stables, by which practices the mine of saltpetre is destroyed. (S. P. Dom. Charles I, vol. cclxiv, f. 115-)

1637, 3rd June. Articles exhibited to the Commissioners for Saltpetre by Christopher Wren, Dean of Windsor, and Rector of Knoyle Magna or Epicopi, Wilts, against Thomas Thornhill, saltpetreman, for damage done by digging for saltpetre in the pigeon-house of the said rectory. There have been two diggings in this pigeon-house, one by Helyar, whom Thornhill then served, about eight years ago, the other by Thornhill in March, 1636-7. On the first occasion, the pigeon-house, built of massy stone walls 20 ft. high, was so shaken that the Rector was forced to buttress tip the east side thereof. On the last occasion the foundation was undermined, and the north wall fell in. The loss to the Rector had been that of three breeds, whereof the least never yielded fewer than -o or 4o dozen, and of the whole flight, which forsook the house, and the Rector stands endangered to the law for dilapidations. Thornhill has refused all recompense, telling the Dean that the King must bear him out. The Dean desires that Thornhill may make full recompense according to the King's pleasure signified on behalf of the Dean, who is registrar of the Garter, at the last chapter of the Order in Whitehall on 18th April last. Underwritten:

8.1. Order of the Lords that Thornhill answer these articles by that day sennight. Whitehall, 3rd June, 1637(S. P. Dom. Charles 1, vol. ccclxi, No. 8.)

The Rise and Progress of the British Explosives Industry
Published under the auspices of the:—
VIIth International Congress of Applied Chemistry
E A Brayley Hodgetts editor
Whittaker and Co. London 1909

A Short Historical Sketch of Gunpowder.

THE question, " Who discovered gunpowder? " is usually answered to-day *unisono:* "Berthold Schwarz, the Freiburg monk."

So our youth has been taught for two generations, and this is quite enough to make any doubt of this assumed fact appear as idle folly.

Nevertheless doubt is justified. The contemporaneous writers, the authors of the middle and of the latter half of the fourteenth century, knew nothing Of the discovery of the monk of Freiburg. The name of Berthold Schwarz' is first mentioned long after "Büchsen" and "Katzen" (small cannon or mortars) were used in firing, and after a "Katzenstadl," i. e., a gun-foundry, as well as an arsenal existed in Augsburg, for instance.

But even those who grant Schwarz the honor of being the first to make use of the preparation of gunpowder in Germany, and to spread the knowledge of its use, deny him part of the merit, of the discovery. They assert that he too belongs to the great number of those "who did *not* discover gunpowder;" at all events be could not have taken out a patent on his "invention," for it had been in use for: centuries. The Chinese had been long acquainted with it; traces of it are found among the Saracens and the Byzantines; it may be assumed, say they, that the discovery is derived from the Chinese, and has passed by various, no longer accurately -determinable, steps, to the Byzantines, and through them has arrived in, Germany; although the Byzatine or "Greek fire" is not identical with modern gunpowder, it is of earlier date, and the latter bears the same relation to the former that an amendment bears to the principal motion, or an additional or improvement pattern to the main patent.

Occupied with these doubts, I find in the ‚Chronicles of Augsburg," composed by the learned Clemens Jager, about the middle of the sixteenth century, the notice that a *Jew, named Typsiles, discovered gunpowder in the year 1853, in Augsburg, and from Augsburg the preparation of gunpowder, its application to military purposes, and the manufacture of fire-arms, spread throughout Germany and over the rest of Europe.*

True, the chronicler Clemens Jager, writes two hundred years after the discovery and the propagation of gunpowder manufacture in Europe, and cannot therefore speak from personal observation or the observation of his contemporaries. But the same is true of the warranters and witnesses of the patent of the monk of Freiburg. Clemens Jager is, however, to be regarded as an earnest and authentic writer, who has studied, his sources carefully. We are compelled to believe that, to make such an assertion with such apodictic certainty, be must have had his good sources and grounds therefor, and that he could assume belief and agreement in his assertion from his fellow citizens in Augsburg, who were acquainted with his sources, and instructed by the traditions of their forefathers on the subject. Indeed, his statement, not. only. remained uncontradicted at the time, but was confirmed and repeated by other chroniclers and other authors of later date.

We may therefore assume as authentic that it was believed in Augsburg, in the sixteenth century, that the discovery or rediscovery of gunpowder by the said Typsiles took place within the walls of that good city.

I acknowledge that this view is founded on a legend as well as that which as that which asserts the authorship of Bertbold Schwarz. In this respect one has not much preference over the other. We also know little more of Schwarz than of Typsiles; in both cases we must be content with the mere names.

But here tbeye is nevertheless a slight difference. "Schwarz" belongs to the names which are so common that they hardly bear the stamp of individuality. Schwarz is a name like Brown or White, like Smith or Jones, like Miller or Baker.

Typsiles, on the contrary, has a meaning. The name is not of Jewish, but of Greek origin, when we consider Typto or Psilos, or regard it as a compound of the two, or of two similar words.

The name points to the Levant, to the Byzantine empire—to Constantinople, which at that time not yet conquered from the Turks, bad still an active inter course with the West; we find, for instance, Byzantine coins everywhere, from Hungary and Roumania to Denmark-and Sweden, and thence to Portugal and Spain. The old German shrines of relies are of Byzantine origin. So also the old imperial crowns. And the Hungarian king's crown, so celebrated for its age and adventures (it was several times sold, stolen, pawned, conquered, robbed, hidden, and yet always reproduced), and regarded by Hungarian as, sacred, is of Bygantine origin.

It is a fact that the Byzantines possessed an explosive substance closely related to modem gunpowder, as it came into use in the middle of the fourteenth century in Germany, and, middle and Western Europe.

These circumstances lead, us to the conjecture that the said Typsilps, be he of Jewish, or Greek Catholic, or Roman Catholic confession—for faith has nothing to do with gunpowder-came from the Orient, and brought thence a knowledge of the preparation of Greek fire into the free imperial city of, Augsburg. the metropolis then of. the Alemannic countries in, Germany, where, by modifications of the technical methods employed, he effected the preparation of our gunpowder.

I do not intend to write an , account of the Greek fire, or the science of gunnery in Constantinople, which passed from the Byzantines to the Turks (as did, for instance., the dome of the churches, and much else), but only,, *en passan*t, to insert two interesting notices.

The "Greek fire", played its part on into the nineteenth century.

During the Greek war for independence in the twenties, the Greeks obtained only occasional successes by land, and these did not prove to be lasting. The separate bands of the, armatoli, klephts and palikari, brave as they were, soon dispersed again.

The truly decisive triumphs of more permanent effect were gained at sea, where a Miavlis and a Sachturis delivered murderous battle to the fleets of Chosren and Ibrahim; and here it was that the activity of the Greeks triumphed over the lethargy of the Turks, the small vessels of the Greeks, so capable of manoeuvering, over the colossal, unwieldy and heavy vessels of the Turks; *and principally by means of fire-ships and the Greek fire.*

These small fire-ships, furnished with this combustible, each manned by nine, or at most twelve men, swarmed about the large Turkish ships, surrounded them, on all sides and endeavored to deprive. them of wind. The Greeks were familiar with the seas and coasts, those of the mainland as well as those of the innumerable islands, which latter had furnished the trained mariners, men of bravery and skill, inured to the perils of war and the sea, whose wants were so few that a handful of black olives sufficed for a day's subsistence. They were versed in the wind and weather of these seas, and could anticipate their character for several days, so as to prepare combined plans of operations in advance. The Turks, on the contrary, generally rode at anchor. "To anchor suits best the believers in Fatalism," *(Mouiller convient aux adeptes du Fatalism*) says the French Vice-Admiral Jurien de la Graviére, in his highly interesting contribution to the history of the Orient, from 1815 to 1830,, which be has furnished in his book La Station du Levant."

When the Turkish ships, finding themselves surrounded by the Greek fire ships, overcoming their fatalistic lethargy finally put themselves in motion, it was generally too late to escape them by a precisely executed manoeuvre. The fire-ship knew how to attach itself and its fire-its Greek fire-which burned on and exploded even - under water, so skillfully that it could not be gotten rid of. The nine or twelve men in the fire-ship pulled rapidly out of danger in a light boat, while, the Greek fire blew the Turkish vessel into the air, or at least tore open a breach of several meters in extent, and thereby usually succeeded in sinking it.

In our torpedoes and torpedo-boats we observe a new form and application of the Greek fire-ships and Greek fire, which has possibly entirely changed naval warfare. At all events the above-mentioned Jurien de la Graviére thought it possible to predict as much. Meanwhile the Germans have every reason to be grateful for the invention of torpedoes, for in 1870 they successfully protected and defended their coasts and seaports.

So much for the notice in regard to *the history of the Greekfire.*

The second notice relates to the *artillery of the Turks*. In the palmiest days of the Turkish Empire, in the fifteenth and sixteenth centuries, the Turkish army excelled in cavalry and artillery. As early as the fifteenth century a gun-foundry existed in Constantinople. In Turkish it is called *top-hané*. In the ear of the Turk the cannon shot does not sound as in our own, "bang," but "top." *Top* is the gun, and *hané* the house. Hence, "gun-house"; and this is precisely what "Katzenstadl" signifies in Augsburg. In the sixteenth century this gun-foundry, this *top-hané*, lying in the suburb Pera, enjoyed an extraordinary celebrity, and the writers of the day (the Genoese Giovanni Antonio Menavino, for instance) do not fail to add to their notice of this

gun-foundry, that they were Greek Jews or Jewish Greeks who conducted the entire establishment, namely, the casting of cannon and the preparation of gunpowder, thus furnishing the elements of war and destruction to the hereditary and arch-fiend of Christendom, as the Turks were called, although then and thereafter in alliance, or at least in most cordial harmony, with the "most' Christian King" of France. Here certain remarks are pertinent,
which I hesitate to communicate. They may be oil on the flames of 'our anti-semitics of to-day. At all events these remarks cast a peculiar, even somewhat comic, retrospective light on the fact that it was also as Clemens Jäger informs us, a Greek "Jew named Typsiles," who furnished the Christians of the West their elements of destruction and war to be used against the Mohammedans of the East.

The matter is therefore compensated.

Let us return, after the communication of these Greco-Turkish notices, from the East to the West, to Germany, to Augsburg. That this metropolis of the Alemanni, like Nürmberg, the metropolis of the Franks, stood then, in the fourteenth century, on the pinnacle of arts and manufactures in Germany, is an indisputable fact. Nürmberg was celebrated for the discovery of painting on glass, Augsburg for that of linen or rag paper (in contradistinction to the old parchment or the East Indian cotton paper). Event his claim is contested by other German cities Ravensburg, for instance, which can, produce a register of the year 1324 written on rag paper, and a linen paper mill of the year 1412. But the claims of Augsburg rest on older documents namely—city accounts of the year 1320, undoubtedly genuine, and written on linen paper. It also possesses such a document, of 1330, and many from 1360 on. In short, there is no doubt that Germany can produce the oldest documents on linen paper—older than those produced by, Spain and Italy—and that, among. the competing German cities it is Augsburg again which contains the oldest of these possessions. The importance of this discovery is apparent when it is remmbered, that the art of printing would not have, spread so rapidly so soon after its discovery, had not linen paper, which surpasses parchment paper in cheapness and cotton paper in durability, already existed.

This same Augsburg, which rejoices in the oldest linen paper, rejoices also in the oldest cannon, i.e., machines from which, by *means of gunpowder, balls* were fired at the enemy. There were then called "Katzen," or "Büchsen" (generally written "Puchsen, "Buchsen," or " Pugxen"). At first they were of wood with iron hoops, and threw stone
balls. Augsburg made use of such machines as early as 1372, in the war again the Bavarian dukes. This is attested by the historian Adelzreiter, and confirmed by the city accounts, in which it may also be seen that the gunpowder manufactured by the city was made from saltpetre. In the city accounts of 1377 "grosze Büchsen," *larqe* guns, which the city ordered to be *cast*, are already mentioned, hence *metallic* cannon. There also existed a "Büchsen-meister," or a master-gunner, appointed by the city.

I select the following extracts from a valuable little paper on "Augsburg and its Former Industries," long ago out of print and forgotten, written and published under commission from the city, by the industrious city recorder, Theodore

Herberger, in 1852, on the occasion of the exposition of arts and manufactures of the Bavarian district of Suabia and Neuberg, which is based upon an accurate study of the archives there, namely, the city accounts.

In 1371 the city had expenditures "for saltpetre for the guns," for saltpetre for the manufacture of powder for the guns. In the account 20 guns are mentioned as being used in firing; moreover, "Trinkgelder " (pour boire) for the vassals who served these guns. The expenditures for the wooden frames on which the guns were supported are reckoned in the account. A year later, 1372, 400 shot were cast for the guns; lead "for casting" occurs in the account, saltpetre and "wilder schwefel" (wild sulphur) for gunpowder. One year later, 1373, the expenditure for copper, lead, "and other material" is reckoned in the account "for 4 guns. Another year later there occurs in the accounts an item "for a mortar, in which powder for cannon is pulverized." Many such and similar items may be cited to show how far advanced Augsburg was when the art of firing with heavy guns began. Master Walther, the master-gunner, was not only paid, in 1373, the uncommonly large sum of 1601 ffl., but, also received a special present in cloth for constructing the guns ordered, and inspecting the preparation of gunpowder in the court of a "canon of St. Moriz." An unusual number of large can-non was manufactured, according to the accounts, in the years 1410 and 1414, and in the year 1416 the master-gunner, Ott, who was also employed to cast bells in foreign cities, cast several large pieces. All this proves the early date of an immense trade in this department. An especially remarkable man appears in Augsburg in the year 1436. Master Heinrich Roggenburger, the master-gunner. His office is more particularly "the casting of guns, large and small," and the firing of them "as dexterously as has ever been seen;" he can also prepare the powder therefor.

Besides, he. is a man remarkably well versed in, the technicalities of his art in other respects also, and in his letter of admission, he is recommended for the following qualities: He can "make cast and projectile apparatus, large and small, the like of which was, never seen in German lands, for this apparatus stands still after the throw without moving or altering its position, and, not requiring to be bound or held; ", these machines throw masses of five or six hundred-weight; besides, he makes lifting machines, by means of which a hundred hundred-weight may be lifted from or, upon a wagon; also shields for guns and war chariots, and bridges which may be carried over land and laid over ditches or running water. More over, he understands the building of houses and towers, water-mills, wind mills and horse-mills, and can make cast, earthen and wooden water conduits to supply the water of wells to bill and valley. Roggenburger received a yearly salary of 110 fl. In the year 1502 the town had a foundry of its own built, which was called Katzenstadl. Here, according to the account of his contremporary. Clemens Sender, Niklas Oberacker cast one hundred metallic pieces and a mortar; among the larger pieces were several forty feet in length. The most noted of all the gun-founders of Angsburg was Gregor Löffler._He was much occupied, not only in Augsburg, but also in foreign countries. in the year 1529 the Government called him to Innsbruck. In this year and in 1537 he had orders to recast all the old pieces which the Emperor and King Ferdinand had in the Tyrol. Among the newly cast cannon were "Karthaune," capable of firing a shot of an hundred-weight. This work gained for him such approbation that he was entrusted with casting the statues designed to decorate the tomb of the Emperor Maximilian.

Thus far the recorder Herberger. The statues in kugsburg cast by Gregor Loffel are, nevertheless, not identical with those colossal life-statues which now surround the tomb of the "last of the knights " in the Franciscan church at Innsbruck, the authors of which were the brothers Stephen and Melchior Godl. The statues of Löffler, representing various saints, twenty-three in number, are found in the same church, in the so-called Silver Chapel," on the south wall.

I will not further expand this chapter on Augsburg gunpowder and Augsburg ordnance; whoever desires to pursue the subject further, him I refer to the Augsburg chronicles of the fourteenth and fifteenth centuries, published by Professor Karl Mayer.

I hasten to conclude. I am aware that this unassuming chat does not solve the problem, but only brings us a trifle nearer the solution. I only desired to instigate doubt and investigation.

Van Nostrand's Science Series Vo. 70.

EXPLOSIVE MATERIALS
A Series of Lectures delivered before the
College de France, at Paris,
By
M. P. E. BERTHELOT

Translated by Marcus Benjamin, Ph. B., F.C.S.

TO WHICH IS ADDED A
SHORT HISTORICAL SKETCH OF GUNPOWDER
Translated from the German by Karl Braun,
By LIEUT. JOHN P. WISSER, U.S.A.

