Feb. 9, 1954 R. F. COLTON 2,668,364

DRYING OF MATERIALS BY INFRARED RADIATION

Filed Oct. 27, 1950 2 Sheets-Sheet 1

Fig. 1

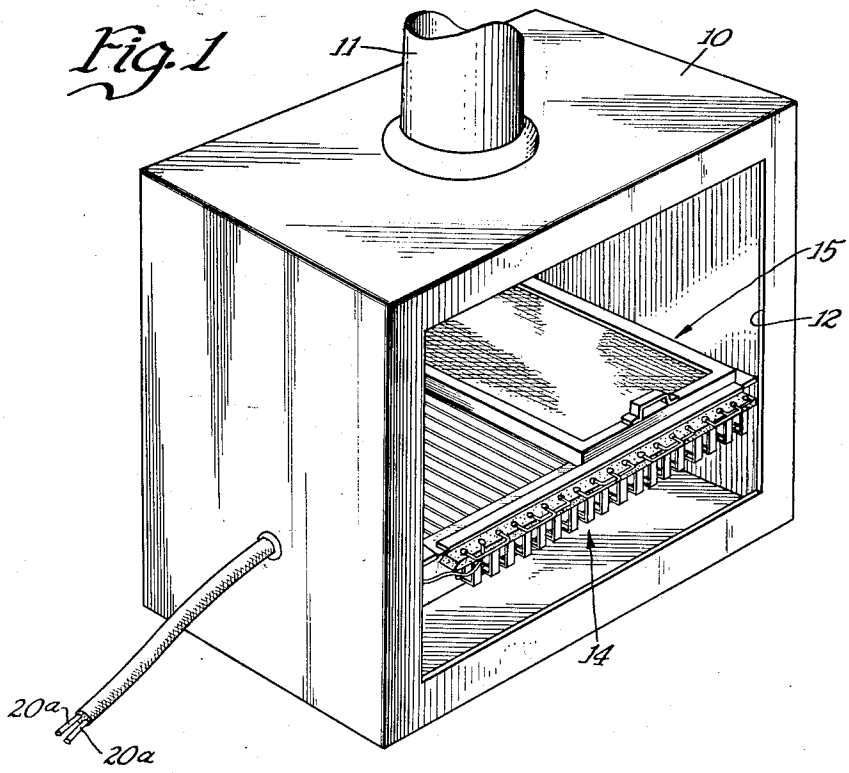
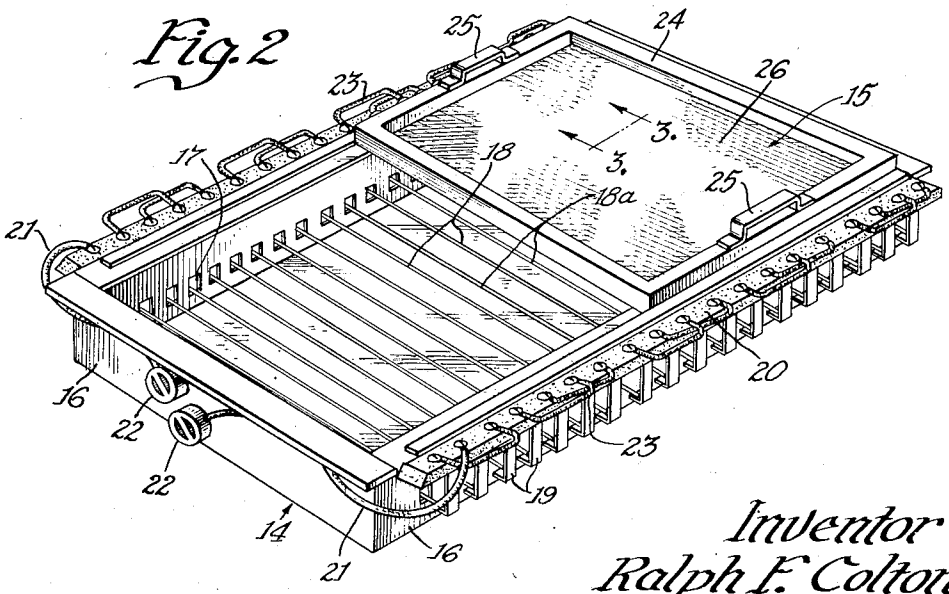


