

Feb. 1, 1949.

N. N. STEPHANOFF

2,460,546

METHOD AND APPARATUS FOR TREATING MATERIALS

Filed Oct. 1, 1942

7 Sheets-Sheet 1

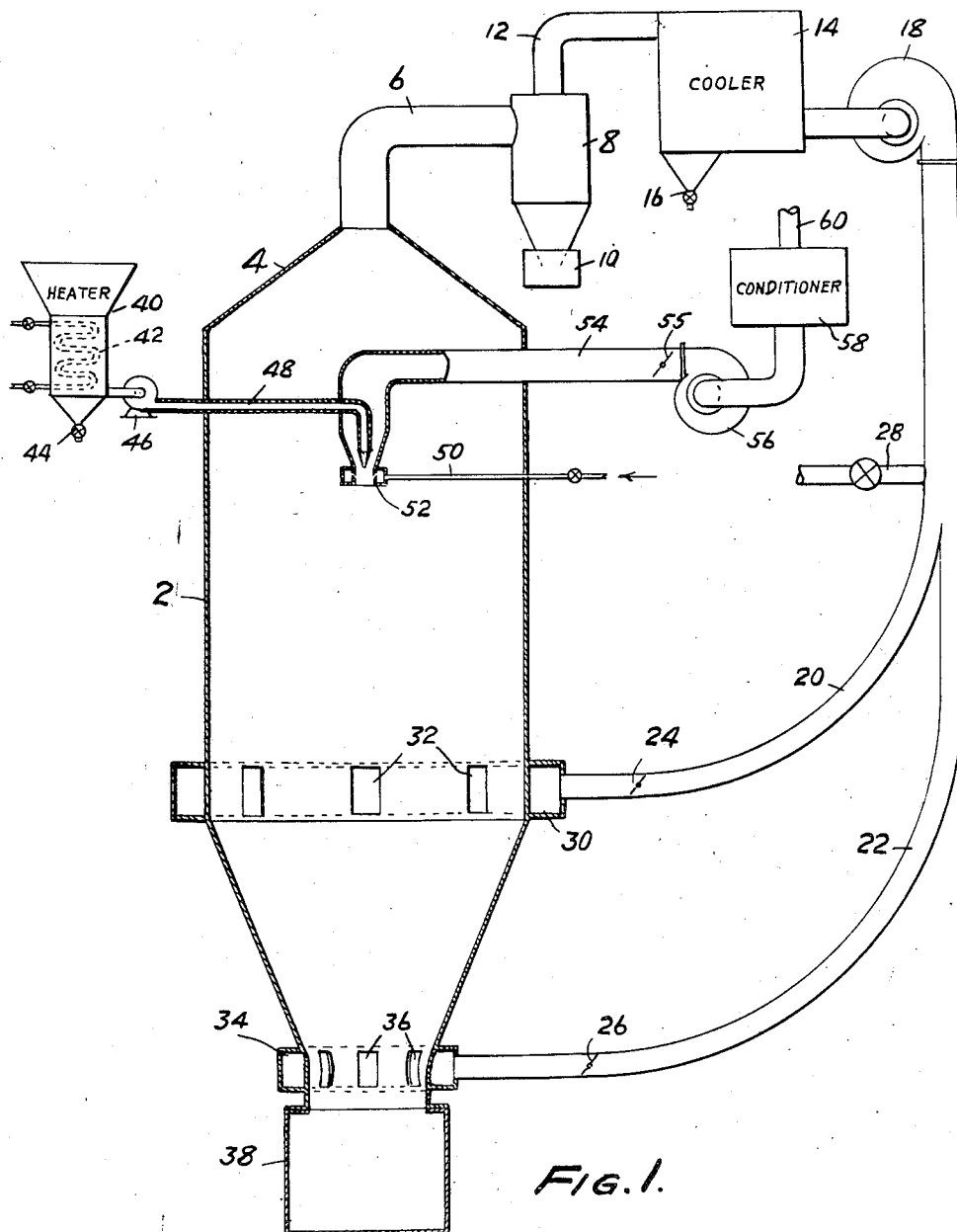


FIG. 1.

INVENTOR

WITNESS:

Robert P. Litchfield

Nicholas N. Stephanoff

BY

I. Sussner & Hardy
ATTORNEYS

Feb. 1, 1949.

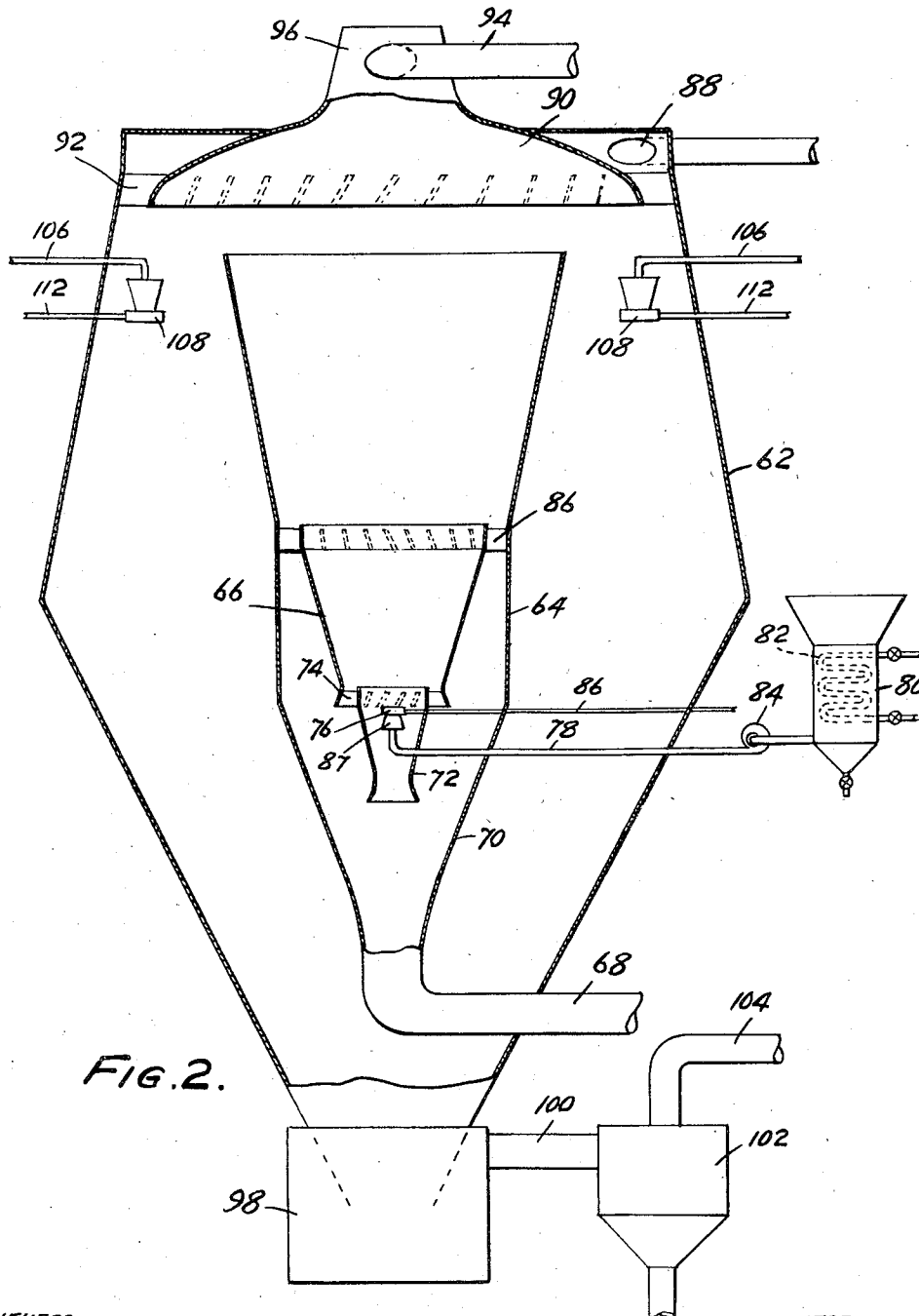
N. N. STEPHANOFF

2,460,546

METHOD AND APPARATUS FOR TREATING MATERIALS

Filed Oct. 1, 1942

7 Sheets-Sheet 2



WITNESS:

Robt. R. Mitchell

INVENTOR

Nicholas N. Stephanoff

BY *Buss & Harding*
ATTORNEYS.

Feb. 1, 1949.

