(No Model.)

G. M. MOWBRAY.

MANUFACTURE OF PYROXYLINE.

No. 350,498. Patented Oct. 12, 1886.

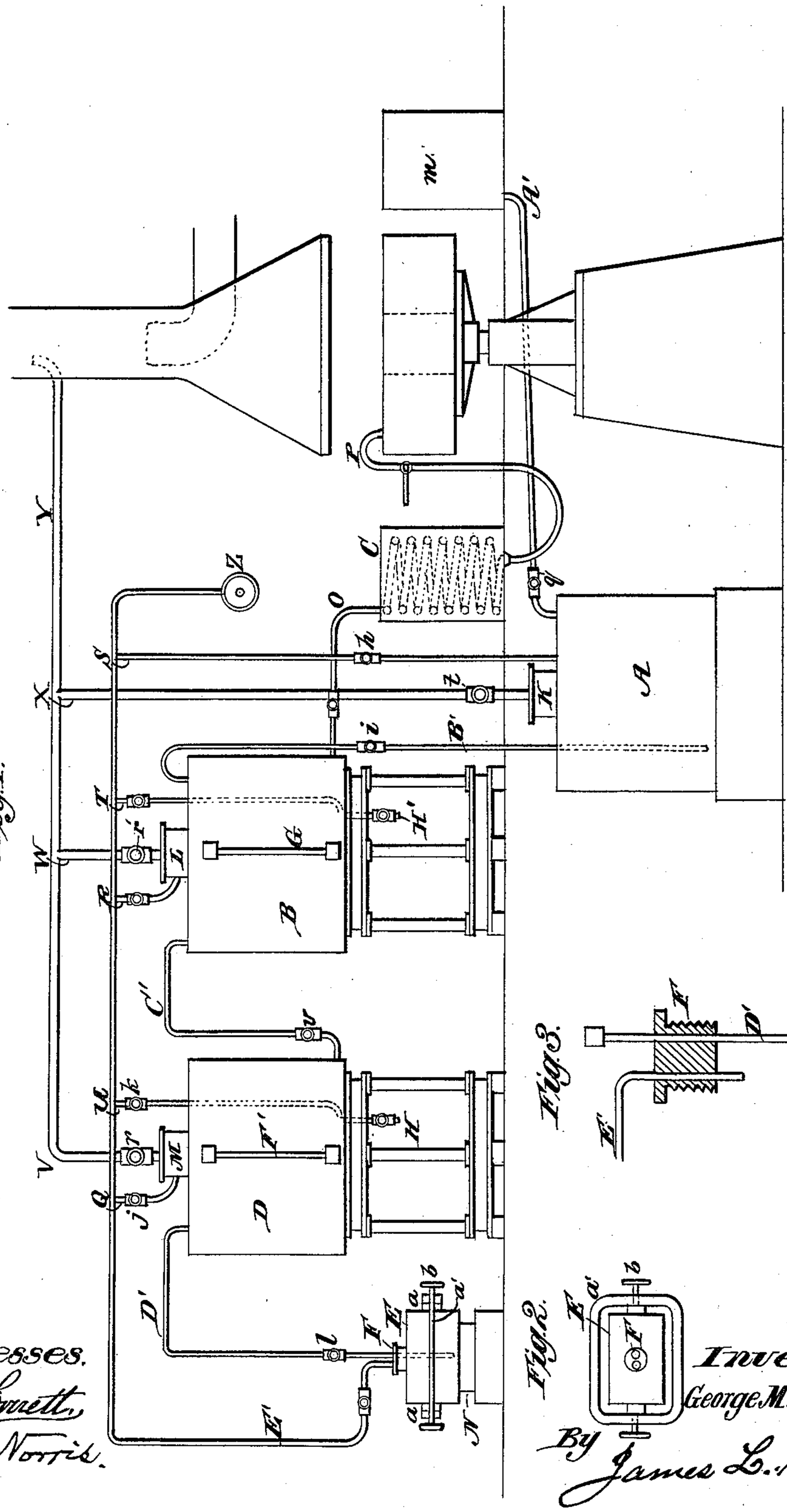

Fig. 1.

Fig. 3.

Fig. 2.

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MANUFACTURE OF PYROXYLINE.

SPECIFICATION forming part of Letters Patent No. 350,498, dated October 12, 1886.

Application filed January 27, 1886. Serial No. 189,969. (No model.)

