(No Model.) 2 Sheets—Sheet 1.

J. R. FRANCE.

MANUFACTURE OF PYROXYLINE COMPOUNDS.

No. 393,751. Patented Dec. 4, 1888.

Fig. 1.

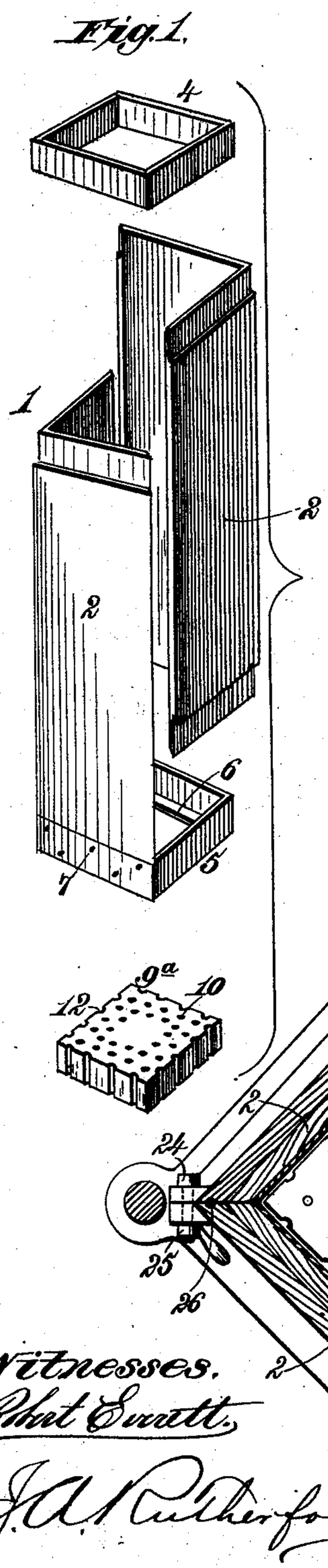


Fig. 2.