Fig. 2



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2 Sheets-Sheet 2

Fig. 3

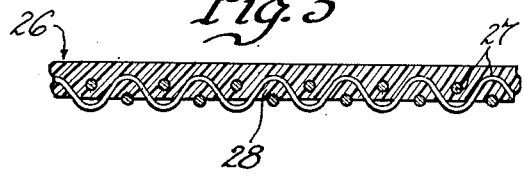


Fig. 4

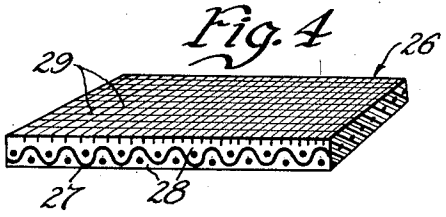


Fig. 5

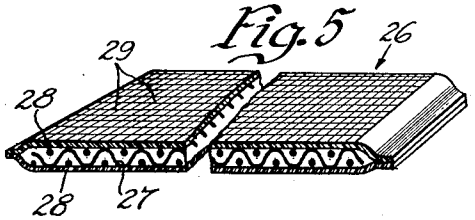


Fig. 6

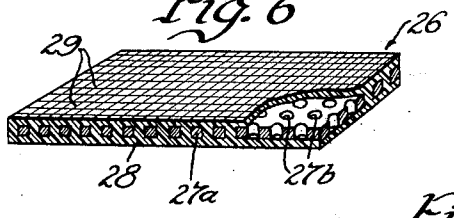


Fig. 7

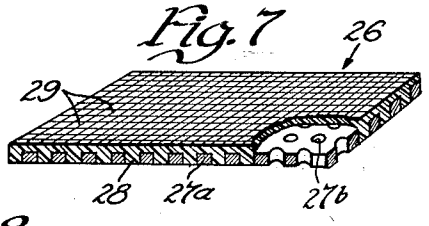


Fig. 8

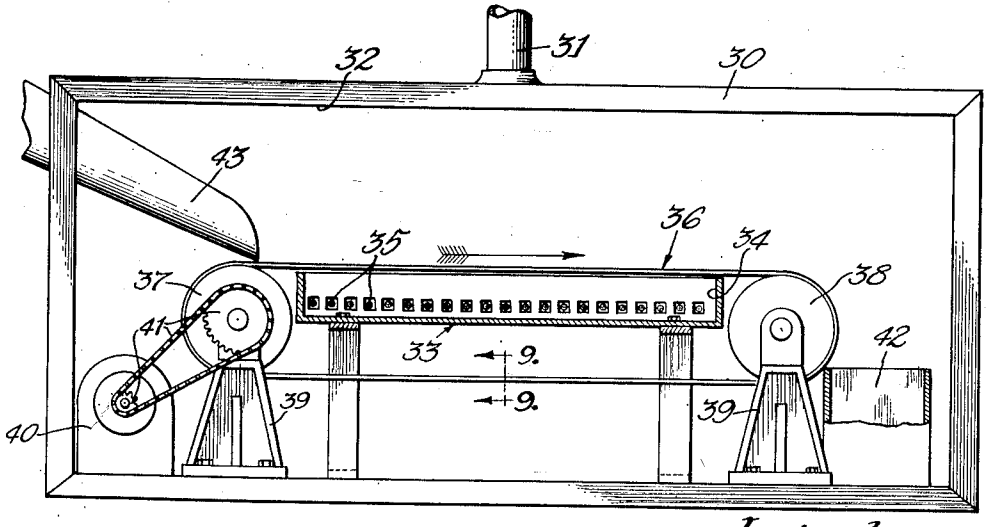
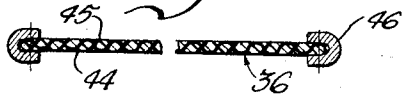


Fig. 9



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Patented Feb. 9, 1954

2,668,364

UNITED STATES PATENT OFFICE

2,668,364

DRYING OF MATERIALS BY INFRARED RADIATION

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Application October 27, 1950, Serial No. 192,574

1 Claim. (Cl. 34—4)

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This invention relates to the drying of materials by infra-red radiation.