AND
A Bibliography of Works on Explosives

New York:
D. Van Nostrand, Publisher
1883

very fearful that some unhappy accident may befall

1639, 27th April. Office of Ordnance. Officers of ordnance to Sec. Windebank. Upon information that there was secretly brought into the house of Robert Davies, of Thames Street, divers barrels of powder which we conceived might be either foreign or embezzled out of some of His Majesty's ships, we granted a search warrant to our messenger, and perceive by his return, that he has found the following: 8 cwt. saltpetre, about 10 bushels small coal, some sulphur, 4 mortars of wood and pestles, 2 brass pans, 6 bushels wood ashes and one searcher' or sieve, whereby it is probable that Davies privately makes powder having all things necessary, and in regard he heretofore used that trade in Whitechapel parish, where by accident he had his house blown up. The neighbours near Davies are very fearful that some unhappy accident may befall if he be suffered either to keep any great quantity of powder in his house or to make powder there, and therefore they have entreated us to make known the same to you that such order may be taken with him as you shall think fit, he having formerly been questioned before the Board for the like occasion and bond taken of him not to make any more powder. (S. P. Dom. Charles 1, Vol. ccccxviii, No. 69.)

The Rise and Progress of the British Explosives Industry
Published under the auspices of the:—
VIIth International Congress of Applied Chemistry
E A Brayley Hodgetts editor
Whittaker and Co. London 1909

Testing Black Powder

C) COMBUSTION.

If good black powder be ignited on white paper, it bums away rapidly,* the smoke ascending vertically, and leaves no residue on the paper. If black spots be found, they indicate, either that the powder contains too much charcoal, or that it has been badly mixed; the same can be said of the sulphur, if yellow spots be left behind. If unburned grains be found, they indicate an imperfect or impure saltpetre. The powder should not burn any holes into the paper, as only moist or otherwise bad powder does so.

Professor Charles E. Munroe has suggested a pyrographic method for examining the quality of black powder. He uses paper sensitized with cyanide of iron, the blue colour of which is destroyed by the sulphites and thiosulphates formed by the combustion of the powder, with the formation of yellow and white spots. A piece of such paper about 8 inches square is moistened and laid on a glass or copper plate. A hollow blunt lead cone holding about 3 cubic centimetres is filled with powder by closing its point with the finger, and then it is reversed on the paper. A small conical heap is thus formed, which is then ignited by an incandescent wire. If the paper be allowed to stand for half a minute, and afterwards washed in running water, only small and uniformly distributed spots will be seen with well-mixed powder; whereas a badly-mixed powder gives large spots of unequal form and division. Powder-cake from incorporating-mills will, according to the perfection of the mixture, show residual particles thrown about nearer or further from the centre of ignition.

In a closed space the combustion of powders of equal composition, but varying in size of grains, gives the same quantity of gases and the same temperature; whilst with combustion in the open air and under ordinary pressure sporting powder bums away much more violently than blasting powder, although both may have the same composition. That this is not the case with combustion in a closed space has been proved by experiments with the author's power-gauge, of which more will be said later on.

Oscar Guttmann
The Manufacture of Explosives
New York McMillan and Co. 1895

Restoring Unserviceable Powder

Restoring Unserviceable Powder.

When powder has been damaged by being stored in damp places, it loses its strength, and requires to be worked over. If the quantity of moisture absorbed do not exceed 7 per cent., it is sufficient to dry it to restore it for service. This is done by exposing it to the sun.

When powder has absorbed more than 7 per cent. of water, it is sent to the powder-mills to be worked over.

When it has been damaged with salt water, or become mixed with foreign matters which cannot be separated by sifting, the saltpetre is dissolved out from the other materials and collected by evaporation.

The Ordnance Manual for The Use of the Officers of the US Army 3rd ed 1861

Baked Black Powder

Wiener Powders (Baked Powders)

Introduced in Russia in 1873, they were prepared by compression of the usual ingredients of black powder, preheated to 120°C. This was done in order to melt the sulphur, and thus achieve its better distribution throughout the mass.

A similar powder was manufactured later in the USA and was known as "Russian Powder". In England, a similar powder was know as "Baked Powder". Tests conducted in 1878 at Woolwich Arsenal indicated that this powder was no better than conventional black powder. The same unfavorable results were obtained by a Colonel deMaria in Italy.

PATR 2700 The Encyclopedia of Explosives and Related Devices

Sulphur.-Sulphur can likewise be used in the state in which it leaves the purifying process described at p. 225. But as the flowers of sulphur always contain a little sulphuric acid, the stick or roll sulphur is preferred, which, at some period of the process, must be pulverised.

The influence which the *charcoal,* by reason of its porosity, inflammability, &c., exerts upon the quality of the powder is very considerable. The quality of the charcoal again, depends upon the material from which it is prepared, and upon the method of its preparation. Proust's experiments have also thrown light upon the former of these points. He found that a mixture of 72 grains of saltpetre was consumed with whilst carbon from rice, Starch, albumen, blood, leather, &e. would produce no detonation. It is obvious from the table, that the soft, woody parts of plants produce the best charcoal, and nitrogenised animal matters the worst. For the same reason, paper is not applicable in consequence of the - size it contains, whilst the fibre of flax and old linen is an excellent material for this purpose. The wood of the bird-cherry is now very frequently used, also that of the elder, poplar, maple, and walnut. In Spain the preference is given to hemp charcoal. Whatever kind of wood is employed, those parts must be thrown aside, which carbonize in the manner of starch, or albumen, &c. For this reason, the bark, which is impregnated with gummy, mucilaginous and extractive matters besides salts, must be peeled off from the wood for charring, all the leaves and smaller branches removed, and wood which is not too old, and yet fully developed should be selected. Branches, from I to 2 inches in thickness, are best for this purpose. It is also found advantageous to expose the pealed boughs. to the rain for some time, which removes still more of the extractive matter.

12 grains of carbon from:	In seconds	Leaving a residue of
Hemp stalks	10	12 grains
King's spear	13	12
Vine branches	12	20
Chick-pea stalks	13	21
Pine-wood	17	30
Common bird-chyerry tree *(Rhamus frangula)*	20	41
Maple *(Evonymus europ.)*	21	27
Hazel	23	30
Horse chesnut wood	26	36
Walnut	29	33
Coke	50	45
Sugar	70	48

The degree of inflammability of charcoal, depends mainly upon its power of conducting heat. If this is slight, the heat communicated to one part, will be the less easily taken from it; it will be extinguished with greater difficulty, and *vice versâ*. Experience has shown the black-charcoal produced at a high temperature, to be more dense and a better conductor of heat, than the product of unfinished carbonization, or the so-called red-charcoal *(charbon roux),* of which mention has been made before (p. 49), In fact a particular kind of charcoal belonging to this class, is now expressly prepared for the manufacture of gunpowder, It can only be procured in a manner which admits of the most accurate regulation of the heat throughout the whole operation. Furnaces are used for this purpose, with cylinders walled into them, Fig. 142 and 143, somewhat resembling the gas furnaces. The three cylinders C, C, C, are of, cast-iron ; the front part projects out of the furnace, and can be closed air-tight by the three discs o, o, o. The hinder part is let into the back of the furnace, so that the wall both supports and closes it. The pipes a, a, Which are seen walled in at this part, and which connect the cylinder with the space t immediately before it, are intended partly for the reception of the test-woods, and partly for conducting the gases and vapours through walled channels to a separate cistern. The flame-fire, which is made upon the grates r r, is more uniformly disseminated by the pierced arch m; it first surrounds the lower halves of the three cylinders, and then the upper ones, by the flues *e, e, e,* and escapes, lastly, by the chimney h. At f there is a damper. When too much ash has collected, it can easily be cleared away through the channels c and d, which at other times are kept closed. The wood for charring fills the middle, narrow portion of the retorts ; the larger pieces being placed on the outside, and the smaller in the interior, having been previously cut to the proper lengths. That the regulation of the process may not be impeded, and a uniform quality of charcoal obtained, it is not advisable to place more than three retorts in one furnace, and these are made only just large enough to hold 100 lbs. of wood; the charcoal is, therefore, rendered somewhat expensive. When all the crevices are luted, the fire is lighted, To effect the decomposition with as little beat as possible is the first object, and the interior must never attain a red heat. The progress of carbonization is estimated by the colour of the escaping vapours, and by the appearance of the test-wood s, which are frequently broken lengthwise, in order to see whether the decomposition has progressed uniformly from end to end. In about five hours the distillation is in full progress. When the vapours appear yellow, and the tests are brittle, and present a yellowish brown, shining fracture, it is time to extinguish the fire, as the heat of the furnace will then suffice to complete the carbonization. When the vapours cease to escape, the lids are quickly removed, and the charcoal, is allowed to cool in well-closed vessels of sheet iron. To avoid long interruptions, the wood is sometimes enclosed in sheet-iron cylinders, which are inserted into the retorts, and exchanged when the operation is finished, for fresh ones. It is questionable whether this method is profitable, as a larger amount of fuel is requisite to cause the heat to penetrate the double casing.

In a well-conducted operation, 34 to 35 per cent of charcoal should remain. This is the usual amount of produce in the charcoal furnaces of Le Bouchet, for instance, where the carbonization is continued during twelve hours for good sporting powder, and for ordinary kinds from eight to nine hours.

Good powder-charcoal *(charbon* roux) should be brown, with veins of a lighter colour, smooth, with a number of cross fissures but none lengthwise; when pounded, it should have the appearance of black shot velvet, [?] should burn with a bluish flame, b slightly flexible, and dissolve almost entirely in caustic potash It is not found advantageous to extinguish the charcoal with water, as, if it is to be used immediately, which is always desirable a calculation must be made for the amount of water, or the proportions in the mixture will not be accurate.

One circumstance, which always occurs during charring, requires particular notice. In the cooler parts of the distilling apparatus, tar is constantly condensed, and on dropping back upon the hot charcoal, is decomposed, leaving a difficulty combustible coal as residue. This, and the half-charred portions, must be carefully separated. They amount, sometimes, to 5 per cent. In furnaces of the beat construction, as at Spandau, for instance, this quantity is reduced to ½ per cent.

By another process, the wood is charred in furnaces resembling those used in the coking Of coal (p. 59). These are constructed with a flat hearth, covered over by a half cylindrical arch, and with two doors, one at each end. In the beginning, when the wood with which the furnace has been charged is ignited, both doors are left open. When the fire has burnt up sufficiently, one of the doors is closed; the other, from which the wood was ignited, is left open for the escape of the smoke, and in order that the Wood may be reached with the rakes, and those pieces, which no longer burn with flame, may be pushed to the back part of the furnace. ' When the, flame is nearly extinguished, the second door is also closed, V diminish the glowing heat. The charcoal may then soon be drawn out and extinguished, in boxes of sheet-iron.

Notwithstanding the saving of time Which is effected by the use of these furnaces they are, nevertheless, not the most profitable, partly on account of the great waste, and production of half-charred wood and tar-charcoal, and chiefly because no definite amount of carbonization can be attained in them, Black charcoal is the only produce they afford.

The ordinary mounds are not adapted to produce charcoal for the powder mills, as the smallest particles of sand introduced into the powder-mixture might strike fire under the machines, and give rise to great danger. Yet, from a very ancient period, a kind of pit carbonization has been practised. These- pits are quadrangular and flat; they are lined with bricks, which are placed upright. A pit 6ft. deep, and 12ft. diameter, is large enough to char 20 cwts. of wood. The margin of the pit must be firm and even; soft, clay-like sand, which is easily formed into balls, and woollen cloths, must also be at band. The wood is bound up in faggots, consisting of some hundreds of pieces, which are arranged with some degree of regularity in two layers, one upon the other, and project about 4 ft. out of the pit. By means of a pole, previously inserted crossways, one row of faggots is easily made somewhat higher than the adjoining one; channel is thus left, which must be open in front, as it serves for the admission of the materials for igniting. Straw and shavings are inserted and ignited, and the whole contents thus set on fire; the month of the channel is then immediately stopped up with faggots, to avoid the admission of an excess of air. The flame gradually makes way, consumes at last the pole, the channel becomes closed, and the mass of wood sinks together. When the fire is extinguished, the pit is no longer filled by that which remains; the same number of faggots are therefore, gradually added, as were at first used. The regular stratification being thus destroyed, it becomes necessary, in order to obtain a uniform state of carbonization throughout, to loosen the mass in some parts, and force it together in others. When the flame is everywhere extinguished, the process is finished. The air is then quickly excluded, by throwing the wetted woollen cloths upon the even surface of the charcoal. On the top is thrown a layer of sand, which is firmly stamped down. In three or four days the charcoal may be drawn, and must be carefully separated from half-charred pieces, and from earthy particles. As much ,is 16 or 17 per cent of charcoal is obtained, by this very imperfect process. Neither the form of the pit, nor the use of sand, can be recommended. Sometimes round iron pans with lids are employed, instead of the pits, which afford a similar kind of coal, and a produce of 23 per cent.

The charcoal from the pits, furnaces, and pans, is black-charcoal, and is in the form of long, sonorous rods, which must not be contaminated with tar-charcoal. In contradistinction to this, the charcoal from the cylinder is called " distilled *charcoal*."

It is always advisable to separate any sand or other impurity from the charcoal, before putting it through any further operation ; this is either done by band, or,- as at the period of the French Revolution, when no time was to be lost, by being thrown with shovels against a current of air, which carried away with it sand and dust.

CHEMICAL TECHNOLOGY OR, CHEMISTRY, APPLIED TO THE ARTS AND TO MANUFACTURES.

BY DR. F. KNAPP,
PROFESSOR AT THE UNIVERSITY OF GIESSEN.
EDITED WITH NUMEROUS NOTES AND ADDITIONS:
BY DR. EDMUND RONALDS,
LECTURER ON CHEMISTRY AT THE MIDDLESEX HOSPITAL,
AND
DR. THOMAS RICHARDSON,
OF NEWCASTLE-ON-TYNE.

VOL. 1.
LONDON:
HIPPOLYTE BAILLIERE, PUBLISHER,
1848.

[I am indebted to Louis Woody for supplying me with a photo copy of this.]

the manufacture of pure charcoal

J Soffern
PROJECTILE WEAPONS OF WAR
3rd ed 1858
Republished by The Richmond Publishing Co. 1971

We now come to the manufacture of pure charcoal, which lately has been carried to a great perfection; to which cause, more than any other, the great superiority of gunpowder now manufactured over that of previous times, is mainly attributable. Charcoal, as all are aware, is essentially "carbon"—-that chemical principle which, in a state of absolute purity, constitutes the diamond. Charcoal is formed by exposing animal or vegetable substances to elevated temperatures under circumstances which do not favour combustion ; that is to say, air being totally or partially excluded. The operation of charcoal making depends upon the fact that carbon is indestructible at any temperature, provided air be excluded. As charcoal made from vegetable substances is the kind invariably employed for the purpose of making gunpowder, we may confine our attention exclusively to that variety.

I need scarcely advert to the common plan of making charcoal; namely, by putting billets of wood into a pit, setting fire to them, then Covering them with turf, &c., in such a manner that just air enough may be admitted to effect slow combustion. Until lately charcoal made by this process was employed by the gunpowder manufacturer. Very early in the history of gunpowder it was discovered that light woods, such as willow and alder, were greatly superior to hard woods in yielding good charcoal, but facts of a chemical nature having reference to the further improvement of charcoal were not then known. When we consider how various are the secretions and juices of vegetables-how different in regard to their volatility and destructibility-how variable are the amounts of lime, potash, soda, and other bodies, some of which exist in most vegetables, and which, being devoid of volatility, must remain behind and contaminate the charcoal— it is evident that no inconsiderable amount of chemical knowledge is required _in the manufacture of this substance for gunpowder.

The common plan, then, of manufacturing charcoal is found never to yield a result of the greatest possible purity: in other words, it is not possible to apply the due amount of heat, so that all volatile substances may be driven off, without at the same time partially destroying the charcoal. The process now followed is that of distillation; the wood, cut into billets of proper length and size, being inserted into cast-iron cylinders or retorts, heated to the requisite degree. By this operation not only is the wood effectually charred, but acetic acid, called from its source "'pyroligneous," and tar, and pyroxylic spirit, ordinarily called wood naptha, valuable results which formerly were dissipated, are now saved; moreover, charcoal thus prepared is said to be more free than any other from potash-a fact which seems attributable to the action of acetic acid in dissolving it out.

In France, since the last few years, a process of charcoal manufacture has been adopted, founded on the discovery of M. Violette, that high pressure steam transmitted amongst and through billets of wood, actually produces a similar result to the application of fire; but much better. Engineers have long been aware of the fact, that steam jets playing against vegetable matter, after a time charred them. The process of M. Violette is a practical application of that fact.

For the best kind of sporting powder soft dry wood is that employed; willow and alder are used for Government powder ; any kind of wood is indiscriminately used for the common powder. In India the gram-bush plant (*cytistts cajan*), Parkinsonia, and milk-edge (*euphorbia tiraculli*), are found to answer well.* [* Braddock's Memoir on Gunpowder] Whatever the wood, it should be carefully decorticated; wherefore it is usually felled in May, when the sap is up. The reason of removing the bark is to prevent scintillation, which, in gunpowder, would be an exceedingly dangerous quality. All who are accustomed to charcoal fires, must have noticed how the bark of charcoal shoots into coruscations ; indeed, the experimental chemist carefully selects, for the purpose of showing the combustion of charcoal in oxygen gas, such portions of charcoal as are supplied with bark; and which, in consequence, beautifully scintillate.

