**Nitro-Explosives: A Practical Treatise eBook**

**Nitro-Explosives: A Practical Treatise**

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**NITRO-EXPLOSIVES.**

**CHAPTER I.**

*INTRODUCTORY.*

The Nitro-Explosives—­Substances that have been Nitrated—­The Danger Area—­ Systems of Professors Lodge, Zenger, and Melsens for the Protection of Buildings from Lightning, &c.

The manufacture of the various nitro-explosives has made great advances during late years, and the various forms of nitro-compounds are gradually replacing the older forms of explosives, both for blasting purposes and also for propulsive agents, under the form of smokeless powders.  The nitro-explosives belong to the so-called High Explosives, and may be defined as any chemical compound possessed of explosive properties, or capable of combining with metals to form an explosive compound, which is produced by the chemical action of nitric acid, either alone or mixed with sulphuric acid, upon any carbonaceous substance, whether such compound is mechanically mixed with other substances or not.[A]

[Footnote A:  Definition given in Order of Council, No. 1, Explosives Act, 1875.]

The number of compounds and mixtures included under this definition is very large, and they are of very different chemical composition.  Among the substances that have been nitrated are:—­Cellulose, under various forms, *e.g*., cotton, lignin, &c.; glycerine, benzene, starch, jute, sugar, phenol, wood, straw, and even such substances as treacle and horse-dung.  Some of these are not made upon the large scale, others are but little used.  Those of most importance are nitro-glycerine and nitro-cellulose.  The former enters into the composition of all dynamites, and several smokeless powders; and the second includes gun-cotton, collodion-cotton, nitrated wood, and the majority of the smokeless powders, which consist generally of nitro-cotton, nitro-lignin, nitro-jute, &c. &c., together with metallic nitrates, or nitro-glycerine.

The nitro-explosives consist generally of some organic substance in which the *no*\_{2} group, known as nitryl, has been substituted in place of hydrogen.

Thus in glycerine,

|*Oh*
C\_{3}H\_{5}|*Oh*,
|*Oh*

which is a tri-hydric alcohol, and which occurs very widely distributed as the alcoholic or basic constituent of fats, the hydrogen atoms are replaced by the *no*\_{2} group, to form the highly explosive compound, nitro-glycerine.  If one atom only is thus displaced, the mono-nitrate is formed thus,

|*Ono*\_{2}
C\_{3}H\_{5}|*Oh*;
|*Oh*

and if the three atoms are displaced, C\_{3}H\_{5}(*Ono*\_{2})\_{3}, or the tri-nitrate, is formed, which is commercial nitro-glycerine.

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Another class, the nitro-celluloses, are formed from cellulose, C\_{6}H\_{10}O\_{5}, which forms the groundwork of all vegetable tissues.  Cellulose has some of the properties of the alcohols, and forms ethereal salts when treated with nitric and sulphuric acids.  The hexa-nitrate, or gun-cotton, has the formula, C\_{12}H\_{14}O\_{4}(*Ono*\_{2})\_{6}; and collodion-cotton, pyroxylin, &c., form the lower nitrates, *i.e*., the tetra- and penta-nitrates.  These last are soluble in various solvents, such as ether-alcohol and nitro-glycerine, in which the hexa-nitrate is insoluble.  They all dissolve, however, in acetone and acetic ether.

The solution of the soluble varieties in ether-alcohol is known as collodion, which finds many applications in the arts.  The hydrocarbon benzene, C\_{6}H\_{6}, prepared from the light oil obtained from coal-tar, when nitrated forms nitro-benzenes, such as mono-nitro-benzene, C\_{6}H\_{5}*no*\_{2}, and di-nitro-benzene, C\_{6}H\_{4}(*no*\_{2})\_{2}, in which one and two atoms are replaced by the *no*\_{2} group.  The latter of these compounds is used as an explosive, and enters into the composition of such well-known explosives as roburite, &c.  The presence of nitro groups in a substance increases the difficulty of further nitration, and in any case not more than three nitro groups can be introduced into an aromatic compound, or the phenols.  All aromatic compounds with the general formula, C\_{6}H\_{4}X\_{2}, give, however, three series.  They are called ortho, meta, or para compounds, depending upon the position of *no*\_{2} groups introduced.

Certain regularities have been observed in the formation of nitro-compounds.  If, for example, a substance contains alkyl or hydroxyl groups, large quantities of the para compound are obtained, and very little of the ortho.  The substitution takes place, however, almost entirely in the meta position, if a nitro, carboxyl, or aldehyde group be present.  Ordinary phenol, C\_{6}H\_{5}.*Oh*, gives para- and ortho-nitro-phenol; toluene gives para- and ortho-nitro-toluene; but nitro-benzene forms meta-di-nitro-benzene and benzoic acid, meta-nitro-benzoic acid.[A]

[Footnote A:  “Organic Chemistry,” Prof.  Hjelt.  Translated by J.B.  Tingle, Ph.D.]

If the graphic formula of benzene be represented thus (No. 1), then the positions 1 and 2 represent the ortho, 1 and 3 the meta, and 1 and 4 the para compounds.  When the body phenol, C\_{6}H\_{5}.*Oh*, is nitrated, a compound is formed known as tri-nitro-phenol, or picric acid, C\_{6}H\_{2}(*no*\_{2})\_{3}*Oh*, which is used very extensively as an explosive, both as picric acid and in the form of picrates.  Another nitro body that is used as an explosive is nitro-naphthalene, C\_{10}H\_{6}(*no*\_{2})\_{2}, in roburite, securite, and other explosives of this class.  The hexa-nitro-mannite, C\_{6}H\_{8}(*Ono*\_{2})\_{6}, is formed

[Illustration:  No. 1]

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[Illustration:  *Meta*-DINITRO-*benzene* No.2]

by treating a substance known as mannite, C\_{6}H\_{8}(*Oh*)\_{6}, an alcohol formed by the lactic acid fermentation of sugar and closely related to the sugars, with nitric and sulphuric acids.  It is a solid substance, and very explosive; it contains 18.58 per cent. of nitrogen.

Nitro-starch has also been used for the manufacture of an explosive.  Muhlhauer has described (*Ding.  Poly.  Jour.*, 73, 137-143) three nitric ethers of starch, the tetra-nitro-starch, C\_{12}H\_{16}O\_{6}(ONO\_{2})\_{4}, the penta- and hexa-nitro-starch.  They are formed by acting upon potato starch dried at 100 deg.  C. with a mixture of nitric and sulphuric acids at a temperature of 20 deg. to 25 deg.  C. Rice starch has also been used in its production.  Muhlhauer proposes to use this body as a smokeless powder, and to nitrate it with the spent mixed acids from the manufacture of nitro-glycerine.  This substance contains from 10.96 to 11.09 per cent. of nitrogen.  It is a white substance, very stable and soluble even in cold nitro-glycerine.

The explosive bodies formed by the nitration of jute have been studied by Messrs Cross and Bevan. and also by Muehlhaeuer.  The former chemists give jute the formula C\_{12}H\_{18}O\_{9}, and believe that its conversion into a nitro-compound takes place according to the equation—­

C\_{12}H\_{18}O\_{9} + 3HNO\_{3} = 3H\_{2}O + C\_{12}H\_{15}O\_(6}(NO\_{3})\_{3}.

This is equivalent to a gain in weight of 44 per cent. for the tri-nitrate, and 58 per cent. for the tetra-nitrate.  The formation of the tetra-nitrate appears to be the limit of nitration of jute fibre.  Messrs Cross and Bevan say, “In other words, if we represent the ligno-cellulose molecule by a C\_{12} formula, it will contain four hydroxyl (OH) groups, or two less than cellulose similarly represented.”  It contains 11.5 per cent. of nitrogen.  The jute nitrates resemble those of cellulose, and are in all essential points nitrates of ligno-cellulose.

Nitro-jute is used in the composition of the well-known Cooppal Smokeless Powders.  Cross and Bevan are of opinion that there is no very obvious advantage in the use of lignified textile fibres as raw materials for explosive nitrates, seeing that a number of raw materials containing cellulose (chiefly as cotton) can be obtained at from L10 to L25 a ton, and yield also 150 to 170 per cent. of explosive material when nitrated (whereas jute only gives 154.4 per cent.), and are in many ways superior to the products obtained from jute.  Nitro-lignin, or nitrated wood, is, however, largely used in the composition of a good many of the smokeless powders, such as Schultze’s, the Smokeless Powder Co.’s products, and others.

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*The Danger Area.*—­That portion of the works that is devoted to the actual manufacture or mixing of explosive material is generally designated by the term “danger area,” and the buildings erected upon it are spoken of as “danger buildings.”  The best material of which to construct these buildings is of wood, as in the event of an explosion they will offer less resistance, and will cause much less danger than brick or stone buildings.  When an explosion of nitro-glycerine or dynamite occurs in one of these buildings, the sides are generally blown out, and the roof is raised some considerable height, and finally descends upon the blown-out sides.  If, on the other hand, the same explosion had occurred in a strong brick or stone building, the walls of which would offer a much larger resistance, large pieces of brickwork would probably have been thrown for a considerable distance, and have caused serious damage to surrounding buildings.

It is also a very good plan to surround all danger buildings with mounds of sand or earth, which should be covered with turf, and of such a height as to be above the roof of the buildings that they are intended to protect (see frontispiece).[A] These mounds are of great value in confining the force of the explosion, and the sides of the buildings being thrown against them are prevented from travelling any distance.  In gunpowder works it is not unusual to surround the danger buildings with trees or dense underwood instead of mounds.  This would be of no use in checking the force of explosion of the high explosives, but has been found a very useful precaution in the case of gunpowder.

[Footnote A:  At the Baelen Factory, Belgium, the danger buildings are erected on a novel plan.  They are circular in ground plan and lighted entirely from the roof by means of a patent glass having wire-netting in it, and which it is claimed will not let a splinter fall, even if badly cracked.  The mounds are then erected right up against the walls of the building, exceeding them in height by several metres.  For this method of construction it is claimed that the force exerted by an explosion will expand itself in a vertical direction ("Report on Visits to Certain Explosive Factories,” H.M.  Inspectors, 1905).]

In Great Britain it is necessary that all danger buildings should be a specified distance apart; a license also must be obtained.  The application for a license must give a plan (drawn to scale) of the proposed factory or magazine, and the site, its boundaries, and surroundings, and distance the building will be from any other buildings or works, &c., also the character, and construction of all the mounds, and nature of the processes to be carried on in the factory or building.[A]

[Footnote A:  Explosives Act, 38 Vict. ch. 17.]

[Illustration:  FIG. 1.—­SECTION OF NITRO-GLYCERINE CONDUIT. *a*, lid; *b*, lead lining; *c*, cinders.]

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The selection of a site for the danger area requires some attention.  The purpose for which it is required, that is, the kind of explosive that it is intended to manufacture, must be taken into consideration.  A perfectly level piece of ground might probably be quite suitable for the purpose of erecting a factory for the manufacture of gun-cotton or gunpowder, and such materials, but would be more or less unsuitable for the manufacture of nitro-glycerine, where a number of buildings are required to be upon different levels, in order to allow of the flow of the liquid nitro-glycerine from one building to another through a system of conduits.  These conduits (Fig. 1), which are generally made of wood and lined with lead, the space between the woodwork and the lead lining, which is generally some 4 or 5 inches, being filled with cinders, connect the various buildings, and should slope gently from one to the other.  It is also desirable that, as far as possible, they should be protected by earth-work banks, in the same way as the danger buildings themselves.  They should also be provided with covers, which should be whitewashed in hot weather.

A great deal of attention should be given to these conduits, and they should be very frequently inspected.  Whenever it is found that a portion of the lead lining requires repairing, before cutting away the lead it should be very carefully washed, for several feet on either side of the portion that it is intended to remove, with a solution of caustic soda or potash dissolved in methylated spirit and water, and afterwards with water alone.  This decomposes the nitro-glycerine forming glycerine and potassium nitrate.  It will be found that the mixed acids attack the lead rather quickly, forming sulphate and nitrate of lead, but chiefly the former.  It is on this account that it has been proposed to use pipes made of guttapercha, but the great drawback to their use is that in the case of anything occurring inside the pipes, such as the freezing of the nitro-glycerine in winter, it is more difficult to find it out, and the condition of the inside cannot be seen, whereas in the case of wooden conduits it is an easy matter to lift the lids along the whole length of the conduit.

The buildings which require to be connected by conduits are of course those concerned with the manufacture of nitro-glycerine.  These buildings are—­(1) The nitrating house; (2) the separating house; (3) the filter house; (4) the secondary separator; (5) the deposit of washings; (6) the settling or precipitation house; and each of these buildings must be on a level lower than the preceding one, in order that the nitro-glycerine or acids may flow easily from one building to the next.  These buildings are, as far as possible, best placed together, and away from the other danger buildings, such as the cartridge huts and dynamite mixing houses, but this is not essential.

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All danger buildings should be protected by a lightning conductor, or covered with barbed wire, as suggested by Professor Sir Oliver J. Lodge, F.R.S., Professors Zenger, of Prague, and Melsens, of Brussels, and everything possible should be done to keep them as cool as possible in the summer.  With this object they should be made double, and the intervening space filled with cinders.  The roof also should be kept whitewashed, and the windows painted over thinly with white paint.  A thermometer should be suspended in every house.  It is very essential that the floors of all these buildings should be washed every day before the work-people leave.  In case any nitro-glycerine is spilt upon the floors, after sponging it up as far as possible, the floor should be washed with an alcoholic solution of soda or potash to decompose the nitro-glycerine, which it does according to the equation[A]—­

C\_{3}H\_{5}(NO\_{3})\_{3} + 3KOH = C\_{3}H\_{8}O\_{3} + 3KNO\_{3}.

[Footnote A:  See also Berthelot, *Comptes Rendus*, 1900, 131[12], 519- 521.]

Every one employed in the buildings should wear list or sewn leather shoes, which of course must be worn in the buildings only.  The various houses should be connected by paths laid with cinders, or boarded with planks, and any loose sand about the site of the works should be covered over with turf or cinders, to prevent its blowing about and getting into the buildings.  It is also of importance that stand pipes should be placed about the works with a good pressure of water, the necessary hose being kept in certain known places where they can be at once got at in the case of fire, such as the danger area laboratory, the foreman’s office, &c.  It is also desirable that the above precautions against fire should be tested once a week.  With regard to the heating of the various buildings in the winter, steam pipes only should be used, and should be brought from a boiler-house outside the danger area, and should be covered with kieselguhr or fossil meal and tarred canvas.  These pipes may be supported upon poles.  A stove of some kind should be placed in the corner of each building, but it must be entirely covered in with woodwork, and as small a length of steam pipes should be within the building as possible.

In the case of a factory where nitro-glycerine and dynamite are manufactured, it is necessary that the work-people should wear different clothes upon the danger area than usual, as they are apt to become impregnated with nitro-glycerine, and thus not very desirable or safe to wear outside the works.  It is also necessary that these clothes should not contain any pockets, as this lessens the chance of matches or steel implements being taken upon the danger area.  Changing houses, one for the men, and another for the girls, should also be provided.  The tools used upon the danger area should, whenever the building is in use, or contains explosives, be made of phosphor bronze or brass, and brass nails or wooden pegs should be used in the construction of all the buildings.

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[Illustration:  FIG. 2.—­MELSENS SYSTEM OF LIGHTNING CONDUCTORS.]

*Lightning Conductors.*—­The Explosive Substances Act, 38 Vict. ch. 17, clause 10, says, “Every factory magazine and expense magazine in a factory, and every danger building in a magazine, shall have attached thereto a sufficient lightning conductor, unless by reason of the construction by excavation or the position of such magazine or building, or otherwise, the Secretary of State considers a conductor unnecessary, and every danger building in a factory shall, if so required by the Secretary of State, have attached thereto a sufficient lightning conductor.”

The exact form of lightning conductor most suitable for explosive works and buildings has not yet been definitely settled.  Lightning-rod engineers favour what is known as the Melsens system, due to Professor Melsens, of Brussels, and Professor Zenger, of Prague, but first suggested by the late Professor Clerk-Maxwell.  In a paper read before the British Association, Clerk-Maxwell proposed to protect powder-magazines from the effects of lightning by completely surrounding or encasing them with sheet metal, or a cage of metallic conductors.  There were, however, several objections to his system as he left it.

Professor Melsens[A] has, while using the idea, made several important alterations.  He has multiplied the terminals, the conductors, and the earth-connections.  His terminals are very numerous, and assume the form of an aigrette or brush with five or seven points, the central point being a little higher than the rest, which form with it an angle of 45 deg..  He employs for the most part galvanised-iron wire.  He places all metallic bodies, if they are of any considerable size, in communication with the conducting system in such a manner as to form closed metallic circuits.  His system is illustrated in Fig. 2, taken from *Arms and Explosives*.

[Footnote A:  Belgian Academy of Science.]

This system is a near approximation to J.C.  Maxwell’s cage.  The system was really designed for the protection of powder-magazines or store buildings placed in very exposed situations.  Zenger’s system is identical with that of Melsens, and has been extensively tried by the Austrian military authorities, and Colonel Hess has reported upon the absolute safety of the system.

[Illustration:  Fig. 3.—­FRENCH SYSTEM OF LIGHTNING CONDUCTORS.]

The French system of protecting powder-magazines is shown in Fig. 3, where there are no brush terminals or aigrettes.  The French military authorities also protect magazines by erecting two or more lightning-rods on poles of sufficient height placed close to, but not touching, the walls of the magazine.  These conductors are joined below the foundations and earthed as usual.

In the instructions issued by the Government, it is stated that the lightning-rods placed upon powder-mills should be of such a height, and so situated, that no danger is incurred in igniting the powder-dust in the air by the lightning discharge at the pointed rod.  In such a case a fork or aigrette of five or more points should invariably be used in place of a single point.

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[Illustration:  FIG. 4\_a\_.—­GOVERNMENT SYSTEM OF LIGHTNING CONDUCTORS FOR LARGE BUILDINGS.]

[Illustration:  FIG. 4\_b\_.—­GOVERNMENT SYSTEM OF LIGHTNING CONDUCTORS FOR SMALL BUILDINGS.]

In Fig. 4 (*a* and *b*) is shown the Government method for protecting buildings in which explosives are made or stored.  Multiple points or aigrettes would be better.  Lord Kelvin and Professor Melsens favour points, and it is generally admitted that lightning does not strike buildings at a single point, but rather in a sheet; hence, in such cases, or in the event of the globular form being assumed by the lightning, the aigrette will constitute a much more effective protection than a single point.  As to the spacing of conductors, they may, even on the most important buildings, be spaced at intervals of 50 feet.  There will then be no point on the building more than 25 feet from the conductor.  This “25-feet rule” can be adhered to with advantage in all overground buildings for explosives.

Underground magazines should, whenever possible, also be protected, because, although less exposed than overground buildings, they frequently contain explosives packed in metal cases, and hence would present a line of smaller electrical resistance than the surrounding earth would offer to the lightning.  The conductor should be arranged on the same system as for overground buildings, but be applied to the surface of the ground over the magazines.

In all situations where several conductors are joined in one system, the vertical conductors should be connected both at the top and near the ground line.  The angles and the prominent portions of a building being the most liable to be struck, the conductors should be carried over and along these projections, and therefore along the ridges of the roof.  The conductors should be connected to any outside metal on the roofs and walls, and specially to the foot of rain-water pipes.

All the lightning conductors should be periodically tested, to see that they are in working condition, at least every three months, according to Mr Richard Anderson.  The object of the test is to determine the resistance of the earth-connection, and to localise any defective joints or parts in the conductors.  The best system of testing the conductors is to balance the resistance of each of the earths against the remainder of the system, from which the state of the earths may be inferred with sufficient accuracy for all practical purposes.

Captain Bucknill, R.E., has designed an instrument to test resistance which is based on the Post Office pattern resistance coil, and is capable of testing to approximate accuracy up to 200 ohms, and to measure roughly up to 2,000 ohms.  Mr R. Anderson’s apparatus is also very handy, consisting of a case containing three Leclanche cells, and a galvanometer with a “tangent” scale and certain standard resistances.  Some useful articles on the protection of buildings from lightning will be found in *Arms and Explosives*, July, August, and September 1892, and by Mr Anderson, Brit.  Assoc., 1878-80.

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*Nitro-Glycerine.*—­One of the most powerful of modern explosive agents is nitro-glycerine.  It is the explosive contained in dynamite, and forms the greater part of the various forms of blasting gelatines, such as gelatine dynamite and gelignite, both of which substances consist of a mixture of gun-cotton dissolved in nitro-glycerine, with the addition of varying proportions of wood-pulp and saltpetre, the latter substances acting as absorbing materials for the viscid gelatine.  Nitro-glycerine is also largely used in the manufacture of smokeless powders, such as cordite, ballistite, and several others.

Nitro-glycerol, or glycerol tri-nitrate, was discovered by Sobrero in the year 1847.  In a letter written to M. Pelouse, he says, “when glycerol is poured into a mixture of sulphuric acid of a specific gravity of 1.84, and of nitric acid of a gravity of 1.5, which has been cooled by a freezing mixture, that an oily liquid is formed.”  This liquid is nitro-glycerol, or nitro-glycerine, which for some years found no important use in the arts, until the year 1863, when Alfred Nobel first started a factory in Stockholm for its manufacture upon a large scale; but on account of some serious accidents taking place, its use did not become general.

It was not until Nobel conceived the idea (in 1866) of absorbing the liquid in some absorbent earth, and thus forming the material that is now known as dynamite, that the use of nitro-glycerine as an explosive became general.

Among those who improved the manufacture of nitro-glycerine was Mowbray, who, by using pure glycerine and nitric acid free from nitrous acid, made very great advances in the manufacture.  Mowbray was probably the first to use compressed air for the purpose of keeping the liquids well agitated during the process of nitration, which he conducted in earthenware pots, each containing a charge of 17 lbs. of the mixed acids and 2 lbs. of glycerol.

A few years later (1872), MM.  Boutnny and Faucher, of Vonges,[A] proposed to prepare nitro-glycerine by mixing the sulphuric acid with the glycerine, thus forming a sulpho-glyceric acid, which was afterwards mixed with a mixture of nitric and sulphuric acids.  They claimed for this method of procedure that the final temperature is much lower.  The two mixtures are mixed in the proportions—­Glycerine, 100; nitric acid, 280; and sulphuric acid, 600.  They state that the rise of temperature upon mixing is limited from 10 deg. to 15 deg.  C.; but this method requires a period of twenty-four hours to complete the nitration, which, considering the danger of keeping the nitro-glycerine in contact with the mixed acids for so long, probably more than compensates for the somewhat doubtful advantage of being able to perform the nitration at such a low temperature.  The Boutnny process was in operation for some time at Pembrey Burrows in Wales, but after a serious explosion the process was abandoned.

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[Footnote A:  *Comptes Rendus*, 75; and Desortiaux, “Traite sur la Poudre,” 684-686.]

Nitro-glycerine is now generally made by adding the glycerine to a mixture of sulphuric and nitric acids.  The sulphuric acid, however, takes no part in the reaction, but is absolutely necessary to combine with the water that is formed by the decomposition, and thus to keep up the strength of the nitric acid, otherwise lower nitrates of glycerine would be formed that are soluble in water, and which would be lost in the subsequent process of washing to which the nitro-compound is subjected, in order to remove the excess of acids, the retention of which in the nitro-glycerol is very dangerous.  Nitro-glycerol, which was formerly considered to be a nitro-substitution compound of glycerol, was thought to be formed thus—­

C\_{3}H\_{8}O\_{3} + 3HNO\_{3} = C{3}H\_{5}(NO\_{2})\_{3}O\_{3} + 3H\_{2}O;

but more recent researches rather point to its being regarded as a nitric ether of glycerol, or glycerine, and to its being formed thus—­

C\_{3}H\_{8}O\_{3} + 3 HNO\_{3} = C{3}H\_{5}(NO\_{3})\_{3} + 3H\_{2}O.
             92 227

|OH
The formula of glycerine is C\_{3}H\_{8}O\_{8}, or C\_{3}H\_{5}|OH
|OH

|ONO\_{2}
and that of the mono-nitrate of glycerine, C\_{3}H\_{5}|OH
|OH

|ONO\_{2}
and of the tri-nitrate or (nitro-glycerine), C\_{3}H\_{5}|ONO\_{2}
|ONO\_{2}

that is, the three hydrogens of the semi-molecules of hydroxyl in the glycerine have been replaced by the NO\_{2} group.

In the manufacture upon the large scale, a mixture of three parts by weight of nitric acid and five parts of sulphuric acid are used.  From the above equation it will be seen that every 1 lb. of glycerol should give 2.47 lbs. of nitro-glycerol ((227+1)/92 = 2.47), but in practice the yield is only about 2 lbs. to 2.22, the loss being accounted for by the unavoidable formation of some of the lower nitrate, which dissolves in water, and is thus washed away, and partly perhaps to the presence of a little water (or other non-nitrable matter) in the glycerine, but chiefly to the former, which is due to the acids having become too weak.

**CHAPTER II.**

*MANUFACTURE OF NITRO-GLYCERINE.*

Properties of Nitro-Glycerine—­Manufacture of Nitro-Glycerine—­Nitration—­ The Nathan Nitrator—­Separation—­Filtering and Washing—­The Waste Acids—­ Treatment of the Waste Acid from the Manufacture of Nitro-Glycerine and Gun-Cotton.

*Properties of Nitro-Glycerine.*—­Nitro-glycerol is a heavy oily liquid of specific gravity 1.6 at 15 deg.  C., and when quite pure is colourless.  The commercial product is a pale straw yellow, but varies much according to the purity of the materials used in its manufacture.  It is insoluble in water, crystallises at 10.5 deg.  C., but different commercial samples behave very differently in this respect, and

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minute impurities prevent or delay crystallisation.  Solid nitro-glycerol[A] melts at about 12 deg.  C., but requires to be exposed to this temperature for some time before melting.  The specific gravity of the solid form is 1.735 at +10 deg.  C.; it contracts one-twelfth of its volume in solidifying.  Beckerheim[B] gives the specific heat as 0.4248 between the temperatures of 9.5 deg. and 9.8 deg.  C., and L. de Bruyn gives the boiling point as above 200 deg..

[Footnote A:  Di-nitro-mono chlorhydrin, when added to nitro-glycerine up to 20 per cent., is said to prevent its freezing.]

[Footnote B:  *Isb., Chem.  Tech.*, 22, 481-487. 1876.]

Nitro-glycerine has a sweet taste, and causes great depression and vertigo.  It is soluble in ether, chloroform, benzene, glacial acetic acid, and nitro-benzene, in 1.75 part of methylated spirit, very nearly insoluble in water, and practically insoluble in carbon bisulphide.  Its formula is C\_{3}H\_{5}(NO\_{3})\_{3}, and molecular weight 227.  When pure, it may be kept any length of time without decomposition.  Berthelot kept a sample for ten years, and Mr G. M’Roberts, of the Ardeer Factory, for nine years, without their showing signs of decomposition; but if it should contain the smallest trace of free acid, decomposition is certain to be started before long.  This will generally show itself by the formation of little green spots in the gelatine compounds, or a green ring upon the surface of liquid nitro-glycerine.  Sunlight will often cause it to explode; in fact, a bucket containing some water that had been used to wash nitro-glycerine, and had been left standing in the sun, has in our experience been known to explode with considerable force.  Nitro-glycerine when pure is quite stable at ordinary temperatures, and samples have been kept for years without any trace of decomposition.  It is very susceptible to heat, and even when quite pure will not stand a temperature of 100 deg.  C. for a longer period than a few hours, without undergoing decomposition.  Up to a temperature of 45 deg.  C., however, properly made and purified nitro-glycerine will remain unchanged almost indefinitely.  The percentage composition of nitroglycerine is as follows:—­

Found.  Theory for C\_{3}H\_{5}(N0\_{2})\_{3}.

Carbon 15.62 15.86 per cent.
Hydrogen 2.40 2.20 "
Nitrogen 17.90 18.50 "
Oxygen ... 63.44 "

The above analysis is by Beckerheim.  Sauer and Adou give the nitrogen as 18.35 to 10.54 per cent. by Dumas’ method; but I have never found any difficulty in obtaining percentages as high as 18.46 by the use of Lunge’s nitrometer.  The decomposition products by explosion are shown by the following equation—­

 2C\_{3}H\_{5}(NO\_{3})\_{3} = 6CO\_{2} + 5H\_{2}O + 6N + O;

that is, it contains an excess of 3.52 per cent. of oxygen above that required for complete combustion; 100 grms. would be converted into—­

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Carbonic Acid (CO\_{2}) 58.15 per cent.
Water 19.83 "
Oxygen 3.52 per cent.
Nitrogen 18.50 "

The volume of gases produced at 0 deg. and 760 mm., calculated from the above, is 714 litres per kilo, the water being taken as gaseous.  Nitro-glycerine is decomposed differently if it is ignited as dynamite (i.e., kieselguhr dynamite), and if the gases are allowed to escape freely under a pressure nearly equal to that of the atmosphere.  Sarrau and Vieille obtained under these conditions, for 100 volumes of gas—­

NO 48.2 per cent.
CO 35.9 "
CO\_{2} 12.7 "
H 1.6 per cent.
N 1.3 "
CH\_{4} 0.3 "

These conditions are similar to those under which a mining charge, simply ignited by the cap, burns away slowly under a low pressure (i.e., a miss fire).  In a recent communication, P.F.  Chalon (*Engineering and Mining Journal*, 1892) says, that in practice nitro-glycerine vapour, carbon monoxide, and nitrous oxide, are also produced as the result of detonation, but he attributes their formation to the use of a too feeble detonator.

Nitro-glycerine explodes very violently by concussion.  It may be burned in an open vessel, but if heated above 250 deg.  C. it explodes.  Professor C.E.  Munroe gives the firing point as 2O3 deg.-2O5 deg.  C., and L. de Bruyn[A] states its boiling point as 185 deg..  He used the apparatus devised by Horsley.  The heat of formation of nitro-glycerine, as deduced from the heat of combustion by M. Longuinine, is 432 calories for 1 grm.; and the heat of combustion equals 1,576 cals. for 1 grm.  In the case of nitro-glycerine the heat of total combustion and the heat of complete decomposition are interchangeable terms, since it contains an excess of oxygen.  According to Dr W.H.  Perkin, F.R.S.,[B] the magnetic rotation of nitro-gylcerine is 5,407, and that of tri-methylene nitrate, 4.769 (diff. = .638).  Dr Perkin says:  “Had nitro-glycerine contained its nitrogen in any other combination with oxygen than as -O-NO\_{2}, as it might if its constitution had been represented as C\_{3}H\_{2}(NO\_{2})\_{3}(OH)\_{3}, the rotation when compared with propyl nitrate (4.085) would be abnormal.”

[Footnote A:  *Jour.  Soc.  Chem.  Ind.*, June 1896, p. 471.]

[Footnote B:  *Jour.  Chem.  Soc.*, W.H.  Perkin, 1889, p. 726.]

The solubility of nitro-glycerine in various solvents has been investigated by A.H.  Elliot; his results may be summarised as follows:—­

\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_

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\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_
| |
Solvent. | Cold. | Warm.
\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_|\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_|\_\_\_\_\_\_\_
\_\_\_\_\_\_\_\_\_\_\_
| |
Water | Insoluble | Slightly soluble
Alcohol, absolute | Soluble | Soluble
" 93% | " | "
" 80% | Slowly soluble | "
" 50% | Insoluble | Slightly soluble
Methyl alcohol | Soluble | Soluble
Amyl " | " | "
Ether, ethylic | " | "
" acetic | " | "
Chloroform | " | "
Acetone | " | "
Sulphuric acid (1.845) | " | "
Nitric acid (1.400) | Slowly soluble | "
Hydrochloric acid (1.200) | Insoluble, decomposed| Slowly soluble
Acetic acid, glacial | Soluble | Soluble
Carbolic acid | " | "
Astral oil | Insoluble | Insoluble
Olive " | Soluble | Soluble
Stearine oil | " | "
Mineral jelly | Insoluble | Insoluble
Glycerine | " | "
Benzene | Soluble | Soluble
Nitro-benzene | " | "
Toluene | " | "
Carbon bi-sulphide | Insoluble | Slightly affected
Turpentine | " | Soluble
Petroleum naphtha, 71 deg.-76 deg. B.| " | Insoluble
Caustic soda (1:10 solution) | Insoluble. | Insoluble.
Borax, 5% solution | " | "
Ammonia (.980) | " | " slightly
| | affected.
Ammonium sulph-hydrate | Insoluble, sulphur | Decomposed.
| separates |
Iron sulphate solution | Slightly affected | Affected.
Iron chloride (1.4 grm. Fe | Slowly affected | Decomposed.
to 10 c.c. N\_{2}O) | |
Tin chloride | Slightly affected | Affected.
\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_|\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_|\_\_\_\_\_\_\_
\_\_\_\_\_\_\_\_\_\_\_

Many attempts have been made to prepare nitro-glycerine
explosives capable of withstanding comparatively low
temperatures without freezing, but no satisfactory
solution of the problem has been found. Among
the substances that have been proposed and used with
more or less success, are nitro-benzene, nitro-toluene,
di-nitro-mono-chlorhydrine, solid nitro derivatives

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of toluene,[A] are stated to lower the freezing point
of nitro-glycerine to -20 deg.C. without altering
its sensitiveness and stability. The subject
has been investigated by S. Nauckhoff,[B] who states
that nitroglycerine can be cooled to temperatures (-40
deg. to -50 deg. C.) much below its true freezing
point, without solidifying, by the addition of various
substances. When cooled by means of a mixture
of solid carbon, dioxide, and ether, it sets to a
glassy mass, without any perceptible crystallisation.
The mass when warmed to 0 deg.C. first rapidly liquefies
and then begins to crystallise. The true freezing
point of pure nitro-glycerine was found to be 12.3
deg.C. The technical product, owing to the presence
of di-nitro-glycerine, freezes at 10.5 deg. C.
According to Raoult’s law, the lowering of the
freezing point caused by *m* grms. of a substance
with the molecular weight M, when dissolved in 100
grms. of the solvent, is expressed by the formula:
[Delta] = E(*m*/M), where E is a constant characteristic
for the solvent in question. The value of E for
nitro-glycerine was found to be 70.5 when calculated,
according to Van’t Hoff’s formula, from
the melting point and the latent heat of fusion of
the substance. Determinations of the lowering
of the freezing point of nitro-glycerine by additions
of benzene, nitro-benzene, di-nitro-benzene, tri-nitro-benzene,
p.-nitro-toluene, o.-nitro-toluene, di-nitro-toluene,
naphthalene, nitro-naphthalene, di-nitro-naphthalene,
ethyl acetate, ethyl nitrate, and methyl alcohol,
gave results agreeing fairly well with Raoult’s
formula, except in the case of methyl alcohol, for
which the calculated lowering of the freezing point
was greater than that observed, probably owing to
the formation of complex molecules in the solution.
The results show that, in general, the capacity of
a substance to lower the freezing point of nitro-glycerine
depends, not upon its freezing point, or its chemical
composition or constitution, but upon its molecular
weight. Nauckhoff states that a suitable substance
for dissolving in nitro-glycerine, in order to lower
the freezing point of the latter, must have a relatively
low molecular weight, must not appreciably diminish
the explosive power and stability of the explosive,
and must not be easily volatile at relatively high
atmospheric temperatures; it should, if possible,
be a solvent of nitro-cellulose, and in every case
must not have a prejudicial influence on the gelatinisation
of the nitro-cellulose.

[Footnote A: Eng. Pat. 25,797, November
1904.]

[Footnote B: *Z. Angew. Chem.*,
1905, 18, 11-22, 53-60.]
 *Manufacture of Nitro-Glycerine.*—­Nitro-glycerine
is prepared upon the manufacturing scale by gradually
adding glycerine to a mixture of nitric and sulphuric
acids of great strength. The mixed acids are contained
in a lead vessel, which is kept cool by a stream of
water continually passing through worms in the interior
of the nitrating vessel, and the glycerine is gradually
added in the form of a fine stream from above.
The manufacture can be divided into three distinct
operations, *viz*., nitration, separation, and
washing, and it will be well to describe these operations
in the above order.

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*Nitration.*—­The most essential condition
of nitrating is the correct composition and strength
of the mixed acids. The best proportions have
been found to be three parts by weight of nitric acid
of a specific gravity 1.525 to 1.530, and containing
as small a portion of the oxides of nitrogen as possible,
to five parts by weight of sulphuric acid of a specific
gravity of 1.840 at 15 deg. C., and about 97 per
cent. of mono-hydrate. It is of the very greatest
importance that the nitric acid should be as strong
as possible. Nothing under a gravity of 1.52 should
ever be used even to mix with stronger acid, and the
nitration will be proportional to the strength of
the acid used, provided the sulphuric acid is also
strong enough. It is also of great importance
that the oxides of nitrogen should be low, and that
they should be kept down to as low as 1 per cent.,
or even lower. It is also very desirable that
the nitric acid should contain as little chlorine
as possible. The following is the analysis of
a sample of nitric acid, which gave very good results
upon the commercial scale:—­Specific gravity,
1.525, N\_{2}O\_{4}, 1.03 per cent.; nitric acid (HNO\_{3}),
95.58 per cent.

The amount of real nitric acid (mono-hydrate) and
the amount of nitric peroxide present in any sample
should always be determined before it is used for
nitrating purposes. The specific gravity is not
a sufficient guide to the strength of the acid, as
an acid having a high gravity, due to some 3 or 4
per cent of nitric oxides in solution, will give very
poor nitration results. A tenth normal solution
of sodium hydroxide (NaOH), with phenol-phthalein
as indicator, will be found the most convenient method
of determining the total acid present. The following
method will be found to be very rapid and reliable:—­Weigh
a 100 c.c. flask, containing a few cubic centimetres
of distilled water, and then add from a pipette 1
c.c. of the nitric acid to be examined, and reweigh
(this gives the weight of acid taken). Now make
up to 100 c.c. at 15 deg. C.; shake well, and
take out 10 c.c. with a pipette; drain into a small
Erlenmeyer flask, and add a little of the phenol-phthalein
solution, and titrate with the tenth normal soda solution.

The nitric peroxide can be determined with a solution
of potassium permanganate of N/10 strength, thus:
Take a small conical flask, containing about 10 c.c.
of water, and add from a burette 10 to 16 c.c. of
the permanganate solution; then add 2 c.c. of the acid
to be tested, and shake gently, and continue to add
permanganate solution as long as it is decolourised,
and until a faint pink colour is permanent.
 *Example.* N/10 permanganate 3.16 grms. per litre,
1 c.c. = O.0046 grm. N\_{2}O\_{4}, 2 c.c. of sample
of acid specific gravity 1.52 = 3.04 grms. taken for
analysis. Took 20 c.c. permanganate solution,
O.0046 x 20 =.092 grm. N\_{2}O\_{4}, and (.092
x 100)/3.04 = 3.02 per cent. N\_{2}O\_{4}.
The specific gravity should be taken with an hydrometer
that gives the specific gravity directly, or, if preferred,
the 2 c.c. of acid may be weighed.

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A very good method of rapidly determining the strength
of the sulphuric acid is as follows:—­Weigh
out in a small weighing bottle, as nearly as possible,
2.45 grms. This is best done by running in 1.33
c.c. of the acid (1.33 x 1.84 = 2.447). Wash
into a large Erlenmeyer flask, carefully washing out
the bottle, and also the stopper, &c. Add a drop
of phenol-phthalein solution and titrate, with a
half normal solution of sodium hydrate (use a 100
c.c. burette). Then if 2.45 grms. exactly have
been taken, the readings on the burette will equal
percentages of H\_{2}SO\_{4} (mono-hydrate) if not,
calculate thus:—­2.444 grms. weighed, required
95.4 c.c. NaOH. Then—­

2.444 : 95.4 :: 2.45 : *x* = 95.64
per cent. H\_{2}SO\_{4}.

It has been proposed to free nitric acid from the
oxides of nitrogen by blowing compressed air through
it, and thus driving the gases in solution out.
The acid was contained in a closed lead tank, from
which the escaping fumes were conducted into the chimney
shaft, and on the bottom of which was a lead pipe,
bent in the form of a circle, and pierced with holes,
through which the compressed air was made to pass;
but the process was not found to be of a very satisfactory
nature, and it is certainly better not to allow the
formation of these compounds in the manufacture of
the acid in the first instance. Another plan,
however, is to heat the acid gently, and thus drive
out the nitrous gases. Both processes involve
loss of nitric acid.

Having obtained nitric and sulphuric acids as pure
as possible, the next operation is to mix them.
This is best done by weighing the carboys in which
the acids are generally stored before the acids are
drawn off into them from the condensers, and keeping
their weights constantly attached to them by means
of a label. It is then a simple matter to weigh
off as many carboys of acid as may be required for
any number of mixings, and subtract the weights of
the carboys. The two acids should, after being
weighed, be poured into a tank and mixed, and subsequently
allowed to flow into an acid egg or montjus, to be
afterwards forced up to the nitrating house in the
danger area. The montjus or acid egg is a strong
cast-iron tank, of either an egg shape, or a cylinder
with a round end. If of the former shape, it
would lie on its side, and upon the surface of the
ground, and would have a manhole at one end, upon
which a lid would be strongly bolted down; but if
of the latter shape, the lid, of course, is upon the
top, and the montjus itself is let into the ground.
In either case, the principle is the same. One
pipe, made of stout lead, goes to the bottom, and another
just inside to convey the compressed air, the acids
flowing away as the pressure is put on, just as blowing
down one tube of an ordinary wash-bottle forces the
water up the other tube to the jet. The pressure
necessarily will, of course, vary immensely, and will
depend upon the height to which the acid has to be
raised and the distance to be traversed.

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The mixed acids having been forced up to the danger
area, and to a level higher than the position of the
nitrating house, should, before being used, be allowed
to cool, and leaden tanks of sufficient capacity to
hold at least enough acid for four or five nitrations
should be placed in a wooden house upon a level at
least 6 or 7 feet above the nitrating house.
In this house also should be a smaller lead tank, holding,
when filled to a certain mark, just enough of the
mixed acids for one nitration. The object of
this tank is, that as soon as the man in charge knows
that the last nitration is finished, he refills this
smaller tank (which contains just enough of the mixed
acids), and allows its contents to flow down into
the nitrating house and into the nitrator, ready for
the next nitration. The nitration is usually
conducted in a vessel constructed of lead, some 4
feet wide at the bottom, and rather less at the top,
and about 4 feet or so high. The size, of course,
depends upon the volume of the charge it is intended
to nitrate at one operation, but it is always better
that the tank should be only two-thirds full.
A good charge is 16 cwt. of the mixed acids, in the
proportion of three to five; that is, 6 cwt. of nitric
acid, and 10 cwt. of sulphuric acid, and 247 lbs.
of glycerine.

Upon reference to the equation showing the formation
of nitro-glycerine, it will be seen that for every
1 lb. of glycerine 2.47 lbs. of nitro-glycerine should
be furnished,[A] but in practice the yield is only
a little over 2 lbs., the loss being accounted for
by the unavoidable formation of some of the lower
nitrate of glycerine (the mono-nitrate), which afterward
dissolves in the washing waters. The lead tank
(Fig. 5) is generally cased in woodwork, with a platform
in front for the man in charge of the nitrating to
stand upon, and whence to work the various taps.
The top of the tank is closed in with a dome of lead,
in which is a small glass window, through which the
progress of the nitrating operation can be watched.
From the top of this dome is a tube of lead which is
carried up through the roof of the building. It
serves as a chimney to carry off the acid fumes which
are given off during the nitration. The interior
of this tank contains at least three concentric spirals
of at least 1-inch lead pipe, through which water
can be made to flow during the *whole* operation
of nitrating. Another lead pipe is carried through
the dome of the tank, as far as the bottom, where
it is bent round in the form of a circle. Through
this pipe, which is pierced with small holes, about
1 inch apart, compressed air is forced at a pressure
of about 60 lbs. in order to keep the liquids in a
state of constant agitation during the whole period
of nitration. There must also be a rather wide
pipe, of say 2 inches internal diameter, carried through
the dome of the tank, which will serve to carry the
mixed acid to be used in the operation into the tank.
There is still another pipe to go through the dome,
*viz*., one to carry the glycerine into the tank.
This need not be a large bore pipe, as the glycerine
is generally added to the mixed acids in a thin stream
(an injector is often used).

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[Footnote A: Thus if 92 lbs. glycerine give 227
lbs. nitro-glycerine, (277 x 1)/92 = 2.47 lbs.]

[Illustration: FIG. 5.—­TOP OF NITRATOR.
*A*, Fume Pipe; *B*, Water Pipes for Cooling;
*C*, Acid Mixture Pipe; *E*, Compressed Air;
*G*, Glycerine Pipe and Funnel; *T*, Thermometer;
*W*, Window.]

Before the apparatus is ready for use, it requires
to have two thermometers fixed, one long one to reach
to the bottom of the tank, and one short one just
long enough to dip under the surface of the acids.
When the tank contains its charge, the former gives
the temperature of the bottom, and the latter of the
top of the mixture. The glycerine should be contained
in a small cistern, fixed in some convenient spot upon
the wall of the nitrating house, and should have a
pipe let in flush with the bottom, and going through
the dome of the nitrating apparatus. It must of
course be provided with a tap or stop-cock, which should
be placed just above the point where the pipe goes
through the lead dome.

Some method of measuring the quantity of glycerine
used must be adopted. A gauge-tube graduated
in inches is a very good plan, but it is essential
that the graduations should be clearly visible to the
operator upon the platform in front of the apparatus.
A large tap made of earthenware (and covered with
lead) is fixed in the side of the nitrating tank just
above the bottom, to run off the charge after nitration.
This should be so arranged that the charge may be
at option run down the conduit to the next house or
discharged into a drowning tank, which may sometimes
be necessary in cases of decomposition. The drowning
tank is generally some 3 or 4 yards long and several
feet deep, lined with cement, and placed close outside
the building.

The apparatus having received a charge of mixed acids,
the water is started running through the pipes coiled
inside the tank, and a slight pressure of compressed
air is turned on,[A] to mix the acids up well before
starting. The nitration should not be commenced
until the two thermometers register a temperature
of 18 deg. C. The glycerine tap is then partially
opened, and the glycerine slowly admitted, and the
compressed air turned on full, until the contents
of the apparatus are in a state of very brisk agitation.
A pressure of about 40 lbs. is about the minimum (if
247 lbs. of glycerine and 16 cwt. of acids are in the
tank). If the glycerine tube is fitted with an
injector, it may be turned on almost at once.
The nitration will take about thirty minutes to complete,
but the compressed air and water should be kept on
for an additional ten minutes after this, to give
time for all the glycerine to nitrate. The temperature
should be kept as low as possible (not above 18 deg.
C.).

[Footnote A: At the Halton Factory, Germany,
cylinders of compressed carbon dioxide are connected
with the air pipes so that in the event of a failure
of the air supply the stirring can be continued with
this gas if necessary.]

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The chief points to attend to during the progress
of the nitration are—­

1. The temperature registered by the two thermometers.

2. The colour of the nitrous fumes given off
(as seen through the little window in the dome of
the apparatus).

3. The pressure of the compressed air as seen
from a gauge fixed upon the air pipe just before it
enters the apparatus.

4. The gauge showing the quantity of glycerine
used. The temperature, as shown by either of
the two thermometers, should not be at any time higher
than 25 deg. C.

If it rises much above this point, the glycerine should
be at once shut off, and the pressure of air increased
for some few minutes until the temperature falls,
and no more red fumes are given off.

The nitration being finished, the large earthenware
tap at the bottom of the tank is opened, and the charge
allowed to flow away down the conduit to the next
building, *i.e*., to the separator.

The nitrating house is best built of wood, and should
have a close-boarded floor, which should be kept scrupulously
clean, and free from grit and sand. A wooden
pail and a sponge should be kept in the house in order
that the workman may at once clean up any mess that
may be made, and a small broom should be handy, in
order that any sand, &c., may be at once removed.
It is a good plan for the nitrator to keep a book in
which he records the time of starting each nitration,
the temperature at starting and at the finish, the
time occupied, and the date and number of the charge,
as this enables the foreman of the danger area at any
time to see how many charges have been nitrated, and
gives him other useful information conducive to safe
working. Edward Liebert has devised an improvement
in the treatment of nitro-glycerine. He adds ammonium
sulphate or ammonium nitrate to the mixed acids during
the operation of nitrating, which he claims destroys
the nitrous acid formed according to the equation—­

(NH\_{4})\_{2}SO\_{4} + 2HNO\_{3} = H\_{2}SO\_{4} + 2N\_{2}
+ 4H\_{2}O.

I am not aware that this modification of the process
of nitration is in use at the present time.

The newly made charge of nitro-glycerine, upon leaving
the nitrating house, flows away down the conduit,
either made of rubber pipes, or better still, of woodwork,
lined with lead and covered with lids made of wood
(in short lengths), in order that by lifting them
at any point the condition of the conduit can be examined,
as this is of the greatest importance, and the conduit
requires to be frequently washed out and the sulphate
of lead removed. This sulphate always contains
nitro-glycerine, and should therefore be burnt in
some spot far removed from any danger building or
magazine, as it frequently explodes with considerable
violence.

[Illustration: FIG. 6.—­SMALL NITRATOR.
*N*, Tap for Discharging; *P*, Water Pipes;
*T*, Thermometer; *W*, Windows; *P’*,
Glycerine Pipe.]

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In works where the manufacture of nitro-glycerine
is of secondary importance, and some explosive containing
only perhaps 10 per cent. of nitroglycerine is manufactured,
and where 50 or 100 lbs. of glycerine are nitrated
at one time, a very much smaller nitrating apparatus
than the one that has been already described will
be probably all that is required. In this case
the form of apparatus shown in Fig. 6 will be found
very satisfactory. It should be made of stout
lead (all lead used for tanks, &c., must be “chemical
lead"), and may be made to hold 50 or 100 lbs. as
found most convenient. This nitrator can very
well be placed in the same house as the separator;
in fact, where such a small quantity of nitro-glycerine
is required, the whole series of operations, nitrating,
separation, and washing, &c., may very well be performed
in the same building. It will of course be necessary
to place the nitrator on a higher level than the separator,
but this can easily be done by having platforms of
different heights, the nitration being performed upon
the highest. The construction of this nitrator
is essentially the same as in the larger one, the
shape only being somewhat different. Two water
coils will probably be enough, and one thermometer.
It will not be necessary to cover this form in with
woodwork.
 *The Nathan Nitrator.*[A]—­This nitrator
is the patent of Lt. Col. F.L. Nathan
and Messrs J.M. Thomson and W. Rintoul of Waltham
Abbey, and will probably before long entirely supersede
all the other forms of nitrator on account of its
efficiency and economy of working. With this nitrator
it is possible to obtain from 2.21 to 2.22 parts of
nitro-glycerine from every 1 part of glycerine.
The apparatus is so arranged that the nitration of
the glycerine, the separation of nitro-glycerine produced,
as well as the operation of “after-separation,”
are carried out in one vessel. The usual nitrating
vessel is provided with an acid inlet pipe at the bottom,
and a glass separation cylinder with a lateral exit
or overflow pipe at the top. This cylinder is
covered by a glass hood or bell jar during nitration
to direct the escaping air and fumes into a fume pipe
where the flow of the latter may be assisted by an
air injector. The lateral pipe in the separation
cylinder is in connection with a funnel leading to
the prewash tank. The drawing (Fig. 7) shows
a vertical section of the apparatus; *a* is the
nitrating vessel of usual construction, having at the
bottom an acid inlet pipe with three branches, one
leading to the de-nitrating plant, *c* leading
to the drowning tank, and *d*, which extends upwards
and has two branches, *e* leading to the nitrating
acids tank, and *f* to the waste acid tank.
On the sloped bottom of the nitrating vessel *a*
lies a coil *g* of perforated pipe for blowing
air, and there are in the vessel several coils *h*,
three shown in the drawing, for circulation of cooling

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water. At the top of the vessel there is a glass
cylinder *i*, having a lateral outlet *j*
directed into the funnel mouth of a pipe *k* leading
to the prewash tank. Over the cylinder *i*
is a glass globe *l*, into which opens a pipe
*m* for leading off fumes which may be promoted
by a compressed air jet from a pipe *r* operating
as an injector. Into an opening of the glass
dome *l* is inserted a vessel *n*, which
is connected by a flexible pipe *p* to the glycerine
tank, and from the bottom of *n*, which is perforated
and covered with a disc perforated with holes registering
with those through the bottom, this disc being connected
by a stem with a knob *q* by which it can be
turned so as to throttle or cut off passage of glycerine
through the bottom. *s* is a thermometer for
indicating the temperature of the contents of the vessel.

[Footnote A: Eng. Pat. 15,983, August 1901.]

[Illustration: FIG. 7.—­NATHAN’S
NITRATOR FOR NITRO-GLYCERINE. (*a*) Nitrating
Vessel; (*b*) to Separating Vessel; (*c*)
to Drowning Tank; (*e*) Nitrating Acids enter
(*f*) to the Waste Acids; (*g*) Coils for
Compressed Air; (*h*) Pipes for Cooling Water;
(*i*) Glass Cylinder; (*j*) Outlet to *k*;
(*k*) leading to Prewash Tank; (*l*) Glass
Dome; (*m*) Pipe to lead off for Escape of Fumes;
(*n*) Vessel; (*p*) Pipe conveying Glycerine;
(*q*) Knob to turn off Glycerine; (*r*) Compressed
Air Jet; (*s*) Thermometer.]

In operating with this apparatus the nitrating acid
is introduced into the nitrating vessel by opening
the cock of the pipe *e*. The glycerine is
then run in by introducing *n* and opening the
valve at its bottom, the contents of the vessel being
agitated by air blown through the perforations of
the pipe *g*. When the glycerine is all nitrated
and the temperature has slightly fallen, the circulation
of the water through the coils *h* and the air-stirring
are stopped, and the glycerine supply vessel *n*
is removed. The nitro-glycerine as it separates
from the acids is raised by introducing by the pipe
*f* waste acid from a previous charge, this displacing
the nitro-glycerine upwards and causing it to flow
by the outlet, *j* and pipe *k* to the prewash
tank. When nearly all the nitro-glycerine has
been separated in this manner the acids in the apparatus
may be run off by the pipe *b* to an after separating
vessel for further settling, thus leaving the apparatus
free for another nitration, or the nitrating vessel
itself may be used as an after separating bottle displacing
the nitro-glycerine with waste acid as it rises to
the top, or skimming off in the usual manner.
When the separation of the nitro-glycerine is complete
the waste acid is run off and denitrated as usual,
a portion of it being reserved for the displacement
of the nitro-glycerine in a subsequent operation.

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In a further patent (Eng. Pat. 3,020, 1903) the
authors propose with the object of preventing the
formation and separation of nitro-glycerine in the
waste acids, after the nitro-glycerine initially formed
in the nitrating vessel has been separated and removed,
to add a small quantity of water to the waste acids;
this is carried out as follows. A relatively
small quantity of water is added, and this prevents
all further separation of nitro-glycerine, and at
the same time the strength of the waste acids is so
slightly reduced that their separation and re-concentration
are not affected. “After-separation”
is thus done away with, and the nitro-glycerine plant
simplified and its output increased. After nitration
separation is commenced at a temperature such that
when all the displacing acid has been added, and the
separation of the nitro-glycerine is complete, the
temperature of the contents of the nitrating vessel
shall not be lower than 15 deg. C. A sufficient
quantity of the displacing acid is then run off through
the waste-acid cock to allow of the remaining acids
being air-stirred without splashing over the top.
A small quantity of water, from 2 to 3 per cent. according
to strength of acid; if waste consists of sulphuric
acid (monohydrate), 62 per cent.; nitric acid (anhydrous),
33 per cent. and water 5 per cent.; temperature 15
deg. C., then 2 per cent. of water is added;
if waste acids contain less than 4 per cent. of water
of temperature lower than 15 deg. C., from 3 to
5 per cent. of water may have to be added. The
water is added slowly through the separator cylinder,
and the contents of the nitrator air-stirred, but not
cooled, the temperature being allowed to rise slowly
and regularly as the water is added—­usually
about 3 deg. C. for each per cent. of water added.
When air-agitation has been stopped, the acids are
kept at rest for a short time, in order to allow of
any small quantity of initially formed nitro-glycerine
adhering to the coils and sides of the vessel rising
to the top. When this has been separated by displacement,
the acids are ready for denitration, or can be safely
stored without further precaution.
 *Separation.*—­The nitro-glycerine,
together with the mixed acids, flows from the nitrating
house to the separating house, which must be on a lower
level than the former. The separating house contains
a large lead-lined tank, closed in at the top with
a wooden lid, into which a lead pipe of large bore
is fixed, and which is carried up through the roof
of the building, and acts as a chimney to carry off
any fumes. A little glass window should be fixed
in this pipe in order that the colour of the escaping
fumes may be seen. The conduit conveying the nitro-glycerine
enters the building close under the roof, and discharges
its contents into the tank through the pipe G (Fig.
8). The tank is only about two-thirds filled
by the charge. There is in the side of the tank
a small window of thick plate glass, which enables
the workman to see the level of the charge, and also
to observe the progress of the separation, which will
take from thirty minutes to one hour.

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The tank should be in connection with a drowning tank,
as the charge sometimes gets very dangerous in this
building. It must also be connected by a conduit
with the filter house, and also to the secondary separator
by another conduit. The tank should also be fitted
with a compressed air pipe, bent in the form of a
loop. It should lie upon the bottom of the vat.
The object of this is to mix up the charge in case
it should get too hot through decomposition.
A thermometer should of course be fixed in the lid
of the tank, and its bulb should reach down to the
middle of the nitro-glycerine (which rests upon the
surface of the mixed acids, the specific gravity of
the nitro-glycerine being 1.6, and that of the waste
acids 1.7; the composition of the acids is now 11 per
cent. HNO\_{3}, 67 per cent. H\_{2}SO\_{4},
and 22 per cent. water), and the temperature carefully
watched.

[Illustration: FIG. 8.—­SEPARATOR.
*A*, Compressed Air Pipes; *G*, Nitro-glycerine
enters from Nitrator; *N*, Nitro-glycerine to
*P*; *L*, Lantern Window; *W*, Window
in Side; *S*, Waste Acids to Secondary Separator;
*T*, Tap to remove last traces of Nitro-glycerine;
*P*, Lead Washing Tank; *A*, Compressed
Air; *W*, Water Pipe; *N*, Nitro-glycerine
from Separator.]

If nothing unusual occurs, and it has not been necessary
to bring the compressed air into use, and so disturb
the process of separation, the waste acids may be
run away from beneath the nitro-glycerine, and allowed
to flow away to the secondary separator, where any
further quantity of nitro-glycerine that they contain
separates out after resting for some days. The
nitro-glycerine itself is run into a smaller tank in
the same house, where it is washed three or four times
with its own bulk of water, containing about 3 lbs.
of carbonate of soda to neutralise the remaining acid.
This smaller tank should contain a lead pipe, pierced
and coiled upon the bottom, through which compressed
air may be passed, in order to stir up the charge
with the water and soda. After this preliminary
washing, the nitro-glycerine is drawn off into indiarubber
buckets, and poured down the conduit to the filter
house. The wash waters may be sent down a conduit
to another building, in order to allow the small quantity
of nitro-glycerine that has been retained in the water
as minute globules to settle, if thought worth the
trouble of saving. This, of course, will depend
upon the usual out-turn of nitro-glycerine in a day,
and the general scale of operations.

[Illustration: FIG. 9.—­FILTERING AND
WASHING PLANT. *W*, Lead Washing Tank; *WP*,
Water Pipe; *L*, Lid; *S*, Nitro-glycerine
from Separator; *A, B, C*, Filtering Tanks; *B2*,
Indiarubber Bucket.]

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*Filtering and Washing.*—­The filter
house (Fig. 9), which must of course be again on a
somewhat lower level than the separating house, must
be a considerably larger building than either the
nitrating or separating houses, as it is always necessary
to be washing some five or six charges at the same
time. Upon the arrival of the nitro-glycerine
at this house, it first flows into a lead-lined wooden
tank (W), containing a compressed air pipe, just like
the one in the small tank in the separating house.
This tank is half filled with water, and the compressed
air is turned on from half to a quarter of an hour
after the introduction of the charge. The water
is then drawn off, and fresh water added. Four
or five washings are generally necessary. The
nitro-glycerine is then run into the next tank (A),
the top of which is on a level with the bottom of the
first one. Across the top of this tank is stretched
a frame of flannel, through which the nitroglycerine
has to filter. This removes any solid matters,
such as dirt or scum. Upon leaving this tank,
it passes through a similar flannel frame across another
tank (B), and is finally drawn off by a tap in the
bottom of the tank into rubber buckets. The taps
in these tanks are best made of vulcanite.

At this stage, a sample should be taken to the laboratory
and tested. If the sample will not pass the tests,
which is often the case, the charge must be rewashed
for one hour, or some other time, according to the
judgment of the chemist in charge. In the case
of an obstinate charge, it is of much more avail to
wash a large number of times with small quantities
of water, and for a short time, than to use a lot of
water and wash for half an hour. Plenty of compressed
air should be used, as the compound nitric ethers
which are formed are thus got rid of. As five
or six charges are often in this house at one time,
it is necessary to have as many tanks arranged in
tiers, otherwise one or two refractory charges would
stop the nitrating house and the rest of the nitro-glycerine
plant. The chief causes of the washed material
not passing the heat test are, either that the acids
were not clean, or they contained objectionable impurities,
or more frequently, the quality of the glycerine used.
The glycerine used for making nitro-glycerine should
conform to the following tests, some of which, however,
are of greater importance than others. The glycerine
should—­

1. Have minimum specific gravity at 15 deg.
C. of 1.261.

2. Should nitrify well.

3. Separation should be sharp within half an
hour, without the separation of flocculent matter,
nor should any white flocculent matter (due to fatty
acids) be formed when the nitrated glycerine is thrown
into water and neutralised with carbonate of soda.

4. Should be free from lime and chlorine, and
contain only traces of arsenic, sulphuric acid, &c.

5. Should not leave more than 0.25 per cent.
of inorganic and organic residue together when evaporated
in a platinum dish without ebullition (about 160 deg.
C.) or partial decomposition.

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6. Silver test fair.

7. The glycerine, when diluted one-half, should
give no deposit or separation of fatty acids when
nitric peroxide gas is passed through it. (Nos.
1, 2, 3, and 5 are the most essential.)

The white flocculent matter sometimes formed is a
very great nuisance, and any sample of glycerol which
gives such a precipitate when tried in the laboratory
should at once be rejected, as it will give no end
of trouble in the separating house, and also in the
filter house, and it will be very difficult indeed
to make the nitro-glycerine pass the heat test.
The out-turn of nitro-glycerine also will be very
low. The trouble will show itself chiefly in
the separating operation. Very often 2 or 3 inches
will rise to the surface or hang about in the nitro-glycerine,
and at the point of contact between it and the mixed
acids, and will afterwards be very difficult to get
rid of by filtration. The material appears to
be partly an emulsion of the glycerine, and partly
due to fatty acids, and as there appears to be no
really satisfactory method of preventing its formation,
or of getting rid of it, the better plan is not to
use any glycerine for nitrating that has been found
by experiment upon the laboratory scale to give this
objectionable matter. One of the most useful methods
of testing the glycerine, other than nitrating, is
to dilute the sample one-half with water, and then
to pass a current of nitric peroxide gas through it,
when a flocculent precipitate of elaidic acid (less
soluble in glycerine than the original oleic acid)
will be formed. Nitrogen peroxide, N\_{2}O\_{4},
is best obtained by heating dry lead nitrate (see
Allen, “Commercial Organic Analysis,”
vol. ii., 301).

When a sample of nitro-glycerine is brought to the
laboratory from the filter house, it should first
be examined to see that it is not acid.[A] A weak
solution of Congo red or methyl orange may be used.
If it appears to be decidedly alkaline, it should
be poured into a separating funnel, and shaken with
a little distilled water. This should be repeated,
and the washings (about 400 c.c.) run into a beaker,
a drop of Congo red or methyl orange added, and a
drop or so of N/2 hydrochloric acid added, when it
should give, with two or three drops at most, a blue
colour with the Congo red, or pink with the methyl
orange, &c. The object of this test is to show
that the nitro-glycerine is free from any excess of
soda, *i.e*., that the soda has been properly washed
out, otherwise the heat test will show the sample
to be better than it is. The heat test must also
be applied.

[Footnote A: A. Leroux, *Bul. Soc.
Chim. de Bel.*, xix., August 1905, contends that
experience does not warrant the assumption that free
acid is a source of danger in nitro-glycerine or nitro-cellulose;
free alkali, he states, promotes their decomposition.]