N. N. STEPHANOFF

2,460,546

METHOD AND APPARATUS FOR TREATING MATERIALS

Filed Oct. 1, 1942

7 Sheets-Sheet 3

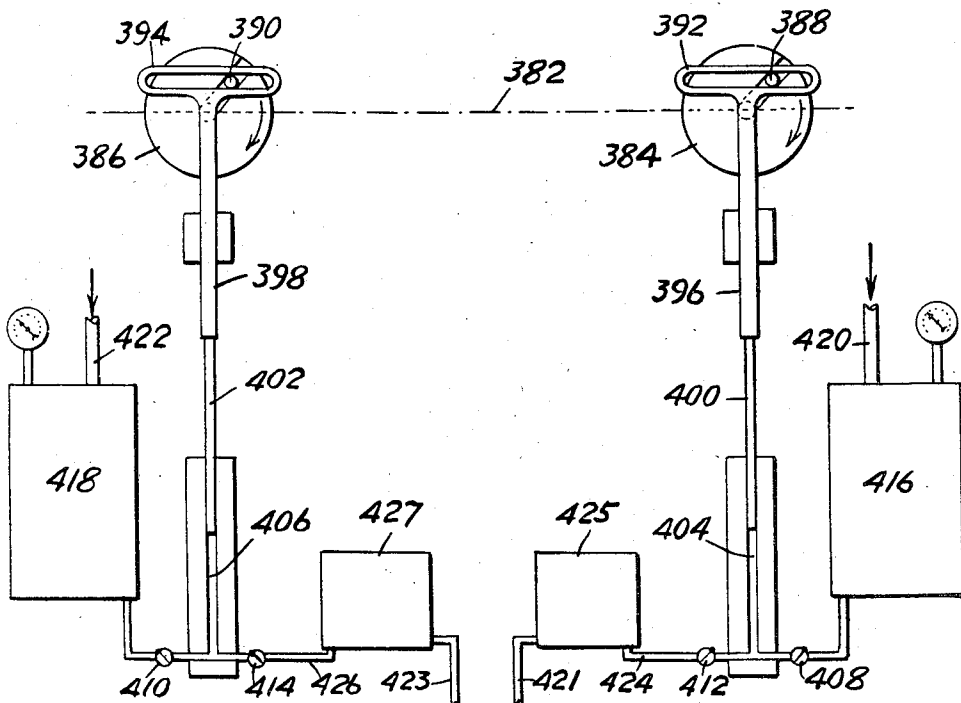


FIG. 9.

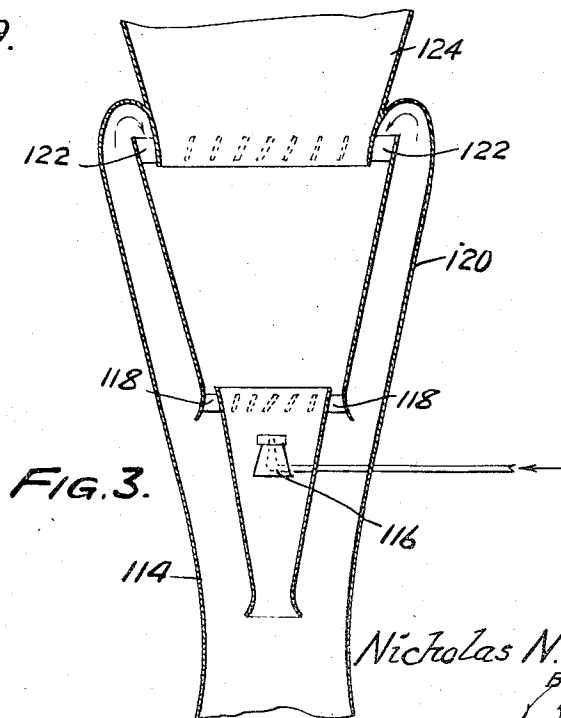


FIG. 3.

WITNESS:

Robert P. Ketchum

INVENTOR

Nicholas N. Stephanoff

BY *Brewer & Hardy*
ATTORNEYS.

Feb. 1, 1949.

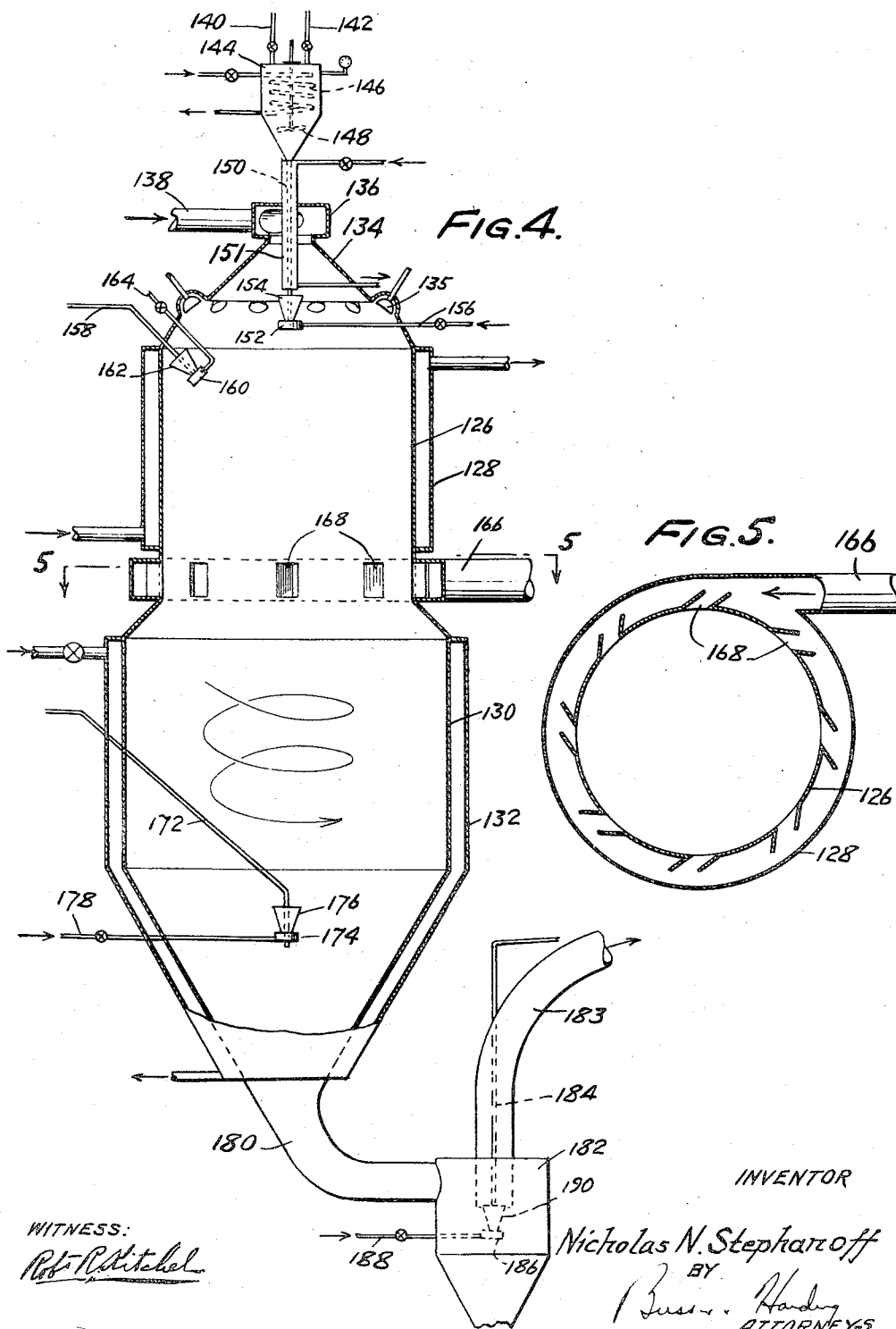
N. N. STEPHANOFF

2,460,546

METHOD AND APPARATUS FOR TREATING MATERIALS

Filed Oct. 1, 1942

7 Sheets-Sheet 4



Feb. 1, 1949.

