

WASC 389
WANBD 12
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Improvements in the mfr
of nitrocellulose and
apparatus Patent 8278

1903

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& W T Thomson (Chemist)
(Displacement)

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WASC 389/05
WAI 597/05

N^o 8278



A.D. 1903

Date of Application, 9th Apr., 1903

Complete Specification Left, 9th Feb., 1904—Accepted, 7th Apr., 1904

PROVISIONAL SPECIFICATION.

“Improvements in the Manufacture of Nitro-cellulose and in Apparatus therefor.”

We, JAMES MILN THOMSON, Manager, and WILLIAM THOMAS THOMSON, Chemist, both of The Royal Gunpowder Factory, Waltham Abbey, Essex, do hereby declare the nature of this invention to be as follows:—

5 This invention relates to improvements in the process of preparation of nitro-cellulose, and to apparatus for use in connection therewith.*

The object of the 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.

10 Our invention consists in combining the acid extraction and preliminary washing operations 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.

15 We have discovered that, if water be 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
20 required for preliminary washing is very greatly reduced.

The apparatus which we have devised 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.

25 In carrying our invention into effect according to one modification, a charge of mixed nitrating acid is introduced into the vessel and the dry cellulose gradually immersed. A thin layer of a suitable liquid, preferably sulphuric acid, of a gravity not exceeding 1.700, is run carefully on the top of the acids, and the whole allowed to stand till nitration has been completed. Water is
30 then allowed to flow very gently over the surface of the sulphuric acid, and when a sufficient layer has been formed, the cock at the bottom of the apparatus is opened and the acid slowly drawn off. We find that the rate of displacement of the acids is a factor which exerts a considerable influence on the properties of the resulting nitro-cellulose and affords a means of regulating the
35 temperature of displacement. A rate of displacement which we find 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
40 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.

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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 we find 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.

This 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 we may carry out our process 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.

Dated this Ninth day of April, 1903.

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30, Cross Street, Manchester.
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COMPLETE SPECIFICATION.

“Improvements in the Manufacture of Nitro-cellulose and in Apparatus therefor.” 25

We, JAMES MILN THOMSON, Manager, and WILLIAM THOMAS THOMSON, Chemist, both of The Royal Gunpowder Factory, Waltham Abbey, Essex, do hereby declare the nature of this invention and in what manner the same is to be performed, to be particularly described and ascertained in and by the following statement:— 30

This invention relates to improvements in the process of preparation of nitro-cellulose, and to apparatus for use in connection therewith.

The object of the 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. 35

Our invention consists in combining the acid extraction and preliminary washing operations 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. 40

We have discovered that, if water be 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. 45

The apparatus which we have devised consists of a cylindrical or rectangular vessel constructed with a perforated false bottom and a cock at its lowest point 50

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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.

Referring now to the accompanying sheet of drawings which illustrate one form of the apparatus adapted for carrying out our invention:—

5 Figure 1 is a side elevation, and

Figure 2 a plan of the nitrating vessel and its accessories.

Figure 3 shows in sectional elevation one of the trough devices for enabling liquids to be added to those in the nitrating vessel without substantial disturbance.

10 In carrying our invention into effect, according to one modification, we employ as the nitrating vessel a rectangular lead lined or earthenware tank *a*, having a perforated false bottom *b* supported by ribs *c*¹ 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
15 the top of the vessel *a* are provided two troughs *f*, having depending aprons *g*. A pipe *h* has two branches *h*¹ leading to the troughs *f*. This pipe *h* is adapted to be connected by a rubber pipe either to the outlet pipe *k*¹ of the sulphuric acid tank *k*, or the water supply pipe *l*. The nitrating acids are supplied through the pipe *m*.

20 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. A thin layer, say $\frac{1}{2}$ 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
25 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*¹, and is allowed to float very gently over the surface of the sulphuric acid, and when a sufficient
30 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. We find that the rate of displacement of the acids 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
35 displacement which we find 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-
40 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.

45 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 we find it can be omitted without any deleterious effect.

50 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.

