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PROCESS OF PREPARING MOLTEN GELATIN SOLUTION

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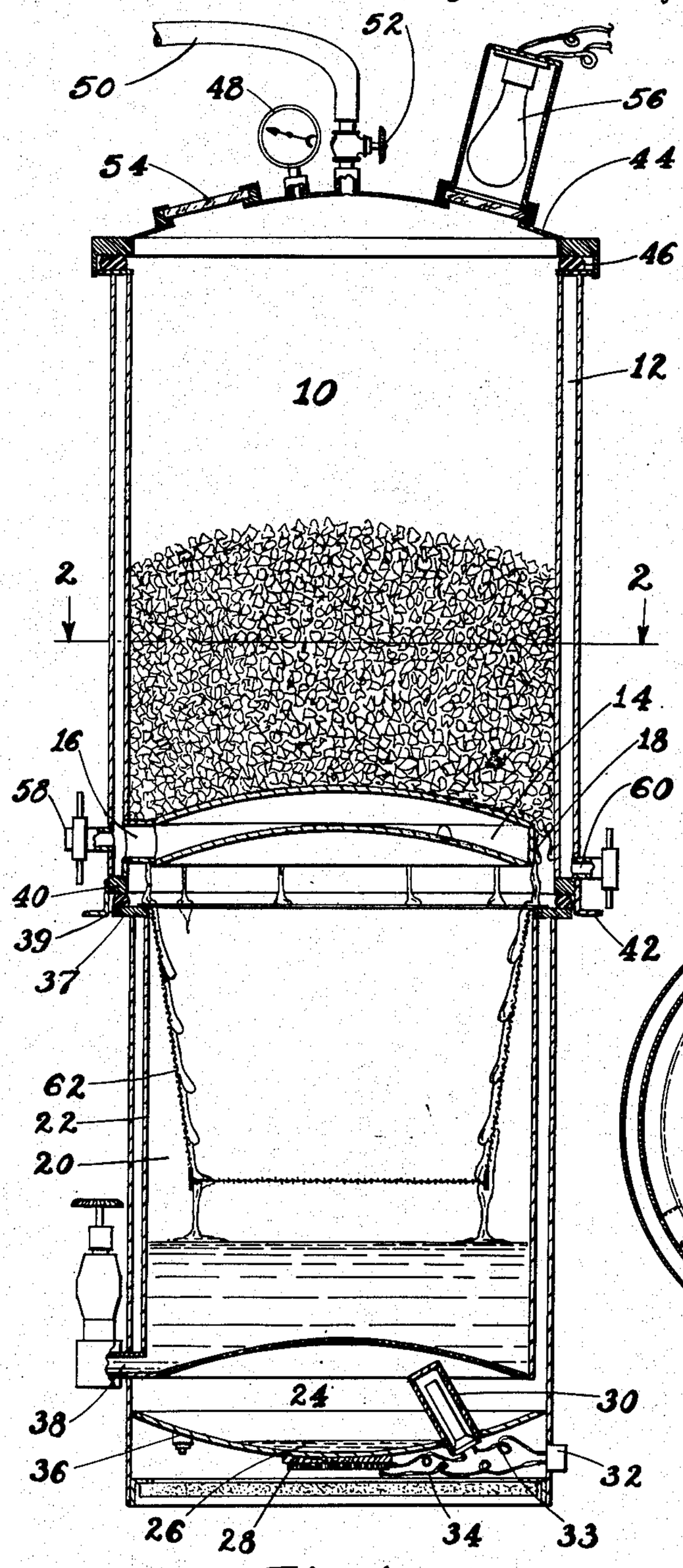