Hemp Charcoal

Takeo Shimizu
Fireworks's The Art,Science and Technique
1st ed 1981

(30) Hemp coal

A black fine Powder which has a somewhat hygroscopic feeling. The apparent specific gravity is 0.22g/cc. The dye adsorption power is the Largest of all the kinds of plant coal. A mixture of hemp coal, sulphur and potassium nitrate burns to produce a violet flame and less fire dust than pine charcoal. It is used to obtain a large force of explosion as a component of black powder or in combination with, potassium chlorate perchlorate for the bursting charge of chrysanthemum shells.

It is more hygroscopic than the pine charcoal; this may be caused by a phosphorus compound which is found in cultivated plants in general. One analysis showed 9.15% moisture and 8.64% ash, and the carbon content may be less than 82%. Such a small carbon content is a defect of this material and the ash contains Si, Cu, K, Al and phosphorus compounds. The wash water (5 grams of hemp coal / 30cc of water) showed a pH value Of 10.5, and changes the colour of phenolphthalein to red. The purification of hemp coal by washing it with water takes much time, because the filtration is very slow due to its alkaline nature. The particle size of commercial hemp coal is less than 20 microns, but it is better to sieve it before use to remove foreign matter.

Manufacture. The following method is typical: An oven is constructed with stones in the shape of a well. The inside diameter of the oven is determined so that the hemp caules can be easily inserted. At first some of the hemp caules are ignited and thrown into the oven, and then the remainder of the caules are heaped on the fire step by step at intervals so that the material thrown in at first does not become ash. Finally, water is poured on the hemp to extinguish the fire. The hemp coal thus produced is dried by itself from the remaining heat. Finally it is crushed to a fine powder and sieved to remove coarse grains and foreign matter.

Effect of Differing Charcoal Types Upon Handmade Lift Powder

Journal of Pyrotechnics Issue 10 Winter 1999
http://www.jpyro.com

Charles Wilson

ABSTRACT

Experimental production of charcoal via the retort method is discussed Charcoals were made from various substances; of special interest were woods belonging to the Salicaceae (willow) family. Lift powders were made using these charcoals and their performance compared using a device for testing powders under conditions similar to those used for propelling *fireworks aerial shells. The author found that handmade powders often outperformed commercially available powders in this application.*

ORGANIC SUBSTITUTES FOR CHARCOAL IN “BLACK POWDER” TYPE PYROTECHNIC FORMULATIONS

Sean Wise
Ronald A. Sassé
Hughes E. Holmes
July 1984
Technical Report ARBRL-TR-02569
Aberdeen Proving Ground, Maryland

ABSTRACT-

A number of crystalline organic compounds have been substituted for the charcoal in black powder in an effort to probe the chemical functionality on charcoal required for combustion. Compounds studied included a large number of polynuclear aromatics and polyphenols to probe the rote of electron transfer reactions as well as an aromatic diacid and some phthalein salts. It was found that polynuclear aromatics-which did not contain any type of oxygen functionally would not sustain combustion. The polyphenols, diacid, and phthalein salts all produced pyrotechnics which sustained combustion and some even burned faster than charcoal-black powder. It was also found that he phenolics which contained catechol/hydroquinone moieties (good organic educing agents) were less reactive than other phenolics. This effect has been attributed to a deactivating reaction between sulfur and the catechol/hydroquinone effect.

One of the mixtures, that containing phenolphthalein, was evaluated further by determining strand-burn rates at various nitrogen pressures to one hundred atmospheres. Combustion rates and photographed features parallel hose of black powder.

INTRODUCTION AND BACKGROUND

Black powder is a mixture of 75 percent potassium nitrate, 10 percent sulfur, and 15 percent charcoal. It is probably the oldest known energetic material and has been used throughout the world for centuries. Even though black powder has been in use for years, the factors that control its combustion properties are not known and certainly not well understood. The reasons for this ambiguity are related to the nature of the composition as it is a heterogeneous mixture of three solids, pressed to about 95 percent theoretical maximum density. Also, charcoal in black powder is a naturally derived substance which contains up to 35 percent tar-like constituents and varies from one source to another. Such variance has been found to have a great impact on the combustion properties of black powder.[1]

Recently poor combustion properties of one lot of black powder has been cited as a cause for weapon malfunctions. [2] One problem area of prime concern is that various lots of black powder made by a particular manufacturer and black powder made by various manufacturers, using apparently equivalent processes, produce a pyrotechnic with different combustion characteristics. In fact, it has been possible to identify "good" and "bad" lots, in relation to device performance without a clear understanding as to the particular differences involved. Such variances are believed to be due to the varying chemical and physical properties of charcoal and to the physical properties of black powder.

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The reasons for the difficulties in characterizing charcoal used in black powder are many. It is an amorphous substance; it reacts and changes on heating; it is a mixture of many components; and only small portions of it dissolve in solvents. Since the material cannot easily be characterized, it has been impossible to learn what reactions might be important in combustion. One hope embraced in this work was to determine if a pure organic compound could be identified that would be an adequate substitute for charcoal and render the same performance, in a reproducible manner, as does "good" black powder. Such a substitute should lead to a more easily studied system to model the combustion processes of black powder. An added benefit may be a new type of pyrotechnic material in which a non-varying chemistry of combustion could exist and uniform physical properties could be maintained.

In choosing organic compounds as substitutes for charcoal in black powder' it is necessary to make an assumption about the important functionality that may contribute to combustion. Since the oxidation of charcoal in combustion is an electron transfer process, it follows that charcoal’s combustion should be made more rapid by functional groups which make electron transfer easier or by easily oxidizable groups present in the material. This hypothesis suggests two classes of model compounds that should be evaluated to study the reactivity of charcoal in black powder: polynuclear aromatics and organic reducing agents.

In the first class of model compounds, we will determine if electrons delocalized over large aromatic pi systems facilitate electron transfer and therefore oxidation. If this is important, then polynuclear aromatic compounds, when substituted for charcoal in black powder, should support combustion.

The second class of compounds studied probes a hypothetical role for the “volatiles” in charcoal during combustion. Rose observed that the "volatiles" in the charcoal play a crucial role in combustion, [l], [4] and fine papers relating volatiles to burning rate were also offered by Hintze [5] and by Kirshenbaum. Gray, March, and Robertson [7] related volatile content to roasting temperature and Sasse' [8] presented complete analysis of charcoal used to make black powder. Although the subject of volatiles appears to be well presented, the mechanisms of combustion are not understood. It is well known that charcoal is not just carbon; it contains 5 to 20 percent (by weight) oxygen, up to 5 percent hydrogen, and smaller amounts of other elements. We suspected that a significant amount of oxygen might be present in the charcoal as catechol or hydroquinone moieties. These compounds are very good organic reducing agents and can easily undergo two-electron oxidations to quinones. In addition, the catechol structure is known to occur in lignin which accounts for approximately 20 to 30 percent of the weight of wood before pyrolysis. [9]

The conditions for the pyrolysis of wood required to make a good black-powder charcoal are stringent but not severe. Thermal analysis has shown that significant amounts of lignin remain in charcoal used for black powder [7], [8] and extreme pyrolysis, to 900C in an inert atmosphere, destroys these organics resulting in a weight loss. Therefore, it is proposed that the lignins originally present act as reducing agents in black powder making charcoal's combustion more facile. To probe the importance of this type of reaction in combustion then, a large number of polyphenolic compounds were evaluated as substitute compounds for charcoal. Some of these were capable of a facile two-electron oxidation and others were not.

Another factor considered in choosing the organic substitutes for charcoal was melting point. Effort was made to choose high-melting materials of the types described above. It was felt that a low-melting-point material would liquefy and agglomerate prior to reacting. This would prevent good mixing of the three components which is required for combustion. The compounds selected are listed in Table 1 showing chemical structure, chemical composition, and melting point.

EXPERIMENTAL -

Characterization of pyrotechnic mixtures was done by Differential Scanning Calorimetry (DSC), and combustion-rate studies of pressed sticks were conducted at one atmosphere. While these two methods by no means constitute complete-characterization, they did permit us to screen a large number of materials; and it was hoped these tests would be adequate to identify some of the functionality required for reaction and combustion. Better characterization will require a more extensive study. One of the mixtures was evaluated further by determining strand-burn rates at various pressures of nitrogen.

A. Preparation of Pyrotechnic Mixtures

The pyrotechnic powders were made by grinding a mixture of 75 parts of potassium nitrate, 1O parts sulfur, and 15 parts of a crystalline organic fuel (by weight) in a mortar and pestle until they passed through a 120-mesh screen. [10] These proportions are used in black powder and no further attempt was made to optimize the stoichiometry. The Maple charcoal used was supplied by Roseville Charcoal Co. of Zanesville, OH and this material was used by the Army Ammunition Plant in Charlestown, IN. [8] Such mixtures were compared to charcoal-black powder which was prepared in the same manner and used as a control in these experiments. Had the samples been ground finer as is done in a jet-mill, they would have burned twice as fast. In an effort to explore the importance of the nitrate oxidizer's melting point, a low melting eutectic of 70 percent potassium nitrate and 30 percent sodium nitrate was prepared, ground, and used in place of the pure potassium nitrate in one experiment. 1t had a melting point near 240° C.

B. Strand-Burning Experiments

The pyrotechnic material was formed into rectangular parallelepipeds by pressing a weighed sample (0.8g) in a constant-volume die where a spacer limited piston travel and controlled dimensions. Internal free volume was kept small; e.g., free volume in the flourescein sample was 5.1 percent. Some samples were inhibited with a coat of cyanoacrylate-based glue. The difference in burn rate between an inhibited and non-inhibited sample is near two.

Many different types of samples were burned at one atmosphere and combustion was recorded on video tape. Burning times were measured by counting picture frames (see Table 2). In most cases, only one sample was prepared of each mixture; therefore, the burning rates are approximate and should be examined with caution. Determination of exact burning rates will require a more extensive study.

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For one pyrotechnic mixture, containing phenolphthalein, strand-burn rates were measured at various high pressures of nitrogen. Cinematography, at 1000 frames per second, was used to record combustion; and burning rates were determined from the slope of the line describing the position history of the regressive surface. This technique and high pressure cell have been described. [10] The samples had a density of 1.86; theoretical maximum density is 1.93.

C. <u>Thermal Analysis</u>

Differential Scanning Calorimetry was performed on a Dupont 990 Thermal Analyzer equipped with a high-pressure DSC cell base. Samples were analyzed as follows: approximately 10 mg of loose pyrotechnic powder was placed in an aluminum sample pan which was covered with a perforated aluminum lid. This was placed in the DSC, flushed with argon and then heated at a rate of 20° C/min. from ambient to 500°C. The phase changes for potassium nitrate, sulfur, and organic compounds were noted; but they are not included in Table 3. The first temperature is the onset value; the second is the peak temperature. -

RESULTS AND DISCUSSION

A. Polynuclear Aromatics

The first group of compounds studied were the polynuclear aromatic materials to determine if delocalizing electrons over a large aromatic pi system could facilitate electron transfer and enhance combustion. Pyrotechnic powders were made with anthracene, tetracene, p-quaterphenyl and rubrene in place of charcoal. None of these mixtures sustained combustion (Table 2). DSC analysis revealed a moderate to weak exothermic reaction normally observed between potassium nitrate and sulfur and no strongly exothermic reactions were observed below 500°C (Table 3).

It might be postulated that none of these materials would be good models for they do not have as extensive a fused aromatic system as does charcoal.

.Graphite, however, has an even more extensive aromatic system than does charcoal and it too does not sustain combustion when used in a pyrotechnic mixture. We believe these results eliminate the possibility that the delocalization of electrons over large fused aromatic systems is a sufficient condition for the facile oxidation of charcoal in black powder.

B. Phenolic Compounds

The second class of compounds studied were phenolics which were further subdivided into two groups; the hydroquinone/catechol type compounds capable of undergoing a two-electron oxidation (quinalizarin, quinizarin, leucoquinizarin, hydroquinone, and catechol) and other phenolics which are not (anthraflavic acid, fluorescein, phenolphthalein, and phenolphthalin). If the two-electron oxidation of the hydroquinone/catechol moiety plays an important role in combustion, then the former group of compounds should burn very well and the latter group should not.

These materials were incorporated into pyrotechnic powders and burned in pressed stick form; the results were surprising. All compounds which easily undergo a two-electron oxidation burned but they burned quite slowly. On the other hand, the polyphenolic compounds which could not undergo this hydroquinone to quinone type oxidation, burned very rapidly. In fact the latter group burned faster than the charcoal-black powder control (See Table 2). The most striking example of this is the comparison of the anthraflavic acid and quinizarin pyrotechnic powders. These two compounds are isomers; -quinizarin is 1,4-dihydroxyanthraquinone and anthraflavic acid is 2,6-dihydroxyanthraquinone. The former compound burned at 0.08 cm/see while the latter burned at 0.44 to 1.4 cm/sec. It is uncertain what is happening in these instances but the DSC data coupled with the combustion of the diketoaromatic, anthraquinone, provides some insight.
:
The DSC analysis of the pyrotechnic powders containing organic compounds with catechol or hydroquinone moteties looked similar to those of charcoal black powder. With black powder there is a double-peaked exotherm associated with the melting point of the potassium nitrate. This peak has been labeled the preignition exotherm and has been attributed to a reaction involving all three components in black powder. In the compositions containing catechol or hydroquinone moieties, this peak is present; and the magnitude of the reaction is similar to that observed in black powder. The next peak in the DSC's of these materials has been labeled the ignition' exotherm because it is during this second exotherm that the greatest amount of heat is released. With black powder this peak is very strong and occurs at about 425°C.

In the catechol/hydroquinone powders, the ignition' peak is reduced in magnitude or it occurs at temperatures in excess of 450°C. It appears that these easily-oxidized systems are being oxidized to ma serials that are much less reactive towards subsequent oxidation. This supposition is supported by the inability of an anthraquinone pyrotechnic powder to sustain combustion. (Anthraquinone is the oxidized form of a hydroquinone, dihydroxyanthraquinone.)

Interestingly, the polyphenolic materials which could not undergo an easy two electron oxidation, such as the anthraflavic acid and the phthaleins, showed little or no exothermic reaction as the potassium nitrate melted; the only reaction observed on the DSC trace was the "ignition" exotherm at about 425°C. Recall that these materials all burned well in their pyrotechnic mixtures. The question one must ask is, “How are these data reconciled with the proposed mechanism describing the role of charcoal's volatiles in the combustion of black powder?" Suggested mechanisms, which include charcoal sulfur reactions, will be discussed.

C. Sulfur Reactions

In their paper on the thermal decomposition of black powder, Blackwood and Bowden [11] discuss the preignition reaction between potassium nitrate, sulfur, and charcoal. They felt that occurred in several steps where the first step is a nonexothermic reduction of sulfur by the organics in charcoal:

S + Charcoal -> H2S + "oxidized" charcoal

This is followed by an exothermic reaction between potassium nitrate and "reduced" sulfur. It may be possible that the species that "oxidizes" the catechol/hydroquinone moieties to some nonreactive compound is the sulfur and not the potassium nitrate. If this is the case, then a sulfurless pyrotechnic powder made with a hydroquinone derivative should burn much better than the equivalent pyrotechnic powder with sulfur. To evaluate this hypothesis, sulfurless powders were made with quinizarin (which contains a hydroquinone moiety) and anthraflavic acid (which does not contain a hydroquinone moiety). Both of these powders burned very rapidly. For the quinizarin this is an increase in rate by a factor of 5-10 and for the anthraflavic acid, little change is observed. It appears then that sulfur is the reactant which turns the hydroquinone/catechol moieties into a less reactive species. The DSC results of the sulfurless compositions support the hypothesis that sulfur is the deactivating species for catechol and hydroquinone systems. When sulfur is not present, these mixtures exhibit stronger ignition exotherms at lower temperatures (see Table 3). Decreases in ignition temperature were 428 to 442°C for quinalizarin, 465 to 430°C for leucoquinizarin, and 475 to 397°C for quinizarin. Other polyphenols exhibited the opposite trend but at a lower magnitude. It seems reasonable that the preignition reactions in charcoal black powders could be due to a reduction of sulfur by catechol moieties originally present in the wood's lignin.

While reduction of sulfur by an organic does not seem to play a crucial role in the burn rate of black powder, perhaps it does play a role in the flame-spread rate of loose granular black powder grains. During the preignition reaction, highly flammable H2S or organic sulfides might be released into the local atmosphere surrounding the powder.* Subsequent

ignition of these gases would accelerate the flame-spread rate. This hypothesis will have to examined carefully both with charcoal-black powder and the model systems.

