contain about ten grams to the liter. On the basis of one gram of ore taken for assay, one cc. of this solution will equal about one per cent. of lead. It is standardized on about 0.30 to 0.40 gram of pure lead, reduced from the acetate. This is dissolved in a little dilute nitric acid, (two water to one acid), then boiled with ten cc. of sulphuric acid and put through the entire process.

An ore may be assayed in thirty minutes by the above method.

While the above method answers in ordinary cases, to provide against bismuth and antimony (of which the basic sulphates might cause trouble), the following modification may be adopted: To the sufficiently cool residue of the sulphuric acid evaporation add ten ec. of dilute sulphuric acid, (I:IO), and about two grams of Rochelle salt. When this is dissolved add forty cc. of water, heat to boiling and proceed as usual.

Line interferes with this method only by a solvent action exercised by the acetate on the brown uranium ferrocyanide of the test. Thirty per cent. of lime in an ore, however, occasions no serious interference. Calcium sulphate must not be allowed in the acetate solution as it would cause a loss of lead as sulphate.

OCTOBER 25, 1893.

THE GELATINE EXPLOSIVES.

BY P. GERALD SANFORD, F. 1. C., F. C. S. Received October 5, 1893.

A MONG the various forms of explosives the material known as gelatine is one of the most important, and is rapidly displacing the older forms of 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.*, the penta and tetra-nitrates, the hexa-nitro-cellulose being known as gun-cotton) dissolved in nitroglycerol, and made up with various proportions of wood pulp, and some nitrate, or other material of a similar nature. Blasting gelatine consists of collodion-cotton and nitroglycerol without any other substance and was patented by 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 India-rubber, and generally consists of ninety-two per cent. to ninety-three per cent. of nitroglycerol and seven per cent. to eight 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:

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

The soluble cotton, which is a mixture of tetra and pentanitro-cellulose, $C_{12}H_{14}(ONO_2)_6$ is soluble in ether-alcohol, in acetone, and in nitroglycerol; the hexa-nitro-cellulose or guncotton, $C_{12}H_{14}O_4(ONO_2)_6$, on the other hand is not soluble in nitroglycerol, or in ether-alcohol, but is soluble in acetone,¹ or acetic ether.

It is very essential, therefore, that the nitro-cotton used in the manufacture of the gelatine compounds 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 a half per cent. The cotton before being used should always be tested by the government heat test, because if the cotton used does not stand this test the gelatine made with it can hardly be expected to do so. The 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. The percentage of nitrogen in the nitrated cotton should be over eleven 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. The cotton, which is generally packed wet in zinc lined wooden boxes,

¹The smokeless powder, "cordite," contains thirty-seven per cent. of gun-cotton dissolved in fifty-eight per cent. of nitroglycerol. This gun-cotton is first dissolved in acetone before admixture with the nitroglycerol.

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 both dangerous and comparatively useless. In order to find out how long any sample of cotton requires to be dried, a sample should be taken from the center of several boxes, well mixed, and about 1,000 grams spread out on a paper tray, weighed, and the whole then placed in the water oven at 100° 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 twenty to thirty per cent. After drying for a period of forty-eight hours, a sample should be again dried in the oven at 100° C. and the moisture determined and so on, at intervals, until the bulk of the cotton is found to be dry, *i. c.*, 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 about two per cent. of moisture from the air at ordinary temperatures. 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 two inches. This current of air can be obtained in any way that may be found convenient, such as by means of a fan, or Roots 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 continually be 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 woodwork 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, etc.) may become very hot, when the slightest friction might cause an 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 oil cloth or linoleum, as being soft it lessens the friction. List shoes should always be worn in this building, and a thermometer hung up somewhere about the center 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 tray), the one nearest to the hot air inlet should be selected; if the temperature of the house is kept at about 40° 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 India-rubber 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, etc. The following are analyses of some typical samples of the three compounds:

	Gelatine.	Gelatine- dynamite.	Blasting gelatine.
Nitroglycerol	60.514	71.128	92.94
Nitro-cellulose	4.888	7.632	7.06
Wood pulp	7.178	4.259	• • • •
Potassium nitrate	27.420	16.720	••••
Water	••••	0.261	••••
	100.000	100.000	100.000

The gelatine and gelatine-dynamite consists 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 test; the higher percentage of cotton too makes it expensive.

When the dry 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 nitroglycerol poured upon it: the whole is then well stirred up, and kept at a temperature of from 40° to 45° C.; it should not be allowed to go much above 40° C. (but higher temperature may be used if the cotton is very obstinate and will not dissolve at 40° C.: great caution must, however, be observed in this case). The mixture should be constantiv worked about by the workman with a wooden paddle for at least half an hour. At a temperature of 40° - 45° the nitroglycerol acts upon the nitro-cotton and forms a jelly. Without heat the gelatinization is very imperfect indeed, and at temperatures under 40° C, takes place very slowly. The limit of temperature is 50° or thereabout : beyond this the jelly should never be allowed to go, and to 50° 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 frame work, 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° C. if it is intended to gelatinize at 45° and about 80° C. if at 50°, 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. The best way to keep the temperature of the water constant is to have a large tank of water raised upon a platform, some five or six feet high, ontside 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 a mixing machine. An ordinary bread-kneading machine does very well. It must, of course, be made of gun-metal. There must be no iron about the working parts, and the bearings must be carefully looked to. A very suitable masticating machine for this purpose is supplied by Messrs. Werner and Pfleiderer, of London, or G. McRoberts' machine may be used. This latter is the form of machine' that is used at Nobel's factories.