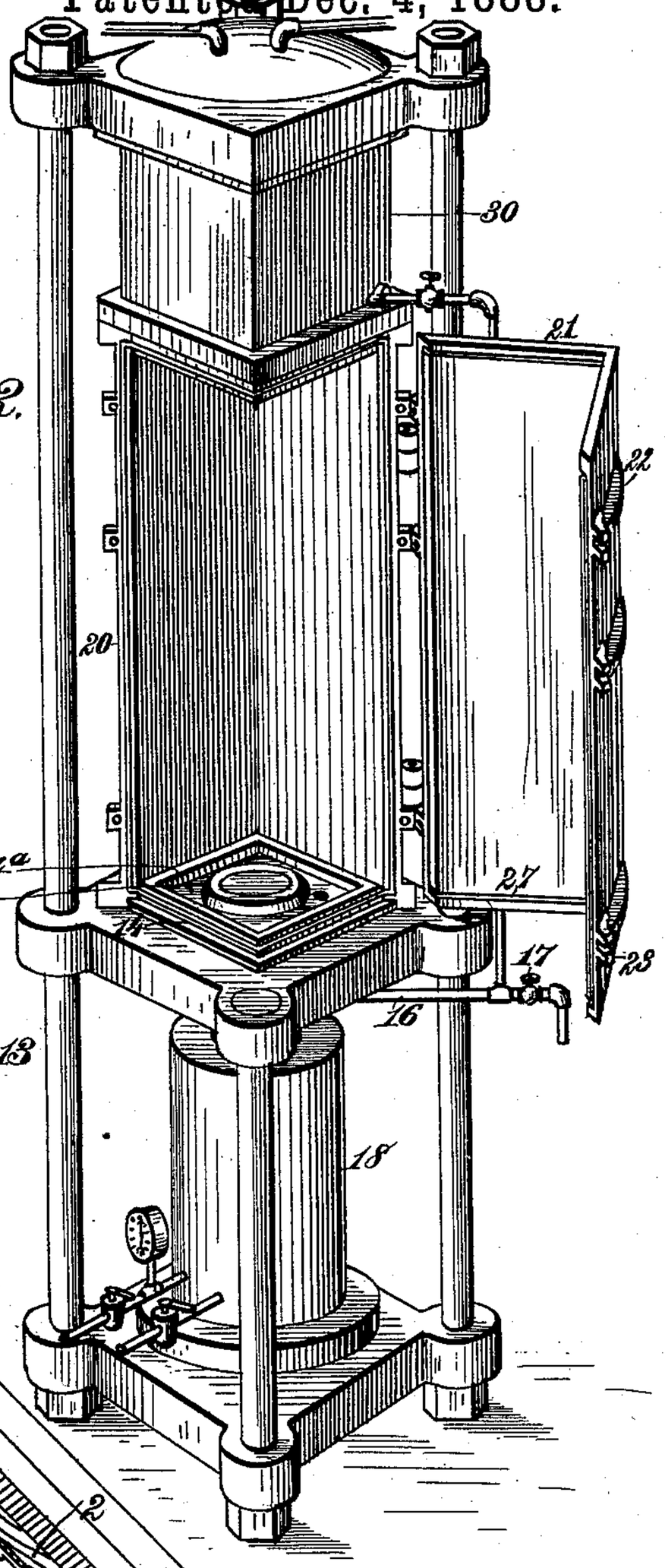
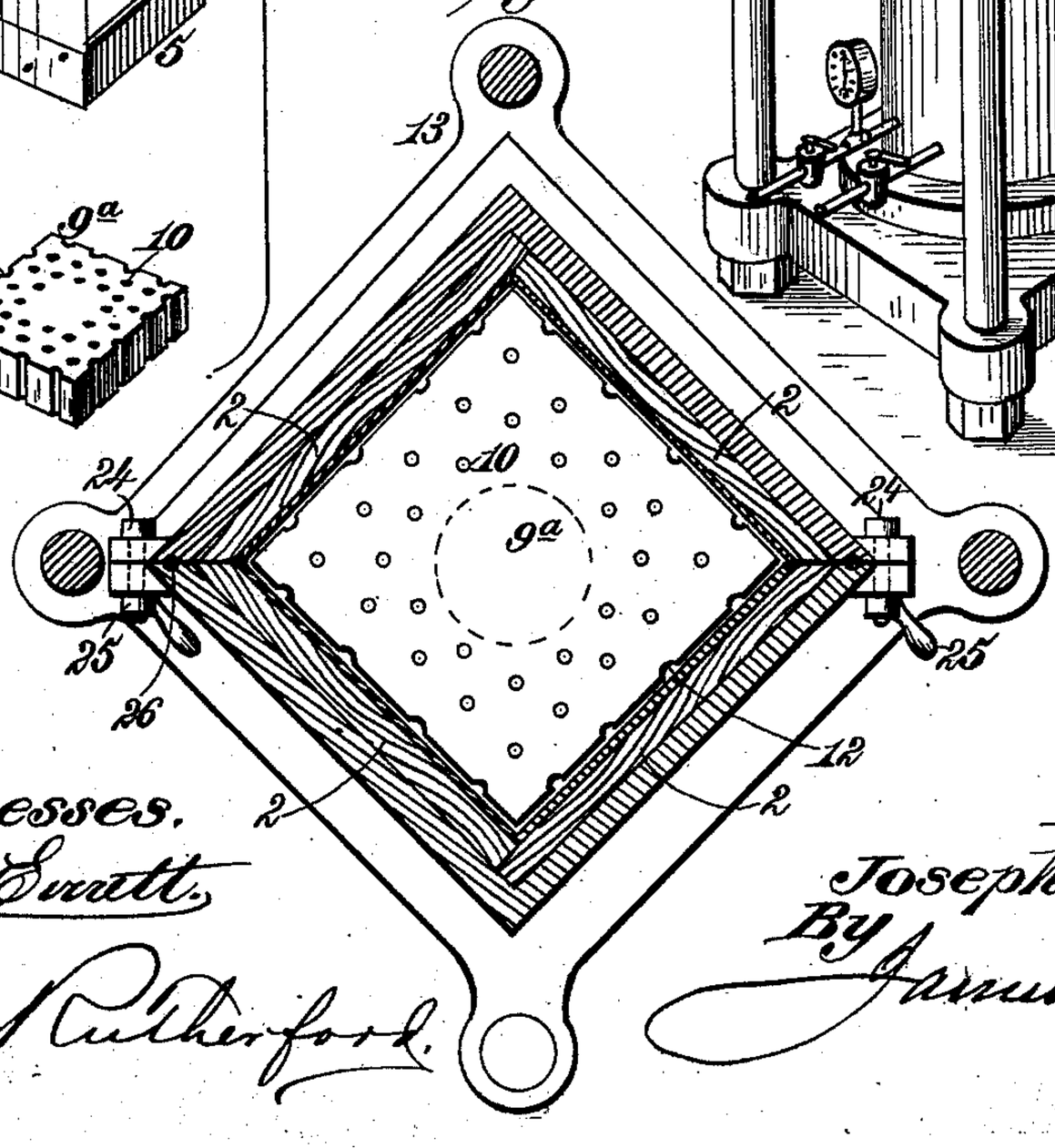