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Upon leaving the filter house, where it has been washed
and filtered, and has satisfactorily passed the heat
test, it is drawn off from the lowest tank in indiarubber
buckets, and poured down the conduit leading to the
precipitating house, where it is allowed to stand for
a day, or sometimes longer, in order to allow the
little water it still contains to rise to the surface.
In order to accomplish this, it is sufficient to allow
it to stand in covered-in tanks of a conical form,
and about 3 or 4 feet high. In many works it
is previously filtered through common salt, which of
course absorbs the last traces of water. It is
then of a pale yellow colour, and should be quite
clear, and can be drawn off by means of a tap (of
vulcanite), fixed at the bottom of the tanks, into
rubber buckets, and is ready for use in the preparation
of dynamite, or any of the various forms of gelatine
compounds, smokeless powders, &c., such as cordite,
ballistite, and many others.

Mikolajezak (*Chem. Zeit.*, 1904, Rep. 174)
states that he has prepared mono- and di-nitro-glycerine,
and believes that the latter compound will form a
valuable basis for explosives, as it is unfreezable.
It is stated to be an odourless, unfreezable oil,
less sensitive to percussion, friction, and increase
of temperature, and to possess a greater solvent power
for collodion-cotton than ordinary nitro-glycerine.
It can thus be used for the preparation of explosives
of high stability, which will maintain their plastic
nature even in winter. The di-nitro-glycerine
is a solvent for tri-nitro-glycerine, it can therefore
be mixed with this substance, in the various gelatine
explosives in order to lower the freezing point.
 *The Waste Acids.*—­The waste acids
from the separating house, from which the nitro-glycerine
has been as completely separated as possible, are run
down the conduit to the secondary separator, in order
to recover the last traces of nitro-glycerine that
they contain. The composition of the waste acids
is generally somewhat as follows:—­Specific
gravity, 1.7075 at 15 deg. C.; sulphuric acid,
67.2 per cent.; nitric acid, 11.05 per cent.; and
water, 21.7 per cent., with perhaps as much as 2 per
cent. of nitric oxide, and of course varying quantities
of nitro-glycerine, which must be separated, as it
is impossible to run this liquid away (unless it can
be run into the sea) or to recover the acids by distillation
as long as it contains this substance. The mixture,
therefore, is generally run into large circular lead-lined
tanks, covered in, and very much like the nitrating
apparatus in construction, that is, they contain worms
coiled round inside, to allow of water being run through
to keep the mixture cool, and a compressed air pipe,
in order to agitate the mixture if necessary.
The top also should contain a window, in order to allow
of the interior being seen, and should have a leaden
chimney to carry off the fumes which may arise from
decomposition. It is also useful to have a glass
tube of 3 or 4 inches in diameter substituted for about
a foot of the lead chimney, in order that the man
on duty can at any time see the colour of the fumes
arising from the liquid. There should also be
two thermometers, one long one reaching to the bottom
of the tank, and one to just a few inches below the
surface of the liquid.

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The nitro-glycerine, of course, collects upon the
surface, and can be drawn off by a tap placed at a
convenient height for the purpose. The cover
of the tank is generally conical, and is joined to
a glass cylinder, which is cemented to the top of
this lead cover, and also to the lead chimney.
In this glass cylinder is a hole into which fits a
ground glass stopper, through which the nitro-glycerine
can be drawn off. There will probably never be
more than an inch of nitro-glycerine at the most, and
seldom that. It should be taken to the filter
house and treated along with another charge.
The acids themselves may either be run to waste, or
better treated by some denitration plant. This
house probably requires more attention than any other
in the danger area, on account of the danger of the
decomposition of the small quantities of nitro-glycerine,
which, as it is mixed with such a large quantity of
acids and water, is very apt to become hot, and decomposition,
which sets up in spots where a little globule of nitro-glycerine
is floating, surrounded by acids that gradually get
hot, gives off nitrous fumes, and perhaps explodes,
and thus causes the sudden explosion of the whole.
The only way to prevent this is for the workman in
charge to look at the thermometers *frequently*,
and at the colour of the escaping fumes, and if he
should notice a rise of temperature or any appearance
of red fumes, to turn on the water and air, and stir
up the mixture, when probably the temperature will
suddenly fall, and the fumes cease to come off.

The cause of explosions in this building is either
the non-attention of the workmen in charge, or the
bursting of one of the water pipes, by which means,
of course, the water, finding its way into the acids,
causes a sudden rise of temperature. If the latter
of these two causes should occur, the water should
at once be shut off and the air turned on full, but
if it is seen that an explosion is likely to occur,
the tank should at once be emptied by allowing its
contents to run away into a drowning tank placed close
outside the house, which should be about 4 feet deep,
and some 16 feet long by 6 feet wide; in fact, large
enough to hold a considerable quantity of water.
But this last course should only be resorted to as
a last extremity, as it is extremely troublesome to
recover the small quantity of nitro-glycerine from
the bottom of this tank, which is generally a bricked
and cemented excavation some few yards from the house.

It has been proposed to treat these waste acids, containing
nitro-glycerine, in Mr M. Prentice’s nitric
acid retort. In this case they would be run into
the retort, together with nitrate of soda, in a fine
stream, and the small quantity of nitro-glycerine,
coming into contact with the hot mixture already in
the retort, would probably be at once decomposed.
This process, although not yet tried, promises to be
a success. Several processes have been used for
the denitration of these acids.

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*Treatment of the Waste Acid from the Manufacture
of Nitro-Glycerine and Gun-Cotton.*—­The
composition of these acids is as follows:—­

 Nitro-glycerine
 and Gun-cotton
 Waste
Acid.

Sulphuric acid 70 per cent. 78 per cent.
Nitric acid 10 " 12 "
Water 20 " 10 "

The waste acid from the manufacture of gun-cotton
is generally used direct for the manufacture of nitric
acid, as it contains a fairly large amount of sulphuric
acid, and the small amount of nitro-cellulose which
it also generally contains decomposes gradually and
without explosion in the retort. Nitric acid
may be first distilled off, the resulting sulphuric
acid being then added to the equivalent amount of nitrate
of soda. Nitric acid is then distilled over and
condensed in the usual way. Very often, however,
the waste acid is added direct to the charge of nitrate
without previously eliminating the nitric acid.
The treatment of the waste acid from the manufacture
of nitro-glycerine is somewhat different. The
small amount of nitro-glycerine in this acid must
always be eliminated. This is effected either
by allowing the waste acid to stand for at least twenty-four
hours in a big vessel with a conical top, where all
the nitro-glycerine which will have separated to
the surface is removed by skimming; or, better still,
the “watering down process” of Col.
Nathan may be employed. In Nathan’s nitrator
every existing trace of nitro-glycerine is separated
from the acids in a few hours after the nitration,
and any further formation of nitro-glycerine is prevented
by adding about 2 per cent. of water to the waste
acids, which are kept agitated during the addition.
The waste acid, now free from nitro-glycerine, but
which may still contain organic matter, is denitrated
by bringing it into contact with a jet of steam.
The waste acid is passed in a small stream down through
a tower of acid-resisting stoneware (volvic stone),
which is closely packed with earthenware, and at the
bottom of which is the steam jet. Decomposition
proceeds as the acid meets the steam, nitric and nitrous
acids are disengaged and are passed out at the top
of the tower through a pipe to a series of condensers
and towers, where the nitric acid is collected.
The nitrous acid may be converted into nitric acid
by introducing a hot compressed air jet into the gases
before they pass into the condensers. Weak sulphuric
acid of sp. gr. 1.6 collects in a saucer in which
the tower stands, and is then passed through a cooling
worm. The weak sulphuric acid, now entirely free
from nitric and nitrous acids, may be concentrated
to sp. gr. 1.842 and 96 per cent. H\_{2}SO\_{4}
by any of the well-known processes, *e.g*., Kessler,
Webb, Benker, Delplace, &c., and it may be used again
in the manufacture of nitro-glycerine or gun-cotton.

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Two points in the manufacture of nitro-glycerine are
of the greatest importance, *viz*., the purity
of the glycerine used, and the strength and purity
of the acids used in the nitration. With regard
to the first of these, great care should be taken,
and a complete analysis and thorough examination,
including a preliminary experimental nitration, should
always be instituted. As regards the second,
the sulphuric acid should not only be strong (96 per
cent.), but as free from impurities as possible.
With the nitric acid, which is generally made at the
explosive works where it is used, care must be taken
that it is as strong as possible (97 per cent. and
upwards). This can easily be obtained if the plant
designed by Mr Oscar Guttmann[A] is used. Having
worked Mr Guttmann’s plant for some time, I
can testify as to its value and efficiency.

[Footnote A: “The Manufacture of Nitric
Acid,” *Jour. Soc. Chem. Ind.*,
March 1893.]

Another form of nitric acid plant, which promises
to be of considerable service to the manufacturer
of nitric acid for the purpose of nitrating, is the
invention of the late Mr Manning Prentice, of Stowmarket.
Through the kindness of Mr Prentice, I visited his
works to see the plant in operation. It consists
of a still, divided into compartments or chambers
in such a manner that the fluid may pass continuously
from one to the other. The nitric acid being
continuously separated by distillation, the contents
of each division vary—­the first containing
the full proportion of nitric acid, and each succeeding
one less of the nitric acid, until from the overflow
of the last one the bisulphate of soda flows away
without any nitric acid. The nitrate of soda is
placed in weighed quantities in the hopper, whence
it passes to the feeder. The feeder is a miniature
horizontal pug-mill, which receives the streams of
sulphuric acid and of nitrate, and after thoroughly
mixing them, delivers them into the still, where,
under the influence of heat, they rapidly become a
homogeneous liquid, from which nitric acid continuously
distils.

Mr Prentice says: “I may point out that
while the ordinary process of making nitric acid is
one of fractional distillation by time, mine is fractional
distillation by space.” “Instead of
the operation being always at the same point of space,
but differing by the successive points of time, I
arrange for the differences to take place at different
points of space, and these differences exist at one
and the same points of time.” It is possible
with this plant to produce the full product of nitric
acid of a gravity of 1.500, or to obtain the acid
of varying strengths from the different still-heads.
One of these stills, capable of producing about 4
tons of nitric acid per week, weighs less than 2 tons.
It is claimed that there is by their use a saving
of more than two-thirds in fuel, and four-fifths
in condensing plant. Further particulars and illustrations
will be found in Mr Prentice’s paper (*Journal
of the Society of Chemical Industry*, 1894, p.
323).

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**CHAPTER III.**

*NITRO-CELLULOSE, &c.*
Cellulose Properties—­Discovery of Gun-Cotton—­Properties
of Gun-Cotton—­ Varieties of Soluble and
Insoluble Gun-Cottons—­Manufacture of Gun-Cotton—­
Dipping and Steeping—­Whirling out the Acid—­Washing—­Boiling—­Pulping—­
Compressing—­The Waltham Abbey Process—­Le
Bouchet Process—­Granulation of Gun-Cotton—­Collodion-Cotton—­Manufacture—­Acid
Mixture used—­Cotton used, &c.—­Nitrated
Gun-Cotton—­Tonite—­Dangers in
Manufacture of Gun-Cotton—­ Trench’s
Fire-Extinguishing Compound—­Uses of Collodion-Cotton—­Celluloid—­
Manufacture, &c.—­Nitro-Starch, Nitro-Jute,
and Nitro-Mannite.
 *The Nitro-Celluloses.*—­The substance
known as cellulose forms the groundwork of vegetable
tissues. The cellulose of the woody parts of
plants was at one time supposed to be a distinct body,
and was called lignine, but they are now regarded
as identical. The formula of cellulose is (C\_{6}H\_{10}O\_{6})\_{X},
and it is generally assumed that the molecular formula
must be represented by a multiple of the empirical
formula, C\_{12}H\_{20}O\_{10} being often regarded as
the minimum. The assumption is based on the existence
of a penta-nitrate and the insoluble and colloidal
nature of cellulose. Green (*Zeit. Farb.
Text. Ind.*, 1904, 3, 97) considers these
reasons insufficient, and prefers to employ the single
formula C\_{6}H\_{10}O\_{5}. Cellulose can be extracted
in the pure state, from young and tender portions
of plants by first crushing them, to rupture the cells,
and then extracting with dilute hydrochloric acid,
water, alcohol, and ether in succession, until none
of these solvents remove anything more. Fine
paper or cotton wool yield very nearly pure cellulose
by similar treatment.

Cellulose is a colourless, transparent mass, absolutely
insoluble in water, alcohol, or ether. It is,
however, soluble in a solution of cuprammoniac solution,
prepared from basic carbonate or hydrate of copper
and aqueous ammonia. The specific gravity of cellulose
is 1.25 to 1.45. According to Schulze, its elementary
composition is expressed by the percentage numbers:—­

Carbon 44.0 per cent. 44.2 per cent.
Hydrogen 6.3 " 6.4 "
Oxygen 49.7 " 49.4 "

These numbers represent the composition of the ash
free cellulose. Nearly all forms of cellulose,
however, contain a small proportion of mineral matters,
and the union of these with the organic portion of
the fibre or tissue is of such a nature that the ash
left on ignition preserves the form of the original.
“It is only in the growing point of certain young
shoots that the cellulose tissue is free from mineral
constituents” (Hofmeister).

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Cellulose is a very inert body. Cold concentrated
sulphuric acid causes it to swell up, and finally
dissolves it, forming a viscous solution. Hydrochloric
acid has little or no action, but nitric acid has,
and forms a series of bodies known as nitrates or
nitro-celluloses. Cellulose has some of the properties
of alcohols, among them the power of forming ethereal
salts with acids. When cellulose in any form,
such as cotton, is brought into contact with strong
nitric acid at a low temperature, a nitrate or nitro
product, containing nitryl, or the NO\_{2} group, is
produced. The more or less complete replacement
of the hydroxylic hydrogen by NO\_{2} groups depends
partly on the concentration of the nitric acid used,
partly on the duration of the action. If the most
concentrated nitric and sulphuric acids are employed,
and the action allowed to proceed for some considerable
time, the highest nitrate, known as hexa-nitro-cellulose
or gun-cotton, C\_{12}H\_{14}O\_{4}(O.NO\_{2})\_{6}, will
be formed; but with weaker acids, and a shorter exposure
to their action, the tetra and penta and lower nitrates
will be formed.[A]

[Footnote A: The paper by Prof. Lunge, *Jour.
Amer. Chem. Soc.*, 1901, 23[8], 527-579,
contains valuable information on this subject.]

Besides the nitrate, A. Luck[A] has proposed to use
other esters of cellulose, such as the acetate, benzoate,
or butyrate. It is found that cellulose acetate
forms with nitro-glycerine a gelatinous body without
requiring the addition of a solvent. A sporting
powder is proposed composed of 75 parts of cellulose
nitrate (13 per cent. N.) mixed with 13 parts
of cellulose acetate.

[Footnote A: Eng. Pat. 24,662, 22nd November
1898.]

The discovery of gun-cotton is generally attributed
to Schoenbein (1846), but Braconnot (in 1832) had
previously nitrated starch, and six years later Pelouse
prepared nitro-cotton and various other nitro bodies,
and Dumas nitrated paper, but Schoenbein was apparently
the first chemist to use a mixture of strong nitric
and sulphuric acids. Many chemists, such as Piobert
in France, Morin in Russia, and Abel in England, studied
the subject; but it was in Austria, under the auspices
of Baron Von Lenk, that the greatest progress was
made. Lenk used cotton in the form of yarn, made
up into hanks, which he first washed in a solution
of potash, and then with water, and after drying dipped
them in the acids. The acid mixture used consisted
of 3 parts by weight of sulphuric to 1 part of nitric
acid, and were prepared some time before use.
The cotton was dipped one skein at a time, stirred
for a few minutes, pressed out, steeped, and excess
of acid removed by washing with water, then with dilute
potash, and finally with water. Von Lenk’s
process was used in England at Faversham (Messrs Hall’s
Works), but was given up on account of an explosion
(1847).

Sir Frederick Abel, working at Stowmarket and Waltham
Abbey, introduced several very important improvements
into the process, the chief among these being pulping.
Having traced the cause of its instability to the
presence of substances caused by the action of the
nitric acid on the resinous or fatty substances contained
in the cotton fibre, he succeeded in eliminating them,
by boiling the nitro-cotton in water, and by a thorough
washing, after pulping the cotton in poachers.

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Although gun-cottons are generally spoken of as nitro-celluloses,
they are more correctly described as cellulose nitrates,
for unlike nitro bodies of other series, they do not
yield, or have not yet done so, amido bodies, on reduction
with nascent hydrogen.[A] The equation of the formation
of gun-cotton is as follows:—­

2(C\_{6}H\_{10}O\_{5}) + 6HNO\_{3} = C\_{12}H\_{14}O\_{4}(NO\_{3})\_{6}
+ 6OH\_{2}.
 Cellulose. Nitric
Acid. Gun-Cotton. Water.

The sulphuric acid used does not take part in the
reaction, but its presence is absolutely essential
to combine with the water set free, and thus to prevent
the weakening of the nitric acid. The acid mixture
used at Waltham Abbey consists of 3 parts by weight
of sulphuric acid of 1.84 specific gravity, and 1
part of nitric acid of 1.52 specific gravity.
The same mixture is also used at Stowmarket (the New
Explosive Company’s Works). The use of
weaker acids results in the formation of collodion-cotton
and the lower nitrates generally.

[Footnote A: “Cellulose,” by Cross
and Bevan, ed. by W.R. Hodgkinson, p. 9.]

The nitrate which goes under the name of gun-cotton
is generally supposed to be the hexa-nitrate, and
to contain 14.14 per cent. of nitrogen; but a higher
percentage than 13.7 has not been obtained from any
sample. It is almost impossible (at any rate
upon the manufacturing scale) to make pure hexa-nitro-cellulose
or gun-cotton; it is certain to contain several per
cents. of the soluble forms, *i.e*., lower nitrates.
It often contains as much as 15 or 16 per cent., and
only from 13.07[A] to 13.6 per cent. of nitrogen.

[Footnote A: Mr J.J. Sayers, in evidence
before the court in the “Cordite Case,”
says he found 15.2 and 16.1 per cent. soluble cotton,
and 13.07 and 13.08 per cent. nitrogen in two samples
of Waltham Abbey gun-cotton.]

A whole series of nitrates of cellulose are supposed
to exist, the highest member being the hexa-nitrate,
and the lowest the mono-nitrate. Gun-cotton was
at one time regarded as the tri-nitrate, and collodion-cotton
as the di-nitrate and mono-nitrate, their respective
formula being given as follows:—­

Mono-nitro-cellulose C\_{6}H\_{9}(NO\_{2})O\_{5} = 6.763 per cent. nitrogen.
Di-nitro-cellulose C\_{6}H\_{8}(NO\_{2})\_{2}O\_{5} = 11.11 " "
Tri-nitro-cellulose C\_{6}H\_{7}(NO\_{2})\_{3}O\_{5} = 14.14 " "

But gun-cotton is now regarded as the hexa-nitrate,
and collodion-cotton as a mixture of all the other
nitrates. In fact, chemists are now more inclined
to divide nitro-cellulose into the soluble and insoluble
forms, the reason being that it is quite easy to make
a nitro-cellulose entirely soluble in a mixture of
ether-alcohol, and yet containing as high a percentage
of nitrogen as 12.6; whereas the di-nitrate[A] should
theoretically only contain 11.11 per cent. On
the other hand, it is not possible to make gun-cotton
with a higher percentage of nitrogen than about 13.7,
even when it does not contain any nitro-cotton that
is soluble in ether-alcohol.[B] The fact is that it
is not at present possible to make a nitro-cellulose
which shall be either entirely soluble or entirely
insoluble, or which will contain the theoretical content
of nitrogen to suit any of the above formulae for
the cellulose nitrates. Prof. G. Lunge gives
the following list of nitration products of cellulose:—­

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[Footnote A: The penta-nitrate C\_{12}H\_{15}O\_{5}(NO\_{3})\_{5}
= 12.75 per cent. nitrogen.]

[Footnote B: In the Cordite Trial (1894) Sir
F.A. Abel said, “Before 1888 there was
a broad distinction between soluble and insoluble nitro-cellulose,
collodion-cotton being soluble (in ether-alcohol) and
gun-cotton insoluble.” Sir H.E. Roscoe,
“That he had been unable to make a nitro-cotton
with a higher nitrogen content than 13.7.”
And Professor G. Lunge said, “Gun-cotton always
contained soluble cotton, and *vice versa*.”
These opinions were also generally confirmed by Sir
E. Frankland, Sir W. Crookes, Dr Armstrong, and others.]

Dodeca-nitro-cellulose C\_{24}H\_{28}O\_{20}(NO\_{2})\_{12}
= 14.16 per cent.
 nitrogen. (= old tri-nitro-cellulose)
Endeca-nitro-cellulose C\_{24}H\_{29}O\_{20}(NO\_{2})\_{11}
= 13.50 per cent.
 nitrogen.
Deca-nitro-cellulose C\_{24}H\_{30}O\_{20}(NO\_{2})\_{10}
= 12.78 per cent.
 nitrogen.
Ennea-nitro-cellulose C\_{24}H\_{31}O\_{20}(NO\_{2})\_{9}
 = 11.98 per cent.
 nitrogen.
Octo-nitro-cellulose C\_{24}H\_{32}O\_{20}(NO\_{2})\_{8}
= 11.13 per cent.
 nitrogen. (= old di-nitro-cellulose)
Hepta-nitro-cellulose C\_{24}H\_{33}O\_{20}(NO\_{2})\_{7}
 = 10.19 per cent.
 nitrogen.
Hexa-nitro-cellulose C\_{24}H\_{34}O\_{20}(NO\_{2})\_{6}
 = 9.17 per cent.
 nitrogen.
Penta-nitro-cellulose C\_{24}H\_{35}O\_{20}(NO\_{2})\_{5}
 = 8.04 per cent.
 nitrogen.
Tetra-nitro-cellulose C\_{24}H\_{36}O\_{20}(NO\_{2})\_{4}
 = 6.77 per cent.
 nitrogen. (= old mono-nitro-cellulose)

It is not unlikely that a long series of nitrates
exists. It is at any rate certain that whatever
strength of acids may be used, and whatever temperature
or other conditions may be present during the nitration,
that the product formed always consists of a mixture
of the soluble and insoluble nitro-cellulose.

Theoretically 100 parts of cotton by weight should
produce 218.4 parts of gun-cotton, but in practice
the yield is a good deal less, both in the case of
gun-cotton or collodion-cotton. In speaking of
soluble and insoluble nitro-cellulose, it is their
behaviour, when treated with a solution consisting
of 2 parts ether and 1 of alcohol, that is referred
to. There is, however, another very important
difference, and that is their different solubility
in nitro-glycerine. The lower nitrates or soluble
form is soluble in nitro-glycerine under the influence
of heat, a temperature of about 50 deg. C. being
required. At lower temperatures the dissolution
is very imperfect indeed; and after the materials have
been left in contact for days, the threads of the
cotton can still be distinguished. The insoluble
form or gun-cotton is entirely *insoluble* in
nitro-glycerine. It can, however, be made to dissolve[A]
by the aid of acetone or acetic ether. Both or
rather all the forms of nitro-cellulose can be dissolved
in acetone or acetic ether. They also dissolve
in concentrated sulphuric acid, and the penta-nitrate
in nitric acid at about 80 deg. or 90 deg. C.

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[Footnote A: Or rather to form a transparent
jelly.]

The penta-nitrate may be obtained in a pure state
by the following process, devised by Eder:—­The
gun-cotton is dissolved in concentrated nitric acid
at 90 deg. C., and reprecipitated by the addition
of concentrated sulphuric acid. After cooling
to 0 deg. C., and mixing with a larger volume
of water, the precipitated nitrate is washed with
water, then with alcohol, dissolved in ether-alcohol,
and again precipitated with water, when it is obtained
pure. This nitrate is soluble in ether-alcohol,
and slightly in acetic acid, easily in acetone, acetic
ether, and methyl-alcohol, insoluble in alcohol.
Strong potash (KOH) solution converts into the di-nitrate
C\_{12}H\_{18}O\_{8}(NO\_{3})\_{2}. The hexa-nitrate
is not soluble in acetic acid or methyl-alcohol.

The lower nitrates known as the tetra- and tri-nitrates
are formed together when cellulose is treated with
a mixture of weak acids, and allowed to remain in
contact with them for a very short time (twenty minutes).
They cannot be separated from one another, as they
all dissolve equally in ether-alcohol, acetic ether,
acetic acid, methyl-alcohol, acetone, amyl acetate,
&c.

As far as the manufacture of explosive bodies is concerned,
the two forms of nitro-cellulose used and manufactured
are gun-cotton or the hexa-nitrate (once regarded
as tri-nitro-cellulose), which is also known as insoluble
gun-cotton, and the soluble form of gun-cotton, which
is also known as collodion, and consists of a mixture
of several of the lower nitrates. It is probable
that it chiefly consists, however, of the next highest
nitrate to gun-cotton, as the theoretical percentage
of nitrogen for this body,. the penta-nitrate, is
12.75 per cent., and analyses of commercial collodion-cotton,
entirely soluble in ether-alcohol, often give as high
a percentage as 12.6.

We shall only describe the manufacture of the two
forms known as soluble and insoluble, and shall refer
to them under their better known names of gun-cotton
and collodion-cotton. The following would, however,
be the formulae[A] and percentage of nitrogen of the
complete series:—­

Hexa-nitro-cellulose C\_{12}H\_{14}O\_{4}(NO\_{3})\_{6}
14.14 per cent.
 nitrogen.
Penta-nitro-cellulose C\_{12}H\_{15}O\_{5}(NO\_{3})\_{5}
12.75 per cent.
 nitrogen.
Tetra-nitro-cellulose C\_{12}H\_{16}O\_{6}(NO\_{3})\_{4}
11.11 per cent.
 nitrogen.
Tri-nitro-cellulose C\_{12}H\_{17}O\_{7}(NO\_{3})\_{3}
 9.13 per cent.
 nitrogen.
Di-nitro-cellulose C\_{12}H\_{18}O\_{8}(NO\_{3})\_{2}
 7.65 per cent.
 nitrogen.
Mono-nitrocellulose C\_{12}H\_{19}O\_{9}(NO\_{3})
 3.80 per cent.
 nitrogen.

[Footnote A: Berthelot takes C\_{24}H\_{40}O\_{20}
as the formula of cellulose; and M. Vieille regards
the highest nitrate as (C\_{24}H\_{18}(NO\_{3}H)\_{11}O\_{9}).
*Compt. Rend.*, 1882, p. 132.]

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*Properties of Gun-Cotton.*—­The absolute
density of gun-cotton is 1.5. When in lumps its
apparent density is 0.1; if twisted into thread, 0.25;
when subjected, in the form of pulp, to hydraulic pressure,
1.0 to 1.4. Gun-cotton preserves the appearance
of the cotton from which it is made. It is, however,
harsher to the touch; it is only slightly hygroscopic
(dry gun-cotton absorbs 2 per cent. of moisture from
the air). It possesses the property of becoming
electrified by friction. It is soluble in acetic
ether, amyl acetate, and acetone, insoluble in water,
alcohol, ether, ether-alcohol, methyl-alcohol, &c.
It is very explosive, and is ignited by contact with
an ignited body, or by shock, or when it is raised
to a temperature of 172 deg. C. It burns with
a yellowish flame, almost without smoke, and leaves
little or no residue. The volume of the gases
formed is large, and consists of carbonic acid, carbonic
oxide, nitrogen, and water gas. Compressed gun-cotton
when ignited often explodes when previously heated
to 100 deg. C.

Gun-cotton kept at 80 deg. to 100 deg. C. decomposes
slowly, and sunlight causes it to undergo a slow decomposition.
It can, however, be preserved for years without undergoing
any alteration. It is very susceptible to explosions
by influence. For instance, a torpedo, even placed
at a long distance, may explode a line of torpedoes
charged with gun-cotton. The velocity of the
propagation of the explosion in metallic tubes filled
with pulverised gun-cotton has been found to be from
5,000 to 6,000 mms. per second in tin tubes, and 4,000
in leaden tubes (Sebert).

Gun-cotton loosely exposed in the open air burns eight
times as quickly as powder (Piobert). A thin
disc of gun-cotton may be fired into from a rifle
without explosion; but if the thickness of the disc
be increased, an explosion may occur. The effect
of gun-cotton in mines is very nearly the same as
that of dynamite for equal weights. It requires,
however, a stronger detonator, and it gives rise to
a larger quantity of carbonic oxide gas. Gun-cotton
should be neutral to litmus, and should stand the
Government heat test—­temperature of 150
deg. F. for fifteen minutes (see page 249).
In the French Navy gun-cotton is submitted to a heat
test of 65 deg. C. (= 149 deg. F.) for eleven
minutes. It should contain as small a percentage
of soluble nitro-cotton and of non-nitrated cotton
as possible.

The products of perfectly detonated gun-cotton may
be expressed by the following equation:—­

2C\_{12}H\_{14}O\_{4}(NO\_{3})\_{6} = 18CO + 6CO\_{2} +
14H\_{2}O + 12N.

It does not therefore contain sufficient oxygen for
the complete combustion of its carbon. It is
for this reason that when used for mining purposes
a nitrate is generally added to supply this defect
(as, for instance, in tonite). It tends also
to prevent the evolution of the poisonous gas, carbonic
oxide. The success of the various gelatine explosives
is due to this fact, *viz*., that the nitro-glycerine
has an excess of oxygen, and the nitro-cotton too
little, and thus the two explosives help one another.

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In practice the gases resulting from the explosion
of gun-cotton are—­ Carbonic oxide, 28.55;
carbonic acid, 19.11; marsh gas (CH\_{4}), 11.17; nitric
oxide, 8.83; nitrogen, 8.56; water vapour, 21.93 per
cent. The late Mr E.O. Brown, of Woolwich
Arsenal, discovered that perfectly wet and uninflammable
compressed gun-cotton could be easily detonated by
the detonation of a priming charge of the dry material
in contact with it. This rendered the use of
gun-cotton very much safer for use as a military or
mining explosive.

As a mining explosive, however, gun-cotton is now
chiefly used under the form of tonite, which is a
mixture of half gun-cotton and half barium nitrate.
This material is sometimes spoken of as “nitrated
gun-cotton.” The weight of gun-cotton required
to produce an equal effect either in heavy ordnance
or in small arms is to the weight of gunpowder in the
proportion of 1 to 3, *i.e*., an equal weight of
gun-cotton would produce three times the effect of
gunpowder. Its rapidity of combustion, however,
requires to be modified for use in firearms. Hence
the lower nitrates are generally used, or such compounds
as nitro-lignose, nitrated wood, &c., are used.

The initial pressure produced by the explosion of
gun-cotton is very large, equal to 18,135 atmospheres,
and 8,740 kilogrammes per square centimetre for 1
kilo., the heat liberated being 1,075 calories (water
liquid), or 997.7 cals. (water gaseous), but the quantity
of heat liberated changes with the equation of decomposition.
According to Berthelot,[A] the heat of formation of
collodion-cotton is 696 cals. for 1,053 grms., or
661 cals. for 1 kilo. The heat liberated in the
total combustion of gun-cotton by free oxygen at constant
pressure is 2,633 cals. for 1,143 grms., or for 1
kilo. gun-cotton 2,302 cals. (water liquid), or 2,177
cals. (water gaseous). The heat of decomposition
of gun-cotton in a closed vessel, found by experiment
at a low density of charge (0.023), amounts to 1,071
cals. for 1 kilo. of the substance, dry and free from
ash. To obtain the maximum effect of gun-cotton
it must be used in a compressed state, for the initial
pressures are thereby increased. Wet gun-cotton
s much less sensitive to shock than dry. Paraffin
also reduces its liability to explode, so also does
camphor.

[Footnote A: “Explosives and their Power,”
trans. by Hake and M’Nab.]

The substance known as celluloid, a variety of nitro-cellulose
nearly corresponding to the formula C\_{24}H\_{24}(NO\_{3}H)\_{8}O\_{12},
to which camphor and various inert substances are
added, so as to render it non-sensitive to shock,
may be worked with tools, and turned in the lathe
in the same manner as ivory, instead of which material
celluloid is now largely used for such articles as
knife handles, combs, &c. Celluloid is very plastic
when heated towards 150 deg. C., and tends to
become very sensitive to shock, and in large quantities
might become explosive during a fire, owing to the

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general heating of the mass, and the consequent evaporation
of the camphor. When kept in the air bath at 135
deg. C., celluloid decomposes quickly. In
an experiment (made by M. Berthelot) in a closed vessel
at 135 deg. C., and the density of the charge
being 0.4, it ended in exploding, developing a pressure
of 3,000 kilos. A large package of celluloid
combs also exploded in the guard’s van on one
of the German railways a few years ago. Although
it is not an explosive under ordinary circumstances,
or even with a powerful detonator, considerable care
should be exercised in its manufacture.
 *The Manufacture of Gun-Cotton.*—­The
method used for the manufacture of gun-cotton is that
of Abel (Spec. No. 1102, 20. 4. 65). It was
worked out chiefly at Stowmarket[A] and Waltham Abbey,[B]
but has in the course of time undergone several alterations.
These modifications have taken place, however, chiefly
upon the Continent, and relate more to the apparatus
and machinery used than to any alteration in the process
itself. The form of cellulose used is cotton-waste,[C]
which consists of the clippings and waste material
from cotton mills. After it has been cleaned and
purified from grease, oil, and other fatty substances
by treatment with alkaline solutions, it is carefully
picked over, and every piece of coloured cotton rag
or string carefully removed. The next operation
to which it is submitted has for its object the opening
up of the material. For this purpose it is put
through a carding machine, and afterwards through a
cutting machine, whereby it is reduced to a state suitable
for its subsequent treatment with acids, that is,
it has been cut into short lengths, and the fibres
opened up and separated from one another.

[Footnote A: The New Explosive Co. Works.]

[Footnote B: Royal Gunpowder Factory.]

[Footnote C: Costs from L10 to L25 a ton.
In his description of the “Preparation of Cotton-waste
for the Manufacture of Smokeless Powder,” A.
Hertzog states that the German military authorities
require a cotton which when thrown into water sinks
in two minutes; when nitrated, does not disintegrate;
when treated with ether, yields only 0.9 per cent.
of fat; and containing only traces of chlorine, lime,
magnesia, iron, sulphuric acid, and phosphoric acid.
If the cotton is very greasy, it must be first boiled
with soda-lye under pressure, washed, bleached with
chlorine, washed, treated with sulphuric acid or HCl,
again washed, centrifugated, and dried; if very greasy
indeed a preliminary treatment with lime-water is
desirable. See also “Inspection of Cotton-Waste
for Use in the Manufacture of Gun-cotton,” by
C.E. Munro, *Jour. Am. Chem.
Soc.*, 1895, 17, 783.]

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*Drying the Cotton.*—­This operation
is performed in either of two ways. The cotton
may either be placed upon shelves in a drying house,
through which a current of hot air circulates, or
dried in steam-jacketed cylinders. It is very
essential that the cotton should be as dry as possible
before dipping in the acids, especially if a wholly
“insoluble” nitro-cellulose is to be obtained.
After drying it should not contain more than 0.5 per
cent. of moisture, and less than this if possible.
The more general method of drying the cotton is in
steam-jacketed tubes, *i.e*., double cylinders
of iron, some 5 feet long and 1-1/2 foot wide.
The cotton is placed in the central chamber (Fig.
10), while steam is made to circulate in the surrounding
jacket, and keeps the whole cylinder at a high temperature
(steam pipes may be coiled round the outside of an
iron tube, and will answer equally well). By
means of a pipe which communicates with a compressed
air reservoir, a current of air enters at the bottom,
and finds its way up through the cotton, and helps
to remove the moisture that it contains. The
raw cotton generally contains about 10 per cent. of
moisture and should be dried until it contains only
1/2 per cent. or less. For this it will generally
have to remain in the drying cylinder for about five
hours. At the end of that time a sample should
be taken from the *top* of the cylinder, and
dried in the water oven (100 deg. C.[A]) for an
hour to an hour and a half, and re-weighed, and the
moisture then remaining in it calculated.

[Footnote A: It is dried at 180 deg. C.
at Waltham Abbey, in a specially constructed drying
chamber.]

[Illustration: FIG. 10.—­COTTON DRIER.]

It is very convenient to have a large copper water
oven, containing a lot of small separate compartments,
large enough to hold about a handful of the cotton,
and each compartment numbered, and corresponding to
one of the drying cylinders. The whole apparatus
should be fixed against the wall of the laboratory,
and may be heated by bringing a small steam pipe from
the boiler-house. It is useful to have a series
of copper trays, about 3 inches by 6 inches, numbered
to correspond to the divisions in the steam oven,
and exactly fitting them. These trays can then
be taken by a boy to the drying cylinders, and a handful
of the cotton from each placed in them, and afterwards
brought to the laboratory and weighed (a boy can do
this very well), placed in their respective divisions
of the oven, and left for one to one and a half hours,
and re-weighed.

When the cotton is found to be dry the bottom of the
drying cylinder is removed, and the cotton pushed
out from the top by means of a piece of flat wood
fixed on a broom-handle. It is then packed away
in galvanised-iron air-tight cases, and is ready
for the next operation. At some works the cotton
is dried upon shelves in a drying house through which
hot air circulates, the shelves being of canvas or
of brass wire netting. The hot air must pass
under the shelves and through the cotton, or the process
will be a very slow one.

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*Dipping and Steeping.*—­The dry cotton
has now to be nitrated. This is done by dipping
it into a mixture of nitric and sulphuric acids.
The acids used must be strong, that is, the nitric
acid must be at least of a gravity of 1.53 to 1.52,
and should contain as little nitric oxide as possible.
The sulphuric acid must have a specific gravity of
1.84 at 15 deg. C., and contain about 97 per
cent. of the mono-hydrate (H\_{2}SO\_{4}). In fact,
the strongest acids obtainable should be used when
the product required is gun-cotton, *i.e*., the
highest nitrate.

The sulphuric acid takes no part in the chemical reaction
involved, but is necessary in order to combine with
the water that is liberated in the reaction, and thus
to maintain the strength of the nitric acid. The
reaction which takes place is the following:—­

2(C\_{6}H\_{10}O\_{5}) + 6HNO\_{3} = C\_{12}H\_{14}(NO\_{3})\_{6}
+ 6 H\_{2}O.
 324
 378 = 594 108.
 Cellulose.
 Gun-Cotton.

Theoretically,[A] therefore, 1 part of cellulose should
form 1.8 part of gun-cotton. Practically, however,
this is never obtained, and 1.6 lb. from 1 lb. of
cellulose is very good working. The mixture of
acids used is generally 1 to 3, or 25 per cent. nitric
acid to 75 per cent. sulphuric acid.

[Footnote A: (594 x 1)/324= 1.83.]

[Illustration: FIG. 11.—­TANK FOR DIPPING
COTTON.]

[Illustration: FIG. 12.—­THE COOLING
PITS.]

The dipping is done in cast-iron tanks (Fig. 11),
a series of which is arranged in a row, and cooled
by a stream of cold water flowing round them.
The tanks hold about 12 gallons, and the cotton is
dipped in portions of 1 lb. at a time. It is
thrown into the acids, and the workman moves it about
for about three minutes with an iron rabble. At
the end of that time he lifts it up on to an iron
grating, just above the acids, fixed at the back of
the tank, where by means of a movable lever he gently
squeezes it, until it contains about ten times its
weight of acids (the 1 lb. weighs 10 lbs.). It
is then transferred to earthenware pots to steep.

[Illustration: FIG. 13.—­COTTON STEEPING
POT.]
 *Steeping.*—­The nitrated cotton, when
withdrawn from the dipping tanks, and still containing
an excess of acids, is put into earthenware pots of
the shape shown in Figs. 12 and 13. The lid is
put on, and the pots placed in rows in large cooling
pits, about a foot deep, through which a stream of
water is constantly flowing. These pits form the
floor of the steeping house. The cotton remains
in these pots for a period of forty-eight hours, and
must be kept cool. Between 18 deg. and 19 deg.
C. is the highest temperature desirable, but the cooler
the pots are kept the better. At the end of forty-eight
hours the chemical reaction is complete, and the cotton
is or should be wholly converted into nitro-cellulose;
that is, there should be no unnitrated cotton.

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[Illustration: FIG. 14.—­HYDRO-EXTRACTOR.]
 *Whirling Out the Acid.*—­The next
operation is to remove the excess of acid. This
is done by placing the contents of two or three or
more pots into a centrifugal hydro-extractor (Fig.
14), making 1,000 to 1,500 revolutions per minute.
The hydro-extractor consists of a machine with both
an inner cylinder and an outer one, both revolving
in concert and driving outwardly the liquid to the
chamber, from which it runs away by a discharge pipe.
The wet cotton is placed around the inner cone.
The cotton, when dry, is removed, and at once thrown
into a large tank of water, and the waste acids are
collected in a tank.[A]

[Footnote A: Care must be taken in hot weather
that the gun-cotton does not fire, as it does sometimes,
directly the workman goes to remove it after the machine
is stopped. It occurs more often in damp weather.
Dr Schuepphaus, of Brooklyn, U.S.A., proposes to treat
the waste acids from the nitration of cellulose by
adding to them sulphuric anhydride and nitric acid.
The sulphuric anhydride added converts the water liberated
from the cellulose into sulphuric acid.]
 *Washing.*—­The cotton has now to be
carefully washed. This is done in a large wooden
tank filled with water. If, however, a river or
canal runs through the works, a series of wooden tanks,
the sides and bottoms of which are pierced with holes,
so as to allow of the free circulation of water, should
be sunk into a wooden platform that overhangs the surface
of the river in such a way that the tanks are immersed
in the water, and of course always full. During
the time that the cotton is in the water a workman
turns it over constantly with a wooden paddle.
A stream of water, in the form of a cascade, should
be allowed to fall into these tanks. The cotton
may then be thrown on to this stream of water, which,
falling some height, at once carries the cotton beneath
the surface of the water. This proceeding is
necessary because the cotton still retains a large
excess of strong acids, and when mixed with water
gives rise to considerable heat, especially if mixed
slowly with water. After the cotton has been well
washed, it is again wrung out in a centrifugal machine,
and afterwards allowed to steep in water for some
time.

[Illustration: FIG. 15\_a\_.—­THE BEATER
FOR GUN-COTTON.]
 *Boiling.*—­The washed cotton is put
into large iron boilers with plenty of water, and
boiled for some time at 100 deg. C. In some works
lead-lined tanks are used, into which a steam pipe
is led. The soluble impurities of unstable character,
to which Sir F.A. Abel traced the liability of
gun-cotton to instability, are thereby removed.
These impurities consist of the products formed by
the action of nitric acid on the fatty and resinous
substances contained in the cotton fibres. The
water in the tanks should be every now and again renewed,
and after the first few boilings the water should
be tested with litmus paper until they are no longer
found to be acid.

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[Illustration: FIG. 15\_b\_.—­WHEEL OF
BEATER.]
 *Pulping.*—­The idea of pulping is
also due to Abel. By its means a very much more
uniform material is obtained. The process is carried
out in an apparatus known as a “Beater”
or “Hollander” (Fig. 15, *a, b*).
It consists of a kind of wooden tank some 2 or 3 feet
deep of an oblong shape, in which a wheel carrying
a series of knives is made to revolve, the floor of
the tank being sloped up so as to almost touch the
revolving wheels. This part of the floor, known
as the “craw,” is a solid piece of oak,
and a box of knives is fixed into it, against which
the knives in the revolving wheel are pressed.
The beater is divided into two parts—­the
working side, in which the cotton is cut and torn between
the knife edges in the revolving cylinder and those
in the box; and the running side, into which the cotton
passes after passing under the cylinder. The wheel
is generally boxed in to prevent the cotton from being
thrown out during its revolution. The cotton
is thus in constant motion, continually travelling
round, and passing between the knives in the revolving
cylinder and those in the box fixed in the wooden
block beneath it. The beater is kept full of
water, and the cotton is gradually reduced to a condition
of pulp. The wheel revolves at the rate of 100
to 150 times a minute.

[Illustration: FIG. 16\_a\_.—­POACHER
FOR WASHING GUN-COTTON.]

[Illustration: FIG. 16\_b\_.—­PLAN OF
THE POACHER.]

[Illustration: FIG. 16\_c\_.—­ANOTHER
FORM OF POACHER.]

When the gun-cotton is judged to be sufficiently fine,
the contents of the beater are run into another very
similar piece of machinery, known as the “poacher”
(Fig. 16, *a, b, c*), in which the gun-cotton
is continuously agitated together with a large quantity
of water, which can be easily run off and replaced
as often as required. When the material is first
run into the poacher from the beater, the water with
which it is then mixed is first run away and clean
water added. The paddle wheel is then set in
motion, and at intervals fresh water is added.
There is a strainer at the bottom of the poacher which
enables the water to be drawn off without disturbing
the cotton pulp. After the gun-cotton has been
in the poacher for some time, a sample should be taken
by holding a rather large mesh sieve in the current
for a minute or so. The pulp will thus partly
pass through and partly be caught upon the sieve,
and an average sample will be thus obtained.
The sample is squeezed out by hand, bottled, and taken
to the laboratory to be tested by the heat test for
purity. It first, however, requires to be dried.
This is best done by placing the sample between coarse
filter paper, and then putting it under a hand-screw
press, where it can be subjected to a tolerably severe
pressure for about three minutes. It is then
rubbed up very finely with the hands, and placed upon
a paper tray, about 6 inches by 4-1/2 inches, which

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is then placed inside a water oven upon a shelf of
coarse wire gauze, the temperature of the oven being
kept as near as possible to 120 deg. F. (49 deg.
C.), the gauze shelves in the oven being kept about
3 inches apart. The sample is allowed to remain
at rest for fifteen minutes in the oven, the door of
which is left wide open. After the lapse of fifteen
minutes the tray is removed and exposed to the air
of the laboratory (away from acid fumes) for two hours,
the sample being at some point within that time rubbed
upon the tray with the hand, in order to reduce it
to a fine and uniform state of division. Twenty
grains (1.296 grm.) are used for the test. (See Heat
Test, page 249.)

If the gun-cotton sample removed from the poacher
stands the heat test satisfactorily, the machine is
stopped, and the water drained off. The cotton
is allowed some little time to drain, and is then dug
out by means of wooden spades, and is then ready for
pressing. The poachers hold about 2,000 lbs.
of material, and as this represents the products of
many hundred distinct nitrating operations, a very
uniform mixture is obtained. Two per cent. of
carbonate of soda is sometimes added, but it is not
really necessary if the cotton has been properly washed.
 *Compressing Gun-Cotton.*—­The gun-cotton,
in the state in which it is removed from the poacher,
contains from 28 to 30 per cent. of water. In
order to remove this, the cotton has to be compressed
by hydraulic power. The dry compressed gun-cotton
is packed in boxes containing 2,500 lbs. of dry material.
In order to ascertain how much of the wet cotton must
be put into the press, it is necessary to determine
the percentage of water. This may be done by
drying 2,000 grains upon a paper tray (previously dried
at 100 deg. C.) in the water oven at 100 deg.
C. for three hours, and re-weighing and calculating
the percentage of water. It is then easy to calculate
how much of the wet gun-cotton must be placed in the
hopper of the press in order to obtain a block of
compressed cotton of the required weight. Various
forms of presses are used, and gun-cotton is sent out
either as solid blocks, compressed discs, or in the
form of an almost dry powder, in zinc-lined, air-tight
cases. The discs are often soaked in water after
compression until they have absorbed 25 per cent. of
moisture.

[Illustration: FIG. 17.—­OLD METHOD.
100 PIECES.]

[Illustration: FIG. 18.—­NEW METHOD.
ONE SOLID BLOCK.]

At the New Explosives Company’s Stowmarket Works
large solid blocks of gun-cotton are pressed up under
a new process, whereby blocks of gun-cotton, for
use in submarine mines or in torpedo warheads, are
produced. Large charges of compressed gun-cotton
have hitherto been built up from a number of suitably
shaped charges of small dimensions (Fig. 17), as it
has been impossible to compress large charges in a
proper manner. The formation of large-sized blocks
of gun-cotton was the invention of Mr A. Hollings.

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Prior to the introduction of this method, 8 or 9 lbs.
had been the limit of weight for a block. This
process has been perfected at the Stowmarket factory,
where blocks varying from the armour-piercing shell
charge of a few ounces up to blocks of compressed gun-cotton
mechanically true, weighing 4 to 5 cwts. for torpedoes
or submarine mines, are now produced. At the
same time the new process ensures a uniform density
throughout the block, and permits of any required density,
from 1.4 downwards, being attained; it is also possible
exactly to regulate the percentage of moisture, and
to ensure its uniform distribution. The maximum
percentage of moisture depends, of course, upon the
density. By the methods of compression gun-cotton
blocks hitherto employed, blocks of a greater thickness
than 2 inches, or of a greater weight than 9 lbs.,
could not be made, but with the new process blocks
of any shape, size, thickness, or weight that is likely
to be required can be made readily and safely.
The advantages which are claimed for the process may
be enumerated as follows:—­(1.) There is
no space wasted, as in the case with built-up charges,
through slightly imperfect contact between the individual
blocks, and thus either a heavier charge—­i.e.,
about 15 per cent. more gun-cotton—­can
be got into the same space, or less space will be occupied
by a charge of a given weight. (2.) The metallic cases
for solid charges may be much lighter than for those
built-up, since with the former their function is
merely to prevent the loss of moisture from wet gun-cotton,
or to prevent the absorption of moisture by dry gun-cotton.
They can thus be made lighter, as the solid charge
inside will prevent deformation during transport.
With built-up charges the case must be strong enough
to prevent damage, either to itself or to the charge
it contains. For many uses a metal case, however
light, may be discarded, and one of a thin waterproof
material substituted. (3.) The uniform density of charges
made by this process is very favourable to the complete
and effective detonation of the entire mass, and to
the presence of the uniform amount of moisture in
every part of the charge. (4.) Any required density,
from the maximum downwards, may be obtained with ease,
and any required amount of moisture left in the charge.
These points are of great importance in cases where,
like torpedo charges, it is essential to have the centre
of gravity of the charge in a predetermined position
both vertically and longitudinally, and the charge
so fixed in its containing case that the centre of
gravity cannot shift. The difficulty of ensuring
this with a large torpedo charge built up from a number
of discs and segments is well known. Even with
plain cylindrical or prismatic charges a marked saving
in the process of production is effected by this new
system. The charges being in one block they are
more easily handled for the usual periodical examination,
and they do not break or chafe at the edges, as in

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the case of discs and cubes in built-up charges.
A general view of the press is given in Fig. 19.
The gun-cotton in a container is placed on a cradle
fixed at an angle to the press. The mould is
swivelled round, and the charge pushed into it with
a rammer, and it is then swivelled back into position.
The mould is made up of a number of wedge pieces which
close circumferentially on the enclosed mass, which
is also subjected to end pressure. Holes are provided
for the escape of water.

[Illustration: FIG. 19.—­A 4-CWT.
BLOCK OF GUN-COTTON BEING TAKEN FROM HYDRAULIC PRESS.]
 *The Waltham Abbey Process.*—­At the
Royal Gunpowder Factory, Waltham Abbey, the manufacture
of gun-cotton has been carried out for many years.
The process used differs but little from that used
at Stowmarket. The cotton used is of a good quality,
it is sorted and picked over to remove foreign matters,
&c., and is then cut up by a kind of guillotine into
2-inch lengths. It is then dried in the following
manner. The cotton is placed upon an endless
band, which conducts it to the stove, or drying closet,
a chamber heated by means of hot air and steam traps
to about 180 deg. F.; it falls upon a second
endless band, placed below the first; it travels back
again the whole length of the stove, and so on until
delivered into a receptacle at the bottom of the farther
end, where it is kept dry until required for use.
The speed at which the cotton travels is 6 feet per
minute, and as the length of the band travelled amounts
to 126 feet, the operation of drying takes twenty-one
minutes. One and a quarter lb. are weighed out
and placed in a tin box; a truck, fitted to receive
a number of these boxes, carries it along a tramway
to a cool room, where it is allowed to cool.
 *Dipping.*—­Mixed acids are used in
the proportion of 1 to 3, specific gravity nitric
acid 1.52, and sulphuric acid 1.84. The dipping
tank is made of cast iron, and holds 220 lbs. of mixed
acids, and is surrounded on three sides by a water
space in order to keep it cool. The mixed acids
are stored in iron tanks behind the dipping tanks,
and are allowed to cool before use. During the
nitration, the temperature of the mixed acids is kept
at 70 deg. F., and the cotton is dipped in quantities
of 1-1/2 lb. at a time. It is put into a tin
shoot at the back of the dipping tank, and raked into
the acids by means of a rabble. It remains in
the acids for five or six minutes, and is then removed
to a grating at the back, pressed and removed.
After each charge of cotton is removed from the tank,
about 14 lbs. of fresh mixed acids are added, to replace
amount removed by charge. The charge now weighs,
with the acids retained by it, 15 lbs.; it is now
placed in the pots, and left to steep for at least
twenty-four hours, the temperature being kept as low
as possible, to prevent the formation of soluble cotton,
and also prevent firing. The proportion of soluble
formed is likely to be higher in hot weather than cold.
The pots must be covered to prevent the absorption
of moisture from the air, or the accidental entrance
of water, which would cause decomposition, and consequent
fuming off, through the heat generated by the action
of the water upon the strong acids.

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The excess of acids is now extracted by means of hydro-extractors,
as at Stowmarket. They are worked at 1,200 revolutions
per minute, and whirled for five minutes (10-1/2 lbs.
of waste acids are removed from each charge dipped).
The charge is then washed in a very similar manner
to that previously described, and again wrung out
in a centrifugal extractor (1,200 revolutions per
minute). The gun-cotton is now boiled by means
of steam in wooden tanks for eight hours; it is then
again wrung out in the extractors for three minutes,
boiled for eight hours more, and again wrung out;
it is then sent to the beater and afterwards to the
poacher. The poachers hold 1,500 gals. each,
or 18 cwt. of cotton. The cotton remains six
hours in the poachers. Before moulding, 500 gals.
of water are run into the poacher, and 500 gals. of
lime water containing 9 lbs. of whiting and 9 gals.
of a caustic soda solution. This mixture is of
such a strength that it is calculated to leave in
the finished gun-cotton from 1 to 2 per cent. of alkaline
matter.

By means of vacuum pressure, the pulp is now drawn
off and up into the stuff chest—­a large
cylindrical iron tank, sufficiently elevated on iron
standards to allow room for the small gauge tanks and
moulding apparatus below. It holds the contents
of one poacher (18 cwt.), and is provided with revolving
arms to keep the pulp stirred up, so that it may be
uniformly suspended in water.

Recently a new process, invented by J.M. and W.T.
Thomson (Eng. Pat. No. 8,278, 1903), has
been introduced at the Waltham Abbey Factory.
The object of this invention is the removal of the
acids of nitration from the nitrated material after
the action has been completed, and without the aid
of moving machinery, such as presses, rollers, centrifugals,
and the like. The invention consists in the manufacture
of nitrated celluloses by removing the acids from
the nitrated cellulose directly by displacement without
the employment of either pressure or vacuum or mechanical
appliances of any kind, and at the same time securing
the minimum dilution of the acids. It was found
that if water was carefully run on to the surface
of the acids in which the nitro-cellulose is immersed,
and the acids be slowly drawn off at the bottom of
the vessel, the water displaces the acid from the
interstices of the nitro-cellulose without any undesirable
rise in temperature, and with very little dilution
of the acids. By this process almost the whole
of the acid is recovered in a condition suitable for
concentration, and the amount of water required for
preliminary washing is very greatly reduced. The
apparatus which is used for the purpose consists of
a cylindrical or rectangular vessel constructed with
a perforated false bottom and a cock at its lowest
point for running off the liquid. Means are also
provided to enable the displacing water to be run
quietly on to the surface of the nitrating acids.[A]

[Footnote A: In a further patent (Eng. Pat.
7,269, 1903, F.L. Natham), J.M. Thomson
and W.T. Thomson propose by use of alcohol to
replace the water, used in washing nitro-cellulose,
and afterward to remove the alcohol by pressing and
centrifuging.]

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The apparatus is shown in Fig. 2O, side elevation,
and in Fig. 21 a plan of the nitrating vessel and
its accessories is given. In Fig. 20 is shown
in sectional elevation one of the trough devices for
enabling liquids to be added to those in the nitrating
vessel without substantial disturbance.

[Illustration: FIG. 20.—­SECTIONAL
ELEVATION OF THOMSON’S APPARATUS, *a*,
Tank; *b*, False Bottom; *c*, Bottom; *c’*,
Ribs; *d*, Draining Outlet; *e*, Grid; *f*,
Troughs, with Aprons *g*; *h*, Pipe, with
Branches *h’*, leading to Troughs, *f*;
*k’*, Outlet Pipe of the Sulphuric Acid
Tank *k*; *l*, Water Supply Pipe; *m*,
Pipe to supply of Nitrating Acids; *o*, Perforations
of Trough *f*; *p*, Cock to remove Acid.]

In carrying out this invention a rectangular lead-lined
or earthenware tank *a* is employed, having a
false bottom *b*, supported by ribs *c’*,
over the real bottom *c*, which slopes down to
a draining outlet pipe *d*, provided with a perforated
grid or plate *e*, adapted to prevent choking
of the outlet. Suitably supported near the top
of the vessel *a* are provided two troughs, *f*
having depending aprons *g*, a pipe *h* has
two branches *h’*, leading to the troughs,
*f*. This pipe *h* is adapted to be
connected by a rubber pipe either to the outlet pipe
*k’* of the sulphuric acid tank *k*
or the water supply pipe *l*. The nitrating
acids are supplied through the pipe *m*.
A charge of mixed nitrating acids is introduced into
the vessel *a* say up to the level *n*, and
the dry cellulose thrown into the acids in small quantities
at a time, being pushed under the surface in the usual
way.

[Illustration: FIG. 21.—­PLAN OF THOMSON’S
APPARATUS, *a*, Tank; *b*, False Bottom;
*c’*, Ribs; *e*, Grid; *f*, Troughs;
*g*, Aprons; *h* and *h’*, Pipes
to Troughs *f*; *k*, Sulphuric Acid Tank;
*m*, Pipe to Nitrating Acids Tank; *o*,
Perforations of Troughs; *p*, Cock to remove
Acid.]

A thin layer, say half an inch, of a suitable liquid,
preferably sulphuric acid, of a gravity not exceeding
that of the waste acid to be produced, is run carefully
on the top of the acids by means of the troughs *f*,
which are perforated as shown at *o*, so that
the sulphuric acid runs down the aprons *g*,
and floats on the nitrating acids. The whole is
then allowed to stand till nitration has been completed.
Water is then supplied to the troughs by way of the
pipes *l*, *h*, and *h’*, and
is allowed to float very gently over the surface of
the sulphuric acid, and when a sufficient layer has
been formed, the cock *p* at the bottom of the
apparatus is opened, and the acid slowly drawn off,
water being supplied to maintain the level constant.
It is found that the rate of displacement of the acids

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is a factor which exerts a considerable influence on
the properties of the resulting nitro-cellulose, and
affords a means of regulating the temperature of displacement.
A rate of displacement which has been found suitable
is about two inches in depth of the vessel per hour
when treating highly nitrated celluloses, but this
rate may, in some cases, be considerably increased.
The flow of water at the top of the apparatus is regulated
so that a constant level is maintained. By this
means the water gradually and entirely displaces the
acids from the interstices of the nitro-cellulose,
the line of separation between the acids and the water
being fairly sharply defined throughout. The flow
of water is continued until that issuing at the bottom
is found to be free from all trace of acid. The
purification of the nitro-cellulose is then proceeded
with as usual, either in the same vessel or another.

In the process above described, the object of the
introduction of a small layer of sulphuric acid is
mainly to prevent the fuming which would otherwise
take place, and is not essential, as it is found it
can be omitted without any deleterious effect.
In order to use the mixed acids in the most economical
manner, the waste acid from a previous operation may
be used for a first nitration of the cellulose; being
afterwards displaced with fresh acids which carry
the nitration to the required degree before they are
in turn displaced by water. The apparatus may
be used merely for the removal of the acid, in which
case the nitration is carried out in other vessels
in the usual way, and the nitro-cellulose removed to
the displacement apparatus where it is just covered
with waste acid, and the displacement then proceeded
with as above described. In some cases the process
is carried out in an ordinary nitrating centrifugal,
using the latter to effect preliminary drying after
acid extraction. This gives a great advantage
over the usual method of working ordinary centrifugal
nitrating apparatus, because the acid being removed
before the centrifugal is run, practically all danger
of firing therein disappears, and a greater proportion
of the waste acid is recovered.

In some cases the acids and water may be supplied
by perforated pipes, lying along the edges of the
nitrating vessel, and these edges may, if desired,
be themselves made inclined, like the sides of the
troughs *f*. In the case of effecting nitration
in centrifugals as above, the displacing sulphuric
acid and water may thus be supplied round the edges
of the machines, or removal troughs such as *f*
may be used. It will be obvious that any inert
liquid of suitable specific gravity may be used instead
of sulphuric acid, as a separation layer.
 *Moulding.*—­By means of the small
measuring tank above referred to, the gun-cotton pulp
is drawn off from the stuff chest, and run into moulds
of the shapes and sizes required. Thence a large
proportion of the water is drawn off by means of tubes
connected with the vacuum engine, the moulds having
bottoms of fine wire gauze, in order to prevent the
pulp from passing through. Hydraulic pressure
of about 34 lbs. on the square inch is then applied,
which has the effect of compressing the pulp into a
state in which it has sufficient consistency to enable
it to be handled with care, and also expels a portion
of the remaining water.

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*Compressing.*—­The moulded gun-cotton
is now taken to the press house, which is situated
at some distance from the rest of the factory.
Here the moulds are subjected to powerful hydraulic
pressure, from 5 to 6 tons per square inch, and is
compressed to one-third of its previous bulk.
The slabs or discs thus formed are kept under pressure
for a short time, not exceeding a minute and a half,
to give the requisite density. It should, when
removed, be compact, and just sink in water, and should
perceptibly yield to the pressure of the fingers.
There are perforations in the press blocks, to allow
of the escape of gases, if formed, by reason of sufficient
heat being generated. The men working the press
are placed under cover, behind strong rope mantlets
having eye tubes which command a view of the press.
 *Packing.*—­The finished slabs and
discs are dipped into a solution of soda and carbolic
acid, and packed in special wood metal-lined cases.
When it is to be sent abroad, the metal lining, which
is made of tinned copper, is soldered down, but both
the outer wooden and inner metal cases are fitted
with air-tight screw-plugs, so that when necessary
water can be added without unfastening the cases.
 *Reworked gun-cotton* does not make such good
discs as new pulped gun-cotton, probably because
the fibrous tenacity of the gun-cotton has been destroyed
by the amount of pressure it has previously undergone,
so that when repulped it resembles fine dust, and
a long time is required to press it into any prescribed
form. It is generally boiled for eight hours to
open up the fibre and remove alkali, then broken up
by hand with wooden mallets, pulped, and then used
with fresh gun-cotton in the proportion of 1 to 5
parts.
 *Manufacture at Le Bouchet.*—­At Le
Bouchet gun-cotton was made thus:—­200 grms.
of cotton were steeped for an hour in 2 litres of a
mixture of 1 volume concentrated nitric and 2 volumes
sulphuric acid. The cotton was then removed and
pressed, whereby 7/10ths of the waste acids was recovered.
After this it was washed for one to one and a half
hours in running water, strongly pressed again; allowed
to lie for twenty-four hours in wood-ash lye; then
well washed in running water; pressed, and finally
dried on a wide linen sheet, through which was forced
air heated to 60 deg. C. The average yield from
100 parts of cotton was 165 parts of gun-cotton.
The strong pressings of the gun-cotton, while still
impregnated with acids, caused subsequent washings
to be difficult and laborious.
 *Granulation of Gun-Cotton.*—­Gun-cotton
is often required in the granulated form for use either
alone or with some form of smokeless powder.
This is done under the patent of Sir Frederick Abel
in the following manner:—­The gun-cotton
from the poacher is placed in a centrifugal machine,
very similar to the hydro-extractors before mentioned,
and used for wringing out the acids. In this machine

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it loses water until it only contains 33 per cent.,
and is at the same time reduced to a more or less
fibrous state. It is then taken to the granulating
room, where it is first passed through sieves or perforations,
which break up the mass into little pieces like shot.
The material is then transferred to a revolving drum
made of wood or stout leather, which is kept constantly
revolving for some time. The material is occasionally
sprinkled with water. The drum in turning, of
course, carries the granules partially round with
it, but the action of gravity causes them to descend
constantly to the lowest point, and thus to roll over
one another continually. The speed of the drum
must not be too rapid. None of the granules must
be carried round by centrifugal force, but it must
be fast enough to carry them some little distance
up the side of the drum. After removal from the
drum the granules are dried upon shelves in the drying
house.

