A FLASHING TEST FOR GUNPOWDER.

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Among the methods in use for the determination of the condition and quality of gunpowder, is the "flashing test." According to the *Ordnance Instructions U. S. Navy, pg.* 345, "about eight drams of powder are poured on a glass plate so as to form a conical heap, and 'flashed' by applying a hot iron; no residuum should be left and only a few smoke marks should be seen on the plate." Capt. Smith, R. A., in his *Handbook of the Manufacture and Proof of Gunpowder, pg.* 83, proceeds in the same way, but he places the powder in a thimble-shaped, copper cylinder "which is then inverted on the flashing plate. This provides for the particles being arranged in pretty nearly the same way each time, which is an all-important point in flashing. The decomposition of the powder will be more thorough if it be thrown together in a conical heap, than if it be spread out in a thin layer on the plate, hence, for comparison of different powders, they should be placed on the plates as nearly as possible under the same conditions.

If the powder has been thoroughly and effectually incorporated the small charge placed on the plate will 'flash' or puff off when touched with a hot iron, leaving only smoke marks on the plate. A badly incorporated powder will, on the other hand, leave specks of undecomposed saltpetre and sulphur, and will therefore give a dirty residue. But the 'flashing' test, though apparently most simple, is one which, like the examination by eye and hand, requires experience to enable the observer to form an accurate judgment. Though a very badly incorporated powder could be detected at once, it is by no means easy to judge between two powders, both tolerably good, as to which has undergone the most thorough incorporation. Flashing should therefore be constantly practised with all classes of powders, and it is useful to keep some samples of bad powders to flash occasionally for comparison. Powder which has once been subjected to and injured by damp will be found to flash very badly, no matter how carefully its incorporation may have been performed. This arises from a partial solution of the saltpetre having taken place, causing a consequent disturbance of the incorporation."

Comdr. J. D. Marvin, U. S. N., in his *Objects and Resources of the Naval Experimental Battery, pg.* 18 repeats the above directions and suggests weighing the plate on which the flash has been made,

but, as he provides no means to prevent the absorption of moisture and oxygen and the escape of the hydrogen and ammonium sulphides, the method is of no value.

In the *Comptes Rendus* 78, 1138 ; 1874, Col. Chabrier proposes, what he terms a *pyrographic* method for the examination of gunpowder and a detailed account is given in the *Revue d' Artillerie* 4, 396 ; 1874, of its application by the *Comité de L'Artillerie* in determining the relative value of wheel mills, stamp mills and *moulins à tonneaux* in effecting incorporation and of the length of time necessary in each case to produce the desired result.

This method consist in flashing the powder on sheets of paper, colered blue with iodide starch. Sheets of uniform tint, 0.30 meter long by 0.15 meters wide, are dampened and placed on a plate of glass of the same size. A half gram of powder is then trailed on the paper, following the longer axis. It is flashed by a red hot, iron wire and it is found that the centre of the blue paper is bleached, while black spots and streaks appear on the white ground and white spots on the blue ground. The size and shape of the bleached space and the number and arrangement of the spots and streaks are determined by the character and amount of the powder used. Col. Chabrier does not give the rationale of his process but it is to be inferred from the fact that he styles these results *pyrographic* images, that he believes the bleaching to be due to the heat evolved by the combustion. The well known experiment of the bleaching of starch paste, colored blue with iodine, by heating in a test tube, is an example of the same kind.

This process is an advance upon the older one but in applying it some years ago I found it difficult to prepare papers of the same degree of blueness and that the evanescent character of the color made it difficult to preserve the test papers intact for any considerabe length of time ; so, as before, we must either practice the method continually or else flash powders, which we have kept as standards for comparison, with each set of tests we make, in order to arrive at any good results. Or, finally, we may photograph the test papers, but this involves considerable labor and the loss of the color.

Since the flashing test is the simplest, readiest, and in the hands of an expert, the best test for the incorporation of powder, and, since it also fairly indicates the amount of deterioration which a powder has undergone during transportation and storage, it has seemed to me desirable to seek some method by which the record could be made permanent. Such a record could then be filed at

the factory with the other data concerning a given powder, or, in the case of the Government, they could be inclosed with the quarterly returns of the inspecting officers at distant stations, to be examined by some expert in the Bureau. Specimens of the tests of standard powders could also be furnished inspecting officers, to guide them in the interpretation of the results of their tests, and finally, a sample of the required test might be attached to the specifications for a gunpowder to be purchased.

After some search I believe that I have secured such a permanent record, by employing a paper colored with Turnbull's Blue, upon which to make my flash. This paper is the same as that used in the "Blue Print Process" of photography, and is easily procured in commerce. The use of the paper was suggested by the following facts. When gunpowder burns, the reaction which takes place may, according to Debus, *Proc. U. S. Naval Institute*, 9, 76, 1883, be represented by the reaction $16\ KNO_3 + 13C + 5S = 3K_2CO_3 + 5K_2SO_4 + 9\ CO_2 + CO \times 8\ N_2$.

Since, however, in ordinary gun powders there is more carbon and sulphur than is required by the above equation, secondary, endothermic reactions take place, which may be combined and represented as follows: $3K_2SO_4 + 2K_2CO_3 + 7C + 7S = 5K_2S_2 + 9C\ O_2$.

Further, on exposure to the air, the polysulphides formed are oxidized to thiosulphates. I have observed that, in my experiments, the characteristic smell of the latter was most noticeable when the powder was badly incorporated.

