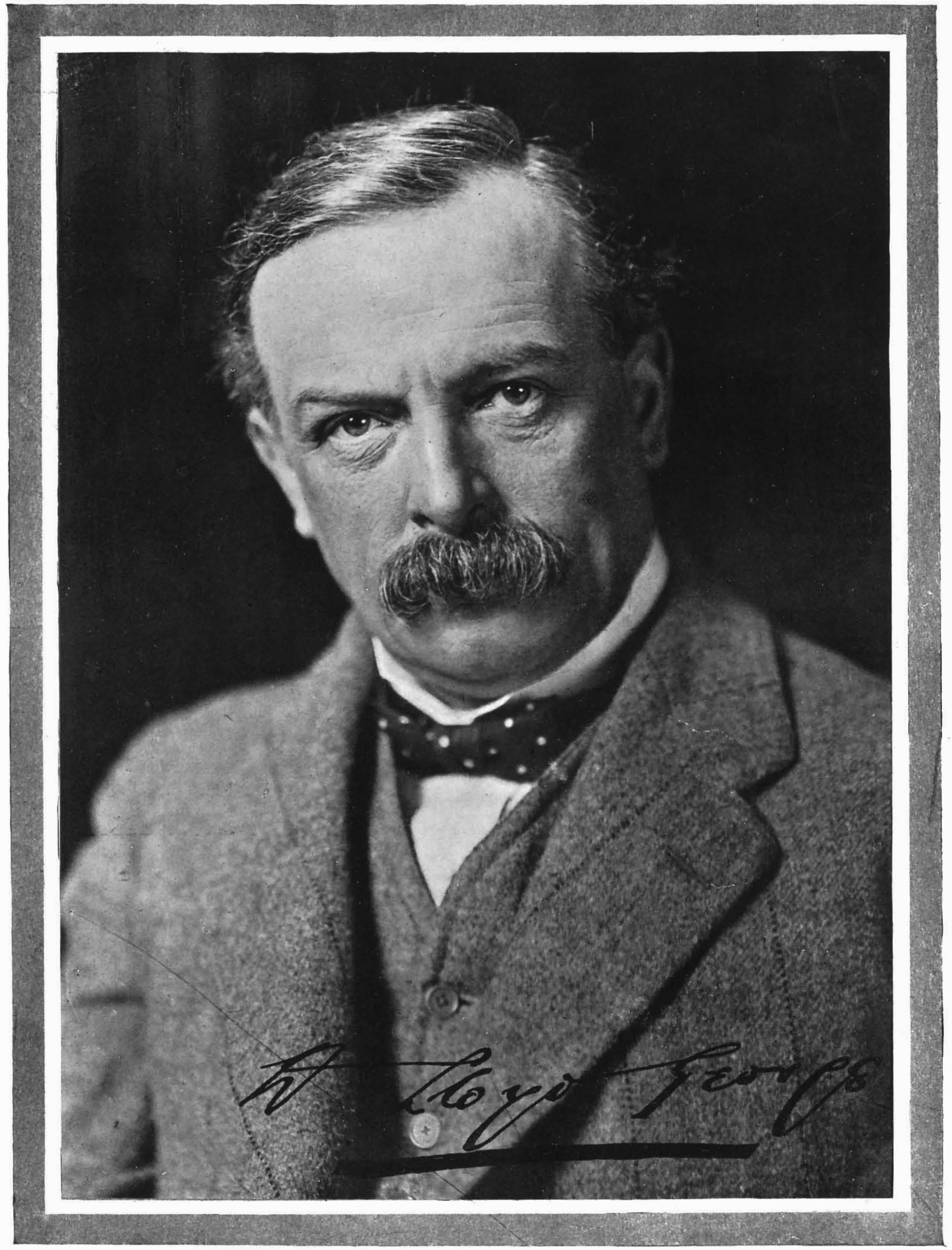

(Photo by E. H. Mills.)

EXPLOSIVES

BY

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SECOND EDITION

Vol. I
HISTORY AND MANUFACTURE

WITH 77 ILLUSTRATIONS

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DEDICATED

By Permission

To the Right Honourable

DAVID LLOYD GEORGE, M.P.

PRIME MINISTER

who during the great European War also served

his King and Country as

Chancellor of the Exchequer

Minister of Munitions

and

Secretary of State for War

"*Arma Virumque Cano*"

Virgil.

PREFACE TO SECOND EDITION

THE fact that a second edition of this work has been called for only a year after the publication of the first indicates that it was really wanted. The Great War has meantime completed the second year of its course, but has not caused the introduction of any very novel explosives, despite sensational statements of some journalists. Certain aspects of the manufacture of explosives have, however, become of greater importance, and have therefore been treated in greater detail in this edition. Picric acid, trinitrotoluene and other nitro-aromatic compounds were formerly merely by-products of the dye industry, and consequently their manufacture seemed only to call for brief notice in a work on explosives. Now, however, they are being made on a very large scale in factories specially erected, and their supply has become a matter of national importance in every country in Europe. Before the War nitric acid made from the air could hardly anywhere compete with that manufactured from sodium nitrate, but the blockade of Germany has altered this. Thus does history repeat herself, for in the Napoleonic wars England cut off the supply of potassium nitrate from India to France and caused a great development of the French saltpetre industry. The blockade has also caused Germany to pay more attention to chlorate and perchlorate explosives and those made with liquid oxygen.

The publication of *Les Poudres et Explosifs*, by L. Vennin and G. Chesneau, has enabled me to improve the description of French explosives and methods. As in the first edition, but little space has been given to explosive substances that have not any commercial, military or theoretical importance. A systematic account of all classes of explosives, organic and inorganic, will be found in the work of Vennin and Chesneau just mentioned.

I have spared no trouble to make the work as reliable and useful as possible to those engaged in making and dealing with the explosives used in this titantic struggle, and I hope that in this way I have assisted slightly, in spite of the fact that I am detained far from the principal theatres of war. There are of course some matters in connexion with explosives which cannot be published.

My best thanks are given to my former fellow student, G. C. Jones, for very kindly undertaking the revision of the proofs and the preparation of the index, thus not only relieving me of much work, but also greatly expediting the publication of this edition. My former colleague, William Barbour, has made a number of useful suggestions and supplied me with copies of some papers which I could not otherwise have obtained in time.

A. MARSHALL.

NAINI TAL, INDIA.
February, 1917.

PREFACE TO FIRST EDITION

Since the late Mr. Oscar Guttmann published his work on the *Manufacture of Explosives* in 1895 no comprehensive book on this subject has appeared in English. In the interval the explosives industry has undergone many changes: every branch of it has developed enormously—even that of black powder; and scientific investigations have thrown light on many of the problems that arise in the manufacture and use of explosives. Especially during the last few years many obscure points have been cleared up. It is hoped therefore that the present work will be found to supply a real want.

In a single book of moderate size it is not possible to treat in detail every point that arises in connexion with explosives. Consequently it has been necessary to restrict its scope in some directions. The methods of using explosives belong rather to the subjects of ballistics, blasting, etc., and their full discussion would alone require a larger work than this. Therefore they have only been referred to briefly. Details of manufacture, although often of much practical importance, can only be learnt properly in the factory: consequently they have been omitted in many cases. Proposals made in patent specifications have not been dealt with unless they possess practical or theoretical importance; for more detailed information concerning patents relating to explosives the reader should refer to works such as those of R. Escales. Subjects which are treated fully in the ordinary scientific or technical textbooks have only been dealt with in so far as they throw new light on problems connected with explosives.

On the other hand, an endeavour has been made to increase the usefulness of the book by collecting allied facts from scattered sources, and placing them in juxtaposition with one another. Some subjects, which are only mentioned briefly, or not at all, in other books have been treated more fully than their intrinsic importance would otherwise have called for. Numerous references to original papers, etc., have been given to assist those who require more detailed information concerning the subjects dealt with. Considerable space has been given to matters connected with the difficult and intricate question of the stability of nitro-cellulose and allied compounds.

I am indebted to my wife for her valued help in revising the book. My thanks are also due to Mr. W. Rintoul, Mr. J. Thorburn, and Mr. W. R. Moore for assistance in revising the proofs.

It is my earnest hope that the book may be of help to my country in the present time of emergency.

A. MARSHALL.

Naini Tal, India.

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LIST OF PRINCIPAL ABBREVIATIONS

JOURNALS, ETC.

A. and E.	*Arms and Explosives.*
Ang.	*Zeitschrift für angewandte Chemie.*
A.R.	*Annual Reports of H.M. Inspectors of Explosives.*
Ber.	*Berichte of the German Chemical Society.*
Bull.	*Bulletin of U.S. Bureau of Mines.*
Chem. Ind.	*Die chemische Industrie.*
Chem. Trade. J.	*Chemical Trade Journal.*
Compt. Rend.	*Comptes Rendus.*
C.Z.	*Chemiker-Zeitung.*
J. Soc. Chem. Ind.	*Journal of the Society of Chemical Industry.*
P. et S.	*Mémorial des Poudres et Salpêtres.*
Phil. Trans.	*Philosophical Transactions of the Royal Society.*
Proc. R.S.	*Proceedings of the Royal Society.*
S.R.	*Special Reports of H.M. Inspectors of Explosives.*
S.S.	*Zeitschrift für das gesamte Schies- und Sprengstoffwesen*
Trans. Chem. Soc.	*Transactions of the Chemical Society.*

BOOKS

Chalon.	*Les Explosifs Modernes.*
Cundill and Thomson.	*Dictionary of Explosives.*
Hime.	*Gunpowder and Ammunition*, by Lieut.-Colonel Hime.
Manufacture.	*The Manufacture of Explosives*, by O. Guttmann.
Monumenta.	*Monumenta Pulveris Pyrii*, by O. Guttmann.
Twenty Years' Progress.	*Twenty Years' Progress in Explosives*, by O. Guttmann.
Rise and Progress.	*The Rise and Progress of the British Explosives Industry.*
Worden.	*The Nitro-cellulose Industry*, by Worden.
Zschokke.	*Militarische Spengtecknik*, by B. Zschokke.
Vennin and Chesneau.	*Les Poudres et Explosifs*, 1914.

OTHER ABBREVIATIONS

b.p.	boiling-point.	G/c.	guncotton.
c.c.	cubic centimetres.	m.p.	melting-point.
coll. cot.	collodion cotton.	N/c.	nitro-cellulose.
D/n/g.	dinitroglycerine.	N/g.	nitro-glycerine.
D/n/t.	dinitrotoluene.	sp. gr.	specific gravity.
g.	grammes.	T/n/t.	trinitrotoluene.

Temperatures are always in degrees Centigrade unless otherwise stated.

INTRODUCTION

Explosion : Explosive : Gas Evolution : Heat Liberation : Sensitiveness : Constituents of Explosives : Oxygen Carriers : Combustible Constituents : Nitroaromatic Compounds : Nitric Esters : Smokeless Powders : Endothermic Compounds : Velocity of Explosion : Incomplete Detonation : Stability : Summary

Explosion.

WHEN gas or vapour is released so suddenly as to cause a loud noise an explosion is said to occur, as, for instance, the explosion of a steam boiler or a cylinder of compressed gas. Great and increasing use is made of explosive processes in gas, petrol, and oil engines for driving machinery of all kinds. In these engines the material that explodes is a mixture of air with combustible gas, vapour, or finely-comminuted liquid, and in the explosion these are suddenly converted into water vapour and the oxides of carbon, which latter are gases. Although all these things are liable to explode, none of them are called explosives; this term is confined to liquid and solid substances, which produce much more violent effects than exploding gaseous mixtures, because they occupy much smaller volumes originally.

Explosive.

An explosive is a solid or liquid substance or mixture of substances which is liable, on the application of heat or a blow to a small portion of the mass, to be converted in a very short interval of time into other more stable substances largely or entirely gaseous. A considerable amount of heat is also invariably evolved, and consequently there is a flame.

Gas Evolution.

That evolution of gas (or vapour) is essential in an explosion is rendered evident by considering thermit. This consists of a mixture of a metallic oxide, generally oxide of iron, with aluminium powder. When suitably ignited the aluminium is converted into oxide and the iron or other metal is set free in a short interval of time with the evolution of an enormous quantity of heat, but there is no explosion. It is indeed because no gas is evolved that thermit can be used, as it is, for local heating and welding.

Heat Liberation.

It is also an essential condition that heat should be evolved in an explosive reaction, otherwise the absorption of energy due to the work done by the explosion would cool the explosive and consequently slow down the reaction until it ceased, unless heat were supplied from without. Ammonium carbonate, for instance, readily decomposes into carbon dioxide, ammonia, and water, but in so doing it absorbs heat; consequently the reaction is much too slow to be explosive. Ammonium nitrate, on the other hand, is decomposed into oxygen, nitrogen, and water, with the evolution of heat, and is consequently liable to explode. A violent impulse is required to start the explosion, but once it is started the energy (or heat) liberated suffices to propagate the explosion, unless the conditions be such that the energy is dissipated more rapidly than it is liberated.

Sensitiveness.

Another essential for an explosive is that the reaction shall not set in until an impulse is applied. If the reaction set in spontaneously, it is obvious that its energy cannot be utilized in the form of an explosion. A mixture of sodium and water evolves hydrogen with the liberation of heat, but reaction sets in immediately the two substances come in contact with one another. Different explosives require impulses of very different strengths to cause them to explode. Some, such as diazobenzene nitrate, are exploded by a slight touch; these explosives are of no practical utility as they are too unsafe. Others, such as fulminate of mercury, are exploded by a moderate blow or a small flame; these are used principally for charging caps and detonators, a small quantity serving to explode a large amount of some other less sensitive explosive. Most of the explosives now used can be exploded by a blow only if it be extremely violent, and many of them cannot be exploded by a flame in the open in ordinary circumstances. The tendency is to use less sensitive explosives because they are safer to handle, but it should never be forgotten that the term "safe," when applied to an explosive, is only a comparative one. The duty of an explosive is to explode, and if it is not treated with proper respect it will, sooner or later, explode at the wrong time with extremely unpleasant results.

Before the subject of explosives was understood so well as it is now, inventors were very liable to think an explosive was very powerful, and therefore valuable merely because it was very sensitive, whereas too great a degree of sensitiveness is really a most objectionable feature. In the middle of the nineteenth century many such mixtures as potassium chlorate and picric acid were proposed through this want of comprehension of a fundamental condition.

Constituents of Explosives.

The explosive gaseous mixtures used in gas and oil engines to which reference has been made are composed of a combustible material, consisting largely of carbon and hydrogen, and air, the useful constituent of which is oxygen. Similarly, nearly all commercial explosives are composed partly of combustible elements, of which carbon and hydrogen are the most important, and partly of oxygen combined, but not directly with the hydrogen and carbon. On explosion the oxygen combines with the hydrogen to form water, and with the carbon to form carbon monoxide or dioxide, or a mixture of the two. It is the heat set free in this combustion that is the main or entire cause of the rise of temperature. The formation of these two oxides of carbon liberates very different quantities of heat; 12 grammes of carbon unite with 16 grammes of oxygen to form 28 grammes of carbon monoxide with the liberation of 29 large Calories, and the same quantity of carbon unites with 32 grammes of oxygen with the liberation of 97 large Calories.

Consequently an explosive is considerably more efficient if it contains sufficient oxygen to oxidize the carbon entirely to dioxide, but the effect is reduced to some extent by the relatively high specific heat of carbon dioxide. In some classes of explosives, however, a very high temperature is objectionable; this is the case with smokeless powders and explosives for use in coal mines. Smokeless powders, therefore, are generally made of such a composition that the greater part of the carbon is oxidized only to monoxide. But there is always some carbon dioxide formed, for it takes up some of the oxygen from the water vapour and liberates hydrogen, or if the total quantity of oxygen be very small there may even be free carbon produced. In the case of safety explosives for coal mines, the temperature of explosion is also sometimes kept low by restricting the proportion of oxygen, but this means is not free from objection because carbon monoxide is poisonous. Other methods are therefore adopted in some safety explosives to reduce the temperature.

Oxygen Carriers.

The oxygen may either be contained in a separate compound, such as saltpetre, which is mixed mechanically with the combustible material, or the two may be combined together in a single compound, as is the case with nitro-glycerine, trotyl, and many other modern explosives. The substances rich in oxygen are often referred to as "oxygen carriers"; those most used are nitrates, chlorates, and perchlorates, in which the oxygen is united to nitrogen and chlorine respectively. Ordinary gunpowder, or "black powder," belongs to the class of explosives that have separate oxygen carriers, in this case saltpetre. The table on page 4 shows the properties of the principal oxygen carriers.

It will be seen from this table that the proportion of available oxygen is about the same in the chlorates as in the corresponding nitrates, but whereas the chlorates decompose with the evolution of a small amount of heat, the nitrates require a considerable amount of heat to split them up, except in the case of the ammonium compound. Explosives containing chlorates are consequently much more powerful than those containing nitrates, but they are also very sensitive unless special measures are adopted to render them more inert. The perchlorates require considerably less heat to decompose them than the nitrates, and have more available oxygen. As they are now produced at quite low cost by electrolytic methods, it is not surprising to find that they are being used more and more for the manufacture of explosives. Ammonium nitrate and perchlorate decompose with the evolution of heat, this being due to the formation of water, but the available oxygen is diminished by the same cause. Ammonium nitrate can be detonated by itself, although only with difficulty, and then gives a large volume of gas at a comparatively low temperature. In consequence of this low temperature it has been found very useful as a constituent of safety explosives for use in coal mines, but it also forms part of many other high explosives. Ammonium perchlorate suffers under the disadvantage that amongst its products of explosion is the poisonous gas, hydrogen chloride, or hydrochloric acid.

Oxygen carrier	Molecular weight	Density	Reaction	Heat evolved		Oxygen available	
				per mol.	per 100 grams.	per 100 grams.	per 100 c.c.
Nitrates.							
Potassium .	101·1	2·08	$2KNO_3=K_2O+N_2+5O$	−75·6	−74·8	39·5	82
Sodium .	85·0	2·26	$2NaNO_3=Na_2O+N_2+5O$	−60·5	−71·3	47	106
Calcium .	164·1	2·36	$Ca(NO_3)_2=CaO+N_2+5O$	−70·6	−43·0	49	115
Barium .	261·5	3·2	$Ba(NO_3)_2=BaO+N_2+5O$	−94·4	−36·1	31	98
Lead . .	331·1	4·58	$Pb(NO_3)_2=PbO+N_2+5O$	−54·6	−16·5	24	111
Ammonium	80·1	1·71	$NH_4NO_3=2H_2O+N_2+O$	+27·6	+34·5	20	34
Chlorates.							
Potassium .	122·6	2·00	$KClO_3=KCl+3O$	+11·9	+ 9·7	39	78
Sodium .	106·5	2·29	$NaClO_3=NaCl+3O$	+13·1	+12·3	45	1103
Barium .	304·3	3·18	$Ba(ClO_3)=BaCl_2+6O$	+25·9	+ 8·5	31·5	100
Perchlorates.							
Potassium .	138·6	2·54	$KClO_4=KCl+4O$	− 7·8	− 5·6	46	117
Sodium .	122·5	—	$NaClO_4=NaCl+4O$	−12·4	−10·2	52	—
Barium .	336·3	—	$Ba(ClO_4)_2=BaCl_2+8O$	− 4·3	− 1·3	38	—
Ammonium	17·5	1·89	$2NH_4ClO_4=2HCl+3H_2O$ $+5O+N_5$	+29·5	+25·1	34	65

Potassium permanganate and bichromate have also been used, but they possess no special advantages. Permanganate explosives are often inconveniently sensitive. Attempts have also been made to use liquid oxygen, which has the advantage of being cheap and containing 100 per cent. of available oxygen, but the difficulties of employing a liquid which boils at 200° C. below the ordinary temperature are so great that these attempts were given up. The Germans are, however, making great efforts to develop these explosives for work in mines, so as to set free a corresponding quantity of nitrates for military use. For the same reason the German authorities are encouraging the use of chlorates and perchlorates.

Combustible Constituents.

In black powder the combustibles are charcoal and sulphur; in blasting explosives many sorts of organic matter have been used or proposed, and some inorganic substances, such as potassium ferrocyanide, ammonium oxalate, and antimony sulphide, but those in common use are not very numerous. For explosives containing nitroglycerin an absorbent material must be used, and of these wood meal is the most usual, but flour and starch are constituents of some nitro-glycerine explosives, and in a few cases such substances as tan meal and prepared horse-dung are present. Cork charcoal has great absorptive power, but its high cost prevents its use. Ordinary charcoal is a constituent of some explosives, as also is coal-dust. American dynamites often contain resin and sulphur, and these constituents are sometimes met with in other explosives. Oily materials, such as castor oil, vaselin, and paraffin wax, reduce the sensitiveness of an explosive, and one or other of them may usually be found in a chlorate blasting explosive. The addition of aluminium greatly increases the heat of explosion; it is present in the explosives of the ammonal type.

Nitro-aromatic Compounds.

Modern high explosives very frequently contain nitro-derivatives of the aromatic compounds obtained from coal tar, especially the mono- di- and tri-nitro-derivatives of benzene, toluene, and naphthalene. The nitro-groups in these compounds contribute oxygen for the explosive reaction. The trinitro-compounds of substances containing only one benzene ring are explosives in themselves; trinitrotoluene, for instance. Trinitrotoluene is not only a constituent of composite explosives, but is also very largely used by itself as a charge for shell and submarine mines, and for other military and naval purposes, for which its insensitiveness combined with its great violence render it suitable. Picric acid (trinitrophenol) is also much used for these purposes, and trinitrocresol to a less extent. Although they detonate with great violence, these trinitro-compounds do not contain sufficient oxygen to oxidize the whole of the carbon they contain even to the stage of carbon monoxide. Their power as explosives is, therefore, increased by mixing them with oxygen carriers. Commercial explosives containing trinitrotoluene always have also some other constituent which can supply the deficient oxygen.

Nitric Esters.

Nitro-glycerine and the nitro-celluloses are the principal members of another very important group of substances that can be used as explosives without admixture. Strictly speaking, they are not nitro-derivatives, but nitric esters. The more highly nitrated celluloses, such as gun cotton, contain enough oxygen to convert all the hydrogen into water and the carbon into monoxide, and even some of it into dioxide. Nitro-glycerine, $C_3H_5N_3O_9$, not only has enough to oxidize entirely all its hydrogen and carbon, but also has a little oxygen left over. Nitro-glycerine is the most powerful explosive compound known, but its power is increased by dissolving in it a small proportion of nitro-cellulose, which utilizes the excess of oxygen and at the same time converts it into a gelatinous solid known as blasting gelatin.

Smokeless Powders.

All smokeless powders consist largely of nitrocellulose, which has been more or less gelatinized and converted into a compact colloid by means of a suitable solvent; many of them contain practically nothing else, but in others there is a considerable proportion of nitro-glycerine. Small percentages of mineral jelly, inorganic nitrates, and other substances are also added, in many cases to improve the ballistics or the stability. Powders for rifled arms are always colloided as completely as possible, whether they be for small-arms or ordnance, to make them burn slowly and regularly, but in shot-gun powders the original structure of the nitro-cellulose is not always destroyed entirely, as they are required to burn comparatively rapidly.

Endothermic Compounds.

There are some explosive compounds which do not depend for their action on oxidation or reduction. These are endothermic substances, which decompose with the evolution of gas and heat; they are usually rather sensitive. The only compounds of this class that are of commercial importance are fulminate of mercury, $Hg(CNO)_2$, and lead azide, PbN_6, both of which are used only for exploding other explosives.

Velocity of Explosion.

There are other endothermic explosive compounds in which the heat liberated on decomposing into their elements is only of minor importance compared with the larger amount set free by the redistribution of the oxygen. Such are tetryl and mono- and dinitro-naphthalene.

The heat and gas evolved are the two principal factors which govern the power of an explosive, i.e. the amount of work it can do in the way of displacing objects. But the time taken by the explosion is also a matter of great importance. The rate of explosion is measured by making a column of the explosive, confining it, if necessary, in a metal tube, and measuring the time that the explosive wave takes to travel a known distance. In black powder and similar nitrate mixtures the velocity of explosion is only a few hundred metres a second, but with modern high explosives the velocity of detonation is from two to seven thousand metres a second. This naturally makes them much more violent and destructive. Explosives of the gunpowder type are used when earth or soft rock is to be blasted, or when the material must not be broken up too much. Propellants for use in firearms are required to burn slowly; for rifled arms they must be slower even than gunpowder. They are not exploded by means of another high explosive, but merely lit by a powerful flame, and should then burn by concentric layers. The rate of burning increases with the pressure in the gun, but for completely gelatinized powders it is less than a metre a second.

Incomplete Detonation.

The more insensitive explosives, such as trinitrotoluene, if fired with a weak detonator are only partially decomposed. Not only is some of the

explosive merely scattered, but the velocity of the explosive wave is low. Consequently the effect produced is comparatively small.

Stability.

Another important property of an explosive is its stability. It should retain its properties and composition unchanged when stored even for a long period. Above all it should not be liable to explode or ignite spontaneously. Nitro-cellulose unfortunately is liable to this defect, and consequently special precautions have to be taken in the case of smokeless powders and other explosives containing it.

Summary.

The most important properties of explosives are: power, sensitiveness, velocity of explosion, stability and temperature of explosion. The power depends upon the temperature of explosion and the quantity of gas and vapour evolved. The prices of the constituents and the ease and safety of manufacture are also of importance. All these factors are dependent on the composition of the explosive and some of them on its physical state.

PART I

HISTORICAL

CHAPTER I

EARLY HISTORY

Gunpowder : Confusion of terms : Incendiary mixtures : Greek fire : Wild-fire : Saltpetre : The Chinese : The Indians : Friar Bacon : The Arabs : Invention of firearms : Summary : Gibbon

Gunpowder

SINCE the very earliest times man has been searching for more and more effective means of killing his fellows and the beasts and birds that threatened his safety or provided his food or clothing, but there is reason to believe that the first explosive, gunpowder, was not known before the thirteenth century. This is a mixture of three substances, saltpetre, sulphur and charcoal, two of which have been known from time immemorial, for sulphur occurs native in a state of considerable purity in some volcanic districts, and charcoal is made by simply heating wood. The early history of gunpowder and explosives generally is therefore closely connected with the discovery of methods of preparing and purifying saltpetre.

Confusion of terms.

The investigation of this and other similar matters is rendered difficult not only by the scarcity of early records, but also by the great uncertainty as to their true interpretation. When saltpetre, gunpowder and guns were discovered or invented, new words were not made, but old terms were adopted which had previously been used for somewhat similar objects. Our word "powder," for instance, means any dust-like material, but the term smokeless powder is now used to denote a class of substances which have nothing in common with dust. "Gun" is from the old English "gonne," which was used to denote an instrument for throwing projectiles before the introduction of gunpowder. Similarly the Arabic "bunduq" (بندوق) now used for any rifle or sporting gun, formerly meant a pellet shot from a small catapult used for sporting purposes. Saltpetre (sal petræ) merely means salt of the rock, and the other Latin term for the same material, "nitrum" (nitron, nitre), formerly meant soda or any other white efflorescence. Both nitron and natron in late Latin were derived from the Arabic نطرون (Ntrun'), some of the vowels being usually omitted in writing that language as in shorthand. Similar difficulties occur with the terms in other languages. Nevertheless, considerable progress has lately been made in ascertaining the early history of gunpowder and fire-arms, and various wild statements as to the great antiquity of the knowledge of gunpowder in some countries are now quite discredited, as it is found that the evidence upon which these statements were made will not bear scrutiny.