N. N. STEPHANOFF

2,460,546

METHOD AND APPARATUS FOR TREATING MATERIALS

Filed Oct. 1, 1942

7 Sheets-Sheet 5

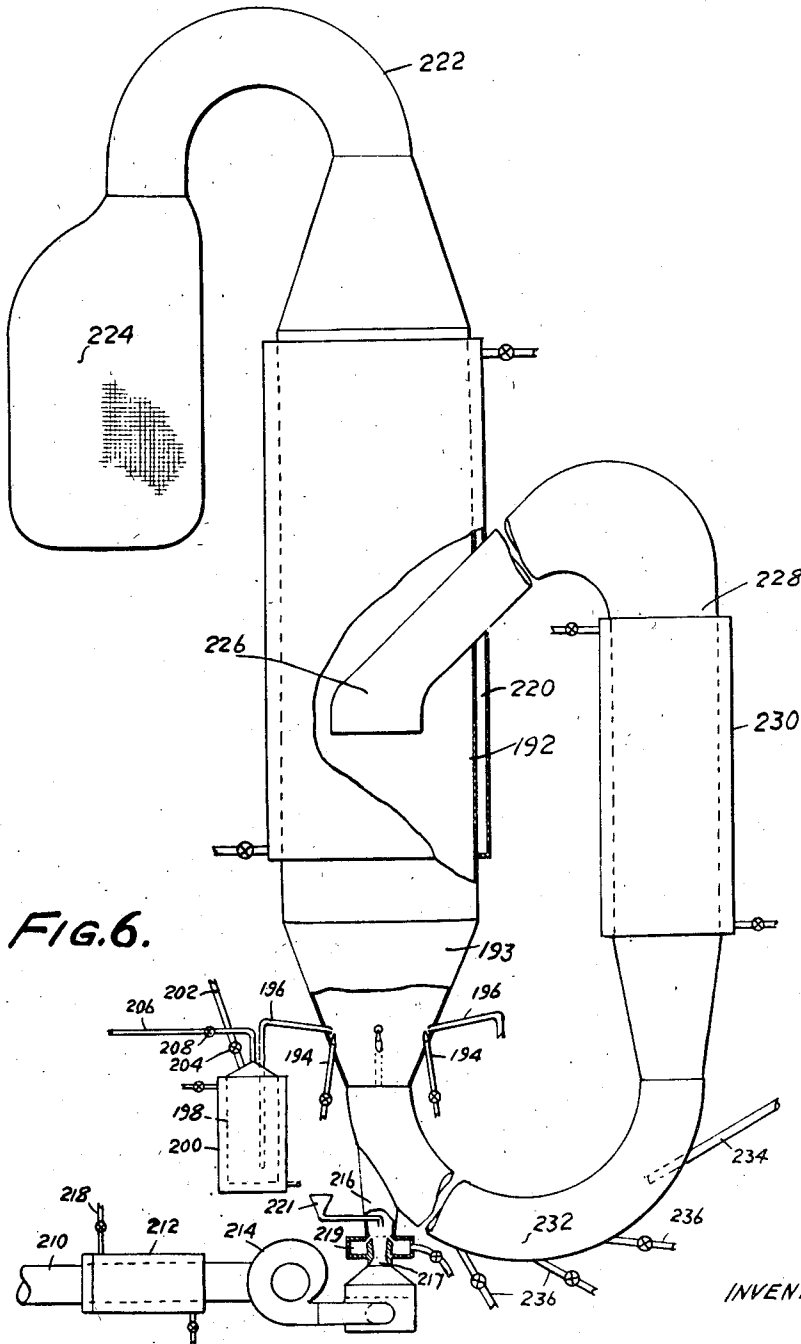


FIG. 6.

INVENTOR

WITNESS:

Robert R. Mitchell

Nicholas N. Stephanoff

BY

James H. Hickey
ATTORNEYS.

Feb. 1, 1949.

N. N. STEPHANOFF

2,460,546

METHOD AND APPARATUS FOR TREATING MATERIALS

Filed Oct. 1, 1942

7 Sheets-Sheet 6

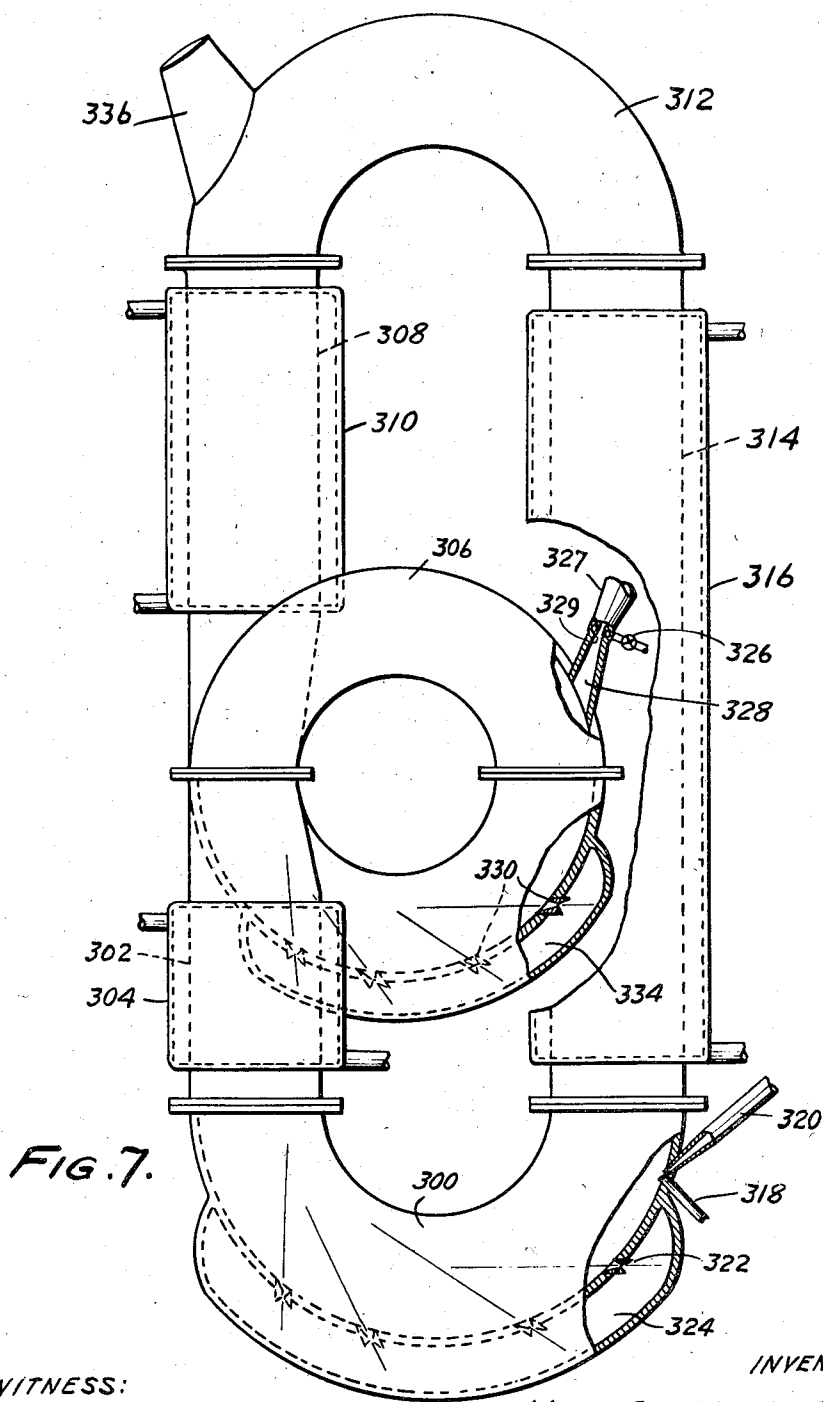


FIG. 7.

WITNESS:

Robert P. Kitchel

INVENTOR

Nicholas N. Stephanoff

BY *Bessie R. Harding*
ATTORNEYS.

Feb. 1, 1949.