Fig. 1

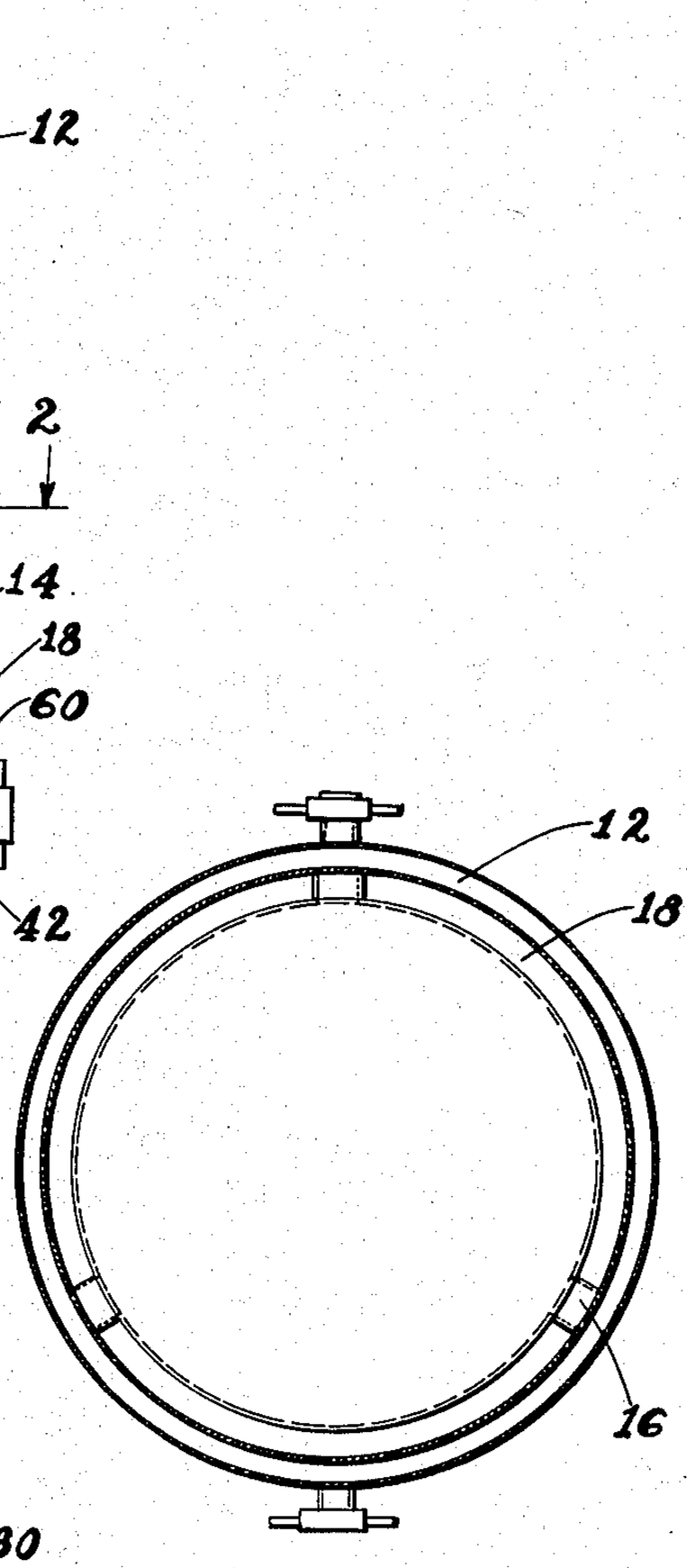

Fig. 2

INVENTOR.
John Otto Scherer

BY

Burton & Parker
ATTORNEY.

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PROCESS OF PREPARING MOLTEN GELATIN SOLUTION

John Otto Scherer, Detroit, Mich., assignor to R. P. Scherer Corporation, Detroit, Mich., a corporation of Michigan

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6 Claims. (Cl. 106—135)

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This invention relates to improvements in the process of preparing molten gelatin solutions. It is a division of my United States application, Serial No. 496,817, filed July 30, 1943, now abandoned.

An object is to provide an improved process of preparing a molten gelatin solution of the desired composition and consistency and relatively free from air bubbles.

More particularly the invention relates to the preparation of molten gelatin solutions which are relatively viscous and which are suitable for the casting of bands or ribbons such as are employed in the fabrication of hermetically sealed gelatin capsules.

A further object is to provide an improved process whereby gelatin solutions having the desired characteristics may be prepared quickly, cheaply and with the minimum of attention on the part of the operator.