Gun-cotton is also dissolved in acetone or acetic
ether until it has taken the form of a jelly.
It is then rolled into thin sheets, and when dry cut
up into little squares. In the manufacture of
smokeless powders from nitro-cellulose, nitro-lignine,
&c., the various substances are mixed with the gun-cotton
or collodion-cotton before granulating.
 *Collodion-Cotton.*—­In the manufacture
of collodion or soluble cotton the finer qualities
of cotton-waste are used and the acids used in the
dipping tanks are much weaker. The manufacture
of collodion-cotton has become of more importance
than gun-cotton, by reason of its use for the manufacture
of the various forms of gelatine, such as gelatine
dynamite, gelignite, forcite, &c., and also on account
of its extensive use in the manufacture of many of
the smokeless powders. It is also used for the
manufacture of “collodion,” which is a
solution of collodion-cotton in ether-alcohol; for
the preparation of celluloid, and many other purposes.
It is less explosive than gun-cotton, and consists
of the lower nitrates of cellulose. It is soluble
in nitro-glycerine, and in a mixture of 2 parts of
ether and 1 of alcohol; also in acetone, acetic ether,
and other solvents. MM. Menard and Domonte
were the first to prepare a soluble gun-cotton, and
its investigation was carried on by Bechamp, who showed
that its properties and composition were different
to those of gun-cotton.
 *Manufacture.*—­The cotton used is
cotton-waste.[A] It is thought by some that Egyptian
cotton is preferable, and especially long fibre varieties.
The strength of the acids used is, however, of more
importance than the quality of the cotton. The
percentage composition of the acid mixture which gives
the best results is as follows:—­Nitric acid,
23 per cent.; sulphuric acid, 66 per cent.; and water,
11 per cent; and has a specific gravity of 1.712 (about).
It can be made by mixing sulphuric acid of specific
gravity 1.84 with nitric acid of specific gravity 1.368

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in the proportions of 66 per cent. and 34 per cent.
respectively. (The production of the penta-nitro-cellulose
is aimed at if the collodion-cotton is for use as
an explosive.) If the acids are much weaker than this,
or potassium nitrate and sulphuric acid is used, the
lower nitrates will be formed. The product, while
being entirely soluble in ether-alcohol or nitro-glycerine,
will have a low nitrogen content, whereas a material
with as high a nitrogen as 12 or 12.6 is to be aimed
at.

[Footnote A: Raw cotton is often used.]

The cotton should not be allowed to remain in the
dipping tanks for more than five minutes, and the
acid mixture should be kept at a temperature of 28
deg. C. or thereabouts; and the cotton should
be removed after a few minutes, and should not be
pressed out, as in the case of gun-cotton, but at
once transferred to the pots and allowed to steep for
forty-eight hours. (Some prefer twenty-four hours,
but there is more chance in this case of the product
containing non-nitrated cellulose.) When the nitration
is complete, the collodion-cotton is removed from the
pots, and treated in exactly the same manner as described
under gun-cotton. The produce should be entirely
soluble in ether-alcohol and nitro-glycerine, and contain
as near 12.7 per cent. of nitrogen as possible.
The theoretical nitrogen is for the penta-nitro-cellulose
12.75 per cent. This will, however, seldom if
ever be obtained. The following are some of the
results I have obtained from different samples:—­

Nitrogen.
(1.) (2.) (3.)
German make 11.64 11.48 11.49 per cent.
Stowmarket 12.57 12.60 11.22 "
Walsrode 11.61 12.07 11.99 "
Faversham 12.14 11.70 11.60 "

and the following was the analysis of a sample (No.
1) of German-made collodion-cotton, which made very
good blasting gelatine:—­

\_
Soluble cotton (collodion) 99.118 per cent.| Nitrogen = 11.64 per cent.
Gun-cotton 0.642 " \_|
Non-nitrated cotton 0.240 "
Total ash 0.25 "

It should contain as little non-nitrated or unconverted
cotton and as little gun-cotton as possible, as they
are both insoluble in nitro-glycerol. The quality
and composition of any sample of collodion-cotton
can be quickly inferred by determining the percentage
of nitrogen by means of the nitrometer and the use
of the solubility test.[A] A high nitrogen content
coupled with a high solubility is the end to be aimed
at; a high nitrogen with a low solubility shows the
presence of gun-cotton, and a low nitrogen, together
with a low solubility, the presence of unnitrated
cotton. Where complete solubility is essential
and the percentage of nitrogen less important, Dr
Lunge recommends nitration with a mixture of equal
parts of sulphuric and nitric acids containing from
19 to 20 per cent. of water.

[Footnote A: See Analysis of Explosives.]

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Mr T.R. France claims to have invented some improvements
in the manufacture of soluble nitro-cellulose.
His object has been to produce an article as uniform
as possible. His explanation of the imperfect
action of the acids is that, however uniform the mixed
acids may be in strength and proportions, and however
carefully the operations of nitrating, &c., may be
conducted, there are variable elements found in different
samples of cotton. The cotton fibre has for its
protection a glazed surface. It is tubular and
cellular in structure, and contains a natural semi-fluid
substance composed of oil or gum, which varies in nature
according to the nature of the soil upon which the
cotton is grown. The tubes of the fibre seem
to be open at one end only when the fibre is of normal
length. When, therefore, the cotton is subjected
to the action of the mixed acids, the line of least
resistance seems to be taken by them, *viz*., the
insides of the tubes constituting the fibre of the
cotton, into which they are taken by capillary attraction,
and are subject to change as they progress, and to
the increased resistance from the oil or gum, &c.,
in their progress, and therefore to modified action,
the result of which is slower and slower action, or
chemical change. He also thinks it is possible
that the power of capillary attraction is balanced
in the tubes by air contained therein, after a little,
sufficiently so to prevent the acids from taking full
effect. To get over this, Mr France uses his cotton
in a fine state, almost dust, in fact, and then nitrates
in the usual mixture of acids at 40 deg. to 90 deg.
F., the excess of acids being removed by pressure.
He says he does not find it necessary to wash this
fine cotton dust in an alkaline solution previous
to nitration. His mixed acids consist of 8 parts
HNO\_{3} = 42 deg. B., and 12 parts H\_{2}SO\_{4}
= 66 deg. B., and he stirs in the dipping tank
for fifteen minutes, the temperature being 50 deg.
F. to 100 deg. F., the temperature preferred
being 75 deg. F.
 *"Nitrated” Gun-Cotton.*—­The
nitrates that are or have been mixed with gun-cotton
in order to supply oxygen are potassium nitrate, ammonium
nitrate, and barium nitrate (tonite). The total
combustion of gun-cotton by potassium nitrate corresponds
to the equation:—­

10[C\_{24}H\_{18}(NO\_{3}H)\_{11}O\_{9}] + 82KNO\_{3} =
199CO\_{2} + 41K\_{2}CO\_{3} + 145H\_{2}O + 96N\_{2},

or 828 grms. of nitrate for 1,143 grms. of gun-cotton,
or 42 per cent. nitrate and 58 per cent. gun-cotton.
The explosive made at Faversham by the Cotton Powder
Company, and known as tonite No. 1, consists of very
nearly half gun-cotton and half barium nitrate.
The relations by weight of total combustion would
be 51.6 of gun-cotton to 48.4 of barium nitrate.
The average composition of tonite I have found by analysis
to be 51 per cent. gun-cotton to 49 per cent. barium
nitrate. The heat liberated is practically the
same as for an equivalent weight of KNO\_{3}; but the
barium nitrate mixture weighs 2,223 grms. instead of
1,971 grms., or one-eighth more. The advantage
in mixing a nitrate with gun-cotton is that it supplies
oxygen, and by converting all the carbon into carbonic
acid, prevents the formation of the poisonous gas
carbonic oxide (CO). The nitrates of potassium
and barium are also used admixed with nitro-cellulose
in several of the sporting smokeless powders.

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*The Manufacture of Tonite.*—­The explosive
tonite was patented by Messrs Trench, Faure, and Mackie,
and is manufactured at Faversham and Melling at the
works of the Cotton Powder Company, and at San Francisco
by the Tonite Powder Company. It consists of
finely divided and macerated gun-cotton incorporated
with finely ground nitrate of barium which has been
carefully recrystallised. It is made by acting
upon carbonate of barium[A] with nitric acid.
The wet and perfectly purified, finely pulped gun-cotton
is intimately mixed up between edge runners with about
the same weight of nitrate, and the mixing and grinding
continued until the whole has become an intimately
mixed paste. This paste is then compressed into
cartridges, formed with a recess at one end for the
purpose of inserting the detonator. The whole
is then covered with paraffined paper.

[Footnote A: Witherite, BaCO\_{3} + 2HNO\_{3} =
Ba(NO\_{3})\_{2} + CO\_{2} + H\_{2}O.]

The tonite No. 2 consisted of gun-cotton, nitrates
of potash and soda, charcoal and sulphur. Tonite
No. 3[A] is composed as follows:—­Gun-cotton,
19 per cent.; di-nitro-benzol, 13 per cent.; and barium
nitrate, 68 per cent. or similar proportions.
It is a yellowish colour, and being slower in its
explosive action, is better adapted for blasting soft
rock.

[Footnote A: Tonite No. 1 was patented by Messrs
Trench, Faure, and Mackie, and tonite Nos. 2 and 3
by Trench alone.]

Tonite is extensively used in torpedoes and for submarine
blasting, also for quarries, &c. Large quantities
were used in the construction of the Manchester Ship
Canal. Among its advantages are, that the English
railways will take tonite on the same footing as gunpowder;
it is a very dense material; if wetted it can easily
be dried in the sun; it very readily explodes by the
use of a proper detonator; while it burns very slowly
and without the least danger; the cartridges being
waterproofed, it can be employed in wet bore holes,
and it can be tamped with water; and finally, as it
contains sufficient oxygen to oxidise the carbon, no
carbonic oxide (CO) gas is formed, *i.e*., its
detonation is perfect. It is a very safe explosive
to use, being little susceptible to either blows or
friction.

Not long ago, a committee, composed of Prof.
P. Bedson, Drs Drummond and Hume, Mr T. Bell, one
of H.M. Inspectors of Coal Mines, and others,
in considering the problem whether the fumes produced
by the combustion of tonite were injurious to health,
carried out a series of experiments in coal mines
for this purpose. The air at the “intake”
was analysed, also the air of the “return,”
and the smoky air in the vicinity of the shot holes.
The cartridge was surrounded by the flame-extinguishing
mixture, and packed in a brown paper bag. During
the first experiment nineteen shots were fired (=
6.29 lbs. tonite). The “return” air
showed only a trace of carbonic oxide gas (CO).
At the second experiment thirteen shots were fired
(= 4.40 lbs. tonite), and analysis of the air of the
“return” showed that CO was present in
traces only, whilst the fumes contained only 1.9 to
4.8 parts per 10,000.

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*Dangers in connection with the Manufacture of Guncotton,
&c.*—­Of all the nitro compounds, the
least dangerous to manufacture are gun-cotton and
collodion-cotton. The fact that the Stowmarket
Factory is within five minutes’ walk of the
town shows how safe the manufacture of this explosive
is regarded. With the exception of the nitration
and the compression into blocks or discs, the whole
process is worked with a large excess of water, and
the probability of an explosion is thus reduced to
a minimum. Among the precautions that should,
however, be taken, are—­first, the careful
extraction of the resinous and soluble substances from
the cotton before nitration, as it was shown many
years ago by Sir F.A. Abel that the instability
of the gun-cotton first manufactured in England and
Austria was chiefly due to these compounds. They
are generally removed by boiling the cotton in a soda
solution.

The actual nitration of cotton is not a dangerous
operation, but the operations of wringing in the hydro-extractors,
and washing the nitro-cotton after it leaves the
first centrifugal machine, are somewhat so. Great
care should be taken that the wrung-out nitro-cotton
at once comes in contact with a large excess of water,
*i.e*., is at once immersed entirely in the water,
since at this stage it is especially liable to decomposition,
which, once started, is very difficult to stop.
The warmer the mixture and the less water it contains,
the more liable it is to decomposition; hence it is
that on warm and damp days the centrifugal machines
are most likely to fire. The commencement of decomposition
may be at once detected by the evolution of red fumes.
Directly the gun-cotton is immersed in the large quantity
of water in the beater and poacher it is safe.

In order that the final product may be stable and
have good keeping qualities, it is necessary that
it should be washed completely free from acid.
The treatment in the beater and poacher, by causing
the material to assume the state of a fine pulp, in
contact with a large quantity of water, does a good
deal to get rid of the free acid, but the boiling
process is absolutely necessary. It has been proposed
to neutralise the free acid with a dilute solution
of ammonia; and Dr C.O. Weber has published some
experiments bearing upon this treatment. He found
that after treatment with ammonia, pyroxyline assumed
a slightly yellowish tinge, which was a sure sign
of alkalinity. It was then removed from the water,
and roughly dried between folds of filter paper, and
afterwards dried in an oven at 70 deg. C. After
three hours, however, an explosion took place, which
entirely destroyed the strong copper oven in which
the nitro-cotton (about one oz.) had been drying.
The explosion was in some respects remarkable.
The pyroxyline was the di-nitro-cellulose (or possibly
the penta-nitro?), and the temperature was below the
igniting point of this material (40 deg. C. would
have been a better temperature). Dr Weber determined

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the ignition point of his di-nitro-cellulose, and found
it to be 194 deg. to 198 deg. C., and he is therefore
of opinion that the explosion was due to the treatment
of the partially washed material with ammonia.
A certain quantity of ammonium nitrate was probably
formed, and subsequently dried upon the nitro-cellulose,
in a state of very fine subdivision. The faintest
trace of acid would then be sufficient to bring about
the explosive ignition of the ammonium nitrate.

The drying of gun-cotton or collodion-cotton is also
a somewhat dangerous operation. A temperature
of 40 deg. C. (104 deg. F.) should not be
exceeded, and thermometers should be placed in the
nitro-cotton, and the temperature frequently observed.
An electric alarm thermometer is also a useful adjunct
to the cotton drying house. Great care must also
be taken that there are no exposed hot-water pipes
or stoves in the drying house, as the fine gun-cotton
dust produced by the turning or moving of the material
upon the shelves would settle upon such pipes or stoves,
and becoming hot, would be very sensitive to the least
friction. The floor also should be covered with
linoleum or indiarubber. When hot currents of
air are made to pass over the surface of gun-cotton,
the gun-cotton becomes electrified. It is important,
therefore, to provide some means to carry it away.
Mr W.F. Reid, F.I.C., was the first to use metal
frames, carriers, and sieves, upon which is secured
the cloth holding the gun-cotton, and to earth them.

The compression of gun-cotton into blocks, discs,
&c., is also attended with considerable risk.
Mr O. Guttmann, in an interesting paper upon “The
Dangers in the Manufacture of Explosives” (*Jour.
Soc. Chem. Ind.*, No. 3, vol. xi., 1892),
says: “The compression of gun-cotton into
cartridges requires far more care than that of gunpowder,
as this is done in a warm state, and gun-cotton even
when cold, is more sensitive than gunpowder.
When coming out of the centrifugal machines, the gun-cotton
should always pass first through a sieve, in order
to detect nails or matches which may by chance have
got into it. What has been said as to gunpowder
presses applies still more to those for gun-cotton,
although the latter are always hydraulic presses.
Generally the pistons fit the mould perfectly, that
is to say, they make aspiration like the piston of
a pump. But there is no metal as yet known which
for any length of time will stand the constant friction
of compression, and after some time the mould will
be wider in that part where the greatest compression
takes place. The best metal for this purpose
has proved to be a special steel made by Krupp, but
this also is only relatively better; for pistons I
prefer hard cast iron. If the position of the
moulds and pistons is not exactly the same in all cases,
what the Germans call ‘Ecken’ (English
‘binding’) will take place, *viz*.,
the mould will stand obliquely to the piston, and a
dangerous friction will result.” “Of

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course, it is necessary to protect the man working
the hydraulic valves during compression. At Waltham
Abbey they have a curtain made of ship’s hawsers,
which is at the same time elastic and resistant.”
Mr Guttmann has found that a partition wall 12 inches
thick, made of 2-inch planks, and filled with ground
cinders, gives very effective protection. A door
in this partition enables the workman to get to the
press, and a conical tube penetrates the wall, enabling
the man to see the whole work from a safe standpoint.
The roof, or one side of the building, should be of
glass, so as to give the explosion a direction.
 *Trench’s Fire-extinguishing Compound*
is manufactured by the Cotton Powder Company at Faversham,
and is the invention of Mr George Trench, F.C.S.,
the manager of the Company. The object of the
invention is to surround the cartridges of tonite,
when used in coal mines, with a fire-extinguishing
compound. If a charge of tonite, dynamite, or
gelatine dynamite is put inside a few ounces of this
mixture, and then fired, not the least trace of flame
can be observed, and experiments appear to show that
there is no flame at all. The compound consists
of sawdust impregnated with a mixture of alum and
chlorides of sodium and ammonia. Fig. 22 shows
the manner of placing the tonite cartridge in the paper
bag, and surrounding it with the fire-extinguishing
compound, *aa*. The attachment of the fuse
and detonator is also shown.

[Illustration: FIG. 22.—­TRENCH’S
FIRE-EXTINGUISHING CARTRIDGE.]

The following report (taken from the *Faversham
News*, 22nd Oct. 1887) of experiments conducted
in the presence of several scientific and mining men
will show its value:—­“A large wrought-iron
tank, of 45 cubic feet capacity, had been sunk level
with the ground in the middle of the yard; to this
tank the gas had been laid on, for a purpose that will
be explained later on. The charges were fired
by means of electricity, a small dynamo firing machine
being placed from 30 to 40 yards away from the ‘mine.’”
Operations were commenced by the top of the tank being
covered over and plastered down in order to make it
air-tight; then a sufficient quantity of coal gas
was placed in it to make it highly inflammable and
explosive, the quantity being ascertained by a meter
which had been fixed specially for the purpose.
Whilst the gas was being injected the cartridge was
prepared.

The first experiment was to try whether a small charge
of tonite—­fired without the patent extinguisher—­would
ignite the gas. The gas having been turned on,
a miner’s lamp was placed in the “tank,”
but this was extinguished before the full quantity
of gas had gone through the meter. However, the
gas being in, the charge of 1-1/4 oz. tonite was placed
in the “mine,” the detonator was connected
by means of long wires to the dynamo machine, and
the word was given to “fire.” With
a tremendous report, and a flash of fire, the covering

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of the mine flew in all directions, clearly showing
that the gas had exploded. The next cartridge
(a similar charge) was prepared with the patent compound.
First of all a brown paper case of about 2 inches
diameter was taken, and one of the tonite cartridges
was placed in the centre of it, the intervening space
between the charge and-the case being packed with the
“fire-extinguishing compound.” The
mine having had another supply of gas injected, the
protected cartridge was placed inside and fired.
The result was astonishing, the explosion not being
nearly so loud, whilst there was not the least flash
of fire. “Protected” and “unprotected”
charges were fired at intervals, gas being turned
into the tank on each occasion. Charges of tonite
varying from 1 to 6 oz. were also used with the compound.
The report was trifling, whilst no flash could be
seen.
 *Uses of Collodion-Cotton.*—­The collodion
or soluble gun-cotton is used for a variety of purposes.
The chief use is, however, for the manufacture of
the various explosive gelatine compounds, of which
blasting gelatine is the type. It is also very
extensively used in the manufacture of smokeless powders,
both military and sporting—­in fact, very
few of them do not contain it. In some, however,
nitro-lignose or nitrated wood is used instead.
This, however, is chemically the same thing, *viz*.,
nitro-cellulose, the cellulose being derived from
the wood fibre. It is more used in this connection
than the higher nitrate gun-cotton. Another use
to which it has been applied very extensively, of
recent years, is in the manufacture of “celluloid.”
It is used in photography for the preparation of the
films on the sensitised plates, and many other purposes.
Dissolved in a solution of two parts ether and one
of alcohol, it forms the solution known as collodion,
used for a variety of purposes, such as a varnish,
as a paint for signals; in surgery, for uniting the
edges of wounds.

Quite lately, Mr Alfred Nobel, the well-known inventor
of dynamite, has patented the use of nitro-cellulose,
hydro- or oxy-cellulose, as an artificial substitute
for indiarubber. For this purpose it is dissolved
in a suitable non-volatile or slightly volatile “solvent,”
such as nitro-naphthalene, di-nitro-benzene, nitro-toluene,
or its homologues; products are obtained varying from
a gelatinous consistency to the hardness of ebonite.
The proportions will vary from about 20 per cent. of
nitro-cellulose in the finished product, forming
a soft rubber, to 50 per cent. nitrating celluloid,
and the “solvent” chosen will depend on
the use to which the rubber substitute is to be put,
the liquids giving a more elastic substance, whilst
mixtures of solids and liquids may be employed when
the product is to be used at high temperatures.
By means of rollers steam heated, the incorporation
may be accomplished without the aid of a volatile
liquid, or the nitro-cellulose may be employed wet,
the water being removed after “solution.”

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It is advisable to use the cellulose nitrated only
just enough to render it suitable, in order to reduce
the inflammability of the finished product. Mr
W. Allen, M.P., of Gateshead, proposed to use celluloid
for cartridge cases, and thus to lighten ammunition,
and prevent jambing, for the case will be resolved
into gases along with the powder. Extractors
will also be done away with.
 *Celluloid* is an intimate mechanical mixture
of pyroxyline (gun-cotton or collodion-cotton) with
camphor, first made by Hyatt, of Newark, U.S.A., and
obtained by adding the pyroxyline to melted camphor,
or by strongly compressing the two substances together,
or by dissolving the constituents in an appropriate
solvent, *e.g*., alcohol or ether, and evaporating
to dryness. A combination of the two latter methods,
*i.e*., partial solution, with pressure, is now
usually adapted. The pyroxyline employed is generally
the tetra- and penta-nitrated cellulose, the hexa-nitrate
(gun-cotton) being but seldom used on account of its
explosive properties.

Care is taken to prevent the formation of the hexa-nitrate
by immersing the cellulose in only moderately strong
nitric acid, or in a warm mixture of nitric and sulphuric
acids. The paper, either in small pieces or in
sheets, is immersed for about twenty-five minutes in
a mixture of 2 parts of nitric acid and 5 parts of
sulphuric acid, at a temperature of about 30 deg.
C., after which the nitrated cellulose is thoroughly
washed with water to remove the last traces of free
acid, pressed, and whilst still moist, mixed with
the camphor.

In the process of Trebouillet and De Besancele, the
cellulose, which may be in the form of paper, cotton,
or linen, is twice nitrated—­first in the
acid mixture employed in a previous operation; and
secondly, in a fresh mixture of 3 parts sulphuric
acid of 1.83 specific gravity, and 2 parts concentrated
nitric acid containing nitrous acid. After each
nitration the mass is subjected to pressure, and is
then carefully washed with water, to which, at the
last, a small quantity of ammonia or caustic soda is
added to remove the final traces of acid. The
impregnation of the pyroxyline with the camphor is
effected in a variety of ways.

The usual proportion of the constituents is 2 parts
pyroxyline and 1 part camphor. In Trebouillet
and De Besancele’s process, 100 parts of pyroxyline
are intimately mixed with from 40 to 50 parts camphor,
and moulded together by strong pressure in a hot press,
and afterwards dried by exposure to air, desiccated
by calcium chloride or sulphuric acid. The usual
method is, however, to dissolve the camphor in the
least possible quantity of alcohol, and sprinkle the
solution over the dry pyroxyline, which is then covered
with a second layer of pyroxyline, and the whole again
treated with the camphor solution, the addition of
pyroxyline and camphor solution being repeated alternately
until the requisite amount of celluloid mixture is
obtained.

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The mass, which sinks together in transparent lumps,
is worked for about an hour between cold iron rollers,
and then for the same period between rollers which
can be gently heated by steam. The layer of celluloid
surrounding the rollers is then cut away and again
pressed, the resulting cake, which is now about 1
cm. thick, being cut into plates of about 70 cm. long
and 30 cm. broad. These are placed one above the
other, and strongly pressed together by hydraulic
pressure at a temperature of about 70 deg. for twenty-four
hours. The thick cakes are once more cut into
plates of the desired thickness, and placed in a chamber
heated from 30 deg. to 40 deg. for eight to fourteen
days, whereby they become thoroughly dry, and are
readily made into various articles either by being
moulded while warm under pressure, cut, or turned.
Occasionally other liquids, *e.g*., ether and wood
spirit, are used in place of alcohol as solvents for
the camphor.

Celluloid readily colours, and can be marbled for
manufacturing purposes, &c. It is highly inflammable
and not explosive even under pressure, and may be
worked under the hammer or between rollers without
risk. It softens in boiling water, and may be
moulded or pressed. Its specific gravity varies
slightly with its composition and with the degree of
pressure it has received. It is usually 1.35.
It appears to be merely a mixture of its components,
since by treatment with appropriate solvents the camphor
may be readily extracted, and on heating the pyroxyline
burns away while the camphor volatilises.

The manufacture of pyroxyline for the purpose of making
celluloid has very much increased during recent years,
and with this increase of production improved methods
of manufacture have been invented. A series of
interesting papers upon the manufacture of pyroxyline
has been published by Mr Walter D. Field, of New York,
in the *Journal of the American Chemical Society*[A]
from which the following particulars are taken:—­

[Footnote A: Vol. xv., No. 3, 1893; Vol. xvi.,
No. 7, 1894; Vol. xvi., No. 8, 1894. Figs. 19,
20, 21, 22, and 23 are taken from Mr Field’s
paper.]
 *Selection of the Fibre.*—­Cotton fibre,
wood fibre, and flax fibre in the form of raw cotton,
scoured cotton, paper, and rags are most generally
used, and give the best results. As the fibres
differ greatly in their structure, they require different
methods of nitrating. The cotton fibre is a flattened
hollow ribbon or collapsed cylindrical tube, twisted
a number of times, and closed at one end to form a
point. The central canal is large, and runs nearly
to the apex of the fibre. Its side walls are
membraneous, and are readily penetrated by the mixed
acids, and consequently the highest nitration results.
In the flax fibre the walls are comparatively thick,
the central canal small; hence it is to be presumed
that the nitration must proceed more slowly than in
the case of cotton. The New Zealand flax gives

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the most perfectly soluble nitrates of any of the
flaxes. Cotton gives a glutinous collodion, and
calico a fluid collodion. One of the largest
manufacturers of pyroxyline in the States uses the
“Memphis Star” brand of cotton. This
is an upland cotton, and its fibres are very soft,
moist, and elastic. Its colour is light creamy
white, and is retained after nitration. The staple
is short, and the twist inferior to other grades,
the straight ribbon-like filaments being quite numerous.
This cotton is used carded, but not scoured. This
brand of cotton contains a large quantity of half
and three-quarter ripe fibre, which is extremely thin
and transparent, distributed throughout the bulk of
the cotton (Monie., Cotton Fibre, 67). Mr Field
says, “This is a significant fact when it is
known that from this cotton an extremely soluble pyroxyline
can be produced.”

Pyroxyline of an inferior grade as regards colour
only can be produced from the cotton wastes of the
trade. They must be scoured before they are fit
for nitrating. Paper made from the pulps of sulphite
and sulphate processes is capable of yielding a very
soluble pyroxyline. It can be nitrated at high
temperatures and still yield good results. Tissue
paper made from flax fibre is also used after being
cut into squares.

Mowbray (U.S.P., No. 443, 105, 3rd December 1890)
says that a pure cotton tissue paper less than 1/500
inch in thickness, thin as it is, takes on a glutinous
or colloid surface, and thus requires some thirty minutes
to enable the nitration to take place. With a
thicker paper only the surface would be nitrated.
He therefore uses a fibre that has been saturated with
a solution of nitrate of soda, and afterwards dried
slowly, claiming that the salt crystallises in the
fibre, or enters by the action termed osmose, and
opens up the fibre to the action of the acid.
This process would only be useful when the cotton
is to be nitrated at a low temperature. At a
high temperature it would be unnecessary.

Dietz and Wayne (U.S.P., No. 133, 969) use ramie,
rheca, or China grass for producing a soluble pyroxyline.
That made from ramie is always of uniform strength
and solubility, and requires a smaller quantity of
solvent to dissolve it than that made from cotton.
Mr Field’s experience, however, is entirely
contrary to this statement. Such is the influence
of the physical form of the fibre on the process of
nitration, that when flax fibre and cotton fibre are
nitrated with acid mixtures of exactly the same strength,
and at the same temperature, the solution of the first
is glutinous or thick, and the second fluid or thin.
By simply nitrating at a higher temperature than the
cotton, the flax will yield a pyroxyline giving an
equally fluid collodion.

The presence of chlorine in the fibre must be carefully
avoided, as such a fibre will yield an acid product
which cannot be washed neutral. The fibre must
be dry before nitration; and this is best done, according
to Mr Field, by using the form of drier used in drying
wool.

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*Nitration of the Fibre.*—­Mixed cotton
and flax fibre in the form of paper, from 2/1000 to
3/1000 inch thick, and cut into 1-inch squares, is
nitrated by the Celluloid Manufacturing Company, and
the same paper, left in long strips, 1 inch wide,
is used for nitration by the Xylonite Manufacturing
Company, of North Adams, Mass. (U.S.A.).

The Celluloid Company introduce the cut paper into
the mixed acids by means of a hollow, rapidly revolving
tube, flared at the lower end, and immersed in the
mixed acids. The centrifugal force of the revolving
tube throws the paper towards the sides of the vessel,
leaving the centre of the vessel ready for fresh paper.

The Xylonite Company simply cut the paper into long
strips, and introduce it into the mixed acids by means
of forks. The arrangement used by this Company
for holding the mixed acids is a cylindrical vessel
divided into a number of sections, the whole revolving
like a turntable, thus allowing the workman to nitrate
successively each lot of paper at a given point.
This Company did not remove the acid from the paper
after its immersion, but plunged it immediately into
the water, thus losing a large proportion of the waste
acid. The Celluloid Company, by using the paper
in smaller pieces, and more paper to a pound of acid,
and wringing the mixed acid from the paper before
immersion in water, had a better process of nitration.

Other manufacturers use earthenware vessels, and glass
or steel rods, hooked at one end, having small pieces
of rubber hose pulled over the other end to prevent
the hand from slipping. The form of vessel in
general use is that given in Fig. 23. It is large
enough to nitrate 1 lb. of cotton at a time.
The hook at one end of the rod enables the workman
to pull the pyroxyline apart, and thus ensures saturation
of the fibre. In the winter the room in which
the nitrating is done must be kept at a temperature
of about 70 deg. F. in order to secure equality
in the batches.

[Illustration: FIG. 23.—­VESSEL FOR
NITRATING COTTON OR PAPER.]

The nitrating apparatus of White and Schupphaus (U.S.P.,
No. 418, 237, 89) Mr Field considers to be both novel
and excellent. The cage (Fig. 24), with its central
perforated cylinder (Fig. 25), is intended to ensure
the rapid and perfect saturation of the tissue paper
used for nitrating. The patentees say that no
stirring is required with their apparatus. This,
says Mr Field, might be true when paper is used, or
even cotton, when the temperature of nitration is
from 30 deg. to 35 deg. C., but would not be true
if the temperature were raised to 50 deg. to 55 deg.
C. The process is as follows:—­ The paper
is nitrated in the cage (Fig. 25), the bottom of which
is formed by the flanged plate C, fastened to the
bottom of the internal cylinder B. After nitration
the cage is carried to a wringer, which forms the basket,
and the acids removed. Finally, the cage is taken
to a plunge tank, where the paper is removed from

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the cage by simply pulling out the central perforated
cylinder B. Fig. 26 shows the nitrating pot, with its
automatic cover. The plunge tank is shown in
plan and section in Figs. 28 and 29. This apparatus
is suitable for the nitration of cotton fibre in bulk
at high or low temperatures. Other methods that
have been patented are Mowbray’s (U.S.P., No.
434, 287), in which it is proposed to nitrate paper
in continuous lengths, and Hyatt’s (U.S.P., No.
210, 611).

[Illustration: FIG. 24.—­CENTRAL PERFORATED
CYLINDER.]

[Illustration: FIG. 25.—­THE CAGE.
WHITE AND SCHUPPHAUS’ NITRATING APPARATUS.]

[Illustration: FIG. 26.—­CELLULOID
NITRATING POT.]

[Illustration: FIG. 27.—­ANOTHER VIEW.]

[Illustration: FIGS. 28, 29.—­PLUNGE
TANK, IN PLAN AND SECTION.]
 *The Acid Mixture.*—­Various formulae
have been published for producing soluble nitro-cellulose.
In many instances, although the observations were
correct for the single experiment, a dozen experiments
would have produced a dozen different products.
The composition of the acids used depends upon the
substance to be nitrated, and the temperature at which
the nitration will be worked. Practically there
are three formulae in general use—­the one
used by the celluloid manufacturers; another in which
the cotton is nitrated at high temperatures; and a
third in which the temperature of the immersion is
low, and the time of nitration about six hours.
Of the three, the best method is the last one, or
the one in which the cotton is immersed at a low temperature,
and then the reaction allowed to proceed in pots holding
from 5 to 10 lbs. of cotton. The formula used
by the celluloid manufacturers for the production
of the low form of nitrated product which they use
is:—­

Sulphuric acid 66 parts by weight.
Nitric acid 17 " "
Water 17 " "

Temperature of immersion, 30 deg. C. Time, twenty
to thirty minutes.

The cellulose is used in the form of tissue paper
2/1000 inch thick, 1 lb. to 100 of acid mixture.
The nitro-cellulose produced by this formula is very
insoluble in the compound ethers and other solvents
of pyroxyline, and is seemingly only converted or
gelatinised by the action of the solvent. The
next formula produces a mixture of tetra-and penta-nitro-celluloses
hardly soluble in methyl-alcohol (free from acetone),
but very soluble in anhydrous compound ethers, ketones,
and aldehydes:—­

Nitric acid, sp. gr. 1.435 8 lbs.
Sulphuric acid, sp. gr. 1.83 15-3/4 lbs.
Cotton 14 oz.

Temperature of nitration, 60 deg. C. Time of
immersion, forty-five minutes.

The 60 deg. of temperature is developed by mixing
the acids together. The cotton is allowed to
remain in the acid until it feels “short”
to the rod.

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The following table, due to Mr W.D. Field, shows
very plainly the great variation in the time of the
immersion and the temperature by seemingly very slight
causes. It extends over fourteen working days,
during which time it rained four days. The formula
used is that given above, except that the specific
gravity of the nitric acid is somewhat lower.
The product obtained differs only from that produced
by using nitric acid of specific gravity 1.43 in being
soluble in methyl-alcohol. From 30 to 35 lbs.
of pyroxyline were produced in each of the fourteen
days.

A careful examination of this table will prove very
instructive. The increase in yield varies from
31 per cent. to nothing, and the loss runs as high
as 10 per cent., yet care was taken to make the product
uniform in quality. On the days it rained there
was a loss, with the exception of the fourth day,
when there was neither a loss nor a gain. On the
days it was partly clear, as just before or after
rain, the table shows a loss in product. We can
explain this fact by reason of the moisture-absorbing
qualities of the cotton. On the rainy days it
would absorb the moisture from the air until, when
immersed in the acids, they were weakened, and the
fibre dissolved more or less in weakened acid, producing
what is known as “burning” in the batch.
It will also be noticed that on days which show a
loss, the time of the immersion was correspondingly
short, as on the a loss, the time of the immersion
was correspondingly short, as on the tenth, twelfth,
and seventh days.

\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_
\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_
| | | |
| | Specific Gravity. | Time. |
| |\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_|\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_|
| | | | | | | |
| |H\_{2}S0\_{4}.|HNO\_{3}.|Hours.|Minutes.|Hours.|Minutes.|
|\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_|\_\_\_\_\_\_\_\_\_\_\_\_|\_\_\_\_\_\_\_\_|\_\_\_\_\_\_|\_\_\_\_\_\_\_\_|\_\_\_\_
\_\_|\_\_\_\_\_\_\_\_|
| | | | | | | |
| 1. Clear | 1.838 | 1.4249 | ... | 20 | 4 | ... |
| 2. " | 1.837 | 1.4249 | ... | 20 | 2 | ... |
| 3. Cloudy | 1.837 | 1.4226 | ... | 45 | 2 | ... |
| 4. Rain | 1.837 | 1.420 | ... | 20 | 1 | 20 |
| 5. Clear | 1.8377 | 1.42 | 1 | 15 | 2 | ... |
| 6. Rainy | 1.8391 | 1.422 | ... | 35 | 1 | 40 |
| 7. Cloudy | 1.835 | 1.4226 | ... | 20 | ... | 35 |
| 8. Clear | 1.835 | 1.422 | ... | 35 | 1 | 10 |
| 9. Partly Clear| 1.824 | 1.4271 | ... | 20 | 1 | ... |
|10. " | 1.83 | 1.4271 | ... | 10 | ... | 25 |
|11. Cloudy | 1.832 | 1.425 | ... | 10 | ... | 50 |
|12. Rainy | 1.822 | 1.425 | ... | 10 | ... | 20 |

The lesson this table teaches is, that it is almost
impossible to nitrate cellulose in small quantities,
and get uniform results, when the nitration is carried
on at high temperatures. As regards the solubility
of pyroxyline, Parks found that nitro-benzene, aniline,
glacial acetic acid, and camphor, dissolved in the
more volatile solvents methyl-alcohol and alcohol-ether,
were much the best solvents for producing a plastic,
as they are less volatile, and develop greater solvent
action under the influence of heat. Nitro-benzene
gives a solution that is granular; it seems to merely
convert the pyroxyline, and not to dissolve it; but
on the addition of alcohol, a solution is at once
obtained, and the granular appearance disappears,
and the solution becomes homogeneous. The acid
mixture and the method of nitrating have much to do
with the action of the various solvents, so also has
the presence of water.

Dr Schupphaus found that propyl and isobutyl alcohols
with camphor were active solvents, and the ketones,
palmitone, and stearone in alcohol solution, also
alpha- and beta-naphthol, with alcohol and anthraquinone
(diphenylene diketone) in alcoholic solution, and also
iso-valeric aldehyde and its derivatives, amyliden-dimethyl
and amyliden-diethyl ethers.

August Sayer (U.S.P., No. 470,451) finds diethyl-ketone,
dibutyl-ketone, di-pentyl-ketone, and the mixed ketones,[A]
methyl-ethyl, methyl-propyl, methyl-butyl, methyl-amyl,
and ethyl-butyl ketones are active solvents of pyroxyline;
and Paget finds that although methyl-amyl oxide is
a solvent, that ethyl-amyl oxide is not.

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[Footnote A: Ketones are derived from the fatty
acids by the substitution of the hydroxyl of the latter
by a monad positive radical. They thus resemble
aldehydes in constitution. The best-known ketone
is acetone CH\_{3}CO.CH\_{3}. Mixed ketones are
obtained by distilling together salts of two different
fatty acids. Thus potassic butyrate and potassic
acetate form propyl-methyl-ketone—­

C(C\_{2}H\_{5})H\_{2}
|
CO.CH\_{3}]

The solvents of pyroxyline can be divided into general
classes—­First, those which are solvents
without the aid of heat or solution in alcohol; second,
those that are solvents when dissolved in alcohol.
These solvents are those which also develop a solvent
action when heated to their melting point in combination
with pyroxyline.

Mr W.D. Field groups the solvents of pyroxyline
into classes thus: Two of the monohydric alcohols;
compound ethers of the fatty acids with monohydric
alcohols, aldehydes; simple and mixed ketones of the
fatty acid series. These four classes include
the greater number of the solvents of pyroxyline.
Those not included are as follows:—­Amyl-nitrate
and nitrite, methylene-di-methyl ether, ethidene-diethyl
ether, amyl-chloracetate, nitro-benzene and di-nitro-benzene,
coumarin, camphor, glacial acetic acid, and mono-,
di-, and tri-acetin.

Richard Hale uses the following solvent:—­Amyl-acetate,
4 volumes; petroleum naphtha, 4 volumes; methyl-alcohol,
2 volumes; pyroxyline, 4 to 5 ounces to the gallon
of solvent. Hale used petroleum naphtha to hasten
the drying qualities of the varnish, so that it would
set on the article to be varnished before it had a
chance to run off. It is, however, the non-hygroscopic
character of the solvent that makes the varnish successful.
This formula is very largely used for the production
of pyroxyline varnish, which is used for varnishing
pens, pencils, &c., also brass-work and silver-ware.

The body known as oxy-cellulose[A] is formed by the
action of nitric acid upon cellulose when boiled with
it. The quantity formed is about 30 per cent.
of cellulose acted upon. When washed free from
acid, it gelatinises. It is then soluble in dilute
alkalies, and can be reprecipitated from solution
by alcohol, acids, or saline solutions. Messrs
Cross and Bevan assign to it the formula C\_{18}H\_{26}O\_{16}.
It dissolves in concentrated sulphuric acid, and with
nitric acid forms a nitro body of the formula C\_{18}H\_{23}O\_{16}3(NO\_{2}),
which is prepared as follows:—­The gelatinous
oxy-cellulose is washed with strong nitric acid until
free from water, and is then diffused through a mixture
of equal volumes of strong sulphuric and nitric acids,
in which it quickly dissolves. The solution, after
standing for about an hour, is poured in a fine stream
into a large volume of water, by which the “nitro”
body is precipitated as a white flocculent mass.
The product, after drying at 110 deg. C., was
found upon analysis to contain 6.48 per cent. nitrogen.

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[Footnote A: “On the Oxidation of Cellulose,”
by C.F. Cross and E.J. Bevan, *Jour.
Chem. Soc.*, 1883, p. 22.]

MISCELLANEOUS NITRO-EXPLOSIVES.
 *Nitro-Starch.*—­It is only recently
that, by means of the process introduced by the “Actiengesellschaft
Dynamit Nobel,” it has been possible to make
this explosive upon the manufacturing scale. Nitro-starch
has been known since 1883, when Braconnot discovered
it, and called it xyloidine. Its formula is C\_{6}H\_{8}O\_{3}(NO\_{3})\_{2},
but Dr Otto Muehlhaeusen has lately succeeded in preparing
higher nitrated compounds, *viz*.:—­

(*a.*) C\_{6}H\_{7-1/2}O\_{2-1/2}(NO\_{3})\_{2-1/2}.

(*b.*) C\_{6}H\_{7}O\_{4}(NO\_{3})\_{3}.

Or doubling the molecule of starch:—­

Nitrogen.
i. Tetra-nitro-starch C\_{12}H\_{16}O\_{6}(ONO\_{2})\_{4} 11.11 per cent.
ii. Penta-nitro-starch C\_{12}H\_{15}O\_{5}(ONO\_{2})\_{5} 12.75 "
iii. Hexa-nitro-starch C\_{12}H\_{14}O\_{4}(ONO\_{2})\_{6} 14.14 "

He regards them as true ethers (esters) of nitric
acid. Thus on treatment with sulphuric acid,
these compounds yield NO\_{3}H, the residue O.NO\_{2}
thus appearing to be replaced by the sulphuric acid
residue. On treatment with a solution of ferrous
chloride, nitric oxide and “soluble” starch
are regenerated. On shaking with sulphuric acid
over mercury, all the nitrogen is split off as NO.

Tetra-nitro-starch is prepared upon the large scale
as follows:—­A quantity of potato-starch
is taken and exposed in some suitable desiccating
apparatus at a temperature of 100 deg. C. until
all the moisture which it contains is completely driven
off. It is then reduced to a fine powder by grinding,
and dissolved in nitric acid of specific gravity 1.501.
The vessel in which this solution is accomplished is
made of lead, and must be provided with two jackets,
cooled by means of water. It should further be
fitted with a screw-agitator, in order to keep the
nitric acid circulating freely. The charge of
starch is introduced through an opening in the cover
of this digesting vessel, and the proportions of acid
to starch are 10 kilogrammes of starch to 100 kilos.
of acid. The temperature is kept within the limits
20 deg. to 25 deg. C. When the solution of the
starch is complete, the liquid is conducted into a
precipitating apparatus, which is also provided with
a cooling jacket, for the purpose of regulating the
temperature. The bottom of this vessel is double
and perforated, and here is placed a layer of gun-cotton
to act as a filter. This vessel is filled with
spent nitro-sulphuric acid obtained as a waste product
from the nitro-glycerine manufactory, and the solution
of starch in nitric acid is sprayed into it through
an injector worked by compressed air, whereby the
nitro-starch is thrown down in the form of a fine-grained
powdery precipitate.

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In order to precipitate 100 kilos. of the acid solution
of starch, it is necessary to employ 500 kilos. of
spent nitro-sulphuric acid. As it is precipitated
the nitro-starch collects on the gun-cotton filter,
and the acid liquor is run off through a tap placed
beneath the perforated double bottom of the vessel,
and of course below the filter pad. The precipitated
starch is further cleansed from acid by repeated washings
and by pressure, until all trace of acidity has been
eliminated, and the substance exhibits a neutral reaction.
The next step is to treat the nitro-starch with a 5
per cent. solution of soda, in contact with which it
is allowed to stand for at least twenty-four hours.
The product is then ground up until a sort of “milk”
or emulsion is obtained, and lastly treated with a
solution of aniline, so that when pressed into cake,
it contains about 33 per cent. of water, and 1 per
cent. of aniline.

Dr Muehlhaeusen, working on these lines in the laboratory,
prepared nitro-starch which contained 10.96 and 11.09
per cent. of nitrogen. When in the state of powder
it is snow-white in colour; it becomes electrified
when rubbed; it is very stable, and soluble even in
the cold in nitro-glycerine. He has also prepared
a tetra-nitro-starch containing 10.58 and 10.50 per
cent. of nitrogen, by pouring water into a solution
of starch in nitric acid which had stood for several
days. The substance thus produced in the laboratory
had all the properties of that prepared by the other
process.

The production of penta-nitro-starch is effected by
adding 20 grms. of rice-starch—­previously
dried at a temperature of 100 deg.C., in order to
eliminate all moisture—­to a mixture of 100
grms. of nitric acid, specific gravity 1.501, and
300 grms. of sulphuric acid, specific gravity 1.8 (some
tetra-nitro-starch is also formed at the same time).
After standing in contact with these mixed acids for
one hour the starch has undergone a change, and the
mass may now be discharged into a large quantity of
water, and then washed, first with water, and finally
with an aqueous solution of soda. The yield in
Dr Muehlhaeusen’s experiments was 147.5 per cent.

The substance thus formed is now heated with ether-alcohol,
the ether is distilled off, and the penta-nitro-starch
appears as a precipitate, whilst the tetra-nitro-starch,
which is formed simultaneously, remains in solution
in the alcohol. As obtained by this process, it
contained 12.76 and 12.98 per cent. nitrogen, whilst
the soluble tetra-nitro-starch contained 10.45 per
cent.

Hexa-nitro-starch is the product chiefly formed when
40 grms. of dry starch are treated with 400 grms.
of nitric acid, specific gravity 1.501, and allowed
to stand in contact for twenty-four hours; 200 grms.
of this mixture are then poured into 600 c.c. of sulphuric
acid of 66 deg. B. The result of this manipulation
is a white precipitate, which contains 13.52-13.23
and 13.22 per cent. nitrogen; and consists, therefore,
of a mixture of penta- and hexa-nitro-starch.

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The experiments undertaken with these substances demonstrated
that those prepared by precipitating the nitro-starch
with strong sulphuric acid were less stable in character
or properties than those which were precipitated by
water or weak sulphuric acid. Dr Muehlhaeusen
is of opinion that possibly in the former case a sulpho-group
may be formed, which in small quantity may occasion
this instability.

The following table shows the behaviour of these substances
prepared in different ways and under various conditions:—­

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| | SAMPLES. |
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| | | | | | |
| | A. | B. | C. | D. | E. |
| Ignition-point |175 deg. C. |170 deg. C. |152 deg. C. |121 deg. C. |155 deg. C. |
| Stability |Stable |Stable |Unstable|Unstable|Unstable|
| Per cent. of N. | 11.02 | 10.54 | 12.87 | 12.59 | 13.52 |
| 96 per cent. alcohol| Sol. | Sol. | Insol. | Insol. | Insol. |
| Ether | Insol. | Insol. | Insol. | Insol. | Insol. |
| Ether-alcohol | Sol. | Sol. | Sol. | Sol. | Sol. |
| Acetic Ether | Sol. | Sol. | Sol. | Sol. | Sol. |
|\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_|\_\_\_\_\_\_\_\_|\_\_\_\_\_\_\_\_|\_\_\_\_\_\_\_\_|\_\_\_\_\_\_\_\_|\_
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These samples were prepared as follows:—­

A. From 1 part nitric acid and 2 parts sulphuric acid
(containing 70 per
 cent. H\_{2}O).
B. From 1 part nitric acid and water.
C. From 1 part nitric and 3 parts H\_{2}SO\_{4} (con.).
D. From 1 part nitric and 3.5 parts con. H\_{2}SO\_{4}.
E. From 1 part nitric and 3 parts con. H\_{2}SO\_{4}.

Dr Muehlhaeusen is of opinion that these compounds
may be turned to practical account in the production
of good smokeless powder. He recommends the following
proportions and method. Six grms. of nitro-jute
and 2 grms. of nitro-starch are mixed together, and
moistened with acetic ether. These ingredients
are then worked together into a uniform mass, and
dried at a temperature ranging between the limits 50
deg. to 60 deg. C. He has himself prepared such
a smokeless powder, which proved to contain 11.54
per cent. of nitrogen, and was very stable. Further
details of Dr Muehlhaeusen’s work upon nitro-starch
can be found in *Dingler’s Polytechnisches
Journal*, paper “Die hoehren Salpetersaeureaether
der Staerke,” 1892, Band 284, s. 137-143, and
a Bibliography up to 1892 in *Arms and Explosives*,
December 1892.

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M. Berthelot gives the heat of formation of nitro-starch
as 812 cals. for 1 grm., and the heat of total combustion
as equal to 706.5 cals. for 207 grms., or for 1 grm.
3,413 cals. The heat of decomposition could only
be calculated if the products of decomposition were
given, but they have not as yet been studied, and
the quantity of oxygen contained in the compound is
far from being sufficient for its complete combustion.
Berthelot and Vieille found the average velocities
for nitro-starch powder, density of charge about 1.2,
in a tin tube 4 mm. external diameter, to be, in two
experiments, 5,222 m. and 5,674 m. In a tin tube
5.5 mm. external diameter, the velocity was 5,815
m., and in lead tube 5,006 m. (density 1.1 to 1.2).
The starch powder is hygroscopic, and is insoluble
in water and alcohol. When dry it is very explosive,
and takes fire at about 350 deg. F. Mr Alfred
Nobel has taken out a patent (Eng. Pat. No.
6,560, 88) for the use of nitro-starch. His invention
relates to the treatment of nitro-starch and nitro-dextrine,
for the purpose of producing an explosive powder,
to be used in place of gunpowder. He incorporates
these materials with nitro-cellulose, and dissolves
the whole in acetone, which is afterwards distilled
off. A perfect incorporation of the ingredients
is thus brought about.
 *Nitro-Jute.*—­It is obtained by treating
jute with nitric acid. Its properties have been
studied by Messrs Cross and Bevan (*Jour. Chem.
Soc.*, 1889, 199), and by Muehlhaeusen. The
latter used for its nitration an acid mixture composed
of equal parts of nitric and sulphuric acids, which
was allowed to act upon the jute for some time.
He found that with long exposure, *i.e*., from
three to four hours in the acids, there was a disintegrating
of the fibre-bundles, and the nitration was attended
by secondary decomposition and conversion into products
soluble in the acid mixture. Cross and Bevan’s
work upon this subject leads them to conclude that
the highest yield of nitrate is represented by an increase
of weight of 51 per cent. They give jute the
empirical formula C\_{12}H\_{18}O\_{9} (C = 47 per cent.
H = 6 per cent., and O = 47 per cent.), and believe
its conversion into a nitro compound to take place
thus:—­

C\_{12}H\_{18}O + 3HNO\_{3} = C\_{12}H\_{15}O\_{6}(NO\_{3})\_{3}
+ 3H\_{2}O.

This is equivalent to a gain in weight of 44 per cent.
for the tri-nitrate, and of 58 per cent. for the
tetra-nitrate. The formation of the tetra-nitrate
appears to be the limit of nitration of jute-fibre.
In other words, if we represent the ligno-cellulose
molecule by a C\_{12} formula, it will contain four
hydroxyl (OH) groups, or two less than cellulose similarly
represented. The following are their nitration
results:—­

Acids used.—­I. HNO\_{3} sp. gr. 1.43,
and H\_{2}SO\_{4} = 1.84 equal parts.
 II.
1 vol. HNO\_{3}(1.5), 1 vol. H\_{2}SO\_{4}(1.84).
 III.
1 vol. HNO\_{3}(1.5), 75 vols. H\_{2}SO\_{4}(1.84).

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I. = 144.4; II. = 153.3; III. = 154.4 grms.; 100 grms.
of fibre being used in all three cases.

Duration of exposure, thirty minutes at 18 deg.
C.

The nitrogen was determined in the products, and equalled
10.5 per cent. Theory for C\_{12}H\_{15}O\_{6}(NO\_{3})\_{3}
= 9.5 per cent. and for C\_{12}H\_{15}O\_{6}(NO\_{3})\_{4}
= 11.5 per cent. These nitrates resemble those
of cellulose, and are in all essential points nitrates
of ligno-cellulose.

Muehlhaeusen obtained a much lower yield, and probably,
as pointed out by Cross and Bevan, a secondary decomposition
took place, and his products, therefore, probably
approximate to the derivatives of cellulose rather
than to those of ligno-cellulose, the more oxidisable,
non-cellulose, or lignone constituents having been
decomposed. In fact, he regards his product as
cellulose penta-nitrate (C\_{12}H\_{16}O\_{5}(ONO\_{2})\_{5}).
The *Chemiker Zeitung*, xxi., p. 163, contains
a further paper by Muehlhaeusen on the explosive nitro-jute.
After purifying the jute-fibre by boiling it with
a 1 per cent. solution of sodium carbonate, and washing
with water, he treated 1 part of the purified jute
with 15 parts of nitro-sulphuric acid, and obtained
the following results with different proportions of
nitric to sulphuric acids:—­

Yield Ignition Nitrogen.
per cent. Point.
Experiment I.—­ 1. HNO\_{3} 1. H\_{2}SO\_{4} 129.5 170 deg. C. 11.96%
" II. " 2. " 132.2 167 deg. C. 12.15%
" III. " 3. " 135.8 169 deg. C. 11.91%

An experiment made with fine carded jute and the same
mixture of acids as in No. II. gave 145.4 per
cent. nitro-jute, which ignited at 192 deg. C.,
and contained 12 per cent. nitrogen. This explosive
is not at present manufactured upon the large scale,
and Messrs Cross and Bevan are of opinion that there
is no very obvious advantage in the use of lignified
textile fibre as raw materials for explosive nitrates,
seeing that a large number of raw materials containing
cellulose (chiefly as cotton) can be obtained at a
cheaper rate, and yield also 150 to 170 per cent. of
explosive material when nitrated, and are in many ways
superior to the products obtained hitherto from jute.
 *Nitro-mannite* is formed by the action of nitric
acid on mannite, a hex-acid alcohol closely related
to sugar. It occurs abundantly in manna, which
is the partly dried sap of the manna-ash (*Fraxinus
ornus*). It is formed in the lactic acid fermentation
of sugar, and by the action of nascent hydrogen on
glucose and cellulose, or on invert sugar. Its
formula is C\_{6}H\_{8}(OH)\_{6} and that of nitro-mannite
C\_{6}H\_{8}(NO\_{3})\_{6}. Mannite crystallises
in needles or rhombic prisms, which are soluble in
water and alcohol, and have a sweet taste. Nitro-mannite
forms white needle-shaped crystals, insoluble in water,
but soluble in ether or alcohol. When rapidly
heated, they ignite at about 374 deg. F., and
explode at about 590 deg. F. It is more susceptible
to friction and percussion than nitro-glycerine, and
unless pure it is liable to spontaneous decomposition.
It is considered as the nitric ether of the hexatomic
alcohol mannite. It is formed by the action of
a mixture of nitric and sulphuric acids upon mannite—­

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C\_{6}H\_{8}(OH)\_{6} + 6HNO\_{3} = C\_{6}H\_{8}(NO\_{3})\_{6}
+ 6H\_{2}O.

Its products of explosion are as shown in the following
equation:—­

C\_{6}H\_{8}(OH)\_{6} = 6CO\_{2} + 4H\_{2}O + 3N\_{2} +
O\_{2}.

Its percentage composition is as follows:—­Carbon,
15.9 per cent.; hydrogen, 1.8 per cent.; nitrogen,
18.6 per cent.; and oxygen, 63.7 per cent. Its
melting point is 112 to 113 deg. C., and it solidifies
at 93 deg.. When carefully prepared and purified
by recrystallisation from alcohol, and kept protected
from sunlight, it can be kept for several years without
alteration.

Nitro-mannite is more dangerous than nitro-glycerine,
as it is more sensitive to shock. It is intermediate
in its shattering properties between nitro-glycerine
and fulminate of mercury. It explodes by the shock
of copper on iron or copper, and even of porcelain
on porcelain, provided the latter shock be violent.
Its heat of formation from its elements is +156.1
calories. It is not manufactured upon the commercial
scale.

Besides the nitro compounds already described, there
are many others, but they are of little importance,
and are none of them made upon the large scale.
Among such substances are *nitro-coal*, which
is made by the action of nitric acid on coal; *nitro-colle*,
a product which results from the action of nitric
acid on isinglass or gelatine, soaked in water.
It is then treated with the usual acids.

Another method is to place strong glue in cold water
until it has absorbed the maximum amount of the latter.
The mixture is solidified by the addition of nitric
acid, nitrated in the usual way, and well washed.
Abel’s *Glyoxiline* is only nitrated gun-cotton
impregnated with nitro-glycerine. Nitro-lignine
is only nitro-cellulose made from wood instead of
cotton; and nitro-straw is also only nitro-cellulose.
The explosive known as *Keil’s Explosive*
contains nitro-glucose. Nitro-molasses, which
is a liquid product, has also been proposed, and nitro-saccharose,
the product obtained by the nitration of sugar.
It is a white, sandy, explosive substance, soluble
in alcohol and ether. When made from cane sugar,
it does not crystallise; but if made from milk sugar,
it does. It has been used in percussion caps,
being stronger and quicker than nitro-glycerine.
It is, however, very sensitive and very hygroscopic,
and very prone to decomposition. Nitro-tar, made
from crude tar-oil, by nitration with nitric acid
of a specific gravity of 1.53 to 1.54. Nitro-toluol
is used, mixed with nitro-glycerine. This list,
however, does not exhaust the various substances that
have been nitrated and proposed as explosives.
Even such unlikely substances as horse dung have been
experimented with. None of them are very much
used, and very few of them are made upon the manufacturing
scale.

**CHAPTER IV.**

*DYNAMITE AND GELATINES.*

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Kieselguhr Dynamite—­Classification of Dynamites—­Properties
and
Efficiency of Ordinary Dynamite—­Other Forms
of Dynamite—­Gelatine and
Gelatine Dynamites, Suitable Gun-Cotton for, and Treatment
of—­Other
Materials used—­Composition of Gelignite—­Blasting
Gelatine—­Gelatine
Dynamite—­Absorbing Materials—­Wood
Pulp—­Potassium Nitrate, &c.—­
Manufacture and Apparatus used, and Properties of
Gelatine Dynamites—­
Cordite—­Composition and Manufacture.
 *Dynamite.*—­Dynamite consists of nitro-glycerine
either absorbed by some porous material, or mixed
with some other substance or substances which are
either explosives or merely inert materials. Among
the porous substances used is kieselguhr, a silicious
earth which consists chiefly of the skeletons of various
species of diatoms. This earth occurs in beds
chiefly in Hanover, Sweden, and Scotland. The
best quality for the purpose of manufacturing dynamite
is that which contains the largest quantity of the
long tubular *bacillariae*, and less of the round
and lancet-shaped forms, such as *pleurosigmata*
and *diclyochae*, as the tube-shaped diatoms
absorb the nitro-glycerine better, and it becomes packed
into the centre of the silicious skeleton of the diatoms,
the skeleton acting as a kind of tamping, and increasing
the intensity of the explosion.

Dynamites are classified by the late Colonel Cundill,
R.A., in his “Dictionary of Explosives”
as follows:—­

1. Dynamites with an inert base, acting merely
as an absorbent.

2. Dynamites with an active base, *i.e*.,
an explosive base. No. 2 may be again divided
into three minor classes, which contain as base—­

(*a.*) Charcoal.

(*b.*) Gunpowder or other nitrate, or chlorate
mixture.

(*c.*) Gun-cotton or other nitro compound (nitro-benzol,
&c.).

The first of these, *viz*., charcoal, was one of
the first absorbents for nitro-glycerine ever used;
the second is represented by the well-known Atlas
powder; and the last includes the well-known and largely
used gelatine compounds, *viz*., gelignite and
gelatine dynamite, and also tonite No. 3, &c.

In the year 1867 Nobel produced dynamite by absorbing
the nitro-glycerine in an inert substance, forming
a plastic mass. In his patent he says: “This
invention relates to the use of nitro-glycerine in
an altered condition, which renders it far more practical
and safe for use. The altered condition of the
nitro-glycerine is effected by causing it to be absorbed
in porous unexplosive substances, such as charcoal,
silica, paper, or similar materials, whereby it is
converted into a powder, which I call dynamite, or
Nobel’s safety powder. By the absorption
of the nitro-glycerine in some porous substance it
acquires the property of being in a high degree insensible
to shocks, and it can also be burned over a fire without
exploding.”

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Ordinary dynamite consists of a mixture of 75 per
cent. of nitro-glycerine and 25 per cent. of kieselguhr.
The guhr as imported (Messrs A. Haake & Co. are the
chief importers) contains from 20 to 30 per cent. of
water and organic matter. The water may be very
easily estimated by drying a weighed quantity in a
platinum crucible at 100 deg. C. for some time
and re-weighing, and the organic matter by igniting
the residue strongly over a Bunsen burner. Before
the guhr can be used for making dynamite it must be
calcined, in order not only to get rid of moisture,
but also the organic matter.

A good guhr should absorb four times its weight of
nitro-glycerine, and should then form a comparatively
dry mixture. It should be pale pink, red brown,
or white. The pink is generally preferred, and
it should be as free as possible from grit of all
kinds, quartz particles, &c., and should have a smooth
feeling when rubbed between the finger and thumb, and
should show a large quantity of diatoms when viewed
under the microscope. The following was the analysis
of a dried sample of kieselguhr:—­Silica,
94.30; magnesia, 2.10; oxide of iron and alumina, 1.3;
organic matter, 0.40; moisture, 1.90 per cent.

The guhr is generally dried in a reverberatory muffle
furnace. It is spread out on the bottom to the
thickness of 3 or 4 inches, and should every now and
then be turned over and raked about with an iron rabble
or hoe. The temperature should be sufficiently
high to make the guhr red hot, or the organic matter
will not be burnt off. The time occupied in calcining
will depend of course upon the quality of the guhr
being operated upon. Those containing a high
percentage of water and organic matter will of course
take longer than those that do not. A sample of
the calcined guhr should not contain more than 0.5
per cent. of moisture and organic matter together.

After the guhr is dry it requires to be sifted and
crushed. The crushing is done by passing it between
iron rollers fixed at the bottom of a cone or hopper,
and revolving at a moderate speed. Beneath the
rollers a fine sieve should be placed, through which
the guhr must be made to pass.

The kieselguhr having been dried, crushed, and sifted,
should be packed away in bags, and care should be
taken that it does not again absorb moisture, as if
it contains anything above about five-tenths per cent.
of water it will cause the dynamite made with it to
exude. The guhr thus prepared is taken up to
the danger area, and mixed with nitro-glycerine.
The nitro-glycerine used should be quite free from
water, and clear, and should have been standing for
a day or two in the precipitating house. The
guhr and nitro-glycerine are mixed in lead tanks (about
1-1/2 foot deep, and 2 to 3 feet long), in the proportions
of 75 of the nitro-glycerine to 25 of the guhr, unless
the guhr is found to be too absorbent, which will
cause the dynamite to be too dry and to crumble.
In this case a small quantity of barium sulphate,
say about 1 per cent., should be added to the guhr.
This will lessen its absorbing powers, or a highly
absorptive sample of guhr may be mixed with one of
less absorptive power, in the proportions found by
experiment to be the best suited to make a fairly moist
dynamite, but one that will not exude.

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The mixing itself is generally performed in a separate
house. In a series of lead-lined tanks the guhr
is weighed, placed in a tank, and the nitro-glycerine
poured on to it. The nitro-glycerine may be weighed
out in indiarubber buckets. The whole is then
mixed by hand, and well rubbed between the hands,
and afterwards passed through a sieve. At this
stage the dynamite should be dry and powdery, and
of a uniform colour.