Early Incendiary Mixtures.

Long before the discovery of saltpetre, incendiary materials had been used in warfare, such as pitch, sulphur, petroleum and other oils. Burning brands were frequently attached to arrows or were thrown by means of engines (catapults), and the descriptions of the effects produced by these early "fire-arms" is often so fanciful and exaggerated that they have been thought to imply the use of gunpowder, with which they really have no connexion. A ball of burning pitch mixed with sulphur and naphtha thrown against a wooden building or ship would cause a fire, which if not quickly extinguished might prove disastrous. Such incendiary mixtures were known in England as "wild-fire." The prompt application of a bucket of water or some sand would, however, remove the danger. Hence, although isolated instances occur in ancient history where great success was achieved with these incendiary mixtures, they must generally have proved ineffective.

Greek Fire A D. 668.

The one notable exception to this is the "Greek-fire" or "sea-fire," the secret of which prevented the conquest of Constantinople and Europe by the Moslems for several centuries. About the year A.D. 668, some forty-six years after the flight of Mohamed from Mecca to Medina, the Arabs, still at the height of their conquering enthusiasm, commenced to beleaguer Constantinople by land and sea, when an architect named Kallinikos fled from Heliopolis in Syria to the Imperial city and imparted the secret of the "sea-fire." This repeatedly spread such terror and destruction among the Moslem fleet, that it was the principal cause of the siege being eventually raised after seven years. In A.D. 716 to 718, the Arabs again appeared before Constantinople with eighteen hundred ships, but again were defeated by the fire; so effectually, that after a stormy passage only five galleys re-entered the port of Alexandria to relate the tale of their various and almost incredible disasters.

Russian naval forces were similarly defeated in 941 and 1043, and the Pisans at the end of the eleventh century.

What, then, was the nature of this "sea-fire"? From the contemporary accounts we know that it was discharged from tubes or siphons in the bows of the ships, but its mode of preparation was kept a close secret and it was never used successfully by any but the Greek rulers of Byzantium. There appears to be no doubt that naphtha was the principal ingredient, and it may also have contained sulphur and pitch. Colonel H. W. L. Hime came to the conclusion that it must have been mixed with quicklime, the slaking of which by the sea-water raised the temperature to the ignition point of the sulphur.[1] I have made a number of attempts to produce ignition in this

way, but although a fairly high temperature was reached the sulphur never caught fire. The heat set free by the slaking of the lime would be ample to raise the temperature to the ignition point if there were no loss of heat, but the reaction is a slow one compared with an explosion, for instance, and consequently much of the heat is dissipated. It seems more probable that the naphtha was simply discharged from a squirt or fire-engine (sipho), and that it was ignited by means of a flame in front of the orifice, and that the secret consisted in the method of constructing the squirt or pump, and of using it so as not to injure the users. If this be so, the Greek fire did not differ greatly from the flame-projectors now employed by the Germans.

Wild-fire.

A D. 1250.

Later the name "Greek fire" was given also to combustible materials which were ignited and then thrown by ballistæ or other machines, and were used on land. These compositions were semi-solid masses of sulphur, pitch, naphtha and other substances that burn readily, and when saltpetre had been discovered this also was added. Such mixtures may more correctly be called "wild-fire." They were much used by the Moslems in the Crusades. Thus Joinville, the faithful and devoted companion of St. Louis in the disastrous sixth Crusade (A.D. 1250), says that "it came flying through the air like a winged long-tailed dragon, about the thickness of a hogshead, with the report of thunder and the velocity of lightning; and the darkness of the night was dispelled by this deadly illumination." Nevertheless, the Greek fire on this occasion did very little damage. That men like St. Louis and Joinville, usually absolutely fearless, should have been terrified by such a cause and described it in such exaggerated language seems to have been due to the fact that they looked upon it as a product of the Devil. By 1250, however, the Arabs were acquainted with saltpetre, and it is quite likely that they mixed some with the incendiary, causing it to burn far more fiercely. Similar language is used in describing the incendiary missiles discharged by the Moors in Spain in battles and sieges of about the same date.

Saltpetre.

Saltpetre (potassium nitrate) is formed in the decomposition of animal and vegetable matters. Under favourable conditions it forms an efflorescence on the ground. It must have been by the investigation of such efflorescences that saltpetre was first discovered. These efflorescences are never pure and seldom contain more than a small percentage of potassium nitrate. The ancients did not clearly distinguish such deposits of saltpetre from the similar ones of soda which are found in some localities. The first preparation of saltpetre of even moderate purity from such a deposit would require considerable chemical knowledge, and it could only have been done in a country where the deposits are plentiful, that is, in a country sufficiently warm to accelerate the decomposition of the organic matter and having a regular and prolonged dry season during which the deposit would collect and not be washed away. The climate of Western Europe is consequently not favour-

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[1] *Gunpowder and Ammunition*, London, 1904.

able, and moreover scientific knowledge and investigation were very backward in Europe in the early Middle Ages. The people who were most proficient in this branch of knowledge at that time were the Arabs or rather the Arabic-speaking people of Spain, Northern Africa and Syria, and many parts of these countries have climates suitable for the formation of saltpetre deposits. Consequently, it is not surprising that it is in Arabic that the first clear reference to saltpetre is to be found. This is in the writings of Abd Allah ibn al-Baythar, a Spanish Arab who died at Damascus in 1248. It seems probable that the Arabs and Egyptians knew saltpetre in a fairly pure state about 1225.

The Chinese. The Chinese apparently became acquainted with saltpetre at about the same period, and it is possible that they were the original discoverers of saltpetre. The Egyptians called it "Chinese snow,"[1] and it is significant that Chingis, the Mongol conqueror, brought Chinese engineers with him in 1218 to reduce the fortifications of the cities of Persia.[2] The statements made by the early Jesuit fathers as to the great antiquity of the manufacture of gunpowder in China, have been shown to be inaccurate and founded on erroneous translations.[3] Marco Polo, who was in the Far East from about 1274 to 1291, says concerning the city of Chan-Glu in Part II., Chapter L., of his book: "In this city and the district surrounding it they make great quantities of salt, by the following process; in the country is found a salsuginous earth; upon this when laid in heaps, they pour water, which in its passage through the mass imbibes the particles of salt, and is then collected in channels from whence it is conveyed to very wide pans not more than four inches deep. In these it is well boiled and then left to crystallize. The salt thus made is white and good, and is exported to various parts." The material prepared in this way could not fail to contain a considerable proportion of saltpetre, moreover the soil in the province of Che-li, in which the city mentioned seems to have been situated, is known to be rich in saltpetre. But from Marco Polo's statement it is probable that the product was used as common salt. In fact, the Chinese appear to have used saltpetre as ordinary salt even at much later periods.

Chinese fireworks. In the chronicles called *Tung-Klan-Kang-mu* there is an account of the siege of Pien-King (now Kai-fung-fu) in 1232, and this was translated into French by Reinaud and Favé in the *Journal Asiatique* for October 1849:

> "At that time use was made of the 'ho-pao' or fire pao, called 'Tchin-tien-loui' or 'thunder that shakes the sky.' For this purpose an iron pot was used which was filled with 'yo.' As soon as a light was applied, the pao rose and fire spread in every direction. Its noise resembled that of thunder

> and could be heard more than 100 lis (thirty-three English miles); it could spread fire over more than a third of an acre. This fire even penetrated the breast plates on which it fell."
>
> "The Mongols constructed with ox-hides a passage which enabled them to reach right to the foot of the rampart. They commenced to sap the walls, and made holes in them in which they could remain sheltered from the men above. One of the besieged proposed that they should hang fire-paos from iron chains and let them down the face of the wall. When they reached the places that were mined, the paos burst and shattered the enemies and the ox-hides, so as not to leave a vestige of them."
>
> "In addition, the besieged had at their disposition some 'arrows of flying fire' (fei-ho-tiang): to an arrow was attached a substance susceptible of taking fire; the arrow flew suddenly in a straight line and spread flames over a width of ten paces. No one dared approach. The fire-paos and arrows of flying fire were much feared by the Mongols."

This arrow may have been a squib or a rocket, or merely an arrow to which a saltpetre mixture was attached. The effects described could hardly have been produced without the use of saltpetre, nor the great noise without an explosive, but we need not take literally the statement that it could be heard thirty-three miles away.

By A.D. 1259 the Chinese had made a further advance. The same annals state: "In the first year of the period Khai-King was made an appliance called 'tho-ho-tsiang,' that is to say, 'lance with violent fire.' A 'nest of grains' was introduced into a long bamboo tube, which was set light to. A violent flame came out and then the 'nest of grains' was shot forth with a noise like that of a pao, which could be heard at a distance of about 500 paces." This was evidently the device now known as a Roman candle.

The Indians. Statements have been made with regard to the antiquity of gunpowder in India upon similarly incorrect evidence. It is improbable that the refining of saltpetre can have been discovered in India, as the habits of mind of the educated classes would prevent their interesting themselves in such matters, and the institution of caste would render it impossible for them to handle many of the materials involved. But the same institution has enabled the saltpetre industry to be developed very widely, when once the process had been discovered elsewhere and introduced, as a special caste of saltpetre workers was formed, and India still supplies a large proportion of the saltpetre used. The saltpetre at first must have been very impure, as the methods of refining it were crude.

Friar Bacon 1214 to 1294. About 1249 Roger Bacon wrote an account of the composition and manufacture of saltpetre and gunpowder in his *De Secretis* and *Opus Tertium*. Those in the former work are fairly full, but were concealed by means of

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[1] Hime, *Gunpowder and Ammunition*, p. 17.

[2] Gibbon, chap. lxiv.

[3] Hime, chap. vii.

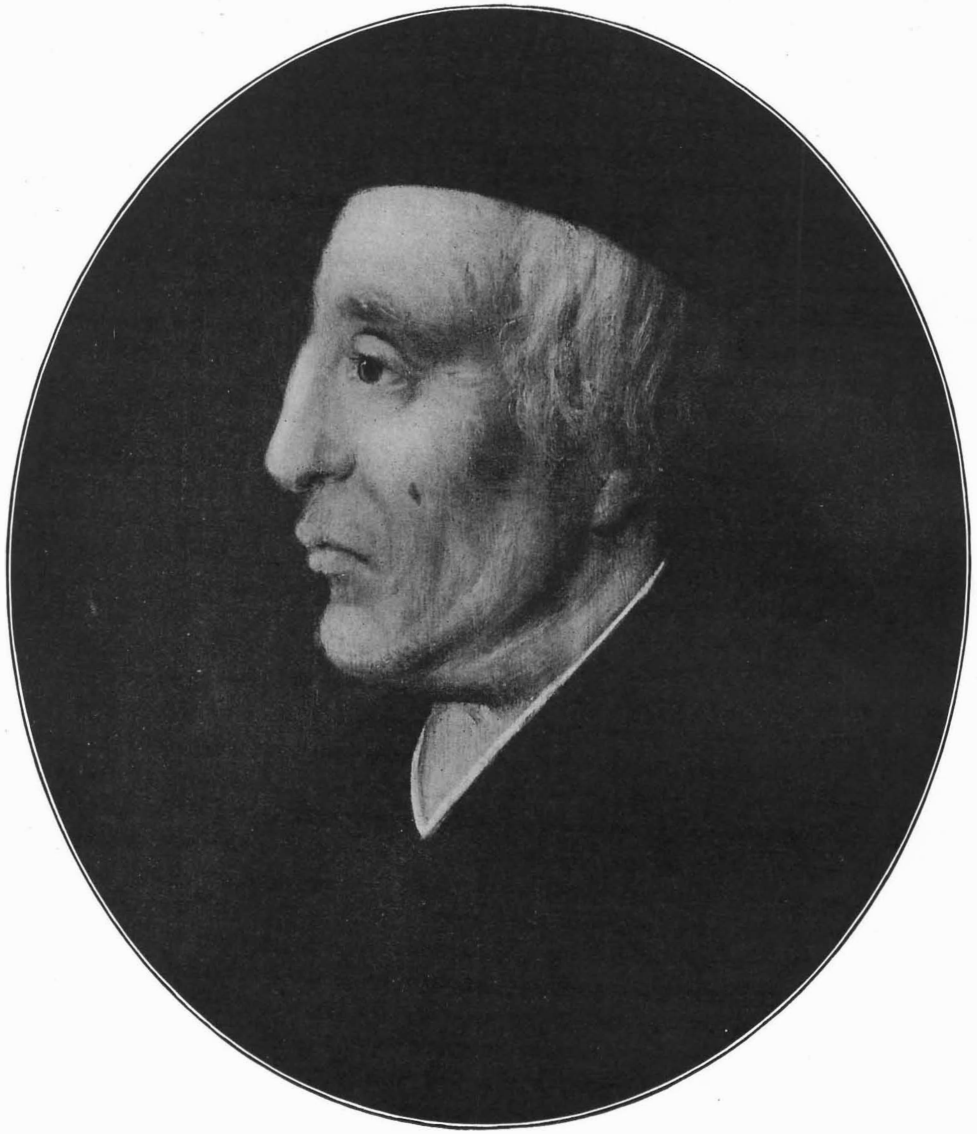

FIG. 1. Portrait of Roger Bacon.
(By kind permission of Lord Sackville, from a photograph by H. F. Corke.)

ciphers, which, however, have been deciphered by Colonel Hime with great ingenuity.[1] Bacon's statements, when not cryptic, are generally vague.

In his *Opus Tertium*, written about 1250, a clearer passage has recently been found by Prof. P. Duhem in a fragment discovered in the Bibliothèque Nationale, Paris. The following free translation has been published by Colonel Hime in the journal of the Royal Artillery for July 1911 :

> "From the flashing and flaming of certain igneous mixtures and the terror inspired by their noise wonderful consequences ensue. As a simple example may be mentioned the noise and flame generated by the powder, known in divers places, composed of saltpetre, charcoal and sulphur. When a quantity of this powder no bigger than a man's finger is wrapped up in a piece of parchment and ignited, it explodes with a blinding flash and a stunning noise. If a larger quantity were used, or if the case were made of some solid material, the explosion would of course be much more violent, and the flash and din altogether unbearable.
>
> "If Greek fire, or any fire of the same species, be employed, nothing can resist the intensity of its combustion.
>
> "These compositions may be used at any distance we please, so that the operators escape all hurt from them, while those against whom they are employed are suddenly filled with confusion."

The Arabs

There can be little doubt that soon after the discovery of saltpetre the Arabs introduced it into their "Greek fire" and other incendiaries. In Europe, saltpetre must have been more scarce than in Africa and Asia. Moreover, the chivalry of Western Europe looked upon such means of war with horror and perhaps were half aware that the use of them must eventually destroy the Order.

In the *Liber Ignium* of Marcus Græcus, which was probably translated into Latin from an Arabic source about 1300,[2] there are several references to such mixtures, but the translator does not appear to have understood the subject he was writing on, and consequently it is not now possible to be sure whether he is endeavouring to describe firebrands, rockets or other fire-works. One "flying fire" (ignis volatilis) is composed of :

Resin	1
Sulphur	1
Saltpetre	2

dissolved in linseed oil and put into a (hollow) reed or piece of wood. This was apparently an incendiary (wild-fire).

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[1] *Gunpowder and Ammunition,* chap. viii. *See* also first edition of this work.

[2] *See* Hime, p. 103.

Another is made of

Sulphur	1
Vine or willow charcoal	2
Saltpetre	6

These were rubbed down together on a marble slab and put into a case (tunica) in different manners according to the effect to be produced. To make a loud noise the case was to be short and wide, and filled only half full, and was to be bound with strong iron wire. Evidently this was a cracker not unlike one described by Bacon. On the other hand, the "flying tunica" was to be thin and long, and filled with the above powder well rammed in. This was apparently an imperfect rocket or squib. The same work contains a second description of these fire-works (recipes 12, 13, 32, 33), but this does not help to clear up the uncertainties.

That the Arabs were probably using saltpetre in their firebrands in 1250, is shown by the passage in Joinville, quoted above (page 13). At the siege of Niebla, in Spain, in 1257, we are told that the Moors "launched stones and darts from machines, and missiles of thunder and fire."

Invention of firearms.

The Chinese do not appear to have developed explosives beyond this point, or to have made the next step, namely, of causing the powder to throw a heavy projectile instead of a ball of fire. Perhaps they made the attempt, but with disastrous results to themselves.

This step could only be taken by a nation which was at once progressive and well acquainted with the working of metals. For some time the development of gunpowder must have been impeded by the scarcity and poor quality of saltpetre. Before any great advance could be made, it was necessary for a considerable organization to grow up for collecting the saltpetre and refining it. In the meantime all the available supply was no doubt absorbed by the makers of warlike combustibles.

In the thirteenth century, therefore, saltpetre was known and used from China to Spain and England, but before the invention of fire-arms its utility can have been but small. No reliable fuse having yet been discovered, hand grenades or bombs can have been of little use and must have been more dangerous to those using them than to the enemy. The fire-works which have been alluded to must have been very uncertain in their action and not without risk to the fire-worker. It does not seem to have occurred to anyone to use explosives to blow up the walls of a besieged town by mining underneath and firing off a large quantity; the primitive powder was no doubt too uncertain in its action and its properties were not well enough known. There is evidence to show that, for getting minerals, gunpowder was not used until the seventeenth century.[1]

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[1] *See* chap. ii.

The real development of gunpowder and its extensive use had to wait, therefore, for the invention of the gun. It is generally considered that this was accomplished by the German monk Berthold Schwartz, as he is named as the inventor in many old manuscripts. There is, however, a curious inconsistency about the dates mentioned. The year 1380 is given by Flavius Blondus, Æneas Sylvius, Baptista Saccus and many others living in the fifteenth century. Other writers have stated that the invention was made in 1354, 1390, and 1393.[1] But on the other hand, there is no doubt that guns were used much earlier. There is a manuscript in the Asiatic museum at Petrograd probably compiled by Shems ed Din Mohammed about 1320 which shows tubes for firing off both arrows and balls by means of powder.[2] In an illuminated manuscript entitled *De Officiis Regum*, written by Walter de Millemete in 1325 and preserved in Christ Church Library, Oxford, there is a drawing, reproduced in Fig. 2, of a rudimentary gun shaped like a bottle, and discharging a dart. A man is applying a light to the touch-hole. On February 11, 1326, the Republic of ~~Venice~~ Florence ordered the provision of iron bullets and metal cannon for the defence of its castles and villages,[3] and in 1338 cannon and powder were provided for the protection of the ports of Harfleur and l'Heure against Edward III.[4]

In two frescoes in the church of the former monastery of St. Leonardo in Leccetto, near Siena, painted by Paolo del Maestro Neri in 1340 are shown a large cylindrical cannon discharging a spherical cannon ball, and many hand-guns.[5]

In 1331 cannon were apparently used by the Moors at the siege of Alicante,[6] and in 1342 in the defence of Algeciras against Alphonso XI of Castille.

The ~~Counts~~ Earls of Derby and Salisbury were present with the Spaniards, and it is supposed that they introduced guns into England. In the following years there are several references in the accounts of the Wardrobe of Edward III of payments on account of saltpetre. Thus Thomas de Roldeston, Clerk of the King's Private Wardrobe in the Tower of London, gives an account for forty shillings for making powder and repairing various arms in the period 1344 to 1347: "Eidem Thomæ super facturam pulveris per ingeniis et emendatione diversarum armaturam XL sol."[7] And an account was discovered by Guttmann delivered by John Cok, Clerk of the King's Great Wardrobe for the date

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[1] Guttmann, *Manufacture of Explosives*, 1895, vol. i., pp. 10–11.

[2] O. Guttmann, *Monumenta Pulveris Pyrii.*

[3] Libris, *Histoire des Sciences mathematiques en Italie*, vol. iv., p. 487; *P. et S.*, vol. vii., p. 33.

[4] *P. et S.*, vol. vii. p. 34.

[5] *See* Guttmann, *Monumenta Pulveris Pyrii*, 1906.

[6] Utescher, *S. S.*, 1914, p. 101.

[7] Guttmann, *Manufacture of Explosives*, vol. i., p. 13; Hunter, *Archæologia*, 1847 vol. xxxii.

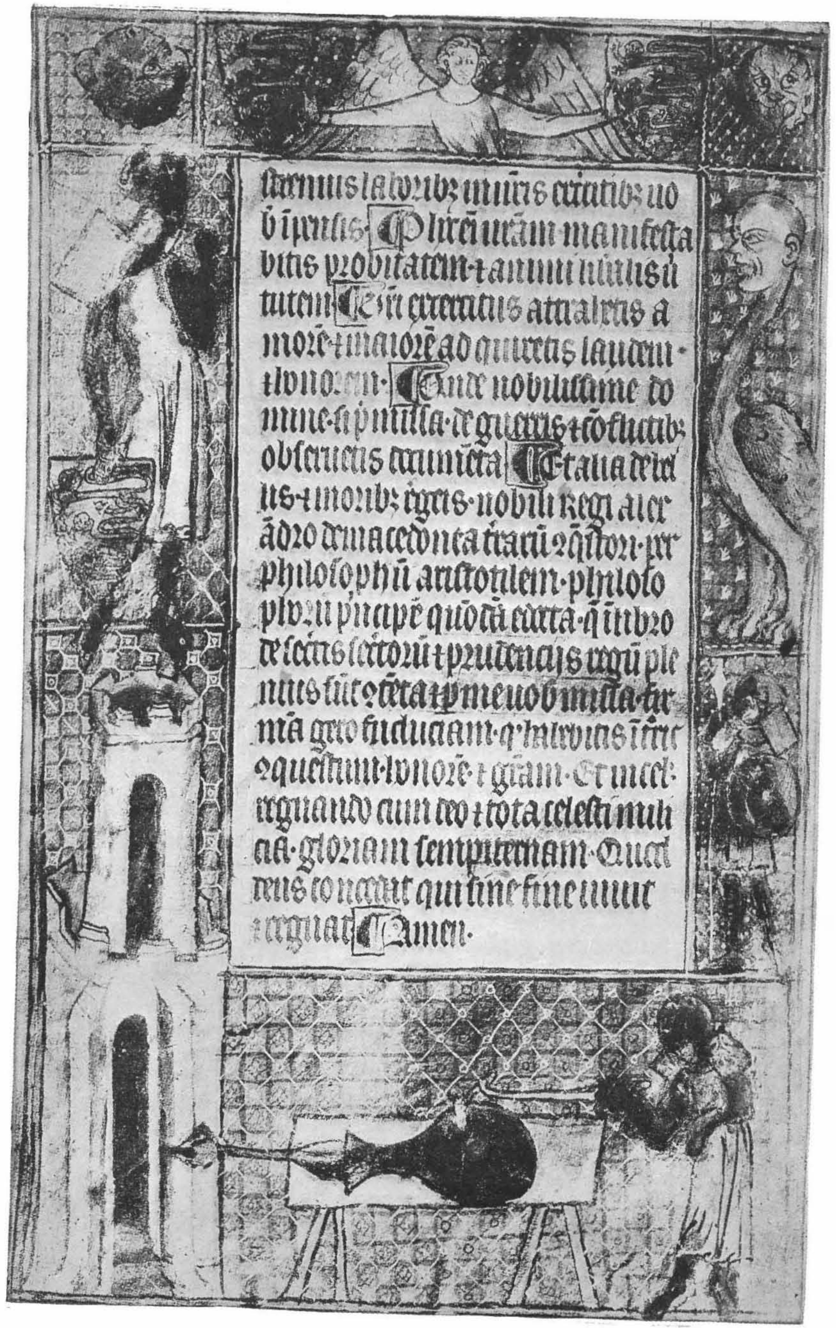

FIG. 2. From Walter de Millemete's Manuscript, A.D. 1326.
(By kind permission of the Dean of Christ Church, Oxford.)

May 10, 1346, for 912 lbs. of saltpetre, and 886 lbs. of quick sulphur for the King's guns:[1] "Et eidem Thomæ de Roldeston per manus Willielmi de Stanes ad opus ipsius Reis pro gunnis suis $\overset{c}{\mathrm{I}}$ XXII lib. sal petræ et DCCC $\overset{xx}{\mathrm{IIIIVI}}$ lib. sulphur vivi per breve Regis datum X die Maii dicto anno $\overset{o}{\mathrm{XX}}$." When all possible allowance has been made for alterations in the meanings of words, there can be no doubt that in 1346 King Edward had guns and powder. On November 25, 1346, and again on September 21, 1347, an official order was given to buy all available saltpetre in the country. On the first occasion 750 lbs. of saltpetre and 310 lbs. of sulphur were obtained; on the second, 2021 lbs. of saltpetre and 466 lbs. of sulphur. The price of the saltpetre was 18*d*. per lb., of the sulphur 8*d*.

At the battle of Crecy (August 26, 1346) guns were used by the English. The French are also believed to have possessed them, but apparently left them behind in order not to be encumbered with them in their pursuit of the English.

Summary. We see then that saltpetre became known about 1225 and that by 1350 fire-arms were in use to a considerable extent in Western Europe.

Saltpetre was apparently discovered by one of the Oriental nations, and was used for making fire-works and incendiary mixtures both in the East and West, but especially the East. The explosive properties of saltpetre mixtures must have been known to many people besides Roger Bacon, but they were of little use until the discovery of fire-arms, which apparently was made in Italy or Germany early in the fourteenth century.

The period of the first development of fire-arms was in many respects an important one. The division of the power in Italy, France and Germany among a great number of petty rulers had given the opportunity for the growth of the free cities on the one hand and the Papacy on the other. The latter had used its power to crush the Albigeois in Southern France, the most civilized and cultured people of the time, and by 1250 had extinguished them with fire and sword. The free cities were frequently richer than important countries, and it was in them that the skill and knowledge were developed which made it possible to construct ordnance and make gunpowder. Only in England did the king retain much power. In the East the prestige of Christianity and the "Franks" was then at its lowest ebb, but a steady advance was to come. The Christian religion had been extirpated from Africa and a great part of Spain, and in Syria the Crusaders had finally failed to retrieve the Holy Sepulchre. The Arabs had long since been obliged to resign most of their conquests to the Turks, who had reduced the Eastern Roman Empire to little more than the city of Constantinople, and that had become the spoil alternately of French, Normans, Venetians and Genoese.

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[1] Public Record Office, London, L.T.R. Enrolled Wardrobe Account No. 4.