D. Other Compounds Studied

The discussion in the preceding paragraphs describes what may be occurring in the preignition exotherm of black powder but DSC and combustion data of other pyrotechnic powders reveal that many types of functional groups can cause this reaction. Powders made with terephthalic acid and the sodium salts of fluorescein and phenolphthalein showed similar preignition exotherms and these materials burned quite rapidly in strand-burning tests. No explanation is offered as to what might be occurring in these cases except to say there are probably alternative explanations for the preignition exotherm. These examples illustrate the very complex chemistry that is involved in any potassium nitrate/sulfur/organic system. The discussion also illustrates the magnitude of the black powder chemistry problem where the "organic" is a very poorly defined material, charcoal.

E. Physical States and Reactivity

A final point should be made concerning the influences of phase changes in relation to reaction. In black powder, exothermic reactions are first observed on melting of the potassium nitrate. In this study, it has been observed that all three components must first melt before exothermic reaction takes place as one might expect. However, in many cases, much higher temperatures were needed before any appreciable reactions were observed with DSC data. This was illustrated by using a low melting point eutectic of potassium nitrate and sodium nitrate in one of the pyrotechnic compositions. It was found that phenolphthalein, sulfur, and the nitrate salts would not react to produce an exotherm at 300°C and the melt had to be heated to over 350 degrees C before the onset of exothermic decomposition was detected. This experiment shows that melting of the components is not a sufficient condition for reaction to take place.

F. Strand-Burn Rates at High Pressure

Strand-burn rates for the system phenolphthalein /KNO_3/S as a function of nitrogen pressure are given in Figure 1. The figure also contains similar data for laboratory prepared black powder made from meal ground in a jet-mill. Had the phenolphthalein mixture been ground in a jet-mill one would expect it to burn twice AS fast. This relationship becomes clearer when comparing the two systems that were hand ground having a particle size of 120 microns. Then the one atmosphere burn rates were nearly equal where the black powder had a burn rate of 0.58 cm/see and the phenolphthalein mixture had a burn rate of 0.42 and 0.48 cm/sec. In contrast, finely ground black powder has a burn rate of 1.0 cm/sec.

The phenolphthalein system has similar burn rates to black powder and the burn-rate curves appear of similar shape. One important characteristic is that both systems exhibit a sharp change or "break" in curvature at a few atmospheres pressure. One is tempted to ascribe this commonality to high temperature equilibrium or to chemical reactions of potassium nitrate, the common element in both systems. Another supporting argument is that black powder reacts above the melting point of potassium nitrate and thus, the system is a liquid-solid reaction; in contrast, the phenolphthalein system reacts above the melting point of all constituents and is a liquid-liquid reaction. Therefore, the break cannot be due to the physical state of the fuel. however, at these temperatures some of the organics may undergo pyrolysis to carbon making the two systems alike. The only firm conclusion is that two pyrotechnic systems have similar breaks in their burning-rate curves.

From the cinematography the burning phenolphthalein "sticks" showed a liquid surface that was in extreme turbulence and liquid drops were propelled by the gas stream. No deconsolidation nor evidence of porous burning was observed and the inhibited "stick" burned in "cigarette fashion." The scenes were very similar to those of black powder, except the drops and liquid film appeared slightly larger.

CONCLUSIONS

The work described in this paper has shown that a variety of functionalized aromatic compounds, such as phenols, acids, and their salts, can support combustion in black powder type pyrotechnic formulations. Since so many types of compounds support combustion, it is impassable to say what functionality is really important in charcoal's combustion. Perhaps the variety of compounds that worked may explain why-so many types of charcoal can function adequately in black powder. The negative results with the unfunctionalized polynuclear aromatics, however, do allow one to conclude that some sort of functionality must be present in the charcoal for rapid combustion to take place. This underscores the need to learn more about the chemical composition of charcoal. The studies done with the phenolic material, in particular the hydroquinone/catechol system, shows the profound effect sulfur can have on combustion. This has led to a hypothetical mechanism explaining sulfurs role in flame spreading which should be explored in future work.

From cinematography and burning-rate curve of the phenolphthalein pyrotechnic, it was shown that this system reacts as well as black powder and has similar physical combustion characteristics to black powder.

A practical outgrowth of this work is the potential that some of these organic substrates might prove to be acceptable substitutes for charcoal in black powder. A synthetic black powder ought to have much more reproducible and uniform combustion characteristics. It is realized that such pyrotechnic mixtures must be extensively tested before they can be seriously considered. Safety tests including drop weight, card gap, friction. and electrostatic sensitivity must be performed.

[1] James E. Rose, "Investigation on Black Powder and Charcoal," IHTR 433, September 1975, Naval Ordnance Station, Indian Head, MD.

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[4] *James E. Rose, "Back Powder - A Modern Commentary," Proceedings of the 10th Symposium on Explosives and Pyrotechnics, 6A-1, 1979, Franklin Research Institute, Philadelphia, PA.*

[5] 1. Hintze, Explosivstoffe Vol. 2, p. 41, 1964.

[6] A. D. Kirshenbaum, J. Ballistic. P. 171, July 1978.

[7] E. Gray, H. March, and J. Robertson, "The influence of charcoal in the Combustion of Back Powder," RARDE, Fort Halstead, Seven Oaks, England. Presented at Basic and Applied Pyrotechnics International Conference, Arcahon, France, October 2982.

[8] R. Sasse', "Characterization of maple Charcoal Used to Make Back Powder," ARBRL-MR-03322 Ballistic Research Laboratory, USA ARRADCOM, Aberdeen Proving Ground, MD, November 1983. (AD-A136-513)

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[10] R. Sassé ', "Strand Burning Rates of Black Powder to One .Hundred Atmosphere," Proc. of the 19th JANNAF Combustion Meeting, CPIA Publication 366, Vol. 1, p. 13, October 1982. (AD-A129-087J

[11] J. D. Blackwood and F. P. Bowden, Proc. Roy. Soc.. London, A 213, 285, 1952.

[12] DSC black powder data show no preignition exotherm when the analysis is performed in an open pan. This supports the hypotheses that gaseous compounds could be involved in the reaction.

SUGAR CHARCOAL

$C_{12}H_{22}O_{11} \longrightarrow 12C + 11H_2O$

SUBMITTED By GEORGE G. MARVIN
CHECKED By HAROLD S. BOOTH AND ALBERT DOLANCE

Charcoal prepared from sugar has long been used when a pure form of carbon is desired. The preparation of this material by treatment of sugar with concentrated sulfuric acid involves long, tedious washing operations and yields a product of questionable purity. The following method has been found to give a product of high purity in a minimum of time.

Procedure

Approximately 100 to 150 g. of pure cane sugar is weighed into a 1200-ml. casserole. The casserole is placed in an electric muffle furnace heated to a temperature of about 800°. The sugar melts, then chars, and volatile products start to burn. The casserole is removed from the furnace and the burning mass is stirred with a long quartz rod to minimize frothing. When the contents cease burning, the casserole is put back into the muffle furnace and heated until the mass begins to solidify into a bulky, porous product. After no more volatile matter is evolved from the solid mass, the casserole is removed from the furnace and allowed to cool. Heating after the mass has solidified will result `only in burning off some of the charcoal to give lower yields.

After the charcoal has cooled, it may easily be removed from the casserole and ground in an agate mortar. It is then sifted to size and stored for use. The grinding of this charcoal to pass a 100-mesh screen is slow but necessary for certain types of work. A yield of about 12 g., corresponding to about 30 per cent of the theoretical, is readily obtainable.

Inorganic Syntheses Volume II 1946

Gunpowder: *The Ingredients*

Sir Edward Thorpe
A Dictionary of Chemistry
In 5 Volumes. Vol. II
Longmans, Green, and Co. London
1916

EXPLOSIVES
Gunpowder
The Ingredients

The *charcoal.* For making the charcoal for gunpowder, soft and light woods are chosen, of an average growth of from two to ten years. The wood should be out in the spring, when it is in full vegetation, as then its bark can readily be removed; in the spring the tree is in fullest sap, but the map is very watery, and contains but little salts in solution.

At Waltham Abbey, the woods used are: the alder buckthorn *(Mamntur fraisgula, L,* improperly called dogwood), alder, and willow *(Salix alba, L.).* The woods are grown in England, and we cut about four inches in diameter.

Désortiaux states that in France the so called dogwood *(Rhamnus fratigula) is* exclusively employed for making the charcoal for Military and sporting powders; he states, how. ever, that it is becoming increasingly difficult to procure, and that they are trying to replace it by willow, or by the wood of the spindle tree. In Germany, 'dogwood' (R. *frangula), willow,* and alder mused; in Russia, alder.

At Waltham Abbey, wood is usually kept for about three years, dogwood in stacks, and willow and alder piled by cords in the open. By the method of carbonisation, there followed, 25 p.c. of black charcoal should be obtained from the wood, rather more from dogwood. The wood is cut into three-feet lengths, which are split if differing much in thickness, and is packed into iron cylindrical cases called slips, 3 feet 6 inches long, and 2 feet 4 inches in diameter. The lid is fastened on, two openings (each about 4 inches in diameter) being left in the bottom of the slip. The slips are then placed in horizontal cylinders, the end of the slip with the opening going to the further end of the cylinder, in which end there are openings corresponding with those in the slips.

The cylinders we closed by tightly fitting iron doors, and are built into the wall, with furnaces underneath, so arranged as to admit of the accurate regulation of the heat throughout the operation of charring ; this occupies with dogwood about 4 hours for R.F.G., and 8 hours for R.F.G2 gunpowders. The flames surround the cylinder, the heat acting as nearly as possible on its whole surface. The gases and volatilised tar from the wood pass out through the openings in the slip and the corresponding holes in the retort, into pipes communicating with the furnace in which they are burnt; this saves a considerable amount of fuel. When the wood has been sufficiently charred, which is known by the violet colour of the flame from the burning gas, indicating the formation of carbonic oxide, the slip is withdrawn by means of tackling, placed in a large iron case ore or cooler, covered with a closely fitting lid, and allowed to remain until all the fire is extinguished, which takes about 4 hours; the charcoal is then emptied into smaller coolers, and sent to store. The charcoal is carefully picked over by hand, to ascertain that it all is properly and evenly burnt, and that no rivets from the slips have broken off. It is then kept from ten days to a fortnight in store before being ground, to obviate the danger from spontaneous combustion (caused by absorption of oxygen from the air) to which charcoal is liable when ground directly after burning.

The smaller the cylinders used, the more uniform is the composition of the charcoal produced, since so high a temperature in not needed to carry the heat to the centre of the charge; in the English powder mills, the tendency has been to use small cylinders, some of which hold . The use of small cylinders, some of which hold only 77 lbs. of wood. The use of small cylinders, however, raises the cost of production (Désortiaux).

Violette states that, for the same temperature, a slow carbonisation gives a much higher yield of charcoal than a quick carbonisation ; the percentage of carbon being also a little higher in the former can.

Instead of fixed carbonising cylinders, movable cylinders are now used in most black. powder factories. No 'slip' in used, but two cylinder are provided for each furnace, one being charged while a carbonisation is proceeding in the other. The charged cylinders are run into the furnace on rails, which support them over the fin. An elaborate arrangement for the regular distribution of the gases and products of distillation of the wood is used, by means of which they can at will be directed into any one of the furnaces or allowed partially to escape by the chimney.

The principal advantages claimed for this system are homogeneity of the charcoal, the ready regulation of the combustion of the gases by means of the distribution apparatus, and the cooling down of the charcoal out of contact with the air, which does away with the possibility of the charcoal taking fire.

In some English factories, vertical movable cylinders are used, the advantages being that a larger number can be fired at the same time, and the moving of the cylinders into the cooling room is greatly facilitated.

Böckmann mentions that some years ago the use of rotating cylinders was introduced in Sweden; the cylinders being turned 90o about their horizontal axes every half-hour during the carbonisation. It is stated that a more uniform carbonisation is obtained, and fuel saved.

Violette, in 1848, introduced the carbonisation of wood by means of superheated steam. The steam was used at a pressure of 1/2 to 1 atmosphere, and was raised to the, required temperature by being passed through a worm of wrought-iron heated by a fire. For the production (from dogwood) of *charbon roux* containing 70 p.c.

carbon, the temperature of the steam had to be about 280°; by using steam heated to about 350° charcoal containing 77 p.c. carbon was produced, and by heating both cylinder and steam to a temperature not exceeding 450o charcoal of 89 p.c. carbon was obtained. The charcoal produced by means of superheated steam is remarkably uniform in composition. The method, however, was abandoned, because it gave a larger yield of *charbon roux,* but not of black charcoal, than the ordinary method of carbonisation in cylinders ; and the lightly-burnt charcoal was then only required for sporting powder. Also, the cost of production of the charcoal by the superheated steam apparatus was greater.

Güttler, in 1887, invented a process for carbonising wood, especially cuttings and pulp, straw, peat, &c, in heated CO_2. Carbon dioxide is stated to be perferable to super, heated steam, on account of the moist state of the charcoal when cooled in the steam. Gases of combustion, as free as possible from oxygen, are actually used. A producer--furnace is arranged by the side of the charring furnace, in which the CO_2 is produced by blowing air though burning coke by means of a fan. The carbonic acid gas is then blown through a tube into the carbonising cylinder during the carbonising of the wood, &c., and the flow of CO_2, is maintained during the cooling, which in consequence of the presence of the gas may be very rapid.

Composition, &c., of the charcoal—Carbonising the wood raises the percentage of carbon, diminishing the percentages of hydrogen and oxygen.

Désortiaux states that woods recently cut have almost the same percentage the mean composition; the mean composition of the dried wood being 49-37 p.c. C 6.14 p.c. H, 43.42 1.07-p.c. ash.

Heintz gives the composition of alder wood minus ash, as 48-63 p.c. C, 5.94 p.c. H, 44.75 p.c. 0, 0.68 p.c. N

On heating, water, carbon monoxide, carbon dioxide, hydrogen, acetic acid, methyl alcohol, and tar are produced.

Violette found that dogwood was converted into slack-baked charcoal (charbon roux) at a temperature of 280°—300° ; at 300° the yield (on the small scale) was about 34 p.c. and the composition of the charcoal was 73.24 p.c. C, 4.25 H, 21.94 p.c. 0 and N, 0.57p.c. ash. Between 350° and 400°, black charcoals are produced, the yield being from 31 p.c. to 28 p.c., the composition ranging from about 77 p.c. to 81 p.c. C. Between 1000° and 1250°, the charcoal obtained was very black and hard, the yield was about 18 p.c., and the composition of the charcoal 82.0 p.c. C, 2.30 p.c. H, 14.10 p.c. 0 and N, 1.60 p.c.. ash, at the lower temperature, and 88.14 p.c. C, 1.42 p.c. H, 9.24 p.c. 0 and N, 1.20 p.c. ash at the higher temperature.

Experiments made at the Chemical Department of the War Department showed no great difference in the specific gravity of charcoals prepared from willow and alder at 394o-558o; their specific gravity at 15.6o was 1.41 to 1.44. Willow charred at the maximum temperature of 394° for 9 1/2 hours had the composition 79.22 p.c. C, 4.02 p.c. H, 15.32 p.c. 0 and N, 1.44 p.c. ash, and specific gravity (at 15.6°) 1.414.

The higher the temperature of carbonization, the less is the inflammability of the charcoal in air, and the greater the thermal conductivity. Violette states that the charcoals prepared from any kind of wood at 300° take fire when heated in the air to 360°-380°; the charcoals from light and porous woods burning more easily than those from hard and close woods. For the same wood he makes the following statement as to the relation between temperature of charring and that of inflammation in air:-

Temperature of charring	Temperature of inflammation
260°—280°	340°¯360°
290°—350°	360°—370°
432°	about 400°
1000°—1500°	600°—800°

The lightly-burnt charcoals are much more absorbent of water than those charred at a high temperature.

The charcoals used for the various kinds of Service black gunpowder range in composition from about 75 p.c., C to 86 p.c. C, according to the nature of the powder. All other conditions being the same (viz. proportion of KNO_3, S, and charcoal, size of powder, density, and moisture),the charcoal burnt at lower temperatures (having lower percentage of carbon) gives higher muzzle-velocities and pressures than charcoal burnt at higher temperatures and having higher percentage of carbon ; that is, for gunpowder of the composition 75 p.c. nitre, 10 p.c. sulphur, and 15 p.c. charcoal. The greater inflammability of the lighter-burnt charcoal makes the gunpowder, of which it forms part, quickerburning.

The wood is charred to expel moisture, which would lower the temperature of the explosion products of gunpowder, and to obtain a charcoal of suitable inflammability.