It is now ready to be made up into cartridges, and
should be taken over to the cartridge huts. These
are small buildings surrounded with mounds, and contain
a single cartridge machine. Each hut requires
three girls—­one to work the press, and
two to wrap up the cartridges. The cartridge press
consists of a short cylinder of the diameter of the
cartridge that it is intended to make. Into this
cylinder a piston, pointed with ivory or lignum vitae
wood, works up and down from a spring worked by a lever.
Round the upper edge of the cylinder is fastened a
canvas bag, into which the powdery dynamite is placed
by means of a wooden scoop, and the descending piston
forces the dynamite down the cylinder and out of the
open end, where the compressed dynamite can be broken
off at convenient lengths. The whole machine
should be made of gun-metal, and should be upright
against the wall of the building. The two girls,
who sit at tables placed on each side of the press,
wrap the cartridges in parchment paper. From these
huts the cartridges are collected by boys every ten
minutes or a quarter of an hour, and taken to the
packing room, where they are packed in 5-lb. cardboard
boxes, which are then further packed in deal boxes
lined with indiarubber, and fastened down air tight.
The wooden lids are then nailed down with brass or
zinc nails, and a label pasted on the outside giving
the weight and description of the contents. The
boxes should then be removed to the magazines.
It is well to take a certain number of cartridges
from the packing house at different times during the
day, say three or four samples, and to test them by
the heat test. A sample cut from a cartridge,
about 1 inch long, should be placed under a glass shade,
together with water (a large desiccator, in fact),
and left for some days. A good dynamite should
not, under these conditions, show any signs of exudation,
even after weeks.[A]

[Footnote A: For analysis of dynamite, see chapter
on “Analysis,” and author’s article
in *Chem. News*, 23rd September 1892.]
 *Properties of Kieselguhr Dynamite.*—­One
cubic foot of dynamite weighs 76 lbs. 4 oz. The
specific gravity of 75 per cent. dynamite is, however,
1.50. It is a red or grey colour, and rather greasy
to the touch. It is much less sensitive to shock
than nitro-glycerine, but explodes occasionally with
the shock of a rifle bullet, or when struck. The
addition of a few per cent. of camphor will considerably
diminish its explosive qualities to such an extent
that it can be made non-explosive except to a very

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strong fulminate detonator. The direct contact
of water disintegrates dynamite, separating the nitro-glycerine,
hence great caution is necessary in using it in wet
places. It freezes at about 40 deg. Fahr.
(4 deg. C.), and remains frozen at temperatures
considerably exceeding that point. When frozen,
it is comparatively useless as an explosive agent,
and must be thawed with care. This is best done
by placing the cartridges in a warming pan, which
consists of a tin can, with double sides and bottom,
into which hot water (130 deg. Fahr.) can be poured.
The dynamite will require to be left in for some considerable
time before it becomes soft. On no account must
it be placed on a hot stove or near a fire, as many
serious accidents have occurred in this way.

Frozen dynamite is a hard mass, with altered properties,
and requires 1.5 grm. of fulminate instead of 0.5
grm. to explode it. Thawing may also cause exudation
of the nitro-glycerine, which is much more sensitive
to shock, and if accidentally struck with an iron
tool, may explode. It is a dangerous thing to
cut a frozen cartridge with a knife. Ramming is
even more dangerous; in fact it is not only dangerous,
but wasteful, to use dynamite when in a frozen state.

Dynamite explodes at a temperature of 360 deg.
Fahr., and is very sensitive to friction when hot.
In hot countries it should never be exposed to the
rays of the sun. It should, however, not be kept
in a damp or moist place, as this is liable to cause
exudation. Sunlight, if direct, can cause a slow
decomposition, as with all nitro and nitric compounds.
Electric sparks ignite, without exploding it, at least
when operating in the open air.

Dynamite, when made with neutral nitro-glycerine,
appears to keep indefinitely. Sodium or calcium
carbonate to the extent of 1 per cent. is often added
to dynamite to ensure its being neutral. If it
has commenced to undergo change, however, it rapidly
becomes acid, and sometimes explodes spontaneously,
especially if contained in resisting envelopes.
Nevertheless, neutral and well-made dynamite has been
kept for years in a magazine without loss of its explosive
force. If water is brought into contact with
it, the nitro-glycerine is gradually displaced from
the silica (guhr). This action tends to render
all wet dynamite dangerous.

It has been observed that a dynamite made with wood
sawdust can be moistened and then dried without marked
alteration, and from 15 to 20 per cent. of water may
be added to cellulose dynamite without depriving it
of the power of exploding by strong detonator (this
is similar to wet gun-cotton). It is, however,
rendered much less sensitive to shock. With regard
to the power of No. 1 dynamite, experiments made in
lead cylinders give the relative value of No. 1 dynamite,
1.0; blasting gelatine, 1.4; and nitro-glycerine,
1.4. The heat liberated by the sudden explosion
of dynamite is the same as its heat of combustion,[A]
and proportionate to the weight of nitro-glycerine
contained in the mixture. The gases formed are
carbonic acid, water, nitrogen, and oxygen.

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[Footnote A: Berthelot, “Explosives and
their Power.”]

The “explosive wave” (of Berthelot) for
dynamite is about 5,000 metres per second. At
this rate the explosion of a cartridge a foot long
would only occupy 1/24000 part of a second, while
a ton of dynamite cartridges about 7/8 diameter, laid
end to end, and measuring one mile in length, would
be exploded in one-quarter of a second by detonating
a cartridge at either end.[A] Mr C. Napier Hake, F.I.C.,
the Inspector of Explosives for the Victorian Government,
in his paper, “Notes on Explosives,” says:
“The theoretical efficiency of an explosive
cannot in practice be realised in useful work for
several reasons, as for instance in blasting rock—­

“1. Incomplete combustion.

“2. Compression and chemical changes induced
in surrounding material.

“3. Energy expended in cracking and heating
of the material which is not displaced.

“4. The escape of gas through the blast-hole
and the fissures caused by the explosion.

“The useful work consists partly in displacing
the shattered masses. The proportion of useful
work obtainable has been variously estimated at from
14 to 33 per cent. of the theoretical maximum potential.”

[Footnote A: C.N. Hake, “Notes on
Explosives,” *Jour. Soc. Chem.
Ind.*, 1889.]

Among the various forms of dynamite that are manufactured
is carbo-dynamite, the invention of Messrs Walter
F. Reid and W.D. Borland. The base is nitro-glycerine,
and the absorbent is carbon in the form of burnt cork.
It is as cheap as ordinary dynamite, and has greater
explosive force, seeing that 90 per cent. of the mixture
is pure nitro-glycerine, and the absorbent itself
is highly combustible. It is also claimed that
if this dynamite becomes wet, no exudation takes place.

Atlas powder is a dynamite, chiefly manufactured in
America at the Repanno Chemical Works, Philadelphia.
It is a composition of nitro-glycerine, wood-pulp,
nitrate of soda, and carbonate of magnesia. This
was the explosive used in the outrages committed in
London, by the so-called “dynamiters.”
Different varieties contain from 20 to 75 per cent.
of nitro-glycerine.

The Rhenish dynamite, considerably used in the mines
of Cornwall, is composed of 70 parts of a solution
of 2 to 3 per cent. of naphthalene in nitro-glycerine,
3 parts of chalk, 7 parts of sulphate of barium, and
20 of kieselguhr.

Kieselguhr dynamites are being largely given up in
favour of gelatine explosives. The late Colonel
Cundill, in his “Dictionary of Explosives,”
gives a list of about 125 kinds of dynamites.
Many of these, however, are not manufactured.
Among the best known after the ordinary No. 1 dynamite
are forcite, ammonia dynamite, litho-fracteur, rendock,
Atlas powder, giant powder, and the various explosive
gelatines. They all contain nitro-glycerine,
mixed with a variety of other substances, such as absorbent
earths, wood-pulp, nitro-cotton, carbon in some form
or other, nitro-benzol, paraffin, sulphur, nitrates,
or chlorates, &c. &c.

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*Blasting Gelatine and Gelatine Dynamite.*—­The
gelatine explosives chiefly in use are known under
the names of blasting gelatine, gelatine dynamite,
and gelignite. They all consist of the variety
of nitro-cellulose known as collodion-cotton, *i.e*.,
a mixture of the penta- and tetra-nitrates dissolved
in nitro-glycerine, and made up with various proportions
of wood-pulp, and some nitrate, or other material of
a similar nature. As the gun-cotton contains
too little oxygen for complete combustion, and the
nitro-glycerine an excess, a mixture of the two substances
is very beneficial.

Blasting gelatine consists of collodion-cotton and
nitro-glycerine without any other substance, and was
patented by Mr Alfred Nobel in 1875. It is a
clear, semi-transparent, jelly-like substance, of a
specific gravity of 1.5 to 1.55, slightly elastic,
resembling indiarubber, and generally consists of
92 per cent. to 93 per cent. of nitro-glycerine, and
7 to 8 per cent. of nitro-cotton. The cotton
from which it is made should be of good quality.
The following is the analysis of a sample of nitro-cellulose
which made very good gelatine:-

Soluble cotton 99.118 per cent.
Gun-cotton 0.642 "
Non-nitrated cotton 0.240 "
Nitrogen 11.64 "
Total ash 0.25 "

The soluble cotton, which is a mixture of the tetra-
and penta-nitrates, is soluble in ether-alcohol, and
also in nitro-glycerine, and many other solvents,
whereas the hexa-nitrate (gun-cotton), C\_{12}H\_{14}O\_{4}(ONO\_{2})\_{6},
is not soluble in the above liquids, although it is
soluble in acetone or acetic ether. It is very
essential, therefore, that the nitro-cotton used in
the manufacture of the gelatine explosives should
be as free as possible from gun-cotton, otherwise little
lumps of undissolved nitro-cotton will be left in the
finished gelatine. The non-nitrated or unconverted
cotton should also be very low, in fact considerably
under 1/2 per cent.

The nitro-cotton and the nitro-glycerine used should
always be tested before use by the heat test, because
if they do not separately stand this test, it cannot
be expected that the gelatine made from them will do
so. It often occurs, however, that although both
the ingredients stand this test separately before
being mixed, that after the process of manufacture
one or other or both fail to do so.

The nitro-cotton most suitable for gelatine making
is that which has been finely pulped. If it is
not already fine enough, it must be passed through
a fine brass wire sieve. It will be found that
it requires to be rubbed through by hand, and will
not go through at all if in the least degree damp.
It is better, therefore, to dry it first. The
percentage of nitrogen in the nitrated cotton should
be over 11 per cent. It should be as free as
possible from sand or grit, and should give but little
ash upon ignition, not more than 0.25 per cent.

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The cotton, which is generally packed wet in zinc-lined
wooden boxes, will require to be dried, as it is very
essential indeed that none of the materials used in
the manufacture of gelatine should contain more than
the slightest trace of water. If they do, the
gelatine subsequently made from them will most certainly
exude, and become dangerous and comparatively valueless.
It will also be much more difficult to make the nitro-cotton
dissolve in the nitro-glycerine if either contains
water.

In order to find out how long any sample of cotton
requires to be dried, a sample should be taken from
the centre of several boxes, well mixed, and about
1,000 grms. spread out on a paper tray, weighed, and
the whole then placed in the water oven at 100 deg.
C., and dried for an hour or so, and again weighed,
and the percentage of moisture calculated from the
loss in weight. This will be a guide to the time
that the cotton will probably require to be in the
drying house. Samples generally contain from 20
to 30 per cent. of water. After drying for a
period of forty-eight hours, a sample should be again
dried in the oven at 100 deg. C., and the moisture
determined, and so on at intervals until the bulk of
the cotton is found to be dry, *i.e*., to contain
from 0.25 to 0.5 per cent. of moisture. It is
then ready to be sifted. During the process of
removing to the sifting house and the sifting itself,
the cotton should be exposed to the air as little
as possible, as dry nitro-cotton absorbs as much as
2 per cent. of moisture from the air at ordinary temperatures
and average dryness.

The drying house usually consists of a wooden building,
the inside of which is fitted with shelves, or rather
framework to contain drawers, made of wood, with brass
or copper wire netting bottoms. A current of hot
air is made to pass through the shelves and over the
surface of the cotton, which is spread out upon them
to the depth of about 2 inches. This current
of air can be obtained in any way that may be found
convenient, such as by means of a fan or Root’s
blower, the air being passed over hot bricks, or hot-water
pipes before entering the building. The cotton
should also be occasionally turned over by hand in
order that a fresh surface may be continually exposed
to the action of the hot air. The building itself
may be heated by means of hot-water pipes, but on
no account should any of the pipes be exposed.
They should all be most carefully covered over with
wood-work, because when the dry nitro-cotton is moved,
as in turning it over, very fine particles get into
the air, and gradually settling on the pipes, window
ledges, &c., may become very hot, when the slightest
friction might cause explosion. It is on this
account that this house should be very carefully swept
out every day. It is also very desirable that
the floor of this house should be covered with oilcloth
or linoleum, as being soft, it lessens the friction.

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List shoes should always be worn in this building,
and a thermometer hung up somewhere about the centre
of the house, and one should also be kept in one of
the trays to give the temperature of the cotton, especially
the bottom of the trays. The one nearest to the
hot air inlet should be selected. If the temperature
of the house is kept at about 40 deg. C. it will
be quite high enough. The building must of course
be properly ventilated, and it will be found very
useful to have the walls made double, and the intervening
space filled with cinders, and the roof covered with
felt, as this helps to prevent the loss of heat through
radiation, and to preserve a uniform temperature,
which is very desirable.

The dry cotton thus obtained, if not already fine
enough, should be sifted through a brass sieve, and
packed away ready for use in zinc air-tight cases,
or in indiarubber bags. The various gelatine compounds,
gelignite, gelatine dynamite, and blasting gelatine,
are manufactured in exactly the same way. The
forms known as gelatine dynamite differ from blasting
gelatine in containing certain proportions of wood-pulp
and potassium nitrate, &c. The following are
analyses of some typical samples of the three compounds:—­

Gelatine Blasting
Gelignite. Dynamite. Gelatine.

Nitro-glycerine 60.514 71.128 92.94 per
cent.
Nitro-cellulose 4.888 7.632 7.06
 "
Wood-pulp 7.178 4.259 ...
 "
Potassium nitrate 27.420 16.720 ...
 "
Water ... 0.261 ...
 "

The gelignite and gelatine dynamites consist, therefore,
of blasting gelatine, thickened up with a mixture
of absorbing materials. Although the blasting
gelatine is weight for weight more powerful, it is
more difficult to make than either of the other two
compounds, it being somewhat difficult to make it
stand the exudation and melting tests. The higher
percentage of nitro-cotton, too, makes it expensive.

When the dry nitro-cotton, which has been carefully
weighed out in the proportions necessary either for
blasting gelatine or any of the other gelatine explosives,
is brought to the gelatine making house, it is placed
in a lead-lined trough, and the necessary quantity
of pure dry nitro-glycerine poured upon it.
The whole is then well stirred up, and kept at a temperature
of from 40 deg. to 45 deg. C. It should not be
allowed to go much above 40 deg. C.; but higher
temperatures may be used if the nitro-cotton is very
obstinate,[A] and will not dissolve. Great caution
must, however, be observed in this case. The
mixture should be constantly worked about by the workman
with a wooden paddle for at least half an hour.
At a temperature of 40 deg. to 45 deg. the nitro-glycerine
acts upon the nitro-cotton and forms a jelly.
Without heat the gelatinisation is very imperfect
indeed, and at temperatures under 40 deg. C. takes
place very slowly.

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[Footnote A: Generally due to the nitro-cotton
being damp.]

[Illustration: FIG. 30.—­WERNER, PFLEIDERER,
& PERKINS’ MIXING MACHINE.]

The limit of temperature is 50 deg. C. or thereabouts.
Beyond this the jelly should never be allowed to go,
and to 50 deg. only under exceptional circumstances.

The tank in which the jelly is made is double-lined,
in order to allow of the passage of hot water between
its inner and outer linings. A series of such
tanks are generally built in a wooden framework, and
the double linings are made to communicate, so that
the hot water can flow from one to the other consecutively.
The temperature of the water should be about 60 deg.
C. if it is intended to gelatinise at 45 deg.
C., and about 80 deg. if at 50 deg. C.; but this
point must, of course, be found by experiment for the
particular plant used. An arrangement should be
made to enable the workman to at once cut off the
supply of hot water and pass cold water through the
tanks in case the explosive becomes too hot.

[Illustration: FIG. 31.—­MR M’ROBERTS’
MIXER FOR GELATINE EXPLOSIVES.]

The best way to keep the temperature of the water
constant is to have a large tank of water raised upon
a platform, some 5 or 6 feet high, outside the building,
which is automatically supplied with water, and into
which steam is turned. A thermometer stuck through
a piece of cork and floated upon the surface of the
tank will give the means of regulating the temperature.

When the jelly in the tanks has become semi-transparent
and the cotton has entirely dissolved, the mixture
should be transferred to the mixing machine.
The mixing machines are specially designed for this
work, and are built in iron, with steel or bronze
kneading- and mixing-blades, according to requirements.

A suitable machine for the purpose is that known as
the Nito-Universal Incorporator, shown in Fig. 30,
which has been specially constructed by Messrs Werner,
Pfleiderer, & Perkins, Ltd., after many years’
experience in the mixing of explosive materials, and
is now almost exclusively adopted in both Government
and private factories. Mr George M’Roberts’[A]
mixing machine, however, which is shown in Fig. 31,
is still used in some factories for dynamite jelly.

[Footnote A: See *Jour. Soc. Chem.
Ind.*, 1890, 267.]

If it is intended to make gelignite, or gelatine dynamite,
it is at this point that the proper proportions of
wood-pulp[A] and potassium nitrate should be added,
and the whole well mixed for at least half an hour,
until the various ingredients are thoroughly incorporated.

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[Footnote A: Most of the wood-pulp used in England
is obtained from pine-trees, but poplar, lime, birch,
and beech wood are also used. It is chiefly imported
as wood-pulp. The pulp is prepared as follows:—­The
bark and roots are first removed, and the logs then
sawn into boards, from which the knots are removed.
The pieces of wood are afterwards put through a machine
which breaks them up into small pieces about an inch
long, which are then crushed between rollers.
These fragments are finally boiled with a solution
of sodium bisulphite, under a pressure of about 90
lbs. per square inch, the duration of the boiling
being from ten to twelve hours. Sulphurous acid
has also been used. Pine-wood yields about 45
per cent. and birch about 40 per cent. of pulp when
treated by this process. The pulp is afterwards
bleached and washed, &c.

Birch. Beech. Lime. Pine. Poplar.
Cellulose 55.52 45.47 53.09 56.99 62.77 per cent.
Resin 1.14 0.41 3.93 0.97 1.37 "
Aqueous extract 2.65 2.47 3.56 1.26 2.88 "
Water 12.48 12.57 10.10 13.87 12.10 "
Lignine 28.21 39.14 29.32 26.91 20.88 “]

The following analysis of woods is by Dr H. Mueller:—­These
mixing machines can either be turned by hand, or a
shaft can be brought into the house and the machine
worked by means of a belt at twenty to thirty revolutions
per minute. The bearings should be kept constantly
greased and examined, and the explosive mixture carefully
excluded. When the gelatine mixture has been
thoroughly incorporated, and neither particles of nitrate
or wood meal can be detected in the mass, it should
be transferred to wooden boxes and carried away to
the cartridge-making machines to be worked up into
cartridges.

[Illustration: FIG. 32.—­PLAN OF THE
BOX CONTAINING THE EXPLOSIVE, IN M’ROBERTS’
MACHINE.]

The application of heat in the manufacture of the
jelly from collodion-cotton and nitro-glycerine is
absolutely necessary, unless some other solvent is
used besides the nitro-glycerine, such as acetone,
acetic ether, methyl, or ethyl-alcohol. (They are
all too expensive, with the exception of acetone and
methyl-alcohol, for use upon the large scale.) These
liquids not only dissolve the nitro-cellulose in the
cold, but render the resulting gelatine compound less
sensitive to concussion, and reduce its quickness
of explosion (as in cordite). They also lower
the temperature at which the nitro-glycerine becomes
congealed, *i.e*., they lower the freezing point[A]
of the resulting gelatine.

[Footnote A: It has been proposed to mix dynamite
with amyl alcohol for this purpose. Di-nitro-mono-chlorhydrine
has also been proposed.]

The finished gelatine paste, upon entering the cartridge
huts, is at once transferred to the cartridge-making
machine, which is very like an ordinary sausage-making
machine[A] (Fig. 33). The whole thing must be
made of gun-metal or brass, and it consists of a conical
case containing a shaft and screw. The revolutions
of the shaft cause the thread of the screw to push
forward the gelatine introduced by the hopper on the
top to the nozzle, the apex of the cone-shaped case,
from whence the gelatine issues as a continuous rope.
The nozzle is of course of a diameter according to
the size of cartridge required.

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[Footnote A: G. M’Roberts, *Jour.
Soc. Chem. Ind.*, 31st March 1890, p.
266.]

[Illustration: FIG. 33.—­CARTRIDGE-MAKING
MACHINE FOR GELATINE EXPLOSIVES.]

The issuing gelatine can of course be cut off at any
length. This is best done with a piece of hard
wood planed down to a cutting edge, *i.e*., wedge-shaped.
Mr Trench has devised a kind of brass frame, into which
the gelatine issuing from the nozzle of the cartridge
machine is forced, finding its way along a series
of grooves. When the frame is full, a wooden
frame, which is hinged to one end of the bottom frame,
and fitted with a series of brass knives, is shut
down, thereby cutting the gelatine up into lengths
of about 4 inches.

It is essential that the cartridge machines should
have no metallic contacts inside. The bearing
for the screw shaft must be fixed outside the cone
containing the gelatine. One of these machines
can convert from 5 to 10 cwt. of gelatine into cartridges
per diem, depending upon the diameter of the cartridges
made.

After being cut up into lengths of about 3 inches,
the gelatine is rolled up in cartridge paper.
Waterproof paper is generally used. The cartridges
are then packed away in cardboard boxes, which are
again packed in deal boxes lined with indiarubber,
and screwed down air tight, brass screws or zinc or
brass nails being used for the purpose. These
boxes are sent to the magazines. Before the boxes
are fastened down a cartridge or so should be removed
and tested by the heat test, the liquefaction test,
and the test for liability to exudation. (Appendix,
p. 6, Explosives Act, 1875.) A cartridge also should
be stored in the magazine in case of any subsequent
dispute after the bulk of the material has left the
factory.

The object of the liquefaction test is to ensure that
the gelatine shall be able to withstand a fairly high
temperature (such as it might encounter in a ship’s
hold) without melting or running together. The
test is carried out as follows:—­A cylinder
of the gelatine dynamite is cut from the cartridge
of a length equal to its diameter. The edges must
be sharp. This cylinder is to be placed on end
on a flat surface (such as paper), and secured by
a pin through the centre, and exposed for 144 consecutive
hours to a temperature of 85 deg. to 90 deg.
F., and during such time the cylinder should not diminish
in height by more than one-fourth of an inch, and the
cut edges should remain sharp. There should also
be no stain of nitroglycerine upon the paper.

The exudation test consists in freezing and thawing
the gelatine three times in succession. Under
these conditions there should be no exudation of nitro-glycerine.
All the materials used in the manufacture of gelatine
explosives should be subjected to analytical examination
before use, as success largely depends upon the purity
of the raw materials. The wood-pulp, for instance,
must be examined for acidity.

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*Properties of the Gelatine Compounds.*—­Blasting
gelatine is generally composed of 93 to 95 parts nitro-glycerine,
and 5 to 7 parts of nitro-cellulose, but the relative
proportions of explosive base and nitro-glycerine,
&c., in the various forms of the gelatine explosives
do not always correspond to those necessary for total
combustion, either because an incomplete combustion
gives rise to a greater volume of gas, or because
the rapidity of decomposition and the law of expansion
varies according to the relative proportions and the
conditions of application. The various additions
to blasting gelatine generally have the effect of lowering
the strength by reducing the amount of nitro-glycerine,
but this is sometimes done in order to change a shattering
agent into a propulsive force. If this process
be carried too far, we of course lose the advantages
due to the presence of nitro-glycerine. There
is therefore a limit to these additions.[A]

[Footnote A: Mica is said to increase the rapidity
of explosion when mixed with gelatine.]

The homogeneousness and stability of the mixture are
of the highest importance. It is highly essential
that the nitro-glycerine should be completely absorbed
by the substances with which it is mixed, and that
it should not subsequently exude when subjected to
heat or damp. It is also important that there
should be no excess of nitro-glycerine, as this may
diminish instead of augment the strength, owing to
a difference in the mode of the propagation of the
explosive wave in the liquid, and in the mixture.
Nitro-glycerine at its freezing point has a tendency
to separate from its absorbing material, in fact to
exude. When frozen, too, it requires a more powerful
detonation to explode it, but it is less sensitive
to shock. The specific gravity of blasting gelatine
is 1.5 (i.e., nearly equal to that of nitro-glycerol);
that of gun-cotton (dry) is 1.0.

Blasting gelatine burns in the air when unconfined
without explosion, at least in small quantities and
when not previously heated, but it is rather uncertain
in this respect. It can be kept at a moderately
high temperature (70 deg. C.) without decomposition.
At higher temperatures the nitro-glycerine will partially
evaporate. When slowly heated, it explodes at
204 deg. C. If, however, it contains as much
as 10 per cent. of camphor, it burns without exploding.
According to Berthelot,[A] gelatine composed of 91.6
per cent. nitro-glycerine and 8.4 per cent. of nitro-cellulose,
which are the proportions corresponding to total combustion,
produces by explosion 177CO\_{2}+ 143H\_{2}O + 8N\_{2}.

[Footnote A: Berthelot, “Explosives and
their Powers.”]

He takes C\_{24}H\_{22}(NO\_{3}H)\_{9}O\_{11} as the formula
of the nitro-cellulose, and 51C\_{3}H\_{2}(NO\_{3}H)\_{3}
+ C\_{24}H\_{22}(NO\_{3}H)\_{9}O\_{11} as the formula of
the gelatine itself, its equivalent weight being 12,360
grms. The heat liberated by its explosion is equal
to 19,381 calories, or for 1 kilo. 1,535 calories.
Volume of gases reduced temperature equals 8,950 litres.
The relative value[A] of blasting gelatine to nitro-glycerine
is as 1.4 to 1.45, kieselguhr dynamite being taken
as 1.0.

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[Footnote A: Roux and Sarran.]

**CHAPTER V.**

*NITRO-BENZOL, ROBURITE, BELLITE, PICRIC ACID, &c.*
Explosives derived from Benzene—­Toluene
and Nitro-Benzene—­Di- and
Tri-nitro-Benzene—­Roburite: Properties
and Manufacture—­Bellite:
Properties, &c.—­Securite—­Tonite
No. 3.—­Nitro-Toluene—­
Nitro-Naphthalene—­Ammonite—­Sprengel’s
Explosives—­Picric Acid—­
Picrates—­Picric Powders—­Melinite—­Abel’s
Mixture—­Brugere’s Powders—­
The Fulminates—­Composition, Formula, Preparation,
Danger of, &c.—­
Detonators: Sizes, Composition, Manufacture—­Fuses,
&c.
 *The Explosives derived from Benzene.*—­There
is a large class of explosives made from the nitrated
hydro-carbons—­benzene, C\_{6}H\_{6}; toluene,
C\_{7}H\_{8}; naphthalene, C\_{10}H\_{8}; and also from
phenol (or carbolic acid), C\_{6}H\_{5}OH. The
benzene hydro-carbons are generally colourless liquids,
insoluble in water, but soluble in alcohol and ether.
They generally distil without decomposition. They
burn with a smoky flame, and have an ethereal odour.
They are easily nitrated and sulphurated; mono, di,
and tri derivatives are readily prepared, according
to the strength of the acids used. It is only
the H-atoms of the benzene nucleus which enter into
reaction.

Benzene was discovered by Faraday in 1825, and detected
in coal-tar by Hofmann in 1845. It can be obtained
from that portion of coal-tar which boils at 80 deg.
to 85 deg. by fractionating or freezing.[A] The ordinary
benzene of commerce contains thiophene (C\_{4}H\_{4}S),
from which it may be freed by shaking with sulphuric
acid. Its boiling point is 79 deg. C.; specific
gravity at 0 deg. equals 0.9. It burns with a
luminous smoky flame, and is a good solvent for fats,
resins, sulphur, phosphorus, &c. Toluene was
discovered in 1837, and is prepared from coal-tar.
It boils at 110 deg. C., and is still liquid
at 28 deg. C.

[Footnote A: It may be prepared chemically pure
by distilling a mixture of benzoic acid and lime.]

The mono-, chloro-, bromo-, and iodo-benzenes are
colourless liquids of peculiar odour. Di-chloro-,
di-bromo-benzenes, tri- and hexa-chloro- and bromo-benzenes,
are also known; and mono-chloro-, C\_{6}H\_{4}Cl(CH\_{3}),
and bromo-toluenes, together with di derivatives in
the ortho, meta, and para modifications. The
nitro-benzenes and toluenes are used as explosives.
The following summary is taken from Dr A. Bernthsen’s
“Organic Chemistry":—­

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SUMMARY.
\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_
\_\_\_\_\_\_\_\_
| |
| C\_{6}H\_{5}(N0\_{2}) Nitro-benzene. Liq. B.Pt. 206 deg. C. |
| |
| C\_{6}H\_{4}(NO\_{2})\_{2} Ortho-, meta-, and para- di-nitro-benzenes. |
| Solid. M.P. 118 deg., 90 deg., and 172 deg. C. |
| |
| C\_{6}H\_{3}(NO\_{3})\_{3} S.-Tri-nitro-benzene. Solid. M.P. 121 deg. C. |
|\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_
\_\_\_\_\_\_\_\_\_|
| |
| C\_{6}H\_{4}(CH\_{3})NO\_{2} Ortho-, meta-, and para- nitro-toluenes. |
| B.P. 218 deg., 230 deg., and 234 deg. C, Para compound solid. |
|\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_
\_\_\_\_\_\_\_\_\_|
| |
| C\_{6}H\_{3}(CH\_{3})\_{2}NO\_{2} Nitro-xylene. Liquid. |
|\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_
\_\_\_\_\_\_\_\_\_|
| |
| C\_{6}H\_{2}(CH\_{3})\_{3}NO\_{2} Nitro-mesitylene. Solid. |
|\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_
\_\_\_\_\_\_\_\_\_|
| |
| C\_{6}H\_{3}(CH\_{3})(NO\_{2})\_{2} Di-nitro-toluenes. |
|\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_
\_\_\_\_\_\_\_\_\_|
| |
| C\_{6}H\_{4}Cl(NO\_{2}) Nitro-chloro-benzenes. |
| |
| C\_{6}Br\_{4}(NO\_{2})\_{2} Tetra-bromo-di-nitrobenzene. |
|\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_
\_\_\_\_\_\_\_\_\_|

The nitro compounds are mostly pale yellow liquids,
which distil unchanged, and volatilise with water
vapour, or colourless or pale yellow needles or prisms.
Some of them, however, are of an intense yellow colour.
Many of them explode upon being heated. They are
heavier than water, and insoluble in it, but mostly
soluble in alcohol, ether, and glacial acetic acid.

Nitro-benzene, C\_{6}H\_{5}(NO\_{2}), was discovered
in 1834 by Mitscherlich. It is a yellow liquid,
with a melting point of +3 deg. C. It has an intense
odour of bitter almonds. It solidifies in the
cold. In di-nitro-benzene, the two nitro groups
may be in the meta, ortho, or para position, the meta
position being the most general (see fig., page 4).
By recrystallising from alcohol, pure meta-di-nitro-benzene
may be obtained in long colourless needles. The
ortho compound crystallises in tables, and the para
in needles. They are both colourless. When
toluene is nitrated, the para and ortho are chiefly
formed, and a very little of the meta compound.

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*Nitro Compounds of Benzene and Toluene.*—­The
preparation of the nitro derivatives of the hydrocarbons
of the benzene series is very simple. It is only
necessary to bring the hydrocarbon into contact with
strong nitric acid, when the reaction takes place,
and one or more of the hydrogen atoms of the hydrocarbon
are replaced by the nitryl group (NO\_{2}). Thus
by the action of nitric acid on benzene (or benzol),
mono-nitro-benzene is formed:—­

C\_{6}H\_{6} + HNO\_{3} = C\_{6}H\_{5}.NO\_{2} +H\_{2}O.
 Mono-nitro-benzene.

By the action of another molecule of nitric acid,
the di-nitro-benzene is formed:—­

C\_{6}H\_{5}.NO\_{2} + HNO\_{3} = C\_{6}H\_{4}(NO\_{2})\_{2}
+ H\_{2}O.
 Di-nitro-benzene.

These nitro bodies are not acids, nor are they ethereal
salts of nitrous acid, as nitro-glycerine is of glycerine.
They are regarded as formed from nitric acid by the
replacement of hydroxyl by benzene radicals.
 *Mono-nitro Benzene* is made by treating benzene
with concentrated nitric acid, or a mixture of nitric
and sulphuric acids. The latter, as in the case
of the nitration of glycerine, takes no part in the
reaction, but only prevents the dilution of the nitric
acid by the water formed in the reaction. Small
quantities may be made thus:—­Take 150 c.c.
of H\_{2}SO\_{4} and 75 c.c. HNO\_{3}, or 1 part
nitric to 2 parts sulphuric acid, and put in a beaker
standing in cold water; then add 15 to 20 c.c. of benzene,
drop by drop, waiting between each addition for the
completion of the reaction, and shake well during
the operation. When finished, pour contents of
beaker into about a litre of cold water; the nitro-benzol
will sink to the bottom. Decant the water, and
wash the nitro-benzol two or three times in a separating
funnel with water. Finally, dry the product by
adding a little granulated calcium chloride, and allowing
to stand for some little time, it may then be distilled.
Nitro-benzene is a heavy oily liquid which boils at
205 deg. C., has a specific gravity of 1.2, and
an odour like that of oil of bitter almonds.
In the arts it is chiefly used in the preparation
of aniline.
 *Di-nitro Benzene* is a product of the further
action of nitric acid on benzene or nitro-benzene.
It crystallises in long fine needles or thin rhombic
plates, and melts at 89.9 deg. C. It can be made
thus:—­The acid mixture used consists of
equal parts of nitric and sulphuric acids, say 50
c.c. of each, and without cooling add very slowly 10
c.c. of benzene from a pipette. After the action
is over, boil the mixture for a short time, then pour
into about half a litre of water, filter off the crystals
thus produced, press between layers of filter paper,
and crystallise from alcohol. Di-nitro-benzene,
or meta-di-nitro-benzene, as it is sometimes called,
enters into the composition of several explosives,
such as tonite No. 3, roburite, securite, bellite.

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Nitro-benzene is manufactured upon the large scale
as follows:—­Along a bench a row of glass
flasks, containing 1 gallon each (1 to 2 lbs. benzene),
are placed, and the acids added in small portions at
a time, the workmen commencing with the first, and
adding a small quantity to each in turn, until the
nitration was complete. This process was a dangerous
one, and is now obsolete. The first nitro-benzene
made commercially in England, by Messrs Simpson, Maule,
and Nicholson, of Kennington, in 1856, was by this
process. Now, however, vertical iron cylinders,
made of cast-iron, are used for the nitrating operation.
They are about 4 feet in diameter and 4 feet deep,
and a series are generally arranged in a row, at a
convenient height from the ground, beneath a line of
shafting. Each cylinder is covered with a cast-iron
lid having a raised rim all round. A central
orifice gives passage to a vertical shaft, and two
or more other conveniently arranged openings allow
the benzene and the mixed acids to flow in. Each
of these openings is surrounded with a deep rim, so
that the whole top of the cylinder can be flooded
with water some inches in depth, without any of it
running into the interior of the nitrator. The
lid overhangs the cylinder somewhat, and in the outer
rim a number of shot-holes or tubes allow the water
to flow down all over the outside of the cylinder
into a shallow cast-iron dish, in which it stands.
By means of a good supply of cold water, the top,
sides, and bottom of the whole apparatus is thus cooled
and continually flooded. The agitator consists
of cast-iron arms keyed to a vertical shaft, with
fixed arms or dash-plates secured to the sides of
the cylinder. The shaft has a mitre wheel keyed
on the top, which works into a corresponding wheel
on the horizontal shafting running along the top of
the converters. This latter is secured to a clutch;
and there is a feather on the shaft, so that any one
of the converters can if necessary be put either in
or out of gear. This arrangement is necessary,
as riggers or belts of leather, cotton, or indiarubber
will not stand the atmosphere of the nitro-benzole
house. Above and close to each nitrator stands
its acid store tank, of iron or stoneware.

The building in which the nitration is carried out
should consist of one story, have a light roof, walls
of hard brick, and a concrete floor of 9 to 12 inches
thick, and covered with pitch, to protect its surface
from the action of the acids. The floor should
be inclined to a drain, to save any nitro-benzol spilt.
Fire hydrants should be placed at convenient places,
and it should be possible to at once fill the building
with steam. A 2-inch pipe, with a cock outside
the building, is advisable. The building should
also be as far as possible isolated.

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The acids are mixed beforehand, and allowed to cool
before use. The nitric acid used has a specific
gravity of 1.388, and should be as free as possible
from the lower oxides of nitrogen. The sulphuric
acid has a specific gravity of 1.845, and contains
from 95 to 96 per cent. of mono-hydrate. A good
mixture is 100 parts of nitric to 140 parts of sulphuric
acid, and 78 parts of benzene; or 128 parts HNO\_{3},
179 of H\_{2}SO\_{4}, and 100 of benzene (C\_{6}H\_{6}).
The benzene having been introduced into the cylinder,
the water is turned on and the apparatus cooled, the
agitators are set running, and the acid cock turned
on so as to allow it to flow in a very thin stream
into the nitrator.

Should it be necessary to check the machinery even
for a moment, the stream of acid must be stopped and
the agitation continued for some time, as the action
proceeds with such vigour that if the benzene being
nitrated comes to rest and acid continues to flow,
local heating occurs, and the mixture may inflame.
Accidents from this cause have been not infrequent.
The operation requires between eight to ten hours,
agitation and cooling being kept up all the time.
When all the acid is added the water is shut off,
and the temperature allowed to rise a little, to about
100 deg. C. When it ceases to rise the agitators
are thrown out of gear, and the mixture allowed some
hours to cool and settle. The acid is then drawn
off, and the nitro-benzene is well washed with water,
and sometimes distilled with wet steam, to recover
a little unconverted benzene and a trace of paraffin
(about .5 per cent. together). At many English
works, 100 to 200 gallons, or 800 to 1,760 lbs., are
nitrated at a time, and toluene is often used instead
of benzene, especially if the nitro-benzene is for
use as essence of myrbane. The waste acids, specific
gravity 1.6 to 1.7, contain a little nitro-benzene
in solution and some oxalic acid. They are concentrated
in cast-iron pots and used over again.
 *Di-nitro Benzene* is obtained by treating a
charge of the hydrocarbon benzene with double the
quantity of mixed acids in two operations, or rather
in two stages, the second lot of acid being run in
directly after the first. The cooling water is
then shut off, and the temperature allowed to rise
rapidly, or nitro-benzene already manufactured is taken
and again nitrated with acids. A large quantity
of acid fumes come off, and some of the nitro- and
di-nitro-benzol produced comes off at the high temperature
which is attained, and a good condensing apparatus
of stoneware must be used to prevent loss. The
product is separated from the acids, washed with cold
water and then with hot. It is slightly soluble
in water, so that the washing waters must be kept
and used over again. Finally it is allowed to
settle, and run while still warm into iron trays, in
which it solidifies in masses 2 or 4 inches thick.
It should not contain any nitro-benzol, nor soil a
piece of paper when laid on it, should be well crystallised,
fairly hard, and almost odourless. The chief
product is meta-di-nitro-benzene, melting point 89.8,
but ortho-di-nitro-benzene, melting point 118 deg.,
and para-di-nitro, melting point 172 deg., are also
produced. The melting point of the commercial
product is between 85 deg. to 87 deg. C.

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Di-nitro-toluene is made in a similar manner.
The tri-nitro-benzene can only be made by using a
very large excess of the mixed acids. Nitro-benzene,
when reduced with iron, zinc, or tin, and hydrochloric
acids, forms aniline.
 *Roburite.*—­This explosive is the
invention of a German chemist, Dr Carl Roth (English
patent 267A, 1887), and is now manufactured in England,
at Gathurst, near Wigan. It consists of two component
parts, non-explosive in themselves (Sprengel’s
principle), but which, when mixed, form a powerful
explosive. The two substances are ammonium nitrate
and chlorinated di-nitro-benzol. Nitro-naphthalene
is also used. Nitrate of soda and sulphate of
ammonium are allowed to be mixed with it. The
advantages claimed for the introduction of chlorine
into the nitro compound are that chlorine exerts a
loosening effect upon the NO\_{2} groups, and enables
the compound to burn more rapidly than when the nitro
groups alone are present.

The formula of chloro-di-nitro-benzol is C\_{6}H\_{3}Cl(NO\_{2})\_{2}.
The theoretical percentage of nitrogen, therefore,
is 13.82, and of chlorine 17.53. Dr Roth states
that, from experiments he has made, the dynamic effect
is considerably increased by the introduction of chlorine
into the nitro compound. Roburite burns quickly,
and is not sensitive to shock; it must be used dry;
it cannot be made to explode by concussion, pressure,
friction, fire, or lightning; it does not freeze; it
does not give off deleterious fumes, and it is to
all intents and purposes flameless; and when properly
tamped and fired by electricity, can be safely used
in fiery mines, neither fine dust nor gases being
ignited by it. The action is rending and not
pulverising. Compared to gunpowder, it is more
powerful in a ratio ranging from 2-1/2 to 4 to 1,
according to the substance acted upon. It is
largely used in blasting, pit sinking, quarrying, &c.,
but especially in coal mining. According to Dr
Roth, the following is the equation of its decomposition:—­

C\_{6}H\_{3}Cl(NO\_{2})\_{2} + 9HN\_{4}NO\_{3} = 6CO\_{2}
+ 20N + HCl.

In appearance roburite is a brownish yellow powder,
with the characteristic smell of nitro-benzol.
Its specific gravity is 1.40. The Company’s
statement that the fumes of roburite were harmless
having been questioned by the miners of the Garswood
Coal and Iron Works Colliery, a scientific committee
was appointed by the management and the men jointly
for the purpose of settling the question. The
members of this committee were Dr N. Hannah, Dr D.J.
Mouncey, and Professor H.B. Dixon, F.R.S., of
Owens College. After a protracted investigation,
a long and technical report was issued, completely
vindicating the innocuousness of roburite when properly
used. In the words of *The Iron and Coal Trades’
Review* (May 24, 1889), “The verdict, though
not on every point in favour of the use in all circumstances
of roburite in coal mines, is yet of so pronounced
a character in its favour as an explosive that it is
impossible to resist the conclusion that the claims
put forward on its behalf rest on solid grounds.”

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Roburite was also one of the explosives investigated
by the committee appointed in September 1889 by the
Durham Coalowners’ and Miners’ Associations,
for the purpose of determining whether the fumes produced
by certain explosives are injurious to health.
Both owners and workmen were represented on the committee,
which elected Mr T. Bell, H.M. Inspector of Mines,
as its chairman, with Professor P.P. Bedson and
Drs Drummond and Hume as professional advisers.
The problem considered was whether the fumes produced
by the combustion of certain explosives, one of which
was roburite, were injurious to health. The trial
comprised the chemical analysis of the air at the
“intake,” and of the vitiated air during
the firing of the shots at the “return,”
and also of the smoky air in the vicinity of the shot-holes.
Five pounds and a half of roburite were used in twenty-three
shots. It had been asserted that the fumes from
this explosive contained carbon-monoxide, CO, but
no trace of this gas could be discovered after the
explosion. On another occasion, however, when
4.7 lbs. of roburite were exploded in twenty-three
shots, the air at the “return” showed
traces of CO gas to the extent of .042 to .019 per
cent. The medical report which Drs Hume and Drummond
presented to the committee shows that they investigated
every case of suspected illness produced by exposure
to fumes, and they could find no evidence of acute
illness being caused. They say, “No case
of acute illness has, throughout the inquiry, been
brought to our knowledge, and we are led to the conclusion
that such cases have not occurred.”
 *Manufacture.*—­As now made, roburite
is a mixture of ammonium nitrate and chlorinated di-nitro-benzol.
The nitrate of ammonia is first dried and ground,
and then heated in a closed steam-jacketed vessel to
a temperature of 80 deg. C., and the melted organic
compound is added, and the whole stirred until an
intimate mixture is obtained. On cooling, the
yellow powder is ready for use, and is stored in straight
canisters or made up into cartridges. Owing to
the deliquescent nature of the nitrate of ammonia,
the finished explosive must be kept out of contact
with the air, and for this reason the cartridges are
waterproofed by dipping them in melted wax. Roburite
is made in Germany, at Witten, Westphalia; and also
at the English Company’s extensive works at
Gathurst, near Wigan, which have been at work now
for some eighteen years, having started in 1888.
These works are of considerable extent, covering 30
acres of ground, and are equal to an output of 10
tons a day. A canal runs through the centre, separating
the chemical from the explosive portions of the works,
and the Lancashire and Yorkshire Railway runs up to
the doors. Besides sending large quantities of
roburite itself abroad, the Company also export to
the various colonies the two components, as manufactured
in the chemical works, and which separately are quite
non-explosive, and which, having arrived at their
destination, can be easily mixed in the proper proportions.

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Among the special advantages claimed for roburite
are:—­First, that it is impossible to explode
a cartridge by percussion, fire, or electric sparks.
If a cartridge or layer be struck with a heavy hammer,
the portion struck is decomposed, owing to the large
amount of heat developed by the blow. The remaining
explosive is not in the least affected, and no detonation
whatever takes place. If roburite be mixed with
gunpowder, and the gunpowder fired, the explosion
simply scatters the roburite without affecting it
in the least. In fact, the only way to explode
roburite is to detonate it by means of a cap of fulminate,
containing at least 1 gramme of fulminate of mercury.
Secondly, its great safety for use in coal mines.
Roburite has the great advantage of exploding by detonation
at a very low temperature, indeed so low that a very
slight amount of tamping is required when fired in
the most explosive mixture of air and coal gas possible,
and not at all in a mixture of air and coal dust—­a
condition in which the use of gunpowder is highly
dangerous.

Mr W.J. Orsman, F.I.C., in a paper read at the
University College, Nottingham, in 1893, gives the
temperature of detonation of roburite as below 2,100
deg. C., and of ammonium nitrate as 1,130 deg.
C., whereas that of blasting gelatine is as much as
3,220 deg. C. With regard to the composition
of the fumes formed by the explosion of roburite, Mr
Orsman says: “With certain safety explosives—­roburite,
for instance—­an excess of the oxidising
material is added, namely, nitrate of ammonia; but
in this case the excess of oxygen here causes a diminution
of temperature, as the nitrate of ammonia on being
decomposed absorbs heat. This excess of oxygen
effectually prevents the formation of carbon monoxide
(CO) and the oxides of nitrogen.”

The following table (A), also from Mr Orsman’s
paper, gives the composition of five prominent explosives,
and shows the composition of the gases formed on explosion.
The gases were collected after detonating 10 grms.
of each in a closed strong steel cylinder, having an
internal diameter of 5 inches.

With respect to the influence of ammonium nitrate
in lowering the temperature of explosion of the various
substances to which it is added, it was found by a
French Commission that, when dry and finely powdered,
ammonium nitrate succeeds in depreciating the heat
of decomposition without reducing the power of the
explosive below a useful limit. The following
table (B) shows the composition of the explosives examined,
and the temperatures which accompanied their explosion.

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A
\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_
\_\_\_\_\_\_\_\_\_\_
| | | |
| | | Composition of Gases. |
| |Volume |\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_|
| Explosive. |of Gas | | | | |
| |formed.|CO\_{2}.| CO. |CH\_{4}| N. |
| | | | | &H. | |
|\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_|\_\_\_\_\_\_\_|\_\_\_\_\_\_\_|\_\_\_\_\_|\_
\_\_\_\_\_|\_\_\_\_\_|
| | | | | | |
| | | Per | Per | Per | Per |
| | c.c. | cent. |cent.|cent. |cent.|
|Gunpowder-- | | | | | |
| Nitre 75 parts | | | | | |
| Sulphur 10 ‘’ | 2,214 | 51.3 | 3.5| 3.5 | 41.7|
| Charcoal 15 ‘’ | | | | | |
|Gelignite-- | | | | | |
| Nitro-glycerine 56.5 parts | | | | | |
| Nitro-cotton 3.5 ‘’ | 4,980 | 25 | 7 | ... | 67 |
| Wood-meal 8.0 ‘’ | | | | | |
| KNO\_{3} 32.0 ‘’ | | | | | |
|Tonite-- | | | | | |
| Nitro-Cotton | 3,750 | 30 | 8 | ... | 62 |
| Barium nitrate | | | | | |
|Roburite-- | | | | | |
| Ammonium nitrate, 86 parts | | | | | |
| Di-nitro-chloro-benzol 14 ‘’ | 4,780 | 32 | ... | ... | 68 |
|Carbonite | | | | | |
| Nitro-glycerine 25 parts | | | | | |
| Wood-meal 40 ‘’ | 2,100 | 19 | 15 | 26 | ... |
| Potas. nitrate 34 ‘’ | | | | | |
|\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_|\_\_\_\_\_\_\_|\_\_\_\_\_\_\_|\_\_\_\_\_|\_
\_\_\_\_\_|\_\_\_\_\_|

B
\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_
\_\_\_\_\_\_\_\_
| | | | |
| | Original | Percentage | Final |
| Explosive. | Temperature |NH\_{4}.NO\_{3}| Temperature |
| |Co-efficient.| added. |Co-efficient.|
|\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_|\_\_\_\_\_\_\_\_\_\_\_\_\_|\_\_\_\_\_\_\_\_\_\_\_\_\_|\_\_\_\_
\_\_\_\_\_\_\_\_\_|
| | | | |
|Nitro-glycerine | 3,200 | ... | ... |
|Blasting gelatine | | | |
| (8 per cent. gun-cotton)| 3,090 | 88 | 1,493 |
|Dynamite | | | | *Bellite* is the patent of Mr Carl Lamm, Managing
Director of the Roetebro Explosive Company, of Stockholm,
and is licensed for manufacture in England. It
consists of a mixture of nitrate of ammonia with di-
or tri-nitro-benzol, it has a specific gravity of
1.2 to 1.4 in its granulated state, and 1 litre weighs
800 to 875 grms. Heated in an open vessel, bellite
loses its consistency at 90 deg. C., but does
not commence to separate before a temperature of 200
deg. C. is reached, when it evaporates without
exploding. If heated suddenly, it burns with a
sooty flame, somewhat like tar, but if the source
of heat is removed, it will cease burning, and assume
a caramel-like structure. It absorbs very little
moisture from the air after it has been pressed, and
if the operation has been performed while the explosive
is hot, the subsequent increase of weight is only 2
per cent. When subjected to the most powerful
blow with a steel hammer upon an iron plate, it neither
explodes nor ignites. A rifle bullet fired into
it at 50 yards’ distance will not explode it.
Granulated bellite explodes fully by the aid of fulminating
mercury. Fifteen grms. of bellite fired by means
of fulminate, projected a shot from an ordinary mortar,
weighing 90 lbs., a distance of 75 yards, 15 grms.
of gunpowder, under the same conditions, throwing
it only 12 yards. A weight of 7-1/2 lbs. falling
145 centimetres failed to explode 1 grm. of bellite.

Various experiments and trials have been made with
this explosive by Professor P.T. Cleve, M.P.F.
Chalon, C.N. Hake, and by a committee of officers
of the Swedish Royal Artillery. It is claimed
that it is a very powerful and extremely safe explosive;
that it cannot be made to explode by friction, shock,
or pressure, nor by electricity, fire, lightning, &c.,
and that it is specially adapted for use in coal mines,
&c.; that it can only be exploded by means of a fulminate
detonator, and is perfectly safe to handle and manufacture;
that it does not freeze, can be used as a filling
for shells, and lastly, can be cheaply manufactured.
 *Securite* consists of 26 parts of meta-di-nitro-benzol
and 74 parts of ammonium nitrate. It is a yellow
powder, with an odour of nitro-benzol. It was
licensed in 1886. It sometimes contains tri-nitro-benzol,
and tri-nitro-naphthalene. The equation of its
combustion is given as

C\_{6}H\_{4}(NO\_{2})\_{2} + 10(NH\_{4}NO\_{3}) = 6CO\_{2}
+ 22H\_{2}O + 11N\_{2}

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and, like bellite and roburite, it is claimed to be
perfectly safe to use in the presence of fire damp
and coal dust.[A] The variety known as Flameless Securite
consists of a mixture of nitrate and oxalate of ammonia
and di-nitro-benzol.

[Footnote A: See paper by S.B. Coxon, *North
of Eng. Inst. Mining and Mech. Eng.*,
11, 2, 87.]
 *Kinetite.*—­A few years ago an explosive
called “Kinetite"[A] was introduced, but is
not manufactured in England. It was the patent
of Messrs Petry and Fallenstein, and consisted of
nitro-benzol, thickened or gelatinised by the addition
of some collodion-cotton, incorporated with finely
ground chlorate of potash and precipitated sulphide
of antimony. An analysis gave the following percentages:—­

Nitro-benzol, 19.4 per cent.
Chlorate of potash, 76.9 per cent.
Sulphide of antimony nitro-cotton, 3.7 per cent.

[Footnote A: V. Watson Smith, *Jour. Soc.
Chem. Ind.*, January 1887.]

It requires a very high temperature to ignite it,
and cannot, under ordinary circumstances, when unconfined,
be exploded by the application of heat. It is
little affected by immersion in water, unless prolonged,
when the chlorate dissolves out, leaving a practical
inexplosive residue.[A] It was found to be very sensitive
to combined friction and percussion, and to be readily
ignited by a glancing blow of wood upon wood.
It was also deficient in chemical stability, and has
been known to ignite spontaneously both in the laboratory
and in a magazine. It is an orange-coloured
plastic mass, and smells of nitro-benzol.

[Footnote A: Col. Cundill, R.A., “Dict.
of Explosives,” says: “If, however,
it be exposed to moist and dry air alternately, the
chlorate crystallises out on the surfaces, and renders
the explosive very sensitive.”]
 *Tonite No. 3* contains 10 to 14 per cent. of
nitro-benzol (see Tonite). Trench’s Flameless
Explosive contains 10 per cent. of di-nitro-benzol,
together with 85 per cent. of nitrate of ammonia, and
5 per cent. of a mixture of alum, and the chlorides
of sodium and ammonia.
 *Tri-nitro-Toluene.*—­Toluene, C\_{7}H\_{8},
now chiefly obtained from coal-tar, was formerly
obtained by the dry distillation of tolu-balsam.
It may be regarded as methyl-benzene, or benzene in
which one hydrogen is replaced by methyl (CH\_{3}),
thus (C\_{6}H\_{5}CH\_{3}), or as phenyl-methane, or
methane in which one hydrogen atom is replaced by the
radical phenyl (C\_{6}H\_{5}), thus (CH\_{3}C\_{6}H\_{5}).
Toluene is a colourless liquid, boiling at 110 deg.
C., has a specific gravity of .8824 at 0 deg.
C., and an aromatic odour. Tri-nitro-toluene
is formed by the action of nitric acid on toluene.
According to Haeussermann, it is more advantageous
to start with the ortho-para-di-nitro-toluene, which
is prepared by allowing a mixture of 75 parts of 91
to 92 per cent. nitric acid and 150 parts of 95 to
96 per cent. sulphuric acid to run in a thin stream

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into 100 parts of para-nitro-toluene, while the latter
is kept at a temperature between 60 deg. to 65 deg.
C., and continually stirred. When the acid has
all been run in, this mixture is heated for half an
hour to 80 deg. C., and allowed to stand till
cold. The excess of nitric acid is then removed.
The residue after this treatment is a homogeneous
crystalline mass of ortho-para-di-nitro-toluene,
of which the solidifying point is 69.5 deg. C.
To convert this mass into tri-nitro derivative, it
is dissolved by gently heating it with four times
its weight of sulphuric acid (95 to 96 per cent.),
and it is then mixed with 1-1/2 times its weight of
nitric acid (90 to 92 per cent.), the mixture being
kept cool. Afterwards it is digested at 90 deg.
to 95 deg. C., with occasional stirring, until
the evolution of gas ceases. This takes place
in about four or five hours.

The operation is now stopped, the product allowed
to cool, and the excess of nitric acid separated from
it. The residue is then washed with hot water
and very dilute soda solution, and allowed to solidify
without purification. The solidifying point is
70 deg. C., and the mass is then white, with
a radiating crystalline structure. Bright sparkling
crystals, melting at 81.5 deg. C. may, however,
be obtained by recrystallisation from hot alcohol.
The yield is from 100 parts di-nitro-toluene, 150 parts
of the tri-nitro derivative. Haeussermann states
also that 1:2:4:6 tri-nitro-toluene can be obtained
from ordinary commercial di-nitro-toluene melting
at 60 deg. to 64 deg. C.; but when this is used,
greater precautions must be exercised, for the reactions
are more violent. Moreover, 10 per cent. more
nitric acid is required, and the yield is 10 per cent.
less. He also draws attention to the slight solubility
of tri-nitro-toluene in hot water, and to the fact
that it is decomposed by dilute alkalies and alkaline
carbonates—­facts which must be borne in
mind in washing the substance. This material
is neither difficult nor dangerous to make. It
behaves as a very stable substance when exposed to
the air under varying conditions of temperature (-10
deg. to +50 deg. C.) for several months.
It cannot be exploded by flame, nor by heating it
in an open vessel. It is only slightly decomposed
by strong percussion on an anvil. A fulminate
detonator produces the best explosive effect with
tri-nitro-toluene. It can be used in conjunction
with ammonium nitrate, but such admixture weakens the
explosive power; but even then it is stated to be
stronger than an equivalent mixture of di-nitro-benzene
and ammonium nitrate. Mowbray patented a mixture
of 3 parts nitro-toluol to 7 of nitro-glycerine, also
in the proportions of 1 to 3, which he states to be
a very safe explosive.
 *Faversham Powder.*—­One of the explosives
on the permitted list (coal mines) is extensively
used, and is manufactured by the Cotton Powder Co.
Ltd. at Faversham. It is composed of tri-nitro-toluol
11 parts, ammonium nitrate 93 parts, and moisture
1 part. This explosive must be used only when
contained in a case of an alloy of lead, tin, zinc,
and antimony thoroughly waterproof; it must be used
only with a detonator or electric detonator of not
less strength than that known as No. 6.

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*Nitro-Naphthalene.*—­Nitro-naphthalene
is formed by the action of nitric acid on naphthalene
(C\_{10}H\_{8}). Its formula is C\_{10}H\_{7}NO\_{2},
and it forms yellow needles, melting at 61 deg.
C.; and of di-nitro-naphthalene (C\_{10}H\_{6}(NO\_{2})\_{2}),
melting point 216 deg. C. There are also tri-nitro
and tetra-nitro and [alpha] and [beta] derivatives
of nitro-naphthalene. It is the di-nitro-naphthalene
that is chiefly used in explosives. It is contained
in roburite, securite, romit, Volney’s powder,
&c. Fehven has patented an explosive consisting
of 10 parts of nitro-naphthalene mixed with the crude
ingredients of gunpowder as follows:—­Nitro-naphthalene,
10 parts; saltpetre, 75 parts; charcoal, 12.5 parts;
and sulphur, 12.5 parts. He states that he obtains
a mono-nitro-naphthalene, containing a small proportion
of di-nitro-naphthalene, by digesting 1 part of naphthalene,
with or without heat, in 4 parts of nitric acid (specific
gravity 1.40) for five days.

Quite lately a patent has been taken out for a mixture
of nitro-naphthalene or di-nitro-benzene with ammonium
nitrate, and consists in using a solvent for one or
other or both of the ingredients, effected in a wet
state, and then evaporating off the solvent, care being
taken not to melt the hydrocarbon. In this way
a more intimate mixture is ensured between the particles
of the components, and the explosive thus prepared
can be fired by a small detonator, *viz*., by 0.54
grms. of fulminate. Favier’s explosive
also contains mono-nitro-naphthalene (8.5 parts),
together with 91.5 parts of nitrate of ammonia.
This explosive is made in England by the Miners’
Safety Explosive Co. A variety of roburite contains
chloro-nitro-naphthalene. Romit consists of 100
parts ammonium nitrate and 7 parts potassium chlorate
mixed with a solution of 1 part nitro-naphthalene
and 2 parts rectified paraffin oil.
 *Ammonite.*—­This explosive was originally
made at Vilvorde in Belgium, under the title of the
Favier Explosive, consisting of a compressed hollow
cylinder composed of 91.5 per cent. of nitrate of ammonia,
and 8.5 per cent. of mono-nitro-naphthalene filled
inside with loose powder of the same composition.
The cartridges were wrapped in paper saturated with
paraffin-wax, and afterwards dipped in hot paraffin
to secure their being water-tight. The Miners’
Safety Explosives Co., when making this explosive
at their factory at Stanford-le-Hope, Essex, abandoned
after a short trial the above composition, and substituted
di-nitro-naphthalene 11.5 per cent. for the mono-nitro-naphthalene,
and used thin lead envelopes filled with loose powder
slightly pressed in, in place of the compressed cylinders
containing loose powder. The process of manufacture
is shortly as follows:—­132-3/4 lbs. of
thoroughly dried nitrate of ammonium is placed in
a mill pan, heated at the bottom with live steam, and
ground for about twenty minutes until it becomes so
dry that a slight dust follows the rollers; then 17-1/2

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lbs. of thoroughly dry di-nitro-naphthalene is added,
and the grinding continued for about ten minutes.
Cold water is then circulated through the bottom of
the pan until the material appears of a lightish colour
and falls to powder. (While the pan is hot the whole
mass looks slightly plastic and of a darker colour
than when cold.) A slide in the bottom of the pan
is then withdrawn, the whole mass working out until
the pan is empty; it is now removed to the sifting
machine, brushed through a wire sieve of about 12
holes to the inch, and is then ready for filling into
cartridges. The hard core is returned from the
sifting machine and turned into one of the pans a
few minutes before the charge is withdrawn.

The ammonite is filled into the metallic cartridges
by means of an archimedian screw working through a
brass tube, pushing off the cartridges as the explosive
is fed into them against a slight back pressure; a
cover is screwed on, and they then go to the dipping
room, where they are dipped in hot wax to seal the
ends; they are then packed in boxes of 5 lbs. each
and are ready for delivery. The di-nitro-naphthalene
is made at the factory. Mono-nitro-naphthalene
is first made as follows:—­12 parts of commercial
nitrate of soda are ground to a fine powder, and further
ground with the addition of 15 parts of refined naphthalene
until thoroughly incorporated; it is then placed in
an earthenware pan, and 30 parts of sulphuric acid
of 66 deg. B. added, 2 parts at a time, during
forty-eight hours (the rate of adding H\_{2}SO\_{4}
depends on the condition of the charge, and keeping
it in a fluid state), with frequent agitation, day
and night, during the first three or four days, afterwards
three or four times a day. In all fourteen days
are occupied in the nitration process. It is
then strained through an earthenware strainer, washed
with warm water, drained, and dried. For the
purpose of producing this material in a granulated
condition, which is found more convenient for drying,
and further nitrification, it is placed in a tub,
and live steam passed through, until brought up to
the boiling point (the tub should be about half full),
cold water is then run in whilst violently agitating
the contents until the naphthalene solidifies; it
can then be easily drained and dried. For the
further treatment to make di-nitro-naphthalene, 18
parts of nitro-naphthalene are placed in an earthenware
pan, together with 39 parts of sulphuric acid of 66
deg. B., then 15 parts of nitric acid of 40 deg.
B. are added, in small quantities at a time, stirring
the mixture continually. This adding of nitric
acid is controlled by the fuming, which should be
kept down as much as possible. The operation takes
ten to twelve days, when 100 times the above quantities,
taken in kilogrammes, are taken. At the end of
the nitration the di-nitro-naphthalene is removed to
earthenware strainers, allowed to drain, washed with
hot water and soda until all acid is removed, washed
with water and dried. The di-nitro-naphthalene
gives some trouble in washing, as some acid is held
in the crystals which is liable to make its appearance
when crushed. To avoid this it should be ground
and washed with carbonate of soda before drying; an
excess of carbonate of soda should not, however, be
used.

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*Electronite.*—­This is a high explosive
designed to afford safety in coal getting. This
important end has been attained by using such ingredients,
and so proportioning them, as will ensure on detonation
a degree of heat insufficient under the conditions
of a “blown-out” shot, to ignite fire
damp or coal dust. It is of the nitrate of ammonium
class of permitted explosives. It contains about
75 per cent. of nitrate of ammonium, with the addition
of nitrate of barium, wood meal, and starch. The
gases resulting from detonation are chiefly water
in the gaseous form, nitrogen, and a little carbon
dioxide. It is granulated with the object of
preventing missfires from ramming, to which nitrate
of ammonium explosives are somewhat susceptible.
This explosive underwent some exhaustive experiments
at the experimental station near Wigan in 1895, when
8 oz. or 12 oz. charges were fired unstemmed into
an admixture of coal dust and 10 per cent. of gas,
without any ignition taking place. It is manufactured
by Messrs Curtis’s & Harvey Ltd. at their factory,
Tonbridge, Kent.
 *Sprengel’s Explosives.*—­This
is a large class of explosives. The essential
principle of them all is the admixture of an oxidising
with a combustible agent at the time of, or just before,
being required for use, the constituents of the mixture
being very often non-explosive bodies. This type
of explosive is due to the late Dr Herman Sprengel,
F.R.S. Following up the idea that an explosion
is a sudden combustion, he submitted a variety of
mixtures of oxidising and combustible agents to the
violent shock of a detonator of fulminate. These
mixtures were made in such proportions that the mutual
oxidation or de-oxidation should be theoretically
complete. Among them are the following:—­

1. One chemical equivalent of nitro-benzene to
equivalents of nitric acid.

2. Five equivalents of picric acid to 13 equivalents
of nitric acid.

3. Eighty-seven equivalents of nitro-naphthalene
to 413 equivalents of nitric acid.

4. Porous cakes, or lumps of chlorate of potash,
exploded violently with bisulphide of carbon, nitro-benzol,
carbonic acid, sulphur, benzene, and mixtures of these
substances.