More particularly, this invention contemplates and provides improvements in the utilization of heat radiated in the spectral range of 1μ to 10μ and reaching a material being dried by passing through a fluid-impervious support in contact therewith.

This invention is predicated upon my discovery that important advantages can be realized by utilizing, in the structure of the aforementioned support, a plastic consisting of polymerized ethylene-type molecules. According to this invention such support comprises a foraminous sheet that has been rendered impervious to fluids by having intimately associated therewith a plastic consisting of polymerized ethylene-type molecules. Advantageously, but not necessarily, such plastic may have incorporated therein one or more of certain ground inorganic halides, ground quartz and/or ground glass.

Other features, objects and advantages of this invention will appear from the following description wherein reference is made to the accompanying drawings in which:

Fig. 1 is a perspective view of a drying apparatus embodying the improvements of my present invention, a side wall closure of the housing of such apparatus being removed;

Fig. 2 is a perspective view of the heater, and its associated tray for supporting the material to be dried, as they appear before placement in the housing;

Fig. 3 is a fragmentary sectional view, of the floor or bottom of the tray, that may be regarded as taken in the vertical plane of the line 3—3 of Fig. 2;

Figs. 4 to 7 both inclusive are perspective views of specifically different structural forms of the tray bottom or floor;

Fig. 8 is a side elevational view of a modified drying apparatus, i. e., one wherein the support for the material to be dried is in the form of an endless belt, embodying my invention; and

Fig. 9 is a transverse sectional view of the belt which may be regarded as taken in the vertical plane of the line 9—9 of Fig. 8.

Similar characters of reference refer to similar parts throughout the several views.

Drying apparatus characterized by my present

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invention may take numerous specifically different structural forms. Two specifically different structural forms of such apparatus are depicted in the accompanying drawings. One of these, illustrated in Figs. 1 and 2, comprises a housing 10, adapted to be tightly closed against the inflow of air when in use. Housing 10 is provided with exhaust conduit 11 through which gaseous fluids may be exhausted from the housing 10 by the action of a suitable vacuum pump (not shown) associated with such conduit. As shown in Fig. 1, housing 10 is provided with a side wall opening 12 through which access may be had to the contents of the housing, but it will be understood that when the drying apparatus is in use such opening 12 is tightly closed by a panel (not shown) which is secured and sealed to the housing in any suitable manner. Optional equipments (not shown) with which the housing 10 may be provided, according to the preferences of the user and/or the particular type of drying that is to be effected with infra-red radiation, are refrigerated surfaces for the condensation of vapors, conventional instrumentalities, other than the gaseous fluid-exhausting devices comprising conduit 11, for refrigerating the interior of the housing, devices for indicating the temperature and/or pressure existing within the housing, devices for indicating the temperature of the material being dried, and switching mechanism either manually or automatically operated for controlling the supply of electrical energy to the heater next to be described.

Horizontally mounted within housing 10 in any suitable manner (not shown) is a heater 14 which is adapted removably to support one or more trays; one of such trays being depicted at 15. Heater 14 comprises a rectangular frame 16, apertures 17 in the side members of such frame, wire-like heating elements 18 and 18a extending across such frame and freely through such apertures 17, support-conductors 19 with which the ends of the heating elements 18 and 18a are electrically connected, insulating bars 20 whereon the support-conductors 19 and their respective terminals are mounted, current supply conductors 21 leading from terminal receptacles 22, and jumpers 23 by which the proper circuit relationships between the heating elements 18 and 18a and their electrical connection with the sup-

ply conductors 21, are established. Suitably, but not necessarily, the several heating elements 18 and 18a are connected in series with each other. Although all of the heating elements may be alike, that is not necessarily so. For example, the heating elements 18 may differ from the heating elements 18a as to size, or the material of which formed, or in any other permissible respect. Terminal receptacles 22 are intended for cooperation with mated terminal members (not shown) electrically connected with other supply conductors 20a leading to a suitable source of electrical energy.

The heating elements 18 are black bodies which, when the heater is in use for my purposes, are caused to glow between 250° C. and 1500° C. A hot black body at a temperature between 250° C. and 1500° C. emits at least 60% of its total radiation in the spectral range of from 1μ to 10μ. These phenomena were pointed out by Doctors Levinson and Oppenheimer in their United States Letters Patent No. 2,445,120 which are directed to an infra-red sublimation process of drying that may be advantageously practiced with the aid of the improvements of my present invention. However, the improvements of my present invention are not limited to use in the practice of such sublimation process.