Appended is a table of the percentage composition of some charcoals from gunpowders of Waltham Abbey make, and of a sporting powder, which will serve as types of the charcoal used in the different kinds. of black gunpowder--

------	W.A. Pebble	W.A. Rifle Large Grain	W.A. Rifle Fine Grain	W.A. Fine Grain	Curtis& Harvey Sporting
C	85.26	80.32	75.72	77.88	77.36
H	2.98	3.08	3.70	3.37	3.77
O (and N)	10.16	14.75	18.84	17.60	16.62
Ash	1.60	1.85	1.74	1.15	2.25

(Noble and Abel, Phil. Trans. 1880, 171, 218)

Willow Charcoal

Again, the Ping Lu [Records of Military Art] (+ 1606) gives a theory of the substances that went to compose gunpowder:

The nature of the chemicals *(yao)* used in attack by fire is as follows. Among the principal substances saltpetre and sulphur are the princely ones, charcoal is the ministerial one, the various poisons are adjutants (tso) , and those constituents that produce *chhi* are the envoys *(Shih*). b One must know the suitability of the ingredients before one can master the wonderful (effects) of attacks with incendiaries and explosives. Now the nature of saltpetre is to be linear *(chih)*; the nature of sulphur is to radiate *(hing);* and the nature of charcoal is to take fire *(jan*) . That which is straight by nature governs impact at a great distance, so for propulsion we take nine parts of saltpetre to one part of sulphur. That which goes sideways by nature governs explosion, so for detonation we take seven parts of saltpetre to three parts of sulphur! Charcoal from green willow [Salix babylonica] is most sharp in nature, charcoal from dried fir is slow, while that from the leaves of the white mountain bamboo *(joyeh) is* particularly fiery.

Saltpeter

Foxfire 5
Eliot Wigginton Ed. Anchor Books 1979

"Saltpeter, the chemical that produces the oxygen for the other ingredients when lit off, can be made by putting urine and manure of any kind in a big cement tank mixed with water until you have **about three hundred** gallons mixed up. Then you put on a tight lid and let it sit for about ten months. You have to have a drain pipe and valve at the bottom, and a stainless steel filter screen installed beforehand or you'll have one big mess on your hands. At the end of that time, you run the liquid that drains off through ashes into shallow wooden trays lined with plastic sheeting and let them stand for evaporation in the sun. When the water evaporates, potassium nitrate crystals (saltpeter) will form in the bottom of the trays.

"In the old days in cities, most outhouses were fitted with trays or drawers under the seats that could be pulled out from behind the building. They had night-soil collectors who were paid so much every month by the outhouse owners to keep those drawers emptied, and they'd come around with a special wagon into which they dumped the contents. When the wagon was full, it was hauled out to where another fellow bought the contents and dumped it into concrete tanks where the bacteria works it just like yeast works wine or bread dough. Then the liquid was run through ashes into shallow tiled or plain concrete evaporating trays or basins to recover the saltpeter.

"Today, saltpeter can also be bought in most drug stores in bottles or cans.

▪▪▪

Another of these operations was located in Mammoth Cave. Recently, in a remarkable experiment there, potassium nitrate crystals from saltpeter were produced again in the traditional method. Carol A. Hill, one of the coordinators for the Saltpeter Research Group, describes the procedure that was used that day:

"Before the 1870S, caves were the primary source of nitrate used in the manufacture of gunpowder. Saltpeter mining was one of the first major industries of the new frontier, and one of the principle objectives of exploring new territory was to find saltpeter caves. Caves were mined by individuals and also commercially for national defense purposes during the Revolutionary War, the War of 1812, and the Civil War. Many homesteaders in the Virginias, Kentucky, and Tennessee had their own individual saltpeter caves and from them would make their own gunpowder in home-constructed V-vats or 'hoppers.'

"Making a V-vat entailed using a peg-and-hole construction. The holes were made with a hand auger (Plate 202); the pegs by whittling down the end of a log with a hatchet and then by trimming with a knife (Plate 203) . The frame was then pounded together with a wooden mallet (Plate 204). A froe was used to make the side boards. Bolts of wood that were straight-grained and well-seasoned were the best for this purpose. The glut was used as a wedge to split the log base of the collecting trough. The trough was then hewn out with a foot adze and hatchet. After the hopper was constructed, twigs were laid in the bottom of the vat, and then wheat straw was laid on top of the twigs and along the side boards to help keep the vat from leaking (Plate 206) .

"Cave dirt was tested for its nitrate potential by the following procedure: A footprint or mark was made in the dirt and left for twenty-four hours. If the print was scarcely visible by the next day, then the dirt was deemed high in niter. A mattock was used to break up the cave dirt, and a wooden saltpeter paddle was used for digging and scraping (Plate 207). The dirt was removed from the cave in gunny sacks and poured on top of the twigs and straw in the V-vat. Buckets of water were then poured over the saltpeter dirt to leach it of its nitrate or 'mother liquor.' The mother liquor (also sometimes called 'beer') would run down the sides of the V-vat and into the split-log base and out into the collecting trough (Plate 208). A dipper gourd was often used to transfer the mother liquor into a container (Plate 209). This same liquor was poured again and again over the saltpeter dirt because releaching caused more nitrates to be dissolved. According to the old reports, releaching went on until the solution was of sufficient density to float an egg.

"The next step was to combine the mother liquor rich in calcium nitrate with woodashes that contain high amounts of potassium hydroxide. The best woodashes for this purpose were made by burning hardwoods such as oak and hickory. The mother liquor was either poured directly over the woodashes or the woodashes were leached in barrels and the leachate directly combined with the mother liquor. Upon combination, a white haze could be seen (Plate 2 ~ 0), and this white precipitate (calcium hydroxide or 'curds' as it was called) would slowly sink to the bottom of the barrel. If the solution contained an excess of calcium nitrate, the product was termed 'in the grease.' An excess of woodashes produced a condition called 'in the fey.' The woodash leachate was poured into the mother liquor until the white curds could no longer be seen precipitating out of solution. The remaining solution thus contained the still soluble potassium nitrate. This solution was dipped out into an apple-butter kettle (or 'evaporator'), and a fire started under the kettle. Turnip halves were then thrown into the boiling solution to help keep it from foaming and to take up the dirty brown color. Oxblood (or alum) was also added to the boiling liquid and caused the organic matter to rise to the top of the liquid and form a scum which, with continued boiling, was constantly ladled off. After a few hours of boiling, the hot liquor was poured through cheesecloth in order to filter out the remaining scum and organic material. Upon cooling, fine, hitter, needle-shaped crystals of niter (potassium nitrate) formed in the liquor (Plate 21 ~). These crystals were then collected and dried (Plate 2 ~ 2).

Potassium-nitrate crystals were far superior to calcium or sodium-nitrate crystals because they are non-deliquescent (do not take up moisture from the air) and, hence, would not make the gunpowder wet and unusable. The nitrate crystals thus obtained had to be further refined and purified. This purification procedure was done either by the individual and homemade into gunpowder, or it was done after the saltpeter crystals were sent to a refinery where the final gunpowder was made."

SALTPETRE, NITRATE, OF POTASSA.

SALTPETRE, NITRATE, OF POTASSA.
Wagner's Chemical Technology 1872
(A translation of Rudlof Wagner's
"Handbuch der Chemischen" 8th Edition 1870)
Republished by Lindsay Publications

(KNO_3 = 101.2. In 100 parts, 46.5 Parts Potassa, and 53.5 parts nitric acid.)

SALTPETRE. This salt is to some extent a native as well as a chemical product. The well-known flocculent substance often observable on walls, especially those of stables, is composed in a great measure of nitrates; a similar phenomenon is seen in subterranean excavations, and even in many localities the surface of the soil is covered with an efflorescent saline deposit, consisting largely of nitrate of potassa. These deposits are most common in Spain, Hungary, Egypt, Hindostan, on the banks of the Ganges, in Ceylon, and in some parts of South America, as at Tacunga in the State of Ecuador; while in Chili and Peru nitrate of soda, so-called Chili saltpetre, is found in very large quantities under a layer of clay, the deposit extending over a tract of land some 150 miles in length.

OCCURRENCE OF NATIVE SALTPETRE. Although native saltpetre is met with under a variety of conditions they all agree in this particular, that the salt is formed under the influence of organic matter. As already stated, the salt covers the soil, forming an efflorescence, which increases in abundance, and which if removed has its place supplied in a short time. In this manner saltpetre or nitre as it is sometimes called, is obtained from the slimy mud deposited by the indundations of the Ganges, and in Spain from, the lixiviation of the soil, which can be afterwards devoted to the raising of corn, or arranged in saltpetre beds for the regular production of the salt. The chief and main condition of formation of saltpetre, which succeeds equally well in open fields exposed to strong sunlight, under the shade of trees in forests, or in caverns, is the presence of organic matter, v*iz.,* Humus, inducing the nitre formation by its slow combustion; the collateral conditions are dry air, little or no rain, and the presence in the soil of a weathered crystalline rock containing feldspar, the potassa of which favours the formation of the nitrate of that base. All the known localities where the formation of nitre takes place naturally, including the soil of Tacunga, formed by the weathering of trachyte and tufstone, are provided with feldspar. The nitric acid is due to the slow combustion of nitrogenous organic matter present in the humus, it having been proved that the nitric acid constantly formed in the air in enormously large quantities by the action of electricity and ozone, as evidenced by the investigations of MM. Boussingault, Millon, Zabelin, Schonbein, Froehde, Bottger, and Meissner, has nothing whatever to do with the formation of nitre in the soil, a fact also supported by Dr. Goppelsroder's discovery of the presence of a small quantity of nitrous acid in native saltpetres.

The mode of obtaining saltpetre in the countries where it is naturally formed is very simple, consisting in a process of lixiviation with water, to which frequently some potash is added for the purpose of decomposing the nitrate of lime occurring among the salts of the soil, the solution being evaporated to crystallization. Soils yielding saltpetre are termed Gay earth or Gay saltpetre. The process by which nitrate of potassa is naturally formed is imitated in the artificial heaps known as saltpetre plantations, formerly far more general than at the present time, it having been found that the importation of Indian saltpetre, and the manufacture of nitrate of potassa by conversion from nitrate of soda, are cheaper sources. Thus, saltpetre beds are to be met with only under peculiar conditions, as, for instance, in Sweden, where all landed proprietors are required to pay a portion of their taxes in saltpetre.

MODE OF OBTAINING SALTPETRE. The mode of making, these plantations may be briefly described as follows:—Materials containing, much carbonate of lime—for instance, marl, old building rubbish, ashes, road scrapings, stable refuse, or mud from canals—is mixed with nitrogenous animal matter, all kinds of refuse, and frequently with such vegetable substances as naturally contain nitrate of potassa, such as the leaves and stems of the potato, the leaves of the beet, sunflower plants, nettles, &c. These materials are arranged in heaps of a pyramidal shape to *a* height of 2 to *2 ½ metres,* care being taken to make the bottom impervious to water by a well puddled layer of clay, the heap being in all directions exposed to the action of the atmosphere, the circulation of which is promoted through the heap by of straw. The heap is protected from rain by a roof, and at least once a week watered with lant (stale urine). The formation of saltpetre of course requires a considerable length of time, but, when taught by experience, the workmen suppose a heap *"ripe",* the watering is discontinued, the salt containing saltpetre soon after efflorescing over the surface of the heap to 6 to 10 centims. in thickness; this layer is scraped off, and the operation repeated from time to time until the heap becomes decayed and has to be entirely removed. In Switzerland saltpetre is artificially made by many of the farmers, simply by causing the urine of the cattle, while in stable in the winter time, to be absorbed by a calcareous soil purposely placed under the loose flooring of the stables, which are chiefly built on the slope of the mountains, so that only the door is level with the earth outside, the rest of the building hanging over the slope, and being supported by stout wooden poles; thus a space is obtained, which, freely admitting air, is filled with marl or other suitable material. After two or three years this material is removed, lixiviated with water, mixed with caustic lime and wood ash, and boiled down. The liquor having been sufficiently evaporated, is decanted from the sediment and left for crystallization; the quantity of saltpetre varying from 50 to 200 lbs. for each stable.

TREATMENT OF THE RIPE SALTPETRE EARTH. The crude salt from the heaps is converted into potassic nitrate by the following processes: a. The earth is lixiviated with water, this operation being known as the preparation of raw lye. b. The raw lye is broken, that is to say, it is mixed with a solution of a potash salt in order to convert the nitrates of magnesia and lime present into nitrate of potassa. c. Evaporation of this liquor to obtain crude crystallized saltpetre. d. Refining the crude saltpetre.

PREPARATION OF RAW LYE. The ripe earth is lixiviated to obtain all the valuable soluble matter it being expedient to use as little water as possible in order to save fuel in the subsequent evaporation, for which the liquor is ready when it contains from 12 to 13 per cent. of soluble salts.

BREAKING UP THE RAW LYE. The raw lye sometimes known as soil water, contains the nitrates of lime, magnesia, potassa, soda, the chlorides of calcium, magnesium, and potassium ; also ammoniacal salts and organic matter of vegetable as well as of animal origin. In order to convert the nitrates of lime and magnesia into nitrate of potassa, the raw lye is broken up as it is termed, that is to say, there is added to it a solution of 1 part potassic carbonate in 2 parts water:—

Nitrate of lime, $Ca(NO_3)_2$] [Nitrate of potassa, $4KNO_3$.
Nitrate of magnesia, $Mg(NO_3)_2$] = [Carbonate of lime, $CaCO_3$.
Carbonate of potassa, $2K_2CO_3$] [Carbonate of magnesia, $MgCO_3$.

The chlorides of calcium and magnesium are also decomposed, being converted into carbonates, while chloride of potassium is formed. The addition of the solution of potassa, to the raw lye is continued as long as a precipitate is formed; in order, however, to have some approximative idea of the quantity of carbonate of potash which may be required, a test experiment is made with 1/2 litre of the raw lye.

Sometimes sulphate of potassa is used instead of the carbonate, but in that case the magnesia salts of the raw lye have first to be decomposed by milk of lime, an operation which has to be followed by the evaporation of the fluid. If, after this, sulphate of potassa is added, sulphate of lime is precipitated—

$[Ca(NO_3)_2 + K_2SO_4 = 2KNO_3 + CaSO_4]$.

When chloride of potassium is used for the decomposition of raw lye, the salts of magnesia are first removed by the addition of milk of lime; and the clear supernatant fluid having been decanted from the sediment, there is added a mixture of equal molecules of chloride of potassium and sulphate of soda, the result being the formation of gypsum, while the sodic nitrate generated exchanges with the chloride of potassium, carrying over to the latter the nitric acid, and taking up the chlorine to form common salt.

BOILING DOWN THE RAW LYE. The clarified raw lye decanted from the precipitate of the earthy carbonates consists of a solution inwhich there are present the chlorides of potassium and sodium, nitrate of potassa, carbonate of ammonia, excess of potassic carbonate, and colouring matter. The boiling down of this liquid is effected in copper cauldrons, Fig. 64, so set in the furnace as to admit of the circulation of the hot air and smoke from the fire-place, passing by c c below the heating pan, and thence by g into the chimney. In some works this waste heat is utilized in drying the saltpetre flour. As the bulk of the fluid in the cauldron decreases by evaporation, fresh lye enters by means of a pipe and tap from the pan, d. About the third day the alkaline chlorides begin to be deposited, and the workmen have then to take, great care to prevent these salts from becoming what is technically termed burnt, which might give rise to serious explosions, and for this purpose the liquid is stirred with stout wooden poles. After each stirring the loose saline matte is removed from the boiling liquid by means of perforated copper ladles. However, as a hard deposit is always formed, a peculiar arrangement exhibited in Fig. 64, consisting of a shallow vessel, m, suspended by a chain, k, and weighted with a piece of stone, is lowered into the middle of the cauldron to about 6 centims. from the bottom, the object being to catch the solid particles, which would, when aggregating, form an incrustation, previously to their reaching the bottom of the vessel; and as no ebullition takes place at m, the particles once deposited remain there, and can be readily removed by raising the dish out of the cauldron, and emptying it into a box placed over the cauldron, the bottom of the box being perforated to admit of any liquor which may have been raised with the solid salt to return again to the cauldron, The deposit thus removed consists chiefly of gypsum and carbonate of lime.

When a portion of the impurities contained in the boiling liquid have been removed, the raw lye still frequently contains some chloride of sodium, as this salt is not, as is the case with nitre, more soluble in boiling than in cold water. The abundant crystallization of the saltpetre is a sign that the lye has been sufficiently evaporated; in order, however, to prove this, a small sample is taken, and if on cooling the nitre crystallizes so that the greater part of the sample becomes a solid mass, the liquid is run into tanks and left for 5 or 6 hours, during which time impurities are deposited, and the liquid rendered quite clear. As soon as the temperature of the liquid has fallen to 60o, it is poured into copper crystallization vessel's; after a lapse of 24 hours the crystallization is complete, and the mother-liquor being separated from the salt is employed in a subsequent operation.

REFINING THE CRUDE SALTPETRE. The crude saltpetre is yellow-coloured, and contains on an average some 20 per cent. of impurities, consisting of deliquescent chlorides, earthy salts, and water. The object to be attained by the refining is the removal of these substances. At the present day a large portion of the refined saltpetre met with in commerce is obtained by the refining of the crude saltpetre imported from India. It may be noted that this importation is steadily increasing, there being, in 1860, 16,460,300 kilos., and in 1868, 33,062,000 kilos. of the salt brought to England; and, indeed, the production of saltpetre from natural sources in Europe is now limited to very few and unimportant localities.