No. 1 covers the explosive known as *Hellhoffite*,
and No. 2 is really oxonite, and No. 4 resembles rack-a-rock,
an explosive invented by Mr S.R. Divine, and
consisting of a mixture of chlorate of potash and nitro-benzol.
Roburite, bellite, and securite should perhaps be regarded
as belonging to the Sprengel class of explosives,
otherwise this class is not manufactured or used in
England. The principal members are known as *Hellhoffite*,
consisting of a mixture of nitro-petroleum or nitro-tar
oils and nitric acid, or of meta-di-nitro-benzol and
nitric acid; *Oxonite*, consisting of picric
and nitric acids; and *Panclastite*, a name given
to various mixtures, proposed by M. Turpin, such as
liquid nitric peroxide, with bisulphide of carbon,
benzol, petroleum, ether, or mineral oils.

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*Picric Acid, Tri-nitro-Phenol, or Carbazotic Acid.*—­Picric
acid, or a tri-nitro-phenol (C\_{6}H\_{2}(NO\_{2})\_{3}OH)[2:4:6],
is produced by the action of nitric acid on many organic
substances, such as phenol, indigo, wool, aniline,
resins, &c. At one time a yellow gum from Botany
Bay (*Xanthorrhoea hastilis*) was chiefly used.
One part of phenol (carbolic acid), C\_{6}H\_{5}OH,
is added to 3 parts of strong fuming nitric acid,
slightly warmed, and when the violence of the reaction
has subsided, boiled till nitrous fumes are no longer
evolved. The resinous mass thus produced is boiled
with water, the resulting picric acid is converted
into a sodium salt by a solution of sodium carbonate,
which throws down sodium picrate in crystals.

Phenol-sulphuric acid is now, however, more generally
used, and the apparatus employed for producing it
closely resembles that used in making nitro-benzol.
It is also made commercially by melting carbolic acid,
and mixing it with strong sulphuric acid, then diluting
the “sulpho-carbolic"[A] acid with water, and
afterwards running it slowly into a stone tank containing
nitric acid. This is allowed to cool, where the
crude picric acid crystallises out, and the acid liquid
(which contains practically no picric acid, but only
sulphuric acid, with some nitric acid) being poured
down the drains. The crude picric acid is then
dissolved in water by the aid of steam, and allowed
to cool when most of the picric acid recrystallises.
The mother liquor is transferred to a tank and treated
with sulphuric acid, when a further crop of picric
acid crystals is obtained. The crystals of picric
acid are further purified by recrystallisation, drained,
and dried at 100 deg. F. on glazed earthenware
trays by the aid of steam. It can also be obtained
by the action of nitric acid on ortho-nitro-phenol,
para-nitro-phenol, and di-nitro-phenol (2:4 and 2:6),
but not from meta-nitro-phenol, a fact which indicates
its constitution.[B]

[Footnote A: O. and p. phenolsulphonic acids.

C\_{3}H\_{4}(OH).SO\_{3}H + 3HNO\_{3} = C\_{6}H\_{2}(NO\_{2})\_{3}OH
+ H\_{2}SO\_{4} + 2H\_{2}O.
 (Picric acid).]

[Footnote B: Carey Lea, *Amer. Jour.
Sci.*, (ii.), xxxii. 180.]

Picric acid crystallises in yellow shining prisms
or laminae having an intensely bitter taste, and is
poisonous. It melts at 122.5 deg. C., sublimes
when cautiously heated, dissolves sparingly in cold
water, more easily in hot water, still more in alcohol.
It stains the skin an intense yellow colour, and is
used as a dye for wool and silk. It is a strong
acid, forming well crystallised yellow salts, which
detonate violently when heated, some of them also
by percussion. The potassium salt, C\_{6}H\_{2}(NO\_{2})\_{3}OK,
crystallises in long needles very slightly soluble
in water. The sodium, ammonium, and barium salts
are, however, easily soluble in water. Picric
acid, when heated, burns with a luminous and smoky
flame, and may be burnt away in large quantity without
explosion; but the mere contact of certain metallic
oxides, with picric acid, in the presence of heat,
develops powerful explosives, which are capable of
acting as detonators to an indefinite amount of the
acid, wet or dry, which is within reach of their detonative
influence. The formula of picric acid is

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C\_{6}H\_{2}|(NO\_{2})\_{3}
|OH.

which shows its formation from phenol (C\_{6}H\_{5}OH.),
three hydrogen atoms being displaced by the NO\_{2}
group. The equation of its formation from phenol
is as follows:—­

C\_{6}H\_{5}.OH + 3HNO\_{3} = C\_{6}H\_{2}(NO\_{2})\_{3}OH
+ 3H\_{2}O.

According to Berthelot, its heat of formation from
its elements equals 49.1 calories, and its heat of
total combustion by free oxygen is equal to +618.4
cals. It hardly contains more than half the oxygen
necessary for its complete combustion.

2C\_{6}H\_{2}(NO\_{2})\_{3}OH + O\_{10} = 12CO\_{2} + 3H\_{2}
+ 3N\_{2}.

The percentage composition of picric acid is—­Nitrogen,
18.34; oxygen, 49.22; hydrogen, 1.00; and carbon,
31.44, equal to 60.26 per cent. of NO\_{2}. The
products of decomposition are carbonic acid, carbonic
oxide, carbon, hydrogen, and nitrogen, and the heat
liberated, according to Berthelot, would be 130.6
cals., or 570 cals. per kilogramme. The reduced
volume of the gases would be 190 litres per equivalent,
or 829 litres per kilogramme. To obtain a total
combustion of picric acid it is necessary to mix with
it an oxidising agent, such as a nitrate, chlorate,
&c. It has been proposed to mix picric acid (10
parts) with sodium nitrate (10 parts) and potassium
bichromate (8.3 parts). These proportions would
furnish a third of oxygen in excess of the necessary
proportion.

Picric acid was not considered to be an explosive,
properly so called, for a long time after its discovery,
but the disastrous accident which occurred at Manchester
(*vide* Gov. Rep. No. LXXXI., by
Colonel (now Sir V.D.) Majendie, C.B.), and some experiments
made by Dr Dupre and Colonel Majendie to ascertain
the cause of the accident, conclusively proved that
this view was wrong. The experiments of Berthelot
(*Bull. de la Soc. Chim. de Paris*, xlix.,
p. 456) on the explosive decomposition of picric acid
are also deserving of attention in this connection.
If a small quantity of picric acid be heated in a
moderate fire, in a crucible, or even in an open test
tube, it will melt (at 120 deg. C. commercial
acid), then give off vapours which catch fire upon
contact with air, and burn with a sooty flame, without
exploding. If the burning liquid be poured out
upon a cold slab, it will soon go out. A small
quantity carefully heated in a tube, closed at one
end, can even be completely volatilised without apparent
decomposition. It is thus obvious that picric
acid is much less explosive than the nitric ethers,
such as nitro-glycerol and nitro-cellulose, and very
considerably less explosive than the nitrogen compounds
and fulminates.

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It would, however, be quite erroneous to assume that
picric acid cannot explode when simply heated.
On the contrary, Berthelot has proved that this is
not the case. If a glass tube be heated to redness,
and a minute quantity of picric acid crystals be then
thrown in, it will explode with a curious characteristic
noise. If the quantity be increased so that the
temperature of the tube is materially reduced, no explosion
will take place at once, but the substance will volatilise
and then explode, though with much less violence than
before, in the upper part of the tube. Finally,
if the amount of picric acid be still further increased
under these conditions, it will undergo partial decomposition
and volatilise, but will not even deflagrate.
Nitro-benzene, di-nitrobenzene, and mono-, di-, and
tri-nitro-naphthalenes behave similarly.

The manner in which picric acid will decompose is
thus dependent upon the initial temperature of the
decomposition, and if the surrounding material absorb
heat as fast as it is produced by the decomposition,
there will be no explosion and no deflagration.
If, however, the absorption is not sufficient to prevent
deflagration, this may so increase the temperature
of the surrounding materials that the deflagration
will then end in explosion. Thus, if an explosion
were started in an isolated spot, it would extend
throughout the mass, and give rise to a general explosion.

In the manufacture of picric acid the first obvious
and most necessary precaution is to isolate the substance
from other chemicals with which it might accidentally
come into contact. If pure materials only are
used, the manufacture presents no danger. The
finished material, however, must be carefully kept
from contact with nitrates, chlorates, or oxides.
If only a little bit of lime or plaster become accidentally
mixed with it, it may become highly dangerous.
A local explosion may occur which might have the effect
of causing the explosion of the whole mass. Picric
acid can be fired by a detonator, 5-grain fulminate,
and M. Turpin patented the use of picric acid, unmixed
with any other substance, in 1885. The detonation
of a small quantity of dry picric acid is sufficient
to detonate a much larger quantity containing as much
as 17 per cent. of water.

It is chiefly due to French chemists (and to Dr Sprengel)
that picric acid has come to the front as an explosive.
Melinite,[A] a substance used by the French Government
for filling shells, was due to M. Turpin, and is supposed
to be little else than fused picric acid mixed with
gun-cotton dissolved in some solvent (acetone or ether-alcohol).
Sir F.A. Abel has also proposed to use picric
acid, mixed with nitrate of potash (3 parts) and picrate
of ammonia (2 parts) as a filling for shells.
This substance requires a violent blow and strong
confinement to explode it. I am not aware, however,
that it has ever been officially adopted in this country.
Messrs Designolles and Brugere have introduced military
powders, consisting of mixtures of potassium and ammonium
picrates with nitrate of potassium. M. Designolles
introduced three kinds of picrate powders, composed
as follows:—­

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\_\_\_\_\_\_\_\_\_\_\_\_
| | | | |
| | For Torpedoes | For Guns. | For Small |
| | and Shells. | Ordinary. Heavy. | Arms. |
|\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_|\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_|\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_|\_\_\_
\_\_\_\_\_\_\_\_|
| | | | | |
| Picrate of Potash | 55-50 | 16.4- 9.6 | 9 | 28.6-22.9 |
| Saltpetre | 45-50 | 74.4-79.7 | 80 | 65.0-69.4 |
| Charcoal | ... | 9.2-10.7 | 11 | 6.4- 7.7 |
|\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_|\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_|\_\_\_\_\_\_\_\_\_\_\_|\_\_\_\_\_\_\_|\_\_\_
\_\_\_\_\_\_\_\_|

They were made much like ordinary gunpowder, 6 to
14 per cent. of moisture being added when being milled.
The advantages claimed over gunpowder are greater
strength, and consequently greater ballistic or disruptive
effect, comparative absence of smoke, and freedom
from injurious action on the bores of guns, owing
to the absence of sulphur. Brugere’s powder
is composed of ammonium picrate and nitre, the proportions
being 54 per cent. picrate of ammonia and 46 per cent.
potassic nitrate. It is stable, safe to manufacture
and handle, but expensive. It gives good results
in the Chassepot rifle, very little smoke, and its
residue is small, and consists of carbonate of potash.
It is stated that 2.6 grms. used in a rifle gave an
effect equal to 5.5 grms. of ordinary gunpowder.

[Footnote A: The British Lydite and the Japanese
Shimose are said to be identical with Melinite.]

Turpin has patented various mixtures of picric acid,
with gum-arabic, oils, fats, collodion jelly, &c.
When the last-named substance is diluted in the proportion
of from 3 to 5 per cent. in a mixture of ether and
alcohol, he states that the blocks of picric acid moulded
with it will explode in a closed chamber with a priming
of from 1 to 3 grammes of fulminate. He also
casts picric acid into projectiles, the cast acid
having a density of about 1.6. In this state it
resists the shock produced by the firing of a cannon,
when contained in a projectile, having an initial
velocity of 600 metres. It is made in the following
way:—­The acid is fused in a vessel provided
with a false bottom, heated to 130 deg. to 145 deg.
C. by a current of steam under pressure, or simply
by the circulation under the false bottom of a liquid,
such as oil, chloride of zinc, glycerine, &c., heated
to the same temperature. The melted picric acid
is run into moulds of a form corresponding to that
of the blocks required, or it may be run into projectiles,
which should be heated to a temperature of about 100
deg. C., in order to prevent too rapid solidification.

When cresylic acid (or cresol, C\_{6}H\_{4}(CH\_{3})OH.)
is acted upon by nitric acid it produces a series
of nitro compounds very similar to those formed by
nitric acids on phenol, such as sodium di-nitro-cresylate,
known in the arts as victoria yellow. Naphthol,
a phenol-like body obtained from naphthalene, under
the same conditions, produces sodium di-nitro-naphthalic
acid, C\_{10}H\_{6}(NO\_{2})\_{2}O. The explosive
known as “roburite” contains chloro-nitro-naphthalene,
and romit, a Swedish explosive, nitro-naphthalene.

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*Tri-nitro-cresol*, C\_{7}H\_{4}(NO\_{2})\_{3}OH.—­A
body very similar to tri-nitro-phenol, crystallises
in yellow needles, slightly soluble in cold water,
rather more so in boiling water, alcohol, and ether.
It melts at about 100 deg. C. In France it is
known as “Cresilite,” and mixed with melinite,
is used for charging shells. By neutralising a
boiling saturated solution of tri-nitro-cresol with
ammonia, a double salt of ammonium and nitro-cresol
crystallises out upon cooling, which is similar to
ammonium picrate. This salt is known as “Ecrasite,”
and has been used in Austria for charging shells.
It is a bright yellow solid, greasy to the touch,
melts at 100 deg. C., is unaffected by moisture,
heat, or cold, ignites when brought into contact with
an incandescent body or open flame, burning harmlessly
away unless strongly confined, and is insensitive to
friction or concussion. It is claimed to possess
double the strength of dynamite, and requires a special
detonator (not less than 2 grms. of fulminate) to
provoke its full force. Notwithstanding the excellent
properties attributed to this explosive, Lieut.
W. Walke ("Lectures on Explosives,” p. 181)
says, “Several imperfectly explained and unexpected
explosions have occurred in loading shells with this
substance, and have prevented its general adoption
up to the present time.”
 *The Fulminates.*—­The fulminates are
salts of fulminic acid, C\_{2}N\_{2}O\_{2}H\_{2}.
Their constitution is not very well understood.
Dr E. Divers, F.R.S., and Mr Kawakita (*Chem.
Soc. Jour.*, 1884, pp. 13-19), give the formulae
of mercury and silver fulminates as

OC = N AgOC = N
/ | \ | \
Hg | O and | O
\ | / | /
-C = N AgC = N

whereas Dr H.E. Armstrong, F.R.S., would prefer
to write the formula of fulminic acid

ON.C.OH.
|
C(N.OH),

and A.F. Holleman (*Berichte*, v. xxvi.,
p. 1403), assigns to mercury fulminate the formula

C:N.O
Hg | |
C:N.O,

and R. Schol (*Ber.*, v. xxiii., p. 3505),

C:NO
|| Hg.
C:NO

They are very generally regarded as iso-nitroso compounds.

The principal compound of fulminic acid is the mercury
salt commonly known as fulminating mercury. It
is prepared by dissolving mercury in nitric acid,
and then adding alcohol to the solution, 1 part of
mercury and 12 parts of nitric acid of specific gravity
1.36, and 5-1/2 parts of 90 per cent. alcohol being
used. As soon as the mixture is in violent reaction,
6 parts more of alcohol are added slowly to moderate
the action. At first the mixture blackens from
the separation of mercury, but this soon vanishes,
and is succeeded by crystalline flocks of mercury fulminate
which fall to the bottom of the vessel. During
the reaction, large quantities of volatile oxidation
products of alcohol, such as aldehyde, ethylic nitrate,
&c., are evolved from the boiling liquid, whilst others,

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such as glycollic acid, remain in solution. The
mercury fulminate is then crystallised from hot water.
It forms white silky, delicate needles, which are
with difficulty soluble in cold water. In the
dry state it is extremely explosive, detonating on
heating, or by friction or percussion, as also on
contact with concentrated sulphuric acid. The
reaction that takes place upon its decomposition is
as follows:—­

C\_{2}N\_{2}O\_{2}Hg = Hg + 2CO + N\_{2}
(284)

According to this equation 1 grm. of the fulminate
should yield 235.8 c.c. (= 66.96 litres for 284 grms.).
Berthelot and Vicille have obtained a yield of 234.2
c.c., equal to 66.7 litres for one equivalent 284 grms.

Dry fulminate explodes violently when struck, compressed,
or touched with sulphuric acid, or as an incandescent
body. If heated slowly, it explodes at 152 deg.
C., or if heated rapidly, at 187 deg. C. It is
often used mixed with potassium chlorate in detonators.
The reaction which takes place in this case is 3C\_{2}N\_{2}O\_{2}Hg
+ 2KClO\_{3} = 3Hg + 6CO\_{2} + 3N\_{2} + 2KCl.

On adding copper or zinc to a hot saturated solution
of the salt, fulminate of copper or zinc is formed.
The copper salt forms highly explosive green crystals.
There is also a double fulminate of copper of ammonia,
and of copper and potassium. Silver fulminite,
C\_{2}N\_{2}O\_{2}Ag\_{2}, is prepared in a similar manner
to the mercury salt. It separates in fine white
needles, which dissolve in 36 parts of boiling water,
and are with difficulty soluble in cold water.
At above 100 deg. C., or on the weakest blow,
it explodes with fearful violence. Even when
covered with water it is more sensitive than the mercury
salt. It forms a very sensitive double salt with
ammonia and several other metals. With hydrogen
it forms the acid fulminate of silver. It is used
in crackers and bon-bons, and other toy fireworks,
in minute quantities. Gay Lussac found it to
be composed as follows:—­Carbon, 7.92 per
cent.; nitrogen, 9.24 per cent.; silver, 72.19 per
cent.; oxygen, 10.65 per cent.; and he assigned to
it the formula, C\_{2}N\_{2}Ag\_{2}O\_{2}. Laurent
and Gerhardt give it the formula, C\_{2}N(NO\_{2})Ag\_{2},
and thus suppose it to contain nitryl, NO\_{2}.

On adding potassium chloride to a boiling solution
of argentic fulminate, as long as a precipitate of
argentic chloride forms, there is obtained on evaporation
brilliant white plates, of a very explosive nature,
of potassic argentic fulminate, C(NO\_{2})KAg.CN, from
whose aqueous solution nitric acid precipitates a
white powder of hydric argentic fulminate, C(NO\_{2})HAg.CN.
All attempts to prepare fulminic acid, or nitro-aceto-nitrile,
C(NO\_{2})H\_{2}CN, from the fulminates have failed.
There is a fulminate of gold, which is a violently
explosive buff precipitate, formed when ammonia is
added to ter-chloride of gold, and fulminate of platinum,
a black precipitate formed by the addition of ammonia
to a solution of oxide platinum, in dilute sulphuric
acid.

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Fulminating silver is a compound obtained by the action
of ammonia on oxide of silver. It is a very violent
explosive. Pure mercury fulminate may be kept
an indefinite length of time. Water does not affect
it. It explodes at 187 deg. C., and on contact
with an ignited body. It is very sensitive to
shock and friction, even that of wood upon wood.
It is used for discharging bullets in saloon rifles.
Its inflammation is so sudden that it scatters black
powder on which it is placed without igniting it,
but it is sufficient to place it in an envelope, however
weak, for ignition to take place, and the more resisting
the envelope the more violent is the shock, a circumstance
that plays an important part in caps and detonators.
The presence of 30 per cent. of water prevents decomposition,
10 per cent. prevents explosion. This is, however,
only true for small quantities, and does not apply
to silver fulminate, which explodes under water by
friction. Moist fulminates slowly decompose on
contact with the oxidisable metals. The (reduced)
volume of gases obtained from 1 kilo. is according
to Berthelot, 235.6 litres. The equation of its
decomposition is C\_{2}HgN\_{2}O\_{2} = 2CO + N\_{2} +
Hg.

Fulminate of mercury is manufactured upon the large
scale by two methods. One of these, commonly
known as the German method, is conducted as follows:—­One
part of mercury is dissolved in 12 parts of nitric
acid of a specific gravity of 1.375, and to this solution
16.5 parts of absolute alcohol are added by degrees,
and heat is then slowly applied to the mixture until
the dense fumes first formed have disappeared, and
when the action has become more violent some more
alcohol is added, equal in volume to that which has
already been added. This is added very gradually.
The product obtained, which is mercury fulminate,
is 112 per cent. of the mercury employed. Another
method is to dissolve 10 parts of mercury in 100 parts
of nitric acid of a gravity of 1.4, and when the solution
has reached a temperature of 54 deg. C, to pour
it slowly through a glass funnel into 83 parts of
alcohol. When the effervescence ceases, it is
filtered through paper filters, washed, and dried
over hot water, at a temperature not exceeding 100
deg. C. The fulminate is then carefully packed
in paper boxes, or in corked bottles. The product
obtained by this process is 130 per cent. of the mercury
taken. This process is the safest, and at the
same time the cheapest. Fulminate should be kept,
if possible, in a damp state. Commercial fulminate
is often adulterated with chlorate of potash.
 *Detonators*, or caps, are metallic capsules,
usually of copper, and resemble very long percussion
caps. The explosive is pure fulminate of mercury,
or a mixture of that substance with nitrate or chlorate
of potash, gun-powder, or sulphur. The following
is a common cap mixture:—­ 100 parts of
fulminate of mercury and 50 parts of potassium nitrate,
or 100 parts of fulminate and 60 parts of meal powder.
Silver fulminate is also sometimes used in caps.
There are eight sizes made, which vary in dimensions
and in amount of explosive contained. They are
further distinguished as singles, doubles, trebles,
&c., according to their number. Colonel Cundill,
R.A. ("Dict. of Explosives"), gives the following
list:—­

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No. 1 contains 300 grms. of explosive per 1000. "
 2 " 400 " " " " " " 3
 " 540 " " " " " " 4 "
 650 " " " " " " 5 " 800
 " " " " " " 6 " 1,000 "
 " " " " " 7 " 1,500 " "
 " " " " 8 " 2,000 " "
" " "

Trebles are generally used for ordinary dynamite,
5, 6, or 7 for gun-cotton, blasting gelatine, roburite,
&c.

In the British service percussion caps, fuses, &c.,
are formed of 6 parts by weight of fulminate of mercury,
6 of chlorate of potash, and 4 of sulphide of antimony;
time fuses of 4 parts of fulminate, 6 of potassium
chlorate, 4 of sulphide of antimony, the mixture being
damped with a varnish consisting of 645 grains of
shellac dissolved in a pint of methylated spirit.
Abel’s fuse (No. 1) consists of a mixture of
sulphide of copper, phosphide of copper, chlorate
of potash, and No. 2 of a mixture of gun-cotton and
gun-powder. They are detonated by means of a platinum
wire heated to redness by means of an electric current.
Bain’s fuse mixture is a mixture of subphosphide
of copper, sulphide of antimony, and chlorate of potash.

In the manufacture of percussion caps and detonators
the copper blanks are cut from copper strips and stamped
to the required shape. The blanks are then placed
in a gun-metal plate, with the concave side uppermost—­a
tool composed of a plate of gun-metal, in which are
inserted a number of copper points, each of the same
length, and so spaced apart as to exactly fit each
point into a cap when inverted over a plate containing
the blanks. The points are dipped into a vessel
containing the cap composition, which has been previously
moistened with methylated spirit. It is then removed
and placed over the blanks, and a slight blow serves
to deposit a small portion of the cap mixture into
each cap. A similar tool is then dipped into
shellac varnish, removed and placed over the caps,
when a drop of varnish from each of the copper points
falls into the caps, which are then allowed to dry.
This is a very safe and efficacious method of working.

At the works of the Cotton-Powder Company Limited,
at Faversham, the fulminate is mixed wet with a very
finely ground mixture of gun-cotton and chlorate of
potash, in about the proportions of 6 parts fulminate,
1 part gun-cotton, and 1 part chlorate. The water
in which the fulminate is usually stored is first
drained off, and replaced by displacement by methyl-alcohol.
While the fulminate is moist with alcohol, the gun-cotton
and chlorate mixture is added, and well mixed with
it. This mixture is then distributed in the detonators
standing in a frame, and each detonator is put separately
into a machine for the purpose of pressing the paste
into the detonator shell.

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At the eleventh annual meeting of the representatives
of the Bavarian chemical industries at Regensburg,
attention was drawn to the unhealthy nature of the
process of charging percussion caps. Numerous
miniature explosions occur, and the air becomes laden
with mercurial vapours, which exercise a deleterious
influence upon the health of the operatives. There
is equally just cause for apprehension in respect to
the poisonous gases which are evolved during the solution
of mercury in nitric acid, and especially during the
subsequent treatment with alcohol. Many methods
have been proposed for dealing with the waste products
arising during the manufacture and manipulation of
fulminate of mercury, but according to Kaemmerer,
only one of comparatively recent introduction appears
to be at all satisfactory. It is based upon the
fact that mercuric fulminate, when heated with a large
volume of water under high pressure, splits up into
metallic mercury and non-explosive mercurial compounds
of unknown composition.

In mixing the various ingredients with mercury fulminate
to form cap mixtures, they should not be too dry;
in fact, they are generally more or less wet, and
mixed in small quantities at a time, in a special house,
the floors of which are covered with carpet, and the
tables with felt. Felt shoes are also worn by
the workpeople employed. All the tools and apparatus
used must be kept very clean; for granulating, hair
sieves are used, and the granulated mixture is afterwards
dried on light frames, with canvas trays the bottoms
of which are covered with thin paper, and the frames
fitted with indiarubber cushions, to reduce any jars
they may receive. The windows of the building
should be painted white to keep out the rays of the
sun.

Mr H. Maxim, of New York, has lately patented a composition
for detonators for use with high explosives, which
can also be thrown from ordnance in considerable quantities
with safety. The composition is prepared as follows:—­Nitro-glycerine
is thickened with pyroxyline to the consistency of
raw rubber. This is done by employing about 75
to 85 per cent. of nitro-glycerine, and 15 to 25 per
cent. of pyroxyline, according to the stiffness or
elasticity of the compound desired. Some solvent
that dissolves the nitro-cotton is also used.
The product thus formed is a kind of blasting gelatine,
and should be in a pasty condition, in order that it
may be mixed with fulminate of mercury. The solvent
used is acetone, and the quantity of fulminate is
between 75 to 85 per cent. of the entire compound.
If desired, the compound can be made less sensitive
to shocks by giving it a spongy consistency by agitating
it with air while it is still in a syrupy condition.
The nitro-glycerine, especially in this latter case,
may be omitted. In some cases, when it is desirable
to add a deterring medium, nitro-benzene or some suitable
gum is added.

[Illustration: FIG. 34. METHOD OF PREPARING
THE CHARGE.]

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The method of preparing a blasting charge is as follows:—­A
piece of Bickford fuse of the required length is cut
clean and is inserted into a detonator until it reaches
the fulminate. The upper portion of the detonator
is then squeezed round the fuse with a pair of nippers.
The object of this is not only to secure that the
full power of the detonator may be developed, but
also to fix the fuse in the cap (Fig. 34). When
the detonator, &c., is to be used under water, or
in a damp situation, grease or tallow should be placed
round the junction of the cap with the fuse, in order
to make a water-tight joint. A cartridge is then
opened and a hole made in its upper end, and the detonator
pushed in nearly up to the top. Gun-cotton or
tonite cartridges generally have a hole already made
in the end of the charge. Small charges of dry
gun-cotton, known as primers, are generally used to
explode wet gun-cotton. The detonators (which
are often fired by electrical means) are placed inside
these primers (Fig. 35).

[Illustration: FIG. 35. PRIMER.]

One of the forms of electric exploders used is shown
in Fig. 36. This apparatus is made by Messrs
John Davis & Son, and is simply a small hand dynamo,
capable of producing a current of electricity of high
tension. This firm are also makers of various
forms of low tension exploders. A charge having
been prepared, as in Fig. 34, insert into the bore-hole
one or more cartridges as judged necessary, and squeeze
each one down separately with a *wooden* rammer,
so as to leave no space round the charge, and above
this insert the cartridge containing the fuse and
detonator. Now fill up the rest of the bore-hole
with sand, gravel, water, or other tamping. With
gelatine dynamites a firm tamping may be used, but
with ordinary dynamite loose sand is better. The
charge is now ready for firing.

[Illustration: FIG. 36.—­ELECTRIC EXPLODER.]

**CHAPTER VI.**

*SMOKELESS POWDERS.*
Smokeless Powder in General—­Cordite—­Axite—­Ballistite—­U.S.
Naval
Powder—­Schultze’s E.G. Powder—­Indurite—­Vielle
Poudre—­Rifleite—­
Cannonite—­Walsrode—­Cooppal Powders—­Amberite—­Troisdorf—­Maximite—­
Picric Acid Powders, &c., &c.

The progress made in recent years in the manufacture
of smokeless powders has been very great. With
a few exceptions, nearly all these powders are nitro
compounds, and chiefly consist of some form of nitro-cellulose,
either in the form of nitro-cotton or nitro-lignine;
or else contain, in addition to the above, nitro-glycerine,
with very often some such substance as camphor, which
is used to reduce the sensitiveness of the explosive.
Other nitro bodies that are used, or have been proposed,
are nitro-starch, nitro-jute, nitrated paper, nitro-benzene,
di-nitro-benzene, mixed with a large number of other
chemical substances, such as nitrates, chlorates,
&c. And lastly, there are the picrate powders,
consisting of picric acid, either alone or mixed with
other substances.

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The various smokeless powders may be roughly divided
into military and sporting powders. But this
classification is very rough; because although some
of the better known purely military powders are not
suited for use in sporting guns, nearly all the manufacturers
of sporting powders also manufacture a special variety
of their particular explosive, fitted for use in modern
rifles or machine guns, and occasionally, it is claimed,
for big guns also.

Of the purely military powders, the best known are
cordite, ballistite, and the French B.N. powder, the
German smokeless (which contains nitro-glycerine
and nitro-cotton); and among the general powders, two
varieties of which are manufactured either for rifles
or sporting guns, Schultze’s, the E.C.
Powders, Walsrode powder, cannonite, Cooppal powder,
amberite, &c., &c.
 *Cordite*, the smokeless powder adopted by the
British Government, is the patent of the late Sir
F.A. Abel and Sir James Dewar, and is somewhat
similar to blasting gelatine. It is chiefly manufactured
at the Royal Gunpowder Factory at Waltham Abbey, but
also at two or three private factories, including
those of the National Explosives Company Limited, the
New Explosives Company Limited, the Cotton-Powder Company
Limited, Messrs Kynock’s, &c. As first
manufactured it consisted of gun-cotton 37 per cent.,
nitro-glycerine 58 per cent., and vaseline 5 per cent.,
but the modified cordite now made consists of 65 per
cent. gun-cotton, 30 per cent. of nitro-glycerine,
and 5 per cent. of vaseline. The gun-cotton used
is composed chiefly of the hexa-nitrate,[A] which is
not soluble in nitro-glycerine. It is therefore
necessary to use some solvent such as acetone, in
order to form the jelly with nitro-glycerine.
The process of manufacture of cordite is very similar,
as far as the chemical part of the process is concerned,
to that of blasting gelatine, with the exception that
some solvent for the gun-cotton, other than nitro-glycerine
has to be used. Both the nitro-glycerine and
the gun-cotton employed must be as dry as possible,
and the latter should not contain more than .6 per
cent. of mineral matter and not more than 10 per cent.
of soluble nitro-cellulose, and a nitrogen content
of not less than 12.5 per cent. The dry gun-cotton
(about 1 per cent. of moisture) is placed in an incorporating
tank, which consists of a brass-lined box, some of
the acetone is added, and the machine (Fig. 29), is
started; after some time the rest of the acetone is
added (20 per cent. in all) and the paste kneaded for
three and a half hours. At the end of this time
the Vaseline is added, and the kneading continued
for a further three and a half hours. The kneading
machine (Fig. 29) consists of a trough, composed of
two halves of a cylinder, in each of which is a shaft
which carries a revolving blade. These blades
revolve in opposite directions, and one makes about
half the number of revolutions of the other.
As the blades very nearly touch the bottom of the trough,

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any material brought into the machine is divided into
two parts, kneaded against the bottom, then pushed
along the blade, turned over, and completely mixed.
During kneading the acetone gradually penetrates the
mixture, and dissolves both the nitro-cellulose and
nitro-glycerine, and a uniform dough is obtained which
gradually assumes a buff colour. During kneading
the mass becomes heated, and therefore cold water is
passed through the jacket of the machine to prevent
heating the mixture above the normal temperature,
and consequent evaporation of the acetone. The
top of the machine is closed in with a glass door,
in order to prevent as far as possible the evaporation
of the solvent. When the various ingredients are
formed into a homogeneous mass, the mixture is taken
to the press house, where in the form of a plastic
mass it is placed in cylindrical moulds. The
mould is inserted in a specially designed press, and
the cordite paste forced through a die with one or
more holes. The paste is pressed out by hydraulic
pressure, and the long cord is wound on a metal drum
(Fig. 38), or cut into lengths; in either case the
cordite is now sent to the drying houses, and dried
at a temperature of about 100 deg. F. from three
to fourteen days, the time varying with the size.
This operation drives off the acetone, and any moisture
the cordite may still contain, and its diameter decreases
somewhat. In case of the finer cordite, such as
the rifle cordite, the next operation is blending.
This process consists in mounting ten of the metal
drums on a reeling machine similar to those used for
yarns, and winding the ten cords on to one drum.
This operation is known as “ten-stranding.”
Furthermore, six “ten-stranded” reels are
afterwards wound upon one, and the “sixty-stranded”
reel is then ready to be sent away, This is done in
order to obtain a uniform blending of the material.
With cordite of a larger diameter, the cord is cut
into lengths of 12 inches. Every lot of cordite
from each manufacturer has a consecutive number, numbers
representing the size and one or more initial letters
to identify the manufacturer. These regulations
do not apply to the Royal Gunpowder Factory, Waltham
Abbey. The finished cordite resembles a cord of
gutta-percha, and its colour varies from light to dark
brown. It should not look black or shrivelled,
and should always possess sufficient elasticity to
return to its original form after slight bending.
Cordite is practically smokeless. On explosion
a very thin vapour is produced, which is dissipated
rapidly. This smokelessness can be understood
from the fact that the products of combustion are
nearly all non-condensible gases, and contain no solid
products of combustion which would cause smoke.
For the same muzzle velocity a smaller charge of cordite
than gunpowder is required owing to the greater amount
of gas produced. Cordite is very slow in burning
compared to gunpowder. For firing blank cartridges
cordite chips containing no vaseline is used.
The rate at which cordite explodes depends in a measure
upon the diameter of the cords, and the pressure developed
upon its mechanical state. The sizes of cordite
used are given by Colonel Barker, R.A., as follows:—­

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For the .303 rifle .0375 inch diameter.
 " 12 Pr. B.L. gun
 .05 "
 " " .075
 "
 " 4.7-inch Q.F. gun .100
 "
 " 6-inch Q.F. gun .300
 "
 " heavy guns .40
to .50 "

For rifles the cordite is used in bundles of sixty
strands, in field-guns in lengths of 11 to 12 inches,
and the thicker cordite is cut up into 14-inch lengths.
Colonel Barker says that the effect of heat upon cordite
is not greater as regards its shooting qualities than
upon black powder, and in speaking of the effect that
cordite has upon the guns in which it is used (R.A.
Inst.) said that they had at Waltham Abbey a 4.7-inch
Q.F. gun that had fired 40 rounds of black powder,
and 249 rounds of cordite (58 per cent. nitro-glycerine)
and was still in excellent condition, and showed very
little sign of action, and also a 12-lb. B.L.
gun that had been much used and was in no wise injured.

[Footnote A: The gun-cotton used contains 12
per cent. of soluble gun-cotton, and a nitrogen content
of not less than 12.8 to 13.1 per cent.]

[Illustration: Fig. 37 Scale, 1 inch = 1 foot.
Single Strand Reel.]

[Illustration: FIG. 38.—­“TEN-STRANDING.”]

In some experiments made by Captain Sir A. Noble,[A]
with the old cordite containing 58 per cent. nitro-glycerine,
a charge of 5 lbs. 10 oz. of cordite of 0.2 inch diameter
was fired. The mean chamber crusher gauge pressure
was 13.3 tons per square inch (maximum 13.6, minimum
12.9), or a mean of 2,027 atmospheres (max. 2,070,
min. 1,970). The muzzle velocity was 2,146 foot
seconds, and the muzzle energy 1,437 foot tons.
A gramme of cordite generated 700 c.c. of permanent
gases at 0 deg. C. and 760 mm. pressure.
The quantity of heat developed was 1,260 gramme units.
In the case of cordite, as also with ballistite, a
considerable quantity of aqueous vapour has to be
added to the permanent gases formed. A similar
trial, in which 12 lbs. of ordinary pebble powder was
used, gave a pressure of 15.9 tons per square inch,
or a mean of 2,424 atmospheres. It gave a 45-lb.
projectile a mean muzzle velocity of 1,839 foot seconds,
thus developing a muzzle energy of 1,055 foot tons.
A gramme of this powder at 0 deg. C. and 760
mm. generates 280 c.c. of permanent gases, and develops
720 grm. units of heat.

[Footnote A: *Proc. Roy. Soc.*,
vol. lii., No. 315.]

In a series of experiments conducted by the War Office
Chemical Committee on Explosives in 1891, it was conclusively
shown that considerable quantities of cordite may
be burnt away without explosion. A number of
wooden cases, containing 500 to 600 lbs. each of cordite,
were placed upon a large bonfire of wood, and burned
for over a quarter of an hour without explosion.
At Woolwich in 1892 a brown paper packet containing
ten cordite cartridges was fired into with a rifle
(.303) loaded with cordite, without the explosion
of a single one of them, which shows its insensibility
to shock.

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With respect to the action of cordite upon guns, Sir
A. Noble points out that the erosion caused is of
a totally different kind to that of black powder.
The surface of the barrel in the case of cordite appears
to be washed away smoothly by the gases, and not pitted
and eaten into as with black powder. The erosion
also extends over a shorter length of surface, and
in small arms it is said to be no greater than in the
case of black powder. Sir A. Noble says in this
connection: “It is almost unnecessary to
explain that freedom from rapid erosion is of very
high importance in view of the rapid deterioration
of the bores of large guns when fired with charges
developing very high energies. As might perhaps
be anticipated from the higher heat of ballistite,
its erosive power is slightly greater than that of
cordite, while the erosive power of cordite is again
slightly greater than that of brown prismatic.
Amide powder, on the other hand, possesses the peculiarity
of eroding very much less than any other powder with
which I have experimented, its erosive power being
only one-fourth of that of the other powders enumerated.”

TABLE GIVING SOME OF SIR. A. NOBLE’S EXPERIMENTS.
\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_
\_\_\_\_\_\_\_\_\_\_\_\_
| |
| VELOCITIES OBTAINED. |
|\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_
\_\_\_\_\_\_\_\_\_\_\_\_\_|
| | | | | |
| | In a 40 | In a 50 | In a 75 | In a 100 |
| | Cal. Gun.| Cal. Gun.| Cal. Gun.| Cal. Gun.|
|\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_|\_\_\_\_\_\_\_\_\_\_|\_\_\_\_\_\_\_\_\_\_|\_\_\_\_\_\_\_\_
\_\_|\_\_\_\_\_\_\_\_\_\_|
| | | | | |
| |Foot Secs.|Foot Secs.|Foot Sees.|Foot Secs.|
| | | | | |
|With cordite 0.4 in. diam. | 2,794 | 2,940 | 3,166 | 3,286 |
| " " 0.3 " | 2,469 | 2,619 | 2,811 | 2,905 |
| " ballistite 0.3 in. cubes| 2,416 | 2,537 | 2,713 | 2,806 |
| " French B.N. for | | | | |
| 6-inch guns | 2,249 | 2,360 | 2,536 | 2,616 |
| " prismatic amide | 2,218 | 2,342 | 2,511 | 2,574 |
| | | | | |
|\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_|\_\_\_\_\_\_\_\_\_\_|\_\_\_\_\_\_\_\_\_\_|\_\_\_\_\_\_\_\_
\_\_|\_\_\_\_\_\_\_\_\_\_|
| |
| ENERGIES REPRESENTED BY ABOVE VELOCITIES. |
|\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_
\_\_\_\_\_\_\_\_\_\_\_\_\_|
| | | | | |
| |Foot Tons.|Foot Tons.|Foot Tons.|Foot Tons.|
And again, in speaking of his own experiments, he
says: “One 4.7-inch gun has fired 1,219
rounds, and another 953, all with full charges of cordite,
while a 6-inch gun has fired 588 rounds with full charges,
of which 355 were cordite. In the whole of these
guns, so far as I can judge, the erosion is certainly
not greater than with ordinary powder, and differs
from it remarkably in appearance. With ordinary
powder a gun, when much eroded, is deeply furrowed
(these furrows having a great tendency to develop
into cracks), and presents much the appearance in miniature
of a very roughly ploughed field. With cordite,
on the contrary, the surface appears to be pretty
smoothly swept away, while the length of the surface
eroded is considerably less.”

[Illustration: FIG. 39.—­COMPARATIVE
PRESSURE CURVES OF CORDITE AND BLACK POWDER. *a*,
Charge, 48 lbs. powder; *b*, charge, 13 lbs. 4
oz. cordite; *c*, charge, 13 lbs. 4 oz. powder.
Weight of projectile, 100 lbs. in 6-inch gun.
M.V. Cordite = 1960 feet seconds.]

The pressures given by cordite compared with those
given by black powder in the 6-inch gun will be seen
upon reference to Fig. 39, which is taken from Professor
V.B. Lewes’s paper, read before the Society
of Arts; and due to Dr W. Anderson, F.R.S., the Director-General
of Ordnance Factories.

It has been found that the erosive effect is in direct
proportion to the nitro-glycerine present. The
cordite M.D., which contains only 30 per cent. nitro-glycerine,
gives only about half the erosive effect of the old
service cordite. With regard to the heating effect
of cordite and cordite M.D. on a rifle, Mr T.W.
Jones made some experiments. He fired fifty rounds
of .303 cartridges in fifteen minutes in the service
rifle. Cordite raised the temperature of the
rifle 270 deg. F., and cordite M.D. 160 deg.
F. only.

With regard to the effect of heat upon cordite, there
is some difference of opinion. Dr W. Anderson,
F.R.S., says that there is no doubt that the effect
of heat upon cordite is greater than upon black powder.
At a temperature of 110 deg. F. the cordite used
in the 4.7-inch gun is considerably affected as regards
pressure.

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Colonel Barker, R.A., in reply to a question raised
by Colonel Trench, R.A. (at the Royal Artillery Institution),
concerning the shooting qualities of cordite heated
to a temperature of 110 deg. F., said: “Heating
cordite and firing it hot undoubtedly does disturb
its shooting qualities, but as far as we can see,
not much more than gunpowder. I fear that we
must always expect abnormal results with heated propellants,
either gunpowder or cordite; and when fired hot, the
increase in pressure and velocities will depend upon
the heat above the normal or average temperature at
which firing takes place.” Colonel Barker
also, in referring to experiments that had been made
in foreign climates, said: “Climatic trials
have been carried out all over the world, and they
have so far proved eminently satisfactory. The
Arctic cold of the winter in Canada, with the temperature
below zero, and the tropical sun of India, have as
yet failed to shake the stability of the composition,
or abnormally injure its shooting qualities.”
Dr Anderson is of opinion that cordite should not
be stored in naval magazines near to the boilers.
Professor Vivian B. Lewes, in his recent Cantor Lectures
before the Society of Arts, suggests that the magazines
of warships should be water-jacketed, and maintained
at a temperature that does not rise above 100 deg.
F.
 *Axite.*—­This powder is manufactured
by Messrs Kynock Limited, at their works at Witton,
Birmingham. The main constituents of cordite are
retained although the proportions are altered; ingredients
are added which impart properties not possessed by
cordite, and the methods of its manufacture have been
modified. The form has also been altered.
Axite is made in the form of a ribbon, the cross section
being similar in shape to a double-headed rail.
It is claimed for this powder, that it does not corrode
the barrel in the way cordite does, that with equal
pressure it gives greatly increased velocity, and
therefore flatter trajectory. That the effect
of temperature on the pressure and velocity with axite
is only half that with cordite. That the maximum
flame temperature of axite is considerably less than
that of cordite, and the erosive effect is therefore
considerably less. That the deposit left in the
barrel after firing axite cartridges reduces the friction
between the bullet and the barrel. It is therefore
practicable to use axite cartridges giving higher velocities
than can be employed with cordite, as with such velocities
the latter would nickel the barrel by excessive friction.
It is also claimed that the accuracy is greatly increased.
The following results have been obtained with this
same time, and under the same conditions:—­
 *Axite* Cartridges with 200-grain bullets.
 Velocity 2,726 F.S.
 Pressure 20.95 tons.
 *Axite* Cartridges with 215-grain bullets.
 Velocity 2,498 F.S.
 Pressure 19.24 tons.
 *Axite* Service Cartridges.
 Velocity 2,179 F.S.
 Pressure 15.76 tons.

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*Cordite* Service Cartridges.
 Velocity 2,010 F.S.
 Pressure 15.67 tons.

Five rounds from the Service axite and Service cordite
were placed in an oven and heated to a temperature
of 110 deg. F. for one hour, and were then fired
for pressure. The following results were obtained:—­
*Axite.* *Cordite.*
Before heating 15.76 tons per sq. in. 15.67 tons per sq. in.
After " 16.73 " " 17.21 " "
\_\_\_\_\_ \_\_\_\_\_

Increase .97 = 6.1%
 1.54 = 9.8%

Average Velocities—­
 Before heating 2,150 F.S.
 2,030 F.S.
 After " 2,180 "
 2,090 "
 \_\_\_\_\_
 \_\_\_\_\_

Increase 30 F.S. = 1-1/2%
 60.0 F.S. = 3%

In order to show the accuracy given by axite, seven
rounds were fired from a machine rest at a target
fixed at 100 yards from a rifle. Six of the seven
shots could be covered by a penny piece, the other
being just outside. In order to ascertain the
relative heat imparted to a rifle by the explosion
of axite and cordite, ten rounds each of axite and
cordite cartridges were fired from a .303 rifle, at
intervals of ten seconds, the temperature of the rifle
barrel being taken before and after each series:—­

**THE RISE IN TEMPERATURE OF THE RIFLE BARREL**

With axite was 71 deg. F.
With cordite was 89 deg. F.
Difference in favour of axite 18 deg. F. =
20.2%

The lubricating action of axite is shown by the fact
that a series of cordite cartridges fired from a .303
rifle in the ordinary way, followed by a second series,
the barrel being lubricated between each shot by firing
an axite cartridge alternately with the cordite cartridge.
The mean velocity of the first series of cordite cartridges
was 1,974 ft. per second; the mean velocity of the
second series was 2,071 ft. per second; the increased
velocity due to the lubricating effect of axite therefore
was 97 ft. per second. This powder, it is evident,
has very many very excellent qualities, and considerable
advantages over cordite. It is understood that
axite is at present under the consideration of the
British Government for use as the Service powder.
 *Ballistite.*—­Nobel’s powder,
known as ballistite, originally consisted of a camphorated
blasting gelatine, and was made of 10 parts of camphor
in 100 parts of nitro-glycerine, to which 200 parts
of benzol were then added, and 50 parts of nitro-cotton
(soluble) were then steeped in this mixture, which
was then heated to evaporate off the benzol, and the
resulting compound afterwards passed between steam-heated
rollers, and formed into sheets, which were then finally
cut up into small squares or other shapes as convenient.

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The camphor contained in this substance was, however,
found to be a disadvantage, and its use discontinued.
The composition is now 50 per cent. of soluble nitro-cotton
and 50 per cent. of nitro-glycerine. As nitro-glycerine
will not dissolve its own weight of nitro-cotton (even
the soluble variety), benzol is used as a solvent,
but is afterwards removed from the finished product,
just as the acetone is removed from cordite.
About 1 per cent. of diphenylamine is added for the
purpose of increasing its stability.

The colour of ballistite is a darkish brown.
It burns in layers when ignited, and emits sparks.
The size of the cubes into which it is cut is a 0.2-inch
cube. Its density is 1.6. It is also, by
means of a special machine, prepared in the form of
sheets, after being mixed in a wooden trough fitted
with double zinc plates, and subjected to the heating
process by means of hot-water pipes. It is passed
between hot rollers, and rolled into sheets, which
are afterwards put through a cutting machine and granulated.
Sir A. Nobel’s experiments[A] with this powder
gave the following results:—­The charge
used was 5 lbs. 8 oz., the size of the cubes being
0.2 inch. The mean crusher-gauge pressure was
14.3 tons per square inch (maximum, 2,210; minimum,
2,142), and average pressure 2,180 atmospheres.
The muzzle velocity was 2,140 foot seconds, and the
muzzle energy 1,429 foot tons. A gramme of ballistite
generates 615 c.c. of permanent gases, and gives rise
to 1,365 grm. units of heat. Ballistite is manufactured
at Ardeer in Scotland, at Chilworth in Surrey, and
also in Italy, under the name of Filite, which is
in the form of cords instead of cubes. The ballistite
made in Germany contained more nitro-cellulose, and
the finished powder was coated with graphite.
Its use has been discontinued as the Service powder
in Germany, but it is still the Service powder in
Italy.

[Footnote A: *Proc. Roy. Soc.*,
vol. lii., p. 315.]
 *U.S. Naval Smokeless Powder.*—­This
powder is manufactured at the U.S. Naval Torpedo
Station for use in guns of all calibres in the U.S.
Navy. It is a nitro-cellulose powder, a mixture
of insoluble and soluble nitro-cellulose together
with the nitrates of barium and potassium, and a small
percentage of calcium carbonate. The proportions
in the case of the powder for the 6-inch rapid-fire
gun are as follows:—­Mixed nitro-cellulose
(soluble and insoluble) 80 parts, barium nitrate 15
parts, potassium nitrate 4 parts, and calcium carbonate
1 part. The percentage of nitrogen contained
in the insoluble nitro-cellulose must be 13.30+-0.15,
and in the soluble 11.60+-0.15, and the mean nitration
strength of the mixture must be 12.75 per cent. of
nitrogen. The solvent used in making the powder
is a mixture of ether (sp. gr. 0.720) 2 parts, and
alcohol (95 per cent. by volume) 1 part. The
process of manufacture is briefly as follows:[A]—­The
soluble and insoluble nitro-cellulose are dried separately

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at a temperature from 38 deg. to 41 deg. C.,
until they do not contain more than 0.1 per cent.
of moisture. The calcium carbonate is also finely
pulverised and dried, and is added to the mixed nitro-celluloses
after they have been sifted through a 16-mesh sieve.
The nitrates are next weighed out and dissolved in
hot water, and to this solution is added the mixture
of nitro-celluloses and calcium carbonate with constant
stirring until the entire mass becomes a homogeneous
paste. This pasty mass is next spread upon trays
and re-dried at a temperature between 38 deg. and 48
deg. C., and when thoroughly dry it is transferred
to the kneading machine. The ether-alcohol mixture
is now added, and the process of kneading begun.
It has been found by experiment that the amount of
solvent required to secure thorough incorporation
is about 500 c.c. to each 500 grms. of dried paste.
To prevent loss of solvent due to evaporation, the
kneading machine is made vapour light. The mixing
or kneading is continued until the resulting greyish-yellow
paste is absolutely homogeneous so far as can be detected
by the eye, which requires from three to four hours.
The paste is next treated in a preliminary press (known
as the block press and is actuated by hydraulic power),
where it is pressed into a cylindrical mass of uniform
density and of such dimensions as to fit it for the
final or powder press. The cylindrical masses
from the block press are transferred to the final
press, whence they are forced out of a die under a
pressure of about 500 lbs. per square inch. As
it emerges from the final press the powder is in the
form of a ribbon or sheet, the width and thickness
of which is determined by the dimensions of the powder
chamber of the gun in which the powder is to be used.
On the inner surface of the die are ribs extending
in the direction of the powder as it emerges from the
press, the object of these ribs being to score the
sheets or ribbons in the direction of their length,
so that the powder will yield uniformly to the pressure
of the gases generated in the gun during the combustion
of the charge. The ribbon or sheet is next cut
into pieces of a width and length corresponding to
the chamber of the gun for which it is intended, the
general rule being that the thickness of the grain
(when perfectly dry) shall be fifteen one-thousandths
(.015) of the calibre of the gun, and the length equal
to the length to fit the powder chamber. Thus,
in case of the 6-inch rapid-fire gun the thickness
of the grain (or sheet) is 0.09 of an inch and the
length 32 inches. The sheets are next thoroughly
dried, first between sheets of porous blotting-paper
under moderate pressure and at a temperature between
15 deg. C. and 21.5 deg. C. for three days,
and then exposed to free circulation of the air at
about 21.5 deg. C. for seven days, and finally
subjected for a week or longer to a temperature not
exceeding 38 deg. C. until they cease to lose
weight.

[Footnote A: Lieut. W. Walke, “Lectures
on Explosives,” p. 330.]

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The sheets, when thoroughly dried, are of a uniform
yellowish-grey colour, and of the characteristic colloidal
consistency; they possess a perfectly smooth surface,
and are free from internal blisters or cracks.
The temperature of ignition of the finished powder
should not be below 172 deg. C., and when subjected
to the heat or stability test, it is required to resist
exposure to a temperature of 71 deg. C. for thirty
minutes without causing discoloration of the test
paper.
 *W.A. Powder.*—­This powder is
made by the American Smokeless Powder Company, and
it was proposed for use in the United States Army and
Navy. It is made in several grades according
to the ballistic conditions required. It consists
of insoluble gun-cotton and nitro-glycerine, together
with metallic nitrates and an organic substance used
as a deterrent or regulator. The details of its
manufacture are very similar to those of cordite,
with the exception that the nitro-glycerine is dissolved
in a portion of the acetone, before it is added to
the gun-cotton. The powder is pressed into solid
threads, or tubular cords or cylinders, according
to the calibre of the gun in which the powder is to
be used. As the threads emerge from the press
they are received upon a canvas belt, which passes
over steam-heated pipes, and deposited in wire baskets.
The larger cords or cylinders are cut into the proper
lengths and exposed upon trays in the drying-house.
The powder for small arms is granulated by cutting
the threads into short cylinders, which are subsequently
tumbled, dusted, and, if not perfectly dry, again
placed upon trays in the drying-house. Before
being sent away from the factory, from five to ten
lots of 500 lbs. each are mixed in a blending machine,
in order to obtain greater uniformity. The colour
of the W.A. powder is very light grey, the grains
are very uniform in size, dry and hard. The powder
for larger guns is of a yellowish colour, almost translucent,
and almost as hard as vulcanite. The powder is
said to be unaffected by atmospheric or climatic conditions,
to be stable, and to have given excellent ballistic
results; it is not sensitive to the impact of bullets,
and when ignited burns quietly, unless strongly confined.

Turning now to the smokeless powders, in which the
chief ingredient is nitro-cellulose in some form (either
gun-cotton or nitro-lignine, &c.), one of the first
of these was Prentice’s gun-cotton, which consisted
of nitrated paper 15 parts, mixed with 85 parts of
unconverted cellulose. It was rolled into a cylinder.
Another was Punshon’s gun-cotton powder, which
consisted of gun-cotton soaked in a solution of sugar,
and then mixed with a nitrate, such as sodium or potassium
nitrate. Barium nitrate was afterwards used,
and the material was granulated, and consisted of
nitrated gun-cotton.

The explosive known as tonite, made at Faversham,
was at first intended for use as a gunpowder, but
is now only used for blasting.

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*The Schultze Powder.*—­One of the
earliest of the successful powders introduced into
this country was Schultze’s powder, the invention
of Colonel Schultze, of the Prussian Artillery, and
is now manufactured by the Schultze Gunpowder Company
Limited, of London. The composition of this powder,
as given in the “Dictionary of Explosives”
by the late Colonel Cundall, is as follows:—­

Soluble nitro-lignine 14.83 per cent.
Insoluble " 23.36 "
Lignine (unconverted) 13.14 "
Nitrates of K and Ba 32.35 "
Paraffin 3.65 "
Matters soluble in alcohol 0.11 "
Moisture 2.56 "

This powder was the first to solve the difficulty
of making a smokeless, or nearly smokeless powder
which could be used with safety and success in small
arms. Previously, gun-cotton had been tried in
various forms, and in nearly every instance disaster
to the weapon had followed, owing to the difficulty
of taming the combustion to a safe degree. But
about 1866 Colonel Schultze produced, as the result
of experiments, a nitrated wood fibre which gave great
promise of being more pliable and more easily regulated
in its burning than gun-cotton, and this was at once
introduced into England, and the Schultze Gunpowder
Company Limited was formed to commence its manufacture,
which it did in the year 1868. During the years
from its first appearance, Schultze gunpowder has passed
through various modifications. It was first made
in a small cubical grain formed by cutting the actual
fibre of timber transversely, and then breaking this
veneer into cubes. Later on improvements were
introduced, and the wood fibre so produced was crushed
to a fine degree, and then reformed into small irregular
grains. Again, an advance was made in the form
of the wood fibre used, the fibre being broken down
by the action of chemicals under high temperature,
and so producing an extremely pure form of woody fibre.
The next improvement was to render the grains of the
powder practically waterproof and less affected by
the atmospheric influences of moisture and dryness,
and the last improvement to the process was that of
hardening the grains by means of a solvent of nitro-lignine,
so as to do away with the dust that was often formed
from the rubbing of the grains during transit.

Minor modifications have from time to time also been
made, in order to meet the gradual alteration which
has taken place during this long period in the manufacture
of sporting guns and cartridge cases to be used with
this powder, but through all its evolution this Company
has adhered to the first idea of using woody fibre
in preference to cotton as the basis of their smokeless
powder, as experience has confirmed the original opinion
that a powder can be thus made less sensitive to occasional
differences in loading, and more satisfactory all
round than when made from the cotton base. The
powder has always been regulated so that bulk for bulk
it occupies the same measure as the best black powder,
and as regards its weight, just one half of that of
black.

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The process of manufacture of this powder is briefly
as follows:—­

Wood of clean growth is treated by the well-known
sulphite process for producing pure woody fibre, which
is very carefully purified, and this, after drying,
is steeped in a mixture of nitric and sulphuric acids,
to render it a nitro-compound and the explosive base
of the powder. This nitro compound is carefully
purified until it stands the very high purity requirements
of the Home Office, and is then ground with oxygen-bearing
salts, &c., and the whole is formed into little irregular-shaped
grains of the desired size, which grains are dried
and hardened by steeping in a suitable solvent for
the nitro compound, and after finally drying, sifting,
&c., the powder is stored in magazines for several
months before it is issued. When issued, a very
large blend is made of many tons weight, which ensures
absolute uniformity in the material.

There is in England a standard load adopted by every
one for testing a sporting powder; this charge is
42 grains of powder and 1-1/8 oz. No. 6 shot—­this
shot fired from a 12-bore gun, patterns being taken
at 40 yards, the velocity at any required distance.

The standard muzzle velocity of Schultze gunpowder
is 1,220 feet per second.

The mean 40 yards ditto is 875 feet per second.

The mean 20 yards ditto is 1,050 feet per second.

The internal pressure not to exceed 3.5 tons.

This Company also manufactures a new form of powder,
known as Imperial Schultze. It is a powder somewhat
lighter in gravity; 33 grains occupies the bulk charge,
as compared with the 42 grains of the old. It
follows in its composition much the lines of the older
powder, but it is quite free from smoke, and leaves
no residue whatever.
 *The E.G. Powder.*—­This is one
of the oldest of the nitro powders. It was invented
by Reid and Johnson in 1882. It is now manufactured
by the E.G. Powder Company Limited, at their
factory near Dartford, Kent, and in America by the
Anglo-American E.G. Powder Company, at New Jersey.
The basis of this powder is a fine form of cellulose,
derived from cotton, carefully purified, and freed
from all foreign substances, and carefully nitrated.
Its manufacture is somewhat as follows:—­Pure
nitro-cotton, in the form of a fine powder, is rotated
in a drum, sprinkled with water, and the drum rotated
until the nitro-cotton has taken the form of grains.
The grains are then dried and moistened with ether-alcohol,
whereby the moisture is gelatinised, and afterwards
coloured with aurine, which gives them an orange colour.
They are then dried and put through a sieve, in order
to separate the grains which may have stuck together
during the gelatinising process.

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Since its introduction soon after 1881, E.G. powder
has undergone considerable modifications, and is now
a distinctly different product from a practical point
of view. It is now and has been since 1897 what
is known as a 33-grain powder, that is to say, the
old standard charge of 3 drams by measure for a 12-bore
gun weighs 33 grains, as compared with 42 grains for
the original E.G. and other nitro powders. This
improvement was effected by a reduction of the barium
nitrate and the use of nitro-cellulose of a higher
degree of nitration, and also more gelatinisation in
manufacture. The granules are very hard, and resist
moisture to an extent hitherto unattainable by any
“bulk” powder.

Irregularities of pressure in loading have also a
minimum effect by reason of the hardness of the grains.
The colouring matter used is aurine, and the small
quantity of nitrate used is the barium salt. The
powder is standardised for pressure velocity with
Boulenge chronograph,[A] pattern and gravimetric density
by elaborate daily tests, and is continually subjected
to severe trials for stability under various conditions
of storage, the result being that it may be kept for
what in practice amount to indefinite periods of time,
either in cartridges or in bulk without any alteration
being feared. The E.C. powders are used in sporting
guns. No. 1 and No. 2 E.C. are not at present
manufactured, E.C. No. 3 having taken their place
entirely. Since 1890 these powders have been manufactured
under the Borland-Johnson patents, these improved powders
being for some time known as the J.B. powders.
The E.C. No. 1 was superseded by the E.C.
No. 2, made under the Borland-Johnson patents, and
this in its turn by the E.C. No. 3 (in 1897).

[Footnote A: Invented in 1869 by Major Le Boulenge,
Belgian Artillery. It is intended to record the
mean velocity between any two points, and from its
simplicity and accuracy is largely employed. Other
forms have been invented by Capt. Breger, French
Artillerie de la Marine, and Capt. Holden, R.A.]
 *Indurite* is the invention of Professor C.E.
Munroe, of the U.S. Naval Torpedo Station.
It is made from insoluble nitro-cotton, treated in
a particular manner by steam, and mixed with nitro-benzene.
The *Dupont* powder is very similar to *Indurite*.
M.E. Leonard, of the United States, invented
a powder consisting of 75 parts of nitro-glycerine,
25 parts of gun-cotton, 5 parts of lycopodium powder,
and 4 parts of urea crystals dissolved in acetone.
The French smokeless powder, Vielle poudre (poudre
B), used in the Lebel rifle, is a mixture of nitro-cellulose
and tannin, mixed with barium and potassium nitrates.
It gives a very feeble report, and very little bluish
smoke. The Nobel Company is said to be perfecting
a smokeless powder in which the chief ingredients
are nitro-amido- and tri-nitro-benzene. C.O.
Lundholm has patented (U.S. Pat, 701,591, 1901)
a smokeless powder containing nitro-glycerine 30,
nitro-cellulose 60, diamyl phthalate 10 (or diamyl
phthalate 5, and mineral jelly 5). The diamyl
phthalate is added, with or without the mineral jelly
to nitro-glycerine and nitro-cellulose.

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*Walsrode Powder.*—­The smokeless powder
known as Walsrode powder consists of absolutely pure
gelatinised nitro-cellulose, grained by a chemical
not a mechanical process, consequently the grains
do not need facing with gelatine to prevent their
breaking up, as is the case with many nitro powders.
For this same reason, as well as from the method of
getting rid of the solvent used, the Walsrode has
no tendency whatever to absorb moisture. In fact,
it can lie in water for several days, and when taken
out and dried again at a moderate temperature will
be found as good as before. Nor is it influenced
by heat, whether dry or damp, and it can be stored
for years without being in the least affected.
It is claimed also that it heats the barrels of guns
much less than black powder, and does not injure them.