The tray 15 suitably consists of a frame 24 that is equipped with handles 25 and that is secured to and circumscribes a tray bottom or floor 26. It is through this tray bottom or floor 26 that material being dried by the apparatus of my invention is subjected to infra-red rays emitted by the heating elements 18. According to my invention, such bottom or floor 26 is of a composite construction consisting, essentially, of a foraminous sheet rendered impervious to fluids, and sufficiently chemically inert, by having intimately associated therewith a plastic consisting of polymerized ethylene-type molecules, the foraminous sheet, in turn, contributing to its associated plastic strength and dimensional stability. The foraminous sheet may be rigid or flexible. Figs. 3 to 7 inclusive of the accompanying drawings depict four specifically different forms which the requisite composite structure of the tray bottom or floor 26 may take. Additional variations of such composite structure will suggest themselves to persons skilled in the art to which my invention relates.

Heretofore, when materials have been dried by infra-red rays reaching such materials by passing through the supports therefor, such supports have been formed of glass or nylon. Glass and nylon, unfortunately, are not efficiently transparent to infra-red rays. A seriously substantial loss of radiant energy is experienced whenever heat radiated in the spestral range of 1μ to 10μ is required to pass through a glass or nylon support to a material being dried that is disposed on or in such support. Fortunately, according to the discovery whereon my present invention is predicated, a plastic consisting of polymerized ethylene-type molecules is so very efficiently transparent to infra-red rays that but a relatively small and practically quite tolerable loss of radiant energy is experienced when heat radiated in the spectral range of 1μ to 10μ passes, to a material being dried, through one of my new supports of composite structure, even though the foraminous sheet component of the support is relatively opaque to infra-red radiation. A striking example of this salutary phenomenon will be recorded at a later point in this specification.

The material-to-be-dried support 26 of Fig. 3 comprises a foraminous sheet in the form of a woven fabric 27 with which is associated a plastic 28 consisting of polymerized ethylene-type molecules. Such plastic suitably is tetrafluoethylene polymer $(C_2F_4)_x$, trifluomonochlorethylene polymer $(C_2ClF_3)_x$, or polyethylene $(C_2H_4)_x$. Presently tetrafluoethylene polymer $(C_2F_4)_x$ is marketed in the United States under the trade-mark "Feflon," trifluomonochlorethylene polymer

$(C_2ClF_3)_x$

is being marketed in the United States under the trade-mark "Kel-F," and polyethylene $(C_2H_4)_x$ is being marketed in the United States under various trade-marks of which one is "Polythene." The fabric 27 desirably is woven of spun glass fibers but may be woven of metal wires (e. g., copper or stainless steel wires) or even certain plastic yarns now available. Generally speaking, however, a foraminous sheet woven of plastic yarns is not as suitable for my purposes as is one woven of spun glass fibers or metal wires, because the plastic yarns do not possess desirable degrees of dimensional stability and sometimes disintegrate at elevated temperatures. In the support 26 of Fig. 3 the plastic 28, consisting of polymerized ethylene-type molecules, fills the interstices of the woven fabric 27, and completely covers one face thereof, but at the other face of the woven fabric leaves its warp and woof strands exposed. Although the upper surface of the plastic 28 may be comparatively smooth as shown, its upper surface may be roughened, by scarification or otherwise, if desired.

The material-to-be-dried support 26 of Fig. 4 also comprises a woven fabric 27, woven of spun glass fibers or metal wires or even certain plastic yarns, with which is associated a plastic 28 consisting of polymerized ethylene-type molecules; the plastic suitably being tetrafluoethylene polymer, trifluomonochlorethylene polymer, or polyethylene. In this instance the plastic 28 covers both faces of fabric 27 in addition to occupying its interstices, and one surface of the plastic 28 is suitably roughened, as by criss-cross scarifications 29, for contact with the material to be dried.

The material-to-be-dried support of Fig. 5 differs substantially from the one of Fig. 4 only in that the plastic 28 does not occupy the interstices of the woven fabric 27. In the support 26 of Fig. 5 the plastic 28, as applied to woven fabric 27, is in two sheets which are heat-sealed or otherwise continuously joined together along all or a sufficient number of the margins of such fabric 27.