The final fall of the city was, however, postponed by the appearance of another race who came, like the Turks, from Central Asia. These were the Tartars or Moguls, the greatest conquerors known in human history. Under Chingis they conquered China in 1210 to 1214, Carizme, Transoxiana, and Persia in 1218 to 1224. The cities of the Middle East were reduced with the aid of a staff of skilful Chinese engineers, who perhaps brought with them the secret of saltpetre. Under the successors of Chingis fire and destruction were carried into Russia, Poland and Hungary. At the beginning of the fourteenth century the Mogul Empire declined under the civil wars which almost inevitably result on the death of a monarch in the East. The Turks regained their ascendancy for a time in Western Asia. In 1361 to 1405 Timour or Tamerlane usurped the whole of Chingis' Empire except China, and added to it Northern India, part of Syria and Asia Minor.

But already the forces were being born which were to revolutionize the world. In the cities of Italy, Germany, England and France a spirit of freedom in inquiry, adventure and culture was arising which now dominates the whole earth.

Gibbon

NOTE.—The account of the Greek fire is largely derived from Gibbon's *Decline and Fall.* Although this remarkable work was written in the eighteenth century, yet such is its accuracy that even upon such a difficult and technical matter as this, subsequent research has been able to find no errors in the statements. In a footnote relating to the discovery of gunpowder, Gibbon says:

"The vanity, or envy of shaking the established property of fame, has tempted some moderns to carry gunpowder above the fourteenth and Greek fire above the seventh century. But their evidence, which precedes the vulgar æra of the invention, is seldom clear or satisfactory, and subsequent writers may be suspected of fraud or credulity. In the earliest sieges some combustibles of oil and sulphur have been used, and the Greek fire has *some* affinities with gunpowder both in nature and effects."

It is impossible to sum the matter up better.

CHAPTER II

DEVELOPMENT OF GUNPOWDER

Early manufacture : Early powder-making machinery : Incorporating mill : Stamp mills : Additions to gunpowder : Corned powder : Pressed powder : Breaking down : Composition of gunpowder : Testing gunpowder : Fire-arms : Double-barrelled guns : Rifles : Cannon Projectiles : Incendiary missiles : Shell : Fuses : Hand-grenades : Infernal machines : Fire-works : Military mines : Blasting

Early manufacture.

IN the fourteenth century gunpowder was only used on a small scale and was made in ordinary houses with pestle and mortar. We hear, for instance, that the Rathaus at Lübeck was destroyed by fire in 1360 through the carelessness of powder makers.[1] Berthelot has stated that there were powder mills at Augsburg in 1340, at Spandau in 1344 and Liegnitz in 1348,[2] but Feldhaus could find no confirmation of these statements in the archives of these towns. There is no mention of gunpowder or fire-arms in Augsburg before 1372 to 1373, and the first powder mill was erected at Spandau in 1578. The scale of operations gradually increased, and in 1461 we find the first mention of a "powder-house" in the Tower of London; powder was made there for many years, as also in Porchester Castle.[3] In the sixteenth century mills of considerable size were in existence: the Liebfrauenkirche in Liegnitz suffered at this time from the effects of explosions in a mill near by. In 1554 to 1555 a gunpowder mill is said to have been erected at Rotherhithe, and about 1561 George Evelyn, the grandfather of John Evelyn, the diarist, had mills at Long Ditton and Godstone, having learned the methods of manufacture in Flanders. A few years later he obtained from Queen Elizabeth a monopoly of the manufacture of gunpowder, which he and his sons were able to maintain more or less until 1636, when Samuel Cordewell obtained the monopoly, which was abolished by Parliament in 1641, the year before the outbreak of the Civil War. George Evelyn made a fortune out of gunpowder, and some of his sons did well, but it is doubtful whether

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[1] F. M. Feldhaus, *S.S.*, 1909, p. 275.

[2] *Revue des deux Mondes*, Aug. 15, 1891, p. 817.

[3] *Brit. Exp. Ind.*, pp. 183 *et seq. See also History of the Evelyn Family*, by Helen Evelyn, 1915, pp. 19 and 26.

any one else made much money out of it in England. After the Restoration the monopoly of gunpowder, as of many other materials, was re-established for a time, but does not appear to have been maintained long.

In the reign of Charles I the contractors supplied to the Crown every year 240 lasts of gunpowder at 7*d.* or 8*d.* per lb., and the Crown sold it again at about a shilling, the retail price being about 16*d.* A last consisted of 24 barrels containing 100 lbs. each.[1]

Early powder-making machinery.

At first gunpowder was made by simply pounding up the constituents and mixing them together in a mortar. Often the pestle was suspended from a flexible wooden rod, which acted as a spring to assist the lifting. The very earliest known example of such an appliance is shown in an illustrated manuscript, the "Codex Germanicus," preserved in Munich (No. 600 of the Hof- und Staatsbibliothek). Guttmann assigns to this the very early date of 1350.[2] Similar pictures appear in later manuscripts, such as the "Rust und feuerwerck buych" of the fifteenth century, in the Stadtbibliothek at Frankfurt a/M. The latter also shows the next step in the adoption of machinery, the stamp mill. Guttmann, in his *Monumenta Pulveris Pyrii,* gives reproductions of many old drawings of such plant. In the fifteenth and sixteenth centuries there were generally two stamps working in each mortar. They were raised alternately by a cam projecting from an axle which was turned by hand. At a later date water wheels and treadmills were used as the motive power, and only one stamp worked in each mortar. Horses do not seem to have been used.

Incorporating mill.

There is a picture of an incorporating mill with an edge-runner in a book entitled *Corona e Palma Militare di Arteglieria* by A. Capo Bianco, and published at Venice in 1598. It has only one edge-runner, and the machine is apparently turned by a horse working in the same building.

Stamp mills.

Stamp mills were still used extensively on the Continent at the end of the nineteenth century, but in England they were forbidden. Tilt hammers were also used sometimes, especially in Switzerland, and in more modern times rotating drums containing hard wood balls have been employed.

At first the powder was used in the fine state. In this condition it burned slowly, as the interstices were very small; it was liable to foul the fire-arm very badly after a few rounds, and it was difficult to regulate the effect, which depended very much upon the ramming. Bourne, in his *Art of Shooting in Great Ordnance,* 1587, said: "The powder rammed too hard and the wad also, it will be long before the piece goes off. . . . The powder too loose . . . will make the shotte to come short of the mark. . . . Put up the powder with the rammer head somewhat close, but beat it not too hard." Other disadvantages of the fine powder were that it absorbed moisture very rapidly, and the constituents were liable to separate one from the other if the powder were subjected to much vibration. For although amorphous carbon has much the same absolute density as saltpetre and sulphur, powdered charcoal contains many cavities which make it lighter than the other constituents.

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[1] *Brit. Exp. Ind.*, p. 278.

[2] *Monumenta*, p. 19, Fig. 48.

Additions to gunpowder.

It was partly to prevent this separation of the constituents that the early powder-makers added camphor, sal ammoniac and gum dissolved in spirit to their powder. Thus in the "Codex Germanicus" of the fourteenth century the following recipe is given: "Wiltu ein gut starck pulver machen So nym IIII lb Salniter und I lb swebel und I lb kol/ I uncz salpetri und I uncz salarmaniak Item und ainen XII tail campfer und stoz daz alls wol undeinand/ und tu gepranten wein darczu und stoss damit ab. und derre daz wol an der sunn/ so hastu ein uberstarck beliwig pulver/ dez phunt mer tut denn sust III phunt getun möchten /und ist auch behaltig und wirt lenger pesser."

Translation: "If you want to make a good strong powder, take 4 lb. of saltpetre, 1 lb. of sulphur and 1 lb. of charcoal, 1 oz. of salpratica and 1 oz. of sal ammoniac and one-twelfth part of camphor. Pound it all well up together, and add spirit of wine and mix it in, and dry in the sun. Then you have a very strong powder, of which 1 lb. will do more than 3 lb. otherwise. It also keeps well and becomes better with time."

In another part of the same manuscript it is stated: "Wenn wa nicht gampfer pey ist daz pulver erwirt und verdirbt gern. Aber daz gamfer halt allez pulver auf/ und ist auch kreftig und prunstig in allem pulver wenn man in darin tut." Translation: "When there is no camphor, it crumbles and easily spoils. But the camphor holds all powder together, and is also strong and quick in all powder, if one puts it in."

"Salpratica" was a mixture of saltpetre, camphor and sal ammoniac, made by dissolving them all in spirit, placing the mixture in an unglazed earthenware vessel, and scraping off the efflorescence that was formed. The composition must have been very variable. These volatile materials were also supposed to improve the power of the explosive by increasing the amount of "air."

Corned powder.

The separation of the ingredients was restrained at a later date by "corning" the powder, that is, breaking the cakes into small grains only, instead of to a fine powder. In order to get a hard mill-cake, which would give good grains, the contents of the mortar were moistened before the end of the stamping operation with water, wine or urine. After it had been broken down the grains of the required size were separated by sifting.

The earliest known reference to corned powder is in the "Firebook" of Conrad von Schongau dated 1425.[1] It left less fouling in the fire-arm and burned faster and more regularly than the very fine powder, but it developed greater pressure and consequently it could not be used in the ordnance of the time, but only in hand-guns. The fine powder came to be called "serpentine," apparently from the name of the small cannon.

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[1] M. Jähns, *Geschichte des Kriegswesens*, Leipzig, 1880.

Whitehorne, in his *Certain Waies for the Ordering of Souldiers in Battelray*, 1560, says: "If serpentine powder should be occupied (used) in handguns, it would scant be able to drive their pellets a quoit's cast from their mouths; and if handgun (*i.e.* corned) powder should be used in pieces of ordnance, without great discretion, it would quickly break or mar them."

According to Guttmann, the French powder-mills began in 1525 to grain and classify their powder by passing it through sieves.[1] Apparently corned powder came gradually into use for small-arms and hand-grenades during the fifteenth century, and for big guns in the sixteenth, the construction of these being sufficiently improved by that time. In an engraving by Phillip Galle, after a drawing by John Stradanus, to which the date 1570 has been assigned,[2] the operations of casting and finishing guns are shown, and the manufacture had evidently reached a fairly high state of development by that time.

Pressed powder.

A really hard compact grain could not be made by this method, consequently after a time presses were introduced to compress the mill-cake before corning. According to Guttmann [3] presses were first used for this purpose in 1784. At Faversham in 1789 the powder was compressed by means of a screw press, shown in drawings in a contemporary note-book.[4] In the nineteenth century hydraulic presses were introduced.

Breaking down.

The cake was broken down by hand with wooden mallets, and then pressed through sieves to granulate and sort it. At one time wooden rollers were used to press it through the sieves, but later discs of lignum vitæ were placed in the sieves, which were suspended by means of cords and swung backwards and forwards. Colonel Congreve in 1819 introduced his granulating machine, which is described in Chapter VI.

Composition of gunpowder.

Colonel Hime has given tables to show the development that took place in the composition of gunpowder in the course of time. With some alterations these are reproduced here:

English Gunpowder

Date	Authority	Saltpetre	Charcoal	Sulphur
1250	Roger Bacon	41·2	29·4	29·4
1350	Arderne (Laboratory receipt) .	66·6	22·2	11·1
1560	Whitehorne	50·0	33·3	16·6
1635	Government contract . .	75·0	12·5	12·5
1670	Sir J. Turner	71·4	14·3	14·3
1742	Robins	75·0	12·5	12·5
1781	Bishop Watson . . .	75·0	15·0	10·0

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[1] *Manufacture*, vol. i., p. 17.

[2] *Monumenta*, Fig. 8.

[3] *Manufacture*, vol. ii., p. 204.

[4] *Brit. Exp. Ind.*, Fig. 13 and p. 36.

Foreign Gunpowder

Date	Country	Saltpetre	Charcoal	Sulphur
14th Century	Germany.	66·6	16·6	16·6
1560	Sweden	66·6	16·6	16·6
1595	Germany.	52·2	26·1	21·7
1598	France	75·0	12·5	12·5
1608	Denmark.	68·3	23·2	8·5
1697	Sweden	73·0	17·0	10·0
1882	Germany.	78·0	19·0	3·0

The proportions 6 : 1 : 1, known as "as, as, six," were first adopted in France at the end of the sixteenth century and have been adhered to there more or less ever since.[1]

The fourteenth-century German powder has been substituted for a French powder of about the same date mentioned by Hime, as it is a more satisfactory example. The last item in the list is German cocoa powder, ballistically the best "black" powder ever made.

But in reality the composition was extremely variable. Every powder-maker had his own formula in early days, and in the absence of testing apparatus there was no means of judging which was best. With the invention of corned powder another variable was introduced, the size of the grains, and the confusion became still worse.

Testing gunpowder.

In the Middle Ages the only tests applied to powder were to feel it to ascertain whether it was moist, and to burn a little to see whether much residue was left. The first instrument for testing powder, of which we have any knowledge, is that described by Bourne in his *Inventions and Devices*, published in 1578. It was a small metal cylinder with a heavy lid on a hinge. The lid was prevented from falling by a ratchet, and the angle to which it rose when powder was fired inside the box measured its strength. **1578.**

A much better instrument was that devised by Curtenbach and described by him in his *Halinitro Pyrobolia*, in 1627. This consisted of a heavy conical shot which rested on the mouth of a small mortar and could travel vertically upwards along a stretched wire. It was prevented from falling again by a series of catches. There is a copy of this in the Imperial Museum in Vienna, and Guttmann gives a reproduction of a photograph of it as Fig. 67 of *Monumenta Pulvis Pyrii*. **1627.**

Master-Gunner Nye, in his *Art of Gunnery*, 1647, described the same instrument, and also proposed that the strength of powder be measured by **1647.**

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[1] Chalon, *Explosifs Modernes*, p. 228.

firing bullets from a pistol into clay, or by firing a heavy ball from a mortar and finding out how far it travelled. This last, the *mortar éprouvette*, was adopted by the French and other Governments, and led to considerable improvements in the powders. By the beginning of the eighteenth century the proportions of the constituents were fairly well fixed, and the powders for different guns differed only in the size of grain. In 1742 Robins placed the matter on a more scientific basis by the invention of the ballistic pendulum, by means of which the actual velocity of a projectile could be measured. By the end of the century, practically every country had come to use the proportions, 75 of saltpetre, 15 of charcoal, and 10 of sulphur.

Fire-arms.

To trace in detail the development of fire-arms is beyond the scope of this work. Moreover it has depended far more upon the engineer than the powder-maker, who has nearly always been able to supply powder more powerful than the gun-maker has been able to use, through insufficient engineering skill. At first the chivalry of Western Europe was entirely opposed to the use of fire-arms, but it soon had to acquiesce in the employment of gunpowder in warfare, but made a longer struggle as regards the hunting of animals. Hawking and the chase were the only respectable forms of sport, but poachers were not governed by the same scruples, and laws were consequently passed to prevent the use of fire-arms by them. For instance, in 1555 the Elector Augustus, of Saxony, prohibited the possession of fire-arms by peasants and shepherds, and in 1562 small shot was absolutely prohibited throughout the Duchy of Mecklenburg.[1] Nevertheless, it was not possible to prevent the use of military muskets for sporting purposes. Italy, in this respect, as in so many others, was ahead of Northern Europe. Benvenuto Cellini (*b.* 1500, *d.* 1571) when a young man was very fond of shooting for sport, and made his own gunpowder. He shot with a single bullet and boasted of his skill as a marksman. He makes no mention of there being any prejudice or law against the use of fire-arms.[2]

Double-barrelled guns.

It was not until the double-barrelled gun was introduced that there was any real difference between the military and sporting weapons. Double guns were first made sufficiently light to be practicable in the middle of the seventeenth century; in the eighteenth century the ribs were added, and the flint lock and hammer were introduced.

Rifles.

Rifles were already known in the first half of the sixteenth century, and are said to have been invented by Augustus Kotter, of Nuremberg, in 1520, but for a long time the rifle was used principally for sporting purposes, because the necessity of ramming the bullet down the barrel with its spiral grooving made the loading very slow. Moreover, the powder left much fouling in the grooves, and consequently it was necessary to clean the arm after a few rounds.

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[1] Greener, *Modern Shot Guns*, 2nd ed., p. 1.

[2] *Vita di Benvenuto Cellini*, part i.

With the old musket, on the other hand, the bullet was smaller than the bore, and this trouble did not arise to anything like the same extent. In the seventeenth century the rifle was tried in several continental armies, but in every case it was given up again.[1] For sporting purposes accuracy was of more importance than rapidity of fire, and the rifle was able to hold its own, especially in mountainous countries such as the Tyrol and Switzerland. In the American War of Independence the sporting rifle was necessarily used for military purposes, and the English Government found it advisable to enlist on the Continent a corps of "Jägers" to put against the colonial marksmen. Afterwards the Rifle Brigade was raised, and this proved a success from the first. Robins, the inventor of the ballistic pendulum, had already prophesied that wonderful effects would be produced by the State which could first make the military rifle a practical success.

Since then every part of the rifle has been further improved: the action, the rifling, the sights; and magazines have been added to increase the rate of fire. In 1886 smokeless powder for rifles was introduced, and this has added greatly to the efficiency of the weapon. The final development is the introduction of automatic rifles and machine guns, such as that of Maxim, but this part of the evolution of small-arms is still in progress. The development of the pistol has proceeded on similar lines.

Cannon.

The first guns were tubes or pots, which could withstand only very slight pressures. Then they were made of strips of wrought iron welded together. By the sixteenth century they were being cast in bronze, and by the eighteenth in iron. Until the second half of the nineteenth century a gun consisted simply of a block of cast metal with a smooth bore machined out and a vent drilled near the breech. It is true that breech-loading guns were made at a much earlier date, for examples of them may be seen in the museums, but the crudity of the workmanship is sufficient to explain why they were given up again. In the Crimean war (1854) many of the guns used had seen service in the Napoleonic campaigns. In 1858 a committee recommended the introduction of rifled ordnance into the British naval service, and from that time there has been rapid and continuous improvement in all sorts of guns. The introduction of the buffer has made the guns much steadier; breech-loading guns were re-introduced, and the mechanism of the breech has since then been improved enormously.

To meet the requirements of the longer and more accurate guns the grains of the powder were 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 moulding 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

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[1] *Textbook of Small-Arms*, 1909, pp. 6, 7.

secret, it was being made at Waltham Abbey also two years later. This very large and dense powder was required on account of the great increase in the size of naval guns. In 1882 at the bombardment of Alexandria we had 80-ton guns of 16-inch bore, and in 1886 110-ton guns of 16¼-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.

Projectiles. The first projectiles used were made more or less like arrows with metal "feathers" and arrow-heads,[1] just as the first railway carriages were built like stage-coaches. These were soon found to be unsuitable and were replaced by round shot made of iron, bronze, lead or stone. All these materials remained in use for several centuries, but stone was the most common for large guns, partly because its cost was only a fraction of that of a metal shot of the same size, and partly because the guns would not stand the strain of discharging the heavier materials. Lead and iron bullets were usually used for small-arms, but in an emergency any small handy article was made use of.

Incendiary missiles. Attempts were made very early to throw from guns incendiary missiles such as had been discharged previously from machines, but some difficulty must have been experienced from the flames being extinguished by the rapid motion through the air. At the siege of Weissenburg in 1469 stone balls were used considerably smaller than the bore of the gun, and these were smeared over with incendiary matter and wrapped in a cloth soaked in the same mixture.[2]

Shell. Actual shell could not be used at that time, because it was not known how to cast them in metal. But a sort of weak shell was made of earthenware, or by joining two hemispheres of metal. These were filled with a slow burning powder well rammed in, or other incendiary matter, and were provided with an igniter, which was set light to by the flame from the gunpowder of the propelling charge, but there must have been considerable uncertainty about the ignition, and of course it was much too dangerous to introduce a lighted shell into the bore of a gun which had been charged with serpentine powder by means of a shovel. The difficulty was sometimes overcome by enclosing the powder in a paper cartridge, but this method did not find general acceptance. Red-hot shot could not be used for the same reason, until Stephen Bathory, King of Poland, in 1579 used a thick wet wad to prevent the fire spreading to the charge. Hot shot were used with great effect in the defence of Gibraltar by the English in 1782.

Solid shot are not used now except for practice and experimental purposes.

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[1] See *Monumenta*, Plates 69, 70, 71; Hime, p. 199; *Rise and Progress*, Fig. 3.

[2] Hime, p. 220.

The shell for the early muzzle-loading rifled guns were provided with studs to fit into the rifling and with copper plates (gas-checks) over the base to prevent the escape of the gases past the shell. For some of the early rifled breech-loading guns the shell were coated with lead, but now they are provided with copper bands near the base to take the rifling and prevent the escape of the gases. Originally of course shell were filled with black powder, but now high explosives are used almost exclusively for common shell. Shrapnel shell were devised about 1784 by Lieutenant Shrapnel for use against troops in the open. They were adopted officially in 1803 and consisted of a round shell containing only a small charge of powder, just sufficient to break the envelope into fragments, which continued to travel more or less in the same direction and with the same velocity as the shell did before. After the introduction of rifled cannon the Shrapnel shell developed into a cylindrical missile filled with bullets embedded in rosin with a small charge of black powder, which, when ignited by a time fuse, expels the bullets. Against troops in the open its killing power is great, but it is ineffective against them when entrenched, and it has not the nerve shattering effect of common shell charged with high explosive.

Formerly case shot was used against troops at short range. It consisted of a case containing a large number of bullets, which spread out from the muzzle of the gun, the case being broken up in the bore. The principal sorts of case shot were grape, canister and spherical case. They are not used much now, as their place has been taken by shrapnel shell and machine guns. Chain shot was fired against the rigging of ships: it consisted of two balls or half balls united by a chain, and are said to have been invented by De Witt, Pensioner of Holland, about 1666.

Fuses. For explosive shell the difficulty was to make a satisfactory igniter or fuse. The earliest record of really successful explosive shell is in the accounts on the sieges of Wachtendonck and Bergen-op-Zoom in 1588, the master gunner being an Italian refugee from Parma in the employment of the Dutch. The fuses used were apparently tubes or pipes filled with slow-burning powder, which were driven into the fuse-hole of the shell, and this type was adhered to until the middle of the nineteenth century and later, when concussion and percussion fuses were invented.

The fuses were made to burn 14 to 20 seconds, corresponding to ranges of 1000 to 2000 yards in the mortars, which were always used instead of ordinary guns for throwing shell. The shell were used for the destruction of stone fortifications and ships; against men they were not effective, as there was usually plenty of time to get away from them before they exploded. Until after the introduction of watches, which were invented by Huygens in 1674, no convenient means existed of testing the time of burning of a fuse. In the middle of the eighteenth century fuses were made of beechwood with a hole down the middle filled with fuse powder. The fuse could be cut to any required length. Great accuracy was not demanded of them, until Captain Mercier during the siege of Gibraltar in 1779 proposed to fire shell from guns instead of howitzers or mortars. Short "calculated" fuses were then used so as to make the shell burst over the Spanish working parties. The effect produced was considerable.

Accurate fuses were also required for the Shrapnel shell, which was devised by Lieutenant Shrapnel, R.A., about 1784, and officially adopted in 1803, but they were made upon the same principle until the second half of the nineteenth century.

Hand-grenades.

Hand-grenades seem to have been used to a considerable extent in the first half of the sixteenth century, at which time they were probably made of earthenware. They are said to have been used at the siege of Arles in 1536.[1] Whitehorne, writing in 1560, says that "earthen bottles or posses" had been formerly used, but he recommends "hollow balles of metal, as big as smal boules and ¼ in. thick, cast in mouldes and made of 3 partes of brasse and 1 of tinne." They were charged with "3 partes serpentine, 3 partes fine corne powder and 1 part rosen." A little fine corned powder was used as priming, and he directs that the grenades be "quickly thrown," as they will almost immediately "breake and flye into a thousand pieces."

In the seventeenth century the problem of the fuse for hand-grenades was fairly well solved, and regiments were formed of "Grenadiers," powerful men specially trained to throw grenades. Major Adye, writing in 1802, said grenades could be thrown twenty-six yards.[2]

Infernal machines.

The doubtful honour of having invented infernal machines is ascribed to a Nuremberg citizen in 1517, but there is a drawing of one by Leonardo da Vinci, who lived from 1452 to 1519. In 1645 attempts were made to blow up Swedish ships in Wismar harbour by means of clock-work bombs. The clock-work actuated a flint lock with a revolving steel wheel. Clock-work infernal machines containing a nitro-glycerine explosive were used also by the Irish-American Fenians in 1883 and 1884, but now clock-work is not generally applied in these criminal attempts.

Fire-works.

Fire-works seem to have been made soon after the discovery of gunpowder; references to them are found in the writings of Hassan-er-Rammah, Roger Bacon, Marcus Græcus, and Albertus Magnus in the thirteenth and fourteenth centuries. They were probably made first in China and introduced into Europe in the thirteenth century. They were used to celebrate peace at Vicenza in 1379.[3] The essential features were developed early, and later centuries have added nothing really novel. Improvements have been made in the artistic effects, precision of execution and safety, but the general principles are the same. The introduction of chlorates at the end of the eighteenth century has been of some assistance, but their use has been restricted on account of the dangerous character of many chlorate mixtures. In the seventeenth and eighteenth centuries large sums were spent in Europe on fire-work displays to celebrate special events, but they were not much used in war. Carcasses containing incendiary composition, smoke-balls and light balls were used, however, in the Peninsula.[1] The Indians fired rockets in the defence of Seringapatam in 1792, but they do not appear to have done much damage. At the siege of the same town in 1799 explosive rockets seem to have been used with some effect.[2] Soon after this the Ordnance Office applied to the Royal Laboratory, Woolwich, for the services of some one who understood the manufacture of rockets. The Laboratory referred the Ordnance Office to the East India Company, who replied that they knew of no one who possessed such knowledge.

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[1] *Militär Wochenblatt*, Sept. 11, 1915.

[2] Hime, chap. x.

[3] A. Gnadewitz, *S.S.*, 1915, p. 273

Colonel Congreve of the Hanoverian Army (afterwards Sir W. Congreve) was thus led to make experiments, and he devised the Congreve rocket, the most powerful thing of the kind that had been used in warfare. It proved very effective at Copenhagen and Walcheren in 1807, and at the passage of the Adour in 1813, but it was at the battle of Leipsic that it achieved the greatest renown, for a French infantry brigade in the village of Paunsdorf, unable to withstand their well-directed fire, surrendered there to the Rocket Brigade. At Waterloo also good service was rendered.

Since the Napoleonic wars the improvements in ordnance have been so great that the war rocket is no longer used. For military purposes rockets are only fired now as signals and to illuminate the enemy's position at night, and for the latter purpose they have been displaced to a great extent by star shell.

Military mines.

The use of gunpowder for blowing up the enemy's walls and fortifications commenced in the fifteenth century. Mines charged with gunpowder were used in 1415 by the English at the siege of Honfleur.

Blasting.

