June 15, 1948. M. A. CHAVANNES 2,443,443

APPARATUS FOR PRODUCING FILMS

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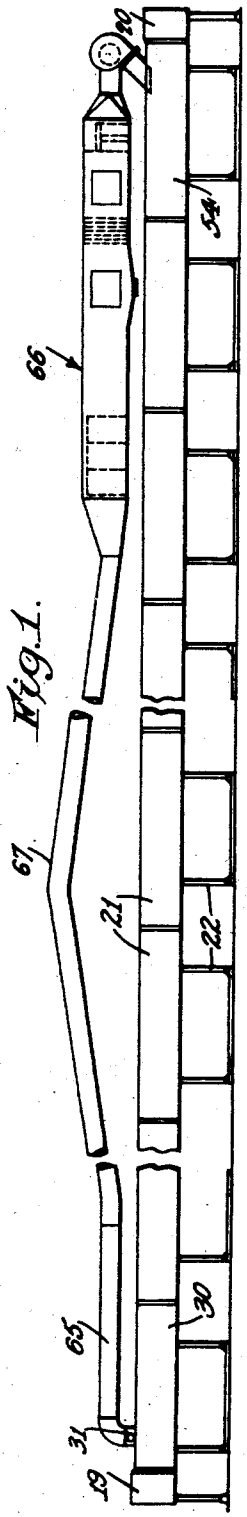


Fig. 1.