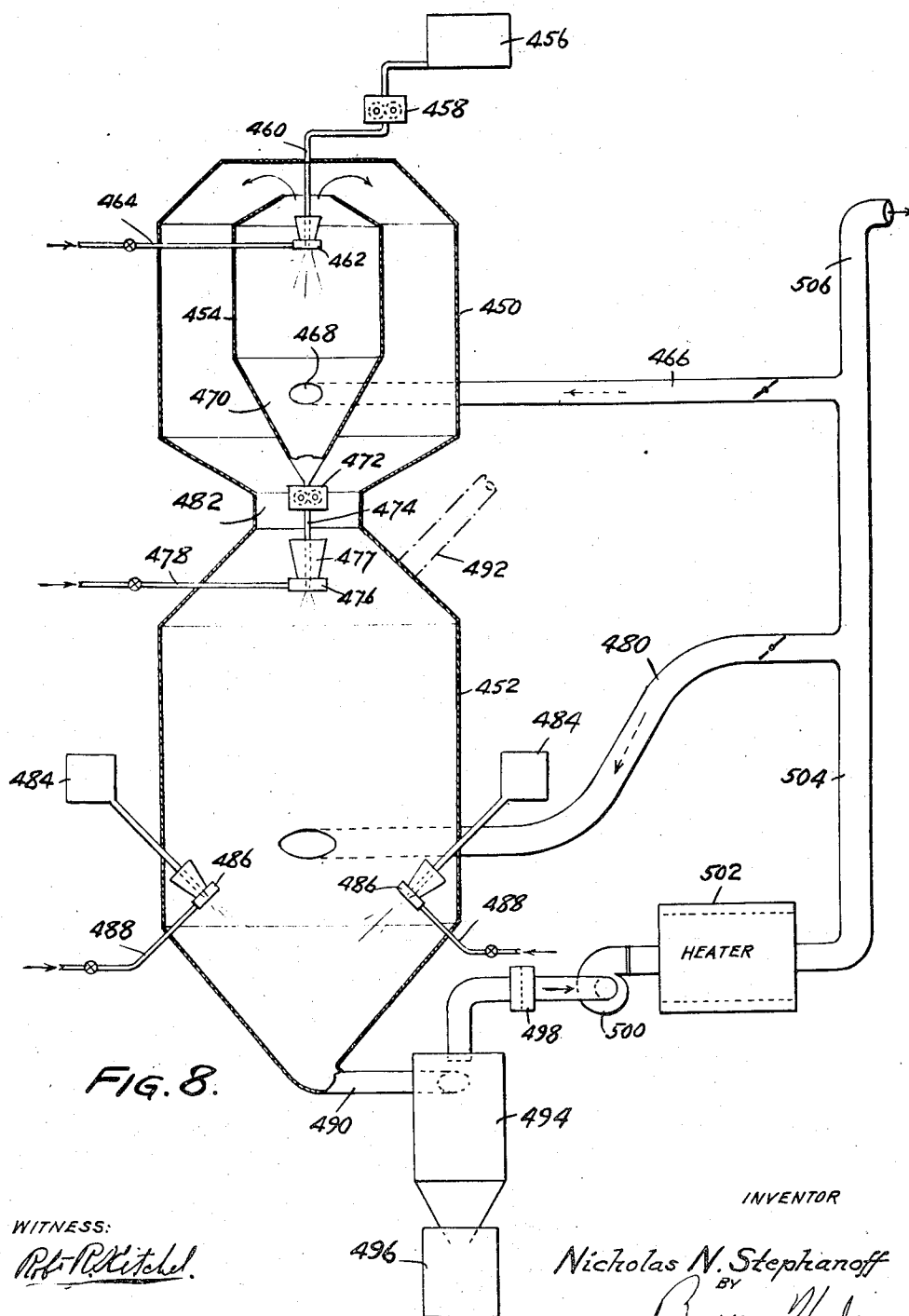
N. N. STEPHANOFF

2,460,546

METHOD AND APPARATUS FOR TREATING MATERIALS

Filed Oct. 1, 1942

7 Sheets-Sheet 7



WITNESS:

R. P. Mitchell

INVENTOR

Nicholas N. Stephanoff
BY
Bussard & Hardy
ATTORNEYS.

UNITED STATES PATENT OFFICE

2,460,546

METHOD AND APPARATUS FOR TREATING MATERIALS

Nicholas N. Stephanoff, Elkton, Md., assignor, by mesne assignments, to C. H. Wheeler Manufacturing Company, a corporation of Pennsylvania

Application October 1, 1942, Serial No. 460,401

32 Claims. (Cl. 260—95)

1

This invention relates to a method and apparatus for treating materials, and particularly to the treatment of materials in the form of droplets or particles to effect the production of solid or semi-solid materials by drying, chemical reaction, physical admixture, coating or the like.

This application is in part a continuation of my applications Serial No. 199,687, filed April 2, 1938, now Patent 2,297,726, issued October 6, 1942, Serial No. 235,139, filed October 15, 1938, now Patent 2,325,080, issued July 27, 1943; and Serial No. 320,788, filed February 26, 1940, now Patent 2,413,420, issued December 31, 1946.

As pointed out in said prior applications, drying in a broad sense may be effected by the atomization of material in high velocity gas or vapor jets. The drying referred to may comprise the mere evaporation of liquid from droplets of a solution or from wet solid or semi-solid particles, or the chilling of droplets of molten material, or may involve the production of solid or semi-solid materials by the chemical decomposition of the material with liberation of volatile breakdown products or constituents, or the polymerization or condensation of liquid materials to form solid or semi-solid products, or may involve the chemical reaction of materials in solution or accompanied by volatile liquids with removal of such volatile liquids and possibly volatile materials produced in the chemical action, or may involve such other operations as result in the ultimate production of solid or semi-solid material in a fine state from a material which is initially of a liquid or semi-solid flowing nature. These various matters will be apparent from the descriptions embodied in said prior applications. The present invention is particularly concerned with those phases of drying involving the production of chemical reactions and admixture or coating of fine particles, generally for the production of more or less homogeneous solid or semi-solid products having the particles thereof in a fine state of subdivision. For example, a material undergoing drying in a broad sense, grinding and/or heating, may be reacted with a gas included in or forming an atmosphere into which it is directed or, in fact, with the gas which may be used in whole or in part for its drying and comminution. Alternatively, reactions may be secured between two non-gaseous substances by their intimate admixture in finely comminuted state. Specifically, in accordance with the present invention, a single or a plurality of materials in suspension or solution in a liquid or even in a moderately finely powered dry state

2

may be projected in finely comminuted form into a common zone wherein violent admixture is effected and reactions, including polymerizations or condensations, accomplished.

As will be evident from the following description, the invention is primarily concerned with the matter of providing proper comminution of one or more materials, and their maintenance in such conditions, at proper temperature or under subjection to radiant energy or the like to produce final products of comminuted nature. The particular reactions or admixtures involved are subject to enormous variations, and while the invention is particularly applicable to the formation of certain products as hereinafter described, it will become obvious that the invention is of quite general applicability.

It is, accordingly, the broad object of the invention to provide methods and apparatus for the accomplishment of the above described results. These and other objects of the invention, particularly relating to details of methods and apparatus, will become apparent from the following description, read in conjunction with the accompanying drawings, in which:

Figure 1 is a diagrammatic sectional view through one form of apparatus designed for carrying out the objects of the invention;

Figure 2 is a similar view of an alternative apparatus particularly designed for the handling of sticky materials or materials requiring a substantial time for the occurrence of reactions or drying, in a broad sense, to produce the final product;

Figure 3 is a fragmentary sectional view showing an alternative arrangement of the apparatus of Figure 2;

Figure 4 is a diagrammatic sectional view through still another form of apparatus provided in accordance with the invention;

Figure 5 is a section taken on the plane indicated at 5—5 in Figure 4;

Figure 6 is a similar view showing still another form of apparatus particularly designed for the accomplishment of grinding of one or more constituents of the material;

Figure 7 is an elevation, partly in section, showing a further form of apparatus designed to carry out the principles of the invention;

Figure 8 is a diagrammatic sectional view of another form of apparatus embodying the invention; and

Figure 9 is a diagrammatic view, partly in section, showing apparatus for the proportioning of materials which are to undergo chemical re-

action or which are to be physically admixed in definite proportions.

In the following description and claims, it will be understood that where the term "gas" or "air" is used it is generally to be regarded as synonymous with "elastic fluid," i. e., it includes the vapor state of a substance below its critical temperature. As pointed out in my Patent No. 2,297,726, evaporation of a liquid, such as water, may be carried out not only in a fixed gas such as air, but in a vapor, including the vapor of the liquid to be evaporated in a superheated or reduced pressure state, for example, steam. Vapors as well as fixed gases may also be used as reagents in producing chemical reactions, as described hereafter. Superheated steam is a thoroughly effective drying medium for materials wetted with water or other liquids, and, in fact, the desirable effects of distillation in steam may be used to produce low temperature drying of high boiling liquids which are immiscible with water. To simplify the description, reference may be made hereafter to specific gases or vapors with the understanding that the terms used are to be broadly construed. Where "drying" is referred to herein, it will be understood that there is included the transition from a liquid to a solid or semi-solid state, though that may not occur by evaporation of a liquid. For example, drying in this broad sense may occur by polymerization of a liquid, as the result of chemical reaction, or by chilling of a molten liquid. The term "wet" is also used in a broad sense to mean a material which has properties of adherence, i. e., a pure liquid, liquid mixtures, wetted solids, tacky semi-solids, or the like.