To all whom it may concern:

Be it known that I, GEORGE M. MOWBRAY, a citizen of the United States, residing at North Adams, in the county of Berkshire and State of Massachusetts, have invented new and useful Improvements in Manufacture of Pyroxyline, of which the following is a specification.

In the specification of a separate application, Case B, filed of even date herewith, setting forth a new process of restoring spent acid in the manufacture of pyroxyline, reference is made to certain apparatus used, consisting, essentially, of, first, a turn-table and eight converting-vessels; second, a centrifugal machine; third, a spent-acid tank; fourth, a concentrated mixed-acids tank; fifth, a building-up-acids tank, and 6, a temperature-controlling coil.

Heretofore in the manufacture of pyroxyline it has been deemed necessary to use vessels made of stoneware for the purpose of converting cellulose into nitro-cellulose or pyroxyline. I have discovered that cast-iron vessels treated so as to diminish the proportion of carbon therein (by exposure to continued heating, surrounded by infusoria or iron peroxide) can be successfully used for this purpose, and that the action of acids upon the metal forms a ferric sulphate, which, being insoluble in the mixed nitric and sulphuric acid used, does not injuriously affect the product. I have also discovered that what is known as "Bessemer steel," homogeneous metal, or mild steel, which of late years has been successfully manufactured into boiler-plates, iron rails, and wire, practically resists the action of these acids, slight formation of ferric sulphate occurring, that precipitates very slowly, owing to its specific gravity closely approximating to that of the mixed acid specific gravity, 1.695 or thereabout, while gaseous nitrous-acid seems to interpose between metal and acid, preventing further action. This discovery enables me to dispense with the operation of coating wrought-iron vessels with paraffine or acid-resisting compounds as practiced heretofore.

By using iron tubing and malleable or cast iron stop-cocks of similar metal I have succeeded in constructing a plant of metal throughout, thus avoiding the great expense of stoneware; also, the weekly paraffining of wrought-iron vessels, materially improving the apparatus, and greatly simplifying and cheapening the manufacture on a large scale. The ease with which large quantities of acid can be moved from a lower level to an upper, so as to thereafter avail of gravity, provided closed vessels of large capacity—say to contain fifteen to thirty thousand pounds of acid, say, of eleven hundred to twenty-two hundred gallons of water capacity—could be obtained, rendered these discoveries the basis of the process described in Application B, before referred to, and instead of flanged tanks with covers that can be lifted, which are necessary to enable the workmen to paraffine the ordinary wrought-iron boiler-plate, a man-hole suffices; indeed, the action of the acids is so trivial that it would take many years of corrosive action to disable a vessel if of the thickness of three-eighths of an inch. Further, by surrounding the ordinary sheet-iron drums in which glycerine has been imported with an elliptic ring and head-blocks, with adjusting-screw at one end, I need no storage-vessels for either nitric or sulphuric acid, substituting these iron drums heretofore used only for transportation. On arrival I place one of them on platform-scales and take the gross weight, then lock the platform-scales so that there is no vibration, and by means of a rubber plug, F, Figs. 1, 2, and 3, which carries one tube reaching to bottom of drum and another tube that permits air to enter on the surface of the liquid, on allowing compressed air to enter the drum the contents are lifted to the required level, while the elliptical link *a*, sustaining the pressure of the head of the drums, there is no injurious strain on the weakest part of the drum. After thus emptying a drum of its contents the tare is now taken and weight of acid noted.

For the process described in my aforesaid Case B, consisting of the addition of a certain proportion of concentrated mixed acids to a certain proportion of spent acid, we really need only three tanks, whose total contents need be less than fifty thousand pounds, in lieu of thirty tanks, &c., whose total capacity is described as amounting to seventeen hundred and fifteen thousand pounds, the value of stock

of acids in the one case being less than twelve hundred dollars, as against forty thousand dollars. Such are the differences that will be found on comparing my apparatus with that described in Patent No. 299,388, dated May 27, 1884, where it will be seen the following vessels are described, but which are wholly unnecessary in my process, Case B, and are therefore excluded in this application.