The standard charge is 30 grains, and it is claimed
that with this charge Walsrode powder will prove second
to none. A large cap is necessary, as the grains
of this powder are very hard, and require a large flame
to properly ignite them. In loading cartridges
for sporting purposes, an extra felt wad is required
to compensate for the small space occupied by the
charge; but for military use the powder can be left
quite loose. The gas pressure of this powder
is low (in several military rifles only one-half
that of other nitros), and the recoil consequently
small; and it is claimed that with the slight increase
of the charge (from 29 to 30 grs.) both penetration
and initial velocity will be largely increased, whilst
the gas pressure and recoil will not be greater.

This powder was used at Bisley, at the National Rifle
Association’s Meeting, with satisfactory results.
It is made by the Walsrode Smokeless and Waterproof
Gunpowder Company. The nitro-cotton is gelatinised
by means of acetic ether, and the skin produced retards
burning. The nitro-cotton is mixed with acetic
ether, and when the gelatinisation has taken place,
the plastic mass is forced through holes in a metal
plate into strips, which are then cut up into pieces
the size of grains. The M.H. Walsrode powder
is a leaflet powder, light in colour, about 40 grains
of which give a muzzle velocity of 1,350 feet and
a pressure of 3 tons. It is, like the other Walsrode
powders, waterproof and heat-proof.
 *Cooppal Powder* is manufactured by Messrs Cooppal
& Co. at their extensive powder works in Belgium.
It consists of nitro-jute or nitro-cotton, with or
without nitrates, treated with a solvent to form a
gelatinised mass. There are a great many varieties
of this powder. One kind is in the form of little
squares; another, for use in Hotchkiss guns, is formed
into 3-millimetre cubes, and is black. Other varieties
are coloured with aniline dyes of different colours.

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*Amberite* is a nitro-cellulose powder of the
42-grain type of sporting gunpowders, and is manufactured
by Messrs Curtis’s & Harvey Limited, at their
Smokeless Powder Factory, Tonbridge, Kent. It
consists of a mixture of nitro-cellulose, paraffin,
barium, nitrate, and some other ingredients.
It is claimed for this powder that it combines hard
shooting with safety, great penetration, and moderate
strain on the gun. It is hard and tough in grain,
and may be loaded like black powder, and subjected
to hard friction without breaking into powder, that
it is smokeless, and leaves no residue in the gun.
The charge for 12 bores is 42 grains by weight, and
1-1/8 oz. or 1-1/16 oz. shot. The powders known
as cannonite[A] and ruby powder, also manufactured
by Messrs Curtis’s & Harvey Limited, are analogous
products having the same general characteristics.

[Footnote A: For further details of cannonite,
see First Edition, p. 181.]
 *Smokeless Diamond*, also manufactured by the
above mentioned firm, is a nitro-cellulose powder
of the 33-grain type of sporting gunpowders. It
was invented by Mr H.M. Chapman. The manufacture
of Smokeless Diamond, as carried out at Tonbridge,
is shortly as follows:—­The gun-cotton, which
is the chief ingredient of this powder, is first stoved,
then mixed with certain compounds which act as moderators,
and after the solvents are added, is worked up into
a homogeneous plastic condition. It then undergoes
the processes of granulation, sifting, dusting, drying,
and glazing. In order to ensure uniformity several
batches are blended together, and stored for some
time before being issued for use.

It is claimed for this powder that it is quick of
ignition, the quickness being probably due to the
peculiar structure of the grains which, when looked
at under the microscope, have the appearance of coke.
The charge for a 12 bore is 33 grains and 1-1/16 oz.
shot, which gives a velocity of 1,050 feet per second,
and a pressure of 3 tons per square inch.
 *Greiner’s Powder* consists of nitro-cellulose,
nitro-benzol, graphite, and lampblack.
 *B.N. Powder.*—­This powder is
of a light grey or drab colour, perfectly opaque,
and rough to the touch. It consists of a mixture,
nitro-cellulose and the nitrates of barium and potassium.
Its composition is as follows:—­

Insoluble nitro-cellulose 29.13 parts
Soluble nitro-cellulose 41.31 "
Barium nitrate 19.00 "
Potassium nitrate 7.97 "
Sodium carbonate 2.03 "
Volatile matter 1.43 "

This powder is a modification of the Poudre B., or
Vieille’s powder invented for use in the Lebel
rifle, and which consisted of a mixture of the nitro-celluloses
with paraffin.
 *Von Foster’s Powder* contains nothing
but pure gelatinised nitro-cellulose, together with
a small quantity of carbonate of lime.

The German *Troisdorf Powder* is a mixture of
gelatinised nitro-cellulose, with or without nitrates.

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*Maximite* is the invention of Mr Hudson Maxim,
and is a nitro-compound, the base being gun-cotton.
The exact composition and method of manufacture are,
however, kept secret. It is made by the Columbia
Powder Manufacturing Company, of New York, and in
two forms—­one for use as a smokeless rifle
powder, and the other for blasting purposes.
 *Wetteren Powder.*—­This powder was
manufactured at the Royal Gunpowder Factory at Wetteren,
and used in the Belgian service. Originally it
was a mixture of nitro-glycerine and nitro-cellulose,
with amyl acetate as solvent. Its composition
has, however, been altered from time to time.
One variety consists chiefly of nitro-cellulose, with
amyl acetate as solvent. It is of a dark brown
colour, and of the consistency of indiarubber.
It is rolled into sheets and finally granulated.
 *Henrite* is a nitro-cellulose powder.
 *Normal Powder.*—­The Swedish powder
known as “Normal” Smokeless Powder, and
manufactured by the Swedish Powder Manufacturing Company,
of Landskrona, Sweden, and used for some years past
in the Swiss Army, is made in four forms. For
field guns of 8.4 calibre, it is used in the form
of cylindrical grains of a yellow colour, of a diameter
of .8 to .9 mm. and density of .790—­about
840 grains of it go to one gun. For rifles, it
is used in the form of grey squares, density .750,
and 1 grm. equals about 1,014 grains. One hundred
rounds of this powder, fired in eighteen minutes,
raised the temperature of the gun barrel 284 deg.
F. A nitro-glycerine powder, fired under the same
conditions, gave a temperature of 464 deg. F.

This powder is said to keep well—­a sample
kept 3-1/2 years gave as good results as when first
made—­is easy to make, very stable, ignites
easily, not very sensitive to shock or friction, is
very light, &c. Eight hundred rounds fired from
a heavy gun produced no injury to the interior of the
weapon. Samples kept for eleven months in the
moist atmosphere of a cellar, when fired gave a muzzle
velocity of 1,450 ft. secs. and pressure of 1,312
atmospheres, and the moisture was found to have risen
from 1.2 to 1.6 per cent. After twenty-three
months in the damp it contained 2 per cent. moisture,
gave a muzzle velocity of 1,478 ft. sees., and pressure
of 1,356 atmospheres. In a 7.5 millimetre rifle,
13.8 grm. bullet, and charge of 2 grms., it gives
a muzzle velocity of 2,035 ft. secs. and a pressure
of 2,200 atmospheres. In the 8.4 cm. field-gun,
with charge of 600 grms., and projectile of 6.7 kilogrammes,
muzzle velocity was equal to 1,640 ft. secs. and pressure
1,750. A sample of the powder for use in the .303
M. rifle, lately analysed by the author, gave the
following result:—­

Gun-cotton 96.21 per cent.
Soluble cotton 1.80 "
Non-nitrated cotton trace.
Resin and other matters 1.99 "
 \_\_\_\_\_\_\_

100.00

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The various forms of powder invented and manufactured
by Mr C.F. Hengst are chiefly composed of nitrated
straw that has been finely pulped. The straw
is treated first with acids and afterwards with alkalies,
and the result is a firm fibrous substance which is
granulated. It is claimed that this powder is
entirely smokeless and flameless, that it does not
foul the gun nor heat the barrel, and is at the same
time 150 per cent. stronger than black powder.

The German “Troisdorf” powder consists
of nitro-cellulose that has been gelatinised together
with a nitrate. Kolf’s powder is also gelatinised
with nitro-cellulose. The powders invented by
Mr E.J. Ryves contain nitro-glycerine, nitro-cotton,
castor-oil, paper-pulp, and carbonate of magnesia.
Maxim powder contains both soluble and insoluble nitro-cellulose,
nitro-glycerine, and carbonate of soda. The smokeless
powder made by the “Dynamite Actiengesellschaft
Nobel” consists of nitro-starch 70 to 99 parts,
and of di- or tri-nitro-benzene 1 to 30 parts.

An American wood powder, known as Bracket’s
Sporting Powder, consists of soluble and insoluble
nitro-lignine, mixed with charred lignine, humus,
and nitrate of soda. Mr F.H. Snyder, of New
York, is the inventor of a shell powder known as the
“Snyder Explosive,” consisting of 94 per
cent. nitro-glycerine, 6 per cent. of soluble nitro-cotton,
and camphor, which is said to be safe in use.
Experiments were made with it in a 6-inch rifled gun,
fired at a target 220 yards away, composed of twelve
1-inch steel plates welded together, and backed with
12-inch and 14-inch oak beams, and weighing 20 tons.
The shots entirely destroyed it. The charge of
explosive used was 10 lbs. in each shell.
 *Comparative Tests of Black and Nitro Powders, from
“American Field."*—­ The results
given in table below were obtained at the German Shooting
Association’s grounds at Coepenick, Berlin.
Penetration was calculated by placing frames, each
holding five cards of 1 millimetre in thickness (equals
.03937 inch), and 3 inches apart, in a bee-line, at
distances of 20 inches. Velocity, pattern, and
penetration were taken at 40 yards from the muzzle
of a 12-gauge choke-bore double-barrel gun. Gas
pressure was taken by a special apparatus. All
shells were loaded with 1-1/8 oz. of No. 3 shot, equal
to 120 pellets, and the number given below represents
the average number in the 30-inch pattern. The
number of sheets passed through gives the average
penetration. One atmosphere equals pressure equal
to 1 kilogramme (2.2 lbs.) on the square centimetre,
hence 1,000 atmospheres equal 2,200 lbs. on the square
centimetre. The E.C., Schultze, and Walsrode
powders were loaded in Elcy’s special shells,
2-1/2 inches long. The averages were taken from
a large number of shots, and the same series of shots
fired under precisely the same conditions.

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| | | | | |
| | Gas | | | |
| | Pressure. | Velocity. | Pattern. | Penetration. |
|\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_|\_\_\_\_\_\_\_\_\_\_\_\_|\_\_\_\_\_\_\_\_\_\_\_|\_\_\_\_\_\_\_\_\_\_\_\_|\_\_
\_\_\_\_\_\_\_\_\_\_\_\_|
| | | | | |
| |Atmospheres.| Metres. | | Sheets. |
| | | | | |
|Fine-grained black| | | | |
|powder, standard | | | | |
|charge | 514.2 | 280 | 78.6 = 66% | 19.O |
| | | | | |
|Coarse-grained | | | | |
|black powder, | | | | |
|standard charge | 473.4 | 281.4 | 78.2 = 65% | 19.4 |
| | | | | |
|Schultze powder, | | | | |
|42 grains | 921.0 | 290.0 | 64.2 = 54% | 20.2 |
| | | | | |
|Schultze powder, | | | | |
|45 grains | 1052.8 | 305.8 | 52.2 = 42% | 20.6 |
| | | | | |
|E.G. smokeless, | | | | |
|42 grains | 920.2 | 298.4 | 81.4 = 67% | 18.8 |
| | | | | |
|Walsrode, | | | | |
|29 grains | 586.4 | 280.6 | 83.0 = 69% | 19.0 |
|\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_|\_\_\_\_\_\_\_\_\_\_\_\_|\_\_\_\_\_\_\_\_\_\_\_|\_\_\_\_\_\_\_\_\_\_\_\_|\_\_
\_\_\_\_\_\_\_\_\_\_\_\_|

Barometer, 760 mm. Thermometer, 30 deg.
C. Hydrometer = 65. Wind, S.W.
 *Picric Powders.*—­The chief of these
is *Melinite*, the composition of which is not
known with certainty. It is believed to be melted
picric acid together with gun-cotton dissolved in
acetone or ether-alcohol. Walke gives the following
proportions—­30 parts of tri-nitro-cellulose
dissolved in 45 parts of ether-alcohol (2 to 1), and
70 parts of fused and pulverised picric acid.
The ether-alcohol mixture is allowed to evaporate
spontaneously, and the resulting cake granulated.
The French claim, however, that the original invention
has been so modified and perfected that the melinite
of to-day cannot be recognised in the earlier product.
Melinite has a yellow colour, is almost without crystalline
appearance, and when ignited by a flame or heated
wire, it burns with a reddish-yellow flame, giving
off copious volumes of black smoke. Melinite as
at present used is said to be a perfectly safe explosive,
both as regards manufacture, handling, and storage.

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*Lyddite*,[A] the picric acid explosive used
in the British service, is supposed to be identical
with the original melinite, but its composition has
not been made public.

[Footnote A: Schimose, the Japanese powder, is
stated to be identical with Lyddite and Melinite (*Chem.
Centr.*, 1906, 1, 1196).]

Picrates are more often used than picric acid itself
in powders. One of the best known is *Brugere’s
Powder*, which is a mixture of 54 parts of picrate
of ammonia and 45 parts of saltpetre. It is stable
and safe to manufacture. It has been used in
the Chassepot rifle with good results, gives little
smoke, and a small residue only of carbonate of potash.

The next in importance is *Designolle’s Powder*,
made at Bouchon, consisting of picrate of potash,
saltpetre, and charcoal. It was made in three
varieties, *viz*., for rifles, big guns, and torpedoes
and shells. These powders are made much in the
same way as gunpowder. The advantages claimed
for them over gunpowder are, greater strength, comparative
absence of smoke, and freedom from injurious action
on the bores of guns.
 *Emmensite* is the invention of Dr Stephen Emmens,
of the United States. The Emmens “crystals”
are produced by treating picric acid with fuming nitric
acid of specific gravity of 1.52. The acid dissolves
with the evolution of red fumes. The liquid,
when cooled, deposits crystals, stated to be different
to picric acid, and lustrous flakes. These flakes,
when heated in water, separate into two new bodies.
One of these enters into solution and forms crystals
unlike the first, while the other body remains undissolved.
The acid crystals are used mixed with a nitrate.

Emmensite has been subjected to experiment by the
direction of the U.S. Secretary for War, and
found satisfactory. A sample of Emmensite, in
the form of a coarse powder, was first tried in a
pistol, and proved superior in propelling power to
ordinary gunpowder. When tested against explosive
gelatine, it did very good work in shattering iron
plates. It is claimed for this explosive that
it enjoys the distinction of being the only high explosive
which may be used both for firearms and blasting.
This view is supported by the trials made by the American
War Office authorities, and shows Emmensite to be
a useful explosive both for blasting and as a smokeless
powder. Its explosive power, as tested, is 283
tons per square inch, and its specific gravity is
1.8.

Abel proposed to use picric acid for filling shells.
His *Picric Powder* consisted of 3 parts of saltpetre,
and 2 of picrate of ammonia. *Victorite* consists
of chlorate of potash, picric acid, and olive oil,
and with occasionally some charcoal. It has the
form of a coarse yellowish grey powder, and leaves
an oily stain on paper, and it is very sensitive to
friction and percussion. The composition is as
follows:—­KClO\_{3} = 80 parts; picric acid,
110 parts; saltpetre, 10 parts; charcoal, 5 parts.
It is not manufactured in England. *Tschiner’s
Powder* is very similar to Victorite in composition,
but contains resin. A list of the chief picric
powders will be found in the late Colonel J.P.
Cundill, R.A.’s “Dictionary of Explosives.”

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**CHAPTER VII.**

*ANALYSIS OF EXPLOSIVES.*
Kieselguhr Dynamite—­Gelatine Compounds—­Tonite—­Cordite—­Vaseline—­
Acetone—­Scheme for Analysis of Explosives—­Nitro-Cotton—­Solubility
Test—­ Non-Nitrated Cotton—­Alkalinity—­Ash
and Inorganic Matter—­Determination of Nitrogen—­Lunge,
Champion and Pellet’s, Schultze-Tieman, and Kjeldahl’s
Methods—­Celluloid—­Picric Acid
and Picrates—­Resinous and Tarry Matters—­
Sulphuric Acid and Hydrochloric Acid and Oxalic Acid—­Nitric
Acid—­ Inorganic Impurities—­General
Impurities and Adulterations—­Potassium
Picrate, &c.—­Picrates of the Alkaloids—­Analysis
of Glycerine—­Residue—­ Silver
Test—­Nitration—­Total Acid Equivalent—­Neutrality—­Free
Fatty Acids—­Combined Fatty Acids—­Impurities—­Oleic
Acid—­Sodium Chloride—­ Determination
of Glycerine—­Waste Acids—­Sodium
Nitrate—­Mercury Fulminate—­Cap
Composition—­Table for Correction of Volumes
of Gases, for Temperature and Pressure
 *Kieselguhr Dynamite.*—­The material
generally consists of 75 per cent. of nitro-glycerine
and 25 per cent. of the infusorial earth kieselguhr.
The analysis is very simple, and may be conducted
as follows:—­Weigh out about 10 grms. of
the substance, and place over calcium chloride in a
desiccator for some six to eight days, and then re-weigh.
The loss of weight gives the moisture. This will
generally be very small, probably never more than
1 per cent., and usually less.

Mr James O. Handy, in order to save time, proposes
to dry dynamite in the following manner. He places
1 grm. of the material in a porcelain crucible 1 inch
in diameter. The crucible is then supported at
the bottom of an extra wide-mouthed bottle of about
600 c.c. capacity. Air, which has been dried
by bubbling through strong sulphuric acid, is now drawn
over the surface of the sample for three hours by
means of an ordinary aspirator. The air should
pass approximately at the rate of 10 c.c. per second.
The tube by which the dry air enters the bottle extends
to within 1 inch of the crucible containing the dynamite.
An empty safety bottle is connected with the inlet,
and another with the outlet of the wide-mouthed bottle.
The first guards against the mechanical carrying over
by the air current of sulphuric acid from the acid
bottle into the sample, whilst the second prevents
spasmodic outbursts of water from the exhaust from
reaching the sample. The method also gave satisfactory
results with nitro-glycerine. The dry substance
may now be wrapped in filter paper, the whole weighed,
and the nitro-glycerine extracted in the Soxhlet apparatus
with ether. The ether should be distilled over
at least twenty-four times.

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I have found, however, that much quicker, and quite
as accurate, results may be obtained by leaving the
dynamite in contact with ether in a small Erlenmeyer
flask for twenty-four hours—­leaving it overnight
is better—­ and decanting, and again allowing
the substance to remain in contact with a little fresh
ether for an hour, and finally filtering through a
weighed filter, drying at 100 deg. C., and weighing.
This gives the weight of the kieselguhr. The
nitro-glycerine must be obtained by difference, as
it is quite useless to evaporate down the ethereal
solution to obtain it, as it is itself volatile to
a very considerable extent at the temperature of evaporation
of the ether, and the result, therefore, will always
be much too low. The dry guhr can, of course,
be examined, either qualitatively or quantitatively,
for other mineral salts, such as carbonate of soda,
&c. An actual analysis of dynamite No. 1 made
by the author at Hayle gave—­ Moisture,
0.92 per cent.; kieselguhr, 26.15 per cent.; and nitro-glycerine,
72.93 per cent., the last being obtained by difference.
 *Nitro-Glycerine.*—­It is sometimes
desired to test an explosive substance for nitro-glycerine.
If an oily liquid is oozing from the substance, soak
a drop of it in filter paper. If it is nitro-glycerine
it will make a greasy spot. If the paper is now
placed upon an iron anvil, and struck with an iron
hammer, it will explode with a sharp report, if lighted
it burns with a yellowish to greenish flame, emitting
a crackling sound, and placed upon an iron plate and
heated from beneath, it explodes sharply.

If a few drops of nitro-glycerine are placed in a
test tube, and shaken up with methyl-alcohol (previously
tested with distilled water, to see that it produces
no turbidity), and filtered, on the addition of distilled
water, the solution will become milky, and the nitro-glycerine
will separate out, and finally collect at the bottom
of the tube.

If to a solution of a trace of nitro-glycerine in
methyl-alcohol, a few drops of a solution, composed
of 1 volume of aniline, and 40 volumes sulphuric acid
(1.84) be added, a deep purple colour will be produced.
This colour changes to green upon the addition of water.
If it is necessary to determine the nitro-glycerine
quantitatively in an explosive, the scheme on page
213 may be followed. Ether is the best solvent
to use. Nitrogen should be determined in the
nitrometer.
 *Gelatine Compounds.*—­The simplest
of these compounds is, of course, blasting gelatine,
as it consists of nothing but nitro-cotton and nitro-glycerine,
the nitro-cellulose being dissolved in the glycerine
to form a clear jelly, the usual proportions being
about 92 per cent. of nitro-glycerine to 8 per cent.
nitro-cotton, but the cotton is found as high as 10
per cent. in some gelatines. Gelatine dynamite
and gelignite are blasting gelatines, with varying
proportions of wood-pulp and saltpetre (KNO\_{3}) mixed

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with a thin blasting gelatine. The method of analysis
is as follows:—­Weigh out 10 grms. of the
substance, previously cut up into small pieces with
a platinum spatula, and place over calcium chloride
in a desiccator for some days. Reweigh.
The loss equals moisture. This is generally very
small. Or Handy’s method may be used.
The dried sample is then transferred to a small thistle-headed
funnel which has been cut off from its stem, and the
opening plugged with a little glass wool, and round
the top rim of which a piece of fine platinum wire
has been fastened, in order that it may afterwards
be easily removed from the Soxhlet tube. The
weight of this funnel and the glass wool must be accurately
known. It is then transferred to the Soxhlet
tube and exhausted with ether, which dissolves out
the nitro-glycerine. The weighed residue must
afterwards be treated in a flask with ether-alcohol
to dissolve out the nitro-cotton.

But the more expeditious method, and one quite as
accurate, is to transfer the dried gelatine to a conical
Erlenmeyer flask of about 500 c.c. capacity, and add
250 c.c. of a mixture of ether-alcohol (2 ether to
1 alcohol), and allow to stand overnight. Sometimes
a further addition of ether-alcohol is necessary.
It is always better to add another 300 c.c., and leave
for twenty minutes or so after the solution has been
filtered off. The undissolved portion, which
consists of wood-pulp, potassium nitrate, and other
salts, is filtered off through a linen or paper filter,
dried and weighed.
 *Solution.*—­The ether-alcohol solution
contains the nitro-cotton and the nitro-glycerine
in solution.[A] To this solution add excess of chloroform
(about 100 c.c. will be required), when the nitro-cellulose
will be precipitated in a gelatinous form. This
should be filtered off through a linen filter, and
allowed to drain. It is useless to attempt to
use a filter pump, as it generally causes it to set
solid. The precipitated cotton should then be
redissolved in ether-alcohol, and again precipitated
with chloroform (20 c.c. of ether-alcohol should be
used). This precaution is absolutely necessary,
if the substance has been treated with ether-alcohol
at first instead of ether only, otherwise the results
will be much too high, owing to the gelatinous precipitate
retaining very considerable quantities of nitro-glycerine.
The precipitate is then allowed to drain as completely
as possible, and finally allowed to dry in the air
bath at 40 deg. C., until it is easily detached
from the linen filter by the aid of a spatula, and
is then transferred to a weighed watch-glass, replaced
in the oven, and dried at 40 deg. C. until constant
in weight. The weight found, calculated upon
the 10 grms. taken, gives the percentage of nitro-cellulose.

[Footnote A: If the substance has been treated
with ether alone in the Soxhlet, the nitro-glycerine
will of course be dissolved out first, and the ether-alcohol
solution will only contain the nitro-cellulose.]

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*The Residue* left after treating the gelatine
with ether-alcohol is, in the case of blasting gelatine,
very small, and will probably consist of nothing but
carbonate of soda. It should be dried at 100 deg.
C. and weighed, but in the case of either gelignite
or gelatine dynamite this residue should be transferred
to a beaker and boiled with distilled water, and the
water decanted some eight or ten times, and the residue
finally transferred to a tarred filter and washed
for some time with hot water. The residue left
upon the filter is wood-pulp. This is dried at
100 deg. C. until constant, and weighed.
The solution and washings from the wood are evaporated
down in a platinum dish, and dried at 100 deg.
C. It will consist of the potassium nitrate, and any
other mineral salts, such as carbonate of soda, which
should always be tested for by adding a few drops of
nitric acid and a little water to the residue, and
again evaporating to dryness and re-weighing.
From the difference in weight the soda can be calculated,
sodium nitrate having been formed. Thus—­

Na\_{2}CO\_{3} + 2HNO\_{3} = 2NaNO\_{3} + CO\_{2} + H\_{2}O.

Mol. wt. = 106 = 170

(170 — 106 = 64) and *x* = (106 x *d*)/64

where *x* equals grms. of sodium carbonate in
residue, and *d* equals the difference in weight
of residue, before and after treatment with nitric
acid.

The nitro-glycerine is best found by difference, but
if desired the solutions from the precipitation of
the nitro-cellulose may be evaporated down upon the
water bath at 30 deg. to 40 deg. C., and finally
dried over CaCl\_{2} until no smell of ether or chloroform
can be detected, and the nitro-glycerine weighed.
It will, however, always be much too low. An actual
analysis of a sample of gelatine dynamite gave the
following result:—­

Nitrocellulose (collodion) 3.819 per cent.
Nitro-glycerine 66.691 "
Wood-pulp 16.290 "
KNO\_{3} 12.890 "
Na\_{2}CO\_{3} *Nil.*
Water 0.340 "

This sample was probably intended to contain 30 per
cent. of absorbing material to 70 per cent. of explosive
substances. Many dynamites contain other substances
than the above, such as paraffin, resin, sulphur, wood,
coal-dust, charcoal, also mineral salts, such as carbonate
of magnesia, chlorate of potash, &c. In these
cases the above-described methods must of course be
considerably modified. Paraffin, resin, and most
of the sulphur will be found in the ether solution
if present. The solution should be evaporated
(and in this case the explosive should in the first
case be treated with ether only, and not ether-alcohol),
and the residue weighed, and then treated on the water
bath with a solution of caustic soda. The resin
goes into solution, and is separated by decantation
from the residue, and precipitated by hydrochloric
acid, and collected on a tarred filter (dried at 100
deg. C.), and dried at 100 deg. C. and weighed.
The nitro-glycerine residue is treated with strong
alcohol, decanted, and the residue of paraffin and
sulphur washed with alcohol, dried, and weighed.

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To separate the paraffin from the sulphur the residue
is heated with a solution of ammonium sulphide.
After cooling the paraffin collects as a crust upon
the surface of the liquid, and by pricking a small
hole through it with a glass rod the liquid underneath
can be poured off, and the paraffin then washed with
water, dried, and weighed. Sulphur is found by
difference. Mr F.W. Smith (*Jour.
Amer. Chem. Soc.*, 1901, 23 [8], 585-589)
determines the sulphur in dynamite gelatine as follows:—­About
2 grms. are warmed in a 100 c.c. silver crucible on
the water bath with an alcoholic solution of sodium
hydroxide, and where the nitro-glycerine is decomposed,
the liquid is evaporated to dryness. The residue
is fused with 40 grms. of KOH and 5 grms. of potassium
nitrate, the mass dissolved in dilute acetic acid
and filtered, and the sulphates precipitated in the
usual way. If camphor is present, it can be extracted
with bisulphide of carbon after the material has been
treated with ether-alcohol. In that case the
sulphur, paraffin, and resin will also be dissolved.
The camphor being easily volatile, can be separated
by evaporation. Let the weight of the extract,
freed from ether-alcohol before treatment with bisulphide
of carbon, equal A, and the weight of extract after
treatment with CS\_{2} and evaporation of the same
equal B; and weight of the residue which is left after
evaporation of the CS\_{2} and the camphor in solution
equal C, the percentage of camphor will be A —
B — C. The residue C may contain traces of nitro-glycerine,
resin, or sulphur.

Camphor may be separated from nitro-glycerine by means
of CS\_{2}. If the solution of camphor in nitro-glycerine
be shaken with CS\_{2}, the camphor and a little of
the nitro-glycerine will dissolve. The bisulphide
solution is decanted, or poured into a separating
funnel and separated from the nitro-glycerine.
The two solutions are then heated on the water bath
to 20 deg. C. and then to 60 deg. C., and
afterwards in a vacuum over CaCl\_{2} until the CS\_{2}
has evaporated from them. The camphor evaporates,
and leaves the small quantity of nitro-glycerine which
had been dissolved with it. The other portion
is the nitro-glycerine, now free from CS\_{2}.
The two are weighed and their weights added together,
and equals the nitro-glycerine present. There
is a loss of nitro-glycerine, it being partly evaporated
along with the CS\_{2}. Captain Hess has shown
that it is equal to about 1.25 per cent. This
quantity should therefore be added to that found by
analysis. Morton Liebschutz, in a paper in the
*Moniteur Scientifique* for January 1893, very
rightly observes that the variety of dynamites manufactured
is very great, all of them having a special composition
which, good or bad, is sometimes of so complicated
a nature that the determination of their elements
is difficult.

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The determination of nitro-glycerine in simple dynamite
No. 1 is easy; but not so when the dynamite contains
substances soluble in ether, such as sulphur, resin,
paraffin, and naphthalene. After detailing at
length the methods he employs, he concludes with the
observation that the knowledge of the use of acetic
acid—­in which nitro-glycerine dissolves—­for
the determination of nitro-glycerine may be serviceable.
Mr F.W. Smith[A] gives the following indirect
method of determining nitro-glycerine in gelatine
dynamite, &c. About 15 grms. of the sample are
extracted with chloroform in a Soxhlet apparatus,
and the loss in weight determined. In a second
portion the moisture is determined. A third portion
of about 2 grms. is macerated with ether in a small
beaker, the ethereal extract filtered, and the process
of extraction repeated three or four times. The
united filtrates are allowed to evaporate spontaneously,
and the residue warmed gently on the water bath with
5 c.c. of ammonium sulphide solution, and 10 c.c.
of alcohol until the nitro-glycerine is decomposed,
after which about 250 c.c. of water and sufficient
hydrochloric acid to render the liquid strongly acid,
are added, and the liquid filtered. The precipitate
is washed free from acid, and then washed through the
filter with strong alcohol and chloroform into a weighed
platinum dish, which is dried to constant weight at
50 deg. C. The contents of the dish are now transferred
to a silver crucible, and the sulphur determined.
This amount of sulphur, deducted from the weight of
the contents of the platinum dish, gives the quantity
of substances soluble in chloroform with the exception
of the nitro-glycerine, moisture, and sulphur.
The amount of the former substances *plus* the
moisture and sulphur, deducted from the total loss
on extraction with chloroform, gives the quantity of
nitro-glycerine. Nitro-benzene may be detected,
according to J. Marpurgo, in the following manner:—­In
a porcelain basin are placed two drops of liquid phenol,
three drops of water, and a fragment of potash as
large as a pea. The mixture is boiled, and the
aqueous solution to be tested then added. On prolonged
boiling nitro-benzene produces at the edge of the liquid
a crimson ring, which on the addition of a solution
of bleaching powder turns emerald-green. And
nitro-glycerine in ether solution, by placing a few
drops of the suspected solution, together with a drop
or two of aniline, upon a watch-glass, evaporating
off the ether, and then adding a drop of concentrated
sulphuric acid to the residue, when, if nitro-glycerine
is present, the H\_{2}SO\_{4} will strike a crimson
colour, due to the action of the aniline sulphate
upon the nitric acid liberated from the nitro-glycerine.

[Footnote A: “Notes on the Analysis of
Explosives,” *Jour. Amer. Chem.
Soc.*, 1901, 23 [8], 585-589.]

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*Tonite.*—­The analysis of this explosive
is a comparatively easy matter, and can be performed
as follows:—­Weigh out 10 grms., or a smaller
quantity, and boil with water in a beaker, decanting
the liquid four or five times, and filter. The
aqueous solution will contain the nitrate of barium.
Then put the residue on the filter, and wash two or
three times with boiling water. Evaporate the
filtrate to dryness in a platinum dish. Dry and
weigh. This equals the Ba(NO\_{3})\_{2}. If
the sample is tonite No. 3, and contains di-nitro-benzol,
treat first with ether to dissolve out this substance.
Filter into a dish, and evaporate off the ether, and
weigh the di-nitro-benzol, and afterwards treat residue
with water as before. The residue is dried and
weighed, and equals the gun-cotton present. It
should then be treated with a solution of ether-alcohol
in a conical flask, allowed to stand some three hours,
then filtered through a weighed filter paper, dried
at 40 deg. C., and weighed. This will give
the gun-cotton, and the difference between this last
weight and the previous one will give the collodion-cotton.
A portion of the residue containing both the gun-cotton
and the soluble cotton can be tested in the nitrometer,
and the nitrogen determined.
 *Cordite.*—­This explosive consists
of gun-cotton (with a little collodion-cotton in it
as impurity), nitro-glycerine, and vaseline—­the
proportions being given as 30 per cent. nitro-glycerine,
65 per cent. gun-cotton, and 5 per cent. vaseline.
Its analysis is performed by a modification of the
method given for gelatines. Five grms. may be
dissolved in ether-alcohol in a conical flask, allowed
to stand all night, and then filtered through a linen
filter. The residue is washed with a little ether,
pressed, and dried at 40 deg. C., and weighed.
It equals the gun-cotton. The solution contains
the nitro-glycerine, soluble cotton, and vaseline.
The cotton is precipitated with chloroform, filtered
off, dried, and weighed. The two ether-alcohol
solutions are mixed, and carefully evaporated down
in a platinum dish upon the water bath at a low temperature.
The residue is afterwards treated with strong 80 per
cent. acetic acid, which dissolves out any nitro-glycerine
left in it. The nitro-glycerine is then obtained
by difference, or the method suggested to me privately
by Mr W.J. Williams may be used. The residue
obtained by evaporation of the ether-alcohol solution,
after weighing, is treated with alcoholic potash to
decompose the nitro-glycerine, water is added and the
alcohol evaporated off. Some ether is then added,
and the mixture shaken, and the ether separated and
evaporated, and the residue weighed as vaseline.

The moisture should, however, be determined by the
method devised by Mr Arthur Marshall, F.I.C., of the
Royal Gunpowder Works, Waltham Abbey, which is carried
out as follows:—­The cordite or other explosive
is prepared in the manner laid down for the Abel heat
test, that is t say, it is ground in a small mill,
and that portion is selected which passes through
a sieve having holes of the size of No. 8 wire gauge,
but not through one with holes No. 14 wire gauge.

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[Illustration: FIG. 40.—­MARSHALL’S
APPARATUS FOR MOISTURE IN CORDITE.]

The form of apparatus used is shown in Fig. 40.
It consists of an aluminium dish A, having the dimensions
shown, and the glass cone B weighing not more than
30 grms. Five grms. of the cordite are weighed
into the aluminium dish A. This is covered with the
cone B, and the whole is accurately weighed, and is
then placed upon a metal plate heated by steam from
a water bath. It is left upon the bath until all
the moisture has been driven off, then it is allowed
to cool for about half-an-hour in a desiccator and
is weighed. The loss in weight gives accurately
the moisture of the sample. For cordite of the
original composition, one hour’s heating is
sufficient to entirely drive off the moisture; for
modified cordite containing 65 per cent. of gun-cotton,
two hours is enough, provided that there be not more
than 1.3 per cent. of moisture present.

If the proportion of nitro-glycerine be higher, a
longer heating is necessary. The aluminium dish
must not be shallower than shown in the figure, for
if the distance between the substance and the edge
of the glass cone be less than half an inch, some
nitro-glycerine will be lost. Again, the sample
must not be ground finer than stated, else some of
the moisture will be lost in the grinding and sieving
operations, and the result will be too low. In
order to be able to drive off all the moisture in
the times mentioned, it is essential that the glass
cone shall not fit too closely on the aluminium dish,
consequently the horizontal ledge round the top of
the dish should be bent, so as to render it slightly
untrue, and leave a clearance of about 0.02 inch in
some places. If these few simple precautions
be taken, the method will be found to be very accurate.
Duplicate determinations do not differ more than 0.01
per cent.[A]

[Footnote A: “Determination of Moisture
in Nitro-glycerine Explosives,” by A. Marshall,
*Jour. Soc. Chem. Ind.*, Feb. 29,
1904, p. 154.]
 *The Vaseline* (C\_{16}H\_{34}), or petroleum jelly,
used has a flash-point of 400 deg. F. It must
not contain more than 0.2 per cent. volatile matter
when heated for 12 hours on the water bath, and should
have a specific gravity of 0.87 at 100 deg. F.,
and a melting point of 86 deg. F. It is obtained
during the distillation of petroleum, and consists
mainly of the portions distilling above 200 deg.
C. It boils at about 278 deg. C.
 *Acetone* (CH\_{3}CO.CH\_{3}), or dimethyl ketone,
is formed when iso-propyl alcohol is oxidised with
potassium bichromate and sulphuric. It is also
produced in considerable quantities during the dry
distillation of wood, and many other organic compounds.
Crude wood spirit, which has been freed from acetic
acid, consists in the main of a mixture of acetone
and methyl-alcohol. The two substances may be
roughly separated by the addition of calcium chloride,
which combines with the methyl-alcohol. On subsequent
distillation crude acetone passes over, and may be
purified by conversion into the bisulphite compound.

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Acetone is usually prepared, however, by the dry distillation
of crude calcium or barium acetate.

(CH\_{3}.COO)\_{2}Ca = CH\_{3}.CO.CH\_{3} + CaCO\_{3}.

The distillate is fractionated, and the portion, boiling
between 50 deg. and 60 deg. C., mixed with strong
solution of sodium bisulphite. The crystalline
cake of acetone sodium bisulphite, which separates
on standing, is well pressed, to free it from impurities,
decomposed by distillation with dilute sodium carbonate,
and the aqueous distillate of pure acetone dehydrated
over calcium chloride. Acetone is a colourless,
mobile liquid of sp. gr. .792 at 20 deg. C.,
it boils at 56.5 deg. C., has a peculiar, pleasant,
ethereal odour, and is mixible with water, alcohol,
and ether in all proportions.

The acetone used in the manufacture of cordite should
conform to the following specification:—­

SPECIFICATION FOR ACETONE.

1. The acetone to be not more than 0.802 specific
gravity at 60 deg. F. When mixed with distilled
water it must show no turbidity, and must leave no
residue on evaporation at 212 deg. F. On distillation,
four-fifths by volume of the quantity taken must distil
over at a temperature not exceeding 138 deg.
F. The residual matter left after this distillation
must not contain, besides acetone, any ingredient
that is not a bye-product incidental to the manufacture
of acetone.

2. One c.c. of 0.10 per cent. solution in distilled
water of pure permanganate of potash, added to 100
c.c. of the acetone, must retain its distinctive colour
for not less than 30 minutes. This test should
be made at a temperature of 60 deg. F.

3. The acetone tested by the following method
must not show more than 0.005 per cent. of acid, calculated
to acetic acid:—­

To 50 c.c. of the sample diluted with 50 c.c. of distilled
water, with 2 c.c. of phenol-phthalein solution (1
gramme to 1,000 c.c. of 50 per cent. alcohol) added
as an indicator, add from a burette N/100 sodium hydrate
solution (1 c.c. 0.0006 gramme acetic acid), and calculate
to acetic acid in the usual manner.

The water used for the dilution of the acetone must
be carefully tested for acidity, and the pipettes
used for measuring should not be blown out, as it
would be possible thus to neutralise nearly 2 c.c.
of the soda solution.

The presence of water in a sample of acetone may be
detected by Schweitzer and Lungwitz’s method
(*Chem. Zeit.*, 1895, xix., p. 1384), which
consists in shaking together equal volumes of acetone
and petroleum ether (boiling point, 40 deg. to 60
deg. C.), when if present a separation of the
liquid in layers will take place.
 *Estimation of Acetone.*—­Kebler (*Jour.
Amer. Chem. Soc.*, 1897, 19, 316- 320)
has improved Squibb’s modification of Robineau
and Rollins’ method. The following solutions
are required:—­

(1.) A 6 per cent. solution of hydrochloric acid.

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(2.) A decinormal solution of sodium thiosulphate.

(3.) Alkaline potassium iodide solution prepared by
dissolving 250 grms. of potassium iodide in water,
made up to a litre; dissolving 257 grms. of sodium
hydroxide (by alcohol) in water, likewise made up to
a litre. After allowing the latter to stand,
800 c.c. of the clear solution are added to the litre
of KI.

(4.) Sodium hypochlorite solution: 100 grms.
of bleaching powder (35 per cent.) are mixed with
400 c.c. of water: to this is added a hot solution
of 120 grms. of crystallised sodium carbonate in 400
c.c. of water. After cooling, the clear liquid
is decanted, the remainder filtered, and the filtrate
made up to a litre; to each litre is added 25 c.c.
of sodium hydroxide solution (sp. gr. 1.29).

(5.) An aqueous solution of the acetone, containing
1 or 2 per cent. of acetone.

(6.) Bicarbonated starch solution prepared by treating
0.125 grm. of starch with 5 c.c. of cold water, then
adding 20 c.c. of boiling water, boiling a few minutes,
cooling, and adding 2 grms. of sodium bicarbonate.

To 20 c.c. of the potassium iodide solution are added
10 c.c. of the diluted aqueous acetone, an excess
of the sodium hypochlorite solution is then run in
from a burette and well shaken for a minute. The
mixture is then acidified with the hydrochloric acid
solution, and while agitated, an excess of sodium
thiosulphate solution is added, the mixture being
afterwards allowed to stand a few minutes. The
starch indicator is then added, and the excess of
thiosulphate re-titrated. The relation of the
sodium hypochlorite solution to the sodium thiosulphate
being known, the percentage of acetone can be readily
calculated.[A]

[Footnote A: See “The Testing of Acetone,”
Conroy, *Jour. Soc. Chem. Ind.*,
31st March 1900, vol. xix.]

Dr S.J.M. Auld has recently (*Jour. Chem.
Soc.*, Feb. 15, 1906, vol. xxv.) worked out a volumetric
method for the estimation of acetone, depending on
the formation of bromoform, and its subsequent hydrolysis
with alcoholic potash. The hydrolysis is probably
expressed thus—­

3CHBr\_{3} + 9KOH + C\_{2}H\_{5}OH = 3CO + C\_{2}H\_{4}
+ 9KBr + 7H\_{2}O

as it has been shown by Hermann and Long that exactly
3 volumes of carbon monoxide to 1 of ethylene are
evolved. The residual potassium bromide is estimated
by means of standard silver nitrate solution.
Bromoform is specially suitable for this purpose for
several reasons. It is very readily formed by
the action of bromine and potash on acetone, and although
very volatile in steam, it is not liable to loss due
to its own evaporation. Further, its high molecular
weight and large percentage of bromine conduce to
accurate results, 58 grms. of acetone being responsible
for the formation of 357 grms. of KBr. The method
of carrying out the analysis is as follows:—­

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A known quantity of the solution to be tested, containing
acetone to the extent of 0.1 to 0.2 grm., is pipetted
into a 500 c.c. round-bottom flask, diluted with a
little water, and mixed with 20 to 30 c.c. of a 10
per cent. solution of caustic potash. The flask
is connected with a long reflex condenser, and is
also fitted with a dropping funnel containing a solution
of bromine in potassium bromide (200 grms. of Br and
250 grms. of KBr to 1 litre of water). The bromine
solution is allowed to flow into the mixture until
it has acquired a faint yellow tinge, the flask and
its contents being then heated on the water bath at
about 70 deg. C. for half-an-hour. Bromine
solution is added drop by drop until the slight coloration
is permanent, excess of bromine being got rid of by
boiling for a minute or two with a little more caustic
potash. The mixture is then distilled until the
distillate is free from bromoform, halogen being tested
for in the usual manner. Water is added to the
contents of the flask if necessary. It may be
here observed that no acetone can be detected in the
distillate by means of the mercuric oxide test, and
free bromine is also absent. The condenser having
been washed out with a little alcohol, in order to
remove any traces of bromoform which may have collected,
the distillate and washings are mixed with 50 c.c.
of alcohol and sufficient solid caustic potash to
make an approximately 10 per cent. solution. The
mixture is then heated on the water bath under a reflux
condenser until the bromoform is completely decomposed.
This generally occupies about three-quarters of an
hour. The liquid is allowed to cool, evaporated
to smaller bulk if necessary, and exactly neutralised
with dilute nitric acid. It is then diluted with
water to 500 c.c., and an aliquot part titrated with
N/10 silver nitrate solution, using potassium chromate
as indicator; 240 parts of bromine correspond to 58
parts of acetone. The complete analysis can be
performed in one and a half to two hours. It is
imperative that the bromine used should be pure, as
crude bromine frequently contains bromoform.
The method is suitable for the estimation of acetone
in wood-spirit, the spirit being diluted to 10 times
its volume, and 5 c.c. of this solution employed for
the determination. For example—­

(1.) Three c.c. of a solution containing 9.61 per
cent. acetone gave 1.7850 grm. KBr. Acetone
found = 9.66 per cent.

(2.) Ten c.c. of a solution containing 0.96 per cent.
acetone gave 0.5847 grm. KBr. Acetone found
= 0.95 per cent.
 *Nitro-Cotton.*—­The first thing upon
opening a case of wet cotton, or in receiving a sample
from the “poacher,” that requires to be
determined is the percentage of water that it contains.
It is best done by weighing out about 1,000 grms.
upon a paper tray, which has been previously dried
in the oven at 100 deg. C. for some time, and
become constant in weight. The trayful of cotton
is then placed in a water oven, kept at 100 deg.
C., and dried as long as it loses water. The
loss gives the percentage of water. It varies
from 20 to 30 per cent. as a rule in “wet”
cotton.

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OUTLINE SCHEME FOR THE ANALYSIS OF NITRO-EXPLOSIVES
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| |
| Exhaust dried substance with Anhydrous Ether in Soxhlet’s Fat |
| Extraction Apparatus. |
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\_\_\_\_\_\_\_\_\_\_\_\_|
| |
| *Solution*--Divide into two parts *A.* and *B.* |
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\_\_\_\_\_\_\_\_\_\_\_\_|
| |
| *A.* |
| |
| Allow ether to evaporate spontaneously. Dry residue in vacuo over |
| H\_{2}SO\_{4} and weigh. Equals nitro-glycerine, resin, camphor, and |
| paraffin. |
| |
| The nitro-glycerine in this residue may be decomposed by heating |
| with a solution of alcoholic potash. Water may then be added, and the |
| alcohol evaporated off on the water bath. From this solution the |
| resin may be precipitated by HCl, filtered off, dried, and weighed. |
| Solution containing the paraffin is treated with AmS solution and |
| heated. On cooling the paraffin separates, and may be separated. |
| Residue may be shaken with CS\_{2} to remove camphor. |
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| |
| *B.* |
| |
| Add phenol-phthalein and titrate with alcoholic potash, 1 c.c. normal |
| KHO = .330 grm. *resin*, and add considerably more KHO. Evaporate, |
| dissolve residue in water, shake with ether, and separate. |
|\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_
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| |
| *Ethereal Solution* evaporated leaves paraffin. |
|\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_
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| |
| *Aqueous Solution*-- |
| Add bromide, acidify with HCl, separate any resin and precipitate, |
| filtrate with BaCl\_{2} BaSO\_{4} x .1373 = Sulphur. |
|\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_
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| |
| *Residue*-- |
NOTE.—­Camphor is found by difference.
Sulphur is only partially soluble in ether. It
is better, therefore, to extract some of the original
substance with water, and treat residue with alcoholic
KHO. Add bromide, acidify, and precipitate as
BaSO.

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*The Solubility Test.*—­The object
of this test is to ascertain, in the case of gun-cotton,
the percentage of soluble (penta and lower nitrates)
cotton that it contains, or in the case of soluble
cotton, the quantity of gun-cotton. The method
of procedure is as follows:—­Five grms. of
the sample which has been previously dried at 100
deg. C., and afterwards exposed to the air for
two hours, is transferred to a conical flask, and 250
c.c. ether-alcohol added (2 ether to 1 alcohol).
The flask is then corked and allowed to digest, with
repeated shaking, for two or three hours. The
whole is then transferred to a linen filter, and when
the solution has passed through the filter, is washed
with a little ether, and pressed in a hand-screw press
between folds of filter paper. The sample is then
returned to the flask, and the previous treatment repeated,
but it will be sufficient for it to digest for one
hour the second time. The filter is then again
pressed first gently by hand, then in the press, and
afterwards opened up and the ether allowed to evaporate.
The gun-cotton is then removed from the filter and
transferred to a watch-glass, and dried in the water
oven at 100 deg. C. When dry it is exposed to
the air for two hours and weighed. It equals
the amount of gun-cotton and unconverted cotton in
the 5 grms. The unconverted cotton must be determined
in a separate 5 grms. and deducted.

The method of determining the soluble cotton now used
in the Government laboratories is as follows:—­Fifty
grains of the nitro-cotton are dissolved in 150 c.c.
of ether-alcohol, and allowed to stand, with frequent
shakings, in a 200 c.c. stoppered measure for six hours;
75 c.c. of the clear solution are then drawn off by
the aid of a pipette and evaporated in a dish on the
water bath, and finally in the water oven at 120 deg.
F. (49 deg. C.), until constant in weight.
The weight found equals the quantity of soluble cotton
in the 75 c.c., which, multiplied by 4, equals the
percentage, thus: Suppose that 2.30 grains was
the weight found, then

(2.3 x 150)/75 = 4.6 in 50 = 9.20 per cent.

A method for the determination of soluble nitro-cellulose
in gun-cotton and smokeless powder has been published
by K.B. Quinan (*Jour. Amer. Chem.
Soc.*, 23 [4], 258). In this method about 1
grm. of the finely divided dry sample to be analysed
is placed in an aluminium cup 1.9 inch in diameter
and 4-1/8 inch deep. It is then covered and well
stirred with 50 c.c. of alcohol, 100 c.c. of ether
are then added, and the mixture is stirred for several
minutes. After removing the stirrer, the cup is
lightly covered with an aluminium lid, and is then
placed in the steel cup of a centrifugal machine,
which is gradually got up to a speed of 2,000 revolutions
per minute, the total centrifugal force at the position
occupied by the cups (which become horizontal when
in rapid rotation) is about 450 lbs. They are
rotated at the full speed for ten to twelve minutes,
and the machine is then gradually stopped. By
this time the whole of the insoluble matter will be
at the bottom of the cup, and the supernatant solution
will be clear. It is drawn off to within a quarter
of an inch of the bottom (without disturbing the sediment),
with the aid of a pipette.

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Care must be taken that the solution thus withdrawn
is perfectly clear. About 10 to 15 c.c. of colloid
solution and a film of insoluble matter remain at
the bottom of the cup; these are stirred up well, the
stirrer is rinsed with ether-alcohol, about 50 c.c.
of fresh ether-alcohol are added; the mixture is again
treated in the centrifugal apparatus for about eight
minutes; the whole washing process is then repeated
until all soluble matter has been removed. This
may require about seven or eight (or for samples with
much insoluble matter ten or twelve or more) washings,
but as the extraction proceeds, the period of rotation
may be somewhat reduced. After extraction is
completed, the insoluble matter is transferred to a
Gooch crucible with the usual asbestos pad, dried at
100 deg. C., and weighed. The residue may,
if wished, be dried and weighed in the aluminium cup,
but then it cannot be ignited. The whole time
for an analysis exclusive of that required for drying,
is from one to two hours—­average time, 1-1/4
hour. The results are satisfactory both as to
accuracy and rapidity. Acetone-soluble nitro-cellulose
may be determined by the same method.
 *The Unconverted or Non-nitrated Cotton.*—­However
well the cotton has been nitrated, it is almost certain
to contain a small quantity of non-nitrated or unconverted
cotton. This can be determined thus:—­Five
grms. of the sample are boiled with a saturated solution
of sodium sulphide, and then allowed to stand for
forty-eight hours, and afterwards filtered or decanted,
and again boiled with fresh solutions of sulphide,
and again filtered, washed first with dilute HCl and
then with water, dried, and weighed. The residue
is the cellulose that was not nitrated, plus ash, &c.
It should be ignited, and the weight of the ash deducted
from the previous weight.

Acetone, and acetic-ether (ethyl-acetate) may also
be used as solvents for the nitro-cellulose.
Another process is to boil the gun-cotton, &c., in
a solution of sodium stannate made by adding caustic
soda to a solution of stannous chloride, until the
precipitate first formed is just re-dissolved.
This solution dissolves the cellulose nitrates, but
does not affect the cellulose. Dr Lunge found
the following process more satisfactory in the case
of the more highly nitrated products:—­The
reagent is an alcoholic solution of sodium-ethylate
prepared by dissolving 2 to 3 grms. of sodium in 100
c.c. of 95 per cent. alcohol, and mixing the filtered
solution with 100 c.c. of acetone. It has no effect
upon cellulose, but decomposes nitro-cellulose with
the formation of a reddish brown compound, which is
soluble in water. In the determination, 5 grms.
of gun-cotton are heated to 40 deg. or 50 deg.
C. on the water bath with 150 c.c. of the reagent,
the liquid being shaken at intervals for twenty to
thirty minutes; or the mixture may be allowed to stand
for a few hours at the ordinary temperature.
The brown-red solution is decanted from the undissolved

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residue, and the latter washed with alcohol and with
water, by decantation, and then on the filter with
hot water, to which a little hydrochloric acid is
added for the final washings. For ordinary work
this cellulose is dried immediately and weighed, but
in exact determinations it is washed with alcohol,
again treated with 50 c.c. of the reagent, and separated
and washed as before. The cellulose thus obtained,
gives no trace of gas in the nitrometer, and duplicate
determinations agree within 0.1 to 0.2 per cent. when
the weight of unchanged cellulose amounts to about
0.2 grm. Gun-cotton, which is completely soluble
in acetone, contains only traces of cellulose, and
when as much as 0.85 per cent. is present it does
not dissolve entirely. This method is not applicable
to the determination of cellulose in lower nitrated
products, and Dr Lunge attributes this to the fact
that these being prepared with less concentrated acid
invariably contain oxy-cellulose.
 *Alkalinity.*—­Five grms. of the air-dried
and very finely divided sample are taken from the
centre of the slabs or discs, and digested with about
20 c.c. of N/2 hydrochloric acid, and diluted with
water to about 250 c.c., and shaken for about fifteen
minutes. The liquid is then decanted, and washed
with water until the washings no longer give an acid
reaction. The solution, together with the washings,
are titrated with N/4 sodium carbonate, using litmus
as indicator.
 *Ash and Inorganic Matter.*—­This is
best determined by mixing 2 or 3 grms. of the nitro-cotton
in a platinum crucible with shavings of paraffin,
heating sufficiently to melt the paraffin, and then
allowing the contents of the crucible to catch fire
and burn away quietly. The temperature is then
raised, and the carbonaceous residue incinerated,
cooled, weighed, &c., and the percentage of ash calculated.
Schjerning proceeds in the following way:—­He
takes 5 grms. of the nitro-cotton in a large platinum
crucible, he then moistens it with a mixture of alcohol
and ether, in which paraffin has been dissolved to
saturation, and filtered and mixed with one-fourth
of its volume of water. Some fragments of solid
paraffin are then added, and the ether set on fire.
Whilst this is in progress the crucible is kept in
an oblique position, and is rotated so that the gun-cotton
may absorb the paraffin uniformly. The partially
charred residue is now rubbed down with a rounded glass
rod, and the crucible is covered and heated for from
fifteen to twenty minutes over the blow-pipe, the
lid being occasionally removed. The residue is
soon converted into ash, which is weighed, and then
washed out into a porcelain basin and treated with
hydrochloric acid heated to 90 deg. C. The oxide
of iron, alumina, lime, and magnesia are thus dissolved,
and the silica remains as insoluble residue.
The rest of the analysis is conducted according to
the well-known methods of separation. The percentage
of ash as a whole is generally all that is required.

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*Examination of Nitrated Celluloses with Polarised
Light.*—­Dr G. Lunge (*Jour. Amer.
Chem. Soc.*, 1901, 23 [8], 527) has formed
the following conclusions:—­The most highly
nitrated products appear blue in polarised light,
but those containing between 13.9 and 13.0 per cent.
of nitrogen cannot be distinguished from each other
by polarisation. As the percentage of nitrogen
rises, the blue colour becomes less intense, and here
and there grey fibres can be observed, though not
in proportion to the increase in the nitrogen.
Below 12.4 per cent. of nitrogen, the fibres show
a grey lustre, which usually appears yellow when the
top light is cut off. Below 10 per cent. of nitrogen,
the structure is invariably partially destroyed and
no certain observations possible. It is only possible
to distinguish with certainty, firstly any unchanged
cellulose by its flashing up in variegated (rainbow)
colours; and secondly, highly nitrated products (from
12.75 per cent. N upwards), by their flashing
up less strongly in blue colours. The purple
transition stage in the fibres containing over 11.28
per cent. of N (Chardonnet) was not observed by Dr
Lunge.
 *Determination of Nitrogen by Lunge Nitrometer.*—­The
determination of the percentage of nitrogen in a sample
of gun-cotton or collodion is perhaps of more value,
and affords a better idea of its purity and composition,
than any of the foregoing methods of examination, and
taken in conjunction with the solubility test, it
will generally give the analyst a very fair idea of
the composition of his sample. If we regard gun-cotton
as the hexa-nitro-cellulose, the theoretical amount
of nitrogen required for the formula is 14.14 per
cent., and in the same way for collodion-cotton, which
consists of the lower nitrates, chiefly, however, of
the penta-nitrate, the theoretical nitrogen is 12.75
per cent., so that if in a sample of nitro-cotton
the nitrogen falls much lower than 14 per cent., it
probably contains considerable quantities of the lower
nitrates, and perhaps some non-nitrated cellulose
as well (C\_{6}H\_{10}O\_{5})\_{x}, which of course would
also lower the percentage of nitrogen.

The most expeditious method of determining the nitrogen
in these nitro bodies is by the use of Lunge’s
nitrometer (Fig. 41), and the best way of working
the process is as follows:—­Weigh out with
the greatest care 0.6 grm. of the previously dried
substance in a small weighing bottle of about 15 c.c.
capacity, and carefully add 10 c.c. of concentrated
sulphuric acid from a pipette, and allow to stand
until all the cotton is dissolved. The nitrometer
should be of a capacity 150 to 200 c.c., and should
contain a bulb of 100 c.c. capacity at the top, and
should be fitted with a Greiner and Friederich’s
three-way tap. When the nitro-cotton has entirely
dissolved to a clear solution, raise the pressure tube
of the nitrometer so as to bring the mercury in the
measuring tube close up to the tap. Open the

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tap in order to allow of the escape of any air bubbles,
and clean the surface of the mercury and the inside
of the cup with a small piece of filter paper.
Now close the tap, and pour the solution of the nitro-cotton
into the cup. Rinse out the bottle with 15 c.c.
of sulphuric acid, contained in a pipette, pouring
a little of the acid over the stopper of the weighing
bottle in case some of the solution may be on it.
Now lower the pressure tube a little, just enough
to cause the solution to flow into the bulb of the
measuring tube, when the tap is slightly opened.
When the solution has run in almost to the end, turn
off the tap, wash down the sides of the bottle, and
add to the cup of the nitrometer; allow it to flow
in as before, and then wash down the sides of the cup
with 10 c.c. of sulphuric acid, adding little by little,
and allowing each portion added to flow into the bulb
of the nitrometer before adding the next portion.
Great care is necessary to prevent air bubbles obtaining
admission, and if the pressure tube is lowered too
far, the acid will run with a rush and carry air along
with it.

[Illustration: FIG. 41.—­ORDINARY FORM
OF LUNGE NITROMETER.]

The solution being all in the measuring tube, the
pressure tube is again slightly raised, and the tube
containing the nitro-cotton solution shaken for ten
minutes with considerable violence. It is then
replaced in the clamp, and the pressure relieved by
lowering the pressure tube, and the whole apparatus
allowed to stand for twenty minutes, in order to allow
the gas evolved to assume the temperature of the room.
A thermometer should be hung up close to the bulb
of the measuring tube. At the end of the twenty
minutes, the levels of the mercury in the pressure
and measuring tubes are equalised, and the final adjustment
obtained by slightly opening the tap on the measuring
tube (very slightly), after first adding a little
sulphuric acid to the cup, and observing whether the
acid runs in or moves up. This must be done with
very great care. When accurately adjusted, it
should move neither way. Now read off the volume
of the NO gas in cubic centimetres from the measuring
tube. Read also the thermometer suspended near
the bulb, and take the height of the barometer in millimetres.
The calculation is very simple.

EXAMPLE—­COLLODION-COTTON.

0.6[A] grm. taken. Reading on measuring tube
= 114.6 c.c. NO. Barometer—­ 758
mm. Temperature—­15 deg. C.

[Footnote A: 0.5 grm. is enough in the case of
gun-cotton.]

Since 1 c.c. NO = 0.6272 milligramme N, and correcting
for temperature and pressure by the formula

760 x (1 + *d*^{2}) (*d* = .003665), for
temperature 15 deg. = 801.78,[A]

then

(114.6 x 100 x 750 x .6272)/(801.7 x. 6) = 11.22 per
cent. nitrogen.

[Footnote A: See Table, page 244.]

The nitrogen in nitro-glycerine may of course be determined
by the nitrometer, but in this case it is better to
take a much smaller quantity of the substance.
From 0.1 to 0.2 grm. is quite sufficient. This
will give from 30 to 60 c.c. of gas, and therefore
a measuring tube without a 100 c.c. bulb must be used.

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EXAMPLE.

0.1048 grm. nitroglycerine taken gave 32.5 c.c.
NO. Barometer, 761 mm. Temperature, 15 deg.
C.

Therefore,

(3.25 x 100 x 761 x .6272)/(801.78 x.1048) = 18.46
per cent. N. Theory = 18.50 per cent.

Professor Lunge has devised another form of nitrometer
(Fig. 42), very useful in the nitrogen determination
in explosives. It consists of a measuring tube,
which is widened out in the middle to a bulb, and is
graduated above and below into 1/10 c.c. The capacity
of the whole apparatus is 130 c.c.; that of each portion
of the tube being 30 c.c., and of the bulb 70 c.c.
The upper portion of the graduated tube serves to
measure small volumes of gas, whilst larger volumes
are read off on the lower part.

[Illustration: FIG. 42. FIG. 43. SOME
NEW FORMS OF NITROMETER.]

F.M. Horn (*Zeitschrift fuer angewandte Chemie*,
1892, p. 358) has devised a form of nitrometer (Fig.
43) which he has found especially useful in the examination
of smokeless powders. The tap H is provided with
a wide bore through which a weighed quantity of the
powder is dropped bodily into the bulb K. From 4 to
5 c.c. of sulphuric acid which has been heated to 30
deg. C. are then added through the funnel T,
the tap H being immediately closed. When the
powder has dissolved—­a process which may
be hastened by warming the bulb very carefully—­the
thick solution is drawn into the nitrometer tube N,
and the bulb rinsed several times with fresh acid,
after which operation the analysis is proceeded with
in the usual way.

Dr Lunge’s method of using a separate nitrometer
in which to measure the NO gas evolved to the one
in which the reaction has taken place, the gas being
transferred from the one to the other by joining them
by means of indiarubber tubing, and then driving the
gas over by raising the pressure tube of the one containing
the gas, the taps being open, I have found to be a
great improvement.

1 c.c. NO gas at 0 deg. and 760 mm.
Equals 0.6272 milligrammes (N) nitrogen.
 " 1.343 " nitric oxide.
 " 2.820 " (HNO\_{3}) nitric
acid.
 " 3.805 " (NaNO\_{3}) sodium
nitrate.
 " 4.523 " (KNO\_{3}) potassium
nitrate.
 *Champion and Pellet’s Method.*—­This
method is now very little used. It is based upon
the fact that when nitro-cellulose is boiled with ferrous
chloride and hydrochloric acid, all the nitrogen is
disengaged as nitric oxide (NO). It is performed
as follows:—­A vacuum is made in a flask,
fitted with a funnel tube, with a glass stopper on
the tube; a delivery tube that can also be closed,
and which dips under a solution of caustic soda contained
in a trough, and the end placed under a graduated tube,
also full of caustic soda. From 0.12 to 0.16 grm.
cotton dissolved in 5 to 6 c.c. of sulphuric acid
is allowed to flow into the flask, which contains
the ferrous chloride and hydrochloric acid, and in

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which a vacuum has been formed by boiling, and then
closing the taps. The solution is then heated,
the taps on the delivery tube opened, and the end placed
under the collecting tube, and the NO evolved collected.
The NO gas is not evolved until the solution has become
somewhat concentrated. Eder substituted a solution
of ferrous sulphate in HCl for ferrous chloride.
Care must be taken that the flask used is strong enough
to stand the pressure, or it will burst.