In order to make the nature of the invention clear, there will first be described various alternative types of apparatus for carrying out the improved methods forming the subject-matter of this application, whereupon there will be then discussed the application of the invention to particular materials and products, and more particularly to the production of finely divided powders designed to be molded for the formation of resins of thermosetting or thermoplastic types.

Referring first to Figure 1, there is illustrated therein an apparatus particularly desirable for the polymerization of various monomers to secure fine, moldable powders. The apparatus comprises a large shell, indicated at 2, providing a chamber wherein a chemical reaction may occur, the term "reaction" being herein used in the broad sense to include not only the interaction of two materials, but a decomposition, polymerization, depolymerization, or the like, of a single material. The shell or chamber 2 is provided with a conical upper end 4 with the vertex of which there communicates an outlet passage 6 leading to a centrifugal separator, diagrammatically indicated at 8, connected with a receiver 10 for fine particles separated therein from a carrying gas or vapor. The gas outlet from the separator is indicated at 12 and may communicate, in particular cases where such is desirable, with a cooler 14 in which condensation may occur, condensed liquid being withdrawn at 16. The gas which leaves the cooler 14 may then be recirculated by means of a blower 18 through conduits 20 and 22, controlled by valves 24 and 26, back to the shell 2, excess being vented through an outlet 28, which may be suitably controlled by a valve, as indicated. The upper conduit 20 communicates with a chest 30 from which the recirculated gas is introduced through nozzle openings 32 tangentially into an

intermediate portion of the shell 2 so as to rotate in a predetermined direction within said shell. (If the shell is of rectangular or other polygonal form the gas may be caused to flow across a plane face through edge slots.) Similarly, the lower conduit 22 communicates with a chest 34 from which gas is directed tangentially into the lower portion of the chamber, through nozzles 36, preferably to rotate in a direction opposite the direction of rotation of the gas entering from the chamber 30. Desirably this latter gas enters the chamber at the lower portion of a tapering extension thereof below which is located a receiver 38 for solid material.

The material subject to treatment may be contained within a vessel diagrammatically indicated at 40 in which it may be heated or cooled as desired by the use of a coil 42 through which there circulates a suitable heating or cooling medium. A valved outlet 44 at the bottom of the vessel 40 permits the draining thereof. The material in the form of a liquid (by which term there may be included a flowable state in general, even though flow may not occur except through the application of high pressure if the material is plastic in nature) may be pumped from the chamber 40 by means of a pump 46 through a passage 48 to be introduced into high velocity jets of gas issuing from a nozzle chamber 52 to which the gas in highly compressed form is introduced through a pipe 50. The particular nozzle arrangement used is subject to considerable variation and may take any of the various forms particularly set forth in my application Serial No. 320,788, referred to above. In general, the liquid material flows out of, or is extruded under pressure from, a tube or other device providing one or more openings, with or without previous admixture of gas, and is thereupon subjected to one or more high velocity jets of gas, desirably flowing at supersonic velocities, whereby it is finally dispersed into the atmosphere of gas below the dispersing nozzle. Such jets have in at least a portion thereof a velocity of flow at least equal to the velocity of sound in the fluid of the jet having the same pressure and temperature as said portion of the jet, and are involved in all of the modifications of the invention described herein, it being understood that such jets are referred to as high velocity jets. These conditions of dispersion may be the same as those described in said application and in general it is desirable to have the dispersing action so adjusted so as to secure very minute particles. In general, it is also desirable that the dispersing nozzles be so arranged in a tangential fashion to the axis of the assembly that rotation is imparted to the dispersion, preferably in a direction opposite the rotation of the surrounding atmosphere of gas issuing from the nozzles 32.

The nozzle ring 52 has opening thereinto a conduit 54 through which, to be mixed with and carry the dispersion there is introduced, under control of a valve 55, a suitable gas handled by a fan or pump 56 preceded or followed by a suitable conditioner 58 to which the gas is introduced through a passage 60. This conditioner may be a heater or cooler, depending upon the effects desired, and has a primary effect in maintaining the temperature of the particles of the dispersion at a desired value.

While the type of action effected is hereafter described in greater detail, the operation of the apparatus may be briefly described with reference to the polymerization of a material such as

5

styrene. In such case, the styrene, preferably in a partially polymerized form and, in fact, undergoing active polymerization in the chamber 40, may be fed through the conduit 48 to be dispersed by compressed air, steam or other gas having a temperature suitable to complete the polymerization. The auxiliary air or gas introduced at 54 should likewise be at a suitable temperature to accomplish the completion of the polymerization, while the gas introduced at 30 may also have its temperature suitably controlled, for example, through a heater located in the conduit 20, so that as the dispersed particles flow downwardly against the counter-current of upwardly flowing gas, the polymerization will be finished to the desired extent to obtain a molding powder suitable for introduction into molds. In order to avoid any sticking of the material to the apparatus, the gas introduced from the chest 34 is desirably cooled, with the result that particles of sufficiently large size not to be floated upwardly out of the apparatus will fall in the solid state into the receiver 38, the velocities of the gases being suitably adjusted by means of valves 24, 26 and 55 to insure, preferably, that the major amount of the material will enter the receiver. Very fine particles produced as the result of the dispersion and polymerization will emerge from the chamber 2 through the conduit 6 to be separated in the conventional centrifugal separator 8. Any styrene vapors will be condensed in cooler 14.

The average time of reaction to which the particles are subjected after atomization may be readily controlled by suitable adjustment of the velocities and quantities of the various gases entering the apparatus. As will be evident from the description of the flow, the counter-current and counter-rotational flows which occur will tend to maintain particles in a floating condition within the chamber to an extent determined by the relative upward, downward and various rotational velocities. At the same time, the temperature conditions of the particles are closely maintained within proper limits, and since the particles or droplets are very small, no portions thereof become either abnormally heated or abnormally cooled, so that a completely uniform product will result eliminating entirely the difficulties resulting in batch processes wherein an exothermic action in the mid portion of the material may overheat it due to the heat insulating action of the surrounding material. If desired, the shell may be insulated to eliminate undesirable effects of radiation of heat either into or out of the apparatus, depending upon its internal temperatures as contrasted with the temperature of the surrounding atmosphere. (Certain polymerizations, such as those of isobutylene or the like, require very low temperatures within the apparatus.)

The apparatus just described is of a simple type particularly designed for such instances as involve fairly rapid reaction with insurance of the production of a solid, non-sticky particle in a short space of time, so that no difficulties are involved in sticking of the material to the walls of the chamber from which the material is, however, substantially insulated by the flowing gas currents entering at 30 and 34, which centrifugally flow along the walls and so provide a dynamic barrier in the form of a vortex resisting the passage of moist particles or droplets to the walls, these particles, by their inertia, particularly if initially rotated in opposition to the gas

6

vortex, tending to remain in the central portion of the apparatus. The various nozzle openings in ring 52 should be so diverted as to cause the dispersion to be confined to the central portion of the apparatus.

The apparatus of Figure 2, however is designed for more difficult cases where a longer time is required for the production of solid particles which will not stick to the walls. In this apparatus there is provided a shell 62 in which are located a plurality of inner chambers, all desirably of circular cross section, indicated at 64 and 66, the latter taking the form of a cone as illustrated. Into the chamber 64 there is led through a conduit 68 and a diverging portion 70 a suitable gas which may be either inert or reactive, designed primarily for preventing contact of the particles with the walls of the apparatus. Below the conical chamber 66, and desirably concentric therewith, is a passage 72 joined to the chamber 66 by a circular series of nozzles 74 arranged to effect the flow of an annular portion of the gas in, for example, a clockwise direction as viewed from above, and as particularly illustrated. The material introduced at 78 to the dispersing nozzle 76 by means of pump 84 from a preliminary heating chamber 80 designed, for example, to be heated by a coil 82, is dispersed by high pressure gas entering at 86, the dispersing nozzle being again of the type illustrated in my application Serial No. 320,788, and provided with a conical approach 87 to the nozzle ring so that the dispersion is admixed with some of the gas entering at 68. Desirably this nozzle produces a dispersion rotating opposite the direction of rotation imparted to the gas by nozzles 74, i. e., in this case in a counter-clockwise direction. As a result of this arrangement, the gas flowing from the nozzles 74 provides an insulating dynamic barrier through which the dispersed particles will not penetrate to come in contact with the walls of the chamber. If the chamber were of great vertical height, however, the particles might well come into contact with it following diffusion into the flowing barrier, and so the height is limited to such extent that contact cannot occur. To insure that contact does not thereafter occur, there is located between the chambers 66 and 64 a further ring of nozzles 86, in this case designed to receive further portions of the gas entering at 68 and to impart to it a rotation which in this case will be counter-clockwise as viewed from above, thereby insulating from the upper portions of the walls of the chamber 64 the diffused or partially diffused portions of the dispersion and the gas passing through the nozzles 74. The velocities of flow may be so adjusted by suitable proportioning of the nozzle passages and their tangential components of direction that by the time the uppermost end of the chamber 64 is reached, the particles will not yet have an opportunity to come into contact with the walls. It will be obvious that this nested arrangement of chambers may be carried still further to produce as great a height as is desired through which the particles in dispersed form may be moving to insure completion of the reaction.