The method of refining saltpetre is based upon the fact that nitrate of potassa is far more soluble in hot water than are the chlorides of sodium and potassium. 600 liters of water are poured into a large cauldron, and 24 cwt. of the crude saltpetre are added at a gradually increasing temperature; as soon as the solution boils, 36 cwts. more crude saltpetre are added. Supposing the crude nitre to contain 20 per cent. of alkaline chlorides, the whole of the nitre will be dissolved in this quantity of water, while a portion of the chlorides will remain undissolved even at the boiling-point. The non-dissolved salt is removed by a perforated ladle, and the scum raising to the surface of the boiling liquid by the aid of a flat strainer. The organic matter present in the solution is removed by the aid of a solution of glue—from 20 to 50 grms. of glue dissolved in 2 litres of water are taken for each hundredweight of saltpetre. In order that the saltpetre may crystallize, the quantity of water is increased to 1000 litres, and as soon as this water is added the organic matter entangled in the glue rises as a scum to the surface and is removed. The operation having progressed so far, and the liquid being rendered quite clear, it is kept at a temperature of 88o for about twelve hours, and then carefully ladled into copper crystallizing vessels, constructed with the bottom a little higher at one end than at the other. The solution would yield on cooling large crystals of saltpetre, but this is purposely prevented by keeping the liquid in motion by means of stirrers, as to produce the so-called flour of saltpetre, which is really the salt in a finely divided state. This is next transferred to wooden boxes termed wash-vessels, 10 feet long by 4 feet wide, provided with a double bottom, the inner one being perforated; between the two bottoms holes are bored through the sides of the vessel and when not required plugged with wooden pegs. Over the flour of saltpetre contained in these wooden troughs, 60 lbs. of a very concentrated solution of pure nitrate of potassa are poured, and allowed to remain for two to three hours, tile plugs being left in the holes. The plugs are then removed, the liquor run off, the holes again plug, and the operation twice repeated, first with a fresh 60 lbs., and next with 24 lbs. of the solution of nitrate of potassa, followed in each case by an equal quantity of cold water. The liquors which are run off in these operations are of course collected, the first being added to the crude saltpetre solution, while the latter, being solutions of nearly pure nitre, are again employed. The saltpetre is next dried at a gentle heat in a shallow vessel, sifted, and packed in casks.

PREPARATION ON NITRATE OF POTASSA FROM CHILE-SALTPETRE. During the last twenty years the preparation of nitrate of potassa from Chili-saltpetre has become an important branch of manufacturing industry. The product obtained by any of the following processes is called "converted-saltpetre," to distinguish it from the preceding preparation. The method of procedure may be one of the following –

The nitrate of soda is decomposed by means of chloride of potassium—

100 kilos. of sodic nitrate
87.9 kilos. of potassium chloride

yield

119.1 kilos. potassa nitrate.
68.8 kilos. common salt.

MM. Longehamp, Anthon, and Kuhlmann first suggested this mode of preparation, which is now generally used on the large scale, as the decomposition of both salts is very complete, and as the common salt as well as the saltpetre can be utilized. The chloride of potassium is obtained by the decomposition of carnallite, or by means already mentioned.

Equivalent quantities of nitrate of soda and of chloride of potassium are dissolved in water contained in a cauldron of some 4000 litres cubic capacity. As the nitrate of soda of commerce (Chili - saltpetre) does not, as regards purity, very much from 96 per cent., some 7 cwts. are usually taken, while of the chloride of potassium, which varies in purity from 60 to 90 per cent., a quantity is taken corresponding, as regards the amount of pure chloride, to the quantity of nitrate of soda. The chloride of potassium is first dissolved, the hot solution being brought to a sp. gr. = 1.2 to 1.21, next the nitrate of soda is added, and the liquid brought, while constantly heated, to a sp. gr. = 1.5. The chloride of sodium continuously deposited is removed by perforated ladles, and placed on a sloping plank so that the mother-liquor may flow back into the cauldron, care being taken to wash this salt afterwards, so as to remove all the nitrate of potassa, the washings being poured back into the cauldron. When the liquid in the cauldron has been brought to 1.5 sp. gr.—an aqueous solution of nitrate of potassa at 15o, with a sp. gr. = 1.144, contains 21.074 per cent. of that salt—the fire is extinguished, the liquid left to clear, the common salt still present carrying down all impurities, and when clear it is ladled into crystallizing vessels, which being very shallow, the crystallization is finished in twenty-four hours. The mother-liquor having been run off, the crystals are thoroughly drained and covered with water, which is left in contact with the salt for some seven to eight hours, and then run off; this operation is repeated during the next day; the mother-liquor, and washings are poured back into the cauldron at a subsequent operation.

2. Nitrate of soda is first converted into chloride of sodium by means of chloride of barium, nitrate of baryta being formed, and in its turn converted into nitrate of potassa by the aid of sulphate of potassa:—

a. 85 kilos. of nitrate of soda	yield	130.5 kilos. nitrate of baryta.
122 kilos. of chloride of barium		58.5 kilos. of common salt

b. 130.5 kilos. of nitrate of baryta	87.2 kilos. of potassic sulphate,
require for conversion into	or
nitrate of potassa	69.2 kilos. of potassic carbonate

When sulphate of potassa is used, permanent-white, barite-white, or sulphate of baryta is obtained as a by-product, while if carbonate of potassa is used, carbonate of baryta remains, and of course may be readily re-converted into chloride of barium. In order to estimate the advantages of either process, the following points must be kept in view :—a. Taking into consideration that it is profitable to convert native carbonate of baryta into chloride of barium—for instance, by exposing witherite to the hydrochloric acid fumes produced in alkali works by the decomposition of salt and to precipitate an aqueous solution with dilute sulphuric acid to obtain permanent white, it may be inferred that it will also pay to obtain it as a by-product. b. Notwithstanding the complication of this process, it is advantageous as producing a far purer nitrate of potassa.

Nitrate of soda is converted by means of potash into the nitrate of that base, pure soda being obtained as a by-product:—

85 kilos Chili-saltpetre		101.2 kilos. of potassic nitrate
69.2 kilos. carbonate of potassa	yield	53 kilos. of soda (calcined).

This mode of manufacturing saltpetre was first introduced into Germany during the Crimean War (1854-55) by M. Wöllner, of Cologne, who established works to prepare saltpetre in this way, and very soon after, during the continuance of the war, five other manufactories of potash-saltpetre had been established on this method. In 1862 the production amounted to 7,500,000 lbs. of potash-saltpetre, the carbonate of potassa required being obtained from beet-root molasses, the soda resulting as a by-product even superior to that produced by Leblanc's process.

4. Nitrate of soda being decomposed by caustic potassa yields potassic nitrate and caustic soda.

According to M. Lunge's description, this process, first suggested by MM. Landann and Gentele, afterwards modified by M. Schnitzer, and practically applied by M. Nollner, is carried on in Lancashire in the following manner:—There is added to a caustic potash lye of 1.5 sp. gr., containing about 50 per cent. of dry caustic potassa, an equivalent quantity of nitrate of soda, and the whole, after a short time, crystallized. The nitrate of potassa having been separated from the mother-liquor, that fluid, the density of which has been greatly decreased by the reaction, is by evaporation again brought to its former density, and yields on cooling another crop of crystals of potash-saltpetre. Usually there then only remains a solution containing caustic soda with saline impurities; sometimes, however, a third crop of crystals is obtained. The deposit during the evaporation is chiefly carbonate of soda derived from the chloride of sodium contained in the potassium chloride from which the caustic potassa is made, this chloride being also converted into carbonate. The small quantities of undecomposed chlorides of potassium and sodium and sulphate of lime are retained in the mother-liquor, which is evaporated to dryness and ignited, yielding a dry caustic soda of a bluish-colour. The crystallized nitrate of potassa is now carefully refined to remove all impurities to about 0.1 per cent. of chloride of sodium, converted into saltpetre-flour, and treated as already described. Notwithstanding that the various operations have been carried on in iron vessels, the salt does not contain any of this metal, nor is the colour in any way affected. The flour is dried in a room 2 metres wide by 5 metres in length, built of brick-work, similarly to the chloride of lime rooms, and having a pointed arched roof 2 metres in height. The saltpetre-flour is spread on a wooden floor, under which extends a series of hot-air pipes, keeping the temperature at 70°, and very rapidly effecting the drying.

TESTING THE SALTPETRE. If, when perfectly pure, saltpetre is carefully fused, and allowed to cool, it becomes a white mass, exhibiting a coarsely radiated fracture; even so small a quantity as 1/80th of chloride of sodium causes the fracture to appear somewhat granular; with 1/40th the centre is not at all radiated, and is less transparent; and with 1/30th the radiation is only slightly perceptible at the edges of the fracture. Nitrate of soda has the same effect. This method of testing the purity of nitre, due to M. Schwartz, is employed in Sweden, where every landowner pays a portion of his taxes in saltpetre of a specified degree of purity. A great number of methods of testing saltpetre have been suggested by various authors for the purposes of the manufacture of gunpowder, not, however, in sufficiently general use to interest the reader. Werther's test for chlorine and sulphuric acid is by solutions of the nitrates of baryta and silver; the silver solution is such that each division of the burette corresponds to 0.004 grm. of chlorine, and with the baryta solution to 0.002 grm. of sulphuric acid. According to Reich's plan, 0.5 grm. of dried and pulverized saltpetre is ignited to a dull red heat, with from 4 to 6 times its weight of pulverized quartz; the nitric acid is expelled, the loss of weight consequently indicating the quantity, the sulphates and chlorides not being decomposed at a dull red heat. If the loss = d, we have, 1.874 nitrate of potassa, or 1.574 d nitrate of soda.

QUANTITATIVE ESTIMATION OF THE NITRIC ACID SALTPETRE. This method, due to Dr. A. Wagner, is based upon the fact of that when saltpetre, or any other nitrate, is ignited, access of air being excluded, with an excess of oxide of chromium and carbonate of soda, the nitric acid oxidises the chromic oxide according to the formula $Cr2O3 + NO5 = 2CrO3 + NO2$. 76.4 parts, by weight, of oxide of chromium are oxidised to chromic acid by 54 parts of nitric acid, or of 1 of chromic oxide by 0.7068 of nitric acid. The operation is performed by taking from *0.3* to 0.4 grm. of the nitrate, mixing it intimately with 3 grms. of chromic oxide and 1 grm. of carbonate of soda, introducing this mixture into a hard German glass combustion tube, one end of which is drawn out, and a vulcanised india-rubber tube attached to it, which is made to dip for about a quarter of an inch into water, while to the other open end, by means of a cork and glass tube bent at right angles, an apparatus is fitted for the evolution of carbonic acid gas which is made to pass through the tube before igniting it, and kept passing through all the time until the tube is quite cool again after ignition. The contents of the tube are placed in warm water, and after filtration the chromic acid is estimated by Rose's method. This process of estimating nitric acid has been found to yield very accurate results.

USES OF SALTPETRE. This salt is employed for many purposes, the most important being:— 1. The manufacture of gunpowder. 2 The manufacture of sulphuric and nitric acids. 3 Glass-making, to refine the metal as it is formed. 4. As oxidant and flux in many metallurgical operations. By the ignition of 1 part of nitre and 2 of argol, in some cases refined argol (cream of tartar), "*black- flux" is* formed consisting, of an intimate mixture of carbonate of potassa and finely divided charcoal. The ignition of equal parts of saltpetre and cream of tartar gives "white flux", consisting of a mixture of carbonate of potassa and undecomposed saltpetre; both these mixtures are often used. Black flux may also be made by intimately mixing carbonate of potassa with lamp-black and white flux. 5. When mixed with common salt and some sugar in the salting and curing of meat. 6. For preparing fluxing and detonating powders. Baumes fluxing powder is a mixture of 3 parts of nitre, 1 of pulverised sulphur, and 1 of sawdust from resinous wood; if some of this mixture be placed with a small copper or silver coin in a nutshell and ignited, the coin is melted in consequence of the formation of a readily fusible metallic sulphuret, while the nutshell is not injured. Detonating powder is a mixture of 3 parts saltpetre, 2 carbonate potassa, and 1 pulverised sulphur; this powder when placed on a piece of sheet-iron, and heated over a lamp, will explode with a loud report, yielding a large volume of gas:-

Saltpetre, $6KNO_3$, Nitrogen, $6N$.
Potassic carbonate, $2K_2CO_3$, = Carbonic acid, $2CO_2$,

Sulphur, 5S, Sulphate of Potassa, $5K_2SO_4$.

7. For manure in agriculture. 8. In many pharmaceutical preparations. 9. For the preparation of Heaton steel.

SODIC NITRATE. This salt, also known as cubical saltpetre, Chili-saltpetre, nitrate of soda, $NaNO_3$, containing in 100 parts 36.47 soda, and 63.53 parts nitric acid, is found native in the district of Atacama and Tarapaca, near the port of Uquique, in Peru, in layers termed *"caleche"* or *terra "salitrosa",* 0.3 to 1.0 metre in thickness, and extending, over more than 150 miles, nearly to Copiapo, in the north of Chili. The deposit chiefly consists of the pure, dry, hard salt, and is close to the surface of the soil. It is also found in other parts of Peru mixed with sand, in some places close to the surface of the soil, in others at a depth of 2.6 meters. Valparaiso being the great exportation depot for Peru, Bolivia, and Chili, both surface and deep soil salts are met with in the trade of that important port. The unrefined Chili-saltpetre is crystalline, brown or yellow, and somewhat moist; but the salt sent to the European markets is commonly semi-refined by being dissolved in water and evaporated to dryness. The composition of a sample in 100 parts is:—

Nitrate of soda	94.03
Nitrate of soda	0.31 [?]
Chloride of sodium	1.52
Chloride of potassium	0.54
Sulphate of soda	0.92
Iodide of soda	0.29
Chloride of magnesium	0.96
Boric acid	traces
Water	1.96

Being deliquescent the salt is not employed in the manufacture of gunpowder, but may be used for blasting powder. It is largely used for the preparation of sulphuric and nitric acids; for purifying caustic soda; for making chlorine in the manufacture of bleaching; for the preparation of arseniate of soda ; in the curing of meat; glass-making; in the preparation of red-lead; in large quantities in the conversion of crude pig-iron into steel, by Hargreaves's and by Heaton's processes ; for preparing nitrate of potassa ; and for the preparation of artificial manures and composts, it being used unmixed as a manure for grain crop.

It may be from the analysis of nitrate of soda quoted above that that salt contains a small quantity of iodine, which at Tarapaca is extracted from the mother-liquor remaining from the re-crystallisation. According to M. L. Krafft the iodine amounts to 0.59 grm. in 1 kilo. of crude nitrate; 40 kilos. of iodine being prepared per day. M. Nollner thinks that the formation of the nitre deposits in Chili and other parts of South America has taken place under the influence of marine plants containing iodine. In order to give some idea of the large and increasing exportation of Chili-saltpetre, we quote from the published statistics, that in 1830, 18,700 cwts., and in 1869, 2,965,000 cwts., were shipped.

Golden Powder I

HOFFMANN-LA ROCHE INC.
PRESENTS GOLDEN POWDER

As presented at the
13th Symposium on
Explosives and Pyrotechnics
Hyatt on Hilton Head Island, SC
Dec. 2-4, 1986

Videotape Script prepared by Hoffmann-La Roche Inc. 340 Kingsland Street Nutley, NJ 07110

Fireworks displays like those you've just seen are used to celebrate important events in many parts of the world. old and young alike enjoy the spectacle and ear shattering sound of pyrotechnics. But we all know that the use of fireworks and other explosives sometimes results in accidental injury to those handling them. one of the most important considerations in the handling of any type of munitions is safety. Safety precautions extend from manufacture through shipping, storage and end use. The development of safer material for use in munitions products has been a highly desirable goal of all suppliers in this industry.

Now let's look at another group of pyrotechnic devices. They look like fireworks we have seen in the past—but with one very important difference.

The munitions for this display were made with Golden Powder, a new propellant that has a safety profile superior to black powder—with comparable power.

In addition to its safety, Golden Powder exhibits an array of unique and useful properties that makes it one of the most exciting developments in explosives since gunpowder was discovered by the Chinese thousands of years ago.

Who discovered Golden Powder?

Oddly enough it wasn't a professional chemist or engineer, but rather a sports rifleman with a lifelong interest in vintage firearms and black powder shooting. The shortcomings of black powder and other similar powders prompted him to experiment through trial and error with new chemical combinations in hopes of finding safer, non-corrosive materials with good performance characteristics. What he found was that two commonly available substances, potassium nitrate and ascorbic acid, when treated together produced a substance with unique properties never before anticipated by trained munitions experts. While originally developed for use in breech and muzzle loading small arms, the advantages of Golden Powder's properties grew with each succeeding test.