For blasting minerals gunpowder does not appear to have been used until the seventeenth century. The first recorded blasts were made by Gaspar Weindl at Schemnitz, in Hungary, and from there the method was introduced into Germany in 1627. Prince Rupert, son of the Queen of Bohemia and nephew of Charles I, is said to have brought the practice of blasting to England in 1629, but this is doubtful; 1670 is a more probable date.[3] In 1689 Thomas Epsly Senior started the use of gunpowder in the Cornish mines.[4] The late Mr. Oscar Guttmann, in his book on *Blasting*, published in 1906, gave the following concise account of the further progress:

"When bore-holes first came into use they were made with iron-mouthed

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[1] *Rise and Progress*, p. 174.

[2] Hime, p. 129.

[3] *Brit. Exp. Ind.*, p. 255.

[4] Feldhaus, *S.S.*, 1908, p. 218.

borers, fairly large—nearly 3 inches in diameter, and then closed with a wooden plug, termed the 'shooting plug.'

"Henning Hutman in 1683 employed a kind of drilling-machine. In 1685 clay tampings, and in 1686 firing-tubes, began to be used. In 1689 paper cartridge cases were used to replace the older form of leather, and in 1717 bore-holes of smaller diameter came into vogue. The use of the chisel-borer dates from 1749, blasting the untouched breast from 1767 (first at Zinnwald)."

It is only by blasting operations that many of the engineering feats of modern times have been made possible. In constructing means of communication, such as roads, canals and railways, immense quantities of explosives have been used.

CHAPTER III

PROGRESS OF EXPLOSIVES IN THE EIGHTEENTH AND NINETEENTH CENTURIES

Berthollet, Chlorate : Igniters : Forsyth's detonator lock : Fulminates : Caps : Fuses : Gun-cotton : Nitro-glycerine : Ammonium nitrate explosives : Sprengel explosives : Coal-mine dangers : Cheddite : Inspection of explosives : Smokeless powders : Picric acid : Trotyl

In the nineteenth century commenced the active application of science to explosives, with the result that this industry like so many others developed enormously. In this chapter no attempt will be made to give in detail the history of each invention; only the principal discoveries will be mentioned, and an attempt will be made to show how one has led up to and assisted another.

Berthollet, chlorate.

When chemistry was put on a firm basis at the end of the eighteenth century, there was a great increase in the number of chemical compounds which could be made in the laboratory. No man had more influence upon chemical science than Count Claude L. Berthollet (1748-1822). Amongst the substances which he discovered was potassium chlorate, or at least he in 1786 first showed clearly how it could be prepared in the pure state, and he investigated and described its properties; for it seems to have been known to Glauber (1603–1668). Berthollet found that potassium chlorate, if substituted for saltpetre, produced a more powerful (or violent) explosive, and proposed in 1788 to manufacture gunpowder with it. But the results were most disastrous. A party had been made up to see the first of the new powder made in the mills: M. and Mme. Lavoisier, M. Berthollet, the Commissary, M. de Chevraud and his daughter, the engineer, M. Lefort, and others. Whilst the mixture was being incorporated in a stamp-mill the party went to breakfast. Lefort and Mlle. de Chevraud were the first to return, and as they did so the charge exploded with great violence, throwing them to a great distance and causing them such injuries that they both died in a few minutes.

In spite of repeated attempts it has not been found possible to make a satisfactory propulsive explosive with chlorate; the explosion is always liable to be too violent and uncontrollable.

Until the invention of Cheddite all the chlorate mixtures proposed were too sensitive to be used with safety even as blasting explosives. Cundill and Thomson's *Dictionary of Explosives* issued in 1895 includes the descriptions of 150 mixtures containing potassium chlorate, but with the exception of a few cap and fuse compositions none of these have proved to be of practical use.

Igniters.

The first fire-arms were set off by means of a lighted match, which was applied to a priming of fine powder. Rain or wind seriously interfered with the operation. In the eighteenth century the flint lock was devised and a lighted match was no longer necessary. In its best form the priming powder was contained in a small chamber which was uncovered only at the instant when the descending flint struck a spark from the steel. Although this was a great improvement it left much to be desired as regards ease of loading, rapidity of ignition, and fouling of the touch-hole. Hence the persevering attempts to devise an easier and simpler method.

Forsyth's detonator lock.

In 1805 the Rev. A. J. Forsyth, a Scotch minister, made a sporting gun with a detonator lock, and in the next year submitted his invention to the Master-General of Ordnance, who asked him to adapt it to the requirements of the Service. Forsyth's device consisted of a receptacle or magazine shaped like a scent-bottle, which was attached to the lock of the gun. It contained a detonating mixture of potassium chlorate, charcoal and sulphur. By rotating the magazine a small quantity of this was caused to fall into a small hole in a plug communicating with the touch-hole of the gun, and on again rotating the magazine it was brought into such a position that the portion of detonating priming could be set off by the fall of the hammer.

Forsyth spent some £600 in trying to produce a satisfactory device for military purposes, and he claimed to have succeeded, but the Government authorities were not convinced and did not adopt it. At the time they only paid Forsyth's expenses, but they granted £1000 to his relatives shortly after his death.

For sporting purposes Forsyth's invention had some success, but the profits must have been largely swallowed up by the numerous lawsuits that he instituted to protect it from 1811 to 1819. Before very many years had elapsed Forsyth's device was displaced by the copper tube or cap, containing fulminate of mercury.

Fulminates.

Fulminates of gold and silver have long been known, and their discovery was ascribed to Basil Valentine, a fictitious person of the fifteenth century. They were perhaps invented by Cornelius Drebbel, a Dutchman, about 1600.[1] Pepys, in his diary for November 11, 1663, recounts a conversation with a Dr. Allen, who told him about *Aurum fulminans,* "of which a grain . . . put in a silver spoon and fired, will give a blow like a musquett and strike a hole through the silver spoon downward, without the least force upward."

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[1] F. M. Feldhaus, *S.S.*, 1909, p. 258,

Mercury fulminate.

The fulminates of gold and silver are, however, too sensitive and dangerous for any practical use, but they have played their part as toys and scientific curiosities. Liebig, who was born in 1803, when a boy saw a quack in the market-place of Darmstadt make fulminating silver. The alcohol he recognized from the smell of a garment which the quack had cleaned with liquid from the same bottle. He went home and succeeded in making the substance. In 1823, when he was in Paris with Gay Lussac, he investigated the fulminates at the suggestion of the latter, and isolated fulminic acid. By that time the comparatively stable fulminate of mercury was well known, having been described by Edward Howard, F.R.S., in a paper before the Royal Society in 1800. It is stated that it was manufactured in France in 1819.

Fulminate cap.

There is some uncertainty as to who first invented the fulminate of mercury cap, but it seems that several people were working at the idea at the same time and contributed towards the final success. According to H. Wilkinson,[1] J. Shaw, of Philadelphia, invented a steel cap in 1814; in 1815 a pewter cap, and in 1816 a copper cap. The London gun-maker, Joseph Egg, seems to have adopted the idea from Shaw. The Paris gun-makers, Prélat and Deboubert, in 1820 patented caps filled with fulminates of silver and mercury respectively. E. Goode Wright, of Hereford, in 1823 published a paper[2] on the fulminate of mercury cap, and subsequent workers seem to have derived much information from it. Frederick Joyce was the first to make a real success of the percussion cap about 1824. The firm of Joyce and Co. claim an earlier date, but although experiments may have been made in previous years there appears to be no evidence of manufacture on a considerable scale.[3]

The capped cartridge.

The next important step was to combine shot, powder and cap in one cartridge, which could be inserted in the breech of the arm. Many attempts had been made from very early times to make breech-loading fire-arms, but workmanship and knowledge of engineering were not sufficiently advanced to make a success of it. In 1836 Lefaucheux introduced his pin-fire breech-loading shot-gun, the barrels of which were made to drop as in the modern shot-gun to allow the cartridges to be introduced. This gun, although it had many imperfections, combined all the principal features of those made at the present day. About 1853 the English and French gun-makers introduced the central fire hammer gun, which fired cartridges having a cap in the middle of the base of the cartridges, but the first really successful central fire gun was that made by Daw in 1861.[4]

Breech-loading rifle

In 1841 the Prussians adopted a breech-loading rifle, the "Zündnagel-

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[1] *Engines of War*, p. 187.

[2] *Phil. Mag.*, vol. lxii., p. 203.

[3] The account of the discovery of the percussion cap is largely taken from the paper by E. Wyndham Hulme, B.A., in the *Rise and Progress of the British Explosives Industry*, London, 1909. *See also* Utescher, *S.S.*, 1914, p. 101.

[4] Greener, *Modern Shot Guns*, 2nd ed., 1891, p. 4.

gewehr," invented in 1838 by Dreyse. In this ignition was effected by a needle being driven right through the base of the cartridge into a disc of fulminating material. After a few rounds the rifle could not be fired from the shoulder in consequence of the escape of flame. The needles also rusted and broke. But in spite of its defects the gain in rapidity of fire caused it to be maintained as the general arm in the wars of 1848, 1866, and 1870.[1]

Chassepôt rifle.

The French adopted the Chassepôt in 1866. This was a considerable improvement upon the Prussian rifle; escape of gas at the breech was prevented by means of a rubber washer. About the same time a committee sat in England to decide upon a rifle, and finally selected that of Mr. Jacob Snider, an American. But at the suggestion of Colonel Boxer the cartridge case was made of brass instead of thin paper as in previous rifles. This not only greatly improved the accuracy of shooting, but effectually prevented the escape of gas at the breech.[2]

Safety fuse.

The old method of firing a blasting charge was to lay a train of powder up to it, or use a quill or rush filled with powder. The time taken by these to burn was very uncertain, however, and this caused numerous accidents in the mines. This led Mr. William Bickford, of Tuckingmill, Cornwall, in 1831 to devise his "miner's safety-fuse," wherein a continuous thin core of powder was contained in cable of jute and string.[3] This gradually came more and more into use, and the fuse was improved in quality as experience was gained in its manufacture. For use in wet places a special quality was made covered with tape and varnished. Soon after 1840 the Bickford fuse was adopted by the English military authorities. In 1836 a factory was started in America; in 1839 in France, and in 1844 in Germany. Before 1840 guttapercha-covered fuse had been adopted for blasting under water. Various modifications have since been invented, including fuse cased in metal, "Colliery Fuse," which emits no sparks,[4] and various sorts of "instantaneous fuse," which burn very rapidly and enable many shots to be fired simultaneously.

Shell fuses.

As stated in the last chapter the fuses of shell were originally arranged to be ignited by the flash of the powder charge in the gun. The invention of the percussion cap, however, made it possible to start the action of the fuse in another and more certain manner. In 1846 Quartermaster Freeburn, R.A., invented the first English time fuse started by the concussion of the discharge; and in 1850 Commander Moorson, R.N., brought forward the first percussion fuse which was actuated by the shock of impact of the shell.[5] These two types of fuse are still in use and are made to screw into either the

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[1] *Textbook of Small-Arms*, 1909, chap. ii.

[2] *Textbook of Small-Arms*, 1909.

[3] Eng. Pat. No. 6159 of 1831.

[4] Patented by Sir G. Smith in 1886.

[5] Hime, p. 245.

nose or base of the shell. Very often both methods are combined in a "time and percussion fuse." Shell were used with great effect by the Russian fleet against the Turkish at Sinope in 1853.[1]

Gun-cotton. 1846.

Gun-cotton was discovered in 1845–1846 by C. F. Schönbein, Professor of Chemistry at Basle, in the course of some experiments which he was making upon highly oxidized bodies, following up a train of thought suggested by his discovery of ozone in 1844. Pélouze had made an explosive in 1838 by the action of nitric acid on cotton, but he did not take the important step of mixing sulphuric with the nitric acid, and he did not make any practicable application of his explosive. Schönbein at once recognized its importance as an explosive and kept the method of preparation secret, whilst he endeavoured to sell the process to various Governments. He showed that when fired in a musket gun-cotton produced the same velocity as a much greater weight of gunpowder. Professor Böttger, of Frankfort-on-Main, discovered gun-cotton in 1846, independently of Schönbein, but he entered into an arrangement with him to share the profits of the invention. Several others, attracted by the great stir that was caused by the invention, endeavoured to make gun-cotton, and some of them succeeded, but Schönbein was able to maintain the start he had obtained. In the autumn of 1846 he came over to England and gave some very successful demonstrations at Woolwich and Portsmouth and before the British Association. In the name of John Taylor he took out a British Patent,[2] and he entered into an agreement for three years with John Hall and Sons that they should have the sole right to manufacture gun-cotton at their powder works at Faversham, and in return should pay one-third of the net profit with a minimum of £1000 down, and the same each year. But before a year had elapsed, on July 14, 1847, there was an explosion of the gun-cotton which destroyed the factory and killed twenty-one men. After this Hall and Sons refused to proceed with the manufacture. About the same time there were disastrous gun-cotton explosions at Vincennes and Bouchet, and these produced such an effect that no more gun-cotton was manufactured in England or France for some sixteen years.

Meantime Schönbein had offered his process to the German Union (Deutscher Bund) for 100,000 thalers, and a committee had been formed to consider the matter, on which Liebig represented the state of Hesse, and Baron von Lenk, who was secretary, represented Austria. This committee sat until 1852, but finally refused to buy the process, partly on political grounds. The individual members of the Union were then able to make separate negotiations, and at the suggestion of von Lenk, who had done most of the actual work of the committee, Austria acquired the process for 30,000 gulden.

von Lenk.

In 1852 the Emperor of Austria appointed a committee to inquire into the use of gun-cotton for military purposes, and with some interruptions this

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[1] Rusch, *S.S.*, 1908, p. 189.

[2] No. 11407, October 8, 1846.

continued to sit until 1865. In 1853 a factory was erected at Hirtenberg to carry out von Lenk's method of making gun-cotton. In this the purification was somewhat more thorough than in Schönbein's original process, for instead of merely washing with water until neutral, von Lenk washed for three weeks, then boiled with dilute potash solution for fifteen minutes, washed again for several days, impregnated the yarn with water-glass, and finally dried. Von Lenk also constructed some batteries of 12-pounder guns to be fired with gun-cotton cartridges. These guns were so much damaged by firing, however, that no other nations adopted them. About 1860 von Lenk introduced bronze guns, which were less liable to burst than iron ones, and these not only had a propulsive charge of gun-cotton, but also had shells containing a bursting charge of the same explosive. It was found, however, that these often burst in the bore, and this was evidently due to the very sudden shock of the discharge exploding the shell charge, for when gunpowder was used to fire the gun-cotton shells, no bursts took place. On July 20, 1863, the magazine at Hirtenberg exploded, and this seems to have finally decided the Austrian authorities to give up gun-cotton as a propulsive explosive, and von Lenk was then allowed to communicate the method of manufacture to other Powers. In 1865 another magazine exploded on Steinfelder Heath, near Vienna, and on October 11, 1865, the manufacture was officially stopped in Austria.

The further development then took place in other countries. Von Lenk made communications to the Emperor Napoleon III, and experiments were started in France. In 1864 he took out an American patent.

In 1862 and 1863 von Lenk took out patents in England in the name of Révy to protect his methods of purification,[1] and in the latter year he came over with the permission of the Emperor to describe his process to a committee of the British Association. The same year Messrs. Prentice and Co. started to make gun-cotton at Stowmarket by von Lenk's process, but an explosion occurred there soon after.

Abel.

Under the direction of Frederick Abel, the Chemist of the English War Department, manufacture was also started about the same time on a small scale in the Royal Gunpowder Factory at Waltham Abbey. This made it possible for Abel to carry out those experiments and researches which led to a revolution in the explosives industry, and have rendered gun-cotton one of the safest explosives in manufacture and use. In 1865 he took out a patent for pulping gun-cotton and pressing it into blocks,[2] and in 1866 and 1867 he published his *Researches on Gun-cotton*.[3] He showed that by pulping

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[1] Eng. Pats. 1090 of 1862 and 2720 of 1863.

[2] Eng. Pat. No. 1102 of 1865. The pulping of gun-cotton had, however, been carried out at Le Bouchet in France (*see* Escales, *Schiessbaumwolle*, 1905, p. 15).

[3] *Phil. Trans. of Royal Society*, 1866, p. 269; and 1867, p. 181.

gun-cotton in the same way as is done with rags, etc., in the manufacture of paper, he not only got it in a more convenient state for pressing into blocks, but the violent mechanical treatment removed much of the impurity, and the gun-cotton was reduced to a condition in which it was much easier to wash it thoroughly. The object of compressing the pulped gun-cotton was to restrain the violence with which it exploded in the gun,[1] but although it was better than von Lenk's yarn it was still uncontrollable; it damaged the guns, and the accuracy of the shooting was unsatisfactory. It was not until some seventeen years later that a successful smokeless military powder was made. Gun-cotton was therefore only used for blasting purposes. Prentice and Co., of Stowmarket, adopted Abel's process of purification and have continued to use it ever since. In 1868 its utility was much increased by the discovery by Abel's assistant, E. A. Brown,[2] that dry compressed gun-cotton could be caused to explode very violently by a detonator containing fulminate of mercury, this appliance having been already used by Nobel[3] for detonating nitro-glycerine. Brown afterwards made the further discovery that a slab of wet gun-cotton could be exploded by means of a small primer of dry gun-cotton. This rendered it possible to store the greater part of the gun-cotton in the wet state, a great advantage for military purposes, and in this field gun-cotton as originally prepared under the superintendence of Abel still holds its ground to some extent.

Other uses of nitro-cellulose.

In 1847 Maynard discovered that nitro-cellulose was soluble in a mixture of ether and alcohol, although it did not dissolve in either liquid alone, and this led to the invention of the collodion photography by Scott Archer in 1851, and to other uses of collodion. Celluloid, made by dissolving nitro-cellulose in camphor with the aid of heat and pressure, was patented by J. W. and I. S. Hyatt in 1870.[4] The artificial silk industry may be said to have started in 1884 when Count Hilaire de Chardonnet took out his first patent.[5]

Nitro-glycerine.

In 1846 nitro-glycerine was discovered by Sobrero, Professor of Chemistry at Turin, who had been assistant to Pélouze in 1838 when he made his first experiments on nitrating various bodies. But no practical application was made of it except that very small quantities were used in medicine as a cure for angina pectoris. People were no doubt deterred by the dangerous nature of the material and the inconvenience of dealing with a liquid explosive, as also by the difficulty in causing it to explode. In 1859 to 1861, however, Alfred Nobel and his father made experiments with it, and found that it could be exploded by means of a detonator containing fulminate, and in 1862

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[1] *See Chem. News*, 1866, (4) p. 250, and 1871, (24) p. 141.

[2] Eng. Pat. No. 3115 of 1868.

[3] Eng. Pat. No. 1345 of 1867.

[4] Amer. Pat. 105,338, July 12, 1870. *See J. Soc. Chem. Ind.*, 1914, p. 225.

[5] French Pat. 165,345.

commenced to manufacture it at Heleneborg, near Stockholm. Many serious accidents occurred in the transport and use of the explosive, and in 1864 an explosion took place at the Heleneborg works, which destroyed them, killed the head chemist and Nobel's brother, and caused his father a paralytic stroke from which he never recovered. Nobel, however, was not deterred by this, and proceeded to re-erect his factory at Vintervikken, and build a new one at Krümmel in Germany. He was convinced that nitro-glycerine was the most powerful explosive known or likely to be discovered, and he allowed nothing to prevent him turning its properties to profitable account. The continual catastrophes, however, caused the various states to pass laws restricting or prohibiting the transport and use of nitro-glycerine. In consequence Nobel searched for means to make the material safer and more convenient to handle, and discovered that kieselguhr had the power to absorb three times its weight of nitro-glycerine.[1] This, combined with the fulminate detonator mentioned in the same specification, produced a very convenient and fairly safe explosive. Nobel then proceeded to exploit his inventions, and he did this with such success that by 1873 fifteen factories had been built or founded in the various countries of Europe and America. In 1875 he made another important invention,[2] that of blasting gelatine. The nitro-glycerine in this explosive was solidified by having about 8 per cent. of collodion cotton dissolved in it. As compared with kieselguhr dynamite this has two advantages: the nitro-glycerine is not liable to be displaced from it by water, a defect which in the case of dynamite has led to many accidents; and, secondly, the substance added is itself an explosive, and consequently blasting gelatine is 25 per cent. more powerful than dynamite. Gelatinized nitro-glycerine containing a small proportion of collodion has been made a constituent of many blasting explosives, the nature of the other constituents and the method of preparation being modified to produce explosives suitable for blasting different sorts of rock and other materials.[3]

Ammonium nitrate explosives.

In 1867 the Swedish chemists, C. J. Ohlsson and J. H. Norrbin, took out a patent for explosives consisting of ammonium nitrate, either by itself or in admixture with other substances such as charcoal, sawdust, naphthalene, picric acid, nitro-glycerine or nitro-benzene. They were led to their invention by theoretical calculations, which showed that a very large amount of heat and gas was given off in the explosion of these mixtures. They selected the proportions so that all the carbon should be converted to carbon dioxide and the hydrogen to water. Considerable difficulty was, however, experienced in igniting the charges, and consequently they usually added some nitro-glycerine to assist the explosion. Afterwards they used the fulminate detonator. The explosive was used to some extent in Sweden. Early in the seventies Alfred Nobel bought up the invention and took out further patents in connexion with it, but great difficulties were experienced in consequence of the very hygroscopic nature of ammonium nitrate. Soon after this Nobel invented blasting gelatine, and he did not take much active interest in ammonium nitrate explosives for some time.

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[1] Eng. Pat. No. 1345 of 1867.

[2] Eng. Pat. No. 4179 of 1875.

[3] A very interesting account of Alfred Nobel and his inventions was contributed by de Mosenthal to the *J.S.C.I.* for 1899, p. 443.

Sprengel explosives.

In 1871 Dr. Hermann Sprengel, F.R.S., the celebrated inventor of the mercury vacuum pump, took out patents for a whole series of mining explosives to be made by mixing an oxidizing substance with a combustible one[1] "in such proportions that their mutual oxidation and de-oxidation should be theoretically complete." The essential feature was that the two constituents were to be mixed together on the spot just before the explosive was required, and the mixture was to be exploded by means of a fulminate detonator. As oxidizing agents he mentions amongst others nitric acid and chlorate of potash; as combustibles, a very large number of substances including nitro-benzene, nitro-naphthalene, carbon bisulphide, petroleum, picric acid. Liquid nitric acid is a most objectionable material to handle, nevertheless several inventors have taken out patents for Sprengel explosives containing nitric acid either enclosed in glass tubes or absorbed in fossil flour or other similar material.[2] Needless to say, they have never found favour. In addition to its other disadvantage there is the serious danger that the nitric acid may come in contact with the detonator and cause a premature explosion. This actually happened in 1884 to the inventor Punshon.

Sprengel explosives consisting of chlorate of potash and a liquid combustible material have, however, been used to a considerable extent. The American, S. R. Divine, took out a patent in 1880 for mixtures of this sort and several English patents in the following years.[3] One of these mixtures, under the name of Rackarock, was used on October 10, 1885, for the great blasting operation of Hell Gate in New York Harbour. On this occasion 240,399 lb. of Rackarock were used together with 42,331 lb. of dynamite.

There are considerable advantages in transporting separately two such inert substances as nitro-benzene and chlorate of potash, but against this must be put the difficulty and inconvenience of mixing the constitutents in the right proportions on the spot. If made up beforehand the cartridges are dangerously sensitive and become more so on keeping. Under the English Explosives Act this mixing is considered to constitute the manufacture of an explosive, and consequently may only be carried out in a duly licensed

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[1] Brit. Pats. Nos. 921 and 2642 of 1871. *Jour. Chem. Soc.*, 1873, p. 796; *S.S.*, 1907, p. 184.

[2] Hellhoff, Brit. Pats. 1315 of 1879, 1285–87 and 2775 of 1880. Punshon, Brit. Pats. 2242 of 1880, 2428 of 1883. Bichel, French Pat. 171,169 of 1885.

[3] Eng. Pat. Nos. 5584 and 5596 of 1881, 1461 of 1882, 5624–25 of 1883.

factory. Therefore Sprengel explosives have never been used in the British Isles, but they were introduced by the Americans into China and Siberia when the first railways were built there, and one of them is now licensed in Italy.[1]

Oxyliquit.

A somewhat similar explosive Oxyliquit, invented by Linde, consisted of liquid oxygen absorbed in wadding, charcoal, or other organic material. It was found that these mixtures would not detonate readily, so kieselguhr was substituted as absorbent with an addition of liquid petroleum. This detonated all right, but was more sensitive to a blow or spark than dynamite. It was very inconvenient to use, as the cartridge had to be fired within five or fifteen minutes of its preparation, according to the diameter. The explosive was tried in 1899 by a commission in Austria, and on a fairly large scale in the building of the Simplon tunnel, but in spite of the low cost of liquid oxygen it was found that the practical difficulties were very great.

During the War, however, the use of explosives of this class is being encouraged in Germany in order that the available supply of nitrates may be used as far as possible for military purposes. The industrial use of chlorate explosives is being extended for the same reason.

Picric acid.

Sprengel also drew attention to the fact that picric acid by itself could be detonated by a powerful detonator and was a very violent explosive, but no use was made of it in this way until many years later.

Coal-mine dangers.

The revival of ammonium nitrate explosives was due to the demand for such as would not ignite the fire-damp in coal-mines. Numerous disasters due to the explosion of fire-damp led to the appointment of commissions in many of the countries of Europe to inquire into the matter and propose remedies. The nature of the danger was investigated in 1815 by Sir Humphry Davy, and one source of disaster was removed by the substitution of the Davy lamp for the naked light. As time went on gunpowder was used more and more for breaking the coal, and after 1870 dynamite was also used. About 1873 Macnab proposed to insert a cylinder filled with water in front of the charge. Others have suggested wet moss, jelly containing 90 per cent. water and sawdust saturated with a solution of alum and sal ammoniac. But these devices were found to be cumbersome and expensive and not very effective.

Marcelin Berthelot.

In 1870, during the Siege of Paris, Professor M. Berthelot, who had hitherto devoted himself to pure science, was called upon to give his city and country the benefit of his scientific knowledge, and he was thus led to study the subject of explosives and especially to consider the amount of heat or energy liberated in the reactions which take place, for he had been working at thermochemistry for some years and was practically the founder of this branch of science. After the war was over Berthelot's services were still retained by the State in connexion with all matters connected with explosives. On the recommendation of the French Academy of Sciences he was in 1876 appointed a member of the Committee on "Poudres et Salpêtres." In order to deal adequately with the many new inventions he recommended the formation of a special commission. This was done in 1878, and Berthelot was appointed president of the new "Commission des Substances explosives," a position which he occupied for many years.

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[1] Guttmann, *Twenty Years' Progress*, p. 10.

In 1877 a commission was appointed in France to inquire into the question of ignitions of coal-damp. In the report which it made in 1880 it was obliged to admit that there was then no explosive known that would not ignite coal-damp. An English Commission which reported in 1886 was forced to come to an equally unsatisfactory conclusion.

Prussia.