Beyond the upper end of the uppermost chamber, in this case 64, the dispersion meets a downwardly flowing stream of gas spirally proceeding from a chest 96 into which it is introduced tangentially by a conduit 94, the rotation in this case being desirably clockwise and so opposing the net rotation of the flow from the upper end of

64. The flow now proceeds outwardly below the upper portion 90 of the chamber 82, about which there are also located nozzles 92 designed to introduce, flowing in a counter-clockwise direction, additional gas from a chest 88. This last gas forms a boundary layer along the outer wall of the chamber 82 to provide a dynamic barrier preventing the dispersion from reaching this wall. By this time, however, substantially solid particles should be provided which will no longer adhere to the walls. If it is desired to effect the admixture of those particles with other materials, a series of dispersion nozzles 108 may be provided for dispersing, under the action of high pressure gas entering at 112, one or more materials introduced at 106 which may, for example, in the case of a plastic, consist of plasticizing, coloring or other materials to be dispersed and dried in admixture with the previously dispersed and now substantially solid material. In particular, where the resin at this location may be somewhat sticky, a material, ultimately to become a filler, may be dispersed to coat the resin particles, being dispersed from a sludge containing evaporable liquid or introduced directly as a fine, dry powder. These dispersing nozzles are also of the type indicated above desirably provided with conical entrances to the nozzle rings through which a substantial portion of the dispersion may be drawn by ejector effect. The final solid combination product will thereafter pass to the bottom of the apparatus to fall into the collector 98. If fine particles pass through the gas outlet 100, these may be separated in a separator 102 from which spent gas emerges at 104.

In Figure 3 there is illustrated a variation in the form of the innermost portion of the apparatus illustrated in Figure 2, indicating how a portion of the gas may be introduced in a counter-flow direction to the dispersion. In this figure, 114 is the equivalent of the entrance portion 70 in Figure 2, and the dispersion is produced in the dispersing nozzle arrangement indicated at 116. Some of the gas entering at 114 passes through the nozzles at 118 equivalent to the nozzles 74 in an upward direction and with a rotary component of motion opposing that of the dispersion. The gas next introduced, however, is caused to flow downwardly through nozzles 122, being introduced therethrough through the annular passage 120. The rotation provided by the nozzles 122 is opposite that produced by the nozzles 118 with a resulting substantial slowing down of the net upward flow of the dispersion and at the same time with the insulation thereof from the next upwardly extending portion 124 of the walls surrounding the dispersion. From that point on, the flow may be the same as described in the case of Figure 2.

In both the last described figures, the various annular arrays of nozzles may be provided by adjustable louvers the angles of which may be varied to secure the particular flow characteristics desired for particular materials and concentrations thereof. Such adjustments, as well as adjustments of quantities of flow and the like are desirable to insure proper periods of maintenance of the dispersion and conditions of flow thereof.

In Figures 4 and 5, there is illustrated still another form of apparatus embodying the principles of the invention. In this modification, there is provided an upper chamber 126, which may be jacketed, as indicated at 128, for cooling or heat-

ing purposes, below which is a second chamber 130, which may be jacketed for the same purposes, as indicated at 132. The upper portion of the chamber 126 may be conical in form, as indicated at 134, and surrounding it there may be provided suitable lamps 136 for the introduction of radiant energy into the reaction zone within the upper chamber. For example, visible light, infra red radiation or ultra violet radiation may thus be provided. If very intense infra red or heat radiation is desired, the upper end of the chamber may be heated by flames or combustion gases to any desired temperature, such expedient, as well as the use of lamps, being applicable to the apparatus of the various other modifications. At the upper end of the conical top portion 134 of chamber 126, is a chest 136 into which gas may be introduced tangentially through the passage 138 to provide a downwardly flowing vortex through the chamber 126. One or more materials may be introduced through valved connections 140 and 142 into a supply chamber 144 which may be suitably heated or cooled as desired by a medium flowing through a coil 146. In the event several materials are introduced, completed mixture may be effected through the use of a stirring means indicated at 148.

From the receptacle 144 the fluent material passes through a tube 150 which is jacketed for heating or cooling as indicated at 151. The material passing through the tube 150 is dispersed by means of a dispersing nozzle 152 of the type previously mentioned, the nozzle ring of which is provided with a cone 154 for the introduction, by ejector action of the nozzle jets, of surrounding gas which will thereby be admixed with the dispersion and will also surround the same. A second material may be introduced at 158 to be dispersed by the nozzle 160 also supplied with high pressure gas through the connection 164. This nozzle is also provided with a cone 162 for the entry of the surrounding gas. The axes of the two dispersing nozzles intersect so that the dispersions will be admixed within the chamber 126. Gas is provided through passage 166 and nozzle openings 168 to provide a vortical flow met by the downwardly flowing dispersion and serving to a substantial extent to provide a dynamic barrier keeping the dispersion away from the walls of the apparatus. Desirably, as indicated previously, the direction of rotary motion is opposite that imparted to the dispersions in the dispersing nozzles, though to secure extremely good and rapid mixing, the two dispersions may be desirably rotating in opposite directions, in which case the direction of rotation imparted to the gas entering through the openings 168 may be immaterial. As the dispersion and surrounding gas passes down through the chamber 130, the reaction or admixture will be completed and the final product will pass out the lower portion of the apparatus through the connection 180 to the separator 182 from which the exhaust gas emerges by way of the passage 183. In the event that it is desirable to add to the dispersion some further dispersed material, this material may be introduced at 172 to a third dispersing nozzle 174 receiving high pressure gas through pipe 178 and provided with a conical inlet 176 through which by ejector action a substantial portion of the dispersion may be drawn to be intimately admixed with the newly added material. Similarly, within the separator 182 there may be added further material through the connection 184, directing

it to the dispersing nozzle 186 receiving high pressure gas through connection 188 and provided with an inlet cone 190. As an example of the use of the apparatus of Figure 4, there may be cited the production of a mixed plastic composition to be obtained in the ultimate form of a molding powder. For example, a mixture of different partially polymerized materials may be contained in the receptacle 144, the separate materials being introduced at 140 and 142. Such a mixture may consist, for example, of partially polymerized styrene and partially polymerized divinyl benzene introduced separately through 140 and 142. This mixture, preferably undergoing active polymerization, reaches the nozzle 152 and is thereby dispersed. Simultaneously there may be introduced through the connection 158 a partially condensed phenol formaldehyde resin or the like, the condensation of which will be completed simultaneously with the completion of the polymerization within the chamber 126. The resulting mixture may then have, for example, a pigment added thereto in the form of a heavy sludge introduced at 178 and particularly if there is provided a considerable region below the dispersion nozzle 174, evaporation of the carrier liquid of the pigment may completely occur in the lower portion of the apparatus so that the pigment is intimately admixed with the mixed resin particles in a dry state. Thereafter, within the separator there may be dispersed, for example, a plasticizer, which may be desirably introduced in such condition as not to become completely dry and thereby serve to aid in precipitating from suspension ultra fine particles within the separator.

If it is desired to treat a molten metal or similar material in the form of a dispersion, a ribbon or rod thereof may be melted or vaporized in an electric arc and so introduced to a nozzle such as 152. It may be mentioned that in the apparatus of Fig. 4, as well as in the others herein disclosed, chemical reaction may be caused to occur between dispersed material and the dispersing gas as fluid, or other gas as fluid introduced (e. g. at 168) into the apparatus. For example, there may be cited the formation of pigment oxides, carbonates, or the like, of lead formed by disposal of molten lead in a suitable atmosphere of gas or vapor.