Another object is to provide improvements in method of preparing gelatin solutions through the employment of which the operation may be carried out with the minimum of possibility of injury to the gelatin.

Heretofore one practice of preparing a molten gelatin solution has been to soak dry gelatin flakes in a mixture of cold water and glycerin of the proportions desired in the finished solution and thereafter to heat the soaked gelatin mass in a double boiler type of heating apparatus until the gelatin was reduced to the molten state. The gelatin was then allowed to stand for the necessary period of time for the air bubbles to pass off which time period might be 24 to 48 hours.

Another practice has been to soak gelatin flakes in a mixture of glycerin and cold water but with the water present in an amount in excess of that required for the finished solution. This produced a more dilute solution than resulted from the process first described and the air bubbles rose more freely from such dilute solution. This mixture was then melted and following the melting it was concentrated under carefully controlled heat and at sub-atmospheric pressure, reducing the water content to the desired amount. Careful control of heat was necessary in this process to prevent gelatin deterioration and though the time required was reduced, as compared with the practice first described, the concentration step required considerable time. Careful attention was necessary as a boiling gelatin solution foams quickly and violently.

It is not satisfactory to mix dry gelatin flakes with hot water for in such a mixture it is difficult

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to produce a smooth homogeneous solution. It is not satisfactory to fuse dry gelatin flakes to a molten mass because of the danger of burning the gelatin. To accomplish such requires very careful attention and a considerable length of time because of heat control required. Furthermore, a gelatin solution which is sufficiently viscous for the specific purpose hereinabove mentioned is, because of such viscosity, very slow to release contained air and a substantial time period is required to get the air out of the solution.

This invention is designed to overcome the disadvantages exemplified in the two practices hereinabove discussed and to provide a gelatin solution of the required characteristics quickly and economically.

The apparatus disclosed in the drawing is apparatus which has been found suitable for the carrying out of the process.

Other objects, advantages and meritorious features will more fully appear from the following description, appended claims and accompanying drawing wherein:

Figure 1 is a vertical section through apparatus suitable for the carrying out of the invention.

Figure 2 is a horizontal sectional view taken on the line 2—2 of Figure 1.

The invention is applicable to the preparation of gelatin solutions which may vary from what is known as a hard gelatin to a soft gelatin. A hard gelatin might consist of 42% dry gelatin flakes, 16% glycerin and 42% water. A soft gelatin might consist of 40% gelatin flakes, 30% glycerin and 30% water.

The apparatus whereby the improved process may be carried out is here illustrated as comprising a container 10, which has a jacketed side wall portion 12, and a jacketed bottom wall portion 14. This vessel is adapted to serve as the gelatin melting vessel. The bottom wall portion is of less diameter than the inside diameter of the side wall portion whereby the bottom is supported from and communicates with the side wall by means of short tubular sections 16 which bridge the space between the bottom wall and the side wall and establish the fluid communication therebetween. Due to this spacing between the bottom wall and the side wall there is provided a series of discharge openings 18 for molten gelatin. The bottom wall is convex upwardly so that as the gelatin melts it flows down through the openings 18.

The melting vessel 10 superposes an accumulating vessel 20 which vessel 20 has a jacketed side wall 22 and a jacketed bottom wall 24. The jack-

eted side wall portion 22 communicates with the hollow bottom portion 24 as shown. These confined spaces 22 and 24 are hermetically sealed and evacuated of air to within about ⅛'' to ¼'' mercury column of absolute vacuum. A small quantity of air free distilled water 26 is placed in the hollow bottom of the vessel 20. A conventional electric heater 28 is disposed adiacent to the jacketed bottom of the vessel 20. An electric thermostat is indicated at 30. Connecting electric plug 32. is connected with the heater through the thermostat by lead wires 33. An insulating protective bottom wall 34 is provided as shown. At 36 is a connection whereby the jacketed portion of the vessel 20 may be evacuated of air as hereinabove described and through which the distilled water may be placed therein.