Fig. 3.



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(No Model.) 2 Sheets—Sheet 2.

J. R. FRANCE.

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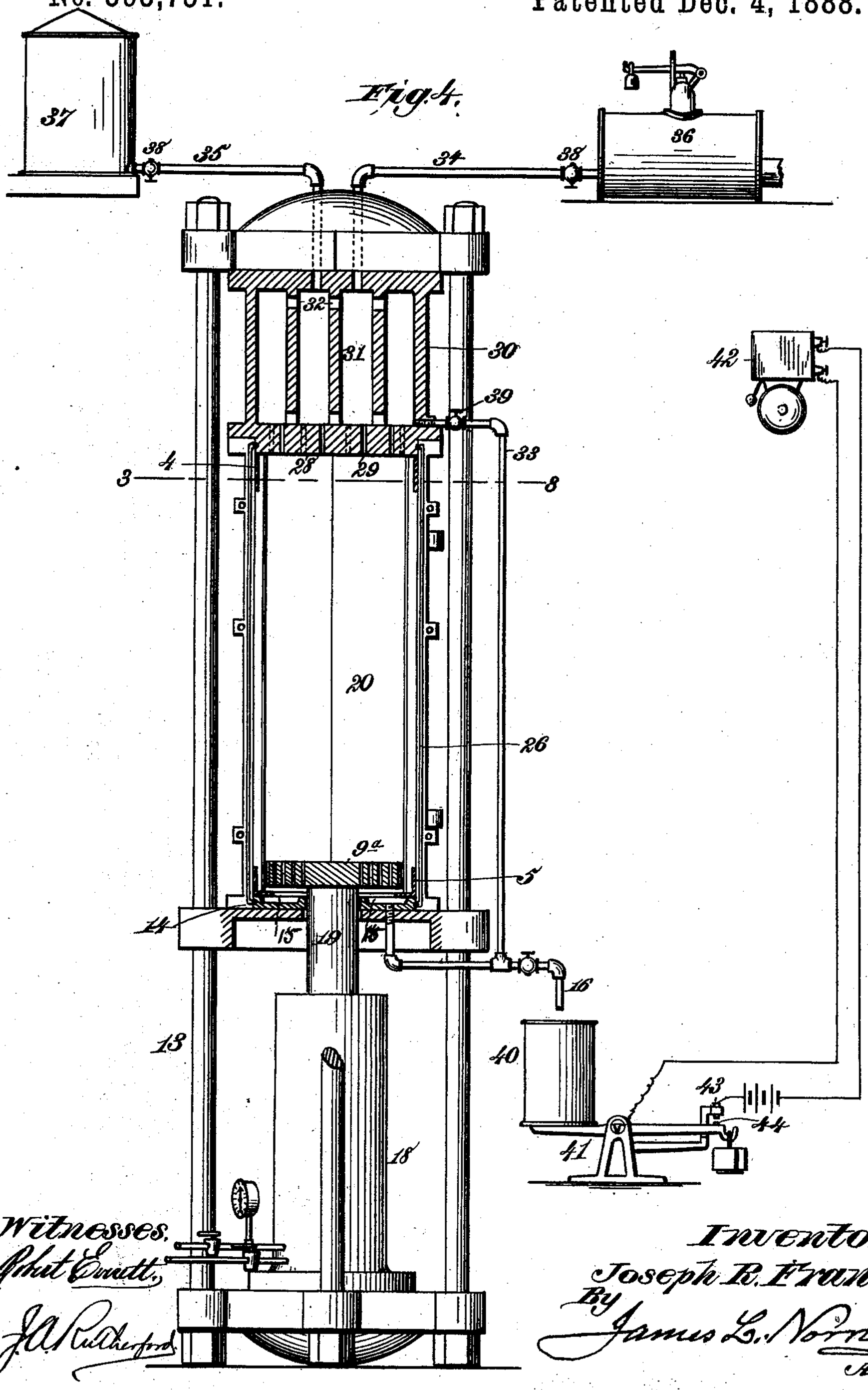
Fig. 4.