The material-to-be-dried support 26 of Fig. 6 comprises a foraminous sheet in the form of a plate 27a provided with a multiplicity of perforations or through-apertures 27b. Plate 27a may be formed of metal or glass or other materials having the requisite physical characteristics. Such plate 27a has its faces covered, and its perforations or through apertures 27b occupied, by a plastic consisting of polymerized ethylene-type molecules; the plastic suitably being tetrafluoethylene polymer, trifluomonochlorethylene polymer or polyethylene. Here again one exposed face of the plastic 28 is suitably roughened, for contact with the material to be dried, as by criss-cross scarifications 29.

The material-to-be-dried support 26 of Fig. 7 differs substantially from the one of Fig. 6 only

in that the plastic does not cover that face of the plate 27a which is not intended to contact the material to be dried.

In using the apparatus of Figs. 1 and 2, the material to be dried, which may be a serum, or a pharmaceutical, or a fruit or spice or vegetable juice or extraction, or practically any other material that feasibly may be dried with the aid of infra-red rays in the spectral range of 1μ to 10μ, is placed upon tray 15. The tray is placed upon heater 14, the side wall opening 12 is closed, conduit 11 is connected with a suction producing means, and the heating elements 18 and/or 18a are electrically energized to cause the passing of infra-red rays, in the spectral range of 1μ to 10μ, to radiate from such heating elements 18 and 18a to and through the tray bottom 26 and into the material to be dried, with the result of causing the material to be dried to give off vapor that is withdrawn from housing 10 through conduit 11. The degree of vacuum, and the temperature, maintained within housing 10 may vary within very wide limits without losing the benefits of my invention. However, when the apparatus of my invention is utilized in performing the sublimation process of the hereinbefore-mentioned Levinson and Oppenheimer Patent No. 2,445,120, the pressure within the housing usually will be maintained below 3mm. of mercury and the temperature within the housing will be maintained in the vicinity of −20° C..

The apparatus of Figs. 8 and 9 comprises a housing 30 adapted to be suitably closed against the inflow of air when it is in use. Such housing is provided with an exhaust conduit 31 through which gaseous fluids may be exhausted from the housing by the operation of a suitable vacuum pump (not shown). Such housing 30 is provided with a side wall opening 32 through which access may be had to the contents of the housing, but it will be understood that when the drying apparatus is in use such opening 32 is tightly closed by a panel (not shown) which is secured and sealed to the housing in any suitable manner. Here again optional equipments (not shown) with which the housing may be provided, according to the preferences of the user and/or the particular type of drying that is to be effected with infra-red radiation are refrigerated surfaces for the condensation of vapors, conventional instrumentalities, other than the gaseous fluid exhausting devices comprising conduit 31, for refrigerating the interior of the housing, devices for indicating the temperature and/or pressure existing within the housing, devices for indicating the temperature of the material being dried, and switching mechanism either manually or automatically operated for controlling the supply of electrical energy to the heater with which the housing 30 is equipped.

Horizontally mounted within housing 30, and conveniently carried on supporting legs as shown, is a heater 33. Such heater comprises, essentially, a tray-like frame or casing 34 and a plurality of horizontally disposed heating elements 35 carried by and extending transversely of the casing 34. Such heating elements 35 are suitable black bodies which, when properly energized, radiate 60% of their rays in the spectral range of 1μ to 10μ. Heater casing 34 is open at the top thereof; if desired, the upper surface of its bottom may be polished or otherwise treated or equipped so as to reflect upwardly heat radiated to it from the heating elements 35. The connections through which electrical energy is supplied to the heating elements 35 have not been shown.