In 1885 the Prussian Government built at Neunkirchen the first testing station for investigating mining explosives and adopted a method of testing, which with slight modifications has been copied by the Governments of England and several other nations. A long iron cylinder was filled with mixtures of coal-damp, coal dust and air, and the explosives were fired. At first the explosive was simply suspended in the gas mixture, and it was found that the gas was ignited every time. Afterwards it was fired from a small mortar without tamping, and it was found that under these conditions kieselguhr dynamite was safe up to 100 grammes, and gelatine dynamite up to about 80.

It was now that ammonium nitrate explosives came to the fore again, as experience showed that considerably larger charges could be used without igniting the gaseous mixture. Two of the first of these were roburite and securite, mixtures of ammonium nitrate with dinitro-benzene. But it was also found possible by suitable admixtures so to alter the character of nitro-glycerine explosives that they can be used in coal-mines with comparative safety. In 1885 Schmidt and Bichel introduced carbonite, a mixture of nitro-glycerine, saltpetre and flour. This has been able to hold its own to the present day and is still considered one of the best safety explosives.

France.

In 1887 another commission was appointed in France to inquire into the matter. Influenced by Berthelot's work and theories it directed its attention mainly to the question of the heat developed by an explosive and the resulting temperature of the products. Explosives having a high temperature of explosion, such as nitro-glycerine (3200°), gun-cotton (2600°) or collodion cotton (2060°), should be mixed with a substance having a low temperature of explosion such as ammonium nitrate (1130°). Three sorts of safety explosives were therefore introduced into France: Grisoutine, a mixture of ammonium nitrate and nitro-glycerine; Blasting Powder P, ammonium nitrate and collodion cotton; and Grisounite, ammonium nitrate and nitro-aromatic compounds such as nitro-naphthalene.

The great drawback to the use of ammonium nitrate is its hygroscopic nature, but the tendency to absorb water from the atmosphere has been overcome to a great extent by coating the grains with paraffin-wax or other waterproofing material, and by enclosing the cartridges in suitable envelopes.

England.

In Germany and England it has long been recognized that the temperature of explosion is only one of the factors in making an explosive safe or dangerous to use in fiery mines, and consequently reliance has been placed more upon trials in testing galleries, which are intended to imitate as nearly as possible the conditions in a mine. The North of England Institute of Mining and Mechanical Engineers appointed a Committee in 1888 to inquire into this matter. Their trial gallery at Hebburn Colliery was completed in 1892, and after experimenting with various explosives until 1895 they recommended the use of several, the majority of which were ammonium nitrate explosives.

It was upon the results obtained by this Committee that the Coal Mines Regulation Act of 1906 was founded. This Act, which is still in force, authorizes the Home Secretary to prohibit the use of any explosive in coal-mines, and to appoint Inspectors of Explosives to administer the Act. A testing gallery was erected in Woolwich Arsenal, and there all explosives had to be tested before they were permitted to be used in coal-mines in the United Kingdom. More recently a larger testing gallery has been erected at Rotherham, where tests can be carried out on lines more nearly approaching those that have been adopted in Germany and Belgium.

Cheddite.

In 1897 F. A. G. Street took out patents for explosives consisting of potassium chlorate mixed with castor-oil in which aromatic nitro-compounds are dissolved. The great sensitiveness of chlorate mixtures is thus overcome. The explosive thus produced is called Cheddite, and is used largely in England, France, and Germany.

Inspection of explosives.

In 1875 was passed the English "Explosives Act," which has had such a great influence upon the development of the explosives industry. Its form was largely due to the late Colonel Sir V. D. Majendie, who was appointed the first permanent Inspector of Explosives to administer its provisions. The necessity for legislation was revealed by an explosion at Messrs. Ludlow's, at Birmingham, which caused the death of fifty-three persons. Colonel Majendie was instructed to report upon it. Previous to this there had been many other accidents large and small, including that of two powder magazines on the banks of the Thames at Erith, which killed thirteen people and did great damage to property.

The Inspectors of Explosives were given power to inspect all magazines and factories and see that operations are carried out in a reasonably safe manner. As a result the number of deaths in explosives factories has been very greatly reduced in spite of the fact that the number of people employed is several times as great:

	Average number killed per annum in explosives factories
1868–1870	43
1871–1874	32
1878–1887	7·5
1888–1897	5·2
1898–1907	6·9
1908–1914	9·0

By the wise and tactful manner in which they carried out their duties Colonel Majendie and his colleagues conferred this great benefit upon the employees in the explosive factories without in any way seriously interfering with the development of the industry. In fact, the precautions, which the inspectors insisted on, have been advantageous to the shareholders as well as to the workpeople and the general public. In 1898 Sir Vivian Majendie died in harness, but the work has been carried on in the same spirit by his successors, Colonel Ford, Captain J. H. Thomson, and Major A. Cooper-Key, and the other Inspectors of Explosives working under them. The provisions of the English Explosives Act of 1875 have been largely adopted in the legislation of foreign countries, British Colonies, and India.

Smokeless powders.

It has already been pointed out that the early attempts of von Lenk and others to make a satisfactory smokeless powder from gun-cotton were unsuccessful because it was much too violent in its effects. The gun-cotton being in a state of fine fibre interspersed with air spaces the explosion travelled through it almost instantaneously. Black powder, on the other hand, being a mechanical mixture, the explosion can only start at the points where the particles of saltpetre are in actual contact with the particles of sulphur and charcoal, consequently the time of explosion is comparatively long.

Schultze powder.

The first successful smokeless powder was that of Major Schultze, of the Prussian Artillery. First, he appears simply to have impregnated little grains of wood with saltpetre,[1] but afterwards he purified the wood to some extent by washing, boiling and bleaching it, and then nitrated it, and purified the nitrated lignose by much the same process as that used by von Lenk for gun-cotton. The grains thus obtained were then impregnated with saltpetre, alone or mixed with barium nitrate.[2] This was introduced about 1865. **1865.** According to an analysis published by Cundill in the *Dictionary of Explosives* the nitro-lignin contained more than 20 per cent. of unnitrated lignin. This and the different physical structure of wood as compared with cotton made the material burn more slowly in the gun, and the rate was still further reduced by the addition of the nitrates of potassium and barium. The explo-

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[1] Sanford, *Nitro-Explosives*, 1896, p. 173.

[2] Eng. Pat. 900 of 1864.

sive was still too violent for rifles, however, but was found to be quite suitable for shot-guns. The Austrian rights to Schultze's invention were acquired by a firm called Volkmann's K.K. priv. Collodinfabriks Gesellschaft, H. Pernice and Co. Volkmann took out Austrian patents in 1870 and 1871, which were kept secret at the time, but Guttmann obtained copies of them and published translations in his *Twenty Years' Progress in Explosives* (Whittaker, 1909). From these it is seen that Volkmann had made the further step of partly gelatinizing the grains by treating them with a mixture of ether and alcohol, whereby the explosion would be restrained still more. This powder was made under the name of Collodin from 1872 to 1875, but then the Austrian Government stopped the manufacture on the ground that it was an infringement of their gunpowder monopoly. The nitro-lignin made as described by Volkmann must have been decidedly impure and, therefore, unstable, and difficulties were no doubt experienced in obtaining uniform results.

A company was formed in England in 1868 to work Schultze's invention, and a factory was established at Eyeworth, in the New Forest, in 1869, and this after a time achieved great success after the methods had been altered by Griffiths. By 1881 Schultze powder had become so popular with sportsmen on account of the light recoil and absence of smoke as compared with black powder, that the London gun-makers found irksome the restrictions upon the quantities they were allowed to store. The manufacturers of this powder have modified their methods from time to time to meet the demands of sportsmen and to keep abreast of the general advance in the technology of explosives, so that the Schultze powders are still amongst the best. In 1883 Schultze started a factory in partnership with Voltz and Lichtenberger at Hetzbach in Hesse-Darmstadt, and powder is still made there under Schultze's patents.[1]

E. C. powder 1882.

The next successful smokeless powder was invented at the works of the Explosives Company at Stowmarket, which formerly belonged to Thos. Prentice and Co. It was protected by patent No. 619, taken out in 1882 by Walter F. Reid and D. Johnson. This was called E. C. Powder (Explosives Company), and consisted of nitro-cotton mixed with the nitrates of potassium and barium with the addition of colouring-matter and small quantities of other organic compounds. It was made into grains which were hardened by being partially gelatinized by means of a solvent, ether-alcohol. A separate company was formed to work the invention, and a factory was started at Green Street Green, near Dartford, in Kent. This is still in existence, and E. C. Powder continues to be much used.

Gelatinized powders.

For use in rifled fire-arms these powders are too quick. For this purpose it has been found necessary to destroy entirely the structure of the original cellulose by thoroughly gelatinizing it. The first to produce a good smokeless

rifle powder was the French engineer Vieille, working on behalf of the French Government in 1884. He incorporated the nitro-cotton with ether-alcohol in a machine such as is used for kneading bread. The resulting paste was rolled out into thin sheets and cut into small squares and dried. The powder was called Poudre B, after General Boulanger. In 1889 a gelatinized nitrocellulose flake powder was introduced in Germany.[1] In 1888 Nobel invented a powder called Ballistite, consisting of a nitro-cotton of low nitration gelatinized with nitro-glycerine, and in the same year an English committee adopted Cordite, a mixture of highly nitrated gun-cotton, nitro-glycerine and vaseline (mineral jelly), gelatinized by means of acetone. A nitro-glycerine powder of the cordite type was adopted in Austria-Hungary in 1893.[2]

Every nation now uses propellants consisting principally of gelatinized nitro-cotton either by itself or mixed with nitro-glycerine. These gelatinized powders when suitably ignited in the gun burn from the surface inwards, consequently the time of explosion can be increased by making the individual pieces of explosive bigger. Without altering the composition powders can be produced suitable for every sort of rifled fire-arm from a pistol to a 14-inch gun.

Picric acid.

Picric acid has been used for a long time as a dye, and was in fact the first artificial dye to be discovered, for its formation was observed in 1771 by Woulfe by the action of nitric acid on silk. Laurent was the first to make it in 1843 by the nitration of phenol and dinitrophenol and to recognize that it is trinitrophenol. The fact that picric acid combines with metals and bases to form explosive picrates has also been known for a long time, and when its manufacture from phenol had reduced its price many mixtures containing them were proposed. Picric acid also was added to explosives as a combustible constituent. None of these mixtures were used to any great extent, however. In 1871 Sprengel demonstrated that picric acid by itself could be detonated by means of fulminate,[3] but this also led to no practical result until E. Turpin in 1885 pointed out the great advantages of using picric acid for filling shell,[4] in consequence of its stability, insensitiveness and violence. This was adopted by the French Government under the name of Melinite. Other high explosives are mostly too sensitive to use in shell : they are liable to explode in the bore of the gun from the shock of discharge. For this reason gunpowder only was previously used, and it still forms the bursting charge of shrapnel shell and other sorts which only require a moderate disruptive

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[1] A. Voigt, *Herstellung der Sprengstoffe*, i., p. 116.

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[1] Von Neyman, *Jahrbuch der Armee und Marine*, Dec. 1914, *S.S.*, 1915, p. 145.

[2] Grotouski, *Mitt. Art.-und Geniewesen* through *S.S.*, 1914, p. 386.

[3] Eng. Pat. 2642 of 1871 ; *J. Chem. Soc.*, 1873, p. 796.

[4] French Pat. 167,512 of Feb. 7, 1885, with additions Oct. 17, 1885, and Sept. 1, 1892. Eng. Pat. 15,089 of 1885. Germ. Pat. 38,734 of Jan. 12, 1886.

power. Picric acid, however, is nearly as insensitive as black powder and can therefore be used with safety for shell. In fact, it requires a very powerful detonating primer to ensure complete detonation. With various modifications picric acid was adopted in almost every country for this purpose. It has the disadvantage, however, that it readily forms picrates if it comes in contact with metals or earthy materials, and these picrates are much more sensitive than picric acid. Its melting point also is inconveniently high.

In his patents Turpin pointed out that the sensitiveness could be reduced still further by compression, or by mixing the picric acid with heavy oils or with collodion. At first collodion was used in this way, but later the acid has generally been used by itself, either in compressed blocks or melted and cast directly into the shell. In 1911 the Civil Court in Paris granted Turpin 100,000 francs in compensation because he had not been permitted to utilize his invention to his own profit.[1] In 1888 picric acid was adopted by the German Army both for filling shell and for military blasting, and about the same date shell were filled in England with molten picric acid under the name of Lyddite, derived from Lydd, the place where the experiments were carried out.

Trotyl. In 1904 the Germans commenced to use trinitrotoluene, otherwise trotyl, instead, as it is free from the disadvantages of picric acid referred to above. This is now used very largely for high explosive shell, and is also often mixed with other substances to form complex explosives.

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[1] *See S.S.*, 1912, p. 57.

PART II

BLACK POWDER

CHAPTER IV

MANUFACTURE OF SALTPETRE

Nitre deposits : French saltpetre industry : Artificial nitre beds : English saltpetre industry : Formation of nitrates : Berthelot's researches : Bacterial action : Indian saltpetre industry : Indian refinery : Chili nitrate deposits : "Conversion" saltpetre : Refining saltpetre : Saltpetre from the atmosphere

So far as is possible in the almost entire absence of all records, an account has been given in Chapter I of the first discovery of saltpetre.

Nitre deposits.

Until the middle of the nineteenth century all saltpetre was obtained by dissolving it from earth and deposits in cellars and caves and similar places, where it had formed naturally. In Europe there are very few localities where nitrate can accumulate in the soil to such an extent that a profit could be made by extracting it. There is no prolonged dry season during which deposits can form without being washed away again. Consequently saltpetre could only accumulate in sheltered places, such as cellars and stables, especially those in which there was much nitrogenous matter undergoing decomposition. As it was of the utmost importance in every country to have a sufficient supply of saltpetre, especially in time of war, its production formed the subject of royal decrees and orders at an early date. In France, officers (salpêtriers commissionés) were appointed in 1540 to search for and extract saltpetre, and no doubt the industry was in existence some time before.[1] This edict was confirmed and renewed in 1572, and again whenever France was waging a serious war. The saltpetre workers operated on the earth of stables, sheep-pens, cattle-sheds, cellars and pigeon-houses, and on the plaster and rubbish removed when houses were pulled down. They had the right to gather material everywhere, with scrapers and brushes in the houses, with picks and shovels in places not inhabited. No building or wall could be pulled down until notice had been given to the saltpetre workers, who stated which parts they wanted reserved.

French saltpetre industry

In the eighteenth century the saltpetre workers in France received many additional privileges. For instance, they could set up their vats and other plant in public halls, private courtyards or wherever they thought fit. The

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[1] Berthelot, *Sur la Force des Matières explosives*, 1883, vol. i., p. 345 *et seq.*

local authorities had to supply the wood required for heating, and provide carts for transporting the plant and the saltpetre to the refinery. As a rule each locality was visited once every three years.

Artificial nitre beds.

Saltpetre was also obtained from artificial nitre-beds, consisting of earth mixed with animal and vegetable matters, ashes, refuse of buildings, lime and marl. This was all placed in a large barn, and collected in heaps mixed with twigs and intersected with holes to allow access of the air. It was turned over also from time to time and watered with urine. Nitrate gradually formed in the mass and was extracted with water. There were many modifications of this process adopted in different places.

In the reign of Louis XIII (1610 to 1643) the annual crop of saltpetre amounted to 3,500,000 lb., but it gradually diminished in the eighteenth century largely on account of the strong objection the people naturally had to the presence of saltpetre workers in their houses and domains. In 1775 the quantity had fallen to 1,800,000 lb., and half the annual requirement was imported from India. If it had not been for the many privileges the nitrate workers enjoyed, the home product could not have competed at all with that imported from India. In 1789, the year of the fall of the Bastille, a great effort was made, however, to revive the industry, and 3,000,000 lb. were obtained. In 1791, however, the National Assembly proposed to abolish the privileges of the saltpetre workers, but war broke out, the harbours of France were blockaded, and it became necessary to produce in the country all the saltpetre for the powder required. The recent increase of chemical knowledge and the hearty co-operation of the greater part of the population made it possible to produce 16,000,000 lb. in a single year, and 5,000,000 in the next. The whole organization was placed under the control of the department of "Poudres et salpêtres," which still continues to regulate matters concerning explosives.

When peace was finally re-established, the renewed competition of Indian saltpetre dealt a severe blow to the industry in France, and in 1840 the bounties were abolished, but it struggled on until the exploitation of the sodium nitrate deposits in Chile and the potash deposits in Germany in the second half of the nineteenth century led to the production of artificial saltpetre. The consequent reduction of price almost entirely killed the French saltpetre industry, and in 1870, when a scientific committee was engaged in providing Paris with all stores necessary for its defence, Berthelot could find only one or two small producers in Champagne.

English saltpetre industry

Until the sixteenth century saltpetre seems mostly to have been imported into England, much of it coming from Spain, but in 1515 Hans Wolf, a foreigner, was appointed to be one of the King's gunpowder makers in the Tower of London and elsewhere. He was to go from shire to shire to find a place where there is stuff to make saltpetre of, and "where he and his laborers shall labor, dig or break in any ground." He is to make compensation to its owners. And in 1531 Thomas à Lee, one of the King's gunners, was appointed principal searcher and maker of gunpowder.[1]

As already stated gunpowder was only manufactured in England on a small scale until the second half of the sixteenth century, when George Evelyn started mills on a comparatively large scale. Consequently there was little difficulty before that time in obtaining sufficient saltpetre, but then it became necessary to grant the saltpetre men special privileges for digging up the floors of stables, dovecots and even private dwellings, and the kingdom was divided into a number of areas in which the collection and working of the saltpetre was assigned to various people. In 1561 Queen Elizabeth granted Gerard Honrick, a Dutchman, £500 (or £300) for teaching two of her subjects how to make saltpetre.[2] In 1588 she granted a monopoly for gathering and working saltpetre to George Evelyn, Richard Hills and John Evelyn. The monopoly extended over the whole of the South of England and the Midlands, except the City of London and two miles outside it. In 1596 Robert Evelyn acquired the rights in London and Westminster from the licensees there. As a rule, however, the Evelyns did not work saltpetre themselves, but bought it from the saltpetre men.

In the reign of Charles I there was considerable friction between the saltpetre men and the public, but it was probably due more to the weakness of the Crown than to any real difficulty in obtaining in England the quantity of saltpetre required, viz. 240 lasts per annum. There was also competition between the saltpetre men and the soap-boilers for wood ashes, which were then practically the only source of potash and were required for the conversion of sodium nitrate into the potassium compound. In 1634 the Lords of the Admiralty gave orders to the Governor and Company of soap-boilers that the saltpetre men were to have the pre-emption of wood ashes, on the ground that saltpetre was a commodity of such necessary use for the King and public that it ought to be preferred before the making of soap.[3] The monopoly of saltpetre was abolished in 1641 at the same time as the monopoly of gunpowder. It was revived for a time after the Restoration, but manufacture then was on a considerably larger scale.

The East India Company, then in its infancy, imported Indian saltpetre into England as early as 1625 and set up a powder mill in Windsor Forest, which, however, was stopped on the ground that it interfered with the King's deer. Next year the Company received a license to erect mills in Surrey, Kent and Sussex. At this time its importations were on a small scale, but when its charter was renewed in 1693 it was stipulated that 500 tons of saltpetre should be supplied every year to the Ordnance. Ever since then, Indian saltpetre has been used very largely in England for the manufacture of gunpowder.

[1] *Brit. Exp. Ind.*, p. 185.

[2] *Brit. Exp. Ind.*, pp. 210, 258.

[3] *Brit. Exp. Ind.*, p. 269.

Formation of nitrates.

Whence come these large quantities of nitrates? The nitrogen of the atmosphere does not form chemical compounds at all readily. Under the influence of lightning, and high tension electricity generally, small quantities of nitric acid are formed and these are carried down by rain into the soil and rendered available for plant life. But Lawes and Gilbert in their researches at Rothamsted found that the amount of nitrogen removed with the crops was more than the total sum of that added as manure and brought down by the rain together with the loss of nitrogen from the soil. No plants can grow without absorbing nitrogen compounds from the soil; and when animal and vegetable matters decompose, a portion of the combined nitrogen is liberated as nitrogen gas, but in spite of this steady loss there is no indication that the surface of the earth as a whole is becoming poorer in combined nitrogen.

Berthelot's researches.

Berthelot investigated this matter about 1876 and discovered that nitrogen and oxygen also combined under the influence of electricity of quite low tension, such as that yielded by two or three electric cells, although only very slowly, and moreover that in the presence of carbohydrates such as dextrine the combination was considerably more rapid.[1] Although the quantities thus obtained in small laboratory experiments only amounted to a few milligrammes in several months, yet, as there are always differences of potential of this order over the whole surface of the globe between the earth and the air above it, he considered that this phenomenon was sufficient to account for all the nitrates and other compounds of nitrogen that are formed.

Bacterial action.

Since then, however, it has been found that this is by no means the only cause of the formation of nitrogen compounds from the nitrogen of the atmosphere. There are bacteria in the soil, which can take up nitrogen from the air and cause it to combine with other elements to form nitrates and more complex bodies such as albuminoids. Some of these, such as Azobacterium Chroococcum Beijerinck, can live and act freely in the soil; others seem only to exist in the nodules that are found on the roots of certain sorts of plants. The Bacterium Radicicola is perhaps the most important of these; it is found on the roots of the leguminosæ (beans, peas), and renders it possible for these crops to grow on otherwise sterile soil, which afterwards is able to support other crops. Some other plants such as the alder tree have similar nodules on their roots. There are some soil bacteria also, which convert nitrates into nitrites, others carry out the reverse change, others again liberate nitrogen from nitrates or nitrites, and several of these actions may be going on simultaneously in the soil.

Bacterial action proceeds most rapidly in a warm moist climate like that of Bengal. The accumulation of nitrate to such an extent, that it can readily be collected in large quantities, also requires that there shall be regular seasons of the year when little or no rain falls. In this respect, Bengal and most other parts of India satisfy the conditions. It was found by Leather that at Pusa in Behar the nitrification takes place mostly in the top 6 or 12 inches of soil and principally at the commencement of the rainy season. There is more nitrate in fallow soil than in that that is covered with crops. The mean quantity that was washed out of the soil into the drain gauges was 70 lb. of nitric nitrogen per acre from fallow soil and 13 lb. from cropped soil.[1] Headden has found that in Colorado soils are often rendered sterile by the presence of too much saltpetre, which at times amounts to 6 per cent. of the soil. It is formed by azobacter.[2]

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[1] *Sur la Force des Matières Explosives*, 1883, vol. i., chap. vi.

Indian saltpetre industry.

Bihar is the principal seat of the saltpetre industry in India, but considerable quantities also come from the United Provinces and the Punjab, and smaller amounts from other parts of India and from Burma. Except what is consumed in the country, the greater part is exported from Calcutta. Fifty or sixty years ago, the average quantity exported was over 30,000 tons per annum, now it is 18,000 to 20,000. In the places where the nitrous earth is collected the natural vegetation is scant, as the soil in many cases is too salt for crops to grow even during the rains. It is obtained in and around existing village sites and on the mud walls of houses and cow-sheds. In the rainy season, lasting from June to October, the process of nitrification goes on in the warm moist soil, assisted by the addition of nitrogenous refuse. The following account by R. W. Bingham[3] gives a clear picture of the industry in the last century:

"It is all made by a peculiar caste called 'nuniah,' and, so far as my experience shows, is principally in the hands of . . . 'mahajuns,' who make yearly advances, charging 12 per cent. for the same. The 'nuniahs' are a tolerably safe class, compared with the ordinary 'riot' (peasant), to deal with, and pay the 'zemindars' (landowners) a comparatively large price (if measured by the 'bigah') for the old walls and old sites in which they revel. The supply of saltpetre from these old sites appears to be practically inexhaustible; for we find the 'nuniah' very busy making up his piles just after the setting in of the rains. This earth he exposes to the sun and rain, and takes care, by erecting walls, etc., that the precious stuff is not wasted away. A casual visitor would not be able to understand what he is after, but when the hot suns of April, May and June come on, then himself and his family boil merrily away, and eliminate saltpetre and salt from this apparently useless soil. Then the 'mahajan' is on the look-out and secures the saltpetre as it is made, and carries it to his own refinery for final manipulation; while the salt which is

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[1] J. W. Leather, *Mem. Dep. Agr. in India*, vol. ii., no. 2, Jan. 1912.

[2] W. P. Headden, Colorado Agr. Col. Expl. Station, *Bulletins* 155, 160, through *Nature*, 1911, p. 364. *J. Ind. Eng. Chem.*, 1914, p. 586.

[3] *Jour. Agricultural and Horticultural Soc.*, xii., p. 107, old series; *Dict. Economic Products of India*, S. 686, vol. vi. part ii. p. 437.

always bitter, and I should say unwholesome, under the name of 'khari nimuk' is sold to the lowest classes of the community at a cheap rate. The business must be a profitable one, as the large bankers of Ghazipore, Patna and Benares are always ready to go into the trade, and to advance money to responsible middlemen. . . . Sometimes these men experience considerable trouble in recovering their advances, but in that case they quietly walk off with the bullocks of the 'nuniah' who . . . never dreams of making a complaint, but begs or borrows from his comrades and friends till he has got money enough to release them by paying back principal and interest; well knowing that he will get no more advances, and will, besides, be put out of caste by his caste-mates if he does not, at all events, pay the original advance. If, on the contrary, he makes more saltpetre than will cover his advance, and he has no particular ceremony going on, he will clandestinely sell his partially refined saltpetre to other petty purchasers, and get drunk while the money lasts, and

Fig. 3. Percolators for Extracting Indian Crude Saltpetre.

ask contemptuously, 'What, am I a poor man that I should work?' The trade is too hazardous a one, and the petty advances spread over too wide an extent of country, to make it worth the while of Europeans with capital to attend to; in consequence it is almost wholly in the hands of the large houses above named (who are connected with Calcutta native firms, and who in turn have their small branches in every petty town in the district)."