If it is intended to make gelignite, or gelatine-dynamite, it is at this point that the proper proportions of wood pulp and potassium nitrate should be added and the whole well mixed for at least half an hour until the various ingredients are throughly incorporated. 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 throughly 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. The application of heat in the manufacture of jelly from collodion-cotton and nitroglycerol is absolutely necessary, unless some other solvent is used besides nitroglycerol, such as acetone, acetic ether, methyl or ethyl alcohol. These compounds 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. They also lower the temperature at which the nitroglycerol becomes congealed, i. e., they lower the freezing point of the resulting gelatine. The finished gelatine upon entering the cartridge huts, is at once transferred to the cartridge-making machine, which is very like an ordinary sausage-making machine.2 The whole thing must be made of gun metal, or brass, and it consists of a conical case containing a shaft and

1 See J. Soc. Chem. Ind., 1890, 267.

²G. McRoberts, J. Soc. Chem. Ind., March 31, 1890, p. 266.

screw. The revolutions of the shaft causes 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. The issuing gelatine can of course be cut off at any length. This is best done with a piece of wood planed down to a cutting edge, i. c., wedge-shaped. It is also essential that the machines should have no metallic contacts inside. The bearings for the shaft must be fixed outside the cone containing the gelatine. One of these machines can convert from five to ten hundred-weight of gelatine into cartridges per diem, depending upon the diameter of the cartridges made. After being cut up into lengths of about three inches, the gelatine is rolled up in cartridge paper; water-proof paper is generally used. The cartridges are then packed away in card-board boxes, which are again packed in deal boxes lined with India-rubber and screwed down air-tight, brass screws, or zinc or brass nails being used for the purpose. These boxes are then ready for the magazines or export. Before the boxes are fastened down, a cartridge or so should be removed and tested by the government heat test, the liquefaction test, and the test for liability to exudation (as described in 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 insure that the gelatine shall be able to withstand a fairly high temperature without melting or running together. The test is carried out as follows: A cylinder of the gelatine is cut from the cartridge of a length equal to its diameter. The edges nust be sharp. This cylinder is to be placed on end on a flat surface (such as paper), and secured by a pin through the center, and exposed for 144 consecutive hours to a temperature of 85°-90° F; and during such time the cylinder should not diminish in height by more than one-fourth inch, and the cut edges should remain sharp. There should also be no stain of nitroglycerol 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 nitroglycerol. All the materials used in the manufacture of gelatime explosives should be subjected to analytical examination before use, as success largely depends upon the purity of the raw materials.⁹

Properties of the Gelatine Compounds .- Blasting gelatine is generally composed of ninety-three to ninety-five parts nitroglycerol and five to seven parts of nitro-cellulose, but the relative proportions of explosive base and nitroglycerol, etc., in the various forms of gelatine compounds 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 vary 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 nitroglycerol, but this is sometimes done in order to change a shattering agent into a propulsive agent. If this process be carried too far we of course lose the advantages due to the presence of nitroglycerol. There is therefore a limit to these additions.² The homogeneousness and stability of the mixture are of the highest importance. It is highly essential that the nitroglycerol 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 nitroglycerol, 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. Nitroglycerol 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 detonator 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 nitroglycerol); that of gun-cotton (dry) 1.0. Blasting gelatine burns in the air when unconfined without explosion, at least in small quantities and when not previously heated; but

¹ For some of the chief methods in use, see J. Anal. Appl. Chem., June, 1892.

² Mica is said to increase the rapidity of explosion when mixed with gelatine.

it is rather uncertain in this respect. It can be kept at a moderately high temperature (70° C.) without decomposition. At higher temperatures the nitroglycerol will partially evaporate : when slowly heated it explodes at 204° C. If however it contains as much as ten per cent. of camphor it burns without exploding. According to Berthelot,' gelatine composed of 91.6 per cent. nitroglycerol and 8.4 per cent. of nitro-cellulose, which are proportions corresponding to total combustion, produces by explosion $177CO_2 + 143H_2O + 8N_3$.

He takes $C_{24}H_{22}(NO_3H)_{*}O_{11}$ as the formula of the nitro-cellulose, and $51C_3H_2(NO_3H)_3 + C_{24}H_{22}(NO_3H)_9O_{11}$ as the formula of the gelatine itself, its equivalent weight being 12360 grams. The heat liberated by its explosion is equal to 19381 calories, or for one kilo., 1535 calories, volume of gases reduced temperature equals 8950 liters. The relative value² of blasting gelatine to nitroglycerol is as 1.4 to 1.45, Kieselguhr dynamite being taken as 1.0. Experiments made in lead cylinders gave the relative value of

Dynamite	1.0
Blasting gelatine	1.4
Nitroglycerol	I.4

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NOTES ON LÖWENTHAL'S METHOD FOR THE DETER-MINATION OF TANNIN.

BY HARRY SNYDER. Received September 22, 1893.

THE determination of tannin is a difficult chemical problem, and to meet this difficulty no less than sixty methods and modifications have been proposed. As early as 1870, Dr. Günther, in the *Chemical News*, said that "none of the different methods for the determination of tannin answer the purpose equally well; that is, a method that yields excellent results for the tannin in nut-galls is not applicable to the tannin in sumac."

¹ Explosives and Their Power, M. Berthelot.

² Messrs. Roux and Sarran.