Depicted at 36 is an endless belt passing around a driven roller 37 and an idler roller 38. Bearings for these rollers may be afforded by suitable standards 39. Rotation of roller 37, to effect movement of belt 36 in the direction of the feathered arrow is effected by an electric motor 40 through the agency of chain-and-sprocket mechanism indicated generally at 41. The upper reach of belt 36 passes closely adjacent to and directly over and in alignment with the top of the heater 33. Indicated at 42 is a receptacle for dried material, and indicated at 43 is a chute through which material to be dried is supplied, either intermittently or more or less continuously, to belt 36. When and if it is desired to maintain in housing 30 a pressure substantially below atmospheric, suitable devices (not shown) may be provided for suitably limiting, or intermittently preventing, the inflow of air with the material to be dried. When the material to be dried is a liquid, the inflow of air therewith will present no difficulty whatsoever. The specific structures and arrangement of the thus far described elements of the apparatus depicted in Figs. 8 and 9, may be varied within wide limits. A very essential feature of the apparatus now under consideration is that the belt 36 shall comprise a suitably flexible foraminous sheet, desirably in the form of a woven fabric 44, that has been rendered impervious to fluids by having intimately associated therewith a plastic 45 which consists of polymerized ethylene-type molecules. Desirably such a fabric 44 is woven of spun glass fibers or metal wires (e. g., copper or stainless steel wires). However, in some cases, such fabric 44 may be woven of certain now available plastic yarns. The plastic 45 desirably fills all of the interstices of the fabric 44 and desirably may more or less cover the two faces of the fabric. Such plastic 45 suitably is tetrafluorethylene polymer, trifluormonochlorethylene polymer, or polyethylene. The lateral edges of belt 36 suitably may be embraced by, and have secured thereto, flexible strips 46 which, as applied, are generally U-shaped in transverse cross section. Such strips 46 prevent lateral escape from the belt of material which has been placed thereon to be dried.

In using the apparatus of Figs. 8 and 9, the side wall opening 32 of housing 30 is closed and, if necessary, desirably sealed. Appropriate conditions of pressure and temperature are then established within housing 30. The elements 35 are energized so that the heater 33 will radiate its heat, at least 60% of which is in the spectral range of from 1μ to 10μ, against the lower surface of the upper reach of the belt 36. Motor 40 is then energized to motivate the belt. Material to be dried is supplied to the belt, through chute 43, either intermittently or more or less continuously. As carried along by the upper reach of the belt, such material is subjected to radiant heat, in the spectral range of 1μ to 10μ moving to it through the belt 36 and more particularly through that component of the belt which is the plastic composed of polymerized ethylene-type molecules. Vapor emitted by the material to be dried is carried away through conduit 31. As the upper reach of the belt passes downwardly around the idler roller 38, dried material falls therefrom into the receptacle 42.

Generally speaking, when the material to be dried is to be subjected to a relatively very high temperature, e. g., a temperature of the order of 375° C., that component of the material-to-

be-dried support which is in the nature of a plastic consisting of polymerized ethylene-type molecules, suitably will be tetrafluoethylene polymer; when the material to be dried need be subjected only to a relatively low temperatpre, e. g., one of the order of 35° C., such component desirably will be polyethylene; and when the material to be dried is to be subjected to temperatures of intermediate degree, such component suitably may be trifluomonochlorethylene polymer.

At the present time my instant invention is industrially significant and important because it materially increases the range of applicability and usefulness of the principles of that infra-red sublimation process described by Doctors Levinson and Oppenheimer in their United States Letters Patent No. 2,445,120. Prior to my instant invention, containers for supporting the material to be dried by such process were either glass or a sheet of plastic such as woven nylon.

In general, glass has limited transparency to radiation in the range from 1μ to 10μ, cutting off all radiation between 2.6μ and 10μ. Special heat-resisting glass such as is used for chemical or biological glassware has somewhat better transmission, being transparent to radiation between 1μ and 2.6μ and also being somewhat transparent in the range from 3μ to 4μ. Even glass made with maximum transparency to infra-red radiation, such as Corning No. 7280, absorbs between 40 per cent and 80 per cent of radiation in the preferred range of use. The brittleness of glass is a further limiting factor in commercial use. Plastics in sheet or woven form, while flexible and also somewhat more transparent to infra-red radiation than glass, have a high coeffcient of expansion, limited strength, and are not sufficiently stable dimensionally or thermally to permit their use over a wide range of temperatures, such as from minus 40° C. to 125° C.