There is indicated at 38 a valve controlled outlet through which molten gelatin may be withdrawn from the vessel 20. A flange ring 37 closes the jacketed side wall space 22 and forms a support for the vessel 10. A gasket 38 is interposed between this ring and the bottom ring 40 of the side wall of the vessel 10. To hold the vessel 10 against displacement upon vessel 20 a flange ring 42 is provided which depends below the vessel 10 and embraces the flange ring 37 of the vessel 20.

Upper vessel 10 is provided with a cover 44 adapted to be received over the open top of the vessel. A suitable gasket 46 is provided to form an air tight seal between such cover and vessel 10. 48 is a vacuum gauge for indicating the pressure within the container and 50 is an air evacuating pipe having a control valve 52. This pipe may be connected with suitable vacuum producing means to withdraw air from the container 10. A glass window 54 is provided to view the operation within the container and 56 indicates a light bulb which may be connected in an electric circuit to illuminate the interior.

A valve controlled steam pipe connection 58 leads into the lower part of the jacketed side wall from one side and a valve controlled outlet pipe connection 60 leads therefrom at the opposite side. Steam may be taken in through the intake connection 58 into the jacketed portion of the vessel 10 and steam condensate may be withdrawn through the outlet pipe 60.

I first mix the dry gelatin flakes with the desired quantity of water, and preferably plus a small excess of water over that desired in the finished solution. The water is cool water. This mixing is carried out in a suitable apparatus such as a pony mixer which thoroughly stirs the mass, say from ten to thirty minutes, until the gelatin picks up the liquid and is thoroughly soaked. Gelatin solutions of the character described contain gylcerin and my liquid mixture is one of cold water and glycerin. The glycerin is provided in the proportionate amount required for the finished solution and the water is provided in approximately such amount or preferably, as above stated, slightly in excess of that required in the finished solution.

This soaked gelatin mass, with the gelatin in the solid but impregnated state, is then placed in the melting vessel 10. The discharge openings 18 are not large enough to permit the soaked gelatin flakes to fall therethrough. After the soaked gelatin mass has been placed in the vessel 10, the cover 44 is placed thereon and the connection 50 with a suitable conventional vacuum producing system, not shown, is established and the valve 52 is opened so as to permit withdrawal of air from the container. This evacuation of air is preferably carried down to an absolute pressure of from one-half inch to three-quarter inch of mercury and is maintained for the necessary amount of time to withdraw substantially all of the air from the gelatin mass. In the practice as presently pursued a time period of about twenty minutes evacuation has been found satisfactory.

Inasmuch as the object is to get substantially all of the air out a portion even of the water is withdrawn, but not so as to reduce the water content below that required for the finished solution. It is for this reason that a small excess of water is originally provided. Withdrawal of water vapor flushes the air out and dilutes the small amount of air content remaining. As presently practiced, about 90% or more of the air is evacuated.

Following this step of evacuating the air from the gelatin mass while maintaining such mass hermetically sealed against atmosphere the valve 52 is closed. Heating steam at 212° F. is then admitted through the valve controlled inlet 58 into the jacketed side wall and bottom portion of the container 10. The gelatin mass in contact with this warm surface will melt and drain down through the openings 18 as rapidly as the same is melted and before it has been heated sufficiently long to injure the gelatin. The construction is such that the circumferential drain outlet 18 provides what might be termed a reverse funnel effect. The surface flow area increases the further the melted gelatin flows from the center of the bottom 14 of vessel 10. The purpose is to permit the molten gelatin to flow away as rapidly as it becomes molten and prevent the formation of a molten gelatin pool subjected to injurious heating. If gelatin is subjected to this heat in the melting pot for a protracted period of time it will break down and lose its Blum or jelly-like strength. Gelatin is a good heat insulator and unless the melted gelatin is withdrawn as it melts the molten gelatin pool which is formed will inhibit the melting of the solid gelatin which superposes the liquid pool.

Disposed within the lower container 20 is an annular tapered flow directing element 62 which is shown in Fig. 1 as removably supported from the upper margin of said vessel. Such element 62 directs the molten gelatin into the vessel 20 without any appreciable splashing thereof. This draining will continue until all the gelatin within the upper vessel has been melted and drained down.