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UNITED STATES PATENT OFFICE.

JOSEPH R. FRANCE, OF NEW YORK, N. Y.

MANUFACTURE OF PYROXYLINE COMPOUNDS.

SPECIFICATION forming part of Letters Patent No. 393,751, dated December 4, 1888.

Application filed January 5, 1888. Serial No. 259,849. (No specimens.)

To all whom it may concern:

Be it known that I, JOSEPH R. FRANCE, a citizen of the United States, residing at New York, in the county of New York and State of New York, have invented new and useful Improvements in Manufacture of Pyroxyline Compounds, of which the following is a specification.

My invention relates to the manufacture of pyroxyline compounds; and the purpose thereof is to provide a novel method of treatment whereby the pulp may be properly dehydrated without danger of explosion, with economy to the manufacturer, and in quantities adapted to the daily requirements of the factory. In the manipulation of nitro-cellulóse, preparatory to its treatment by solvents and following the nitration of the pulp and its subsequent washing, it is necessary to free it from the considerable body of water absorbed or adherent thereto before it can be converted into celluloid.

It is well known that trinitro-cellulose or gun-cotton is explosive at a higher temperature, and is therefore less dangerous, than dinitro-cellulose, which is the soluble fiber forming the basis of the various compounds of celluloid. In dehydrating the latter various methods have been proposed. In one instance the pulp has been placed in a closed vessel containing a suitable absorbent packed in bags, the particles of pulp being free to move in the vessel and come into close proximity to the absorbent or the bags containing the same. The vessel is then rotated until the maximum quantity of moisture has been abstracted from the pulp. Inasmuch, however, as it has been found necessary to employ very heavy hydraulic pressure to expel part of the water only from the pulp, it will be seen that the method referred to above cannot be relied upon to remove the requisite percentage of moisture unless a very considerable period of time and absorbents having a marked affinity for water are employed. In another instance the wet pulp has been molded into cakes or layers and built up in a pile of such cakes with alternate pads of bibulous paper. The pile thus formed is subjected to enormous hydraulic pressure, and the water expelled from the pulp is absorbed and retained by the bibulous pads lying between the cakes. This method will remove the moisture, provided sufficient pressure is exerted, and if the absorbents possess the necessary capillary attraction they will retain it and prevent its reabsorption. It is a well-known fact, however, that pressure upon nitro-cellulose beyond a certain point is extremely liable to produce an explosion, and especially so in cases where the pressure is exerted for the purpose of expelling the water wherein the material at a certain stage of the process approximates a desiccated condition. It may be accepted as an ascertained fact that all the known methods of dehydrating cellulose which involve a sufficient degree of pressure to expel the proper amount of water are unavoidably dangerous by reason of the liability to explosion, which will always exist in a greater or less degree.

In other processes of manipulation cold air has been employed from which the moisture has been removed, the air being driven into and through the pulp or through a drying-room containing it. Heated air has also been used in a similar manner, the pulp being treated while in a wet state, in combination with a liquid solvent, until it is reduced to a semi-liquid state. It has also been proposed to grind the pyroxyline-pulp, mixing in at the same time camphor and such pigments or coloring materials as may be desired. After grinding, the pulp, still impregnated with water, is formed into cakes, and each cake, laid between bibulous pads, is placed upon a metallic sheet, a pile of such cakes being formed upon the press-bed. To effect the desiccation by this method, the pressure exerted is about seven thousand five hundred pounds to the square inch, the pile being exposed to such pressure for a time varying from forty minutes to one hour. The danger from explosion in this method is the same as that already mentioned, save that it is enhanced by the increased degree of pressure exerted.