This 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-
55 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 we may carry out our process in an ordinary nitrating centri-

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fugal, 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. 5

In some cases the acids and water may be supplied by perforated or like 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 removable 10 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.

Having now particularly described and ascertained the nature of our said invention, and in what manner the same is to be performed, we declare that 15 what we claim is:—

1.—In the manufacture of nitro-cellulose, removing the acids of nitration from the nitrated cellulose directly by displacement, substantially as described.

2.—The process of manufacture of nitro-cellulose, consisting in nitrating the cellulose in a large vessel, then running on an inert liquid layer of suitable 20 specific gravity, and afterwards supplying water and slowly drawing off the acids from below, so that they are gradually displaced, and the nitrated cellulose washed, substantially as described.

3.—The process of manufacture of nitro-cellulose, consisting in nitrating the cellulose in a centrifugal machine, removing the acids of nitration directly by 25 displacement, and then running the centrifugal so as to effect preliminary drying, substantially as described.

4.—An apparatus for effecting the nitration of cellulose, comprising a vessel having a bottom outlet with a false bottom, and provided with means for supply- 30 ing displacing liquids so as not to disturb and mix with the nitrating acids, substantially as described.

5.—The nitrating apparatus hereinbefore described with reference to the accompanying drawings.

Dated this 9th day of February, 1904.

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Fig.3.