The responsibility for continued development and commercialization of Golden Powder was assumed by Golden Powder of Texas, Inc. of Dallas, Texas. Realizing that this unique product would require extensive testing in far more sophisticated chemical and ballistics laboratories, Golden Powder of Texas contacted their supplier of ascorbic acid, Hoffmann-La Roche Inc. of Nutley N.J., and proposed further joint evaluation, support and development.

Hoffmann-La Roche, the world's leading manufacturer of ascorbic acid, was interested in further extending the uses of ascorbic acid into other industries. Recognizing the potential of this innovative product, Roche and Golden Powder of Texas agreed to work together to bring Golden Powder and its potential applications to the attention of industry and to offer licenses under the related patents and technology.

An independent ballistics testing facility, H.P. White Laboratory of Maryland, was retained by Hoffmann-La Roche to conduct performance tests on Golden Powder. Test results from the laboratories of both companies confirmed Golden Powder's excellent properties and performance.

The following sequences taken at Hoffmann-La Roche in Nutley, N.J., illustrate a laboratory process for producing Golden Powder.

In the laboratory raw materials composed of potassium nitrate, ascorbic acid and water are combined in a flask, dissolved by heating, and poured into a drying pan. The pan is then heated i'n an 0 oven at 120 Celsius for approximately 3 hours. The resulting
residue is pulverized into a powder.

To obtain the granular form, the powder is placed in a Chillsonator which compacts it into larger particles. The compacted material is then milled to a specific particle size.

Overall this is a very simple and clean process. The raw materials and Golden Powder are totally water soluble. Easy cleanup by water hosing is all that is required. Unlike the typical dark and messy character of black powder plants, Golden Powder manufacturing facilities would remain bright and clean.

Moreover, as you have seen, Golden Powder can be handled safely with procedures that are unsuitable to black powder. Anyone who has worked with black powder knows it is unstable, and can be detonated by rough handling or high temperatures. This instability also makes it unsuitable for shipping via common carrier.

Golden Powder, on the other hand, has been approved for shipping via common carrier. It has been tested by the Bureau of Mines and, based on their recommendations, the Department of Transportation has classified it as a Class B explosive.

To graphically illustrate the stability of Golden Powder we have placed the material on a steel plate. our lab assistant will now strike it with a hammer. The lack of visible reaction is typical of Golden Powder's resistance to the impact of the hammer.

While Golden Powder exhibits an unusual degree of tolerance to rough handling under laboratory conditions, the usual safety precautions should, nevertheless, be taken by all personnel working with it as when working with any explosive material.

So far we have seen Golden Powder heated, milled, sieved and impacted by the force of a hammer. What happens if it is put in a press and subjected to thousands of pounds of pressure?

This machine, a Carver press, can be used to mold Golden Powder into various solid configurations. For example, it can be molded into a consumable form which has the potential to replace conventional brass or steel cartridges. The molded form can be fired as such in rifles or any suitable type of explosive device.

Another important characteristic of solid forms of Golden Powder is an even burn rate. To demonstrate this we have set up a rod of the material in a bench vise. Ignition is by a standard match. Through adjustments to the composition of the material and compaction pressures, various burn rates are possible. Notice the minimal amount of residue after burning. The major products of combustion are water, carbon dioxide, and potassium carbonate. Golden Powder, unlike black powder, has no sulfur by-products.

Let's move now out of Roche's experimental labs. Here in the rolling hills of Harford County, Maryland is the only private, unaffiliated, ballistics laboratory in the world, H.P. White.

Their engineers have set up a test rig to provide chamber pressure and velocity readings for an experimental load of Golden Powder. The firing pin is attached to the white cord. After the firing, chamber pressure was recorded at 5,600 PSI while the speed of the bullet measured 1,439 feet per second.

The chamber pressure and velocity of this and previous tests indicate more consistent performance compared to black powder. These charts graph the consistency of Golden Powder compared to Black Powder on an equal weight basis.

Another unique feature of all forms of Golden Powder is their ability to burn cleanly with minimal, non-corrosive residue. To illustrate the non-corrosive nature of Golden Powder residue we asked the engineers at H.P. White to prepare corrosion test plates for Golden Powder and black powder.

The plate on the left is black powder.

The plate on the right is Golden Powder.

Both materials are burned and the residue on the test plate is then stored for more than 3 days in a humid atmosphere.

Now let's wash off the residue on each plate with plain water.

You can see that the metal under the Golden Powder residue cleans up smooth and bright. The residue on the black powder section requires cleaning with solvents rather than water. Here the metal is pitted and stained showing the residue's corrosive action.

The non-corrosive property of Golden Powder extends to its use in many other applications. The development of several practical devices, such as fireworks and flares, is currently being pursued by Golden Powder of Texas at RTF Industries in Marshall, Texas. The following explosive displays were videotaped on the Marshall test range.

These fireworks, for example, use Golden Powder as an igniter and booster for the explosive charges.

This flare is made with compressed Golden Powder. The burn rate on the flare is adjustable depending on its use.

Black smoke "location flares" like these have numerous military and law enforcement uses.

This military "Artillery Burst Simulator" used in battle maneuvers has also been fitted with demonstration charges using Golden Powder.

The potential for Golden Powder's use in gas generators is. shown here using a prototype generator and a compressed load of Golden Powder.

A dramatic demonstration of Golden Powder's use as a propellent will be shown on the test firing range. This miniature rocket fueled with Golden Powder is mounted on a supporting cable.

Here's ignition!

This tiny, 6 inch rocket was so powerful it snapped the cable and bent the supporting rod eyebolt.

To demonstrate Golden Powder's use as an alternative to Black Powder, we are using it to fire tracer bullets, shotgun shells, and that old standby, the breech loader.

Interest has also been expressed outside the United States for additional applications of Golden Powder. This interest includes use as an igniter, or for surface treatment of solid propellants to improve or change their burning rate.

Classified as a Class B explosive, Golden Powder's versatility in handling allows it to be colored, molded, milled, extruded and pressed to any desired configuration, leading to an almost unlimited variety of applications. It also has the ability.to act as a basic compound to which additional ingredients can be added to achieve the desired properties.

The Hoffmann-La Roche Vitamins and Fine Chemicals Division has been pleased to bring you the preceding information on what is one of the most exciting new uses for ascorbic acid. If you wish further information on its manufacture and use in any application, please contact your Hoffmann-La Roche representative.

Golden Powder II

Golden Powder: A New Explosive/Propellant
Based on Ascorbic Acid

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Nutley, N.J. 07110
(2071.) 235-5000

The search for Black Powder substitutes is an old one dating back into the 19th century. In l846, nitro-cellulose was discovered and the quest to find explosives or propellants with safer and improved performance characteristics is still ongoing. Within this chain of discoveries, we should like to present a new explosive, discovered and patented by Earl F. Kurtzl, which we have developed in collaboration with Golden Powder of Texas Inc.

This powder, called "Golden Powder" because of its appearance in early experiments or of its perceived potential value, is a simple explosive made from potassium nitrate and ascorbic acid. The exact chemical composition is not known due to the transformation of most of the ascorbic acid into compounds of unknown structure, presumably polymeric in nature.

The process is a very simple one. The two compounds, ascorbic acid and potassium nitrate in a weight ratio of 38:62 are heated, in the presence of water and a small amount of potassium bicarbonate, until a "melt" is formed and the color turns golden-brown. It is then immediately cooled, broken into pieces, and crushed to a powder. It can be processed further, e.g., by compaction or molding, granulation, or any other suitable process.

U.S. Patent #4,497,676, February 5, 1985.

Laboratory Process

A typical laboratory recipe is as follows: Weigh 105.7 g of potassium nitrate, 65.2 g of ascorbic acid, 3.7 g of potassium bicarbonate, and 128.5 g of deionized water into a 250 ml Erylemenmer flask. (2) Potassium bicarbonate is added as a precaution to prevent the formation of nitric acid due to acidic impurities present in the raw materials. Agitate the slurry using a magnetic stirring bar. The temperature falls several degrees during the solid dissolution process. Heat the solution to 600C to completely dissolve the solids. When the solids are dissolved, the solution will be pale yellow.

Pour the solution into a 45 cm x 37 cm pyrex dish. Some material will crystallize out in the coo- dish but will re-dissolve Later. The solution will form a Layer 2-3 m-- thick. Place the dish into an oven preheated to 7-200C. During the first 1 1/2 hr. of heat treatment, the majority of the water is removed. The dried solid will rise to a thickness of 5-10 mm and turn from yellow to brown in color. The best performing material is heated for 3 hr. The tray is removed from the oven, covered with aluminum foil and allowed to cool to room temperature.

The heat-treated material is a brittle sponge like solid which breaks up easily when touched. As soon as cool, the solid is removed from

the tray and ground into a powder using a mortar and pestle. At this point, we have Golden Powder in its crude state. The powder is hygroscopic and care should be taken to minimize exposure to water or humid atmosphere to avoid caking.

[2.] Potassium nitrate and potassium bicarbonate are reagent-grade material. The ascorbic acid is Hoffmann-La Roche, U.S.P. grade material.

-Heat Treatment

While the water is vaporized, crystals of potassium nitrate 10-50 microns in size are formed. These crystals are visible in the final product under a scanning electron microscope (Figure 1). The photograph shows the cross-section of a typical particle. The lighter particles seem to be crystals of potassium nitrate surrounded by a matrix of ascorbic acid "polymer". Golden powder is similar to other composite propellants where the oxidizer is coated by the fuel. In this case, the oxidizer is potassium nitrate and the fuel is ascorbic acid polymer.

Figure 1
Scanning Electron Micrograph of Golden Powder (200X)

Although we do not know the reactions which take place during the heat treatment, several observations have been made. The potassium nitrate is essentially unaffected by the heating so the reacting component is ascorbic acid. During the treatment, gaseous products are given off which causes the powder to rise. These products have been identified as carbon dioxide and water and account for a weight loss of 10-12% during the heating (Figure 2). This loss is in addition to the water used to dissolve the ascorbic acid and potassium nitrate. The reaction progress can be followed by monitoring the ascorbic acid content of the powder.

Several temperatures have been used for the heat treatment ranging from 105°C to 140oC (Figure 3). As we would expect, the degradation is more rapid at higher temperatures. For convenience on a laboratory scale, we chose 120°C as our working temperature. The best powder contains 2-5% residua ascorbic acid which is produced after about 3 hr at 120^{0}C. Higher temperatures are possible with good control on the heating time and temperature. Overheating of the powder results in the formation of carbon and a decrease in performance and safety.

The physical properties of golden powder are summarized below in Table I. Golden Powder has several advantageous properties as a propellant. It can be molded without any binders into a solid fuel for use as consumable cartridges. The heat of combustion is 5% higher and the gas volume produced is 10% greater than an equivalent amount of black powder. The residue on ignition is only 28% compared to about 50% using black powder. In addition, the residue from burning golden powder is water soluble, unlike many other propellants.

Table I

Physical Properties of Golden Powder and Black Powder

	Golden Powder	Black Powder
color	Golden to medium brown	Black
Bulk Density (20-50 mesh)	.88-.90 gm/cc	
Heat of Combustion	718 cal/gm	684 cal/gm)
Gas Volume on Combustion	298 cc/gm	271 cal/gm
Residue on Combustion	28% (H_2O soluble)	50%
Ignition Temperature	333° C	313°C (4)

4. Initiation temperature from differential calorimentry on Gearhart-Owen Industries Superfine, FFFG Black Powder.

Using differential scanning calorimetry, the ignition temperature of golden powder was determined to be 333°C (Figure 4). The ignition temperature is 20^{0}C higher than that measured for black powder in the same equipment. Scanning calorimetric studies show a two-stage exotherm over a temperature range of 333°C to 455°C.

Ballistic Performance

Although Golden Powder offers a wide spectrum of applications, one area which has attracted the attention of end users is its use as a black powder substitute. Golden Powder can be easily granulated to any grade of gun powder. The crude powder can be compacted to pellets or sheets which can be milled to appropriate grain sizes. We have made granulation's of golden powder which pass through a 20-mesh screen but are retained on a 40-mesh screen. This material was tested ballistically in a .45 caliber, 32 inch rifled test barrel. Muzzle velocities where measured using lumiline screens and the peak pressures measured using lead crushers. The ballistic data from three separate lots or golden powder are summarized in Table 2.

Table 2

Ballistic Performance of Golden Powder

60 Grain loading in 32 inch, 45 caliber, 138, grain, Hornady #6060 lead balls and Connecticut Valley Arms #11 percussion

Golden Powder Lot 5 Shot Average

	Muzzle Velocity (ft./sec)	Peak Chamber Pressure (LUP,)
1	1,363	5,300
2	1,375	5,000
3	1,383	5,400
Range (3 Lots)	1,330—1,410	4,600-5,500
SD (3 Lots)	20.2	230

These muzzle velocities are comparable to black powder at significantly lower chamber pressures. The ballistic results are extremely reproducible from shot to shot and from lot to lot. The standard deviation of velocities over the fifteen shots was only 20.2 ft./sec. and the standard deviation of peak pressures was 230 LUP.

As we stated earlier, the best performing material was powder in which the ascorbic acid has been reacted to a residual level of 2-5%. The ballistic performance of golden powder has been measured as a function of the ascorbic assay (Figure 5). The muzzle velocity of the powder, which has a residual ascorbic acid assay -less than 5%, is double that of powder which has an ascorbic acid assay greater than 30%.

Safety

One of the advantageous properties of golden powder is its safety. Unlike black powder, golden powder can be shipped as a flammable solid following the recommendation of the Bureau of Mines. They recommend a DOT classification as a Class B Explosive. The Bureau of Mines testing included thermal stability at 75oC for 48 hours during which golden powder was stable. No detonation of golden powder occurred during the blasting cap sensitivity test, the package burn test, and the squib test. Golden powder did not ignite on the Association of American Railroads Bureau of Explosives strip friction test in 10 out of 70 trials under 500 psig, which is equivalent to 100 pounds of friction force.

Summary

Golden Powder is a new explosive product based on ascorbic acid. Its combustion characteristics are comparable to black powder but with several other distinct advantages. Golden powder is safer to handle and transport. It forms about half the residue as black powder when burned. The residue formed is non-corrosive and is water-soluble. Golden powder is easily molded into solid fuel elements which burn at a well controlled rate. When used as gunpowder, the performance is comparable to black powder but is significantly more reproducible. The inherent safety of' the powder allows its shipment as a flammable solid by common carrier. With these characteristics, golden powder is a product with many potential applications.

Golden Powder III

Golden Powder' is a new energetic material derived from ascorbic acid and potassium nitrate, with combustion characteristics that offer distinct advantages over black powder.

As you'll see from the test results on the following pages, Golden Powder is safer to handle and transport than black powder. Compared to black powder it forms considerably less residue when burned, and that residue is both water-soluble and non-corrosive. Golden Powder itself is water-soluble and directly compressible - easily molded into solid fuel elements that burn at a well-controlled rate.

As a black powder substitute, Golden Powder can be easily granulated to any grade or mesh size of gunpowder. The crude powder itself can be compacted into pellets or sheets which can be milled to appropriate granular sizes.

While comparable to black powder in combustion performance, Golden Powder is more reproducible. That higher degree of consistency results in a more reliable material with more dependable results.

Initially, Golden Powder will be used primarily as a propellant in firearms ammunition (i.e., in breech and muzzle-loading rifles). As an igniter, propellant, booster or gas generator, Golden Powder's future applications are almost unlimited.

The physical properties of Golden Powder offer distinct advantages as a propellant:

- It can be directly compressed into a solid fuel for use as consumable cartridges or in delay fuses.
- Levels Of C02 and CO, two of the combustion products of Golden Powder, can be altered in formulation for specific applications.
- Golden Powder is water-soluble.
- The residue from burning Golden Powder is also water-soluble.
- Both Golden Powder and its residue are noncorrosive.

The following table highlights other advantages of Golden Powder over black powder.

Using differential scanning calorimetry, the ignition temperature of Golden Powder was determined to be 330ºC (FIG. 5), or 20ºC higher than black powder (FIG. 6). Scanning calorimetric studies show a two-stage exotherm over a temperature range of 330ºC to 455ºC.

Perhaps the most compelling advantage of Golden Powder as an energetic material is its superior safety.

Based on the recommendation of the Bureau of Mines, the DOT has assigned Golden Powder the classification of a Class B explosive. That means that unlike black powder, Golden Powder can be shipped as a flammable solid.

The Bureau of Mines testing included thermal stability at 750C for 48 hours during which Golden Powder was stable. No detonation of Golden Powder occurred during the blasting cap sensitivity test, the package burn test, and the squib test. (FIG. 7)

Golden Powder did not ignite on the Association of American Railroads Bureau of Explosives strip friction test in 10 out of 10 trials under 500 psig, which is equivalent to 100 pounds of friction force. (FIG. 8)

Although comparable to black powder on a per shot basis, Golden Powder is highly superior in consistency of performance and burn rate.