The same chemists (*Compt. Rendus*, lxxxiii.
707) also devised the following method for determining
the NO\_{2} in nitro-glycerine:—­A known
quantity of a solution of ferrous sulphate of previously
ascertained reducing power is placed in a flask, acidified
with hydrochloric acid, and its surface covered with
a layer of petroleum oil. About .5 grm. of the
nitro-glycerine is then introduced, and the flask heated
on the water bath. When the sample is completely
decomposed, the liquid is heated to boiling to remove
nitric oxide, and the excess of ferrous sulphate ascertained
by titration with standard permanganate; 56 of iron
(Fe) oxidised by the sample correspond to 23 of NO\_{2}
in the sample of nitro-glycerine.
 *The Schultze-Tieman Method* for determining
nitrogen in nitro-explosives, especially nitro-cellulose
and nitro-glycerine.—­The figure (No. 44)
shows the general arrangement of the apparatus.
I am indebted for the following description of the
method of working it to my friend, Mr William Bate,
of Hayle. To fill the apparatus with the soda
solution, the gas burette is put on the indiarubber
stopper of basin W, and firmly clamped down. Then
the taps A and C are opened, and B closed. When
the burette is filled with soda solution half-way
up the funnel Y, A and C are closed, and B opened.
The arrows show the inlet and outlet for the cooling
water that is kept running through the water jacket
round the nitrometer tube. To collect the gas,
raise the nitrometer off the rubber stopper, and place
the gas tube from the decomposition apparatus in the
glass dish W and under the opening of the nitrometer.

[Illustration: Fig. 44. SCHULTZE-TIEMAN
APPARATUS.]

For the estimation of nitrogen in nitro-cellulose
take .5 to .65 grm., and place in the decomposition
flask *f* (Fig. 45), washing in with about 25
c.c. of water by alternately opening clips D and E.
The air in the flask is driven out by boiling, whilst
the air is shut off by the tube *i* dipping into
the basin W, which is filled with the soda lye, and
tube K is placed in the test tube R, which contains
a few c.c. of water. As soon as all the air is
completely driven out, clips D and E are closed, and
the gas jet is taken away. (This flask must be a strong
one, or it will burst.) Into test tube R, 25 c.c.
of concentrated solution of protochloride of iron
and 10 to 15 c.c. concentrated hydrochloric acid are
poured, which are sucked up into the developing flask
*f* by opening clip E, air being carefully kept

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from entering. The clip E is now closed, and
tube *i* is put underneath the burette, and the
development of NO gas is commenced by heating the
contents of the flask *f*. When the pressure
of the gas in the flask has become greater than the
pressure of the atmosphere, the connecting tube begins
to swell at *i*, whereupon clip D is opened,
and the boiling continued with frequent shaking of
the bulb, until no more nitrous gas bubbles rise up
into the soda lye, the distilling over of the HCl
causes a crackling noise, the clip D is closed, and
E opened. The burette is again put hermetically
on the indiarubber stopper in basin W, and the apparatus
is left to cool until the water discharged through
P shows the same temperature as the water flowing
through (into the cooling jacket) Z. If the level of
the soda solution in the tube X is now put on exactly
the same level as that in the burette by lowering
or elevating the tube X as required, the volume of
NO obtained in c.c. can be read off within 1/10 c.c.,
and the percentage of nitrogen calculated by the usual
formula.

[Illustration: FIG. 45.—­Decomposition
Flask for Schultze-Tieman Method.]

The solution of protochloride of iron is obtained
by dissolving iron nails, &c., in concentrated HCl,
the iron being in excess. When the development
of hydrogen ceases, it is necessary to filter warm
through a paper filter, and acidify filtrate with
a few drops of HCl. The soda solution used has
a sp. gr. of 1.210 to 1.260; equals 25 deg. to 30 deg.
B. The nitro-cellulose is dried in quantities of 2
grms. at 70 deg. C. during eight to ten hours,
and then three hours in an exiccator over H\_{2}SO\_{4}.
The results obtained with this apparatus are very
accurate. The reaction is founded upon that of
MM. Champion and Pellet’s method.
 *The Kjeldahl Method of Determining Nitrogen.*—­This
method, which has been so largely used by analysts
for the determination of nitrogen in organic bodies,
more especially perhaps in manures, was proposed by
J. Kjeldahl,[A] of the Carlsberg Laboratory of Copenhagen.
It was afterwards modified by Jodlbauer, of Munich,[B]
and applied to the analysis of nitro-explosives by
M. Chenel, of the Laboratoire Centrale des Poudres,
whose method of procedure is as follows:—­0.5
grm. of the finely powdered substance is digested
in the cold with a solution of 1.2 grm. of phenol
and 0.4 grm. phosphoric anhydride in 30 c.c. of sulphuric
acid. The mixture is kept well shaken until the
solution is complete. From 3 to 4 grms. of zinc-dust
is then cautiously and gradually added, the temperature
of the mass being kept down until complete reduction
has been effected. Finally, 0.7 grm. of mercury
is added, and the process continued in the usual way,
according to Kjeldahl; that is, the liquid is distilled
until all the ammonia has passed over, and is absorbed
in the standard acid. The distillate is then
titrated with standard ammonia.

[Footnote A: J. Kjeldahl, *Zeitschrift Anal.
Chem.*, 1883, xxii., p. 366.]

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[Footnote B: Jodlbauer, *Chemisches Centralblatt*,
1886, pp. 434-484. See also *Arms and Explosives*,
1893, p. 87.]

The NO\_{2} group is at the moment of solution fixed
upon the phenol with the production of mono-nitro-phenol,
which is afterwards reduced by the action of the zinc-dust
into the amido derivative. During the subsequent
combustion, the nitrogen of the amido-phenol becomes
fixed in the state of ammonia. M. Chenel is perfectly
satisfied with the results obtained, but he points
out that the success of the operation depends upon
the complete conversion of the phenol into the mono-nitro
derivatives. This takes place whenever the organic
compound forms a *clear solution* in the cold
sulphuric acid mixture. Substances like collodion
or gun-cotton must be very finely divided for successful
treatment. The following table shows some of
the results obtained by M. Chenel:—­

\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_
| | |
| | Total Nitrogen. |
| Substances Analysed. |\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_|
| | | |
| | Calculated. | Found. |
| |\_\_\_\_\_\_\_\_\_\_\_\_\_|\_\_\_\_\_\_\_\_|
| | | |
| Saltpetre (KNO\_{3}) | 13.86 | 13.91 |
| | | 13.82 |
| | | 13.73 |
| | | 13.96 |
| Ammonium nitrate | 35.00 | 35.31 |
| | | 34.90 |
| | | 34.96 |
| Barium nitrate | 10.72 | 10.67 |
| | | 10.62 |
| Nitro-glycerol | 18.50 | 18.45 |
| Di-nitro-benzol[A] | 16.67 | 16.78 |
| | | 16.57 |
| Para-nitro-phenol | 10.07 | 10.03 |
| Picric acid[A] | 18.34 | 18.42 |
| | | 18.43 |
| Ammonium picrate | 22.76 | 22.63 |
| | | 22.67 |
| Di-nitro-ortho-cresol | 14.14 | 14.10 |
| | | 13.98 |
| Tri-nitro-meta-cresol | 17.28 | 17.57 |
| | | 17.27 |
|\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_|\_\_\_\_\_\_\_\_\_\_\_\_\_|\_\_\_\_\_\_\_\_|

[Footnote A: Dr. Bernard Dyer obtained 18.39
per cent. for picric acid and 16.54 per cent. for
di-nitro-benzol.—­*Jour. Chem.
Soc.*, Aug. 1895.]

When Chenel endeavoured to apply Jodlbauer’s
modification of Kjeldahl’s process to the examination
of the tri- and tetra-nitrated naphthalenes, he found
that good results were not obtainable, because these
compounds do not dissolve completely in the cold sulphuric
acid. It may, however, be used if they are previously
converted into the naphthylamines, according to the
plan proposed by D’Aguiar and Lautemann (*Bull.*

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Soc. Chim., vol. iii., new series, p. 256).
This is rapidly effected as follows:—­Twelve
grms. of iodine are gradually added to a solution of
2 grms. of phosphorus in about 15 or 20 c.c. of bisulphide
of carbon, this solution being contained in a flask
of 250 c.c. capacity. The flask and its contents
are heated on the water bath at 100 deg. C. with
constant attention, until the last traces of the carbon
bisulphide have distilled away. It is then cooled,
and the iodide of phosphorus is detached from the sides
of the flask by shaking, but not expelled. The
next step is to add about 0.5 to 0.6 grm. of the substance
that is to be analysed, after which 8 grms. of water
are introduced, and the flask is agitated gently two
or three times. As soon as the reaction becomes
lively, the contents of the flask are well shaken.
It is usually finished about one minute after the addition
of the water. The flask is now cooled, and 25
c.c. of sulphuric acid, together with 0.7 grm. of
mercury, are gradually added; hydriodic acid (HI) forms,
and the temperature of the flask must be raised sufficiently
to expel it. The remaining part of the operation
is as in the ordinary Kjeldahl process.

M. Chenel has found this process the best for the
analysis of the nitro-naphthalenes, and for impervious
substances like collodion or gun-cotton. Personally,
I have never been able to obtain satisfactory results
with this process in the analysis of nitro-cellulose,
and I am of opinion that the process does not possess
any advantage over the nitrometer method, at any rate
for the analysis of gun-cotton.

Table giving the Percentages of Nitrogen and Oxide
of Nitrogen in Various Substances used in or as Explosives:

 Name
 FORMULAE NITROGEN NO\_{2}
 per
cent. per cent.

Nitroglycerine C\_{3}H\_{5}(ONO\_{2})\_{3}
 18.50 = 60.70
Hexa-nitro-cellulose C\_{12}H\_{14}O\_{4}(ONO\_{2})\_{6}
 14.14 = 46.42
Penta-nitro-cellulose C\_{6}H\_{8}O\_{5}(ONO\_{2})\_{5}
 11.11 = 36.50
Nitro-benzene C\_{6}H\_{5}NO\_{2}
 11.38 = 37.39
Di-nitro-benzene C\_{6}H\_{4}(NO\_{2})\_{2}
 16.67 = 54.77
Tri-nitro-benzene C\_{6}H\_{3}(NO\_{2})\_{3}
 19.24 = 63.22
Nitro-toluene C\_{7}H\_{7}NO\_{2}
 10.21 = 33.49
Nitro-naphthalene C\_{10}H\_{7}NO\_{2}
 8.09 = 26.53
Di-nitro-naphthalene C\_{10}H\_{6}(NO\_{2})\_{2}
 12.84 = 42.12
Nitro-mannite C\_{6}H\_{7}(NO\_{3})\_{6}
 23.59 = 77.37
Nitro-starch C\_{6}H\_{8}O\_{4}(HNO\_{3})
 6.76 = 22.18
Picric acid
 (Tri-nitro-phenol) C\_{6}H\_{2}OH(NO\_{2})\_{3}
 18.34 = 60.15
Chloro-nitro-benzene C\_{6}H\_{3}Cl(NO\_{2})\_{2}
 13.82 = 45.43
Ammonium nitrate NH\_{4}NO\_{3}

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35.00 =
Sodium nitrate NaNO\_{3}
 16.47 =
Potassium nitrate KNO\_{3}
 13.86 =
Nitric acid HNO\_{3}
 22.22 =
Barium nitrate Ba(NO\_{3})\_{2}
 10.72 =
 *Analysis of Celluloid.*—­The finely
divided celluloid is well stirred, by means of a platinum
wire, with concentrated sulphuric acid in the cup of
a Lunge nitrometer, and when dissolved the nitrogen
determined in the solution in the usual way.
To prevent interference from camphor, the following
treatment is suggested by H. Zaunschirm (*Chem.
Zeit.*, xiv., 905). Dissolve a weighed quantity
of the celluloid in a mixture of ether-alcohol, mixed
with a weighed quantity of washed and ignited asbestos,
or pumice-stone, dry, and disintegrate the mass, and
afterwards extract the camphor with chloroform, dry,
and weigh: then extract with absolute methyl-alcohol,
evaporate, weigh, and examine the nitro-cellulose in
the nitrometer.
 *Picric Acid and Picrates.*—­Picric
acid is soluble in hot water, and to the extent of
1 part in 100 in cold water, also in ether, chloroform,
glycerine, 10 per cent. soda solution, alcohol, amylic
alcohol, carbon bisulphide, benzene, and petroleum.
If a solution of picric acid be boiled with a strong
solution of potassium cyanide, a deep red liquid is
produced, owing to the formation of potassium iso-purpurate,
which crystallises in small reddish-brown plates with
a beetle-green lustre. This, by reaction with
ammonium chloride, gives ammonium iso-purpurate (NH\_{4}C\_{8}H\_{4}N\_{5}O\_{6}),
or artificial murexide, which dies silk and wool a
beautiful red colour. On adding barium chloride
to either of the above salts, a vermilion-red precipitate
was formed, consisting of barium iso-purpurate.
With ammonio-sulphate of copper, solutions of picric
acid give a bright green precipitate. Mr A.H.
Allen gives the following methods for the assay of
commercial picric acid, in his “Commercial Organic
Analysis":—­
 *Resinous and Tarry matters* are not unfrequently
present. They are left insoluble on dissolving
the sample in boiling water. The separation is
more perfect if the hot solution be exactly neutralised
by caustic soda.
 *Sulphuric Acid, Hydrochloric Acid, and Oxalic Acid*,
and their salts are detected by adding to the filtered
aqueous solution of the sample solutions of the picrates
of barium, silver, and calcium. These salts are
readily made by boiling picric acid with the carbonates
of the respective metals and filtering: other
soluble salts of these methods may be substituted
for the picrates, but they are less satisfactory.
 *Nitric Acid* may be detected by the red fumes
evolved on warming the sample with copper turnings.
 *Inorganic Impurities and Picrates of Potash and
Sodium*, &c., leave residues on cautious ignition.

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*General Impurities and Adulterations* may be
detected and determined by shaking 1 grm. of the sample
of acid in a graduated tube with 25 c.c. of ether,
the pure acid dissolves, while any oxalic acid, nitrates,
picrates, boric acid, alum, sugar, &c., will be left
insoluble, and after removal of the ethereal liquid,
may be readily identified and determined. For
the detection and determination of water and of oxalic
acid, 50 c.c. of warm benzene may be advantageously
substituted for ether. Sugar may be separated
from the other impurities by treating the residue insoluble
in ether or benzene with rectified spirit, in which
sugar and boric acid alone will dissolve. If
boric acid be present, the alcoholic solution will
burn with a green flame. Mono- and di-nitrophenic
acids lower the melting point (122 deg. C).
Their calcium salts are less soluble than the picrate,
and may be approximately separated from it by fractional
crystallisation, or by precipitating the hot saturated
solution of the sample with excess of lime water.
Picric acid may be determined by extracting the acidulated
aqueous solution by agitation with ether or benzene,
and subsequently removing and evaporating off the
solvent. It may also be precipitated as the potassium
salt.
 *Potassium Picrate* [KC\_{6}H\_{2}(NO\_{2})\_{3}O].
When a strong solution of picric acid is neutralised
by carbonate of potash, this salt is thrown down in
yellow crystalline needles, which require 260 parts
of cold or 14 parts of hot water for their solution.
In alcohol it is much less soluble.
 *Ammonium Picrate* is more soluble in water than
the above, and sodium picrate is readily soluble in
water, but nearly insoluble in solution of sodium
carbonate.
 *Picrates of the Alkaloids.*—­Picric
acid forms insoluble salts with many of the alkaloids,
and picric acid may be determined in the following
manner:—­To the solution of picric acid,
or a picrate, add a solution of sulphate of cinchonine
acidulated with H\_{2}SO\_{4}. The precipitated
picrate of cinchonine [C\_{20}H\_{24}N\_{2}O(C\_{6}H\_{2}N\_{3}O\_{7})\_{2}]
is washed with cold water, rinsed off the filter into
a porcelain crucible or dish, the water evaporated
on the water bath, and the residual salt weighed.
Its weight, multiplied by .6123, gives the quantity
of picric acid in the sample taken.
 *Analysis of Glycerine.*[A] Glycerine that is
to be used for the manufacture of nitro-glycerine
should have a minimum specific gravity of 1.261 at
15 deg. C. This can be determined, either by the
aid of a Sartorius specific gravity balance, or by
using an ordinary specific gravity bottle. One
of 10 or 25 c.c. capacity is very convenient.

[Footnote A: See also Sulman and Berry, *Analyst*,
xi., 12-34, and Allen’s “Commercial Organic
Analysis,” vol. ii., part i.]
 *Residue*[A] left upon evaporation should not
be more than 0.25 per cent. To determine this,
take 25 grms. of the glycerine, and evaporate it at
a temperature of about 160 deg. C. in a platinum
basin, and finish in an air bath. Weigh until
constant weight is obtained. Afterwards incinerate
over a bunsen burner, and weigh the ash.

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[Footnote A: Organic matter up to .6 per cent.
is not always prejudicial to the nitrating quantities
of a glycerine.]
 *Silver Test.* A portion of the sample of glycerine
to be tested should be put in a small weighing bottle,
and a quarter of its bulk of N/10 silver nitrate solution
added to it, then shake it, and place in a dark cupboard
for fifteen minutes. It must be pronounced bad
if it becomes black or dark brown within that time
(acrolein, formic, and butyric acids).

The German official test for glycerine for pharmaceutical
purposes is much more stringent, 1 c.c. of glycerine
heated to boiling with 1 c.c. of ammonia solution
and three drops of silver nitrate solution must give
neither colour or precipitate within five minutes.
 *Nitration.* Fifty grms. of the glycerine are
poured from a beaker into a mixture of concentrated
nitric acid (specific gravity 1.53) and sulphuric
acid (1.84), mixed in the proportions of 3 HNO\_{3}
to 5 H\_{2}SO\_{4} (about 400 c.c. of mixed acids).
The mixed acids should be put into a rather large
beaker, and held in the right hand in a basin of water,
and the glycerine slowly poured into them from a smaller
one held in the left. A constant rotatory motion
should be given to the beaker in which the nitration
is performed. When all the glycerine has been
added, and the mixture has been shaken for a few minutes
longer, it is poured into a separator, and allowed
to stand for some time. It should, if the glycerine
is a good one, have separated from the mixed acids
in ten minutes, and the line of demarcation between
the nitro-glycerine and the acid should be clear and
sharp, neither should there be any white flocculent
matter suspended in the liquid. The excess of
acids is now drawn off, and the nitro-glycerine shaken
once or twice with a warm solution of carbonate of
soda, and afterwards with water alone. The nitro-glycerine
is then drawn off into a weighed beaker, the surface
dried with a piece of filter paper, and weighed; 100
parts of a good glycerine should yield about 230 of
nitro-glycerine. A quicker method is to take only
10 c.c. of the glycerine, of which the specific gravity
is already known, nitrate as before, and pour into
a burette, read off the volume of nitro-glycerine in
c.c. and multiply them by 1.6 (the specific gravity
of nitro-glycerine), thus: 10 grms. gave 14.5
c.c. nitro-glycerine, and 14.5 x 1.6 = 23.2 grms.,
therefore 100 would give 232 grms. nitro-glycerine.
The points to be noted in the nitration of a sample
of glycerine are: the separation should be sharp,
and within half an hour or less, and there should be
no white flocculent matter formed, especially when
the carbonate of soda solution is added.
 *Total Acid Equivalent.* Mr G.E. Barton
(*Jour. Amer. Chem. Soc.*, 1895)
proposes to determine thus: 100 c.c. of glycerine
are diluted to 300 c.c. in a beaker, a few drops of
a 1 per cent. solution of phenolphthalein and 10 c.c.
of normal caustic soda solution are added; after boiling,
the liquid is titrated with normal hydrochloric acid
(fatty acids are thus indicated and roughly determined).

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*Neutrality.* The same chemist determines the
neutrality of glycerine thus: 50 c.c. of glycerine
mixed with 100 c.c. of water and a few drops of alcoholic
phenolphthalein[A] are titrated with hydrochloric acid
or sodium hydroxide; not more than 0.3 c.c. normal
hydrochloric acid or normal soda solution should be
required to render the sample neutral; raw glycerines
contain from .5 to 1.0 per cent. of sodium carbonate.

[Footnote A: Sulman and Berry prefer litmus as
indicator.]
 *Determination of Free Fatty Acids.* A weighed
quantity of the glycerine is shaken up with some neutral
ether in a separating funnel, the glycerine allowed
to settle, drawn off, and the ether washed with three
separate lots of water. The water must have been
recently boiled, and be quite free from CO\_{2}.
All the free fatty acid is now in the ether, and no
other soluble acid. A drop of phenolphthalein
is now added, a little water, and the acidity determined
by titration with deci-normal baryta solution, and
the baryta solution taken calculated as oleic acid.
 *Combined Fatty Acid.* About 30 grms. of the
glycerine are placed in a flask, and to it is added
about half a grm. of caustic soda in solution.
The mixture is heated for ten minutes at 150 deg.
C. After cooling some pure ether is added to it, and
enough dilute H\_{2}SO\_{4} to render it distinctly
acid. It is well shaken. All the fatty acids
go into the ether. The aqueous solution is then
removed, and the ether well washed to remove all H\_{2}SO\_{4}.
After the addition of phenolphthalein the acid is
titrated, and the amount used calculated into oleic
acid. From this total amount of fatty acids the
free fatty acid is deducted, and the quantity of combined
fatty acids thus obtained.
 *Impurities.* The following impurities may be
found in bad samples of glycerine:—­Lead,
arsenic, lime, chlorine, sulphuric acid, thio-sulphates,
sulphides, cyanogen compounds, organic acids (especially
oleic acid and fatty acids[A]), rosin products, and
other organic bodies. It is also said to be adulterated
with sugar and glucose dextrine. Traces of sulphuric
acid and arsenic may be allowed, also very small traces
indeed of lime and chlorine.

[Footnote A: These substances often cause trouble
in nitrating, white flocculent matter being formed
during the process of washing.]

The organic acids, formic and butyric acids may be
detected by heating a sample of the glycerine in a
test tube with alcohol and sulphuric acid, when, if
present, compound ethers, such as ethylic formate and
butyrate, the former smelling like peaches and the
latter of pine-apple, will be formed.
 *Oleic Acid*, if present in large quantity, will
come down upon diluting the sample with water, but
smaller quantities may be detected by passing a current
of nitrogen peroxide, N\_{2}O\_{4} (obtained by heating
lead nitrate), through the diluted sample, when a
white flocculent precipitate of elaidic acid, which
is less soluble than oleic acid, will be thrown down.
By agitating glycerol with chloroform, fatty acids,
rosin oil, and some other impurities are dissolved,
while certain others form a turbid layer between the
chloroform and the supernatant liquid. On separating
the chloroform and evaporating it to dryness, a residue
is obtained which may be further examined.

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*Sodium Chloride* can be determined in 100 c.c.
of the glycerine by adding a little water, neutralised
with sodium carbonate, and then titrated with a deci-normal
solution of silver nitrate, using potassium chromate
as indicator.
 *Organic Impurities* of various kinds occur in
crude glycerine, and are mostly objectionable.
Their sum may be determined with fair accuracy by
Sulman and Berry’s method: 50 grms. of the
sample are diluted with twice its measure of water,
carefully neutralised with acetic acid, and warmed
to expel carbonic acid; when cold, a solution of basic
lead acetate is added in slight but distinct excess,
and the mixture well agitated. The formation
of an abundant precipitate, which rapidly subsides,
is an indication of considerable impurity in the sample.
To ascertain its amount, the precipitate is first
washed by decantation, and then collected on a tared,
or preferably a double counter-poised filter, where
it is further washed, dried at 100 deg. to 105 deg.
C., and weighed. The precipitate and filter paper
are then ignited separately in porcelain, at a low
red heat, the residues moistened with a few drops
of nitric acid and reignited; the weight of the lead
oxide deducted from that of the original precipitate
gives the weight of the organic matter precipitated
by the lead. Raw glycerines contain from 0.5
to 1.0 per cent.
 *Albuminous Matters.* An approximate determination
of the albuminous matters may be made by precipitating
with basic lead acetate as already described, and
determining the nitrogen by the Kjeldahl method; the
nitrogen multiplied by 6.25 gives the amount of albuminous
matter in the precipitate.
 *The Determination of Glycerine.* The acetin
method of Benedikt and Canton depends upon the conversion
of glycerine into triacetin, and the saponification
of the latter, and reduces the estimation of glycerine
to an acidmetric method. About 1.5 grm. of crude
glycerine is heated to boiling with 7 grms. of acetic
anhydride, and 3 to 4 grms. of anhydrous sodium acetate,
under an upright condenser for one and a half hours.
After cooling, 50 c.c. of water are added, and the
mixture heated until all the triacetin has dissolved.
The liquid is then filtered into a large flask, the
residue on the filter is well washed with water, the
filtrate quite cooled, phenolphthalein is added and
the fluid exactly neutralised with a dilute (2 to
3 per cent.) solution of alkali. Twenty-five c.c.
of a 10 per cent. caustic soda solution, which must
be accurately standardised upon normal acid, are then
pipetted into the liquid, which is heated to boiling
for ten minutes to saponify the triacetin, and the
excess of alkali is then titrated back with normal
acid. One c.c. of normal acid corresponds to
.03067 grm. of glycerine.
 *Precautions.*—­The heating must be
done with a reflux condenser, the triacetin being
somewhat volatile. The sodium acetate used must
be quite anhydrous, or the conversion of the glycerine
to triacetyl is imperfect. Triacetin in contact
with water gradually decomposes. After acetylation
is complete, therefore, the operations must be conducted
as rapidly as possible. It is necessary to neutralise
the free acetic acid very cautiously, and with rapid
agitation, so that the alkali may not be locally in
excess.

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*The Lead Oxide Method.*—­Two grms.
of sample are mixed with about 40 grms. of pure litharge,
and heated in an air bath to 130 deg. C. until
the weight becomes constant, care being taken that
the litharge is free from such lead compounds and
other substances as might injuriously affect the results,
and that the heating of the mixture takes place in
an air bath free from carbonic acid. The increase
in weight in the litharge, minus the weight of substance
not volatilisable from 2 grms. of glycerine at 160
deg. C., multiplied by the factor 1.243, is taken
as the weight of glycerine in the 2 grms. of sample.
The glycerine must be fairly pure, and free from resinous
substances and SO\_{3}, to give good results by this
process.
 *Analysis of the “Waste Acids” from
the Manufacture of Nitro-Glycerine or Gun-Cotton.*
Determine the specific gravity by the specific gravity
bottle or hydrometer, and the oxides of nitrogen by
the permanganate method described under nitro-glycerine.
Now determine the total acidity of the mixture by
means of a tenth normal solution of sodium hydrate,
and calculate it as nitric acid (HNO\_{3}), then determine
the nitric acid by means of Lunge nitrometer, and
subtract percentage found from total acidity, and
calculate the difference into sulphuric acid, thus:—­

Total acidity equals 97.46 per cent.—­11.07
per cent. HNO\_{3} = 86.39 per cent., then (86.39
x 49)/63 = 67.20 per cent. H\_{2}SO\_{4}.

Then analysis of sample will be:—­

\_
Sulphuric acid = 67.20 per cent. |
Nitric acid = 11.07 " |- Specific gravity = 1.7075.
Water = 12.73 " \_|

This method is accurate enough for general use in
the nitric acid factory. The acid mixture may
be taken by volume for determining nitric oxide in
nitrometer. Two c.c. is a convenient quantity
in the above case, then 2 x 1.7075 (specific gravity)
= 3.414 grms. taken, gave 145 c.c. NO (barometer
= 748 mm, and temperature = 15 deg.C.) equals 134.9
c.c. (corr.) and as 1 c.c. NO = .0282 grm.
HNO\_{3} 135 x .0282 = .378 grm. = 11.07 per cent. nitric
acid.
 *Sodium Nitrate.* Determine moisture and chlorine
by the usual methods, and the total, NaNO\_{3}, by
means of nitrometer—­0.45 grm. is a very
convenient quantity to work on (gives about 123 c.c.
gas); grind very fine, and dissolve in a very little
hot water in the cup of the nitrometer; use about
15 c.c. concentrated H\_{2}SO\_{4}. One cubic cent.
of NO equals .003805 grm. of NaNO\_{3}. The insoluble
matter, both organic and inorganic, should also be
determined, also sulphate of soda and lime tested
for.

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*Analysis of Mercury Fulminate (Divers and Kawakita’s
Method).*—­A weighed quantity of mercury
fulminate is added to excess, but measured quantity
of fuming hydrochloric acid contained in a retort
connected with a receiver holding water. After
heating for some time, the contents of the retort and
receiver are mixed and diluted, and the mercury is
precipitated by hydrogen sulphide. By warming
and exposure to the air in open vessels the hydrogen
sulphide is for the most part dissipated. The
solution is then titrated with potassium hydroxide
(KOH), as well as another quantity of hydrochloric
acid, equal to that used with the fulminate. As
the mercury chloride is reconverted into hydrochloric
acid by the hydrogen sulphide, and as the hydroxylamine
does not neutralise to litmus the hydrochloric acid
combined with it, there is an equal amount of hydrochloric
acid free or available in the two solutions.
Any excess of acid in the one which has received the
fulminate will therefore be due to the formic acid
generated from the fulminate. Dr. Divers and
M. Kawakita, working by this method, have obtained
31.31 per cent. formic acid, instead of 32.40 required
by theory. (*Jour. Chem. Soc.*, p.
17, 1884.)

Divers and Kawakita proceed thus: 2.351 grms.
dissolved, as already described, in HCl, and afterwards
diluted, gave mercury sulphide equal to 70.40 per
cent. mercury. The same solution, after removal
of mercury, titrated by iodine for hydroxylamine,
gave nitrogen equal to 9.85 per cent., and when evaporated
with hydroxyl ammonium chloride equal to 9.55 per
cent. A solution of 2.6665 grms. fulminate in
HCl of known amount, after removal of mercury by hydrogen
sulphide, gave by titration with potassium hydrate,
formic acid equal to 8.17 per cent. of carbon.
Collecting and comparing with calculation from formula
we get—­

Calc. I. II.
 III.

Mercury 70.42 70.40 ... ...
Nitrogen 9.86 9.85 9.55 ...
Carbon 8.45 ... ... 8.17
Oxygen 11.27 ... ... ...
 \_\_\_\_\_\_\_

100.00
 *The Analysis of Cap Composition.*—­Messrs
F.W. Jones and F.A. Willcox (*Chem.
News*, Dec. 11, 1896) have proposed the following
process for the analysis of this substance:—­Cap
composition usually consists of the ingredients—­potassium
chlorate, antimony sulphide, and mercury fulminate,
and to estimate these substances in the presence of
each other by ordinary analytical methods is a difficult
process. Since the separation of antimony sulphide
and mercury fulminate in the presence of potassium
chlorate necessitates the treatment of the mixture
with hydrochloric acid, and this produces an evolution
of hydrogen sulphide from the sulphide, and a consequent
precipitation of sulphur; and potassium chlorate cannot
be separated from the other ingredients by treatment
with water, owing to the appreciable solubility of
mercury fulminate in cold water.

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In the course of some experiments on the solubility
of mercury fulminate Messrs Jones and Willcox observed
that this body was readily soluble in acetone and
other ethereal solvents when they were saturated with
ammonia gas, and that chlorate of potash and sulphide
of antimony were insoluble in pure acetone saturated
with ammonia; these observations at once afforded
a simple method of separating the three ingredients
of cap composition. By employing this solution
of acetone and ammonia an analysis can be made in
a comparatively short time, and yields results of
sufficient accuracy for all technical purposes.
The following are the details of the process:—­

A tared filter paper is placed in a funnel to the
neck of which has been fitted a piece of rubber tubing
provided with a clip. The paper is moistened
with a solution of acetone and ammonia, the cap composition
is weighed off directly on to the filter paper and
is then covered with the solution of acetone and ammonia
and allowed to stand thirty-four hours. It is
then washed repeatedly with the same solution until
the washings give no coloration with ammonium sulphide,
and afterwards washed with acetone until washings
give no residue on evaporation dried and weighed.
The paper is again put in the funnel and washed with
water until free from potassium chlorate, dried and
weighed.

If *c* = weight of composition taken, *d* = " " filter paper, *a* = " after first extraction, *b* = " " second extraction,
 then *c+d-a* = weight of
fulminate, *c+d-a-b*
= " " KClO\_{3}, *b-d*
 = " " sulphide of antimony.

The composition should be finely ground in an agate
mortar.

The results of the analysis by this method of two
mixtures of known composition are given below—­

\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_
\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_
| | | |
| | A | B |
| | | |
| | Percentage | Percentage | Percentage | Percentage |
| | Taken. | Found. | Taken. | Found. |
|\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_|\_\_\_\_\_\_\_\_\_\_\_\_|\_\_\_\_\_\_\_\_\_\_\_\_|\_\_\_\_\_\_\_\_\_\_\_\_
|\_\_\_\_\_\_\_\_\_\_\_\_|
| | | | | |
| Antimony Sulphide | 36.47 | 36.25 | 37.34 | 37.22 |
| Potassium Chlorate | 33.25 | 33.71 | 46.03 | 46.43 |
| Mercury Fulminate | 30.27 | 30.02 | 16.61 | 16.34 |
|\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_|\_\_\_\_\_\_\_\_\_\_\_\_|\_\_\_\_\_\_\_\_\_\_\_\_|\_\_\_\_\_\_\_\_\_\_\_\_
|\_\_\_\_\_\_\_\_\_\_\_\_|

Dr. H.W. Brownsdon’s (*Jour. Soc.
Chem. Ind.*, xxiv., April 1905) process is
as follows:—­The cap composition is removed
by squeezing the cap with pliers, while held over

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a porcelain basin of about 200 c.c. capacity, and
removing the loosened foil and broken composition by
means of a pointed wooden chip. Composition adhering
to the shell or foil is loosened by alcohol, and washed
into the dish by means of alcohol in a small wash
bottle. The shell and foil are put to one side
and subsequently weighed when dry. The composition
in the dish is broken down quite fine with a flat-headed
glass rod, and the alcohol evaporated on the water
bath till the residue is moist, but not quite dry,
25 c.c. of water are then added, and the composition
well stirred from the bottom. After the addition
of 0.5 grm. of pure sodium, thiosulphate, the contents
of the dish, is well stirred for two and a half minutes.
One drop of methyl orange is then added, and the solution
titrated with N/20 sulphuric acid, which has been
standardised against weighings of 0.05-0.1 grm. fulminate
to which 25 c.c. of water is added in a porcelain
dish, then 0.5 grm. of thiosulphate, and after stirring
for two and a half minutes, titrated with N/20 sulphuric
acid. The small amount of antimony sulphide present
does not interfere with the recognition of the end
point. After titration, the solution is filtered
through a small 5-1/2 cm. filter paper, which retains
the antimony sulphide. The filter paper containing
the Sb\_{2}S\_{3} is well washed and then transferred
to a large 6 by 1 test tube. Five c.c. of strong
hydrochloric acid are added, and the contents of the
tube boiled gently for a few seconds until the sulphide
is dissolved and all the H\_{2}S driven off or decomposed:
2-3 c.c. of a saturated solution of tartaric acid
are added, and the contents of the tube washed into
a 250 c.c. Erlenmeyer flask. The solution
is then nearly neutralised with sodium carbonate,
excess of bi-carbonate added, and after the addition
of some starch solution titrated with N/20 iodine
solution. This method for small quantities of
stibnite is both quick and accurate, the error being
about +-0.0003 grm. Sb\_{2}S\_{3} at the outside.

The tendency of this method is to give slightly low
figures for the fulminate, but since these are uniform
within a negligible error, it does not affect the
value of the results as a criterion of uniformity.
The following test results were obtained by Dr Brownsdon:—­

\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_
\_\_\_\_\_
| | | |
| Fulminate Taken. | Fulminate Found. | Error. |
| Grm. | Grm. | Grm. |
| | | |
| 0.0086 | 0.0083 | -0.0003 |
| 0.0082 | 0.0081 | -0.0001 |
| 0.0074 | 0.0071 | -0.0003 |
| 0.0068 | 0.0066 | -0.0002 |
|\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_|\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_|\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_
\_|
| | | |
TABLE FOR CORRECTION OF VOLUMES OF GASES FOR TEMPERATURE,
GIVING THE DIVISOR FOR THE FORMULA.

V\_{1} = V x B/(760 x (1 + dt)) (d = 0.003665) 1 +
dt from 0 deg. to 30 deg. C.

\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_
\_\_\_\_
| | | | |
t. | 760x(1+dt). | t. | 760x(1+dt). | t. | 760x(1+dt).
\_\_\_\_\_|\_\_\_\_\_\_\_\_\_\_\_\_\_|\_\_\_\_\_|\_\_\_\_\_\_\_\_\_\_\_\_\_|\_\_\_\_\_|\_\_\_\_\_\_\_\_\_\_\_\_\_
| | | | |
deg.C. | | deg.C. | | deg.C. |
0.0 | 750.000 | 1.7 | 764.7352 | 3.4 | 769.4704
.1 | 760.2785 | .8 | 765.0137 | .5 | 769.7489
.2 | 760.5571 | .9 | 765.2923 | .6 | 770.0274
.3 | 760.8356 | 2.0 | 765.5708 | .7 | 770.3060
.4 | 761.1142 | .1 | 765.8493 | .8 | 770.5845
.5 | 761.3927 | .2 | 766.1279 | .9 | 770.8631
.6 | 761.6712 | .3 | 766.4064 | 4.0 | 771.1416
.7 | 761.9498 | .4 | 766.6850 | .1 | 771.4201
.8 | 762.2283 | .5 | 766.9635 | .2 | 771.6987
.9 | 762.5069 | .6 | 767.2420 | .3 | 771.9772
1.0 | 762.7854 | .7 | 767.5206 | .4 | 772.2558
.1 | 763.0639 | .8 | 767.7991 | .5 | 772.5343
.2 | 763.3425 | .9 | 768.0777 | .6 | 772.8128
.3 | 763.6210 | 3.0 | 768.3562 | .7 | 773.0914
.4 | 763.8996 | .1 | 768.6347 | .8 | 773.3699
.5 | 764.1781 | .2 | 768.9133 | .9 | 773.6485
.6 | 764.4566 | .3 | 769.1918 | 5.0 | 773.9270
\_\_\_\_\_|\_\_\_\_\_\_\_\_\_\_\_\_\_|\_\_\_\_\_|\_\_\_\_\_\_\_\_\_\_\_\_\_|\_\_\_\_\_|\_\_\_\_\_\_\_\_\_\_\_\_\_
\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_
| | | | |
t. | 760x(1+dt). | t. | 760x(1+dt). | t. | 760x(1+dt).
\_\_\_\_\_|\_\_\_\_\_\_\_\_\_\_\_\_\_|\_\_\_\_\_|\_\_\_\_\_\_\_\_\_\_\_\_\_|\_\_\_\_\_|\_\_\_\_\_\_\_\_\_\_\_\_\_
| | | | |
deg.C. | | deg.C. | | deg.C. |
5.1 | 774.2055 | .9 | 787.5755 | .7 | 800.9454
.2 | 774.4841 |10.0 | 787.8540 | .8 | 801.2239
.3 | 774.7626 | .1 | 788.1325 | .9 | 801.5025
.4 | 775.0412 | .2 | 788.4111 |15.0 | 801.7810
.5 | 775.3197 | .3 | 788.6896 | .1 | 802.0595
.6 | 775.5982 | .4 | 788.9682 | .2 | 802.3381
.7 | 775.8768 | .5 | 789.2467 | .3 | 802.6166
.8 | 776.1553 | .6 | 789.5252 | .4 | 802.8952
.9 | 776.4339 | .7 | 789.8038 | .5 | 803.1737
6.0 | 776.7124 | .8 | 790.0823 | .6 | 803.4522
.1 | 776.9909 | .9 | 790.3609 | .7 | 803.7308
.2 | 777.2695 |11.0 | 790.6394 | .8 | 804.0093

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.3 | 777.5480 | .1 | 790.9179 | .9 | 804.2879
.4 | 777.8266 | .2 | 791.1965 |16.0 | 804.5664
.5 | 778.1051 | .3 | 791.4750 | .1 | 804.8449
.6 | 778.3836 | .4 | 791.7536 | .2 | 805.1235
.7 | 778.6622 | .5 | 792.0321 | .3 | 805.4020
.8 | 778.9407 | .6 | 792.3106 | .4 | 805.6806
.9 | 779.2193 | .7 | 792.5892 | .5 | 805.9591
7.0 | 779.4978 | .8 | 792.8677 | .6 | 806.2376
.1 | 779.7763 | .9 | 793.1463 | .7 | 806.5162
.2 | 780.0549 |12.0 | 793.4248 | .8 | 806.7947
.3 | 780.3334 | .1 | 793.7033 | .9 | 807.0733
.4 | 780.6120 | .2 | 793.9819 |17.0 | 807.3518
.5 | 780.8905 | .3 | 794.2604 | .1 | 807.6303
.6 | 781.1690 | .4 | 794.5390 | .2 | 807.9089
.7 | 781.4476 | .5 | 794.8175 | .3 | 808.1874
.8 | 781.7261 | .6 | 795.0960 | .4 | 808.4660
.9 | 782.0047 | .7 | 795.3746 | .5 | 808.7445
8.0 | 782.2832 | .8 | 795.6531 | .6 | 809.0230
.1 | 782.5617 | .9 | 795.9317 | .7 | 809.3016
.2 | 782.8403 |13.0 | 796.2102 | .8 | 809.5801
.3 | 783.1188 | .1 | 796.4887 | .9 | 809.8587
.4 | 783.3974 | .2 | 796.7673 |18.0 | 810.1372
.5 | 783.6959 | .3 | 797.0458 | .1 | 810.4175
.6 | 783.9544 | .4 | 797.3244 | .2 | 810.6943
.7 | 784.2330 | .5 | 797.6029 | .3 | 810.9728
.8 | 784.5115 | .6 | 797.8814 | .4 | 811.2514
.9 | 784.7901 | .7 | 798.1600 | .5 | 811.5299
9.0 | 785.0686 | .8 | 798.4385 | .6 | 811.8084
.1 | 785.3471 | .9 | 798.7171 | .7 | 812.0870
.2 | 785.6257 |14.0 | 798.9956 | .8 | 812.3655
.3 | 785.9042 | .1 | 799.2741 | .9 | 812.6441
.4 | 786.1828 | .2 | 799.5527 |19.0 | 812.9226
.5 | 786.4613 | .3 | 799.8312 | .1 | 813.2011
.6 | 786.7398 | .4 | 800.1098 | .2 | 813.4797
.7 | 787.0184 | .5 | 800.3883 | .3 | 813.7582
.8 | 787.2969 | .6 | 800.6668 | .4 | 814.0368
\_\_\_\_\_|\_\_\_\_\_\_\_\_\_\_\_\_\_|\_\_\_\_\_|\_\_\_\_\_\_\_\_\_\_\_\_\_|\_\_\_\_\_|\_\_\_\_\_\_\_\_\_\_\_\_\_
\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_
| | | | |
t. | 760x(1+dt). | t. | 760x(1+dt). | t. | 760x(1+dt).
\_\_\_\_\_|\_\_\_\_\_\_\_\_\_\_\_\_\_|\_\_\_\_\_|\_\_\_\_\_\_\_\_\_\_\_\_\_|\_\_\_\_\_|\_\_\_\_\_\_\_\_\_\_\_\_\_
| | | | |
deg.C. | | deg.C. | | deg.C. |
19.5 | 814.3153 |23.0 | 824.0642 | .5 | 833.8131
.6 | 814.5938 | .1 | 824.3427 | .6 | 834.0916
.7 | 814.8724 | .2 | 824.6213 | .7 | 834.3702
.8 | 815.1500 | .3 | 824.8998 | .8 | 834.6487
.9 | 815.4925 | .4 | 825.1784 | .9 | 834.9273
20.0 | 815.7080 | .5 | 825.4569 |27.0 | 835.2058
.1 | 815.9865 | .6 | 825.7354 | .1 | 835.4843
.2 | 816.2651 | .7 | 826.0140 | .2 | 835.7629
.3 | 816.5436 | .8 | 826.2925 | .3 | 836.0414
.4 | 816.8222 | .9 | 826.5711 | .4 | 836.3200
.5 | 817.1007 |24.0 | 826.8496 | .5 | 836.5985
.6 | 817.3792 | .1 | 827.1281 | .6 | 836.8770

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.7 | 817.6578 | .2 | 827.4067 | .7 | 837.1556
.8 | 817.9363 | .3 | 827.6852 | .8 | 837.4341
.9 | 818.2149 | .4 | 827.9638 | .9 | 837.7127
21.0 | 818.4934 | .5 | 828.2423 |28.0 | 837.9912
.1 | 818.7719 | .6 | 828.5208 | .1 | 838.2697
.2 | 819.0505 | .7 | 828.7994 | .2 | 838.5483
.3 | 819.3290 | .8 | 829.0779 | .3 | 838.8268
.4 | 819.6076 | .9 | 829.3565 | .4 | 839.1054
.5 | 819.8861 |25.0 | 829.6350 | .5 | 839.3839
.6 | 820.1646 | .1 | 829.9135 | .6 | 839.6624
.7 | 820.4432 | .2 | 830.1921 | .7 | 839.9410
.8 | 820.7217 | .3 | 830.4706 | .8 | 840.2195
.9 | 821.0003 | .4 | 830.7492 | .9 | 840.4981
22.0 | 821.2788 | .5 | 831.0277 |29.0 | 840.7766
.1 | 821.5573 | .6 | 831.3062 | .1 | 841.0551
.2 | 821.8859 | .7 | 831.5848 | .2 | 841.3337
.3 | 822.1144 | .8 | 831.8633 | .3 | 841.6122
.4 | 822.3930 | .9 | 832.1419 | .4 | 841.8908
.5 | 822.6715 |26.0 | 832.4204 | .5 | 842.1693
.6 | 822.9500 | .1 | 832.6989 | .6 | 842.4478
.7 | 823.2286 | .2 | 832.9775 | .7 | 842.7264
.8 | 823.5071 | .3 | 833.2560 | .8 | 843.0049
.9 | 823.7857 | .4 | 833.5346 | .9 | 843.2835
| | | |30.0 | 843.5620
\_\_\_\_\_|\_\_\_\_\_\_\_\_\_\_\_\_\_|\_\_\_\_\_|\_\_\_\_\_\_\_\_\_\_\_\_\_|\_\_\_\_\_|\_\_\_\_\_\_\_\_\_\_\_\_\_
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**CHAPTER VIII.**

*FIRING POINT OF EXPLOSIVES, HEAT TESTS, &c.*
Horsley’s Apparatus—­Table of Firing
points—­The Government Heat-Test Apparatus
for Dynamites—­Nitro-Glycerine, Nitro-Cotton,
and Smokeless Powders—­Liquefaction and
Exudation Tests—­Page’s Regulator for
Heat-Test Apparatus—­Specific Gravities
of Explosives—­Table of Temperature of Detonation,
Sensitiveness, &c.
 *The Firing Point of Explosives.*—­The
firing point of an explosive may be determined as
follows:—­A copper dish, about 3 inches deep,
and 6 or more wide, and fitted with a lid, also of
copper, is required. The lid contains several
small holes, into each of which is soldered a thick
copper tube about 5 mm. in diameter, and 3 inches
long, with a rather larger one in the centre in which
to place a thermometer. The dish is filled with
Rose’s metal, or paraffin, according to the
probable temperature required. The firing point
is then taken thus:—­After putting a little
piece of asbestos felt at the bottom of the centre
tube, the thermometer is inserted, and a small quantity
of the explosive to be tested is placed in the other
holes; the lid is then placed on the dish containing
the melted paraffin or metal, in such a way that the
copper tubes dip below the surface of the liquid;
the temperature of the bath is now raised until the
explosive fires, and the temperature noted. The
initial temperature should also be noted.

THE FIRING POINT OF VARIOUS EXPLOSIVES (by C. E. Munroe).
(Horsley’s Apparatus used.)

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|
| deg.C.
Nitro-glycerine, 5 years old (a single drop taken) | 203-205
Gun-cotton (compressed military cotton, sp. gr. 1.5) | 192-201
Air-dried gun-cotton, stored for 4 years | 179-187
Ditto, stored for 1 year | 187-189
Air-dried collodion-cotton, long staple “Red Island |
cotton,” 3 years old | 186-191
Air-dried collodion, 3 years old, stored wet | 197-199
Hydro-nitro-cellulose | 201-213
Kieselguhr dynamite, No. 1 | 197-200
Explosive gelatine | 203-209
Mercury fulminate | 175-181
Gunpowder (shell) | 278-287
Hill’s picric powder (shells) Been in store 10 years. | 273-283
Ditto (musket) Composed of—­ | 282-290
Ammonium picrate 42.18 % |
Potassium picrate 53.79 " |
Charcoal (alder) 3.85 " |
\_\_\_\_\_\_\_\_ |
|
99.82 |
Forcite, No. 1 | 187-200
Atlas powder (75% NG) | 175-185
Emmensite, No. 1 Sample had been stored in | 167-184
magazine for some months in |
a wooden box. |
" No. 2 Stored in tin case. | 165-177
" No. 5 " " | 205-217
\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_|\_
\_\_\_\_\_\_\_\_\_
| |
| deg.C. |
Powder used in Chassepot rifle | 191 | By Leygue & Champion.
French gunpowder | 295 | " "
Rifle powder (picrate) | 358 | " "
Cannon | 380 | " "
\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_|\_\_\_\_\_\_\_\_\_|\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_
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Horsley’s apparatus consists of an iron stand
with a ring support, holding a hemispherical iron
vessel or bath in which solid paraffin is put.
Above this is another movable support, from which
a thermometer is suspended, and so adjusted that its
bulb is immersed in the material contained in the
iron vessel. A thin copper cartridge-case, 5/8
inch in diameter and 1-15/16 inch long, is suspended
over the bath by means of a triangle, so that the
end of the case is just 1 inch below the surface of
the molten material. On beginning the experiment
of determining the firing point of any explosive,
the material in the bath is heated to just above the
melting point; the thermometer is inserted in it, and
a minute quantity of the explosive is placed in the
bottom of the cartridge-case. The initial temperature
is noted, and then the cartridge-case containing the
explosive is inserted in the bath. The temperature
is quickly raised until the contents of the cartridge-case
flash off or explode, when the temperature is noted
as the *firing point*.

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[Illustration: FIG. 46.—­HEAT TEST
APPARATUS.]

Professor C.E. Munroe, of the U.S. Torpedo
Station, has determined the firing point of several
explosives by means of this apparatus.
 *The Government Heat Test (Explosives Act, 1875):
Apparatus required.*—­A water bath, consisting
of a spherical copper vessel *(a)*, Fig. 46, of
about 8 inches diameter, and with an aperture of about
5 inches; the bath is filled with water to within
a quarter of an inch of the edge. It has a loose
cover of sheet copper about 6 inches in diameter *(b)*
and rests on a tripod stand about 14 inches high *(c)*,
which is covered with coarse wire gauze *(e)*,
and is surrounded with a screen of thin sheet copper
*(d)*. Within the latter is placed an argand
burner *(f)* with glass chimney. The cover
*(b)* has four holes arranged, as seen in Fig.
II., No. 4 to contain a Page’s[A] or Scheibler’s
regulator, No. 3 the thermometer, Nos. 1 and 2 the
test tubes containing the explosive to be tested.
Around the holes 1 and 2 on the under side of the
cover are soldered three pieces of brass wire with
points slightly converging (Fig. III.); these
act as springs, and allow the test tubes to be easily
placed in position and removed.

[Footnote A: See *Chem. Soc. Jour.*,
1876, i. 24. F.J.M. Page.]
 *Test Tubes*, from 5-1/4 to 5-1/2 inches long,
and of such a diameter that they will hold from 20
to 22 cubic centimetres of water when filled to a
height of 5 inches; rather thick glass is preferable.
Indiarubber stoppers, fitting the test tubes, and
carrying an arrangement for holding the test papers,
*viz*., a narrow glass tube passing through the
centre of the stopper, and terminating in a platinum
wire hook. A glass rod drawn out and the end
turned up to form a hook is better.
 *The Thermometer* should have a range from 30
deg. to 212 deg. F., or from 1 deg. to 100 deg.
C. A minute clock is useful.
 *Test Paper.*—­The test paper is prepared
as follows:—­45 grains (2.9 grms.) of white
maize starch (corn flour), previously washed with cold
water, are added to 8-1/2 oz. of water. The mixture
is stirred, heated to boiling, and kept gently boiling
for ten minutes; 15 grains (1 grm.) of pure potassium
iodide (previously recrystallised from alcohol, absolutely
necessary) are dissolved in 8-1/2 oz. of distilled
water. The two solutions are thoroughly mixed
and allowed to get cold. Strips or sheets of
white English filter paper, previously washed with
water and re-dried, are dipped into the solution thus
prepared, and allowed to remain in it for not less
than ten seconds; they are then allowed to drain and
dry in a place free from laboratory fumes and dust.
The upper and lower margins of the strips or sheets
are cut off, and the paper is preserved in well-stoppered
or corked bottles, and in the dark. The dimensions
of the pieces of test paper used are about 4/10 inch
by 8/10 inch (10 mm. by 20 mm.).[A]

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[Footnote A: When the paper is freshly prepared,
and as long as it remains in good condition, a drop
of diluted acetic acid put on the paper with a glass
rod produces no coloration. In process of time
it will become brownish, when treated with the acid,
especially if it has been exposed to sunlight.
It is then not fit for use.]

In Germany zinc-iodide starch paper is used, which
is considered to be more sensitive than potassium
iodide.
 *Standard Tint Paper.*—­A solution
of caramel in water is made of such concentration
that when diluted one hundred times (10 c.c. made up
to 1 litre) the tint of this diluted solution equals
the tint produced by the Nessler test in 100 c.c.
water containing .000075 grm. of ammonia, or .00023505
grm. AmCl. With this caramel solution lines
are drawn on strips of white filter paper (previously
well washed with distilled water, to remove traces
of bleaching matter, and dried) by means of a quill
pen. When the marks thus produced are dry, the
paper is cut into pieces of the same size as the test
paper previously described, in such a way that each
piece has a brown line across it near the middle of
its length, and only such strips are preserved in
which the brown line has a breadth varying from 1\2
mm. to 1 mm. (1/50 of an inch to 1/25 of an inch).
 *Testing Dynamite, Blasting Gelatine, and Gelatine
Dynamite.*—­Nitro-glycerine preparations,
from which the nitro-glycerine can be extracted in
the manner described below, must satisfy the following
test, otherwise they will not be considered as manufactured
with “thoroughly purified nitro-glycerine,”
*viz*., fifteen minutes at 160 deg. F. (72
deg. C.).
 *Apparatus required.*—­A funnel 2 inches
across (*d*), a cylindrical measure divided into
grains (*e*), Fig. 47.
 *Mode of Operation.*—­About 300 (19.4
grms.) to 400 grains (26 grms.) of dynamite (*b*),
finely divided, are placed in the funnel, which is
loosely plugged by freshly ignited asbestos (*a*).
The surface is smoothed by means of a flat-headed
glass rod or stopper, and some clean washed and dried
kieselguhr (*c*) is spread over it to the depth
of about 1/8 inch. Water is then poured on from
a wash bottle, and when the first portion has been
soaked up more is added; this is repeated until sufficient
nitro-glycerine has collected in the graduated measure
(*e*). If any water should have passed through,
it must be removed from the nitro-glycerine by filter
paper, or the nitro-glycerine may be filtered.

[Illustration: FIG. 47.—­APPARATUS
FOR SEPARATING THE NlTRO-GLYCERINE FROM DYNAMITE.]

[Illustration: FIG. 48.—­TEST TUBE
ARRANGED FOR HEAT TEST.]

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*Application of Test.*—­The thermometer
is fixed so as to be inserted through the lid of the
water bath into the water, which is maintained at
160 deg. F. (72 deg. C.), to a depth of 2-3/4
inches. Fifty grains (= 3.29 grms.) of nitro-glycerine
to be tested are weighed into the test tube, in such
a way as not to soil the sides of the tube (use a
pipette). A test paper is fixed on the hook of
the glass rod, so that when inserted into the tube
it will be in a vertical position. A sufficient
amount of a mixture of half distilled water and half
glycerine, to moisten the upper half of the paper,
is now applied to the upper edge of the test paper
by means of a glass rod or camel’s hair pencil;
the cork carrying the rod and paper is fixed into
the test tube, and the position of the paper adjusted
so that its lower edge is about half way down the
tube; the latter is then inserted through one of the
holes in the cover to such a depth that the lower
margin of the moistened part of the paper is about
5/8 inch above the surface cover. The test is
complete when the faint brown line, which after a
time makes its appearance at the line of boundary between
the dry and moist part of the paper, equals in tint
the brown line of the standard tint paper.
 *Blasting Gelatine, Gelatine Dynamite, Gelignite,
&c.*—­Fifty grains (= 3.29 grms.) of
blasting gelatine are intimately mixed with 100 grains
(= 6.5 grms.) of French chalk. This is done by
carefully working the two materials together with
a wooden pestle in a wooden mortar. The mixture
is then gradually introduced into the test tube, with
the aid of gentle tapping upon the table between the
introduction of successive portions of the mixture
into the tube, so that when the tube contains all the
mixture it shall be filled to the extent of 1-3/4
inch of its height. The test paper is then inserted
as above described for nitro-glycerine. The sample
tested must stand a temperature of 160 deg. F.
for a period of ten minutes before producing a discoloration
of the test paper corresponding in tint to the standard
paper.
 *N.B.*—­Non-gelatinised nitro-glycerine
preparations, from which the nitro-glycerine cannot
be expelled by water, are tested without any previous
separation of the ingredients, the temperature being
as above 160 deg. F., and the time being seven
minutes.
 *Gun-Cotton, Schultze Gunpowder, E.C. Powder,
&c.: A. Compressed Gun-Cotton.*—­Sufficient
material to serve for two or more tests is removed
from the centre of the cartridge by gentle scraping,
and if necessary, further reduced by rubbing between
the fingers. The fine powder thus produced is
spread out in a thin layer upon a paper tray 6 inches
by 4-1/2 inches, which is then placed inside a water
oven, kept as nearly as possible at 120 deg.
F. (49 deg. C.). The wire gauze shelves of
the oven should be about 3 inches apart. The
sample is allowed to remain at rest for fifteen minutes
in the oven, the door of which is left wide open.
After the lapse of fifteen minutes the tray is removed
and exposed to the air of the room for two hours,
the sample being at some point within that time rubbed
upon the tray with the hand, in order to reduce it
to a fine and uniform state of division.

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The heat test is performed as before, except that
the temperature of the bath is kept at 170 deg.
F. (66 deg. C.), and regulator set to maintain
that temperature. Twenty grains (1.296 grm.)
are used, placed in the test tube, gently pressed
down until it occupies a space of as nearly as possible
1-5/10 inch in the test tube of dimensions previously
specified. The fine cotton adhering to the sides
of the tube can be removed by a clean cloth or silk
handkerchief. The paper is moistened by touching
the upper edge with a drop of the 50 per cent. glycerine
solution, the tube inserted in the bath to a depth
of 2-1/2 inches, measured from the cover, the regulator
and thermometer being inserted to the same depth.
The test paper is to be kept near the top of the test
tube, but clear of the cork, until the tube has been
immersed for about five minutes. A ring of moisture
will about this time be deposited upon the sides of
the test tube, a little above the cover of the bath.
The glass rod must then be lowered until the lower
margin of the moistened part of the paper is on a level
with the bottom of the ring of moisture in the tube.
The paper is now closely watched, The test is complete
when a very faint brown coloration makes its appearance
at the line of boundary between the dry and moist parts
of the paper. It must stand the test for not
less than ten minutes at 170 deg. F. (The
time is reckoned from the first insertion of the tube
in the bath until the appearance of a discoloration
of the test paper.)
 *B. Schultze Powder, E.C. Powder, Collodion-Cotton,
&c.*—­The sample is dried in the oven
as above for fifteen minutes, and exposed for two hours
to the air. The test as above for compressed gun-cotton
is then applied.
 *C. Cordite* must stand a temperature of
180 deg. F. for fifteen minutes. The sample
is prepared as follows:—­Pieces half an inch
long are cut from one end of every stick selected
for the test: in the case of the thicker cordites,
each piece so cut is further subdivided into about
four portions. These cut pieces are then passed
once through the mill, the first portion of material
which passes through being rejected on account of
the possible presence of foreign matter from the mill.
The ground material is put on the top sieve of the
nest of sieves, and sifted. That portion which
has passed through the top sieve and been stopped by
the second is taken for the test. If the mill
is properly set, the greater portion of the ground
material will be of the proper size. If the volatile
matter in the explosive exceeds 0.5 per cent., the
sifted material should be dried at a temperature not
exceeding 140 deg. F, until the proportion does
not exceed 0.5 per cent. After each sample has
been ground, the mill must be taken to pieces and
carefully cleaned. The sieves used consist of
a nest of two sieves with holes drilled in sheet copper.
The holes in the top sieve have a diameter = 14 B.W.G.,
those in the second = 21 B.W.G.

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If too hard for the mill, the cordite may be softened
by exposure to the vapour of acetone,[A] or reduced,
to the necessary degree of subdivision by means of
a sharp moderately-coarse rasp. Should it have
become too soft in the acetone vapour for the mill,
it should be cut up into small pieces, which may be
brought to any desired degree of hardness by simple
exposure to air. Explosives which consist partly
of gelatinised collodion-cotton, and partly of ungelatinised
gun-cotton, are best reduced to powder by a rasp,
or softened by exposure to mixed ether and alcohol
vapour at a temperature of 40 deg. F. to 100
deg. F.

[Footnote A: Mr W. Cullen *(Jour. Soc.
Chem. Ind.*, Jan. 31, 1901) says:—­
“Undoubtedly the advent of the horny smokeless
powders of modern times has made it a little difficult
to give the test the same scope as it had when first
introduced.” As a rule a simple explanation
can be found for every apparently abnormal result,
and in the accidental retention of a portion of the
solvent used in the manufacture, will frequently be
found an explanation of the trouble experienced.]
 *Ballistite.*—­In the case of ballistite
the treatment is the same, except that when it is
in a very finely granulated condition it need not be
cut up.
 *Guttmann’s Heat Test.*—­This
test was proposed by Mr Oscar Guttmann in a paper
read before the Society of Chemical Industry (vol.
xvi., 1897), in the place of the potassium iodide
starch paper used in the Abel test. The filter
paper used is wetted with a solution of diphenylamine[A]
in sulphuric acid. The solution is prepared as
follows:—­Take 0.100 grm. of diphenylamine
crystals, put them in a wide-necked flask with a ground
stopper, add 50 c.c. of dilute sulphuric acid (10 c.c.
of concentrated sulphuric acid to 40 c.c. of water),
and put the flask in a water bath at between 50 deg.
and 55 deg. C. At this temperature the diphenylamine
will melt, and at once dissolve in the sulphuric acid,
when the flask should be taken out, well shaken, and
allowed to cool. After cooling, add 50 c.c. of
Price’s double distilled glycerine, shake well,
and keep the solution in a dark place. The test
has to be applied in the following way:—­The
explosives that have to be tested are finely subdivided,
gun-cotton, nitro-glycerine, dynamite, blasting gelatine,
&c., in the same way as at present directed by the
Home Office regulations. Smokeless powders are
all to be ground in a bell-shaped coffee mill as finely
as possible, and sifted as hitherto. 1.5 grm. of the
explosive (from the second sieve in the case of smokeless
powder) is to be weighed off and put into a test tube
as hitherto used. Strips of well-washed filter
paper, 25 mm. wide, are to be hung on a hooked glass
rod as usual. A drop of the diphenylamine solution
is taken up by means of a clean glass rod, and the
upper corners of the filter paper are touched with
it, so that when the two drops run together about

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a quarter of the filter paper is moist. This is
then put into the test tube, and this again into the
water bath, which has been heated to 70 deg.
C. The heat test reaction should not show in a shorter
time than fifteen minutes. It will begin by the
moist part of the paper acquiring a greenish yellow
colour, and from this moment the paper should be carefully
watched. After one or two minutes a dark blue
mark will suddenly appear on the dividing line between
the wet and dry part of the filter paper, and this
is the point that should be taken.

[Footnote A: Dr G. Spica (*Rivista*, Aug.
1897) proposes to use hydrochloride of meta-phenylenediamine.]
 *Exudation and Liquefaction Test for Blasting Gelatine,
Gelatine Dynamite, &c.*—­A cylinder of
blasting gelatine, &c., is to be cut from the cartridge
to be tested, the length of the cylinder to be equal
to its diameter, and the ends being cut flat.
The cylinder is to be placed on end on a flat surface
without any wrapper, and secured by a pin passing
vertically through its centre. In this condition
the cylinder is to be exposed for 144 consecutive
hours (six days and nights) to a temperature ranging
from 85 deg. to 90 deg. F. (inclusive), and during
such exposure the cylinder shall not diminish in height
by more than one-fourth of its original height, and
the upper cut surface shall retain its flatness and
the sharpness of its edge.
 *Exudation Test.*—­There shall be no
separation from the general mass of the blasting gelatine
or gelatine dynamite of a substance of less consistency
than the bulk of the remaining portion of the material
under any conditions of storage, transport, or use,
or when the material is subjected three times in succession
to alternate freezing and thawing, or when subjected
to the liquefaction test before described.
 *Picric Acid.*—­The material shall
contain not more than 0.3 part of mineral or non-combustible
matter in 100 parts by weight of the material dried
at 160 deg. F. It should not contain more than
a minute trace of lead. One hundred parts of
the dry material shall not contain more than 0.3 part
of total (free and combined) sulphuric acid, of which
not more than 0.1 part shall be free sulphuric acid.
Its melting point should be between 248 deg. and 253
deg. F.
 *Ammonite, Bellite, Roburite, and Explosives of
similar Composition.*—­ These are required
to stand the same heat test as compressed nitro-cellulose,
gun-cotton, &c.
 *Chlorate Mixtures.*—­The material
must not be too sensitive, and must show no tendency
to increase in sensitiveness in keeping. It must
contain nothing liable to reduce the chlorate.
Chlorides calculated as potassium chloride must not
exceed 0.25 per cent. The material must contain
no free acid, or substance liable to produce free
acid. Explosives of this class containing nitro-compounds
will be subject to the heat test.

