

(*Phaseolus vulgaris*), and the adzuki bean (*Phaseolus radiatus*).

3. The cow pea contains a third globulin, extremely soluble in very dilute salt solutions, which could be precipitated but partially by dialysis in water and completely only in the coagulated form by dialysis in alcohol. This substance closely resembles, in properties and composition, bodies obtained from several other leguminous seeds. Its composition, as found by analysis of two precipitates, one obtained by dialysis in water and the other by further dialysis in alcohol, is as follows :

|               |        |
|---------------|--------|
| Carbon.....   | 53.25  |
| Hydrogen..... | 7.07   |
| Nitrogen..... | 16.36  |
| Sulphur.....  | 1.11   |
| Oxygen.....   | 22.21  |
|               | 100.00 |

## ON THE MANUFACTURE OF DYNAMITE.

BY G. E. BARTON.

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UNDER the general term dynamite are included two classes of mixtures. The first of these consists of nitroglycerol, held by an absorbing medium, such as woodpulp or kieselguhr, together with various other bodies used as fillers, the principal one being sodium nitrate; this latter body also adds to the strength of the dynamite when woodpulp is the absorbent used by furnishing oxygen for the combustion. The second class, more properly called gelatin dynamites, consists of nitroglycerol gelatinized by means of nitrated cellulose, much the same substances being present as fillers, but considerable less absorbing material in proportion to the amount of nitroglycerol. It is to the former class of mixtures that this paper relates, although most of it is equally applicable to the latter. Most of the dynamite used in this country is of the first class, as it is less costly and equally serviceable, except in wet holes.

The choosing of a site for a dynamite factory is a matter into which many factors enter. It is much more convenient, cheaper, and safer to have the part of the plant used in making the nitroglycerol on a hillside, as it is then possible to allow that liquid to flow from building to building in pipe lines. Authorities usually hold that the soil should not be sandy on account of the

danger from sand blowing into the buildings and finally giving rise to friction in some part of the apparatus. The danger from this source is very small, as can be seen from the fact that one factory (the only one known to the author on such soil) has been located for about fifteen years on a sandy hillside without having an accident which could be traced to this cause. The only precautions taken are to carefully clean everything after each day's work, which must be done anyway, and the sweeping out of the sand occasionally as it accumulates before the doors. The sandy soil probably plays a very important part in another direction, for though the most violent thunder storms sweep over the region during many months of the year, there has never been a building struck by lightning. No lightning protectors whatever are used. The explanation of this fact seems to be that the light buildings, with their very light foundations, are much poorer conductors of electricity than the surrounding trees, the roots of which extend into the moist earth below. Another advantage is that the shock of the most violent explosion is not conducted to great distances, as is the case in other soils. In the factory before mentioned a catch box for the nitroglycerol, carried away in the wash waters, in which about one hundred and fifty pounds of that explosive had accumulated (contrary to orders) exploded. It was situated not more than one hundred feet from the storehouse, in which was considerable nitroglycerol at the time, but this did not explode, although the flying débris damaged the building badly.

The manufacture of nitroglycerol is the most important point to be considered in connection with the manufacture of dynamite and the different parts of the process will be taken up in the order in which they occur. The nitration is best conducted in a nitrator of cylindrical shape and made of lead with a bottom sloping slightly toward the point at which the faucet for drawing off the mixture of nitroglycerol and acids is placed. The top is made of lead and has several panes of glass set in it, through which the workman watches the operation. From the center a ventilator of lead passes up through the roof. Two thermometers are fixed, one with the bulb near the surface of the liquid, and the other with it in the lower strata. These are in front of the operator at work, and guide him. The glycerol valve is

controlled with the left hand, and the compressed air for agitating with the right.

The method of mixing by compressed air is beyond doubt the safest, as it gives a more thorough mixing, allows more than one cooling coil to be used, and is less liable to get out of order. A drawback, common to all mechanical agitators, is the unreliability of any piece of machinery exposed to the acid fumes which are always present in and near the nitrating house. The air after leaving the compressor should be passed through an iron drum containing sulphuric acid, and arranged in the same manner as the ordinary bottle for washing gases in the laboratory, in order that any moisture it may contain may be removed.