In closed bomb ballistic pressure tests, Golden Powder exhibits almost identical results as those obtained with black powder. (FIG. 11)

Ballistic performance tests show that shot-to-shot results are extremely reproducible - the standard deviation of velocities over fifteen shots was only 20.2 ft./sec., and the-standard deviation of peak pressure was 230 LUP. (FIG. 12)

Muzzle velocities were measured using lumiline screens and peak pressures were measured using lead crushers.

FIG. 12 - BALLISTIC PERFORMANCE OF GOLDEN POWDER

60 grain loading in 32 inch, 45 caliber, 138 grain, Hornady #6060 lead balls and Connecticut Valley Arms #11 percussion caps.

FIG. 1 - PHYSICAL PROPERTIES OF GOLDEN POWDER AND BLACK POWDER

	I Golden Powder	Black Powder
Color	--Golden to brown	Black
Density	-1.6-1.7 g/cc	1.75-1.8 g/cc
Bulk Density.	88-.90 g/cc (20-50 mesh)	1.05 g/cc (FFFG)
Heat of Combustion	718 cal/gm	684 cal/gm (1)
Gas Volume of Combustion	300 cc/gm	271 cc/gm
Residue on Combustion	28% (water-soluble)	42% (not Completely
Combustion Products	C02, CO, N2, H20, K2CO3 K2SO4,	C02, CO, N2, H2S, K2S, unreacted carbon

The following charts illustrate Golden Powder's stability.

FIG. 2

TEMP	WEIGHT LOSS OVER	
	2 days	28 days

60°C	0.1%	0.5%
75°C	0.4%	2.1%

FIG. 3 - HOLLAND TEST

TEMP	WEIGHT LOSS OVER 8-72 hrs.
110oC	0.5-0.6%

FIG. 4 - BERGMANN-JUNK TEST

TEMP	WEIGHT LOSS OVER 2 hrs.
132oC	0.2%

FIG. 7 - BUREAU OF MINES TESTING
(DEPARTMENT OF INTERIOR)

Blasting Cap Sensitivity Test
No detonation

Impact Sensitivity Test
No explosion in ten trials under 10 inch drop

Package Burn Test
No detonation

Squib Test
No detonation

Fig, 8 FRICTION SENSITIVITY TEST

American Association of Railroads Bureau of Explosives
Friction Test (100 lbs)
No ignition

BAM Test (36 kg maxium)
No ignition

FIG. 9 — IMPACT SENSITIVITY

BAM Test (2kg Ball) 0.95 kgm

FIG. 10 - ELECTROSTATIC SENSITIVITY (AAR-BOE) (at 5,000 volts)

	Positive
Golden Powder	0.503 Joules

Black Powder	0.057 Joules

Figure 12 BALISTIC PERFORMANCE OF GOLDEN POWDER

GOLDEN POWDER LOT	MUZZLE VELOCITY 5 shot mean (FT/SEC.)	PEAK CHAMBER PRESSURE(LUP)
1	1,363	5,300
2	1,375	5,000
3	1,383	5,400
Range (15 shots)	1,330-1,410	4,600-5,500
Standard Deviation (15 shots)	20.2	230

Golden Powder can be obtained from Golden Powder of Texas, Inc.

Golden Powder is a registered trademark of *Golden Powder* of *Texas, Inc., 8300 Douglas, Dallas, Texas 75225*

On Black Powder

CHAO HSUEH-MIN(1736-1796) ON BLACK POWDER

TABOOS

The compounding must not be done in a family which is in mourning. It is especially prohibited in the house where a funeral has been held or where a man has died, for there the misfortune of accidental fire is certain to happen. In case the mourning is for someone outside of the immediate family, and in case the family wishes to buy powder and must use it, a piece of red silk-cloth may be hung in the compounding room to release (the family) from the prohibition of using powder. In a house where fireworks are being made, one must not burn ts'an sha_or bamboo leaves lest by this means the essence of the saltpeter is weakened. During the packing of powder, if a drum is beaten to strike power into the powder, the fire flowers will be brighter. However, during the compounding, the sound of a drum must not be heard lest the powder in consequence acquire the defect of bursting. The ashes on the charcoal must be removed before use. If a charcoal with adhering ashes is used, the resulting powder will usually be impeded. Probably the ashes are the ghosts of charcoal and the charcoal is afraid of them.

Women are not allowed to handle the powder. If the powder is packed by a woman, the crackers will change into fountains and vice versa. Smoking is forbidden in the powder room. The room should be kept quiet and neat, and noisy talk forbidden in order that the soul of the powder may be soothed. Care must be taken to prevent any changes in the powder. The testing of powder must not be carried out any place near the powder house. The filling of the cylinders must not be done near any fire or smoke.

The apparatus for handling the powder must be closed tightly, and the access of wind must be prevented. After long standing in the wind, the powder takes fire spontaneously. Artifices after being loaded with powder, must not be heated again (for drying), for there is danger that the powder may show its behavior spontaneously after long continued warming. The tamping or pounding of the powder must be neither too heavy nor too light, and the amount of the powder may not freely be increased or decreased. The packing of powder by lamplight is not permissible. The opening of the powder container on a rainy day is not permissible. Those who hold established formulas will be limited by them; who understands elemental changes?

Davis, T. L. and Chao Yun-Ts'ung. Chao Hsuenh-Mine Outline of Pyrotechnics a Contribution to the History of Fireworks. Proceedings of the American Academy of Arts and Sciences. 75 (4) 95-107, May 1943.

JOSEPH NEEDHAM ON BLACK POWDER

Socially, the contrast with China is particularly noteworthy. While gunpowder blew up Western military aristocratic feudalism, the basic structure of China bureaucratic feudalism after five centuries or so of gunpowder weapons remained just about the same as it had been before the invention had taken place. The birth of chemical warfare had occurred, we may say, in the T'ang, [+ 644] but it did not find wide military use before the Wu Tai [+10th century] and the Sung, [13th century] and its real proving grounds were the wars between the Sung Empire, the Chin Tartars and the Mongols in the 12th and 13th centuries. There are plenty of examples of its use by the forces of agrarian rebellions and it was employed at sea as well as on land, in seige warfare no less than in the field; but as there were no heavily armored knightly cavalry in China, nor any aristocratic or manorial feudal castles, the new weapon simply supplemented those which had been in use before, and produced no perceptible effect upon the age-old civil and military bureaucratic apparatus, which each new foreign conqueror had to take over and use in his turn.

Needham, Joseph. Science in Traditional China: A Comparative Perspective. Chapter II - The Epic of Gunpowder and Firearms, Developing from Alchemy. Harvard University Press. 1981.

DAVID R. DILLEHAY ON BLACK POWDER -1978.

Subtle changes in raw materials or even in component parts can creep into the system and result in rejects or hazardous items. Sometimes the tolerance on a parameter is at fault. Sometimes it is a change that is not even covered in the specification. Designers and users both should be alert to changes in materials or components that can result from improvements in technology, cost-saving shortcuts by a vendor, environmental requirements (causing process modifications), or even changes in raw material sources. Many examples can be cited where only one vendor's product can meet performance requirements although no discernible difference exists from raw material acceptance tests. These instances retard advancement of pyrotechnics to a science and foster the "black magic" image we would like to shed.

David Dillehay Signal Propellant Evaluation. Sixth International Pyrotechnics Seminar. 1978.

Donald J Haarmann
First published in the *PGII Bulletin* #55 March, 1987

Big Fart

Pétards (ou artifices) pour signaux

"Used during WWI to attract the attention of neighboring troop units or fortifications. The devices consist of boxes filled with about 400kgs of black powder. They produce a report which could be heard for a distance of 3km, and the smoke produced gave the location of the signaling unit."

J Pepin Lehalleur, "Traité des Poudres, Explosifs et Artifices," Balliére et Fils, Paris 1935: In PATR-2700

The Prince of Parma Pissed

William's [The Protestant William the silent, killed with poisoned bullets by the Roman Catholic Blathazar Gérard.] most outstanding enemy was Alexander Farnese, Prince of Parma, governor-general of the Spanish Netherlands. Here was a splendid brute, and against his life, too, sundry attempts were made, though none succeeded.

It was a bridge Parma built that brought about the biggest explosion tile world ever had known. This bridge was over the Scheldt just below Antwerp. It was Parma's idea that control of the Scheldt would make easy the reconquering of all the Low Country provinces that had been inherited by his kingly uncle, Philip of Spain, and with this in mind he had laid Siege to Antwerp. If Antwerp fell, Parma reasoned, so would Protestantism. By force or fraud he had already taken or at least invested the other river or near-river cities farther inland, Ghent, Dendermonde, Mechlin, Brussels. But Antwerp was the key of the campaign, and Antwerp was near the sea. And the Scheldt was broad. And the Zeelanders of the coast were amphibious creatures to whom smuggling was an art.

So Parma dedicated the siege to the Virgin Mary, and he started to build his bridge.

The result was a masterpiece of military engineering, a feat that everybody, always excepting Parma himself, had said could not be done, But it was done. The Scheldt was bridged, chiefly by well-protected boats, and there was a fort at each end. And even the Zeelanders couldn't sneak through. And the folks in Antwerp began to get hungry.

The obvious action for the Dutch to take was fireboats. The Spaniards expected this, and had prepared against it. What they had nor expected were Giambelli's "hell ships."

Gianibelli was a crafty Mantuan who once had offered his services to Philip II and been snubbed. He was not interested in the cause of the States General. He was interested in getting his dreams financed; and the siege of Antwerp, and the Prince of Parma's bridge, made up his opportunity.

The politicians, as politicians will, had whittled down his dream, denying him the funds he demanded, but even so he was allotted a fleet of some thirty small fireboats and two ships, one of seventy, the other of eighty tons. These were appropriately christened *Fortune* and *Hope*. The fireboats were to be a feint, an advance guard that might lull the Spaniards' alertness by its very conventionality. It was with *Hope* and *Fortune* that Giambelli would make his real move.

He stripped them and lined the hold of each with a flooring of brick and mortar one foot thick and five feet wide. Upon this was built a chamber of marble mason-work forty feet long, three and a half feet broad, three and a half feet high. This constituted the crater-for Gianibelli was preparing nothing less than a couple of floating volcanoes.

Into this crater was put seven thousand pounds of gunpowder-and it was good gunpowder, for Giambelli himself had made it. This was roofed with six feet of blue marble slabs, over which was a cone or pyramid of other marble slabs, which cone was filled with millstones, ax hafts, scythe blades, iron hooks, plowshares, cannonballs, and anything else heavy that could be found.

Fortune was equipped with a slow-burning match, carefully timed. *Hope* had an invention of Gianibelli's, a clockwork detonating device, probably the first in history.

On the night of April 5, 1585, a dark night, these two nautical infernal machines, preceded by the fleet of fireboats, were sent downriver toward Parma's bridge.

The "hell ships" were equipped as well with a deck cargo of the customary combustibles-tar, pitch, oil-smeared shavings, and the like. The pilot of each would ignite this just before he slipped over the stem into his rowboat for escape.

The idea was to make them seem standard fireboats, if somewhat larger than was usual.

It worked. The Spaniards, suspecting nothing, swarmed aboard the *Fortune* the moment she hit, and started to stamp out the flames, a task for which they had been trained.

Something went wrong. There was an explosion below, but it was a small one. It frightened a few of the men, but they kept on working. It did no real damage. It was not enough to deter other men from pouring aboard the *Hope,* which hit the bridge at this moment.

Then the *Hope* let go, in full.

Nothing like it ever had been known. Men dropped dead without any wounds, sheerly from concussion. Men were thrown hundreds of feet into the air. The ship itself simply disappeared-as did a good section of the bridge. Windows were smashed in Antwerp three-four miles away. Parma lost many staff officers, some of the best lieutenants he had, and about a thousand seasoned troopers.

He himself was thrown on his face, a shoulder smashed, and for a little while it was thought that he was dead; but he got up, drawing his sword, shouting orders.

Had that explosion been followed by an immediate naval attack, as the plan called for, there is not the slightest doubt that Parma would have lost his bridge, his army as well, perhaps his life. But the Dutch admiral was criminally slow. Nothing was ready. And by the time the attack did come, hours later, well after dawn, Parma, that whirlwind of energy, was ready for it; and it was beaten off. The bridge was saved. Giambelli was disgusted.

When the Prince of Parma did ride into Antwerp, some months later, a conqueror, there had been a plot to kill him and everybody near him-by blowing up a street over which it was calculated he would be sure to pass. Nothing came of this, for it was revealed before the entry; that it gave Robert Catesby, some years later in London the first faint glimmerings of his own plan to blow up the houses of Parliament, no less, complete with King, Queen, Prince of Wales, and all the ministers and peers of the realm.

Donald Barr Chidsey
Goodby to Gunpowder
Crown Publishing NY 1963

"CIA BLACK POWDER" REVISITED

Donald J Haarmann — Is the WiZ
American Fireworks News
No. 35 August 1984

With the sale by Desert Publications of their "CIA Field Expedient Preparation of Black Powders" booklet many pyro's rushed out to their local stores and purchased potato ricers, and isopropyl alcohol by the gallon. Soon after the little woman had left the house, they proceeded to produce the damnedest mess seen in a pyro's kitchen in quite a while, along with black powder of varying qualities.

However, all is not lost as H.W. Voigt and D.S. Downs at the Seventh International Pyrotechnics Seminar presented a paper dealing with black powder igniter pills produced in part with black powder obtained using a modification of the CIA method. Their paper contained several interesting revelations, the first being an early attempt at producing black powder using a "salting out" method [aka, the precipitation method] by one Edward Greene [USP 160,053] of New York, N.Y., January 25, .1875! Greene's method consisted of mixing the sulphur and charcoal in a saturated solution of potassium nitrate, as close to the boiling point of water as practical, and then removing the excess water by connecting the mixing vessel to a vacuum, with constant stirring. [The boiling point of water at 760mm of mercury (atmospheric pressure) is 100° C, however, if the pressure is lowered to say 100mm of mercury the boiling point of water is lowered to only 52° C, therefore a great deal of water can be removed rapidly (flash evaporation) resulting in the "salting out" of the potassium nitrate.] No doubt due to the difficulties in producing the required vacuum, and for other more technical reasons this method was never used.

The second revelation is the fact that although generally credited to the CIA, the production of black powder through the use of alcohol as a dehydrating agent was developed at Frankford Arsenal, by T.J. Hennessy. ("Field Expedient Preparation of Black Powders", Frankford Arsenal Memorandum Report M67-16-1, February 1967.) Now you wife has someone to blame for the mess in her kitchen! If you want to get her off your back have her put the potato ricer to its intended use, *Kartoffelkloesse* which are a lot more difficult to make well than black powder! [Remind me to tell you the story of The WiZ's adventures in making Sauerbraten some time!]

The method they use differed from the "CIA" process in a number of important ways: Whereas the CIA method added alcohol to a mixture of sulphur, charcoal, and potassium nitrate in hot water, Voigt and Downs method mixes the sulphur and activated carbon black (in place of charcoal) in alcohol, along with two other ingredients, and then to the mixture is added the potassium nitrate dissolved in hot water.

The Details provided by them are as follows:

45 grams of K nitrate was dissolved in 45 ml. of water at about 75 C. 2.5 grams of K nitrate were added to compensate f or loss in the filtrate. [A loss of less then 6% as compared with a loss of over 18% for the CIA method.]

6.24 grams of commercial flowers of sulfur [most pyro's do NOT use flowers of sulphur due to the possibility of its containing free acid, so normal pyro grade sulphur should be used.] and 8.76 grams of activated carbon black [not lamp black] were suspended with vigorous agitation in a solution of 0.135 gram of polyvinyl pyrrolidone [a dispersing agent - try wetting sulphur some time!] and 0.6 grams of mercaptan terminated polyacrylic liquid polymer [B.F. Goodrich Co. Hycar MTA - a binding agent, don't worry, you can leave it out] in 135 ml. of 95% ethanol. [Isopropyl alcohol is cheaper and just as good.]

"The al[c]oholic suspension of the fuel components was cooled to 15° C., after which the hot aqueous KNO_3 solution was introduced gradually with vigorous agitation whereby the KNO_3 was precipitated in the form of very fine particles intimately mixed with fuel components." The resulting product was then washed with alcohol and dried.

The process was also tried: 1/ using channel carbon black, and NO Hycar MTA; 2/ using wood charcoal that was ball milled, and NO Hycar MTA; 3/ using maple wood charcoal, colloidal sulphur, and NO Hycar MTA; 4/ and using a 50/50 mixture of maple wood charcoal, and carbon black powder, WITH Hycar MTA. All of these methods produced black powder equal to the standard DuPont [Goex?] black powder when tested in a "Closed Bomb."

At a $1.85 or so a pound for commercial black powder all this would seem a lot of work for little gain, and I would be remiss if I failed to mention that for better or for worse, black powder is listed as an explosive in 18 USC section 841(c), and therefore you would be in effect manufacturing an explosive material. DJH