The industry is conducted on the same lines now as then, except that it is not as remunerative as it used to be. Hooper[1] made analyses of a large number of samples of the earth collected by the "nuniahs": the amount of nitrates in them varied from 1 to 27 per cent., but as a general rule they contain 3 to 5 per cent., also several per cent. of sodium chloride and sulphate. A description of the process of extraction has been given by Leather and Mukerji.[2]

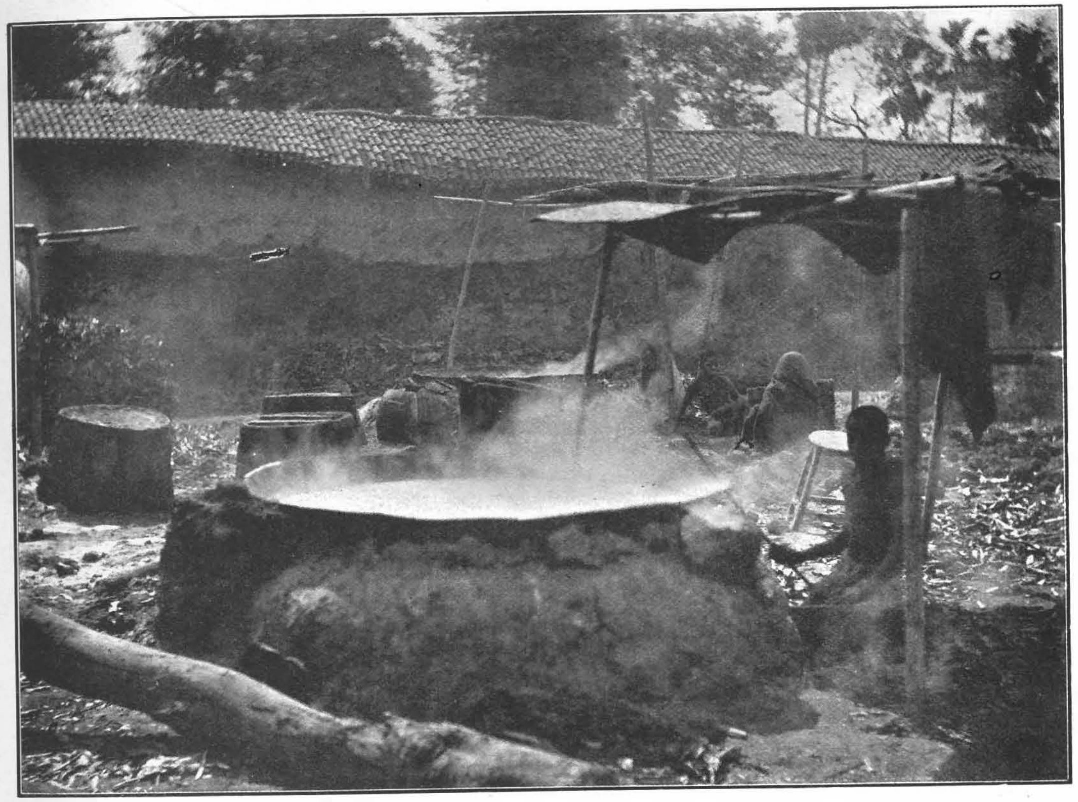

Fig. 4. Evaporating Liquor from Percolator.

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[1] *Agricultural Ledger*, 1905, No. 3.

[2] *Bulletin* No. 24 of the Agricultural Research Institute, Pusa, 1911; *S.S.* 1912, 116 and 136.

The "nuniah" builds an earthen chamber called the "kuria" or "kothi" with wet mud, which is allowed to dry. This chamber (*see* Fig. 3) has either circular walls some 5 or 6 feet in diameter, or oblong walls, and a floor which slopes slightly from back to front. In the front wall is a hole at the level of the bed, which allows the nitrate liquor to drain away into a pot. Above the bottom of this earthen chamber a false bottom is laid, consisting of bamboos and matting placed on a few loose bricks. The nitrate earth is filled in on this with great care. Stones, etc., are removed from it as far as possible, and it is put in slightly moist and trodden down so as to leave no channels, through which the water would run too rapidly. Wood ashes are generally mixed with the earth, so that the potash in them may convert into saltpetre the nitrates of lime and magnesia. A small piece of matting is placed over the top of the earth and water is poured on cautiously. The liquor that percolates through first is fairly strong: it is transferred to shallow earthenware or iron basins and evaporated down (Fig. 4) by means of a fire of wood, leaves or twigs, or in the Punjab to shallow masonry trays in which the concentration takes place through the action of the very dry air and the heat of the sun. The weak liquor that percolates through afterwards, is thrown on to a heap of already extracted earth, where it evaporates: this earth is afterwards extracted again. The strong liquor is boiled down until crystals begin to appear, and then is allowed to cool; the crystals are then fished out, fresh liquor is added to the mother liquor and the concentration is carried out as before. The composition of the crude saltpetre thus produced varies considerably. Hooper gives the analysis of fifty-five samples, and of these the following have been selected by Leather as being typical:

	Farukhabad		Okara		Mozaf-ferpore	Burhan-pura
	I	III	I	III		
Potassium nitrate	66·07	44·92	53·00	26·86	49·36	68·40
Calcium nitrate	—	—	2·60	—	3·28	2·60
Magnesium nitrate	2·54	4·80	—	12·24	7·44	2·12
Sodium chloride	21·84	35·38	34·22	34·80	16·82	17·98
Sodium sulphate	3·65	10·00	3·88	11·20	14·60	3·40
Insoluble matter	·90	1·20	1·10	1·40	1·50	1·70
Water	5·00	3·70	5·20	13·50	7·00	3·80
	100	100	100	100	100	100

No attempt is made to separate the sodium chloride at this stage because there is an excise duty on it, and the salt department only allows the recovery to take place in the refineries, where a proper control can be kept. Some of the crude saltpetre is used as manure, but the greater part goes to the refinery.

Indian refinery.

In the refinery the processes are very similar to those carried out by the "nuniah." There is always a large heap of saltpetre earth, which is worked over and over again, the weak liquors being always thrown on to it. This is extracted in "kothias," but the strong liquors are not evaporated down by themselves; crude saltpetre is dissolved in them at the boiling-point. The quantity added is such that the potassium nitrate is all dissolved to form a boiling saturated solution, whereas the greater part of the sodium chloride remains undissolved (*see* Table of Solubilities on p. 63) together with the insoluble matter. The hot liquid is allowed to settle for a little while, and then run into wooden vats where it is allowed to cool slowly and deposit crystals of potassium nitrate. The residue in the dissolving tank is washed with water to recover the saltpetre in it, and the common salt may be purified by dissolving it in weak nitre liquor, decanting off and evaporating down. The insoluble matter and all weak liquors are added to the heap of earth, which steadily grows from year to year. The mother liquor from the crystallization of the saltpetre is also added to it after it has been used three or four times, as it is then too impure.

The refined saltpetre is in large crystals of a brownish colour. To purify it further and improve the colour it is sometimes subjected to a washing process: it is put in sacks over wooden tubs and cold water is poured through it. This of course dissolves some of the potassium nitrate as well as the impurities, and is consequently returned to the refinery process. Leather and Mukerji give the following analyses of refined saltpetre before and after washing:

	Burhanpura		Savan		Bakramau	
	Un-washed	Washed	Un-washed	Washed	Un-washed	Washed
Potassium nitrate	90·70	94·91	81·98	91·55	88·63	94·70
Potassium sulphate	—	—	5·44	·93	·15	·15
Sodium sulphate	·91	·03	—	—	—	—
Potassium chloride	—	—	2·59	2·51	6·06	2·67
Sodium chloride	5·40	3·12	7·05	1·68	·07	·10
Sand	·30	·20	·20	·35	—	—

Leather has designed a simple plant on more up-to-date lines to carry out the refining process, and is endeavouring to get the Indian refiners to take it up. This consists of a dissolving vessel provided with a stirrer, a filter in which the liquor is rapidly filtered at a high temperature, and a series of coolers in which the saltpetre is caused to crystallize rapidly. The crystals are then freed as far as possible from the mother liquor in a centrifugal machine. The saltpetre produced has a purity of 90 to 93 per cent.

Chili nitrate deposits.

The plain of Tamarugal in Chili is even more favourably situated than Bihar or Bengal. It lies between the Andes and the comparatively low Coast Hills at a height of about 3000 feet above the sea within the tropics. As a rule there is very little rain there, but about once in six or seven years the plain, which is about 45 miles wide, is flooded. The plain slopes gently towards the Coast Hills and as there is no outlet for the water, it collects there and evaporates, and all the nitrate it has dissolved from the entire plain is deposited in a comparatively narrow area. The entire product of the bacterial action upon many hundreds of square miles for many centuries is found in the Chili nitrate beds. As the soil contains sodium compounds and comparatively little potassium, it is principally sodium nitrate that has been desposited.[1]

The following gives the results of analysis of commercial Chili nitrate:

Sodium nitrate	94·20
Potassium nitrate	1·51
Sodium chloride	1·06
Sodium iodate	0·01
Potassium chlorate	0·26
Magnesium sulphate	0·26
Magnesum chloride	0·32
Calcium sulphate	0·07
Insoluble	0·16
Water	2·15
	100

During the Crimean War (1854–1855), the demand for saltpetre was so great that the existing sources of supply in Europe and India did not suffice, and considerable quantities were made from Chili nitrate, which had been supplied to Europe in constantly increasing quantities since 1830. The salt beds at Stassfurt, however, did not commence to yield potassium chloride (Carnallite) until about 1863, therefore other sources of potash had to be used to convert the sodium nitrate from Chili into the corresponding potassium salt. The only sources of potash then available were kelp, and the ash of wood, etc. When the war was over, saltpetre prepared in this way could no longer compete with the natural product from India. But shortly afterwards fresh sources of potash were found in "suint," the dried sweat of sheep, which is washed from the wool, and in the cinder of "vinasse" (Schlempekohle), which is obtained as a by-product in the refining of beet sugar. With the development of the Stassfurt potash industry these lost their importance however.

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[1] *See* Newton, *J. Soc. Chem. Ind.*, 1900, p. 408.

"Conversion" saltpetre.

Large quantities of potassium nitrate are now made by the interaction of Chili nitrate and commercial chloride of potash, which is made by lixiviating "carnallite," a double chloride of potassium and magnesium, occurring in immense deposits near Stassfurt in Germany. In the heated and concentrated mother liquor from a previous operation commercial sodium nitrate (about 95 per cent. purity) and potassium chloride (not less than 80 per cent. purity) are dissolved, the nitrate being in slight excess. Of the four salts that might be present in the solution thus formed, sodium chloride has the least solubility at a high temperature and potassium nitrate the greatest (*see* Table below). At a low temperature potassium nitrate has the lowest solubility. The figures are of course for pure salts dissolved in distilled water, and the presence of other substances in solution would alter the solubilities somewhat, but the figures given in the last two columns, the solubilities of sodium chloride and potassium nitrate in water which is simultaneously saturated with both salts, show that in this case the salts have little effect upon one another's solubility. Consequently in the hot concentrated solution of sodium nitrate and potassium chloride most of the sodium chloride is precipitated out:

$$NaNO_3 + KCl = KNO_3 + NaCl$$

TABLE OF SOLUBILITIES

Temperature	Gramme mols. per 1000 g. water					
	One salt only present				When water is saturated with KNO_3 and NaCl	
	NaCl	KCl	$NaNO_3$	KNO_3	KNO_3	NaCl
0° C.. . .	6·10	3·70	8·54	1·31	1·8	5·6
20 . . .	6·14	4·56	10·3	3·12	3·4	6·5
40 . . .	6·24	5·36	12·2	6·32	6·0	6·4
60 . . .	6·36	6·11	14·5	10·9	9·9	6·0
80 . . .	6·53	6·85	17·4	16·7	16·5	6·2
100 . . .	6·75	7·60	20·9	24·3	28·0	6·9
120 . . .	6·84	8·43	25·1	38·9	38·0	7·2

Calculated from figures in Seidell's *Solubilities of Inorganic and Organic Substances*, 1907. A "gramme-mol." is the molecular weight of a substance in grammes. To find the actual weight of the salts dissolved by 1000 parts of water, multiply the above figures by the corresponding molecular weights: for NaCl 58·5, KCl 74·6, $NaNO_3$ 85·1, KNO_3 101·1.

The solubilities of these different salts in the presence of one another have recently been investigated by J. W. Leather and J. N. Mukerji, whose results do not differ very greatly from the above. At temperatures below 30° C., however, they found that a small proportion of KCl is formed in a solution saturated with KNO_3 and NaCl, and a corresponding amount of solid $NaNO_3$ is deposited. At temperatures above 30° C. on the other hand KCl is deposited and $NaNO_3$ formed in solution, and as the temperature rises the quantities of these salts increase rapidly.[1]

The liquid is boiled for half an hour to complete the reaction as far as possible, then it is run through a filter into shallow cooling tanks; some more water may now be added with advantage to prevent sodium chloride separating out with the potassium nitrate.

The solution is kept stirred whilst it cools, so that the potassium nitrate may separate in small crystals, which do not contain so much mother liquor as large ones. The crystals are drained and then washed with the liquors from the next crystallization. This is best done in a centrifugal machine, as the quantity of washing liquor is thereby reduced to a minimum.

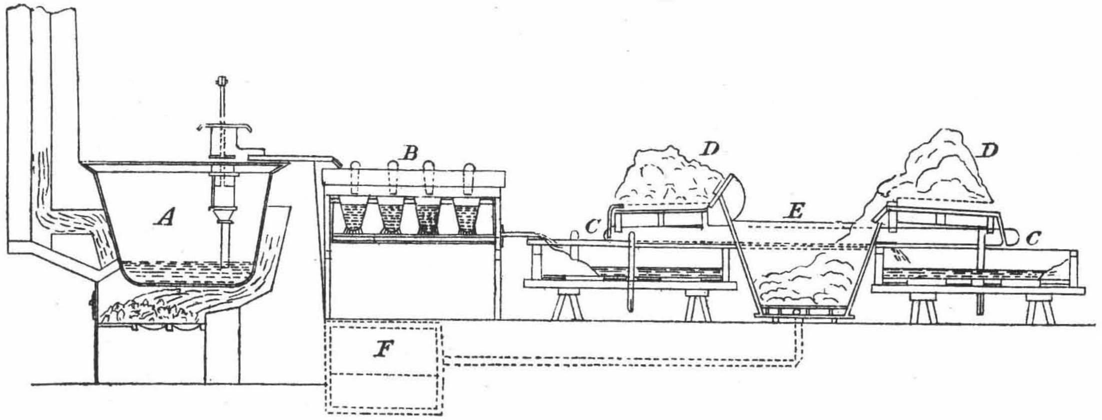

FIG. 5. Plant for Refining Saltpetre at Waltham Abbey.

The crude saltpetre thus obtained still contains several per cent. of sodium chloride and about a half per cent. of magnesium chloride. It is purified by dissolving in the washings of the purified salt, allowing it to crystallize, and washing with water, whereby the percentage of chloride is reduced to 0·05 per cent. or less, and the material is rendered practically free from all other impurities. Finally it is dried.

The sodium chloride formed in the conversion is washed on the filter with liquors containing gradually diminishing amounts of nitrate, until the solid contains only 0·8 per cent. or less. This "saltpetre salt" then contains about 98 per cent. of sodium chloride in the dry state. It is unsuitable for the manufacture of hydrochloric acid, because this would be contaminated with nitrous compounds, and moreover the plant would be strongly attacked. It is therefore either sold to farmers to put on the land or used in copper extraction or other metallurgical processes.

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[1] *Mem. of Dept. of Agriculture in India,* Chemical Series, vol. iii., No. 7, 1914.

"Artificial" or "conversion" saltpetre made as above, is usually supplied by the chemical works to the explosives factory in such a state of purity that no further purification is necessary. Natural saltpetre from India on the other hand always contains a considerable amount of impurity and requires to be refined before use.

Although the quantity of black powder made is still very considerable, it is not nearly so large as it was twenty years ago. The black powder factories now being worked were all in existence at that time, and they mostly have large saltpetre refineries attached, which more than suffice to refine all the material that they require. It has not been found worth while to reconstruct the refineries, as they are still capable of turning out saltpetre of good quality. It would nowadays be possible, however, to erect up-to-date plant that would save much space and some fuel and labour.

Refining Indian saltpetre.

At Waltham Abbey, as at some other English powder factories, Indian saltpetre is used exclusively. The total quantity imported into England every year is, however, only 10,000 tons and the total consumption for making powder, etc., several times that amount. The balance is made up with "conversion" saltpetre.

Waltham Abbey method.

The method of refining still followed at Waltham Abbey is as follows: The crude or "grough" saltpetre is dissolved up in a large iron copper, *A* (Fig. 5), which has a capacity of 500 gallons, and is fitted with a perforated false bottom which prevents the saltpetre adhering to the vessel. For each charge, about 25 cwts. of grough saltpetre are taken, and 5 cwts. of crystals recovered from liquors, and 5 cwts. of crystals left in the crystallizing cisterns. This is all dissolved in about 280 gallons of the washings of the purified saltpetre, which also contains a considerable amount of the salt. The fire is lit under the copper, and in about two hours the saltpetre is dissolved and the liquid boiling. Just before it boils a thick scum rises to the surface consisting mostly of impurities. This is skimmed off and the false bottom is removed, and cold water is added from time to time to induce fresh scum to form, if it will. The fire is then withdrawn and the liquid is allowed to settle for two hours. Then a hand pump is lowered into the copper and the liquid is pumped into filters *B*, where it passes through linen cloth. From here it runs to shallow copper crystallizing troughs *C*. As it cools down, the liquid is kept stirred by a workman in order to make the saltpetre separate in small crystals, and the saltpetre "flour" as it forms is drawn up on to an inclined draining platform *D*, and from there is passed to a washing vat *E*. After the temperature has fallen to about 32° C. (90° F.) the solution is no longer stirred and any crystals that form after that are treated as grough nitre.

Washing. The washing vat *E* is about 6 feet long, by 4 feet wide, by 3 feet 6 inches deep, and is fitted with a false bottom made of wood with small holes bored in it. Below the false bottom is a plug which can be removed to allow the washings to flow away. First the charge is washed with 70 gallons of water sprinkled over it by means of a rose, the plug being left out so that the washings can drain away to a liquor tank *F*. After draining half an hour the plug is inserted and the saltpetre covered with fresh water, which after standing half an hour is also allowed to drain into *F*. Finally the salt is washed by sprinkling with 100 gallons of water, the plug remaining out. The saltpetre is now allowed to drain all night and is then removed to the store-house where it is allowed to dry spontaneously. In about three days the moisture has fallen to 3 or 5 per cent.

Motherliquor. The mother liquors and other impure solutions are boiled down to about a quarter of their original volume. Any scum or deposit that forms during the boiling should be removed and water then be added. The solution is now filtered and allowed to crystallize. The crystals are treated as grough saltpetre and the mother liquor returned to the evaporating pots.

Continental methods. The methods of refining adopted in France, Germany and other countries are substantially the same as that at Waltham Abbey. A small proportion of size is, however, often added in the refining copper to assist the formation of scum.

Saltpetre from the atmosphere. Potassium nitrate could also be made from the calcium nitrate produced from atmospheric oxygen and nitrogen by processes such as that of Birkeland and Eyde as carried out at Notodden, but the calcium chloride obtained as a by-product would be of no value. Or the dilute nitric acid obtained in the Birkeland-Eyde process could be treated with limestone or chalk and potassium chloride :

$$CaCO_3 + 2KCl + 2HNO_3 = 2KNO_3 + CaCl_2 + CO_2 + H_2O.$$

In this case the carbonic acid could be collected and compressed into cylinders and sold. Up to the present, these methods do not appear to have been adopted, but nitric acid and ammonium nitrate are being made on a very large scale, especially in Germany.[1]

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[1] *See* chap. viii.

CHAPTER V

MANUFACTURE OF CHARCOAL AND SULPHUR

Charcoal : Wood used : Distillation : Composition : Brown charcoal : Sulphur : Sicilian sulphur : By-product sulphur : Louisiana sulphur : Refining sulphur : Properties : Functions of sulphur

Wood used. AT one time the charcoal for black powder was made almost exclusively from alder-wood, but later other soft woods were used, and straw charcoal was also introduced for the brown powders for heavy ordnance. Charcoal from soft woods is generally used, especially for the better qualities of gunpowder, because it is more easy to ignite. In England dog-wood is much used, especially for rapid burning powders of small grain ; for larger powders, alder and willow. In Germany alder and willow are the principal woods used ; in Austria, alder and hazel ; in Switzerland, hazel ; in France black alder is used for high class powders, for mining powders common white woods such as white alder, poplar, aspen, birch and hazel ; in Spain, the oleander, yew, willow, hemp stems, and vine ; in Italy, almost exclusively hemp stems.

Charcoal burnt in heaps or kilns has not been used very largely for gunpowder since even the very earliest days, for it was soon found that to produce good powder it was necessary to select the wood carefully and burn it very uniformly. It has therefore been heated in ovens or iron vessels, and the procedure of the present day does not differ materially from that of the fourteenth century.

The wood should be cut in the spring, as the sap in it at that time of the year contains much less inorganic matter, so that although the proportion of sap is larger, yet the percentage of ash in the wood is much smaller. Moreover, wood cut in the spring is much more easily freed from its bark, which also contains a large proportion of ash. The wood is kept at least eighteen months, and generally not less than three years, to allow the sap to dry out of it and other changes to take place. The practice varies considerably as regards protecting the wood from rain : at Dresden it is kept in sheds ; at Spandau in the open ; at Waltham Abbey also the wood is kept in the open, but the dog-wood is covered with thatch, whereas the alder and willow are not.

The wood is split if necessary into pieces about 1 inch thick, and these are placed in an iron cylinder about 2 feet in diameter and 3 feet 6 inches long. This cylinder is then raised by means of suitable tackle and placed in a furnace, which is heated as uniformly as possible. The higher the temperature and the longer the heating, the lower is the percentage of hydrogen and oxygen in the charcoal and the greater is its hardness and the difficulty with which it is ignited. At Waltham Abbey dog-wood for R.F.G. or M.G.^{1} powders is heated 4 hours, for R.F.G.^{2} 8 hours. Alder and willow for R.L.G.^{2} are heated 3½ hours, for R.L.G.^{4}, P., and Prism ^{1} Black 4 hours, for P ^{2} 6 hours.

Distillation.

When the temperature of the wood attains about 280° C. volatile products of decomposition of the wood come off plentifully. These could be condensed by means of a suitable condenser, and worked up into acetate of lime and wood spirit. The charcoal plant of a powder mill is, however, on such a small scale as compared with the factories in which charcoal is produced for metallurgical processes with recovery of the by-products, that it is not usually considered worth while to do this. The volatile products are therefore simply led into the furnace by which the wood is being heated, and so some expenditure of fuel is saved. To enable the gases and volatile products to escape the cylinder has some holes bored in it at one end, and the furnace is provided with a pipe to lead away these products. When the carbonization has proceeded far enough, the flame of the burning gas becomes blue. The furnace is then opened, the cylinder taken out by means of the tackle, and a fresh cylinder of wood put in before the furnace has had time to cool. The cylinder that has been taken out is placed inside a larger cylinder, which has a closely fitting lid, and is there allowed to cool. It is necessary that the cooling should take place out of contact of the air, as otherwise the charcoal will catch fire. Even when cold it at first absorbs large quantities of oxygen from the air, and in so doing may become sufficiently hot to catch fire. Therefore oxygen must only be allowed gradual access to the charcoal: it should not be ground until a week after it has been burnt. Before use it is carefully picked over by hand to remove any that has not been properly burnt, as also any foreign matters that have got into it. Charcoal intended for powders for ordnance should be jet black in colour; its fracture should show a clear velvet-like surface; it should be light and sonorous when dropped on a hard surface, and so soft that it will not scratch polished copper.[1] The yield of such charcoal is 28 to 30 per cent. of the dried wood. For small-arms a more slackly burnt charcoal can be used, and the yield may be as much as 40 per cent. Such charcoal has a reddish-brown colour, which is perceptible in the powder until it has been glazed with graphite.

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[1] *Treatise on Service Explosives,* 1907, p. 10.

Composition.

The composition of some typical charcoals is shown in the Table[1] below. Spanish hemp charcoal is usually burnt in pits holding ½ to 1 ton. When the carbonization has proceeded far enough the pit is covered with a woollen cloth on which earth is placed. This probably accounts for the high percentage of ash in the analysis below.

Description	Carbon	Hydrogen	Oxygen	Ash
From P. Powder, Waltham Abbey . .	85·26	2·98	10·16	1·60
From R.L.G., Waltham Abbey . .	80·32	3·08	14·75	1·85
From R.F.G., Waltham Abbey . .	75·72	3·70	18·84	1·74
From F.G., Waltham Abbey . . .	77·88	3·37	17·60	1·15
Curtis's and Harvey, Sporting . .	77·36	3·77	16·62	2·25
Curtis's and Harvey, Mining . . .	83·74	3·07	10·45	2·74
Spanish, Hemp Charcoal. . . .	76·29	3·31	14·87	5·53
German Sporting Powder. (B. & S.) .	68·8	3·7	27·5	Trace
Austrian Cannon Powder. (K.) . .	81·3	2·8	13·6	2·3
Austrian Small Arm Powder. (K.). .	82·6	2·9	12·5	2·0
Russian Powder. (F)	72·5	2·9	22·3	2·3

Brown charcoal.

The charcoal for brown or "cocoa" powder was made from rye-straw, which was only carbonized very slightly.[2] 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.

For cheap blasting powder and powder for scaring birds and supplying natives of Africa, etc., charcoal of an inferior quality can be used.

SULPHUR

Sicilian sulphur.

Sulphur occurs native in many volcanic districts, especially in Sicily, and until recent times practically the whole of the world's supply came from there. The sulphur in Sicily is mixed with limestone, the ores containing

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[1] Noble and Abel, *Trans. Roy. Soc.*, 1875 and 1879; Noble, *Artillery and Explosives,* 1906, pp. 127, 129. "B. & S." means analysis by Bunsen and Schischkoff, "K" by Karolyi, "F" by Fedeow.

[2] Guttmann, *Manufacture,* vol. i., p. 90; Cundill and Thompson, p. 21; *Treatise on Service Explosives,* 1900, p. 110.

usually from 20 to 40 per cent. of sulphur. Formerly the sulphur was recovered by the wasteful "calcaroni" process. The ore was piled in a large heap and covered over with moistened ash except for a small opening. Combustion was started with burning wood, but the combustion of part of the sulphur provided most of the necessary heat. The sulphur melted out and flowed down on to a prepared floor. Only about 60 per cent. of the sulphur in the ore was recovered by this process, and the large quantities of sulphur dioxide set free were very injurious to the surrounding vegetation. This method has been largely superseded by the introduction of recuperative furnaces invented by Gill and modified by Sanfillipo. About six large chambers are arranged in a series so that the hot gases from one can be made to heat the next. By this process the recovery is about 80 per cent. Attempts have been made to introduce more efficient methods whereby practically the whole of the sulphur could be recovered by melting it out with hot brine or steam, or distilling it with superheated steam. These methods have not attained any great success, however, the obstacles being the absence of any local supply of fuel and the backward state of the country. The sulphur is refined by distillation, the principal distilleries being situated in Marseilles. Some powder mills have small sulphur refineries of their own, as at Waltham Abbey for instance.

Two sorts of sulphur can be obtained by distillation: flowers and stick sulphur. The former consists of minute crystals, which have been deposited on the interior surface of a large chamber or "dome" into which the vapours have been passed. The flowers contain a small percentage of sulphuric acid formed by the action of the air on the sulphur, and consequently are not suitable for the manufacture of explosives. Stick sulphur, on the other hand, is very pure and only requires to be ground.

By-product sulphur.