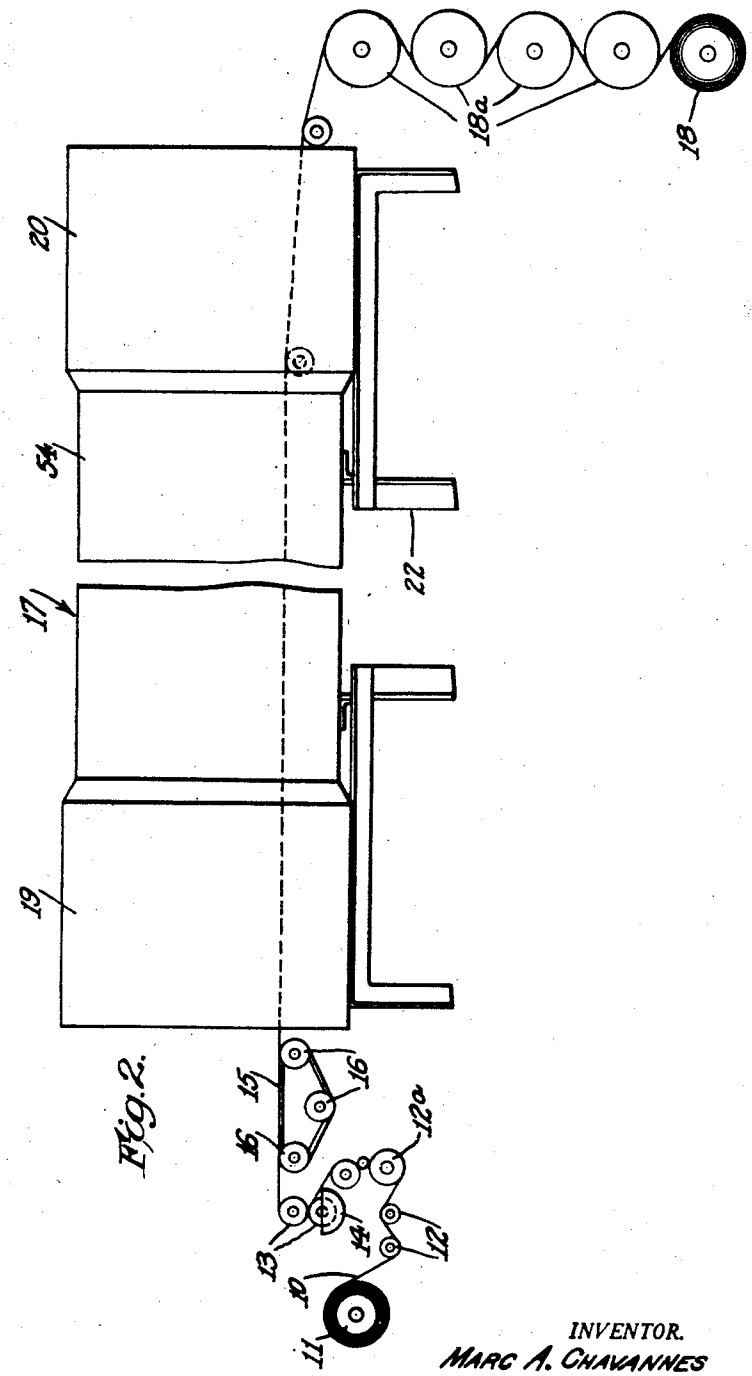


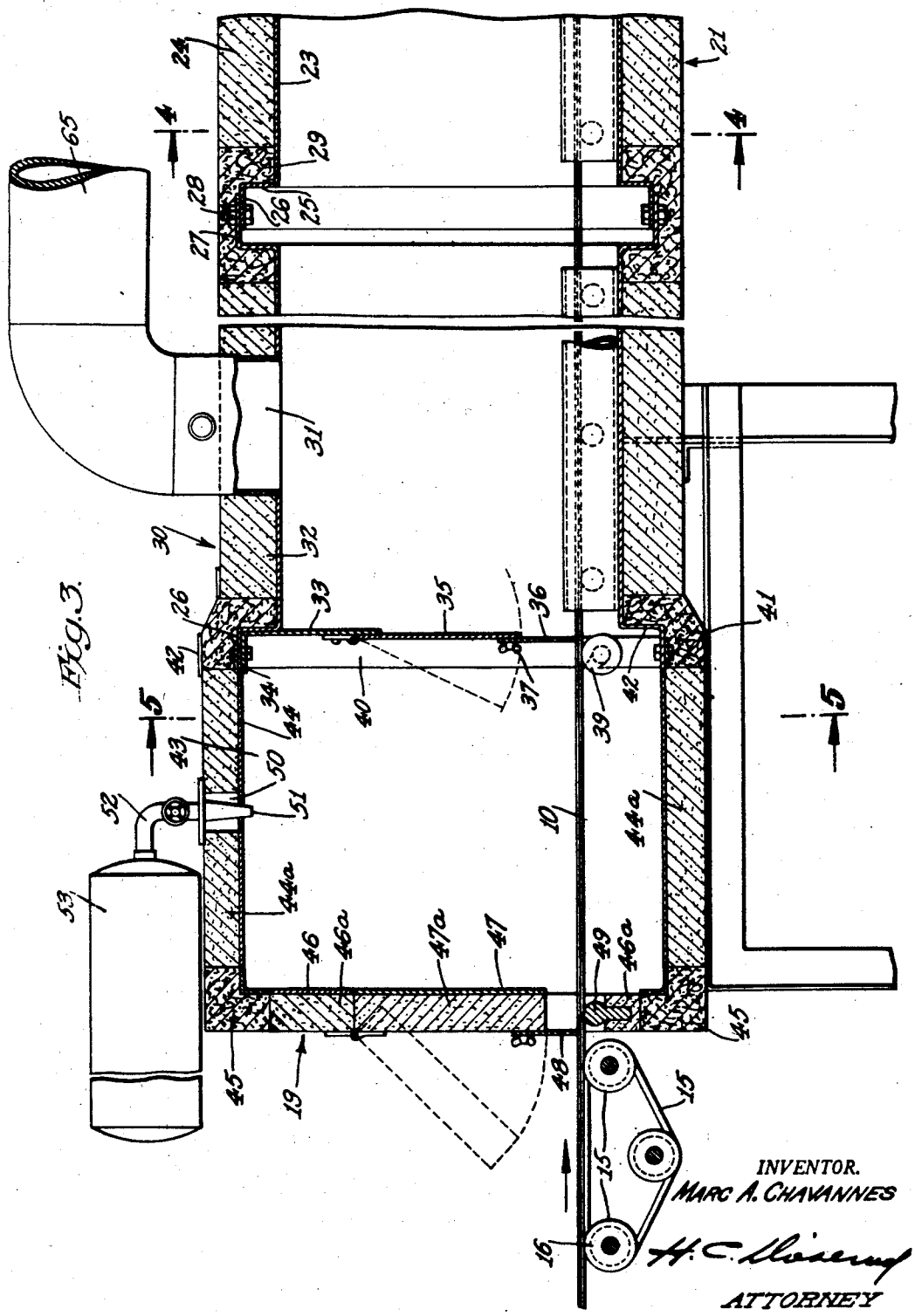
Fig. 2.

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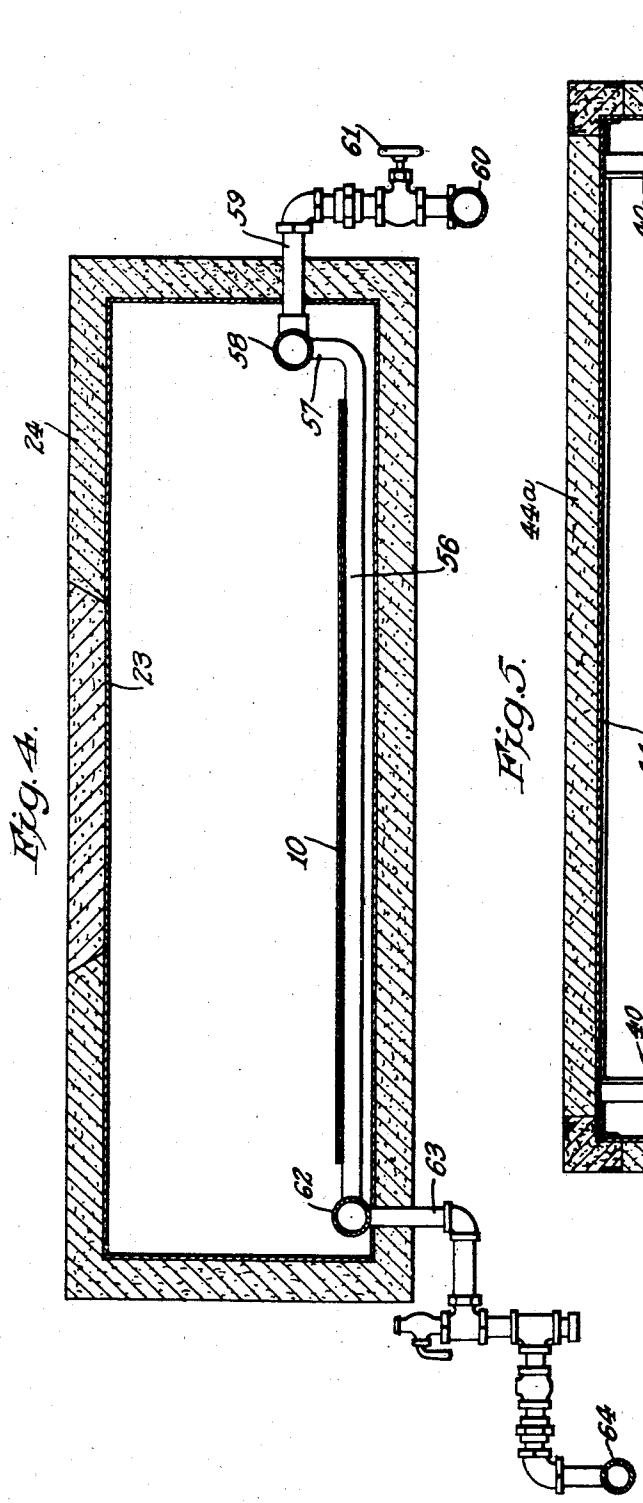