The modification of Figure 6 is desirable primarily under conditions where grinding and reactions such as those previously indicated are to take place simultaneously. In this apparatus, the chamber takes the form of an enlarged stack 192 having a conical lower portion 193 into which a dispersion is projected by means of nozzles 194 projecting high velocity jets across the ends of tubes 196 arranged to receive material in liquid form from one or more receptacles 198, jacketed as indicated at 200 for the purpose of heating or cooling. The nozzle arrangement here shown may be utilized in this form of apparatus, though there may be equally well utilized the improved types of nozzles described in said application Serial No. 320,788. In this modification, the receptacle 198 is shown as provided with means for causing flow under pressure of a quite viscous liquid. To this end the material is arranged to be introduced through a connection 202 containing a valve 204, while gas, such as air, under very high pressure, may be introduced at 206 under control of the valve 208. With this apparatus, liquid may be first introduced, valve 204 closed, and thereupon pressure exerted on a batch there-

of by opening the valve 208. Upon substantial exhaustion of this batch, the valve 208 may be closed and the valve 204 opened to admit a new batch of material, and the operation repeated.

Gas to act as a carrier and also to serve to heat or cool the dispersion may enter a heating or cooling device 212, as indicated at 210, a suitable heating or cooling medium being introduced at 218. A fan 214 may then direct the gas through connection 216 into the lower portion of the tower or stack, which may be jacketed as indicated at 220 to maintain a proper temperature condition therein.

If desired, grinding may be effected in the connection 216 by forming it with a throat 217, similar to a Venturi throat, into and along which high velocity gas jets are directed from nozzles fed from a chest 219, which jets entrain and serve to grind material introduced at 221, for example in the form of a sludge.

Entering the stack as indicated is a connection 226 which passes through the wall thereof and communicates with a vertical chamber 228 which may also be jacketed for heating or cooling purposes as indicated at 230. The lower end of the vertical chamber 228 is connected through a tapering pipe section with an arcuate tube 232 into which there may be introduced, for example, a solid material through a connection 234. Nozzles 236, fed with gas at very high pressure, produce high super acoustic velocity jets in the tube 232, serving to effect grinding of the material introduced at 234, this material being mixed with the material introduced at 196 and 221 in the lower portion of the stack. The material leaving the stack may pass therefrom at 222 to a receiver 224 conventionally indicated as a dust collecting bag, though it will be understood that any type of dust separator or the like may be utilized.

It will be evident that the recirculating arrangement between 226 and 232 may be omitted and grinding effected solely in 216.

The apparatus may, for example, be used for the introduction of filler into a thermoplastic material, the polymerization of which may be completed in the chamber 192. A solid, for example mineral, filler, introduced at 234 or 221 in the form of particles of fairly large size, either in dry or sludge form, will be partially ground as it passes through the high velocity jets emerging from the nozzles 236 or those within passage 216. If the stack 192 is sufficiently high, the large particles will be unable to reach the top thereof under moderate flow velocities of the gases therein, and such large particles will be recirculated through the connection 226 and portion 228 of the apparatus under the action of high velocity gas flow produced by the ejector action of the nozzles 236. By reason of the action of these nozzles, the volume of gas thus recirculated may greatly exceed that rising above the connection 226 and passing out of the upper end of the stack 192. As recirculation of the large particles occurs, grinding will occur with ultimately the escape from the stack in admixture with the thermoplastic material of only very fine particles of the filler or the like. The grinding action involved is essentially similar to that of the apparatus described in my Patent No. 2,325,080.

In Figure 7 there is illustrated still another form of the apparatus designed particularly for the admixture of materials both of which are subject to intense grinding action in addition to

drying or chemical reaction. The apparatus of this figure comprises a lower tubular bend 300 communicating with a vertical tube 302 which may be jacketed as indicated at 304 for the purpose of heating or cooling its interior.

Beyond 302 there is a tube 306 in the form of approximately a single helical turn communicating with a further upright turn 308 which may also be jacketed as indicated at 310. A top tube turn 312 connects 308 with a vertical tube 314, which in turn communicates with the bottom portion 300. The tube 314 may be jacketed as indicated at 316. Material to be dispersed is introduced into the lower tube turn through the connection 318, from which it is ejected and dispersed by means of the high velocity jet from a nozzle 320. The material thus dispersed is subjected to the grinding action of jets from nozzles 322 fed by high pressure gas from a chest 324. The dispersion of this first introduced material upon entrance into the tube section 306, meets a second material introduced at 327 into the throat 329 of a nozzle 328, which is fed with high pressure gas through 326 to produce a dispersion of the second material, which will be intimately admixed with that of the first material, the two materials being then subject to simultaneous grinding under the action of the high velocity jets issuing from nozzles 330 fed with high pressure gas from the chest 334. It will be evident that the grinding or dispersing effect of these nozzles may be accompanied by drying and by chemical reaction or the like, thereby producing either a single compound if the two materials are introduced in proportions for complete reaction or suitable admixtures of various substances, either the original ones or reaction products if complete reaction does not occur. The final finely divided product flows from the apparatus in suspension through the connection 336, from which it may pass to a suitable separator or to a point of further processing.

In Figure 8 there is illustrated another form of apparatus capable of general use in promoting chemical reactions, but also of particular use for securing collectible particles obtained by evaporation of liquid from dilute solutions or suspensions of small percentages of solid materials. If in the various types of apparatus disclosed in my prior applications referred to above, drying of a dilute solution or a low percentage suspension is effected, as for example in the case of fruit or vegetable juices, it is sometimes found that after evaporation is completed, the remaining solid or substantially solid particles are so fine as to be extremely difficult to collect in ordinary collecting apparatus. This may be readily understood when it is considered that the original droplets formed by jet action may be of sizes of the order of a micron or less, and if the solid material therein forms only a small percentage of the original material, the size of the ultimate dry particles may be only a small fraction of a micron. The apparatus of Figure 8 is well adapted for the purpose of insuring the production of dried particles of sufficient size to be collectible.

The apparatus comprises an upper chamber 450 and a lower chamber 452 joined by a neck portion 482 of relatively restricted size. Within the upper chamber 450 there is located an inner chamber 454 which may take, essentially, the form of a dryer such as disclosed herein. The material to be dried is fed from a supply 456 by means of a pump 458 to the region of the high velocity jets produced by nozzles at 462, the nozzle structure

being the same as that heretofore described, and supplied with elastic fluid through the pipe 464. The fine dispersion thus produced within the chamber 454 meets an upwardly flowing current of air entering the tangentially conical portion 470 of chamber 454 at 468, whereby a swirling action of drying air produces evaporation of the liquid in usual fashion. However, in this case, it is desirable to so adjust the amount of air entering at 468 with respect to its quality, i. e., its temperature and humidity, that complete drying of the droplets is not effected, with the result that the larger droplets will collect in the bottom of the cone 470 as a concentrated solution, while some of the finer droplets, which may be completely dried to a form of a fine dust, will escape from the upper portion of the casing 454, thereupon flowing through the annular space between 450 and 454 and through the neck 482 of the apparatus.

The concentrated liquid collecting in the lower portion of the cone 470 will now contain a substantially higher proportion of solids than the original material, and is pumped by means of a pump 472 to the region of the high velocity jets of another nozzle assembly 476, also of the type previously described, and fed with elastic fluid through the pipe 478. As indicated, this nozzle assembly is provided with an approach cone 477 through which, by ejector action, there will be drawn all or a very large percentage of the elastic fluid containing fine particles in suspension passing through the neck 482. As a result, the fine particles which are so carried are intimately mixed with the atomized suspension produced below the assembly 476. The suspension of solid particles which may pass outside the cone 477 will also meet the suspension of droplets before evaporation is completed. The droplets themselves now have a sufficiently high concentration of solid material therein that upon evaporation the solid particles will be of sufficiently large size both in themselves and by possible agglomeration with the previously formed fine solid particles, that they may be readily collected in a collecting apparatus, the drying being in this case completed through the entry of drying air in spiral fashion from tube 480.

If drying alone is to be effected, the suspension of dried particles may pass directly to a collector 494 either through a lower outlet 490 or through an upper outlet at the position indicated in chain lines at 492. In the collector separation occurs and the solid material will drop into a receiver 496.

The remaining air containing the vapors produced by evaporation may be passed through a filter 498 of any suitable type to remove the last traces of dry dust, whereupon recirculation may take place by means of pump 500 through a heater 502, which delivers the heated mixture through conduit 504 for distribution through passages 466 and 480, controlled by suitable valves, to the respective chambers 454 and 452. The excess air and vapor may be bled off at 506. By reason of this recirculating arrangement, it will be evident that the atmosphere used for the drying comprises both air and the vapors of the evaporated solvent or suspending liquid. If these vapors are superheated, they are quite as effective as air in promoting evaporation. In fact, it will be evident that, using no air at all, superheated steam may be solely utilized for evaporating purposes, being introduced at 464 and 478 and, after the apparatus is running for a short

time, completely filling the circulatory system, since the air will have been completely bled off through 506.