Another method proposed is to place the nitrated and washed pulp in a suitable receptacle having escape-openings in its bottom and introduce alcohol upon the pulp, which is then forced down into the mass, expelling the aqueous particles and taking the place of the latter. This method is not essentially dissimilar to the processes long employed in pharmacies and laboratories, depending upon

the different specific gravities of water and alcohol, whereby a body of the latter superimposed upon a mass of matter containing water by absorption will tend to percolate through such matter and drive the aqueous particles before it. The main objection to this process is that the pressure upon the superimposed alcohol causes the latter to mingle rapidly with the water, and in all cases the fluid remaining in the pulp will be found to consist of hydrated alcohol of various strength. It is impossible, therefore, to effect an aqueous displacement in this manner, the best results merely dividing the residuum of water at each repetition of the operation and increasing the proportion of alcohol remaining in the pulp.

It is well ascertained that nitro-cellulose or pyroxyline in a loose state and while in a damp or moist condition, and even when containing only thirty per cent. of water, cannot be ignited or exploded by any ordinary methods. Even when it contains the same percentage of alcohol it is equally safe, since if it be ignited it will burn with a steady alcoholic flame, consuming the nitro-cellulose, but without danger of explosion. In this loose damp state it is practically safe for handling in large quantities.

My invention contemplates the utilization of these facts; and it consists in the novel process hereinafter fully described, and definitely pointed out in the claims.

In carrying my invention into effect I employ apparatus, one suitable form of which is shown in the accompanying drawings, in which—

Figure 1 is a perspective view of the containing-vessel employed, the view showing the parts composing said vessel separated from each other. Fig. 2 is a perspective view of a press adapted to the practice of my invention. Fig. 3 is a horizontal section of Fig. 4, taken in the line 3 3. Fig. 4 is a central vertical section of the entire apparatus.

In the said drawings the reference-numeral 1 designates what I term the "containing" or "pulp-receiving" vessel. It is composed of four walls, 2, inclosing a rectangular space, two of said walls being separably connected to the other two walls. At the upper end these walls are supported in proper relative position by means of a metallic re-enforcing-band, 4, which embraces the upper end of the container. At the lower end the four vertical walls are supported by a metallic band, 5, having a flange, 6, turned at a right angle to the plane of the wall above and having its inner edge projecting within the inner surface of the wall of said container. The lower band, 5, is permanently fastened to the vertical walls of one-half of the container by means of rivets or screws 7. (Seen in Fig. 1.) The container is made of wood lined with zinc to reduce its weight; but other material may be used, if preferred. The walls are gained at top and bottom to bring the re-enforcing bands flush with the outer surfaces.

Within the container or receptacle 1 is placed a false bottom, 9^a, composed of any suitable material—such as wood or metal—and provided with numerous escape-openings, 10, formed in the false bottom itself, as well as with channels 12, formed between the edges of the false bottom 9^a and the inner surface of the vertical wall-pieces 2. This false bottom is vertically movable within the container 1 and rests normally upon the flange 6, formed by turning in the lower edges of the re-enforcing plates or bands 5.

Upon any suitable form of frame, 13, I mount a press-table having a plate, 14, upon which the base of the container may rest. This plate has a flange, 14^a, inclosing a recess, 15, with which a discharge-pipe, 16, communicates, said pipe having a cock, 17. Beneath this skeleton table is arranged the cylinder 18 of a hydraulic press, said cylinder having a plunger, 19, operated in the well-known manner. This plunger is arranged to rise and fall through the skeleton table or base 14 and against the false bottom 9^a, which is constructed to move up and down in the container 1.