Fig. 4.

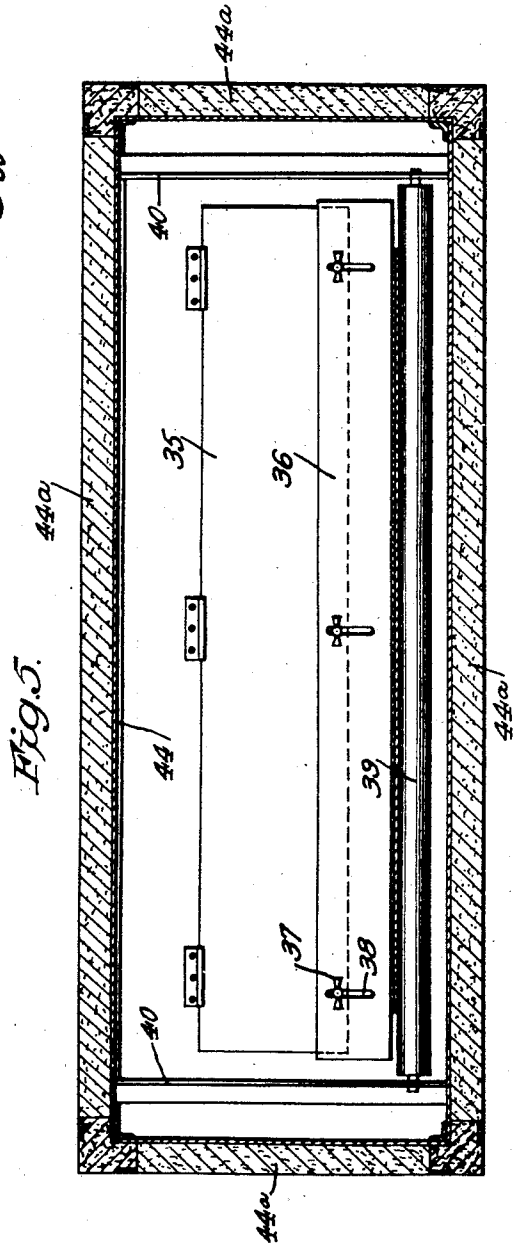


Fig. 5.

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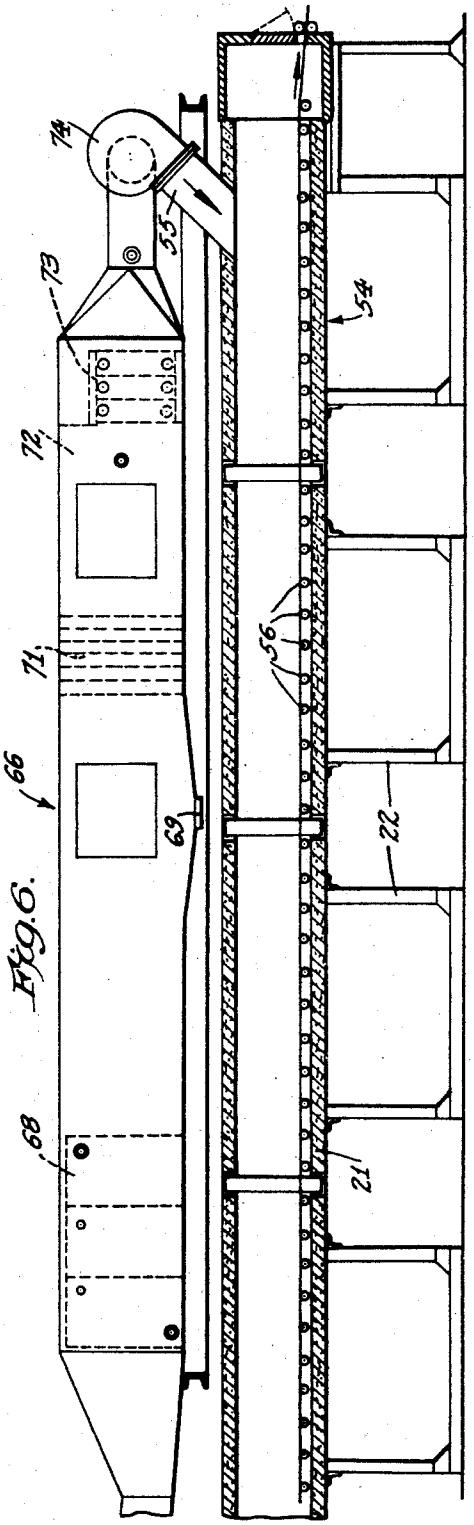