For the manufacture of sulphuric acid elementary sulphur is but little used now, as it pays better to roast various ores in which it is combined with metals, such as copper pyrites and zinc-blende, but it does not pay to extract sulphur as such from these ores. A certain proportion of it comes on the market, however, as it is obtained as a by-product in the Leblanc soda process. The sulphuric acid used in that process is ultimately converted into calcium sulphide, CaS, and for many years this accumulated in great heaps, which were a public nuisance, no method being known by which it could be worked up except at a prohibitive cost. Eventually the Claus-Chance process was devised and perfected, which enabled this to be done. Kiln gas is passed over the "soda-waste," converting it into calcium carbonate and sulphuretted hydrogen: $CaS + H_2O + CO_2 = CaCO_3 + H_2S$. As the sulphuretted hydrogen is rather dilute and variable in concentration, the gas is led through a fresh quantity of the waste, by which it is absorbed, forming the bisulphide: $CaS + H_2S = CaH_2S_2$. When kiln gas is led through this the sulphuretted hydrogen is again given off, but is of double the previous concentration: $CaH_2S_2 + H_2O + CO_2 = CaCO_3 + 2H_2S$. This gas is collected in a gas-holder, and can be fed from there into the chambers where it is converted into sulphuric acid, or it can be mixed with gas from the pyrites burners, whereby sulphur is caused to deposit in accordance with the equation: $2H_2S + SO_2 = 2H_2O + 3S$. The sulphur thus obtained is of considerable purity.

Sulphur is also obtained in the purification of coal-gas from sulphuretted hydrogen and other sulphur compounds.

Louisiana sulphur.

Until recently the market was entirely controlled by an English association, the Anglo-Sicilian Sulphur Company, formed in 1895. Sulphur had been found in Louisiana in 1865, during some boring operations for petroleum, but it was situated underneath 500 feet of quicksand, and all attempts to work it commercially failed until the matter was taken up by Hermann Frasch in 1891, and even then years of work were required and a large amount of capital before success was achieved. The sulphur is mixed with a much smaller proportion of limestone than in Sicily, the ore containing about 70 per cent. sulphur. The method that has been adopted is to put down a pipe of 10 inches diameter until the sulphur deposit is reached, then the hole is continued with a 9-inch drill through the sulphur deposit, which is about 200 feet thick. A 6-inch pipe is passed to the bottom, and a 3-inch pipe through this, both being perforated near their lower ends. Superheated water is passed down the 6-inch pipe, but the sulphur passes up the 3-inch. At first it was raised by means of pumps, but now air is forced down: this mixes with the sulphur and reduces its density, and it is raised to the surface of the ground by the pressure of the water used for melting.

The "Union Sulphur Company" has been so successful that it has acquired the whole of the trade of the United States and also exports considerable quantities. The production amounts to several hundred thousand tons per annum.

The sulphur as it comes up from the well is said to have a purity of 99·93 to 99·98 per cent. It is simply run into great bins, which hold as much as 150,000 tons each. When it has cooled the sides of the bins are removed, the sulphur is broken up, and is then ready for shipment.

The Anglo-Sicilian Sulphur Company finding itself unable to contend with Frasch's Company finally retired from the business, but it had made enormous profits for many years. The Italian Government has formed a compulsory trust to control the marketing of Sicilian sulphur and ensure a living wage to the Sicilian workmen. This has proved very successful and the workers in Sicily are now better off than they have been for many years past.

On the occasion of the presentation to him of the Perkin Medal, Frasch gave a very interesting account of the various difficulties he had to contend with in working out his invention, and this is published in the *J. Soc. Chem. Ind.*, 1912, pp. 168–176.[1]

Refining sulphur. Sicilian sulphur requires to be refined before it can be used, and this is done by distilling it. The crude or "grough" sulphur is placed in an iron pot, which is heated from below by a furnace until the sulphur boils. The vapour passes over into a chamber where the sulphur is deposited on the walls in the form of small crystals, which constitute "flowers of sulphur." If the walls of the chamber are allowed to get hot enough to melt the crystals the sulphur runs down and is tapped off and cast into sticks or rolls. The still is often so arranged that the waste heat from the furnace melts another charge of crude sulphur ready to run into the still as soon as the first charge has been distilled off. The refinery at Waltham Abbey is provided not only with a large chamber or "dome" but also with a condenser leading to a receiver. Only the first portion of vapour is admitted to the dome, then the vapours are turned into the condenser. Flowers of sulphur are not fit for making explosives because they contain a small proportion of sulphuric and sulphurous acids. The flowers from the dome are therefore redistilled.

Properties. Roll sulphur consists of pale yellow brittle crystals belonging to the rhombic system, having a density of 2·07 at 0°. It melts at about 113°, to an amber-coloured liquid, but when the heating is continued above 120° it gradually becomes darker and more viscous. Between 160° and 220° it is so viscous that the vessel containing it can be inverted without losing any. If this viscous amorphous mass be cooled rapidly part of it retains the amorphous condition and is insoluble in carbon bisulphide, which dissolves ordinary rhombic sulphur with ease. Flowers of sulphur always contain a proportion of this insoluble modification. Sulphur boils at 444·5°.

Functions of sulphur. One reason why sulphur is added to black powder is that its temperature of ignition, 261° C, is low, and consequently it makes the powder burn more readily. But another reason is that under the influence of pressure, not only in the press but also in the incorporating mill, it flows and becomes colloidal, cementing together the particles of charcoal and the minute crystals of saltpetre. From the examination of microphotographs Cronquist[2] found that brown charcoal has a similar power of becoming colloidal under pressure. This is why brown gunpowders burn more slowly and regularly than black, and why the percentage of sulphur in them can be reduced or abolished altogether.[3]

Sulphur oxidizes slowly in the air, forming sulphur dioxide and a little sulphuric acid. If a chlorate be present chloric acid is liberated, and this accelerates the oxidation, and there is grave danger of spontaneous ignition occurring.

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[1] *See also S.S.* 1911, p. 236.

[2] *S.S.*, 1906, p. 53.

[3] *See* p. 86.

CHAPTER VI

MANUFACTURE OF GUNPOWDER

Advantages and disadvantages : Composition : Grinding the ingredients : Weighing and mixing : Incorporating or milling : Automatic drenchers : Removing the mill-cake : Breaking down : Pressing : Granulating or corning : Dusting and glazing : Stoving or drying : Finishing and blending : Cut powders : Moulded powders : Blasting powders : Sprengsalpeter : Cahuecit : Petroklastit : Bobbinite : Water-soluble powder : Products of explosion

THE invention of so many other blasting explosives and smokeless powders has greatly restricted the consumption of black powder, but it has been able to hold its own in certain fields in consequence of its advantages : its low price, the ease with which it can be ignited, its insensitiveness to shock, its stability at moderately high temperatures, its regular rate of burning, and the non-corrosive nature of the residue that it leaves in the gun. But against these must be placed its great disadvantages : its want of power and the great quantity of smoke that it evolves. For shot-guns its rate of explosion is suitable, only the recoil and smoke are disagreeable, but for rifles the rate of burning cannot be controlled sufficiently ; for driving the bullets out of shrapnel shell there is no better explosive, and it is still used for armour-piercing shell, because the high explosives used for other sorts of shell will not withstand the great shock of impact without exploding prematurely ; for filling the rings of time fuses for shell no satisfactory substitute has yet been found.

Composition. Guttmann, in his book on the *Manufacture of Explosives*, published in 1895, gave the following as the compositions of the principal powders made at that time :

	Saltpetre	Sulphur	Charcoal
(*a*) *Rifle Powders :*			
Austria-Hungary	75	10	15
Belgium	75·5	12	12·5
China	75	10	15
France[1]	75	10	15
Germany[2]	74	10	16

[1] *Vennin et Chesneau*, p. 322, give the proportions 75 : 12·5 : 12·5.

[2] The proportions afterwards used in Germany for rifle powder were 75 : 9 : 15.

	Saltpetre	Sulphur	Charcoal
(a) *Rifle Powders :*			
Great Britain	75	10	15
Holland	70	14	16
Italy	75	10	15
Persia	75	12·5	12·5
Portugal	75·7	10·7	13·6
Russia	75	10	15
Spain	75	12·5	12·5
Sweden	75	10	15
Switzerland	75	11	14
Turkey	75	10	15
United States	75	10	15
(b) *Cannon Powders :*			
Austria-Hungary	74	10	16
France [1]	75	10	15
Germany	74	10	16
Great Britain	75	10	15
Switzerland	75	10	15
(c) *Sporting Powders :*			
Austria-Hungary	76	9·4	14·6
France	78	10	12
Germany	78	10	12
Great Britain	75	10	15
Switzerland	78	9	13
(d) *Blasting Powders :*			
Austria-Hungary	60·2	18·4	21·4
France	72	13	15
Germany	70	14	16
Great Britain	75	10	15
Italy	70	18	12
Russia	66·6	16·7	16·7

Blasting powders, however, vary in composition far more than this Table indicates, powders with different rates of burning being used for rocks of different degrees of hardness. Thus the French Government factories make three sorts of mining powder :

	Saltpetre	Sulphur	Charcoal
Ordinary Powder	62	20	18
Slow Powder	40	30	30
Strong Powder	72	13	15

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[1] *Vennin et Chesneau*, p. 322, give the proportions 75 : 12·5 : 12·5.

The powders manufactured in Belgium have the following compositions :

	Saltpetre	Sulphur	Charcoal
Rifle Powder	75	12·5	12·5
Cannon Powder	75	12·5	12·5
Sporting Powder	78	10	12
Blasting Powder	75	12	13
Slow Powder or Pulvérin	70	13	14 & 3% wood meal
Slow Powder in cartridges	70	13	14 & 3% dextrine
Export Powder	68	18	22

In France "pulvérin" is also prepared for the manufacture of fireworks, etc. According to Chalon it has the composition 75 : 12·5 : 12·5,[1] but Vennin gives the proportions as 62 : 20 : 18.[2]

Grinding the ingredients.

Before they are mixed together the three ingredients are powdered. As they are not explosive when separate, they can be ground up in any suitable mill. In this respect, however, some reserve must be made as regards the sulphur : this has a great tendency to become electrified, and as it is also very inflammable an electric spark may easily set it alight or cause the explosion of a mixture of sulphur-dust and air. Rapid-moving machinery is therefore to be avoided ; the parts should all be made of metal and "earthed." According to Voigt the drum for pulverizing sulphur should not make more than ten revolutions per minute.[3] In some works the sulphur is mixed with a small proportion of the saltpetre before grinding to prevent this electrification, which has the further disadvantage that it causes the sulphur to cake together and so escape proper grinding. At Waltham Abbey the sulphur is ground under steel edge-runners similar to those used for incorporating the powder.

The charcoal is generally ground in a machine resembling a large coffee mill (*see* Fig. 6), but in some factories ball-mills are used, the charcoal being placed in a drum with bronze balls. The drum is then rotated until the constant falling of the balls on to the charcoal has reduced it to a sufficiently fine state of division.

Sulphur and saltpetre may also be ground in the Excelsior mill, but if the saltpetre is already in fine crystals it need not be ground, but only sifted. In France the saltpetre is mixed with 6 per cent. of charcoal and pulverized in an iron drum with bronze balls. The charcoal makes the saltpetre easier to grind, and this small proportion does not make it explosive. The remainder of the charcoal is mixed with the sulphur and pulverized in a similar drum. After grinding these binary mixtures are passed through a sieve with holes

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[1] *Explosifs Modèrnes*, pp. 228, 263.

[2] *Vennin et Chesneau*, p. 322.

[3] *Herstellung der Sprengstoffe*, vol. i., p. 52.

0·65 mm. in diameter to separate foreign matters. The two binary mixtures are then mixed together by hand before being milled.[1]

Weighing and mixing.

The three ingredients are carefully weighed out, preferably each in separate scales. An extra amount of saltpetre is often taken to allow for the moisture in it, the actual proportions weighed out being sometimes 76·0 : 15·0 : 10·0 instead of 75·0 : 15·0 : 10·0. But the charcoal usually contains quite as high a percentage of moisture as the saltpetre. It is not surprising therefore that analysis sometimes shows a percentage of charcoal which is below the theoretical.

The ingredients are then given a preliminary mixing. In Germany this is done in a rotating drum with lignum vitæ balls. The drum is made of wood and may be lined with leather; iron must be avoided in its construction; the axle must be covered with leather. At Waltham Abbey the mixing is done in a cylindrical drum of copper or gun-metal about 18 inches long and 2 feet 9 inches in diameter. Through the centre passes an axle carrying eight rows of fork-shaped arms, called "flyers." The drum rotates in one direction making 40 revolutions per minute. The axle moves in the other direction and makes 120 revolutions per minute. The ingredients are mixed for five minutes, and then sifted through a fine-mesh sieve of copper or brass wire. The sifting at this stage is very important, as any hard particles left in the charge are likely to cause an explosion in the incorporating mill. For this reason the sifting is sometimes done by hand. The "green" charge is now placed in a waterproof bag ready to be taken to the incorporating mill.

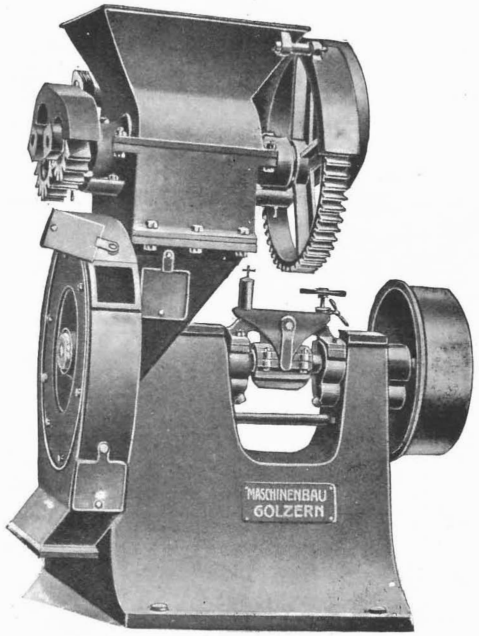

Fig. 6. Excelsior Mill, made by Maschinenbau A.-G. Golzern-Grimma.

Incorporating or milling.

On the Continent stamp-mills are still used to a small extent for incorporating as well as mixing gunpowder. In Germany the stamp-heads may be made of copper, zinc, bronze or other suitable alloy.[2] The charge is placed in a spherical hole in a block of wood, with a piece of specially hard wood inserted at the bottom for the stamp to play upon. The stamping is carried on for about fourteen hours. If continued longer the density of the powder diminishes and the ballistics deteriorate.

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[1] *Vennin et Chesneau*, p. 327.

[2] *Unfallverhüttungsvorschriften*, 1912, p. 182.

In France the use of stamp-mills was definitely abandoned in 1884,[1] the powders there being incorporated in drums and mills. The cheaper sorts of powder are incorporated entirely in drums containing wooden balls. In England also stamp-mills are not used, and the standard method is to grind the ingredients together in incorporating mills. Formerly these consisted of two heavy stone edge-runners working on a stone bedplate. Now it is more usual to have iron runners working on an iron bed-plate; of course iron must not work upon stone, or vice versa, on account of the danger of generating sparks. In the most usual type of mill the runners are 6 or 7 feet in diameter and about 15 inches wide, and weigh about 4 tons each. They rotate on the opposite ends of a horizontal shaft, which is carried by a cross-head, which again is attached to a vertical shaft making about eight revolutions a minute. Usually the two edge-runners are mounted at different distances from the central shaft so that one works the outer part of the charge and the other the inner, but their paths overlap. There are two ploughs of wood covered with leather, which are fixed to the shaft and travel round with it. These continually push the charge away from the centre and the curb respectively, and bring it under the edge-runners again. The mills make $7\frac{1}{2}$ or 8 revolutions per minute.

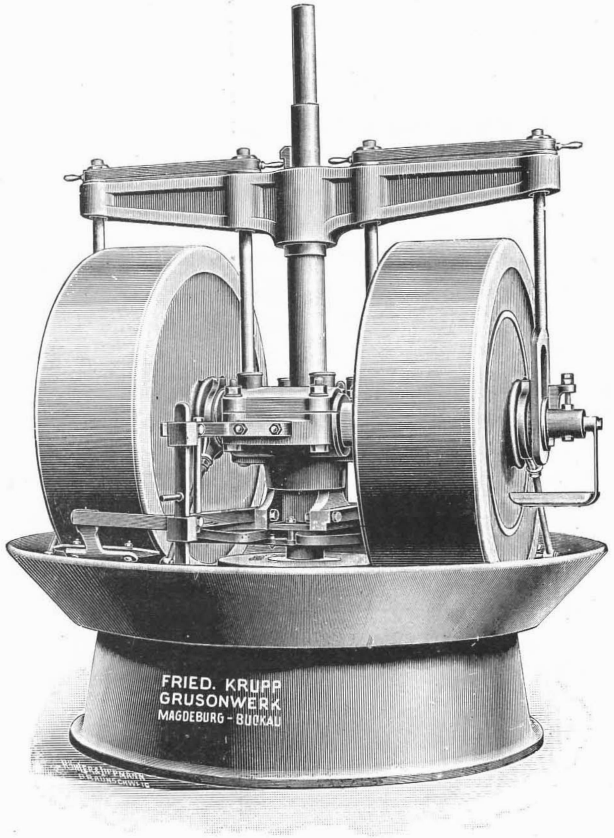

Fig. 7. Gruson Gunpowder Mill.

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[1] *P. et S.*, vol. iii., p. 18.

In the Gruson mill (Fig. 7) the iron runners do not rest on the bed but are suspended a short distance above it, so that there is no danger of a very thin layer of powder being subjected to great friction. The bearings are so suspended that either runner can travel upwards independently of the other when an extra thick portion of charge comes underneath it. The runners weigh about 5½ tons each and rotate equidistantly round the main vertical shaft. The ploughs are made of phosphor bronze, and each runner is also provided with a scraper to prevent the charge being thrown off the bed. The drive is by means of a large bevelled gear-wheel, which may be arranged either above the machine or below it.

In Germany iron runners are not allowed to work on an iron bed-plate unless they are suspended, as in the Gruson mill. If they actually rest upon the bed, it must be made of wood fastened down with brass screws.

By the action of the runners the ingredients are crushed and ground together very intimately without subjecting the mixture to any violent action.

Usually about six incorporating mills are arranged in a row and driven from a common shaft actuated by a single water-wheel or steam-engine. Each mill is separated from the next by a strong masonry wall. Explosions in these mills are fairly frequent in spite of every precaution, but as a rule no very serious damage is done. In 1907 there were nine such explosions in England, but only one man was injured; in 1908 there were five explosions and two men were injured; in 1909 there were seven explosions and no men were injured. The reason why there are so few men killed or injured in these accidents is that as a rule there is no one in the mill house: after the charge has been started the man in attendance goes on to see to other mills and only comes occasionally to see that all is right and to add a little water to the charge if necessary. In France there is less than one explosion in 100,000 milling operations.[1] In Germany no workman is allowed to remain in the building whilst the mill is working at full speed.

A charge consists of 60 to 80 lbs., the time of incorporation varies with the description of the powder: the longer the incorporation the faster the powder burns. Cannon powders were usually milled for three or four hours, rifle powders eight, and sporting powders as much or more. The charge when placed in the mill contains 2 to 3 per cent. of moisture. It must be kept moist the whole time; for this purpose the mill-man adds water from time to time, preferably condensed water from a steam trap. Formerly urine was frequently used instead of water. For fine grain powders 1½ to 3 per cent. of moisture should be present in the finished mill-cake, for larger sizes 3 to 6 per cent.

In France mills similar to the Gruson mill are used, but the charge is only 20 kg. or 25 kg. in the case of mining powder. It contains 8 per cent. moisture when introduced and 2 to 4 per cent. at the finish. The mill makes 10 revo-

[1] *Vennin et Chesneau*, p. 333.

lutions per minute and requires 7 horse-power. The following table gives the times of milling and the densities of the mill cake:

	Time	Density
Military rifle powder, F_3	2 –2½ hours	1·740
Sporting powder, ordinary	1½ ,,	1·725
,, ,, strong	5 ,,	1·80
Dust reworked	½	—
Mining powder	½	1·57

The density is of importance because in France the powder is not pressed. The density can be increased by milling slowly, half a turn per minute, with the outer plough removed.[1]

In Germany the charge is generally about 75 kg. and the mill makes about nine revolutions per minute. The time of milling is:

Military powder	2½–3 hours.
Sporting ,,	4 ,,
Mining ,,	1½ ,,

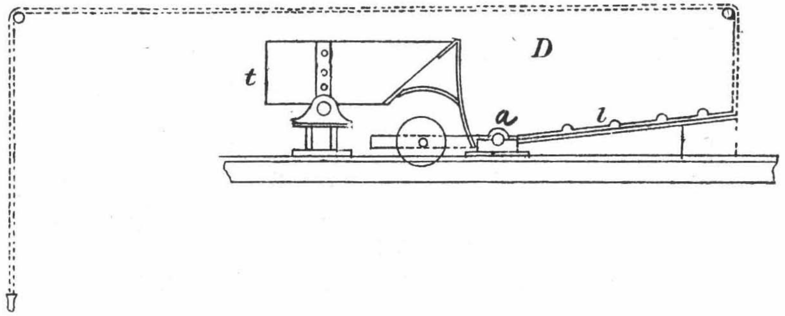

FIG. 8. Drenching Arrangements for Powder Mills.

Before the charge is removed the mill is run slowly for a time to increase the density, but the powder undergoes a pressing operation also, except in the case of mining powder.[2]

Automatic drenchers.

In order to prevent the explosion in one mill being communicated to the other mills of the group, each one is provided in England with an automatic drenching arrangement (*see* Fig. 8). This consists of a lifting board *l*, provided with a counterpoise weight. There is also a tank *t* full of water, supported on a hinge and a leg at one end that rests on a projection from the lifting board. When the latter is lifted, the leg is released, the tank tips forward and the water is poured over the charge in the mill. The axle *a* is common

[1] *Vennin et Chesneau*, p. 332.

[2] Voigt, *Herstellung der Sprengstoffe*, i., pp. 55, 56.

to all the lifting boards of the group of mills, so that if there be an explosion in one of the mills the corresponding board *l* is raised, taking with it all the other similar boards, and all the charges in the group are drenched and so rendered unexplosive. In order to make the mechanism sufficiently sensitive it is important that the boards be not too small and not too near the roof, and that they be directly over the mills. The second point is essential, because if the board be very near the roof a reflected wave of pressure reaches it almost immediately after the direct wave and before the mechanism has had time to act.

Removing the mill-cake.

The mill-cake often becomes caked on to the bed very firmly. Many accidents having been caused by removing this with metal tools, H.M. Inspector of Explosives issued a letter on December 27, 1883, proposing the adoption of the following special rule in all black powder works:

"Whenever it may become necessary in mills or other buildings to remove any powder incrustations (whether from the machinery or elsewhere), which cannot be easily brushed off, such removal is to be effected without the use of any metal tool whatever; the hard powder is to be removed by means of water, supplemented, if need be, when the whole incrustation has been thoroughly saturated, by a suitable wooden instrument gently applied."

Breaking down.

The mill-cake is next reduced to a rough powder by hand or by passing it through gun-metal rolls in a machine somewhat resembling the granulating machine, but simpler.

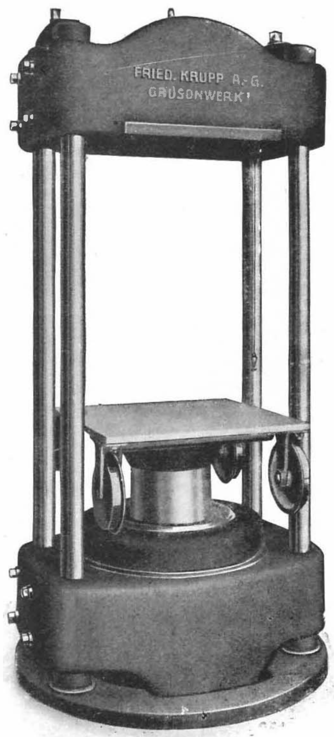

FIG. 9. Gunpowder Press.

Pressing.

Then the mixture is subjected to high pressure in a press. This converts it into a hard mass, the constituents of which have no tendency to separate again from one another, and also increases the density of the powder. Formerly the powder was compressed in a very strong box, but this is no longer done on account of the dangerous friction against the sides.

For moulded powders and blasting cartridges special presses are used, which will be described later. Granulated powders are pressed in presses of the type shown in Fig. 9. The mill-cake is built up on a small trolley: first a plate of copper, bronze, or ebonite is put down, and a temporary frame put round it, then a layer of the mill-cake about $\frac{3}{4}$-inch thick is carefully spread, then another plate and another layer of powder, until about 10 cwt. of mill-cake has been built up with about twenty plates. The temporary frames are then removed and the trolley is wheeled on to the press, and the pressure is gradually applied. The amount of compression required varies with the amount of moisture in the mill-cake and the density to be attained in the finished powder. For a mixture containing about 3 per cent. of moisture it is necessary to apply a pressure of about 400 lb. per square inch of plate surface for $1\frac{1}{2}$ to 2 hours to obtain a density of 1·7. The amount of compression is measured by the motion of the press rather than by the hydraulic pressure; this motion may be 24 or 30 inches according to the dimensions of the press, etc. The pressure is usually released and reapplied several times to obtain a satisfactory result.

Ebonite plates are sometimes preferred to metal because they keep their shape better and yet give sufficiently to transmit the pressure evenly. If the cake be very dry the ebonite may become electrified, however, and so produce very dangerous sparks. In Germany the use of ebonite plates is forbidden, and cloths are laid between the plates and the powder. The four columns of the press should be made of mild steel with an ample margin of strength even if the whole pressure is borne by only two of them. They may with advantage be covered with leather. It was recommended by H.M. Inspector of Explosives[1] that the press should not be worked directly off the hydraulic pump, but from an accumulator, and that the drive of the pump should not be positive, but by friction.

The explosion of a press-house is more destructive than that of any other building in a black powder works, as might be expected, considering that there is about half a ton of powder in one mass strongly compressed. The house should therefore be specially well isolated from other buildings by mounds, etc. In some works the workmen are not allowed to be in the press-house whilst the pressure is on the powder; the pressure can be applied and controlled from another compartment, where there is also an indicator showing the position of the platform of the press.

When sufficiently pressed the pressure is released and the trolley is wheeled away, and the press-cake removed from it by hand or with wooden tools. The outer portion of each slab is rejected as it is not sufficiently compressed; it is added to a later pressing.

Blasting powder is sometimes compressed between rollers.

Granulating or corning.

The broken-up press-cake is put in barrels and taken to the granulating or corning house. Here there is a machine having three or four pairs of gun-metal rolls, through which the press-cake is passed, and a number of automatic sieves, which sort out the grains of the required size (*see* Fig. 10). The top pair of rollers usually has pyramidal teeth: from this the material passes

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[1] Special Report No. 138.

over a sieve to the next pair of rolls, which has smaller teeth. The lowest pair are plain. The pieces that are not fine enough are passed through the machine again; the dust and fine powder are milled for a short time and pressed again. The bearings of one or both rolls of each pair are provided with springs or weights to keep them in position, and are not rigidly fixed. Consequently if an extra hard piece of cake passes through the rolls it is not subjected to great violence: the rolls give way and the piece passes through.