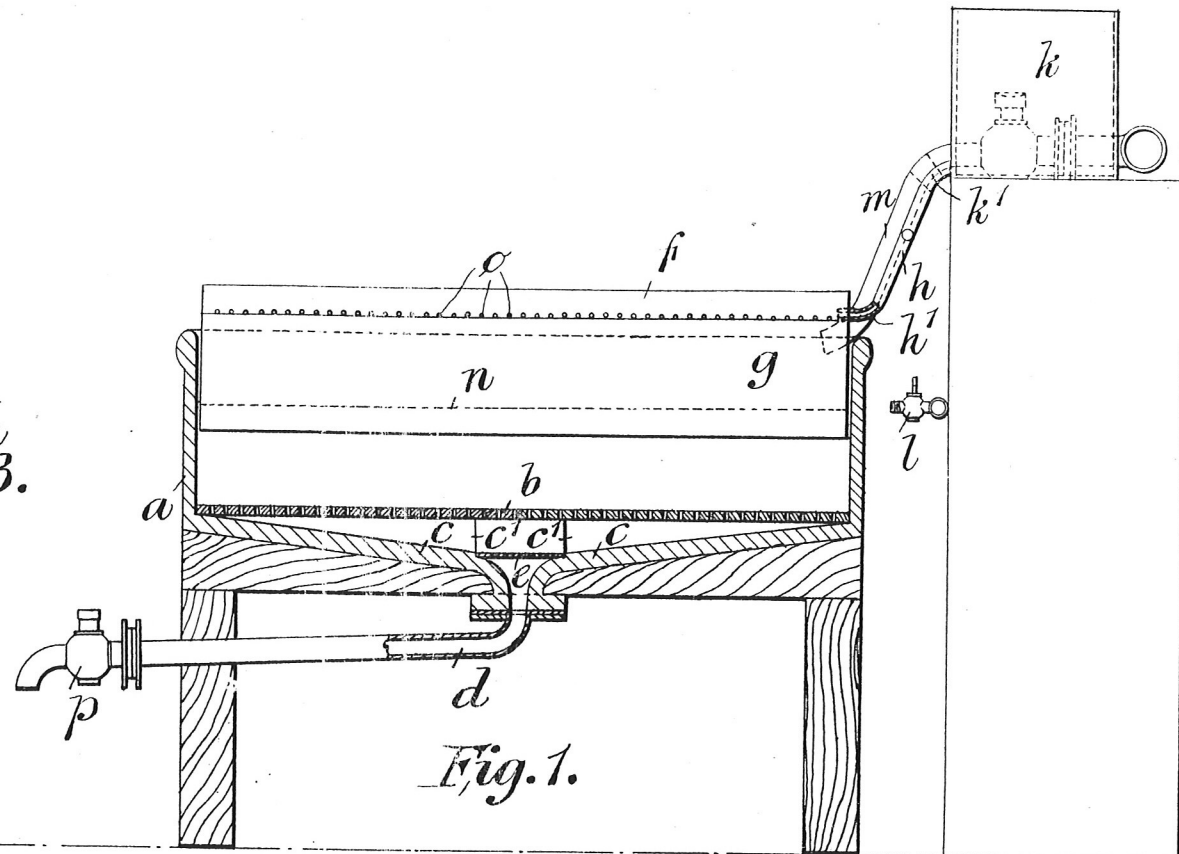
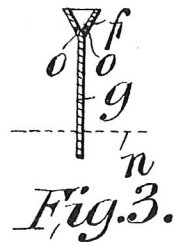
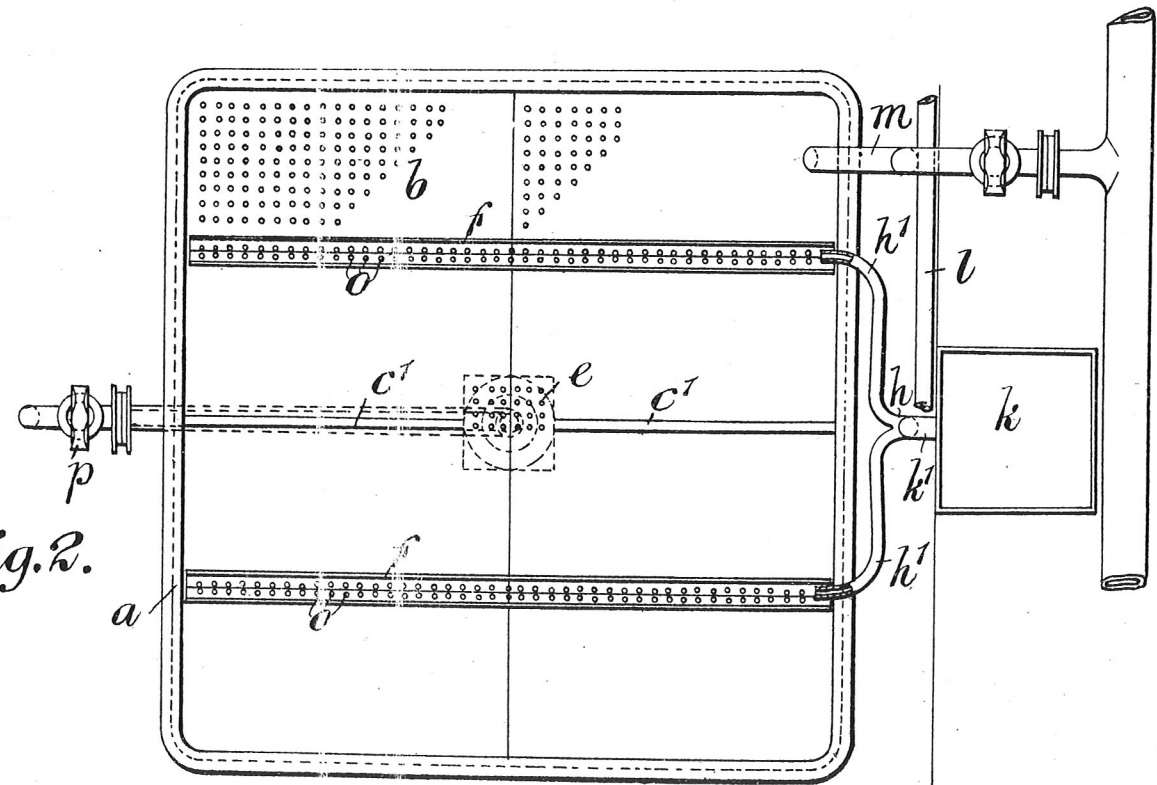


Fig.1.

Fig.2.



[This Drawing is a reproduction of the Original on a reduced scale.]