Fig. 6.

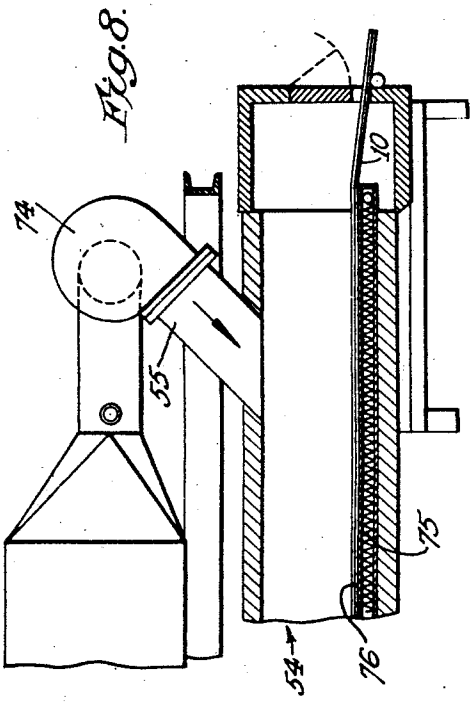


Fig. 8.

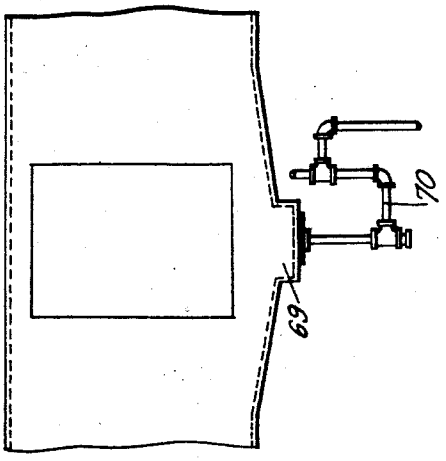


Fig. 7.

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Patented June 15, 1948 2,443,443

UNITED STATES PATENT OFFICE

2,443,443

APPARATUS FOR PRODUCING FILMS

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Application September 22, 1943, Serial No. 503,365

1 Claim. (Cl. 34—77)

1

This invention relates to apparatus for producing films of resinous materials, or similar substances, from suitable solutions of the same and to the improved product resulting from the use of such method and apparatus.

More particularly the invention relates to the apparatus employed in the drying of a layer of film-forming solution applied to a carrier on which the improved film is to be formed.

An object of the invention has been to provide a simple, rapid and inexpensive means for removing the solvent from the film applied to the carrier and for recovering such solvent.

Another object has been to provide apparatus for drying a film which accomplishes this result in such a way as to prevent the formation of a skin over the surface of the film. In achieving this aim, the present invention permits the rapid escape of the solvent vapors and any gases which may be occluded or otherwise embodied in the original film, without forming irregularities in the film, such as pockets or pin-holes, and the like. This is accomplished by carrying out the drying operation in such a way that there is constantly in contact with the surface of the film an atmosphere which is rich in the solvent vapors. Accordingly, a much more uniform and more nearly impervious film is produced than by methods heretofore known. This end is achieved, moreover, rapidly and without any additional steps in the treatment of the film. Furthermore, the new drying method enables the substantially complete drying of the film before it is stripped from the surface of the carrier on which it is formed.

A further object of the invention has been to provide a drying and solvent recovery system which is entirely safe in its operation and free from dangers of explosion and the like.

Other objects, features and advantages of the invention will appear from a detailed description of an illustrative form of the same which will now be given in conjunction with the accompanying drawings, in which:

Fig. 1 is a schematic view in side elevation of the improved dryer of the invention, portions being broken away to condense the drawing;

Fig. 2 is a schematic view in side elevation, on a larger scale, showing the ends of the dryer in relation to other parts of the equipment, the intermediate portion of the dryer being broken out;

Fig. 3 is a longitudinal, vertical section through a portion of the dryer at the wet end;

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Fig. 4 is a transverse, vertical section along the line 4—4 of Fig. 3;

Fig. 5 is a transverse, vertical section along the line 5—5 of Fig. 3;

Fig. 6 is a view partly in longitudinal, vertical section and partly in side elevation, showing a portion of the dryer and associated devices adjacent the dry end of the equipment;

Fig. 7 is an enlarged detail view, in side elevation, of a portion of the solvent recovery system, and

Fig. 8 is an enlarged detail view similar to the left-hand portion of Fig. 6, but showing a modified construction.