The vessels described in before-mentioned patent, that my process enables me to dispense with, are two sulphuric-acid tanks, capacity forty thousand pounds each; two nitric-acid tanks, capacity twelve thousand pounds each; one weighing-tank; eight storage-tanks, capacity one hundred thousand pounds each; large weighing-tank; car and railway for moving same; five temperature-regulating pots; one auxiliary weighing-pot, making a total of twenty-one vessels dispensed with by my less-complicated process. Every unnecessary vessel is a disadvantage for these reasons, that an extended surface tends to weaken the acid by absorption of moisture, and the degradation of the acids is proportioned to the time and extent of metallic surface exposed, which, if of wrought-iron, is imperfectly protected by what is termed "acid-proof coatings." The ability to turn out an equal quantity of product with these vastly-disproportioned means, is attained by the discovery referred to in aforesaid Case B. I repeat here—viz., that the so-called "flock," instead of being as described "flock or flocculent matter detached from the fiber," is simply ferric sulphate $Fe_2(SO_4)_3$, which, being of nearly same specific gravity as the normal standard acid mixture used for conversion, is slow to settle. Being insoluble in the concentrated acids it is innocuous during the process of nitration, but as it is completely soluble when diluted with water, disappears as soon as the pyroxyline is plunged into the washing-water.

I will now describe the vessels I use, and refer to the drawings accompanying this specification, in which—

Figure 1 is a side elevation of an apparatus embodying my invention; Figs. 2 and 3, detail views of drum, elliptic ring, and plug.

The subjoined letters of reference, with annexed explanations and description of modus operandi, will enable any one skilled in the art to avail of my invention.

A, spent-acid tank; B, building up-acids tank; C, temperature-controlling coil; D, concentrated-acids tank; E, drum, in situ for emptying; N, platform-scales; A′, waste-pipe from centrifugal machine to spent-acid tank A; B′, delivery-pipe from spent-acid tank A to building-up acid tank B; C′, delivery-pipe from concentrated-acid tank D to building-acid tank B; D′, delivery-pipe from drum E to concentrated-acids tank D; E′, compressed-air inlet-pipe for admitting compressed air on surface of contents of a drum; F′, concentrated-acids-tank glass gage; G, building-up-acids tank glass gage; H H′, waste-plugs to tanks D and B, respectively; K L M, man-holes to tanks A, B, and D; O, connecting-pipe from building-up-acid tank B to coil C; P, delivery-pipe and controlling stopway for conveying built-up-acid of suitable temperature to replenish nitrating vessels. The compressed-air pipes are Q, R, and S, and should be inserted in or near to the side of the man-hole of tanks A, B, and D, while air-pipes T and U, of about three-quarters of an inch in diameter with stop-cocks to control same, should be inserted from the domes, respectively, of tanks B and D, terminating about six inches from the bottom of tanks to serve as a means of agitating and thoroughly mixing the acids sent into these tanks.

Z is the pressure gage for air-pressure.

V, W, and X are air and nitrous-gas outlet-pipes leading through main Y to ventilation.

Eight nitrating-vessels, fixed on a suitable turn-table, are each charged with from one hundred and fifty to one hundred and eighty kilos of normal standard mixed acid, so as to occupy about three fourths of its capacity. Into each of these charged vessels, at intervals of three and one-half minutes, the operator immerses thirteen hundred and twenty-five grams of tissue-paper strips or bleached-cotton fiber. By the time the eighth vessel is charged—say in about twenty-four and one-half minutes—the tissue-paper or cotton fiber will have been converted into pyroxyline, and must now be transferred to a centrifugal machine, *m*, which, in from one to three minutes, separates five-sixths of its adherent acid, which passes from said machine through pipe A′ into spent-acid tank A. One whole day's accumulation of spent acid is more accurately restored for the succeeding day's use than is possible by returning the spent acid of one preceding nitration to a nitrating-vessel and adding one-fifth of that added spent-acid's volume of concentrated mixed acid, just as it is easier to perform one operation of restoration at the termination of a day's work, instead of about two hundred operations during the day on a smaller scale. For this reason I prefer using a building-up-acid tank, B, instead of making the converting-vessel serve a purpose similar to that which the tank B serves—viz., receiving spent acid, also concentrated mixed acid—the latter in a proportion that, with the spent acid left over in the converting-vessel, makes these acids from three differing sources suitable for nitration. I therefore prefer that one day's accumulation of spent acid from the centrifugal machine *m* shall be made into what I have termed "building-up acid," to serve for the next day's work in restoring the spent acid left in the nitrating-vessels after each immersion to a condition suitable for converting cellulose into nitro-cellulose.