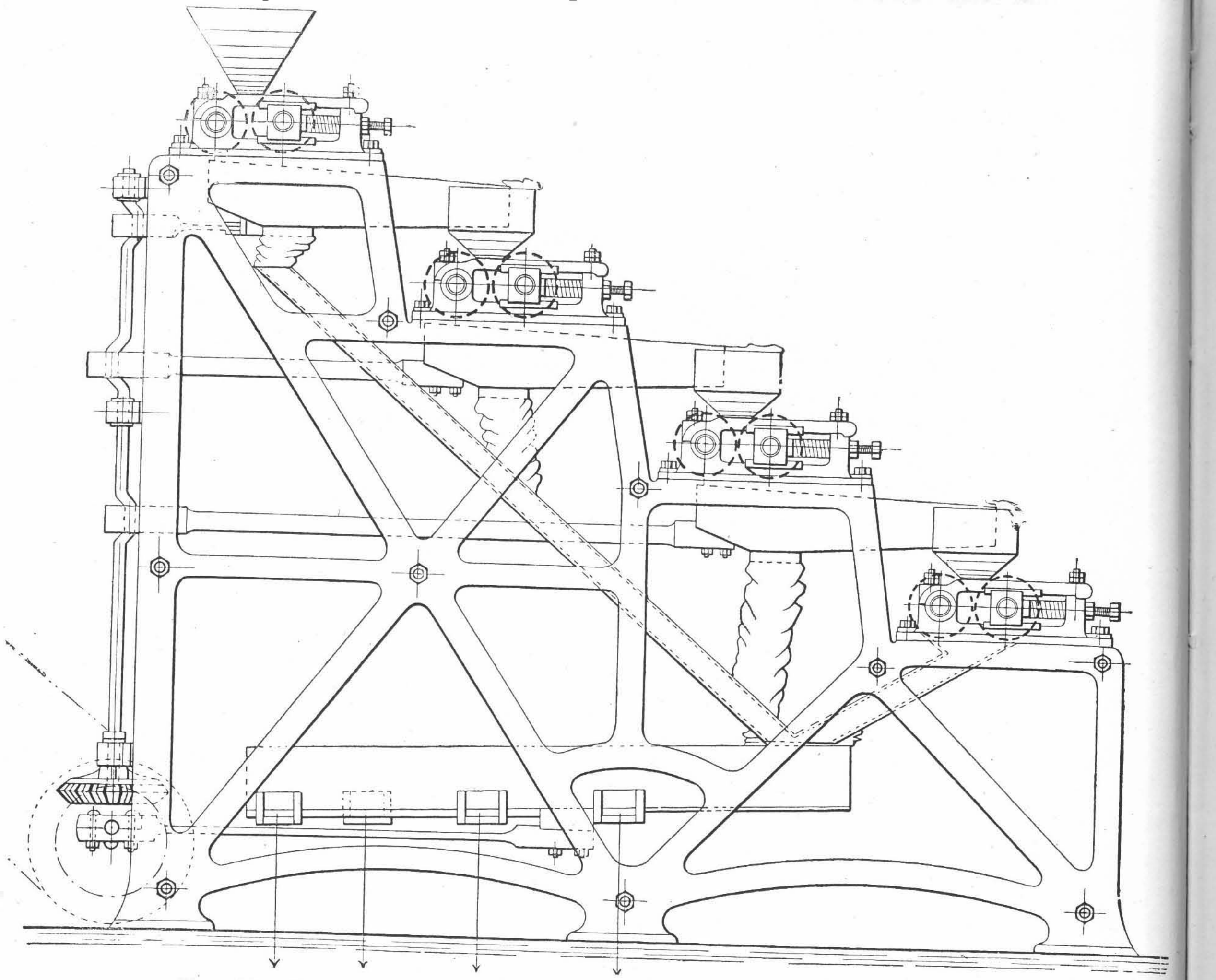

Fig. 10. Corning Machine, made by Maschinenbau A.-G. Golzern-Grimma

This type of granulating machine was invented in 1819 by Colonel Congreve, and is the one in most general use. Various other types have been tried, but none produces such a good angular grain.

In France the granulation is carried out in a horizontal drum covered with metal sheet perforated with fine holes of a size suited to the sort of powder that is to be produced. The broken down mill-cake is placed inside this drum together with pieces of hard wood, which are caused by longitudinal strips to fall continually on to the powder and break it up. A charge of 20 kg. is granulated in ten to twelve minutes and yields 35 to 55 per cent. of grains of the size required.

Dusting. Powders made with dog-wood charcoal produce a lot of dust in the corning process, and it is best to remove this by passing the powder through a dusting reel. This is simply a cylindrical reel set at an angle of about 4° to the horizontal and covered with fine woven wire of copper or brass. It is open at both ends and rotates on its axis, making about forty revolutions per minute. The powder is simply passed through this and caught again in a barrel.

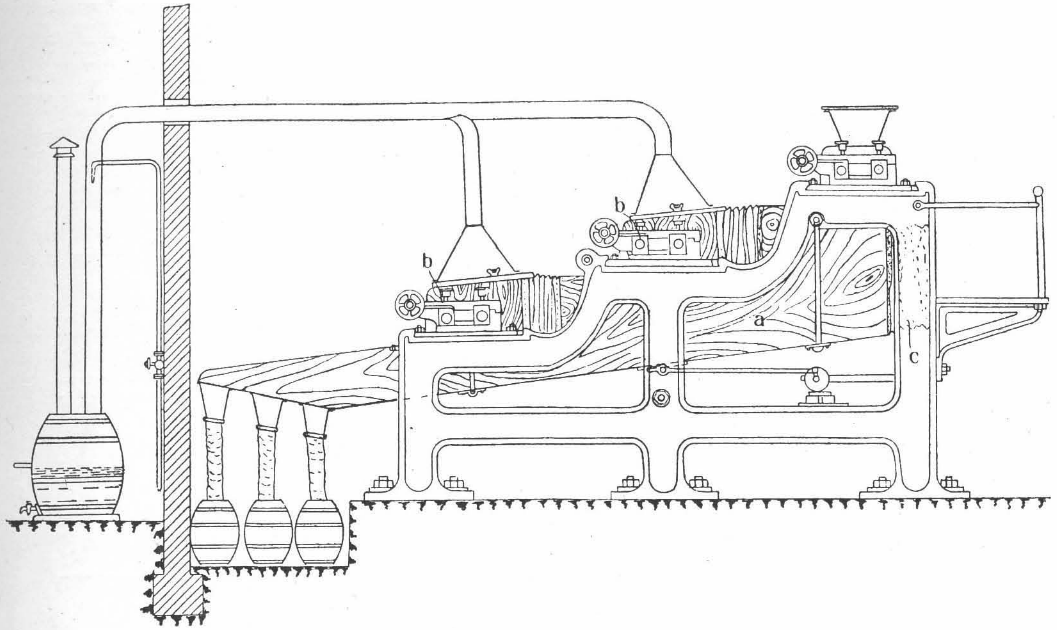

Fig. 11. Corning Machine with Dust-Remover

Glazing. The glazing operation is carried out in wooden drums, which rotate on their axis about thirty times a minute. Cannon powders receive an addition of a small proportion of graphite and are glazed for two or three hours. Rifle and sporting powders, and others that are required to burn quickly, do not receive any graphite, but are glazed longer.

Stoving or drying. The stove may be heated either by forcing hot air through it, or by arranging hot water or steam pipes in it. The easiest and most economical method is to dry with steam, and as black powder is not very sensitive and is not liable to spontaneous decomposition, this method is usually adopted. Only low-pressure steam should be used, the exhaust being open to the air. The powder is placed on canvas trays supported on wooden racks. Inlets and outlets are provided for the air, and the temperature is kept at about 40° C. (104° F.). The time required to dry the powder varies from one to four hours according to the size of grain.

Finishing and blending.

To remove the last traces of dust and give the powder a good "colour" it is now treated for some two hours in the finishing reel, which is covered with fine canvas, and finally thoroughly blended into large uniform batches. The last operation is performed at Waltham Abbey by pouring it into a hopper, which is provided with four delivery shutes, so that the contents of the hopper are divided into four equal portions. By repeating this operation in a systematic manner the desired object is attained very effectually.

Cut powders.

Powder for cannon of large size, 6 to 12-inch bore, was made by cutting the press-cake into cubes. The slabs from the press were passed under a roll armed with longitudinal knives, whereby the cake was cut into strips, and these were then passed under another similar roll in the other direction and so cut into cubes. The glazing, stoving, finishing and blending were very much as for granulated powder, except that the stoving had to be continued for about thirty hours at 55° C. These cut powders are but little made now, as they have been displaced by smokeless powders.

The following Table gives particulars of some of the powders made formerly at Waltham Abbey[1]:

Powder	Size of grains Meshes to 1 in.	Charcoal		Incorporation		Glazing hrs.	Stoving		Finished Powder	
		Wood	Time of burning	Time hrs.	Moisture in mill-cake		hrs.	Temp.	Density	Moisture
RFG	12–20	Dogwood	4 hrs.	4	1·5%	5½	1	100° F.	1·58–1·62	0·9–1·2
RFG[2]	12–20	,,	8 ,,	8	2·5	10	2	100	1·72–1·75	0·9–1·2
RLG[2]	3–6	Alder and willow	3½ ,,	3	2·5	1½	2	110	1·65	1·0–1·3
RLG[4]	2–3	,,	4 ,,	3	4	3	6	115	1·65	1·0–1·3
P	⅝" cubes	,,	4 ,,	3	4–5	4	36	130	1·75	1·0–1·3

Moulded powders.

Moulded powders also are but little used for the same reason, but much the same process is used for making moulded cartridges of mining powder, and also

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[1] *Treatise on Service Explosives*, 1907 ed., pp. 123, 124.

pellets for time and percussion fuses and for other ammunition. The general form of all these articles is practically the same: a hexagonal or round cylinder with a central perforation. The powder is pressed, granulated, dusted and blended as already described, and then taken to the moulding house. Two different types of press are used, worked by hydraulic and mechanical pressure

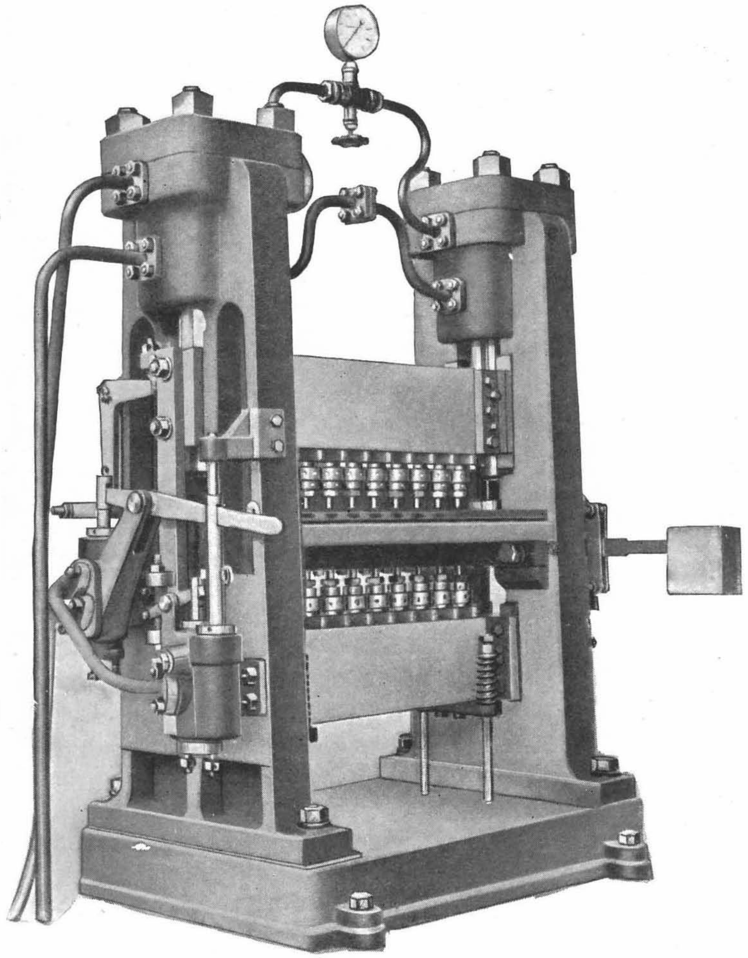

FIG. 12. Hydraulic Automatic Press for Moulded Powders and Blasting Cartridges (Maschinenbau A.-G. Golzern-Grimma)

respectively (*see* Figs. 12 and 13). The general principle is, however, the same. The granular powder is put into a hopper and flows from there into a measure that automatically measures off the right quantity, which then passes into the die. The dies are arranged in rows in a plate, so that whilst one row is being filled another is being pressed. The pressure is usually applied simultaneously from above and below by two different plungers. The central hole is formed by means of a pin which passes through the lower plunger and into the other. Hydraulic presses are safer, but mechanical ones more rapid in their action. For blasting cartridges, machines are also made with rotating tables containing a number of dies: each operation, filling, pressing, removing, is performed at a different position of the table. Such machines are quite automatic and require very little attention.

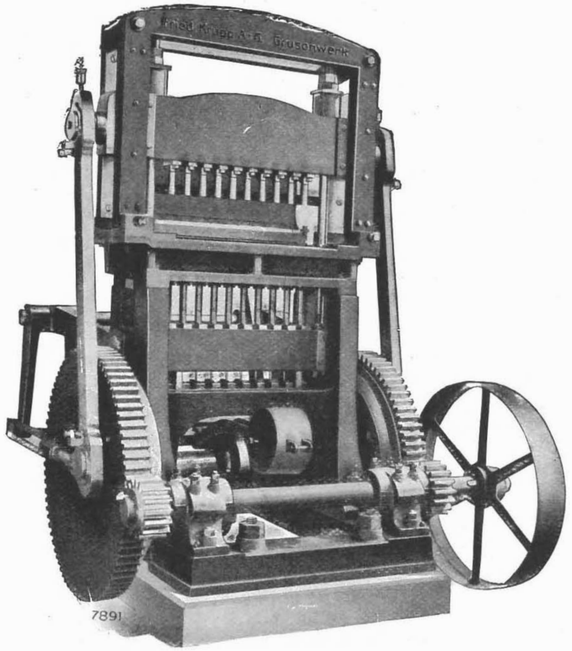

FIG. 13. Mechanical Press for Moulded Powders, etc. (F. Krupp A.-G. Grusonwerk)

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 following Table gives the details of the English and German powders:

Country	Name	Charcoal	Proportions	Density
England .	Prism[1] Black . .	Black .	75 : 10 : 15	1·76
,, .	Prism[1] Brown . .	Brown .	79 : 3 : 18	1·80
,, .	E.X.E. . . .	,, .	77·4 : 5 : 17·6	1·80
,, .	S.B.C. . . .	,, .	79 : 3 : 18	1·85
Germany .	P.P.C./68 . . .	Black .	74 : 10 : 16	1·66
,, .	P.P.C./75 . . .	,, .	74 : 10 : 16	1·76
,, .	P.P.C./82 . . .	Brown .	78 : 3 : 19	1·86
,, .	P.P.C./85 . . .	,, .	80 : 0 : 20	1·88
France [1] .	P.B.. . . .	,, .	78 : 3 : 19	1·85–1·87

[1] *Vennin et Chesneau*, pp. 322, 341.

P.P.C./68 has seven holes, all the others, English and German, only one. The value of the brown straw charcoal is that under the high pressures 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.[1]

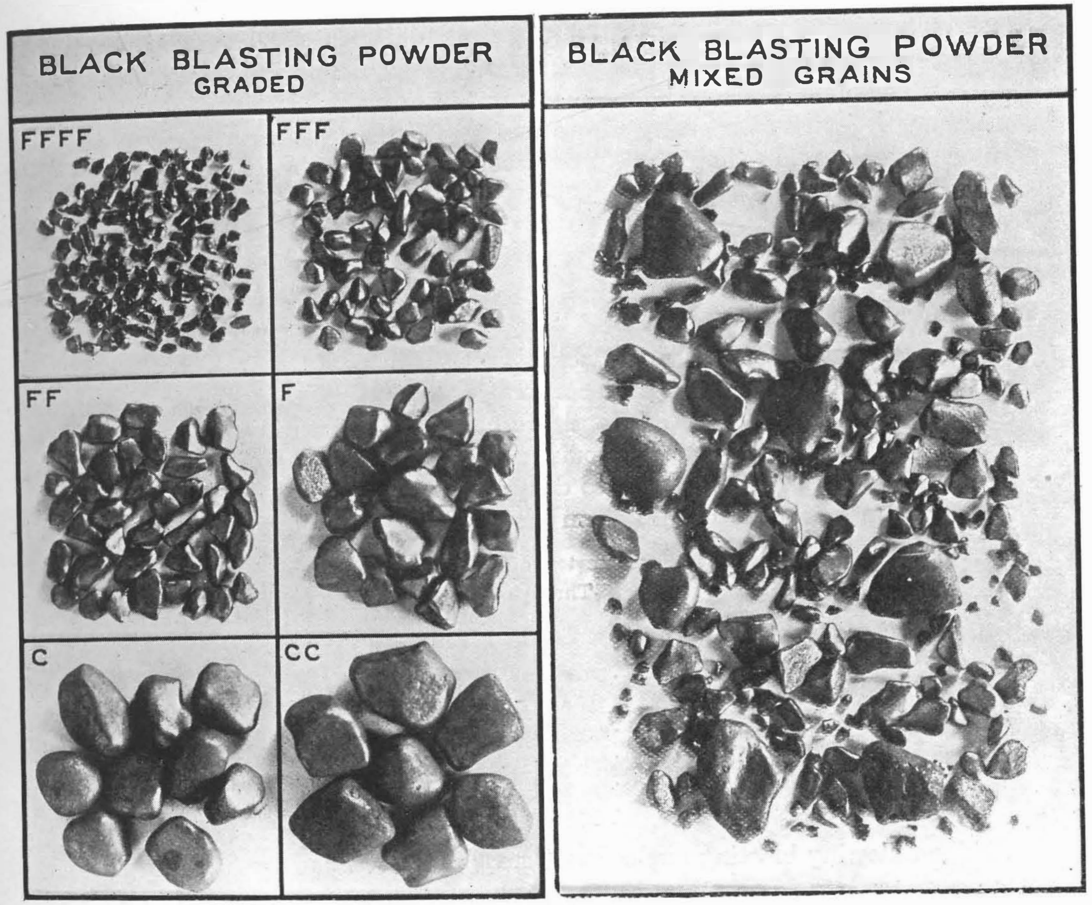

FIG. 14. American Black Blasting Powders (Munroe and Hall)

Blasting powders. The usual composition of black blasting powders has already been stated (p. 74). The violence of the powder can be varied by altering the composition, the density or the size of the grains: the powder is made slower by diminishing the percentage of saltpetre, compressing to a higher density or making

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[1] *See* Cronquist, *S.S.*, 1906, p. 53.

the grains coarser. In France the mining powders manufactured in the State mills are incorporated, not under edge-runners, but in copper drums with wooden and bronze balls, and the granulation is also effected in drums with the aid of a spray of water.

In America enormous quantities of blasting powder are used containing sodium nitrate (Chili saltpetre) instead of the potassium salt. This burns more slowly than ordinary gunpowder, but is more powerful, as it evolves a greater volume of gas and more heat, but its principal advantage is its low price, for it is used for many purposes where in Europe hand labour would be employed. According to Chalon,[1] the composition varies between the following limits :

		Mean
Sodium nitrate	67·3 to 77·1	74
Sulphur	9·4 to 14·3	10
Charcoal	22·9 to 8·6	16

The usual proportions in this " black blasting powder " are given by Munroe and Hall[2] as 73 : 11 : 16. The incorporation is not so thorough as in the case of ordinary black powder, and the charcoal is generally obtained from coarser-grained woods. As the sodium nitrate is hygroscopic, care must be taken not to expose the powder to damp air more than can be helped. The following are the sizes of American black blasting powders :

	Diameters of round holes in screens in $\frac{1}{64}$th inches	
	Through which grains pass	On which grains collect
CCC	40	32
CC	36	24
C	27	18
F	20	12
FF	14	7
FFF	9	3
FFFF	5	2

The following blasting explosives resembling black powder in composition are made in Germany, and are allowed to be sent as goods in unlimited quantities, as they are considered safer to handle than ordinary black powder :

Sprengsalpeter

" Sprengsalpeter " (blasting saltpetre) is a mixture of sodium nitrate, sulphur and brown coal in about the proportions 75 : 10 : 15, and is very largely used in the Stassfurt salt-mines where the soft and brittle nature of the salts, such as carnallite, require an explosive that is milder than ordinary blasting powder. It possesses the advantage of being cheap and not giving rise to poisonous fumes. Harder materials, such as sylvinite and rock-salt, are blasted with a combined charge of nitro-glycerine explosive and Sprengsalpeter.

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[1] *Les Explosifs Modernes*, 3rd ed., 1911, p. 264.

[2] *Primer on Explosives for Coal Miners*, Washington, 1911, p. 16.

Cahuecit.

" Cahuecit " was invented by R. Cahuc some forty years ago and was manufactured at one time at Dartford under the name of Safety Blasting Powder or Carboazotine. It had the composition :

Saltpetre	64
Sulphur	12
Lampblack	7
Bark or wood-pulp	17

to which was added 1 to 5 per cent. of sulphate of iron. After mixing the ingredients roughly in a drum they were introduced together with a considerable bulk of water into a steam-jacketed pan where the mixture was heated with constant stirring until almost dry. The mixture was imperfect in consequence of the tendency of the soluble salts to crystallize out.[1] It is still manufactured in Germany, and has been found good for blasting basalt.[2] The official German definition is : a compressed mixture of not more than 70 per cent. saltpetre, 8 per cent. lampblack, about 12 per cent. flowers of sulphur, at least 10 per cent. cellulose, and a small quantity of iron sulphate.

Petroklastit.

" Petroklastit " (Haloklastit) has approximately the following composition :

Sodium nitrate	69
Potassium nitrate	5
Sulphur	10
Coal tar pitch	15
Potassium bichromate	1

Its strength and sensitiveness as compared with black blasting powder[3] are :

	Trauzl test	Falling weight
Petroklastit	157	100
Black powder	108	65

Its official definition is : a compressed mixture of sodium nitrate, sulphur, coal-tar pitch, saltpetre, and not more than 1 per cent. potassium bichromate, also with an addition of not more than 10 per cent. charcoal. It has been used in stone quarries and potash mines.

Bobbinite.

In English coal-mines the most largely used explosive has been Bobbinite, which is a black powder mixture with an addition of the sulphates of copper and ammonium, or of starch and paraffin-wax. It is the only explosive of this class that was able to pass the Woolwich test for " Permitted Explosives " ; it does not pass the Continental and Rotherham tests. In 1906 a

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[1] Guttmann, *Manufacture*, vol. i., p. 273 ; *see also* Cundill and Thomson, p. 142.

[2] *S.S.*, 1908, p. 97.

[3] Zschokke, pp. 42, 57.

Departmental Committee was held at the Home Office to inquire whether this explosive should be removed from the list. This has not been done, but by the Explosives in Coal-Mines Order of September 1, 1913, its use has been restricted to mines that are not gassy or dusty. In these its use is permitted for a period of five years from January 1, 1914. The following is its composition according to the official definitions, and an analysis made by Hall and Howell:[1]

	Official definitions		Hall and Howell
	First	Second	
Nitrate of potassium	62·0–65·0	63·0–66·0	65·31
Charcoal	17·0–19·5	18·5–20·5	19·52
Sulphur	1·5–2·5	1·5– 2·5	2·63
Sulphate of ammonium Sulphate of copper	13·0–17·0	—	—
Rice or Maize starch	—	7·0– 9·0	8·73
Paraffin wax	—	2·5 –3·5	3·35
Moisture	0·0– 2·5	0·0– 3·0	0·46

In 1914 more than a million lbs. of Bobbinite were used in British mines and quarries.

Water-soluble powder.

Raschig proposes to make a cheap blasting powder consisting of 65 per cent. sodium nitrate and 35 per cent. sodium cresol-sulphonate. These are dissolved in water and the solution is evaporated very rapidly on a rotating drum heated by high-pressure steam. It is claimed that the expensive and dangerous operation of incorporation is thus done away with. It is necessary to select a combustible constituent like the cresol-sulphonate, that has a high solubility of the same order as the nitrate, otherwise there would be a tendency for the substances to separate during the evaporation. Safety explosives containing ammonium nitrate instead of the sodium salt have been registered under the name of "Raschit."[2]

Products of explosion.

The products formed on the explosion of gunpowder were investigated by Bunsen and Schischkoff,[3] Linck,[4] and Karolyi,[5] but the most complete series of experiments was carried out by Noble and Abel.[6] Debus[7] showed

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[1] U.S. Bureau of Mines, *Bull.* 15, 1912, p. 179.
[2] *See Ang.* 1912, p. 1194; Ger. Pat. App. R. 54,360 of 3/2/12; *S.S.*, 1912, p. 292.
[3] *Pogg. Annalen*, 102, 1857, p. 321. [4] *Annalen der Chemie*, 109, 1858, p. 53.
[5] *Pogg. Annalen*, April, 1863; *Phil. Mag.*, Ser. 4, No. 26, 1863, p. 266.
[6] *Phil. Trans.*, 1875, 49.
[7] *Proc. Roy. Soc.*, 30, 1880, p. 198; *Phil. Trans.*, 1882, p. 523.

that they had made an error in giving potassium hyposulphite as a primary product of the explosion. Noble and Abel[1] accordingly corrected their results. The mean percentages from R.L.G. Powder were:

Gases	42·98%
Solids	55·91
Water	1·11

Gases, per cent. by volume		Solids, per cent. by weight	
Carbon dioxide	49·3	Potassium carbonate	61·0
Carbon monoxide	12·5	Potassium sulphate	15·1
Hydrogen	2·2	Potassium sulphide	14·5
Methane	0·4	Potassium sulphocyanide	0·2
Nitrogen	32·9	Potassium nitrate	0·3
Sulphuretted Hydrogen	2·6	Sulphur	8·7

From 1 g. of the powder 271·3 c.c. of gas were produced, measured at 760 mm. and 0° C., and the quantity of heat liberated was 700·7 calories.

The products obtained from mining powder have been given by J. Harger,[2] and the analysis of the gases from American blasting powder has been published by C. M. Young.[3] Hall and Howell[4] have investigated the products from Bobbinite.

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[1] *Phil. Trans.*, 1880, p. 203.
[2] *J. Soc. Chem. Ind.*, 1912, p. 415.
[3] *Bull. Am. Min. Eng.*, 1910, pp. 637–662; *Ang.*, 1911, p. 1886.
[4] *U.S. Bureau of Mines Bull.* 15, 1912, p. 179.