In accordance with the invention, a coating of the desired material is first applied to a carrier web in any suitable manner, preferably in the manner disclosed in my pending application Ser. No. 458,898, now abandoned, filed on September 18, 1942. As disclosed in said prior application, the carrier is preferably formed of strong, relatively heavy paper having applied to one, or both, of its surfaces a firmly adhering coating of a base material, such as polyvinyl alcohol, casein or the like, capable of providing a smooth outer surface on the carrier to which the film-forming coating will but lightly adhere. It will be understood, by reference to my prior application above-mentioned, that the character of the base coating preferably applied to the carrier web will vary with the character of the film to be produced. For instance, a plasticized casein coating has been found well suited for the purpose of a carrier web on which a film of a co-polymer of vinyl acetate and vinyl chloride, is to be formed. Such a base coating may also be used for the production of films of other types of synthetic or natural resins. A polyvinyl alcohol coating may also be used in the formation of most resinous films but such coating should preferably be heavily filled. If the invention is utilized in the production of films from other plastic substances, the base coating on the surface of the web should be such as not to react with the solvent used and not to be affected by the temperature and other conditions employed in the process. Moreover, it should be flexible, so as not to crack in normal handling or in its passage through the coater and dryer, and such that the final film may be readily stripped from it. A variety of thermo-setting resins provide suitable base coatings for a large number of different types of films formed upon the evaporation of various solvents. The web itself, in lieu of being formed of paper, may be formed of other relatively inexpensive, flexible

materials capable of withstanding the strains and conditions to which it is subjected in the conduct of the process. It may, for example, be formed of a cellulose derivative or of asbestos paper, or metal foil, or of fibre glass, or the like. Such materials are, in the main, more durable than paper when subjected to the high temperature conditions met with in the dryer.

Referring now to the drawings, and more particularly to Figs. 1 and 2, the carrier web 10 having the firmly or permanently adhering, smooth coating on one, or preferably both, surfaces may be supplied from a roll 11 and passed around suitable guide rollers 12 and 12a to a coater. Roller 12a is preferably heated by water or steam or the like so as to raise the temperature of the carrier web to substantially that of the film-forming solution. The coater may comprise a pair of cooperating rollers 13, the lower roller being revolved in a bath of the desired film-forming substance in solution in a vat 14. This arrangement, it will be understood, is such that as the web 10 is continuously advanced through the bight of the rollers 13, a thin film or coating of the film-forming substance is applied in a layer of desired thickness to the outer surface of the web. Other suitable means, such as spreader knives, nozzles or hoppers, may be employed, in lieu of the coating rollers, for applying a continuous film or coating of the desired substance in its solvent to the surface of the web. After receiving the film-forming layer or coating, the web is preferably passed over a suction apron 15, carried by a series of rollers 16 and driven by one of the latter at the desired speed, to advance the web without engaging its upper, coated surface. Between the upper roller 13 and the suction apron 15, however, there may, if desired, be provided suitable means for smoothing out the coating or film on the surface of the web, as set forth in my above-mentioned application, or means, such as disclosed in the Chavannes et al. application Ser. No. 479,310, now abandoned, filed March 16, 1943, for vibrating the carrier web to eliminate bubbles from the film.

The suction apron 15 serves to deliver the web to the wet end of the dryer. This is designated generally at 17 in Fig. 2. After the coating or film has been thoroughly dried, it is discharged, on the carrier web, at the dry end of the equipment and is preferably delivered to a take-up reel 18 for storage or subsequent disposition. In advance of the winding reel 18, the web is preferably passed around a series of cooling rolls 18a, which may be water-cooled, to reduce the temperature of the film below the point at which it is tacky or has a tendency to block. The dryer, which may be of any appropriate length, depending upon the character of the film being formed, the amount and character of solvent which must be removed, and the speed at which it is desired to operate the equipment, is preferably formed in a plurality of sections, including a sealing section 19 at the inlet or wet end, a similar sealing section 20 at the discharge or dry end, and a large number of intermediate sections 21. Each of the intermediate sections is preferably only about 7 or 8 feet long and each is supported by a leg structure 22, which may appropriately be of angle-iron construction. The form and arrangement of the parts is such that a gas-and-vapor-tight joint is provided between the successive sections of the dryer while due allowance is made for the expansion and contraction of the several sections due to temperature changes. Thus, while

the complete dryer, which may be between 100 and 300 feet long, or even longer, would be subject to substantial elongation, as its temperature is raised, in starting up, from room temperature to the temperature to be maintained within the dryer, the expansion of each section is relatively small and is readily taken up by the joints provided between the sections.