Full and complete details of this process of nitration and restoration having been given in a separate application, Case B, filed of even

date herewith, it only remains to describe the mode of operating the apparatus, referring to the drawings accompanying this specification. We commence by supposing the spent-acid tank A contains the accumulation of a day's workings received through pipe A′ from the centrifugal machine *m*. First, mark on gage G correctly indicating level of acid in building-up-acid tank B; then close stop-cock O, also stop-cock *q* on pipe A′, between the centrifugal machine and spent-acid tank A; open the compressed-air stop-cock *h* and the spent-acid service stop-cock *i*. The compressed air on the surface of the spent acid causes it to rise in the pipe B′, which transfers it into the building-up-acid tank B. Now note how many inches of rise of level the gage G indicates after transfer, say, sixty inches. (I defer details of charging concentrated mixed acid-tank D for the present.) Close stop-cocks *q′* or pipe A′ and *l* on pipe D′ *h*, and open compressed-air cock *j*. Mark on gage F′ exact level. Now open stop-cock *v* on pipe C′. As soon as the gage G indicates twelve inches increased level beyond the sixty inches before noted, close stop-cock *v* on pipe C′, shut off compressed-air supply by stop-cock T, and open compressed-air-supply cock *k* for agitation; also exhaust-exit *r*. It is better to defer the agitation until the mixture has cooled down to about 70° Fahrenheit. To prepare the concentrated mixed acids for tank D, of which we have just described as having transferred twelve inches to tank B, we proceed as follows: A drum of nitric acid of suitable strength is rolled onto a platform-scale and its gross weight marked down. Lock the scale-beam to prevent vibration, and harness onto the drum the elliptical iron ring *a′*, adjusting the blocks *a a* so as to bear against the drum's heads, tightening the same with the set-screw *b*. Remove the iron screw-plug that closes the drum, and insert the rubber plug F, with its iron pipe and coupling, which must now be connected to the pipe D′, that leads into the concentrated mixed acid tank D. Connect, also, the curved small pipe E′ with compressed-air service, open stop-cock *l*, and take level of glass gage accurately, so as to ascertain how many inches of level are equivalent to a given weight of each acid as a check in future work. When contents of drum have been transferred from the drum E to the tank D, remove the elliptic ring, head-blocks, rubber plug, and its attachments, drain drum, insert tightly the iron plug, ascertain tare of drum, deduct same from gross weight, and set down net contents of drum and compare weight with level of glass gage F′, for purpose of estimating contents when taking stock, &c.

Having transferred, as above described, the contents of four or five drums of nitric acid of proper strength, as described in my aforesaid application, Case B, and noted accurately their net contents, next calculate the proportion of sulphuric acid of suitable strength, as described in the aforesaid Case B, and having rolled out ready for the scale as many drums of sulphuric acid as are required to make up with the nitric acid hereinbefore referred to, what I have termed "concentrated mixed acid," proceed to weigh and transfer precisely as before described for the transfer of the nitric acid. In adding the sulphuric acid there should be sufficient time allowed between each drum of sulphuric acid added for the mixed acids to cool, otherwise if the acids get overheated the tanks may suffer. By this method of transfer one weighing suffices to check from the chemical factory, and for the process, for in all further uses of these acids volumetric measurement by the glass gages is practicable; and as all transfers take place in closed vessels, except to replenish the converting-vessels, there is avoidance of waste, and fumes do not harrass the workmen.

I do not limit my invention to any one of the three varieties of pyroxyline known as, first, gun-cotton; second, collodion, or soluble pyroxyline, and third, xyloidine, as it is applicable to each of these, or a mixture of them, by increased or diminished strength of acids; nor do I confine myself to the exact proportions of acids set down in this specification.

Having thus described my invention, what I claim is—

1. In the manufacture of pyroxyline, the use of steeled cast-iron pots for holding the mixed acids in which the cellulose is immersed during the process of conversion.

2. The use of homogeneous metal, otherwise called "Bessemer process steel," plates for tanks to serve as containing-vessels for the mixed acids used in the process of converting cellulose into nitro cellulose.

3. The use of homogeneous metal, otherwise called "Bessemer process steel," plates for receiving-tanks and storage-tanks of the mixed concentrated and spent acids used in the process of converting cellulose into nitro-cellulose.

4. The combination of a spent-acid tank, a building-up-acid tank, and a concentrating-acid tank with a temperature-regulating coil.

5. The elliptic ring with head-blocks and adjusting-screw, in combination with wrought-iron drum, substantially as described.

6. The perforated plug, in combination with the wrought-iron drum and the compressed-air inlet and liquid outlet, substantially as described.

In testimony whereof I affix my signature in presence of two witnesses.

GEO. M. MOWBRAY.

Witnesses:
HARRY S. MOWBRAY,
MERRITT T. WHITE.