Upon the press-table 14 is mounted the press-box 20, which consists of a rectangular structure, 1, two of the walls being rigidly mounted upon the press-table, surrounding the flange 14^a thereof, while the other two walls, 21, are hinged to the rigid walls. Upon the exterior of these walls are formed or mounted ribs 22, having projecting ends 23, which meet when the press-box is closed and receive bolts 24 and lever-nuts 25, whereby the joints may be tightly closed. To make them air-tight, or as nearly so as practicable, rubber strips 26 are placed in the meeting edges and engage with grooves 27 in the corresponding edges of the hinged section 21.

The press-box is terminated above by a press-head or strong diaphragm, 28, having openings 29, which lead into a close chamber, 30, above. This chamber may, if desired, be divided by partitions 31, having top and bottom openings 32. A discharge-pipe, 33, enters said chamber just above the floor thereof, and communicating with the top thereof are two pipes, 34 and 35, the former leading to an air-compressor, 36, of any suitable construction, and the latter to an alcohol-reservoir, 37. Cocks 38 are placed in said pipes, and a similar shut-off, 39, is placed in the pipe 33, which may conveniently be carried down to and into the discharge-pipe 16.

With apparatus of this construction the mode of procedure is as follows: I take pyroxyline as it comes from the washing-tanks and place it in a centrifugal extractor, which is operated until the pulp contains its own weight of water. I then divide it into lots of about two pounds each, each lot containing one pound of water. I now spread upon the

perforated false bottom 9ª a sheet of muslin, upon which is spread the first lot of wet pulp. A second strip of fabric is then spread upon the top of the pulp, and a second lot of pulp is laid upon the second piece of muslin. This arrangement is repeated until the container is filled, each superimposed layer being compacted as it is laid in place. The container is now closed and placed in the press-box, and the door of the latter is closed tight. Being supported upon the press-table 14, the ram 19 of the hydraulic press rests against the false bottom of the container, and, the press being operated, the ram rises, carrying the false bottom with it and pressing the pulp between the latter and the diaphragm 28. A comparatively light pressure will answer the purpose at this stage, four hundred pounds to the square inch giving excellent results, and with a slowly-moving ram being entirely free from danger. Any suitable form of safety valve or gage may be attached to the pump of the press to open at the pressure indicated. The pulp is allowed to remain under pressure for a short time or until the expelled water has flowed out through the outlets required. When the operation is conducted as set forth, the pyroxyline will, while in the container and under pressure, part with thirty per cent. of the absorbed or adherent water, leaving seventy per cent. of the original bulk still remaining in the pulp. The valve 39 of the upper outlet, 33, is now closed and air is driven through the pipe 34 from the air pump or compressor. Entering the compacted pulp at the top it penetrates down through the same, driving off the expressed water, which has not already escaped, into the chamber 15 and opening the spaces or cells in the pulp, which are filled with unexpressed aqueous particles, thereby leaving the material in excellent condition for the ensuing steps. The currents of air, also driven through the closely-compacted mass under pressure, carry off an additional quantity of the contained water, amounting to twenty per cent. of the entire amount, thereby leaving in the mass about one-half of the original bulk. After the water ceases to flow the air is still for a further brief period driven through the pyroxyline, after which the air-valve is closed and the tap in pipe 35 is opened, admitting alcohol to the chamber 30, whence it drips through the diaphragm 28 and enters the pulp. Knowing the approximate quantity of water still remaining in the latter, the reservoir 37, with which the pipe 35 connects, is supplied with a quantity of alcohol greater by a few pounds than the amount of retained water, and the recess 15 is emptied of the water expressed by the ram and by the air. A vessel, 40, is placed upon a suitable scale, 41, and brought under the lower outlet, 16, the weight of the scale being set so that the beam shall tip when the water known to be in the pyroxyline shall have been received in the vessel. An electric or other call, 42, may be arranged to operate by the movement of the scale-beam to call the operator's attention thereto, electric contacts 43 and 44 being placed on the scale-beam and scale-arm, respectively. The alcohol entering the container at the top penetrates down through the pyroxyline, driving before it the retained fifty per cent. of water, which passes off through the chamber 15 into the vessel upon the scale. The alcohol sinks into and passes through the mass of pyroxyline by gravity, and by reason of its own different specific gravity it floats or rests upon the contained water, driving it down before it as it sinks through the compressed mass. About one hour, more or less, will be required for the completion of this stage of the alcohol treatment. When the scale-beam rises, showing that the quantity of water estimated to remain in the pyroxyline after the treatment by compressed air has been received in the vessel upon the scale, this vessel is removed and at the same time the valve in the alcohol-pipe 35 is closed and the compressed-air valve opened. The vessel removed from the scale is replaced by another to receive any hydrated alcohol which may come away for future redistillation, and air is again blown through the pyroxyline until it retains from forty to fifty per cent. only of alcohol. The valve is now closed, the ram is run down, the press is opened, and the contents of the container are removed to zinc-lined boxes having tight covers. The pyroxyline in the container will be found in thin layers easily separable by means of the interposed muslin. The quantity of camphor necessary to complete the conversion of the pyroxyline is now placed between the layers, which are piled one above another, and coloring-matter and pigments are added. The boxes containing the material are then tightly covered and set one side for the operation of the rolls, which completes the conversion of the pyroxyline into celluloid and other similar compounds.