Referring now to Fig. 3, there is shown an illustrative form of the several sections of the dryer and the connection between them. Each of the sections 21 preferably comprises an inner, hollow shell or casing 23 formed of sheet iron or other suitable material, which may, if necessary, be coated on its inner surface to protect it against the action of the vapors and gases within the dryer. The casing or shell, as shown in Fig. 4, is preferably rectangular in cross section and may suitably be formed of four sheets forming the top, bottom and side walls, these being welded together along their meeting edges. Suitable heat-insulating material is applied to the outer surface of each wall of the casing. This insulating material may appropriately be in the form of slabs 24 of rock wool or asbestos or other material having good heat-insulating qualities. The slabs 24 terminate a slight distance from the ends of the metallic casing, for a purpose to be presently explained. Beyond the ends of the slabs, the metallic casing is provided with an outwardly projecting flange 25 terminating at its outer end in an axial extension. Such extension 26 at the left end of each section is of slightly smaller dimension than the corresponding extension 27 at the right end of the section so that these extensions may be suitably telescoped, as indicated in Fig. 3. They are fastened together by bolts 28 in a manner to permit relative movement as the casings expand due to the application of heat at the interior of the dryer. Between the ends of the slabs 24 and around the joint formed by the telescoping extensions 26 and 27, there is provided any suitable form of packing material 29, such as loose rock wool, or asbestos, or the like. This arrangement is such that substantially gas-and-vapor-tight joints are maintained between the several sections and, at the same time, these joints are heat-insulated.

The section or unit 30 (Fig. 1) adjacent the left end of the dryer, next to the sealing section 19, is of substantially the same construction as the rest of the intermediate sections but is provided at its top with an outlet 31, formed of sheet iron or the like, welded to the top member of the casing. The insulating slab 32 is suitably formed to fit around this outlet. At its left end the unit 30 is substantially closed by a rectangular closure member 33 having an axially extending flange 34 fitting within the flange 26 of the casing and secured thereto by bolts, rivets or welding. Intermediate its upper and lower edges the closure 33 is provided with an elongated opening normally closed by a hinged gate or door 35 carrying at its bottom an extension plate 36 whose vertical position may be adjusted in relation to the gate by means of wing nuts 37 and elongated slots 38 formed in the extension plate. This arrangement is such that in the normal operation of the equipment only a slight clearance is provided between the lower edge of the plate 36 and the upper surface of the web 10 but, whenever required, as in threading the equipment in starting up, the gate 35 may be swung open to provide ample room above the web supporting means. Such support-

ing means may include a roller 39 mounted at an appropriate level in upright angle members 40 secured to the closure plate 33. Around the closure 33 and the flange 26 at the left end of the unit 30, there is provided suitable heat-insulating packing 41 which may be held in place by a sheet metal frame structure 42.

Beyond the closure 33 and extending toward the left therefrom in Fig. 3 is a sealing chamber 43 which may be formed in any suitable manner, similar to the sections 21 and 30, but is preferably slightly larger in transverse cross section. It is preferably formed by an inner sheet metal casing 44 insulated along its top, bottom and side walls by slabs 44a of rock wool or the like. The outer, left end of the chamber 43 is closed by a surrounding angle frame filled with heat-insulating packing 45 and by a wall 46 covered by slabs 46a likewise formed of heat-insulating material. A large central opening in the wall 46 is closed by a swingable door or gate 47 covered by an insulating slab 47a. A vertically adjustable plate 48 is provided along the lower edge of the door 47 to provide just the desired clearance above the carrier web 10 as it passes over a supporting bar 49 which is preferably embedded in the insulation 46a, and extends across the lower portion of the end wall 46. The bar 49 is preferably formed of hardened steel or the like and is provided with a smooth, rounded, upper edge, which permits the web to pass freely over it without undue friction. If desired, the bar may be replaced by a roller similar to the roller 39 or the latter may be replaced by a bar similar to the bar 49. Such a bar permits a somewhat smaller clearance to be provided between the plate 48 and the upper surface of the web 10. The top wall of the chamber 43 is provided with an opening 50 through which projects a nozzle 51 connected by a valve-controlled pipe 52 with a tank 53 containing compressed carbon dioxide or other suitable inert gas, which may be introduced into the chamber 43 and maintained therein at a slightly super-atmospheric pressure, for the purpose to be hereinafter set forth.

The unit 20, at the discharge or dry end of the equipment, may be substantially the same construction as the unit 19 at the wet end. So also, the section 54 immediately preceding the discharge unit 20 may be substantially the same as the unit 30 adjacent the inlet unit 19. However, as shown in Fig. 6, the vapor and gas inlet 55 leading into the unit 20 is preferably inclined at an acute angle to the longitudinal axis of the unit so as to assist in directing the gases and vapors in the direction of such axis. Moreover, since the film carried by the web is dry as it passes into and out of the unit 20, the problem of sealing this unit is simplified. The web and film may, for example, be passed through the nip of cooperating rollers to effect the seal.