Everything being in order and tested as far as possible, the operator sets the lead trough, which connects the discharging faucet with the separator, or drowning tank, as desired, to connect with the drowning tank, starts the water running into the tank so as to give some circulation and consequent stirring there in case it becomes necessary to drown the charge and allows the mixed acids to run into the nitrator from a small tank, into which just enough for one charge has previously been forced from the acid store tank outside the danger area by means of a montejus and compressed air. The cooling coils furnish enough water to keep up a circulation in the drowning tank when allowed to discharge into it. About 1,500 pounds of mixed acid is found to be an economical charge, and to this should be added from 210 to 230 pounds of glycerol, according to the conditions. It is economical to increase the glycerol used, even though the percentage yield decreases, as long as a pound of glycerol adds a pound to the yield of nitroglycerol. The acids used should have the following composition: Sulphuric acid, 61.9 per cent.; nitric acid, 34.5 per cent.; oxides of nitrogen, calculated as nitrogen trioxide, 0.7 per cent., with not more than a trace of chlorine and iron. The glycerol must have approximately the following composition, when examined by the method given in this Journal, 17, 277: Ash, trace; carbonaceous residue, 0.012 per cent.; sodium chloride, 0.002 per cent.; total acid equivalent, 0.05347; permanent specific gravity, 1.2653; specific gravity, 1.2634; higher fatty acids, none; reaction, neutral. During the process of nitration the temperature should not

be allowed to go above 88° F. The yield by this method is greater the shorter the time employed in the process, so that in the winter, when a run can be made in one-half an hour or less, fifteen or twenty pounds more of glycerol can be used without decreasing the percentage yield. This is partly due to the fact that the shorter the time the less nitric acid is blown out of the mixture in agitating. No economical method has been devised for recovering the acid thus lost or for reducing the temperature during the summer to the point easily obtainable in this climate during the winter months.

The nitration being finished, the trough is placed so that the mixture can flow into the separating boxes, which transfer ought not to take over three minutes. It is usually held that the separators should be in another building, but many factories in this country have them in the same one with the nitrator. By this method only one drowning tank is required, while if the buildings are far enough apart for one to escape injury from an explosion in the other, two must be provided. It also avoids the trouble which would otherwise be caused by passing the mixture of acids and nitroglycerol through a long trough.

It is almost impossible to avoid some of the mixture remaining in the hollows of the trough, and this in a few hours, especially during the summer, becomes dangerous.

While an explosion would not necessarily be communicated to either building, it must at least injure the line and delay work. No way would be at hand of disposing of the run then in the nitrator, except by drowning, with the consequent loss of the spent acids. The disadvantage is that an explosion destroys more apparatus and material. The danger in a building containing the separators and nitrator is not increased to any extent, as both operations, when properly conducted, give ample warning before an explosion. The building must, of course, be arranged with several exits.

Two men working with two separators and a nitrator, can easily dispose of twelve runs in a working day of ten hours, which means 5,500 pounds of nitroglycerol. If it is necessary to make more, such a plant can be forced to fourteen or fifteen runs a day, under favorable conditions, by working a little overtime. If still more nitroglycerol be required, it is best to build another

similar plant, far enough removed from the first to avoid danger. This latter plan also has the advantage that a single explosion does not entirely stop the production, which is a thing greatly to be desired, as anyone familiar with the demands of trade for the rapid filling of orders will appreciate.

The separators should be cylindrical lead boxes, containing both cooling coils and an arrangement for agitating with air, the latter for use only in emergencies. The bottom is made slightly sloping towards the point where the cock for drawing off both acids and nitroglycerol is placed. The top should be covered with lead, and have glass windows for viewing the fumes given off during the operation. It is necessary to have a narrow pane of glass in the side next to the cock, by means of which the operator is enabled to see when all the acids have been drawn off, and interrupt the flow before any of the nitroglycerol goes into the second separator. The spent acids having been disposed of, a short lead trough is then set so that the nitroglycerol can flow in a thin sheet into a small tank half filled with water, and connected with the wash-house. A stream of water, sufficient to keep the liquid in this tank at a constant level, is turned on at the same time. The nitroglycerol flows from here into the wash-tank in the wash-house, the stream of water being kept up till the nitroglycerol is drowned, and a sufficient quantity of water passed through the pipe line to clean out the last traces.

The wash-tanks should be made of wood, and lined with lead. The agitation is best done with compressed air. Two faucets are set in each of these tanks, one at the bottom for drawing off the nitroglycerol at the end of the washing, and one above the surface of the explosive through which the wash-waters escape. An alkaline wash must always be used at least once, and the nitroglycerol tested with litmus paper before it is allowed to go to the storehouse. The disposal of the wash-waters is a problem which every manager must solve for himself according to conditions at hand, but it will effect quite a saving if they are allowed to flow through a series of compartments in which the little nitroglycerol left in them has a chance to settle out. The nitroglycerol should be removed from these compartments at least once a week. The lead lining to the wash-tanks should be insisted upon, as otherwise the fibers of the wood are opened by

the action of the acid and alkali, and become saturated with nitroglycerol, making the repairing or destruction of the tanks dangerous. The author has traced at least one explosion to this cause.