It is well known that solutions of the alkalies and the alkaline carbonates decompose Turnbull's Blue and thereby destroy its color. Advantage has been taken of this reaction to increase the distinctness of "Blue Prints," or to make such additions to them or alterations in them as desired. With this I exhibit a specimen of the blue paper, upon which one of the above reactions is written by the aid of a solution of potassium carbonate. The alkaline sulphides and thiosulphates also act upon the blue paper, but with less intensity and with the partial production of a yellow color. By flashing gunpowder then, upon such paper, yellow and white spots will be formed through the action of the substances formed by the reaction.

The test is made as follows: Pieces of the paper, from 15 to 20 centimetres square, are dampened and placed on a sheet of glass or copper. A truncated leaden cone 3 centimetres in capacity is closed

with the finger at the smaller end, filled evenly with powder and inverted on the paper. The result is a conical heap. The heap is immediately fired, either by a hot iron or copper wire, or, as in my experiments, by a fina platinum wire, heated to incandescence by an electric current. The paper is exposed to the action of the residue for thirty seconds and then immediately placed under the spigot and washed with running water. When pulverized gunpowder cake is used it will be found that the space described by the base of the cone, has been blackened and partially bleached by the dampened layers of powder in contact with it; that about this space are black smutches and streaks, and that the whole surface of the paper is marked by white and yellow dots. Where the powder is badly incorporated the spots are coarse, and irregular in shape and distribution; where the incorporation is complete, the spots are fine and quite uniformly distributed over the surface so that the paper appears but of a paler blue, with occasional spots and few streaks.

With this, I forward specimens of tests made with powdered "mill cake." All of the specimens belong to the same "charge," but the first was drawn after the mill had been running four hours; the second, at the end of eight hours; the third, after twelve hours; and the fourth, after sixteen hours. The latter is known as the "finished composition." This length of running is rather unusual, but the charge used at the mills is greater than common. The tests exhibited were made October 19, 1883. I have yet others made April 26, 1883, which are to-day apparently as fresh and distinct as when made. It is believed that the papers show what is described above. That importance is given, in interpreting the results, to uniformity of the bleaching and in the arrangement of the spots, depends upon the fact that gunpowder is a mechanical mixture, and, therefore, that the regularity of the combustion and the uniformity of the accompanying reactions must depend upon the fineness of the ingredients and the intimacy and uniformity of the mixture. If the ingredients are coarse, and the mixture imperfect, the combustion will go on slowly and irregularly, and the resulting globules of residue will be of considerable size and be deposited near the centre of action. If the incorporation is complete, the reaction will take place nearly simultaneously throughout the whole mass, and the globules will be, as a rule, quite small and projected to some distance. This interpretation is for mealed powders having the same formula. I have not yet been able,

personally, to extend my experiments to granulated powders or powders of varying proportions and ingredients, but I believe that this test will form a useful method for the study of these powders.

In order that the indications may be interpreted aright, it is necessary that the conditions, under which the experiments are made, shall be as nearly uniform as possible, and the first of these is that the color of the test paper should be in all cases as nearly as possible of the same depth. The paper may be purchased in an emergency but it varies among manufacturers owing to the many different formulas according to which it is made. Among these I have selected that issued by the Penn. R. R. Co. for use among its operatives.

"Take 10 oz. (283.5 c. c.) of clean water and put in an opaque bottle, add 1¼ oz. (35. 44 grms), of Red Prussiate of Potash, allow this to dissolve. In a second vessel containing 6 oz. (170. 1 c. c.) of water put 2½ oz. (70. 88 grms), of Ammonio-citrate of Iron, allowing this also to dissolve. Add the second liquid to the first and shake thoroughly. Keep closely stoppered and not exposed to light."

"In a room, from which daylight is excluded, but where lamp or gas light may be used, the paper to be printed on, is laid on a table, and the fluid applied with a *clean* sponge. Care should be taken to apply the fluid as evenly as possible, and every part of one side should be gone over. For that reason it would be well to sponge the paper, first in one direction, and afterwards crosswise to the first. When a sheet is sensitized it is put away in a drawer to dry, but never place one sheet on the top of another before they are dry; afterward it makes no difference. Sensitized paper may be kept in a drawer for a week or more, without injuring its sensitive quality."

"In using the fluid, care should be taken to pour out no more than is needed for the time, as it would be apt to spoil the fluid in the bottle if any fluid which had been used, was poured back again. For the same reason the saucer into which the fluid is poured, and the sponge with which it is applied, should be washed out immediately after using and also before using."

For the purposes of this test for gunpowder the dry sheets are now exposed to strong sunlight for four or five hours. When about to use, immerse in running water for five minutes, lay on the

plate of glass and remove the excess of moisture by aid of filter paper or a blotter. The paper must be thoroughly moistened but without "standing" moisture.

Since writing the above I have received from Lt. Commander W. M. Folger, U. S. N., commanding the Naval Experimental Battery, the following statement concerning the testing of a granulated powder by this method.

"In firing a sample of experimental powder lately, I had reason to believe, from its performance in the gun, (calibre 6"), that the powder was badly incorporated. Tested in the manner you suggested with Turnbull's paper and following all your directions, indications were furnished which (when compared with results obtained with a normal sample of approved powder) verified most definitely the value of the method you suggest."