Within each of the units or sections 21, 30 and 54 of the dryer, there is provided suitable heating means, preferably in the form of a series of transversely extending pipes 56. These, as best shown in Fig. 4, preferably have an up-turned end 57 connected with a longitudinally extending header 58. Steam or other heating medium is introduced into the header 58 through a pipe 59 which receives the heating medium at an appropriate temperature and pressure from a main line 60. A control valve 61 serves to regulate the supply of the heating medium. At its opposite end each pipe 56 is connected with a

longitudinally extending header 62 into which the heating medium is discharged and from which it is delivered through a pipe 63 to a main return pipe 64. The details of the piping form no part of the present invention and, therefore, need not be described further. However, it should be pointed out that the headers 58 and 62 and the series of connecting pipes 56 are separate for each of the units or sections of the dryer, thus permitting the introduction of steam or other heating medium under different temperature and pressure conditions into the several sections. In the operation of the equipment, the temperature and heat in-put should increase from the intake or wet end of the dryer toward the outlet or dry end. The temperature of the heating medium supplied to the successive sections need not vary throughout the length of the dryer; a plurality of sections may be connected together as a group, if desired, to receive the heating medium, either in series or in parallel, under substantially the same conditions; but the general increase in temperature indicated above should be maintained. The variation in the temperature of the different portions of the dryer may be brought about either through the supply of the heating medium, or different heating media, from different sources at the required temperature and pressure or through the appropriate regulation of the supply of the heating medium, by the use of the valves 61, from a single source of supply.

The web 10 is preferably passed directly over the pipes 56 and in contact therewith for most efficient heat transfer, the pipes 56 being provided at sufficiently close intervals for the purpose. However, if desired, a sheet metal or similar surface may be provided over the pipes and in contact therewith and may serve as the web supporting and heat transferring medium.

Means are provided for continuously circulating the gases and vapors within the dryer by withdrawing them from a point adjacent the wet end and returning them at a point adjacent the dry end. For this purpose a pipe 65 (Figs. 1 and 2) is connected with the outlet 31 of the unit 30 and serves to deliver the gases and vapors to the intake of a solvent recovery unit 66. An upward bend 67 in the pipe 65 allows for expansion and contraction due to heating and cooling. The recovery unit 66 (Fig. 6) may be of any suitable construction. It preferably includes a condenser section 68 in which the vapors and gases are passed in heat exchange relation to suitable cooling coils through which water or other cooling medium is circulated. A portion of the vapors, which is condensed as a result of this cooling action, is collected in a sump 69 (Figs. 6 and 7) and withdrawn through suitable piping 70 for delivery to storage or any convenient point for reuse. Prior to reuse the recovered solvent may be purified or treated in any required manner. An advantage of the invention, however, is that the solvent is recovered in a concentrated and relatively pure condition so that little treatment is required before reuse. Moreover, the recovery during a continuous period is practically complete.

The gases and uncondensed vapors continue their passage through the recovery unit and are preferably passed through a series of eliminators 71 which provide a tortuous path for the mixture and serve to remove any entrained liquid particles of the solvent. The entrained solvent so eliminated is returned to the sump 69. After passing through the eliminators the gases and

vapors are passed through a chamber 72 into which a small amount of carbon dioxide or other inert gas is introduced for the purpose of replacing any of the gas which may have escaped. The mixture of gas and solvent vapors is then passed through a heater section 73 of any suitable construction, in which the vapors remaining mixed with the gas are super-heated. These super-heated vapors and the gas are then drawn by a blower 74 and forced, under a slight super-atmospheric pressure, through the inlet 55 into the dryer.

In a typical operation of the system, the carrier web having its smooth, protective base coating may be coated with a film or layer of a co-polymer of vinyl acetate and vinyl chloride which may be carried in solution in methyl ethyl ketone. It is then passed into the drying equipment through inlet chamber 43. It will be understood that suitable provisions (not shown) may be made, in the form of endless conveyor chains at the two sides of the dryer, for threading the carrier web through the dryer in starting up. Such threading means may be of any known construction. Within the sealing chamber 43 an inert atmosphere of CO_2 or other inert gas supplied from the tank 53 is maintained under a slight pressure of, say, a fraction of an inch up to several inches of water above atmospheric. This pressure should also be slightly below that maintained within the adjacent portion of the drying chamber which itself is slightly above atmospheric. Accordingly, any leakage which may take place will be from within the sealing chamber to the outside and this leakage will be primarily of the inert gas with but a slight amount of the solvent, which may evaporate in the passage of the web through the sealing chamber 43. The operation of the main section of the dryer, between the closure 33 (Fig. 3) and a corresponding closure at the discharge end of the dryer, is preferably such that the mixture of gas and vapor which is introduced through the inlet 55 is approximately 60% CO_2 and 40% solvent vapors, by weight. This mixture, in passing through the dryer toward the discharge end, will receive substantial quantities of the solvent vapors, removed not only because of the super-heating of the mixture but also because of the heat supplied through the pipes 56 over which the carrier web is passed. The temperature adjacent the inlet end of the dryer should be slightly under the boiling point of the solvent, i. e., about 175° F. in the typical operation being described. This temperature should be gradually increased toward the discharge end of the equipment, where it may be well above the boiling point of the solvent, say around 250° F. These temperatures should be maintained both above and below the carrier. The capacity of the condenser and its operation should be such that the amount of solvent driven off from the film and carried along with the circulating mixture of gases and vapors will be condensed as the mixture is passed through the recovery system. Thus, the mixture which is returned to the dryer through the duct 55 will always contain substantially the same percentage of the vapors.