In the meantime the temperature of the lower vessel will be maintained at such a degree by the electric heater 28 as to maintain the gelatin in the molten state without heat injury. The thermostat may be set to maintain a temperature of 120° F. to 135° F. and this will cause the accumulating vessel 20 to maintain the gelatin mass at the required temperature.

When the melting vessel 10 has been emptied, as observed through the viewing window 54, then the steam supplied to the melting vessel may be cut off and the condensate valve in outlet 60 is opened and the air exhaust pipe 50 disconnected from the vacuum system. The melting tank 10 may then be removed from the lower vessel 20. A cover may then be placed over the lower vessel 20 and such accumulating vessel may be kept at the temperature required to maintain the molten solution in the desired state. It is obvious that

the melting vessel might be kept in its superposed position if such were desired.

By melting the gelatin in this fashion it is possible to provide a molten solution which is relatively free from air bubbles and which does not require careful attention on the part of the operator during its preparation, which is constantly accumulated as formed, and which is retained at the desired temperature during the accumulation. This process is relatively free from any possibility of damage to the gelatin during the formation of the solution. If the steam is left on too long no harm is done. Any bubbles which may have water vapor cores will be collapsed by condensation of the water vapor within them when the atmospheric pressure is restored to the gelatin solution in the accumulation vessel.

What I claim is:

1. In the process of preparing a molten gelatin solution from water-impregnated gelatin, the steps of hermetically sealing the impregnated gelatin mass from the atmosphere and withdrawing substantially all of the air therefrom, and subsequently heating the gelatin mass sufficiently to melt the same while maintaining it hermetically sealed from the atmosphere.

2. In the process of preparing a molten gelatin solution from water-impregnated gelatin, the steps of hermetically sealing the impregnated gelatin mass from the atmosphere and withdrawing substantially all of the air therefrom, subsequently heating the gelatin mass sufficiently to melt the same while maintaining it hermetically sealed from the atmosphere, and withdrawing the melted gelatin from the mass as the same is melted.

3. In the process of preparing a molten gelatin solution from water-impregnated gelatin, the steps of hermetically sealing the impregnated gelatin mass from atmosphere and withdrawing by vacuum substantially all of the air and some of the water therefrom, closing the vacuum connection, and heating the gelatin mass to melt the same while maintaining the hermetic seal thereon.

4. In the process of preparing a molten gelatin solution from water-impregnated gelatin, the steps of hermetically sealing the impregnated gelatin mass from atmosphere and withdrawing by vacuum substantially all of the air and some of the water therefrom, closing the vacuum connection, heating the gelatin mass to melt the same while maintaining the hermetic seal thereon, and withdrawing the melted gelatin from the mass sufficiently rapidly to prevent the formation of a pool of melted gelatin in association with the mass.

5. In the process of preparing a molten gelatin solution from gelatin impregnated with an aqueous liquid mixture, the steps of hermetically sealing said impregnated gelatin mass against atmosphere, withdrawing by vacuum substantially all of the air from the mass, and, while maintaining the hermetic seal thereon, heating the gelatin mass throughout its bottom area to progressively melt the same, and withdrawing the melted gelatin from the mass throughout an area extending substantially circumferentially about and below the bottom of the mass, said withdrawal being sufficiently rapid to prevent formation of a pool of melted gelatin along the bottom of the mass.

6. That process of preparing a molten gelatin solution which comprises impregnating gelatin with water in excess of the amount desired in the molten solution, hermetically sealing the impregnated gelatin mass from the atmosphere, withdrawing by vacuum substantially all of the air and the water vapor equivalent of said excess of water from the gelatin, and, while maintaining it hermetically sealed from the air, applying heat to the gelatin sufficient to melt the same, withdrawing melted gelatin substantially as rapidly as the same in melted and depositing it within an accumulator vessel while maintaining the gelatin within said accumulator vessel in the molten state.

JOHN OTTO SCHERER.

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