From the wash-house, the nitroglycerol may be allowed to flow at once into the storage tanks, which are in a separate building. In this country, where there is no limit placed on the amount of nitroglycerol which may be stored, or the length of time it may be kept, no filtering or drying process is necessary. There should be several tanks in the storehouse, and the nitroglycerol should stand twenty-four hours before being mixed with the absorbent, thus giving time for the small amount of moisture which it still contains to become thoroughly separated. Each tank should be tested with litmus paper every day, and if any signs of acidity are developed, the nitroglycerol must at once be destroyed or rewashed. The pipe line from the wash-house should be so arranged that it may be broken when not in actual use, thus preventing the communication of an explosion from one building to the other. It should also be carefully guarded against freezing in winter, and becoming overheated in summer.

The spent acid, which we lost track of as it left the first separators, flows in the usual form of trough to the second separators, where the process is continued for at least three, preferably five days. These separators are best oblong lead boxes, having a capacity sufficient to accommodate the spent acid from three runs, and are furnished with cooling coils and air agitators, the last for use only in emergencies. The top of these boxes should have the shape of three quadrangular pyramids, set side by side. These pyramids terminate in glass cylinders about twelve inches in height and four in diameter, which have a glass plug ground into them about six inches from the bottom. The tops of these cylinders are covered with ground glass plates in order that the air may be excluded from the surface of the liquid as much as possible. The top plates having been removed, the spent acid is allowed to flow into the box until it is filled nearly to the top of the glass cylinders. The nitroglycerol finds its way to the surface of the liquid, and when it has accumulated to the depth of an inch or so, the lower level is brought opposite the plug in the side of the cylinder by drawing out a little of the spent acids,

the plug is removed and the nitroglycerol allowed to flow out and into a lead saucer just below, from which it passes into a dish set to catch it under the spout of the saucer. It must be at once emptied into a pail and carried to the wash-house to be washed, or else drowned in a small tank provided in the separating house. The latter plan is preferable, as the danger of an explosion from the shock of dropping a pail is much less after the nitroglycerol has been thoroughly washed.

How to dispose of the spent acids from the nitroglycerol manufacture, which contain from seventy to seventy-four per cent. of sulphuric acid and from seven to twelve per cent. of nitric acid, is a question which probably has caused managers of dynamite factories more thought than almost any other problem connected with the business. It can hardly be said to be solved satisfactorily yet, but in this country the weight of opinion is in favor of regaining the acid. For this purpose it is run at once from the second separators into a montejus and blown into a tank situated near the top of a Glover tower from which it can be run directly into the tower without further precautions. The nitric acid is condensed in any of the usual ways, while the sulphuric acid runs into a tank at the foot of the tower. The sulphuric acid comes out at  $58^{\circ}$  B, or over, in a well-managed tower, and the nitric acid can be kept above  $36^{\circ}$  all the time, usually near  $38^{\circ}$  B. Care must be taken to arrange the pipe leading from the montejus at the separating house to the tank at the Glover tower, so that it will drain itself thoroughly, as otherwise a sediment, composed mostly of lead sulphate, will settle out, together with any small amount of nitroglycerol the acids may still contain, and finally cause an explosion.

A few general rules to be observed in the manufacture of nitroglycerol may be mentioned here :

Nitroglycerol should never be carried in pails when it is possible to avoid it.

All receptacles which contain the nitroglycerol and acids at the same time should be covered so that neither water nor other foreign substance may accidentally come in contact with them. The writer has seen heating sufficient to necessitate the drowning of the contents of one of the second separators, caused by a small piece of wood falling into the mixture.

All receptacles for nitroglycerol, or nitroglycerol and acids together, should be set on a slight incline, so that they may be drained thoroughly on emptying them.

No more nitroglycerol should be made in one plant in a day than can be handled by two men working in a combined nitrating and separating house as described above. If more is to be handled, even occasionally, it will pay to have another plant entirely separate. The second plant is very valuable in case of emergencies, either due to explosions or the necessity for repairs to the apparatus.

The quality of the mixed acids and glycerol used must be given careful attention, and both should be bought under specifications embodying the analytical method. The method given by Lunge for the analysis of the acids is best for this purpose, while that given in this Journal, 17, 277, was devised by the author especially for such use in the case of glycerol. The strict enforcement of such specifications has led to the saving of at least \$5,000 in a single year in the case of a factory turning out 9,000 or 10,000 pounds of dynamite per day.