If in lieu of methyl ethyl ketone a solvent such as cyclohexanone is employed, the mixture of inert gas and vapor which is introduced through the inlet 55 may contain a smaller percentage of the vapors than in the case of the methyl ethyl ketone. In general, the higher boiling solvents,

such as cyclohexanone and hexone, appear to have a greater solvent action on the co-polymer of vinyl acetate and vinyl chloride than does the methyl ethyl ketone and, therefore, a smaller percentage of the vapors of the higher boiling solvents will have the desired effect of insuring the production of a uniform film which is free from pin holes and other imperfections. Thus, in the case of cyclo hexanone, the vapors at the inlet 55 may constitute only 25% of the mixture. This is sufficient to prevent the formation of a skin upon the surface of the film during the drying operation and the subsequent puncturing of such a skin by the escape of gases or solvent vapors in the course of the drying action. The vapors in escaping are not required to dissolve a skin before the drying action is speeded up. Accordingly, in the use of the improved method and equipment there is produced a new or greatly improved product, having substantial uniformity of physical as well as chemical properties throughout and having a much greater imperviousness to gases than prior films of the same thickness and greater freedom from impurities, such as dust particles. At the same time this result is achieved in a manner which brings about a rapid and efficient drying of the film and a substantially complete recovery of the solvent vapors. Moreover, since the drying is carried out without the use of air, the solvent vapors are not diluted and mixed with water vapor, so that the necessity of later removing water from the condensed solvent is eliminated.

When the higher boiling solvents are employed the temperatures at different points in the dryer may be correspondingly increased. Thus, when cyclohexanone is used, the temperature adjacent the inlet end of the dryer may be around 275° F. and adjacent the discharge end it may be 350° F. When hexone is used, the two temperatures may be, respectively, 225° F. and 260° F.

In the major portion of the dryer the temperature should be considerably below the melting point of the resinous or plastic material of which the film is to be formed. However, the final temperature, adjacent the discharge end of the dryer, may be substantially at or even slightly above the melting point of the plastic. The length of time during which the film is subjected to a melting temperature, or substantially so, should not be great enough to permit any substantial fusing of the material but it may result in a smoothing of the surface and a further sealing of the pores of the film as the final portion of the solvent is driven off. A special arrangement which may be used for the purpose is shown in Fig. 8. It comprises an electrical heating member 75, in the form of a plurality of resistance coils, mounted in a frame having a sheet metal plate 76 at its top, over which the carrier web 10 is passed. This heating unit, which may be substituted for the heating pipes in the section 54 of the dryer or in this section and the preceding section 21, may be very accurately controlled and arranged to be maintained at a temperature around the melting point of the film. In the case of a film of a co-polymer of vinyl acetate and vinyl chloride having a melting point of, say, 400° F., the temperature in the final section may be as high as, say, 398° F., which is sufficient to effect a heat sealing action. For other films, of higher or lower melting points, the temperature may be carried correspondingly higher or lower. Thus, in forming a film of vinylidene chloride the temperature may be carried as high as 275° F., while in forming films of methyl methacrylate or poly-

styrene, it should not be carried above about 250° F. The web and its supported film will be subjected to the high temperature for only a few seconds.

In lieu of the pipes or resistance coils in the final section or sections of the dryer, there may be provided a group of electric heating bulbs or units adapted to produce radiant heat. These may be provided either above or below the web or at both points and may be very accurately controlled. If provided above the web, they may be mounted in or above the top of the dryer housing and a glass plate capable of passing the infra red rays or other forms of radiant heat may be provided beneath the bulbs. A similar arrangement may be employed beneath the web, if desired. Such an arrangement may, if preferred, be substituted for the entire system of pipes below the web.

In the operation of the dryer the major portion of the solvent will be driven off from the film in the first part of the equipment. As much as 80% of the solvent may be removed in the first half of the dryer and about 15% may be removed in the main part of the remainder of the dryer. The final few per cent, which are most difficult to remove, may be driven off by the high temperature treatment mentioned above. Even this treatment, however, will not remove the final traces of solvent and the film as it leaves the dryer will still have between, say, 1 and 3% of the original solvent.

It is important, as previously indicated, to cool the film before it is wound on the reel 18. This is accomplished by passing it over the series of cooling rollers 18*a* which serve to reduce the temperature of the web and film to a point below that at which the film is tacky.

While illustrative forms of the improved apparatus have been described in considerable detail, it will be understood that numerous changes may be made in the various phases of the invention without departing from its scope.

What I claim is:

Apparatus for drying films formed from a solution of film forming substances which comprises an elongated chamber, a flexible carrier for the film arranged to enter said chamber at one end and emerge from said chamber at its other end, an auxiliary chamber at each end of said first-mentioned chamber, narrow openings being provided at the outer ends of said auxiliary chambers and between the same and said first-mentioned chamber for the pasasge of said carrier without rubbing contact with the film, means for introducing an inert gas under a slight pressure above atmospheric into each of said auxiliary chambers, means for withdrawing vapors and gases at one point in said first-mentioned chamber and returning vapors and gases to said first-mentioned chamber at another point therein, means for condensing and removing a portion of the vapors so withdrawn, said withdrawing and returning means being adapted to maintain a slight pressure above that in the auxiliary chamber in said first-mentioned chamber, and means for heating the remaining vapors and gases returned to said first-mentioned chamber.

MARC ALFRED CHAVANNES.

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