By this process I secure several advantages, one of which is the rapid dehydration of the pyroxyline, enabling the manufacturer to treat a large quantity in a single day and with comparatively small expense. Again, the solvents are distributed through the pyroxyline so evenly and every particle thereof is so thoroughly saturated with the alcohol as to greatly promote and facilitate the subsequent operations attending the treatment by solvents. The dehydration of the pulp also is accomplished without danger, the pressure to which it is subjected being very light and the pulp being at all times in a moist condition. Finally, the process is speedy, simple, safe, and economical, the alcohol used being recovered by distillation, save as to that portion retained in the pulp and afterward combined with camphor.

While I have mentioned alcohol only, I may use any active or non-active solvent in place thereof—such as amyl or chloride of amyl—as well as other well-known solvents.

The container 1 may be formed of any material; but for lightness and economy wood lined with zinc may be employed. I may, however, use vulcanized rubber or metal and make the container of any size. In practice I have usually constructed it with a depth of thirty inches and a diameter of ten inches each way, giving an interior space of three thousand cubic inches. I use these proportions because I have found that dinitro-cellulose, when closely compacted, occupies a space equal to ninety-six cubic inches for each pound avoirdupois of the pulp, and that when compressed at the maximum pressure of four hundred pounds to the square inch it occupies less than half the space named, or about forty cubic inches. Thus a container of the dimensions specified would be adapted to a charge of about thirty pounds of pyroxyline in its hydrated condition—that is to say, with an additional weight of adherent or absorbed water about equal in weight to the weight of the pyroxyline.

What I claim is—

1. The process set forth for the dehydration of dinitro-cellulose or pyroxyline, the same consisting in first subjecting the washed pulp to pressure, and then while still under pressure blowing air through the same, substantially as described.

2. The process herein set forth for the dehydration of dinitro-cellulose and similar compounds, the same consisting in first exposing the pulp to light pressure to remove the excess of water, then forcing air through the compacted mass, and then admitting alcohol to the upper end of the containing-vessel and allowing it to percolate through the pulp by gravity, substantially as described.

3. The process set forth for the dehydration of dinitro-cellulose, consisting in first exposing the pulp to a light pressure, then forcing air through the compacted mass, then while still under pressure percolating alcohol through the pulp by gravity, and finally blowing air through the same to expel the surplus of alcohol, substantially as described.

4. The process set forth for the dehydration of dinitro-cellulose, the same consisting in arranging the pulp in layers with interposed strips of fabric, subjecting it to a comparatively light pressure to expel the excess of water, then forcing air downward through the compacted mass, then while still under pressure percolating alcohol down through the pulp by gravity, and finally blowing air through the same to remove the excess of alcohol, substantially as described.

5. The process set forth for dehydrating dinitro-cellulose, the same consisting in arranging the pulp, after nitration and washing, in successive layers with interposed strips of fabric, subjecting it to light pressure, then forcing air down through the compacted pulp, then percolating alcohol through the same by gravity, then blowing compressed air down through it to remove the excess of alcohol, separating the layers, and finally placing camphor with coloring-matter and pigments between the compressed layers, substantially as described.

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

JOSEPH R. FRANCE.

Witnesses:
EDW. S. FULLER,
GEORGE C. ELLIOTT.