The mixing of the nitroglycerol with the absorbent may be conducted in a building on the same level as the storehouse for that explosive. The best form of mixing machine now in use, is practically the well-known wheel-mill of the powder manufacturer. It consists of a circular wooden trough capable of containing a mixing of 500 pounds of dynamite at a time. In this trough, two large wooden wheels run, each followed by a brass plough which has a strip of rubber fastened at the lower edge, thus cleaning the trough without requiring an actual contact. Such an apparatus requires less time for mixing than the old method of using rakes, and is more thorough. The only danger is from nails, or other foreign substances getting into the trough, and to avoid this the absorbent should always be sifted. This is done best before the different ingredients are put together. The nitroglycerol should be brought from the storehouse in a small lead-lined tank, placed on wheels, and arranged as a push-cart. This is wheeled upon scales set in the floor of the storehouse, counterbalanced, and the required amount of nitroglycerol for one mix, run into it through an opening in the top. It is then wheeled along a platform to the mix-house.



This platform should have guides to prevent the cart from running off. At the mix-house, the nitroglycerol flows from the tank into the mixing trough through a piece of rubber hose attached to a small spigot in the bottom of the tank. The end of this hose is securely fastened to the top of the tank during the weighing and transportation of the nitroglycerol. This plan for transporting the explosive is superior to any of the old methods using pails, since they, even at the best, require some handling, with the consequent danger of dropping. The danger of spilling is also reduced to a minimum by this method.

The dynamite next passes to the pack-houses, where it is packed in paper cartridges of almost any desired size. Many packing machines are in use, but the old method of filling the cartridge by placing the open end over the tube of a tin funnel, and pressing the dynamite into place with a wooden packing stick, is preferable on the score of safety. From the pack-house the cartridges pass to the casing house, which process presents no particular points of difficulty, and from there to the magazines, or cars, as the case may be.

We now come to the part of the manufacture which is carried on outside the danger limits, and which includes the regaining of the spent acids already described. The arrangement of this part of the plant is solely with regard to convenience, and offers no particular difficulties due to the nature of the work with the possible exception of the storehouse for the sodium nitrate, which should be at a reasonable distance from other buildings, and upon which no water should be thrown in case of fire. The sodium nitrate is best ground and dried in a steam-jacketed pan, in which heavy iron rollers run, as in the wheel-mill before mentioned. Steam-jacketed pans are also provided for the wood-pulp, kieselguhr, etc.

The paper cartridges were formerly made by hand, and a large factory required a considerable number of girls for this purpose; but at least one machine is known to the writer, which does satisfactory work for a considerable range of diameters and lengths by a very simple adjustment.

Besides the examination of the mixed acids and glycerol above mentioned, the chemist should make frequent determinations of the nitroglycerol in the finished product, as a check on the mix-

ing and weighing. The spent and regained acids may be analyzed as a check on the working of the regaining plant, while valuable information is often brought out by an examination of the various absorbents.

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## PROTEID OF THE WHITE PODDED ADZUKI BEAN,<sup>1</sup>

(*Phaseolus Radiatus*).

BY THOMAS B. OSBORNE AND GEORGE F. CAMPBELL.

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THIS is a small red bean cultivated in Japan. The seeds used in this investigation were grown in Kansas and sent to us by Professor C. C. Georgeson.

As our object was to discover the nature of the globulin forming the chief proteid constituent of various leguminous seeds, no attempt was made to determine the total amount of proteid contained in this seed, nor to study the other proteid substances occurring in small quantity.

It was impossible, by any means at our command, to remove the closely adhering red seed-coat, but as it was found that this yielded but little coloring matter to salt solution the entire seed was ground until all passed a sieve of fine bolting cloth.

Two kilograms of this meal were treated with eight liters of ten per cent. sodium chloride solution, and after stirring some time the mixture was strained on fine bolting cloth.

After standing long enough to deposit most of the suspended starch the extract was filtered quite clear and saturated with ammonium sulphate. The precipitate so produced was filtered out, suspended in water, and in order to remove the adherent ammonium sulphate, which prevented solution of the proteid in a sufficiently small volume of water, the mixture was dialyzed over night. The proteid was thus completely dissolved. The solution was filtered perfectly clear and again dialyzed four days. A large precipitate of globulin resulted, which was filtered out and a portion collected on a separate paper and washed thoroughly with water and alcohol and dried over sulphuric acid. This was found to weigh 13.21 grams and formed preparation 1.

<sup>1</sup> From the Report of the Connecticut Agricultural Experiment Station for 1896.