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*Page’s Regulator.*—­The most
convenient gas regulator to use in connection with
the heat-test apparatus is the one invented by Prof.
F.J.M. Page, B.Sc.[A] (Fig. 49). It is not
affected by variations of the barometric pressure,
and is simple and easy to fit up. It consists
of a thermometer with an elongated glass bulb 5/8
inch diameter and 3 inches long. The stem of
the thermometer is 5 inches long and 1/8 inch to 3/16
inch internal diameter. One and a half inch from
the top of the stem is fused in at right angles a
piece of glass tube, 1 inch long, of the same diameter
as the stem, so as to form a T. A piece of glass tube
(A), about 7/16 inch external diameter and 1-1/2 inch
long, is fitted at one end with a short, sound cork
(C, Fig. 50). Through the centre of this cork
a hole is bored, so that the stem of the thermometer
just fits into it. The other end of this glass
tube is closed by a tightly fitting cork, preferably
of indiarubber (I), which is pierced by a fine bradawl
through the centre. Into the hole thus made is
forced a piece of fine glass tube (B) 3 inches long,
and small enough to fit loosely inside the stem of
the thermometer.

[Footnote A: *Chemical Soc. Jour.*,
1876, i. 24.]

The thermometer is filled by pouring in mercury through
a small funnel until the level of the mercury (when
the thermometer is at the desired temperature) is
about 1-1/2 inch below the T. The piece of glass tube
A, closed at its upper extremity by the cork I, through
which the fine glass tube B passes into the stem of
the thermometer, is now filled by means of the perforated
cork at its lower extremity on the stem of the thermometer.
The gas supply tube is attached to the top of the tube
A, the burner to the T, so that the gas passes in
at the top, down the fine tube B, rises in the space
between B and the inside wall of the stem of the thermometer,
and escapes by the T. The regulator is set for any
given temperature by pushing the cork C, and consequently
the tubes A and B, which are firmly attached to it,
up or down the stem of the thermometer, until the
regulator just cuts off the gas at the desired temperature.

[Illustration: FIG. 49.—­PAGE’S
REGULATOR.]

[Illustration: FIG. 50.—­PAGE’S
GAS REGULATOR, SHOWING BYE-PASS AND CUT-OFF ARRANGEMENT.]

As soon as the temperature falls, the mercury contracts,
and thus opens the end of the tube B. The gas is thus
turned on, and the temperature rises until the regulator
again cuts off the gas. In order to prevent the
possible extinction of the flame by the regulator,
the brass tube which carries the gas to the regulator
is connected with the tube which brings the gas from
the regulator to the burner by a small brass tap (Fig.
2). This tap forms an adjustable bye-pass, and
thus a small flame can be kept burning, even though
the regulator be completely shut off. It is obvious
that the quantity of gas supplied through the bye-pass
must always be less than that required to maintain
the desired temperature. This regulator, placed
in a beaker of water on a tripod, will maintain the
temperature of the water during four or five hours
within 0.2 deg. C., and an air bath during six
weeks within 0.5 deg. C.

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To sum up briefly the method of using the regulator:—­Being
filled with mercury to about 1\2 inch below the T,
attach the gas supply as in diagram (Fig. 2), the
brass tap being open, and the tube B unclosed by the
mercury. Allow the gas to completely expel the
air in the apparatus. Push down the tube A so
that the end of B is well under the surface of the
mercury. Turn off the tap of the bye-pass until
the smallest bead of flame is visible. Raise
A and B, and allow the temperature to rise until the
desired point is attained. Then push the tubes
A and B slowly down until the flame is just shut off.
The regulator will then keep the temperature at that
point.
 *Will’s Test for Nitro-Cellulose.*—­The
principle of Dr W. Will’s test[A] may be briefly
described as follows:—­The regularity with
which nitro-cellulose decomposes under conditions
admitting of the removal of the products of decomposition
immediately following their formation is a measure
of its stability. As decomposing agent a sufficiently
high temperature (135 deg. C.) is employed, the
explosive being kept in a constantly changing atmosphere
of carbon dioxide, heated to the same temperature:
the oxides of nitrogen which result are swept over
red-hot copper, and are then reduced to nitrogen,
and finally, the rates of evolution of nitrogen are
measured and compared. Dr Will considers that
the best definition and test of a stable nitro-cellulose
is that it should give off at a high temperature equal
quantities of nitrogen in equal times. For the
purposes of manufacture, it is specially important
that the material should be purified to its limit,
*i.e*., the point at which further washing produces
no further change in its speed of decomposition measured
in the manner described.

[Footnote A: W. Will, *Mitt. a. d. Centrallstelle
f. Wissench. Techn. Untersuchungen
Nuo-Babelsberg Berlin*, 1902 [2], 5-24.]

The sample of gun-cotton (2.5 grms.) is packed into
the decomposition tube 15 mm. wide and 10 cm. high,
and heated by an oil bath to a constant temperature,
the oxides so produced are forced over ignited copper,
where they are reduced, and the nitrogen retained
in the measuring tubes. Care must be taken that
the acid decomposition products do not condense in
any portion of the apparatus. The air in the
whole apparatus is first displaced by a stream of
carbon dioxide issuing from a carbon dioxide generator,
or gas-holder, and passing through scrubbers, and this
stream of gas is maintained throughout the whole of
the experiment, the gas being absorbed at the end
of the system by strong solution of caustic potash.
To guard against the danger of explosions, which occasionally
occur, the decomposition tube and oil bath are surrounded
by a large casing with walls composed of iron plate
and strong glass.

Dr Will’s apparatus has been modified by Dr
Robertson,[A] of the Royal Gunpowder Factory, Waltham
Abbey. The form of the apparatus used by him is
shown in Fig. 51.

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*CO\_{2} Holders.*—­Although objection
has been taken to the use of compressed CO\_{2} in
steel cylinders on account of the alleged large and
variable amount of air present, it has, nevertheless,
been found possible to obtain this gas with as little
as 0.02 per cent. of air. Frequent estimations
of the air present in the CO\_{2} of a cylinder show
that even with the commercial article, after the bulk
of the CO\_{2} has been removed, the residual gas contains
only a very small amount of air, which decreases in
a gradual and perfectly regular manner. For example,
one cylinder which gave 0.03 per cent. of air by volume,
after three months’ constant use gave 0.02 per
cent. The advantage of using CO\_{2} from this
source is obvious when compared with the difficulty
of evolving a stream of gas of constant composition
from a Kipps or Finkener apparatus. A micrometer
screw, in addition to the main valve of the CO\_{2}
cylinder, is useful for governing the rate of flow.
A blank experiment should be made to ascertain the
amount of air in the CO\_{2} and the correction made
in the readings afterwards.

[Footnote A: *Jour. Soc. Chem.
Ind.*, June 30, 1902, p. 819.]

[Illustration: Fig 51.—­Will’s
Apparatus for Testing Nitro-cellulose]
 *Measurement of Pressure and Rate of Flow.*—­Great
attention is paid to the measurement of the rate of
flow of gas, which is arrived at by counting with
a stop-watch the number of bubbles of gas per minute
in a small sulphuric acid wash bottle. A mercury
manometer is introduced here, and is useful for detecting
a leak in the apparatus. The rate of flow that
gives the most satisfactory results is 1,000 c.c. per
hour. If too rapid it does not become sufficiently
preheated in the glass spiral, and if too slow there
is a more rapid decomposition of the nitro-cellulose
by the oxides of nitrogen which are not removed.
 *Decomposition Tube.*—­This is of the
form and dimensions given by Dr Will (15 mm. wide
and 10 cm. high), the preheating worm being of the
thinnest hydrometer stem tubing. The ground-in
exit tube is kept in position by a small screw clamp
with trunnion bearings.
 *Bath.*—­To permit of two experiments
being carried on simultaneously, the bath is adapted
for two decomposition tubes, and is on the principle
of Lothar Meyer’s air bath, that is, the bath
proper filled with a high-flashing hydrocarbon oil,
and fitted with a lid perforated with two circular
holes for the spiral tubes, is surrounded by an asbestos-covered
envelope, in the interior of which circulate the products
of combustion of numerous small gas jets. The
stirrer, agitated by a water motor, or, better still,
a hot-air engine, has a series of helical blades curved
to give a thorough mixing to the oil. Great uniformity
and constancy of temperature are thus obtained.
The bath is fitted also with a temperature regulator
and thermometer.

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*Reduction Tube*—­This is of copper,
and consists of two parts, the outer tube and an inner
reaching to nearly the bottom of the former. Into
the inner tube fits a spiral of reduced copper gauze,
and into the annular space between the tubes is fitted
a tightly packed reduced copper spiral. At the
bottom the inlet tube dips into a layer of copper oxide
asbestos, on the top of which is a layer of reduced
copper asbestos. Through the indiarubber cork
passes a glass tube, which leads the CO\_{2} and nitrogen
out of the reduction tube. As the portion of the
tube containing the spirals is heated to redness,
water jackets are provided on both inner and outer
tubes to protect the indiarubber cork.
 *Nitrogen Measuring Apparatus.*—­The
measuring tube with zigzag arrangement is used, having
been found very economical in potash. It is most
convenient to take readings by counterbalancing the
column of potash solution and reading off the volume
of gas at atmospheric pressure. For this purpose
the tap immediately in front of the measuring tube
is momentarily closed, this having been proved to
be without ill effect on the progress of the test.
In all experiments done by this test the air correction
is subtracted from each reading, and the remainder
brought to milligrams of nitrogen with the usual corrections.
As objection has frequently been taken to the test
on the ground of difficulty in interpreting the results
obtained, Dr Robertson made a series of experiments
for the purpose of standardising the test, and at the
same time of arriving at the condition under which
it could be applied in the most sensitive and efficient
manner. A variety of nitro-celluloses having
been tested, there were chosen as typical, of stable
and unstable products, service gun-cotton on the one
hand, and an experimental gun-cotton, Z, on the other.
The first point brought out by these experiments was
the striking uniformity of service gun-cotton, first
in regard to the rectilinear nature of the curve of
evolution of nitrogen, and secondly in regard to the
small range within which a large number of results
is included, 15 samples lying between 6.6 and 8.7
mgms. of nitrogen evolved in four hours. In the
case of service gun-cotton, little difference in the
rate of evolution of nitrogen evolved is obtained on
altering the rate of passage of CO\_{2} gas through
the wide range of 500 c.c. per hour to 2,500 c.c.
per hour. With Z gun-cotton (see Fig. 52), however,
the case is very different. Operating at a rate
of 1,000 c.c. of CO\_{2} per hour, a curve of nitrogen
evolution is obtained, which is bent and forms a good
representation of the inherent instability of the material
as proved to exist from other considerations.
Operating at the rate of 1,500 c.c. per hour, as recommended
by Dr Will, the evolution of nitrogen is represented
by a straight line, steeper, however, than that of
service gun-cotton. The rate of passage of CO\_{2}
was therefore chosen at 1,000 c.c. per hour, or two-thirds
of the rate of Dr Will, and this rate, besides possessing
the advantage claimed of rendering diagnostic the
manner of nitrogen evolution in Z gun-cotton, has
in other cases been useful in bringing out relationships,
which the higher rate would have entirely masked.

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[Illustration: Fig. 52.—­Dr. Robertson’s
results.]

[Illustration: Fig. 53.—­Service Guncotton
for Cordite made at a Private Factory.]

Readings are taken thirty minutes from the time the
nitro-cellulose is heated, and are taken at intervals
of fifteen minutes for about four hours; fresh caustic
potash is added every thirty minutes or so. It
is convenient to plot the results in curves.
The curves given in Fig. 53 are from gun-cotton manufacturers
in England at a private factory. The rate of
evolution of nitrogen is as follows:—­

In 1 hour. In 2 hours. In 3 hours.
 In 4 hours.
 N. N. N.
 N. in milligrammes.
 1.25 2.55 4.5
 5.75
 1.5 3.25 5.25
 6.75
These results are very satisfactory, the gun-cotton
was of a very good quality. Several hours are
necessary to remove all the air from the apparatus.
Dr Will stated fifteen minutes in his original paper,
but this has not been found sufficient. It has
not been satisfactorily proved that Will’s test
can be applied to gelatinised nitro-cellulose powders.
It is convenient to plot the results in curves; the
nitrogen is generally given in cubic centimetres or
in milligrammes, and readings taken every fifteen
minutes. The steepness of the curve is a measure
of the stability of the nitro-cellulose which is being
examined. The steeper the curve the more nitrogen
is evolved per unit of time, and the less stable the
nitro-cellulose. In the case of unstable nitro-celluloses
heated under the conditions described, the separation
of nitrogen is much greater at first than at a later
period. If the nitro-cellulose be very unstable,
explosions are produced. If the separation of
nitrogen is uniform during the prolonged heating,
then the nitro-cellulose may be regarded as “normal.”
If it be desired to determine the absolute amount of
nitrogen separated from a nitro-cellulose, the following
conditions must be observed:—­(1.) Accurate
weighing of the nitro-cellulose; (2.) Determination
of the amount of air in the CO\_{2}, and deduction of
this from the volume of gas obtained; (3.) Reduction
of the volume of the gas to the volume at 0 deg.
C. and 760 mm. pressure.[A]

[Footnote A: See also *Jour. Soc.
Chem. Ind.*, Dec. 1902, pages 1545-1555, on
the “Stability of Nitro-cellulose” and
“Examination of Nitro-cellulose,” Dr
Will.]
 *Bergrnann and Junk*[A] describe a test for nitro-cellulose
that has been in use in the Prussian testing station
for some years. The apparatus consists of a closed
copper bath provided with a condenser and 10 countersunk
tubes of 20 cm. length. By boiling amyl-alcohol
in the bath, the tubes can be kept at a constant temperature
of 132 deg. C. The explosive to be tested is
placed in a glass tube 35 cm. long and 2 cm. wide,
having a ground neck into which an absorption bulb
is fitted. The whole apparatus is surrounded
by a shield, in case of explosion. In carrying

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out the test, 2 grms. of the explosive are placed
in the glass tube and well pressed down. The
absorption bulb is half filled with water, and fitted
into the ground neck of the glass tube, which is then
placed in one of the tubes in the bath previously
brought to the boiling point (132 deg. C.).
The evolved oxides of nitrogen are absorbed in the
water in the bulb, and at the end of two hours the
tubes are removed from the bath, and on cooling, the
water from the bulb flows back and wets the explosive.
The contents of the tube are filtered and washed,
the filtrate is oxidised with permanganate, and the
nitrogen determined as nitric oxide by the Schultze-Tieman
method. The authors conclude that a stable gun-cotton
does not evolve more than 2.5 c.c. of nitric oxide
per grm. on being heated to 132 deg. C. for two
hours, and a stable collodion-cotton not more than
2 c.c. under the same conditions. The percentage
of moisture in the sample to be tested should be kept
as low as possible. A sample of nitro-cellulose
containing 1.97% of moisture gave an evolution of
2.6 c.c. per grm., while the same sample with 3.4%
moisture gave an evolution of over 50 c.c. per grm.
Sodium carbonate added to an unstable nitro-cellulose
diminishes the rate of decomposition, but if sodium
carbonate be intimately mixed with a stable nitro-cellulose
the rate of decomposition will be increased. Calcium
carbonate and mercury chloride have no influence.
If an unstable nitro-cellulose be extracted with
alcohol a stable compound is produced. The percentage
solubility of a nitro-cellulose in ether-alcohol rises
on heating to 132 deg. C. A sample which before
heating had a solubility of 4.7% had its solubility
increased to 82.5% after six hours’ heating.

[Footnote A: *Jour. Soc. Chem.
Ind.*, xxiii., Oct. 15, 1904, p. 953.]

Mr A.P. Sy (*Jour. Amer. Chem.
Soc.*, 1903) describes a new stability test for
nitro-cellulose which he terms “The Elastic Limit
of Powder Resistance to Heat.” The test
consists in heating the powder on a watch glass in
an oven to a temperature of 115 deg. C., after
eight hours the watch glass and powder are weighed
and the process repeated daily for six days or less.
He claims that the powder is tested in its natural
state, all the products of decomposition are taken
into account, whilst in the old tests only the acid
products are shown, and in the Will test only nitrogen,
that it affords an indication of the effect of small
quantities of added substances or foreign matters
on the stability and that it is simple, and not subject
to the variations of the old tests.

Obermueller (*Jour. Soc. Chem. Ind.*,
April 15, 1905) considers Bergmann and Junk’s
test is too complicated and occupies too much time;
he proposes to heat gun-cotton to 140 deg. C.
*in vacuo*, and to measure continuously by means
of a mercury manometer the pressure exerted by the
evolved gases, the latter being maintained at constant
volume; the rate at which the pressure increases is
a measure of the rate of decomposition of the nitro-cellulose.

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SPECIFIC GRAVITIES OF EXPLOSIVES, &C.

Nitro-glycerine 1.6
Gun-cotton (dry) 1.06
 " (25 per cent. water)
1.32
Dynamite No. 1 1.62
Blasting gelatine 1.54
Gelatine dynamite 1.55
Ballistite 1.6
Forcite 1.51
Tonite 1.28
Roburite 1.40
Bellite 1.2-1.4
Carbo-dynamite 1.5
Turpin’s cast picric acid 1.6
Nitro-mannite 1.6
Nitro-starch 1.5
Emmensite 1.8
Mono-nitro-benzene 1.2
Meta-di-nitro-benzene 1.575 at 18 deg.
C.
Ortho-di-nitro-benzene 1.590 "
Para-di-nitro-benzene 1.625 "
British gunpowder, E.X.E. 1.80
 " " S.B.C. 1.85
Cannonite (powder) 1.60
Celluloid 1.35
Cellulose 1.45
Ammonium nitrate 1.707
Mercury fulminate 4.42

TABLE OF THE TEMPERATURE OF DETONATION.

Blasting gelatine 3220 deg.
Nitro-glycerine 3170 deg.
Dynamite 2940 deg.
Gun-cotton 2650 deg.
Tonite 2648 deg.
Picric acid 2620 deg.
Roburite 2100 deg.
Ammonia nitrate 1130 deg.

RELATIVE SENSITIVENESS TO DETONATION (by Professor
C.E. Munroe, U.S. Naval
Torpedo Station).

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\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_
|
| Maximum |
| Distance |
| at which |
| Detonation |
| occurred. |
| CM. |
| |
Gun-cotton | 10 | Nitro-glycerine 86.5 nitro-cotton
| | 9.5, camphor 4 per cent.
Explosive gelatine | 20 | NH\_{4}NO\_{3} 5 parts,
(camphorated) | | C\_{6}H\_{4}(N0\_{3})\_{2} 1 part.
Judson powder, R.R.P. | 25 |
Emmensite (No. 259) | 30 |
Rack-a-rock | 32 | KClO\_{3} 79 parts,
| | C\_{6}H\_{5}(NO)\_{2} 21 parts.
Bellite | 50 |
Forcite No. 1 | 61 |
Kieselguhr dynamite No. 1 | 64 | 75 per cent. nitro-gycerine.
Atlas powder No. 1 | 74 |
\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_|\_\_\_\_\_\_\_\_\_\_\_\_|\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_
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**CHAPTER IX.**

*DETERMINATION OF THE RELATIVE STRENGTH OF EXPLOSIVES.*
Effectiveness of an Explosive—­High and
Low Explosives—­Theoretical Efficiency—­MM.
Roux and Sarrau’s Results—­Abel and
Noble’s—­Nobel’s Ballistic Test—­The
Mortar, Pressure, or Crusher Gauge—­Lead
Cylinders—­ The Foot-Pounds Machine—­Noble’s
Pressure Gauge—­Lieutenant Walke’s
Results—­Calculation of Pressure Developed
by Dynamite and Gun-Cotton—­ Macnab’s
and Ristori’s Results of Heat Developed by the
Explosion of Various Explosives—­Composition
of some of the Explosives in Common Use for Blasting,
&c.

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*The Determination of the Relative Strength of Explosives.*—­Explosives
may be roughly divided into two divisions, *viz*.,
those which when exploded produce a shattering force,
and those which produce a propulsive force. Explosives
of the first class are generally known as the high
explosives, and consist for the most part of nitro
compounds, or mixtures of nitro compounds with other
substances. Any explosive whose detonation is
very rapid is a high explosive, but the term has chiefly
been applied to the nitro-explosives.

The effectiveness of an explosive depends upon the
volume and temperature of the gases formed, and upon
the rapidity of the explosion. In the high explosives
the chemical transformation is very rapid, hence they
exert a crushing of shattering effect. Gunpowder,
on the other hand, is a low explosive, and produces
a propelling or heaving effect.

The maximum work that an explosive is capable of producing
is proportionate to the amount of heat disengaged
during its chemical transformation. This may
be expressed in kilogrammetres by the formula 425Q,
where Q is the number of units of heat evolved.
The theoretical efficiency of an explosive cannot,
however, be expected in practice for many reasons.

In the case of blasting rock, for instance:[A]—­1.
Incomplete combustion of the explosive. 2. Compression
and chemical changes induced in the surrounding material
operated on. 3. Energy expended in the cracking
and heating of the material which is not displaced.
4. The escape of gas through the blast-hole,
and the fissures caused by the explosion. The
proportion of useful work has been estimated to be
from 14 to 33 per cent. of the theoretical maximum
potential.

[Footnote A: C.N. Hake, Government Inspector
of Explosives, Victoria, *Jour. Soc. Chem.
Ind.*, 1889.]

For the purposes of comparison, manufacturers generally
rely more upon the practical than the theoretical
efficiency of an explosive. These, however, stand
in the same relation to one another, as the following
table of Messrs Roux and Sarrau will show:—­

MECHANICAL EQUIVALENT OF EXPLOSIVES.

Theoretical
Work Relative
in
Kilos. Value.

Blasting powder (62 per cent. KNO\_{3}) 242,335 1.0
Dynamite (75 per cent. nitro-glycerine) 548,250 2.26
Blasting gelatine (92 per cent. nitro-glycerine) 766,813 3.16
Nitro-glycerine 794,563 3.28

Experiments made in lead cylinders give—­
 Dynamite
 1.0
 Blasting gelatine
 1.4
 Nitro-glycerine
 1.4

Sir Frederick Abel and Captain W.H. Noble, R.A.,
have shown that the maximum pressure exerted by gunpowder
is equal to 486 foot-tons per lb. of powder, or that
when 1 kilo, of the powder gases occupy the volume
of 1 litre, the pressure is equal to 6,400 atmospheres;
and Berthelot has calculated that every gramme of
nitro-glycerine exploded gives 1,320 units of heat.
MM. Roux and Sarrau, of the Depot Centrales des
Poudres, Paris, by means of calorimetric determinations,
have shown that the following units of heat are produced
by the detonation of—­

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Nitro-glycerine 1,784 heat units.
Gun-cotton 1,123 "
Potassic picrate 840 "

which, multiplied by the mechanical equivalent per
unit, gives—­

Nitro-glycerine 778 metre tons per kilogramme.
Gun-cotton 489 " "
Picrate of potash 366 " "
 *Nobel’s Ballistic Test.*—­Alfred
Nobel was the first to make use of the mortar test
to measure the (ballistic) power of explosives.
The use of the mortar for measuring the relative power
of explosives does not give very accurate results,
but at the same time the information obtained is of
considerable value from a practical point of view.
The mortar consists of a solid cylinder of cast iron,
one end of which has been bored to a depth of 9 inches,
the diameter of the bore being 4 inches. At the
bottom of the bore-hole is a steel disc 3 inches thick,
in which another hole has been bored 3 inches by 2
inches. The mortar (Fig. 54) itself is fitted
with trunnions, and firmly fixed in a very solid wooden
carriage, which is securely bolted down to the ground.
The shot used should weigh 28 lbs., and be turned
accurately to fit the bore of the mortar. Down
its centre is a hole through which the fuse is put.

The following is the method of making an experiment:—­A
piece of hard wood is turned in the lathe to exactly
fit the hole in the steel disc at the bottom of the
bore. This wooden cylinder itself contains a small
cavity into which the explosive is put. Ten grms.
is a very convenient quantity. Before placing
in the mortar, a hole may be made in the explosive
by means of a piece of glass rod of such a size that
the detonator to be used will just fit into it.
After placing the wooden cylinder containing the explosive
in the cavity at the bottom of the bore, the shot,
slightly oiled, is allowed to fall gently down on
to it. A piece of fuse about a foot long, and
fitted with a detonator, is now pushed through the
hole in the centre of the shot until the detonator
is embedded in the explosive. The fuse is now
lighted, and the distance to which the shot is thrown
is carefully measured. The range should be marked
out with pegs into yards and fractions of yards, especially
at the end opposite to the mortar. The mortar
should be inclined at an angle of 45 deg.. In
experimenting with this apparatus, the force and direction
of the wind will be found to have considerable influence.

[Illustration: FIG. 54.—­MORTAR FOR
MEASURING THE BALLISTIC POWER OF EXPLOSIVES. *A*,
Shot; *B*, Steel Disc; *C*, Section of Mortar
(Cast Iron); *D*, Wooden Plug holding Explosive
(*E*); *F*, Fuse.]

Mr T. Johnson made some ballistic tests. He used
a steel mortar and a shot weighing 29 Ibs., and he
adopted the plan of measuring the distance to which
a given charge, 5 grms., would throw the shot.
He obtained the following results:—­

Range
in Feet.

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Blasting gelatine (90 per cent. nitro-glycerine and
nitro-cellulose) 392 Ammonite (60 per cent.
Am(NO\_{3}) and 10 per cent. nitro-naphthalene) 310
Gelignite (60 per cent. nitro-gelatine and gun-cotton)
 306 Roburite (AmNO\_{3} and chloro-nitro-benzol)
 294 No. 1 dynamite (75 per
cent. nitro-gelatine) 264
Stonite (68 per cent. nitro-gelatine and 32 per cent.
wood-meal) 253 Gun-cotton
 234 Tonite
(gun-cotton and nitrates)
 223 Carbonite (25 per cent. nitro-gelatine,
40 per cent. wood-meal,
 and 30 per cent. nitrates)
 198
Securite (KNO\_{3} and nitro-benzol)
 183 Gunpowder
 143
 *Calculation of the Volume of Gas Evolved in an
Explosive Reaction.*—­The volume of gas
evolved in an explosive reaction may be calculated,
but only when they are simple and stable products,
such calculations being made at 0 deg. and 760 mm.
Let it be required, for example, to determine the volume
of gas evolved by 1 gram-molecule of nitro-glycerine.
The explosive reaction of nitro-glycerine may be represented
by the equation.

C\_{3}H\_{5}O\_{3}(NO\_{2})\_{3} = 3CO\_{2} + 2-1/2H\_{2}O + 1-1/2N\_{2} + 1/4O\_{2}
By weight 227 = 132 + 45 + 42 + 8
By volume 2 = 3 + 2-1/2 + 1-1/2 + 1/4

The weights of the several products of the above reactions
are calculated by multiplying their specific gravities
by the weight of 1 litre of hydrogen at 0 deg.
C. and 760 mm. (0.0896 grm). Thus,

One litre of CO\_{2} = 22 x .0896 = 1.9712 grm.
 " H\_{2}O = 9
x " = 0.8064 "
 " N\_{2} = 14
x " = 1.2544 "
 " O\_{2} = 16
x " = 1.4336 "

The volume of permanent gases at 0 deg. and 760 mm.
is constant, and assuming the gramme as the unit of
mass, is found to be 22.32 litres. Thus:—­

Volume of 44 of CO\_{2}, at 0 deg. and 760 mm. = 44/1.9712
= 22.32 litres.
 18
" H\_{2}O " " = 18/0.8044 = 22.32
 "
 28
" N\_{2} " " = 28/1.2544 = 22.32
 "
 32
" O\_{2} " " = 32/1.4366 = 22.32
 "

Therefore

132 grms. of CO\_{2} at 0 deg. C and 760 mm. =
22.32 x 3 = 66.96 litres. 45 " H\_{2}O
 " " = 22.32 x 2-1/2 = 55.80 " 42
 " N\_{2} " " = 22.32 x 1-1/2
= 33.48 "
 8 " O\_{2} " "
= 22.32 x 1/4 = 5.58 "

 \_\_\_\_\_\_\_\_\_\_\_\_

161.82
 "
Therefore 1 gram-molecule or 227 grms. of nitro-glycerine
when exploded, produces 161.82 litres of gas at 0
deg. C and 760 mm.

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To determine the volume of gas at the temperature
of explosion, we simply apply the law of Charles.[A]
Thus—­

V : V’ :: T : T’ or V’
= VT’*T*
in which V represents the original volume.
 V’
 " new volume.
 T
 " original temperature on the absolute
scale.
 T’
 " new temperature of the same scale
In the present case T’ = 6001 deg..

Therefore substituting, we have

V’ = 161.82x6001/273 = 3557 litres

or at the temperature of explosion 1 gram-molecule
of nitro-glycerine produces 3,557 litres of permanent
gas.

[Footnote A: According to the law of Charles,
the volume of any gas varies directly as its temperature
on the absolute scale, provided the pressure remains
constant. Knowing the temperature on the centigrade
scale, the corresponding temperature on the absolute
scale is obtained by adding 273 to the degrees centigrade.]
 *Pressure or Crusher Gauge.*—­There
are many forms of this instrument. As long ago
as 1792 Count Rumford used a pressure gauge. The
so-called crusher gauge was, however, first used by
Captain Sir Andrew Noble in his researches on powder.
Other forms are the Rodman[A] punch Uchatius Eprouvette,
and the crusher gauge of the English Commission on
Explosives. They are all based either upon the
size of an indent made upon a copper disc by a steel
punch fitted to a piston, acted upon by the gases of
the explosive, or upon the crushing or flattening
of copper or lead cylinders.

[Footnote A: Invented by General Rodman, United
States Engineers.]

[Illustration: FIG. 55.—­PRESSURE GAUGE.]

Berthelot uses a cylinder of copper, as also did the
English Commission, but in the simpler form of apparatus
mostly used by manufacturers lead cylinders are used.
This form of apparatus (Fig. 55) consists of a base
of iron to which four uprights *a* are fixed,
set round the circumference of a 4-inch circle; the
lead plug rests upon the steel base let into the solid
iron block. A ring *c* holds the uprights
*d* together at the top. The piston *b*,
which rests upon the lead plug, is a cylinder of tempered
steel 4 inches in diameter and 5 inches in length;
it is turned away at the sides to lighten it as much
as possible. It should move freely between the
uprights *d*. In the top of this cylinder
is a cavity to hold the charge of explosive.
The weight of this piston is 12-1/4 lbs. The shot
*e* is of tempered steel, and 4 inches in diameter
and 10 inches in length, and weighs 34-1/2 lbs.
It is bored through its axis to receive a capped fuse.

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The instrument is used in the following manner:—­A
plug of lead 1 inch long and 1 inch in diameter, and
of a cylindrical form, is placed upon the steel plate
between the uprights *a*, the piston placed upon
it, the carefully weighed explosive placed in the
cavity, and the shot lowered gently upon the piston.
A piece of fuse, with a detonator fixed at one end,
is then pushed through the hole in the shot until it
reaches the explosive contained in the cavity in the
piston. The fuse is lighted. When the charge
is exploded, the shot is thrown out, and the lead cylinder
is more or less compressed. The lead plugs must
be of a uniform density and homogeneous structure,
and should be cut from lead rods that have been drawn,
and not cast separately from small masses of metal.

[Illustration: FIG. 56.—­*b*,
STEEL PUNCH; *c*, LEAD CYLINDER FOR USE WITH
PRESSURE GAUGE.]

The strength of the explosive is proportional to the
work performed in reducing the height of the lead
(or copper) plug, and to get an expression for the
work done it is necessary to find the number of foot-pounds
(or kilogrammetres) required to produce the different
amounts of compression. This is done by submitting
exactly similar cylinders of lead to a crushing under
weights acting without initial velocity, and measuring
the reduced heights of the cylinders; from these results
a table is constructed establishing empirical relations
between the reduced heights and the corresponding
weights; the cylinders are measured both before and
after insertion in the pressure gauge by means of
an instrument known as the micrometer calipers (Fig.
57).[A]

[Footnote A: An instrument called a “Foot-pounds
Machine” has been invented by Lieut. Quinan,
U.S. Army. It consists of three boards,
connected so as to form a slide 16 feet high, in which
a weight (the shot of the pressure gauge) can fall
freely. One of the boards is graduated into feet
and half feet. The horizontal board at the bottom,
upon which the others are nailed, rests upon a heavy
post set deep in the ground, upon which is placed
the piston of the gauge, which in this case serves
as an anvil on which to place the lead cylinders.
The shot is raised by means of a pulley, fixed at
the top of the structure, to any desired height, and
let go by releasing the clutch that holds it.
The difference between the original length and the
reduced length gives the compression caused by the
blow of the shot in falling, and gives the value in
foot-pounds required to produce the different amounts
of compression. (Vide *Jour. U.S. Naval
Inst.*, 1892.)]

[Illustration: FIG. 57.—­MICROMETER
CALIPERS FOR MEASURING DIAMETER OF LEAD CYLINDERS.]

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*The Use of Lead Cylinders.*—­The method
of using lead cylinders to test the strength of an
explosive is a very simple affair, and is conducted
as follows:—­A solid cast lead cylinder,
of any convenient size, is bored down the centre for
some inches, generally until the bore-hole reaches
to about the centre of the block. The volume
of this hole is then accurately measured by pouring
water into it from a graduated measure, and its capacity
in cubic centimetres noted. The bore-hole is then
emptied and dried, and a weighed quantity (say 10
grms.) of the explosive pressed well down to the bottom
of the hole. A hole is then made in the explosive
(if dynamite) with a piece of clean and rounded glass
rod, large enough to take the detonator. A piece
of fuse, fitted with a detonator, is then inserted
into the explosive and lighted. After the explosion
a large pear-shaped cavity will be found to have
been formed, the volume of which is then measured
in the same way as before.

The results thus obtained are only relative, but are
of considerable value for comparing dynamites among
themselves (or gun-cottons). Experiments in lead
cylinders gave the relative values for nitro-glycerine
1.4, blasting gelatine 1.4, and dynamite 1.0. (Fig.
58 shows sections of lead cylinders before and after
use.)

[Illustration: FIG. 58.—­LEAD CYLINDERS
BEFORE AND AFTER USE.]

Standard regulations for the preparation of lead cylinders
may be found in the *Chem. Zeit.*, 1903,
27 [74], 898. They were drawn up by the Fifth
International Congress of App. Chem., Berlin.
The cylinder of lead should be 200 mm. in height and
200 mm. in diameter. In its axis is a bore-hole,
125 mm. deep and 25 mm. in diameter. The lead
used must be pure and soft, and the cylinder used
in a series of tests must be cast from the same melt.
The temperature of the cylinders should be 15 deg.
to 20 deg. throughout. Ten grms. of explosive
should be used and wrapped in tin-foil. A detonator
with a charge of 2 grms., to be fired electrically,
is placed in the midst of the explosive. The
cartridge is placed in the bore-hole, and gently pressed
against the bottom, the firing wires being kept in
central position. The bore-hole is then filled
with dry quartz sand, which must pass through a sieve
of 144 meshes to the sq. cm., the wires being .35 mm.
diameter. The sand is filled in evenly, any excess
being levelled off. The charge thus prepared
is then fired electrically. The lead cylinder
is then inverted, and any residues removed with a
brush. The number of c.c. of water required to
fill the cavity, in excess of the original volume of
the bore-hole, is a measure of the strength of the
explosive. The results are only comparable if
made with the same class of explosive. A result
is to be the mean of at least three experiments.
The accuracy of the method depends on (*a*) the
uniform temperature of the lead cylinder (15 deg. to
20 deg. C. 7); (*b*) on the uniformity of
the quartz sand; (*c*) on the uniformity of the
measurements.

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[Illustration: FIG. 59.—­NOBLE’S
PRESSURE GAUGE.]
 *Noble’s Pressure Gauge.*—­The
original explosive vessels used by Captain Sir A.
Noble in his first experiments were practically exactly
similar to those that he now employs, which consists
of a steel barrel A (Fig. 59), open at both ends,
which are closed by carefully fitted screw plugs,
furnished with steel gas checks to prevent any escape
past the screw. The action of the gas checks
is exactly the same as the leathers used in hydraulic
presses. The pressure of the gas acting on both
sides of the annular space presses these sides firmly
against the cylinder and against the plug, and so
effectually prevents any escape. In the firing
plug F is a conical hole closed by a cone fitting
with great exactness, which, when the vessel is prepared
for firing, is covered with fine tissue paper to act
as an insulator. The two firing wires GG, one
in the insulated cone, the other in the firing plug,
are connected by a very fine platinum wire passing
through a glass tube filled with meal powder.
The wire becomes red-hot when connection is made with
a Leclanche battery, and the charge which has previously
been inserted into the vessel is fired. The crusher
plug is fitted with a crusher gauge H for determining
the pressure of the gases at the moment of explosion,
and in addition there is frequently a second crusher
gauge apparatus screwed into the cylinder. When
it is desired to allow the gases to escape for examination,
the screw J is slightly withdrawn. The gases
then pass into the passage I, and can be led to suitable
apparatus in which their volume can be measured, or
in which they can be sealed for subsequent chemical
analysis.

The greatest care must be exercised in carrying out
experiments with this apparatus; it is particularly
necessary to be sure that all the joints are perfectly
tight before exploding the charge. Should this
not be the case, the gases upon their generation will
cut their way out, or completely blow out the part
improperly secured, in either case destroying the apparatus.
The effect produced upon the apparatus when the gas
has escaped by cutting a passage for itself is very
curious. The surface of the metal where the escape
occurred presents the appearance of having been washed
away in a state of fusion by the rush of the highly
heated products.
 *The Pressure Gauge.*—­The pressure
is found by the use of a little instrument known as
the pressure gauge which consists of a small chamber
formed of steel, inside of which is a copper cylinder,
and the entrance being closed by a screw gland, in
which a piston, having a definite sectional area,
works. There is a gas check E (Fig. 60) placed
in the gland, and over the piston, which prevents
the admission of gas to the chamber. When it
is desired to find the pressure in the chamber of a
gun, one or more of these crushers are made up with
or inserted at the extreme rear end of the cartridge,
in order to avoid their being blown out of the gun
when fired. This, however, often takes place,
in which case the gauges are usually found a few yards
in front of the muzzle. The copper cylinders
which register the pressure are made 0.5 inch long
from specially selected copper, the diameters being
regulated to give a sectional area of either 1/12
or 1/24 square inch.

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[Illustration: FIG. 60.—­CRUSHER GAUGE.
*E*, GAS CHECK.]

Hollow copper cylinders are manufactured with reduced
sectional areas for measuring very small pressures.
It has been found that these copper cylinders are
compressed to definite lengths for certain pressures
with remarkable uniformity. Thus a copper cylinder
having a sectional area of 1/12 square inch, and originally
1/2 inch long, is crushed to a length of 0.42 inch
by a pressure of 10 tons per square inch. By subsequently
applying a pressure of 12 tons per square inch the
cylinder is reduced to a length of 0.393 inch.
Before using the cylinders, whether for experimenting
with closed vessels or with guns, it is advisable to
first crush them by a pressure a little under that
expected in the experiment. Captain Sir A. Noble
used in his experiments a modification of Rodman’s
gauge. (Ordnance Dept., U.S.A., 1861.)
 *By Calculation.*—­To calculate the
pressure developed by the explosion of dynamite in
a bore-hole 3 centimetres in diameter, charged with
1 kilogramme of 75 per cent. dynamite, Messrs Vieille
and Sarrau employ the following formula:—­

P = V\_{o}(1 + Q/273.\_c\_)/(V — *v*).

Where V\_{o} = the volume (reduced to 0 deg. and 760
mm.) of the gases produced by a unit of weight of
the explosive; Q the number of calories disengaged
by a unit of weight of the explosive; *c* equals
the specific heat at constant volume of the gases;
V the volume in cubic centimetres of a unit of weight
of the explosive; *v* the volume occupied by the
inert materials of the explosive. The volume
of gas produced by the explosion of 1 kilogramme of
nitro-glycerine (at 0 deg. and 760 mm.) is 467 litres.

V\_{o} will therefore equal 0.75 x 467 = 350.25.

The specific heat *c* is, according to Sarrau,
.220 (*c*); and according to Bunsen, 1 kilogramme
of dynamite No. 1 disengages 1,290 (Q) calories.
The density of dynamite is equal to 1.5, therefore

V = 1/1.5 = .666.

If we take the volume of the kieselguhr as .1, we
find from above formula that

P = 350(1 + 1290/(273 x .222))/(.600 — .1) =
13,900 atmospheres,

which is equal to 14,317 kilogrammes per square centimetre.
The pressure developed by 1 kilogramme of pure nitro-glycerine
equals 18,533 atmospheres, equals 19,151 kilogrammes.
Applying this formula to gun-cotton, and taking after
Berthelot, Q = 1075, and after Vieille and Sarrau,
V\_{o} = 671 litres, and *c* as .2314, and the
density of the nitro-cellulose as 1.5, we have (V
= O)

P = 671(1 + 1075/(273 x .2314))/.666 = 18,135 atmospheres.

To convert this into pressure of kilogrammes per square
centimetre, it is necessary to multiply it by the
weight of a column of mercury 0.760 m. high, and 1
square centimetre in section, which is equal to increasing
it by 1/30. It thus becomes

P^{k} = (1 + 1/30).

P^{k} = 18,135 x 1.033 = 18,733 kilogrammes.

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The following tables, taken from Messrs William Macnab’s
and E. Ristori’s paper (*Proc. Roy.
Soc.*, 56, 8-19), “Researches on Modern Explosives,”
are very interesting. They record the results
of a large number of experiments made to determine
the amount of heat evolved, and the quantity and composition
of the gases produced when certain explosives and various
smokeless powders were fired in a closed vessel from
which the air had been previously exhausted.
The explosions were carried out in a “calorimetric
bomb” of Berthelot’s pattern.[A]

[Footnote A: For description of “bomb,”
see “Explosives and their Power,” Berthelot,
trans. by Hake and Macnab, p. 150. (Murray.)]

Table Showing Quantity of Heat and Volume and Analysis
of Gas Developed per Gramme with Different Sporting
and Military Smokeless Powders Now In Use

\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_
\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_
| | | | |
Name of Explosive. | Calories | Permanent | Aqueous | Total Volume |
| per grm. | Gases. | Vapour. | of Gas at 0 deg. |
| | | | and 760 mm. |
\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_|\_\_\_\_\_\_\_\_\_\_|\_\_\_\_\_\_\_\_\_\_\_|\_\_\_\_\_\_\_\_\_|\_\_\_\_
\_\_\_\_\_\_\_\_\_\_|
| | cc/grm | cc/grm | cc/grm |
E.C. powder, English | 800 | 420 | 154 | 574 |
S.S. powder | 799 | 584 | 150 | 734 |
Troisdorf, German | 943 | 700 | 195 | 895 |
Rifleite, English | 864 | 766 | 159 | 925 |
B.N., French | 833 | 738 | 168 | 906 |
Cordite, English | 1253 | 647 | 235 | 882 |
Ballistite, German | 1291 | 591 | 231 | 822 |
Ballistite, Italian | 1317 | 58l | 245 | 826 |
and Spanish | | | | |
\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_|\_\_\_\_\_\_\_\_\_\_|\_\_\_\_\_\_\_\_\_\_\_|\_\_\_\_\_\_\_\_\_|\_\_\_\_
\_\_\_\_\_\_\_\_\_\_|

The figures in column headed “Co-efficient of
Potential Energy” serve as a measure of comparison
of the power of the explosives, and are the products
of the number of calories by the volume of gas, the
last three figures being suppressed in order to simplify
the results.

The amounts of water found were calculated for comparison
as volumes of H\_{2}O gas at 0 deg. and 760 mm.

E.C. powder consists principally of nitro-cellulose
mixed with barium nitrate and a small proportion of
camphor.

S.S. of nitro-lignine mixed with barium nitrate and
nitro-benzene.

Troisdorf powder is gelatinised nitro-cellulose; rifleite
gelatinised nitro-cellulose and nitro-benzene.

Cordite contains 58 per cent. nitro-glycerine, 37
per cent. gun-cotton, and 5 per cent. vaseline.

Ballistite (Italian) consists of equal parts nitro-cellulose
and nitro-glycerine, and 1/2 per cent. of aniline.
The German contains a higher percentage of nitro-cellulose.

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TABLE SHOWING THE HEAT DEVELOPED BY EXPLOSIVES CONTAINING
NITRO-GLYCERINE AND NITRO-CELLULOSE IN DIFFERENT PROPORTIONS.

\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_
\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_
Composition of Explosives. | Calories per cent.
\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_|\_\_\_\_\_\_\_\_\_\_\_\_\_\_
\_\_\_\_\_\_\_\_\_\_
Nitro-cellulose | |
(N = 13.3 per cent.). | Nitro-glycerine. |
| |
100 per cent. dry pulp | 0 | 1061
100 " gelatinised | 0 | 922
90 " | 10 per cent. | 1044
80 " | 20 " | 1159
70 " | 30 " | 1267
60 " | 40 " | 1347
50 " | 50 " | 1410
40 " | 60 " | 1467
0 " | 100 " | 1652
\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_|\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_|\_\_\_\_\_\_\_\_\_\_\_\_\_\_
\_\_\_\_\_\_\_\_\_\_
| |
Nitro-cellulose | |
(N=12.24 per cent.) | Nitro-glycerine. |
| |
80 per cent. | 20 per cent. | 1062
60 " | 40 " | 1288
50 " | 50 " | 1349
40 " | 60 " | 1405
| |
\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_|\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_|\_\_\_\_\_\_\_\_\_\_\_\_\_\_
\_\_\_\_\_\_\_\_\_\_|
Nitro-cellulose | |
(N = 13.3 per cent.). | Nitro-glycerine. | Vaseline.
| |
55 per cent. | 40 per cent. | 5 per cent. 1134
35 " | 60 " | 5 " 1280
\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_|\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_|\_\_\_\_\_\_\_\_\_\_\_\_\_\_
\_\_\_\_\_\_\_\_\_\_

TABLE OF RESULTS OBTAINED BY LIEUT. W. WALKE.,
OF THE ARTILLERY, U.S.A, WITH QUINAN’S PRESSURE
GAUGE.

Nitro-glycerine being taken as 100. (From *U.S.
Naval Inst. Jour.*)

\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_
\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_
| | |
| Compression | Order of |
Name of Explosive. | of Lead | Strength. |
| | |
| Inch. | |
Explosive gelatine | 0.585 | 106.17 |
Hellhoffite | 0.585 | 106.17 |
Nitro-glycerine | 0.551 | 100.00 | Standard, N.G.
Nobel’s smokeless powder | 0.509 | 92.38 |
Nitro-glycerine | 0.509 | 92.37 |
Gun-cotton | 0.458 | 83.12 | U.S. naval torpedo
| | | gun-cotton
Gun-cotton | 0.458 | 83.12 | Stowmarket.
Nitro-glycerine | 0.451 | 81.85 | Vouges, N.G.
Gun-cotton | 0.448 | 81.31 |
Dynamite No. 1 | 0.448 | 81.31 |
Dynamite de Traul | 0.437 | 79.31 | *Composition of some of the Explosives in Common
Use.
Ordinary Dynamite.*
Nitro-Glycerine 75 per cent.
Kieselguhr 25 "
 *Amvis.*
Nitrate of Ammonia 90 per cent.
Chloro-di-nitro Benzene 5 "
Wood Pulp 5 "
 *Ammonia Nitrate Powder.*
Nitrate of Ammonia 80 per cent.
Chlorate of Potash 5 "
Nitro-Glucose 10 "
Coal Tar 5 "
 *Celtite.*
Nitro-Glycerine 56-59 parts.
Nitro-Cotton 2-3.5 "
KNO\_{3} 17-21 "
Wood Meal 8-9 "
Ammonium Oxalate 11-13 "
Moisture 0.5-1.5 "
 *Atlas Powders.*
Sodium Nitrate 2.0 per cent.
Nitro-Glycerine 75.0 "
Wood Pulp 21.0 "
Magnesium Carbonate 2.0 "
 *Dauline.*
Nitro-Glycerine 50 per cent.
Sawdust 30 "
Nitrate of Potash 20 "
 *Vulcan Powder.*
Nitro-Glycerine 30 per cent.
Nitrate of Soda 52.5 "
Sulphur 7.0 "
Charcoal 10.5 "
 *Vigorite.*
Nitro-Glycerine 30 per cent.
Nitrate of Soda 60 "
Charcoal 5 "
Sawdust 5 "
 *Rendrock.*
Nitrate of Potash 40 per cent.
Nitro-Glycerine 40 "
Wood Pulp 13 "
Paraffin or Pitch 7 "
 *Ammonia Nitrate Powder.*
Ammonia Nitrate 80 per cent.
Potassium Chlorate 5 "
Nitro-Glucose 10 "
Coal Tar 5 "
 *Hercules Powders.*
Nitro-Glycerine 75 to 40 per cent.
Sugar 1 " 15.66 "
Chlorate of Potash 1.05 " 3.34 "
Nitrate of Potash 2.10 " 31.00 "
Carbonate of Magnesia 20.85 " 10.00 "

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*Carbo-Dynamite.*
Nitro-Glycerine 90 per cent.
Charcoal 10 "
 *Geloxite (Permitted List).*
Nitro-Glycerine 64-54 parts.
Nitro-Cotton 5-4 "
Nitrate of Potash 22-13 "
Ammonium Oxalate 15-12 "
Red Ochre 1-0 "
Wood Meal 7-4 "

The Wood Meal to contain not more than 15% and not
less than 5% moisture.
 *Giant Powder.*
Nitro-Glycerine 40 per cent.
Sodium Nitrate 40 "
Rosin 6 "
Sulphur 6 "
Guhr 8 "
 *Dynamite de Trauzel.*
Nitro-Glycerine 75 parts.
Gun-Cotton 25 "
Charcoal 2 "
 *Rhenish Dynamite.*
Solution of N.G. in Naphthalene 75 per cent.
Chalk, or Barium Sulphate 2 "
Kieselguhr 23 "
 *Ammonia Dynamite.*
Ammonia Nitrate 75 parts.
Paraffin 4 "
Charcoal 3 "
Nitro-Glycerine 18 "
 *Blasting Gelatine.*
Nitro-Glycerine 93 per cent.
Nitro-Cotton 3 to 7 "
 *Gelatine Dynamite.*
Nitro-Glycerine 71 per cent.
Nitro-Cotton 6 "
Wood Pulp 5 "
Potassium Nitrate 18 "
 *Gelignite.*
Nitro-Glycerine 60 to 61 per cent.
Nitro-Cotton 4 " 5 "
Wood Pulp 9 " 7 "
Potassium Nitrate 27 "
 *Forcite.*
Nitro-Glycerine 49 per cent.
Nitro-Cotton 1.0 "
Sulphur 1.5 "
Tar 10.0 "
Sodium Nitrate 38.0 "
Wood Pulp 5 "
 (The N.-G., &c., varies.)
 *Tonite No. 1.*
Gun-Cotton 52-50 per cent.
Barium Nitrate 47-40 "
 *Tonite No. 2.*
Contains Charcoal also.
 *Tonite No. 3.*
Gun-Cotton 18 to 20 per cent.
Ba(NO\_3)\_2 70 " 67 "
Di-nitro-Benzol 11 " 13 "
Moisture 0.5 " 1 "
 *Carbonite.*
Nitro-Glycerine 17.76 per cent.
Nitro-Benzene 1.70 "
Soda 0.42 "
KNO\_3 34.22 "
Ba(NO\_3)\_2 9.71 "
Cellulose 1.55 "
Cane Sugar 34.27 "
Moisture 0.36 "
 \_\_\_\_\_\_\_\_

99.99
 *Roburite.*
Ammonium Nitrate 86 per cent.
Chloro-di-nitro-Benzol 14 "
 *Faversham Powder.*
Ammonium Nitrate 85 per cent.
Di-nitro-Benzol 10 "
Trench’s Flame-extinguishing Compound 5
 "
 *Favierite No. 1.*
Ammonium Nitrate 88 per cent.
Di-nitro-Naphthalene 12 "

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*Favierite No. 2.*
No. 1 Powder 90 per cent.
Ammon. Chloride 10 "
 *Bellite.*
Ammonium Nitrate 5 parts.
Meta-di-nitro-Benzol 1 "
 *Petrofacteur.*
Nitro-Benzene 10 per cent.
Chlorate of Potash 67 "
Nitrate of Potash 20 "
Sulphide of Antimony 3 "
 *Securite.*
Mixtures of Meta-di-nitro-Benzol 26 per cent.
and Nitrate of Ammonia 74 "
 *Rack-a-Rock.*
Potassium Chlorate 79 parts.
Mono-nitro-Benzene 21 "
 *Oxonite.*
Nitric Acid (sp. gr. 1.5) 54 parts.
Picric Acid 46 "
 *Emmensite.*
Emmens Acid 5 parts.
Ammonium Nitrate 5 "
Picric Acid 6 "
 *Brugere Powder.*
Ammonium Picrate 54 per cent.
Nitrate of Potash 46 "
 *Designolle’s Torpedo Powders.*
Potassium Picrate 55 to 50 per cent.
Nitrate of Potash 45 " 50 "
 *Stowite.*
Nitro-Glycerine 58 to 61 parts.
Nitro-Cotton 4.5 " 5 "
Potassium Nitrate 18 " 20 "
Wood Meal 6 " 7 "
Oxalate of Ammonia 11 " 15 "

The Wood Meal shall contain not more than 15% and
not less than 5% by weight of moisture. The explosive
shall be used only when contained in a non-water-proofed
wrapper of parchment—­No. 6 detonator.
 *Faversham Powder.*
Nitrate of Ammonium 93 to 87
Tri-nitro-Toluol 11 " 9
Moisture 1 " —­
 *Kynite.*
Nitro-Glycerine 24-26 parts.
Wood-Pulp 2.5-3.5 "
Starch 32.5-3.5 "
Barium Nitrate 31.5-34.5 "
CaCO\_{3} 0-0.5 "
Moisture 3.0-6.0 "

Must be put up only in water-proof parchment paper,
and No. 6 electric detonator used.
 *Rexite.*
Nitro-Glycerine 6.5-8.5 parts.
Ammonium Nitrate 64-68 "
Sodium Nitrate 13-16 "
Tri-nitro-Tolulene 6.5-8.5 "
Wood Meal 3-5 "
Moisture .5-1.4 "

Must be contained in water-proof case (stout paper),
water-proofed with
Resin and Cerasin—­No. 6 detonator.
 *Withnell Powder.*
Ammonium Nitrate 88-92 parts.
Tri-nitro-Toluene 4-6 "
Flour (dried at 100 deg. C.) 4-6 "
Moisture 0-15 "

Only to be used when contained in a linen paper cartridge,
water-proofed with Carnuba Wax, Parrafin—­No.
7 detonator used.
 *Phenix Powder.*
Nitro-Glycerine 28-31 parts.
Nitro-Cotton 0-1 "
Potassium Nitrate 30-34 "
Wood Meal 33-37 "
Moisture 2-6 "
 *SMOKELESS POWDERS.*

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*Cordite.*
Nitro-Glycerine 58 per cent. +or- .75
Nitro-Cotton 37 " +or- .65
Vaseline 5 " +or- .25
 *Cordite, M.D.*
Nitro-Glycerine 30 per cent. +or- 1
Nitro-Cotton 65 " +or- 1
Vaseline 5 " +or- .25

Analysis of—­
 By W. Mancab and A.E. Leighton.
 *E.C. Powder.*
Nitro-Cotton 79.0 per cent.
Potassium Nitrate 4.5 "
Barium Nitrate 7.5 "
Camphor 4.1 "
Wood Meal 3.8 "
Volatile Matter 1.1 "
 *Walarode Powder.*
Nitro-Cotton 98.6 per cent.
Volatile Matter 1.4 "
 *Kynoch’s Smokeless.*
Nitro-Cotton 52.1 per cent.
Di-nitro-Toluene 19.5 "
Potassium Nitrate 1.4 "
Barium Nitrate 22.2 "
Wood Meal 2.7 "
Ash 0.9 "
Volatile Matter 1.2 "
 *Schultze.*
Nitro-Lingin 62.1 per cent.
Potassium Nitrate 1.8 "
Barium Nitrate 26.1 "
Vaseline 4.9 "
Starch 3.5 "
Volatile Matter 1.0 "
 *Imperial Schultze.*
Nitro-Lignin 80.1 per cent.
Barium Nitrate 10.2 "
Vaseline 7.9 "
Volatile Matter 1.8 "
 *Cannonite.*
Nitro-Cotton 86.4 per cent.
Barium Nitrate 5.7 "
Vaseline 2.9 "
Lamp Black 1.3 "
Potassium Ferro-cyanide 2.4 "
Volatile Matter 1.3 "
 *Amberite.*
Nitro-Cotton 71.0 per cent.
Potassium Nitrate 1.3 "
Barium Nitrate 18.6 "
Wood Meal 1.4 "
Vaseline 5.8 "
 *Sporting Ballistite.*
Nitro-Glycerine 37.6 per cent
Nitro-Cotton 62.3 "
Volatile Matter 0.1 "

The following is a complete List of the Permitted
Explosives as Defined in the Schedules to the Explosives
in Coal Mines Orders of the 20th December 1902, of
the 24th December 1903, of the 5th September 1903,
and 10th December 1903:—­

Albionite.
Ammonal.
Ammonite.
Amvis.
Aphosite.
Arkite.
Bellite No. 1.
Bellite No. 2.
Bobbinite.
Britonite.
Cambrite.
Carbonite.
Clydite.
Coronite.
Dahmenite A.
Dragonite.
Electronite.
Faversham Powder.
Fracturite.
Geloxite.
Haylite No. 1.
Kynite.
Negro Powder.
Nobel’s Ardeer Powder.
Nobel Carbonite.
Normanite.
Pit-ite.
Roburite No. 3.
Saxonite.
Stow-ite.
Thunderite.
Victorite.
Virite.
West Falite No. 1.
West Falite No. 2.

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Emmensite | 0.429 | 77.86 |
Amide powder | 0.385 | 69.87 |
Oxonite | 0.383 | 69.51 |
Tonite | 0.376 | 68.24 | G.C. 52.5%, and
| | | Ba(NO\_{3})\_{2}, 47.5%
Bellite | 0.362 | 65.70 |
Rack-a-rock | 0.340 | 61.71 |
Atlas powder | 0.333 | 60.43 |
Ammonia dynamite | 0.332 | 60.25 |
Volney’s powder No. 1 | 0.322 | 58.44 | Nitrated naphthalene.
" No. 2 | 0.294 | 53.18 | " "
Melinite | 0.280 | 50.82 | Picric acid 70%, and
| | | sol. nitro-cotton 30%.
Silver fulminate | 0.277 | 50.27 |
Mercury | 0.275 | 49.91 |
Mortar powder | 0.155 | 28.13 |
\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_|\_\_\_\_\_\_\_\_\_\_\_\_\_|\_\_\_\_\_\_\_\_\_\_\_|\_\_\_\_\_\_\_\_
\_\_\_\_\_\_\_\_\_\_\_\_\_\_

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| Stibnite Taken. |Sb\_{2}S\_{3}, Found.| Error. |
| Grm. | Grm. | Grm. |
| | | |
| 0.0085 | 0.0084 | -0.0001 |
| 0.0098 | 0.0099 | +0.0001 |
| 0.0160 | 0.0157 | -0.0003 |
| 0.0099 | 0.0100 | +0.0001 |
|\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_|\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_|\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_
\_|

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| Dry, weigh, and exhaust with water preferably in Soxhlet. |
|\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_
\_\_\_\_\_\_\_\_\_\_\_\_|
| | |
| *Solution*-- | *Residue*-- |
| Contains metallic | Dry, weigh, and agitate an aliquot part with |
| nitrates, chlorates, | with H\_{2}SO\_{4} and Hg in nitrometer. If |
| soluble carbonates, | nitro-cellulose is present, treat remainder of |
| the sum of which | residue with ether-alcohol. |
| (except AmCO\_{3}) |\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_|
| can be determined by | |
| evaporating down at | *Solution*-- |
| 100 deg. C. to dryness | Evaporate and weigh. Residue consists of |
| and weighing. | soluble nitro-cellulose. |
| Nitrates can be |\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_|
| determined by | |
| | *Residue*-- |
| | Dry and weigh and determine hexa-nitro- |
| | cellulose in nitrometer, if present. Exhaust |
| | remainder with acetic ether. |
| |\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_|
| | | |
| | *Solution*-- | *Residue*-- |
| | Hexa-nitro-cellulose | Dry and weigh, ignite |
| | (Gun cotton). | and reweigh. Loss = |
| | | *Cellulose*. |
| | |\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_|
| | | |
| | | Residue consists of |
| | | sawdust, charcoal, |
| | | coal, chalk, guhr, |
| | | or mineral matter, &c. |
|\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_|\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_|\_\_\_\_\_\_\_\_\_\_\_\_\_
\_\_\_\_\_\_\_\_\_\_\_\_|

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| | | | | |
| Cordite 0.4 inch | 5,413 | 5,994 | 6,950 | 7,478 |
| Ballistite 0.3 inch cubes | 4,227 | 4,754 | 5,479 | 5,852 |
| French B.N. | 4,047 | 4,463 | 5,104 | 5,460 |
| Prismatic amide | 3,507 | 3,862 | 4.460 | 4,745 |
|\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_|\_\_\_\_\_\_\_\_\_\_|\_\_\_\_\_\_\_\_\_\_|\_\_\_\_\_\_\_\_
\_\_|\_\_\_\_\_\_\_\_\_\_|

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| (25 per cent. silica)| 2,940 | 80 | 1,468 |
| | | | |
|Gun-cotton, 1 | 2,650 | ... | ... |
| | 2,060 | 90.5 | 1,450 |
| | | | |
|Ammonium nitrate | 1,130 | ... | ... |
|\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_|\_\_\_\_\_\_\_\_\_\_\_\_\_|\_\_\_\_\_\_\_\_\_\_\_\_\_|\_\_\_\_
\_\_\_\_\_\_\_\_\_|

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|13. Partly CLear| 1.8378 | 1.4257 | ... | 60 | 1 | 40 |
|14. Cloudy | 1.837 | 1.4257 | 1 | 56 | 4 | 40 |
|\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_|\_\_\_\_\_\_\_\_\_\_\_\_|\_\_\_\_\_\_\_\_|\_\_\_\_\_\_|\_\_\_\_\_\_\_\_|\_\_\_\_
\_\_|\_\_\_\_\_\_\_\_|
| | | |
| |Temp., Deg. C. | Percentage |
| |\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_|\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_|
| | | | | |
| | From | To | Increase. | Loss. |
|\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_|\_\_\_\_\_\_\_|\_\_\_\_\_\_\_|\_\_\_\_\_\_\_\_\_\_\_|\_\_\_\_\_\_\_|
| | | | | |
| 1. Clear | 57 deg. | 62 deg. | 31 | ... |
| 2. " | 60 deg. | 62 deg. | 18 | ... |
| 3. Cloudy | 60 deg. | 62 deg. | 7 | ... |
| 4. Rain | 60 deg. | 63 deg. | 0 | 0 |
| 5. Clear | 58 deg. | 62 deg. | 15 | ... |
| 6. Rainy | 58 deg. | 62 deg. | ... | 2 |
| 7. Cloudy | 62 deg. | 65 deg. | ... | 10 |
| 8. Clear | 60 deg. | 62 deg. | 5 | ... |
| 9. Partly Clear| 50 deg. | 60 deg. | ... | 3 |
|10. " | 58 deg. | 60 deg. | ... | 10 |
|11. Cloudy | 58 deg. | 60 deg. | 8 | ... |
|12. Rainy | 58 deg. | 60 deg. | ... | 10 |
|13. Partly CLear| 50 deg. | 58 deg. | 20 | ... |
|14. Cloudy | 50 deg. | 60 deg. | 16 | ... |
|\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_|\_\_\_\_\_\_\_|\_\_\_\_\_\_\_|\_\_\_\_\_\_\_\_\_\_\_|\_\_\_\_\_\_\_|