According to my instant invention, remarkably excellent transparency of the ethylene-type polymers to infra-red radiation is recognized and made utilizable by combining them with a foraminous sheet. Such foraminous sheet desirably is a woven fabric having strength, flexibility, dimensional stability and, as in the case of woven glass fibers, some transparency to the radiation in the preferred range. In fact, combination of an ethylene-type plastic with somewhat brittle glass fibers produces a flexible composite which has such high tensile strength as to permit its use in long continuous belts having a thickness of as little as 0.015 inch (0.035 mm.). The thin layer of glass fabric in itself is more transparent than the minimum practical thickness of sheets of special infra-red transmitting glass. Tests have been made of the relative transparencies to infra-red rays, of the following:

(1) A glass plate (Corning No. 7280) having a thickness of 2.49 mm.,

(2) A glass plate (Corning No. 7280) having a thickness of 6.00 mm., and

(3) A material-to-be-dried support, of the composite construction characteristic of the present invention, which comprised a foraminous textile sheet woven with thirty-two strands of glass fibers (.015") per inch of width and sixteen strands of glass fibers (.05") per inch of length, with which was intimately associated the plastic tetrafluoethylene polymer, such plastic completely filling the interstices of the fabric and more or less covering the faces thereof.

Percentage of radiation transmitted

Band of Radiation	Glass Plate 1	Glass Plate 2	The Composite Support
	Percent	*Percent*	*Percent*
2.2μ to 2.4μ	[1] 100	[1] 100	[1] 100
2.4μ to 2.6μ	93	85	[1] 100
2.6μ to 4.0μ	50	19	95
4.0μ to 5.0μ	15	5	87
5.0μ to 8.0μ	9	0	35

[1] Approximately.

If the radiant energy is generated at a temperature of 1200° C., the composite support will permit 75% of the energy to pass into the material being dried, while the 2.49 mm. glass just described will pass only 59% and the same glass 6.00 mm. thick only 49%. If the radiant energy is generated at the temperature of 650° C., specified by Doctors Oppenheimer and Levinson to be a preferred temperature for the radiating source, the composite support passes twice the energy of the 2.49 mm. glass and three times that of the 6.00 mm. glass.

Importance of high transparency is twofold:

(1) The energy that fails to pass through the support is absorbed by the support, thereby heating it and causing melting or other thermal destruction of the material being dried. As an example, with an energy source at 1200° C. if the maximum energy per unit of area absorbable by the support without thermal deterioration of the products is 25 units, a maximum input of 83 units may be used safely if the support is of the composite construction described above, while a limitation of 61 units is imposed on the 2.49 mm. glass and 49 units on the 6.00 mm. glass.

(2) Energy most efficiently absorbed by water is in the wave band from 2.6μ to 3.1μ. The energy in this range is important to the achievement of a thoroughly dried product when it has the exceedingly porous structure of the typical lyophilized product. Glass absorbs strongly in this range. Of 100 units of total energy generated by a source at 1200° C., 12.3 units in the range of 2.6 to 3.1μ will pass through the composite support described above while only 7.2 units will pass through the 2.49 mm. of the best glass and only 3.7 units will be available in this desirable range if the same glass is used in the usual plate thickness of 6.00 mm.

Belts of the composite construction described above make it possible to replace batch drying in glass vials or trays with rapid continuous drying at greatly reduced processing expense.

My invention contemplates that significant percentages of ground inorganic halides and/or ground quartz and/or ground glass desirably may be thoroughly intermixed with that plastic component of the material-to-be-tried support which consists of polymerized ethylene-type molecules. Frequently these additives will accord the plastic desirable physical characteristics without significantly or objectionably reducing its transparency to infra-red rays in the spectral range of 1μ to 10μ. When such an additive is an inorganic halide, it usually will be sodium chloride, calcium fluoride, lithium fluoride, or potassium bromide.

Having thus illustrated and described my invention in presently preferred practical embodiments thereof, what I claim as new and desire to secure by Letters Patent of the United States is:

Drying apparatus comprising a container,

means for the elimination of vapors, a radiant heater in said container which emits at least 60% of its radiation in the spectral range of 1μ to 10μ, and a support for material to be dried, also in said container, so disposed with respect to said heater that energy radiating from the heater to material carried by said support passes through said support, asid support comprising a foraminous sheet rendered impervious to fluids by having intimately assoicated therewith a plastic composed of polymerized ethylene-type molecules, the plastic component of the support having intermingled therewith a ground additive chosen from the group which consists of sodium

chloride, calcium fluoride, lithium fluoride, potassium bromide, quartz and glass.

RALPH F. COLTON.

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