If instead of mere evaporation, it is desired to effect coating or admixture, the materials required for this purpose may be fed from supplies 484 through nozzles 486 supplied with elastic fluid through connections 488. The suspensions produced thereby are mixed with the suspension produced by the nozzle 476 in precisely the same fashion as described above in connection with the other modifications.

While evaporation has been referred to as effected by the nozzles 462 and 476, it will be evident that polymerization or condensation may be equally well effected with the particular result that the particles of the product may be built up to a desired size if by a single separating operation they would be too small. In such case, the first separating may produce some completely polymerized or condensed particles, while the remaining material may be transformed into a very viscous state which, upon the second dispersion, will form relatively large droplets.

In describing the above apparatus, mention has been made of admixture of single materials in suitable proportions either to secure a desirable physical admixture or for the purpose of providing chemical reactions. For this purpose the materials should be fed in properly related amounts continuously. The apparatus of Figure 9 is designed for this purpose.

In this figure there is indicated at 382 a shaft driven at a suitable high rate of speed and connected to discs 384 and 386 which carry radially adjustable crank pins 388 and 390 desirably in the same phase relationships, though this phase may be desirably adjustable as indicated hereafter. These pins operate in cross-heads 392 and 394, respectively, carried by plungers 396 and 398, which at their lower ends are reduced to provide pistons 400 and 402 working in cylinders 404 and 406. These cylinders receive, respectively, through connections including check valves 408 and 410, materials from supply tanks 416 and 418. If highly viscous materials are being handled, gas pressures may be maintained on the materials in these tanks through the medium of connections 420 and 422. In such case, the rate of feed may be controlled by control of the pressures, as indicated by suitable gauges, to insure that on the up stroke of each piston the corresponding cylinder will be filled with material and not have therein spaces in which may exist partial vacuum. Stirring means may be present in tanks 416 and 418 to maintain uniform suspensions and admixtures therein.

The cylinders discharge through connections 424 and 426 containing discharge check valves 412 and 414 (sufficiently resisting direct passage of material due to pressure in tanks 416 and 418) into containers 425 and 427 in the nature of air domes to smooth out the fluctuations, and from these cylinders there extend connecting tubes 421 and 423 to the material feeds or nozzle assemblies such, for example, as 76 and 108 of Figure 2, 140, 142, 160 and 174 of Figure 4 or 318 and 326 of Figure 7.

By the use of this apparatus and the proper adjustment of crank pins 388 and 390 radially, and with a suitable high velocity of rotation of the shaft 382, coupled with small size of cylinders 404 and 406, there can be insured a carefully controlled delivery of proportionate amounts of ma-

terials through the assemblies, the amounts being so proportioned as to secure the desired reaction. Substantially continuous streams of materials in finely suspended form will thereupon issue from the nozzle assemblies at an accurately predetermined rate in the case of each to insure complete reaction in the limited zone afforded by the flow through the apparatus. If materials of different viscosities are fed, then to insure simultaneous delivery of portions corresponding to strokes of the pistons, it may be desirable to adjust the phase relationship of the crank pins because of slight lags occurring in passage of the more viscous material to its nozzle assembly due to elastic effects in the feed line.

Instead of using an arrangement of this type, it is also possible to use piston displacement screw pumps or gear pumps suitably geared together to secure corresponding rates of feed.

As will be evident from the general and brief discussions of the various forms of apparatus above, these are applicable to numerous treatments of materials and particularly to the formation of resins and resin compositions.

As an example of the application of the invention to the formation of thermoplastic type resins or artificial rubbers, there may be cited the polymerization of styrene to form a fine moldable powder, sufficiently fine to enter every crevice of a mold and produce an exact reproduction of its interior. The starting product in this case may be either unpolymerized styrene or partially polymerized styrene, by which term would be included either the product directly resulting from partial polymerization of styrene or the product obtained by dissolving fully polymerized styrene or partially polymerized styrene in either unpolymerized styrene or some partially polymerized form thereof. Generally speaking, it is advantageous to start with a quite viscous liquid of this sort still capable of being fed to the apparatus, but of such nature as to reduce to a minimum the actual degree of polymerization to be effected within the spraying apparatus. While the polymerization may take place in the absence of a catalyst in well known fashion, the viscous starting liquid may contain a polymerization catalyst present throughout a preliminary partial polymerizing reaction, or there may be added a polymerizing catalyst just prior to the entrance of liquid into the apparatus. Such catalysts are well known and exemplified by benzoyl peroxide.

If the initial material has been made up in batches and stored temporarily prior to entrance into the spraying apparatus, it is desirable to raise it to the temperature of beginning of polymerization as it approaches the spray nozzles, so that, desirably, active polymerization is taking place at the point where it reaches the nozzles. As the viscous material enters the region of the gas jets, it will be immediately broken up into extremely minute droplets. Each of these droplets will be surrounded by, and in intimate turbulent contact with, the gas which is used for the dispersion. The temperature of this gas should be carefully controlled to a proper degree to promote very active polymerization, but nevertheless, due to the intimate contact of the droplets with the gas, it is impossible for any of them to become over-heated, with the result that the polymerization takes place substantially isothermally despite the fact that the polymerization is exothermic in character. The ratio of gas to the polymerizing material, of course, determines

the net rise of temperature which would tend to occur, but this may be readily controlled by causing the dispersion to enter an upwardly flowing current of cooler air or gas in counter-flow fashion, this gas preferably having a vortical motion, so that substantially the isothermal conditions referred to above exist until the polymerization is completed. Thereafter, as the particles now fully polymerized enter the upwardly flowing gas or air which has just entered the apparatus and which is substantially cool, the temperature drops below any softening point (which may range from about 65° C. to 90° C. for polystyrene) so that the final solid particles are hard and will not tend to adhere to each other or to any walls of the apparatus with which they come into contact. The fine particles flow from the apparatus in suspension to a dust separator or collector from which the final molding powder may be removed. It will be evident that the types of apparatus of Figures 1, 2, 3 and 4 are particularly suited for this process.

The operations just described in connection with styrene are applicable to numerous other polymerizable materials which have polymerizing and final characteristics similar to styrene. Among such substances there may be cited the various methacrylates, methacrylonitrile, substituted styrenes, divinyl benzene and its substituted derivatives, vinyl esters such as vinyl acetate, vinyl hydrocarbons and derivatives, such as vinyl chlorobenzene and vinyl naphthalene, vinyl alcohols and ketones, ethyl methylene malonate, dimethyl itaconate and dimethyl acetylene. Mixtures of these substances or their partially polymerized products with each other may be used as starting substances, for example, a quite desirable molding powder is producible by the admixture of styrene and divinyl benzene in widely varying proportions with the production of products having properties ranging from those of polystyrene to those of the polymer of divinyl benzene.

To the initial material may also be added, in the fashions indicated above, coloring materials, lubricants, fillers, plasticizers, stabilizers (antioxidants) and the like, depending upon the desired final product. The types of materials which may be used and particular temperatures and types of treatments thereof are well known in the art, as already set forth, for example (in connection with specific procedures which in themselves are irrelevant to the present invention) in the patents to Fields Nos. 2,057,673 and 2,057,674, dated October 20, 1935. See also "Plastics in Engineering," by Delmonte, second edition, Penton Publishing Company, for such matters as conditions of reactions, catalysts, light, etc., to be used in applying the present invention to numerous materials, e. g. butadiene, chloroprene, olefine polysulphides, isobutylene, etc.

Instead of starting with the partially polymerized material of the type indicated above, the starting point of the procedure, in the event that polymerization has been completed to form the material in large masses, may be a solution of the fully polymerized material and/or partially polymerized material in a volatile solvent, such as, in the case of polystyrene, various esters or aromatic hydrocarbons, or in the case of acrylate type resins, ketones, esters, and aromatic hydrocarbons. In the initial formation of the polymer in such cases, no particular care need be exercised to eliminate gas bubbles or other cloudiness, since by introducing a solution into the appa-

ratus, dispersing the same with evaporation of solid, and collection of the micronic particles, the final powder may be molded to produce a completely clear and transparent product if that is desired. In this case of the use of solvents, which may vary depending upon the particular resin being handled, mixtures of various resins may be used as well as mixtures of one or more resins with fillers, pigments, coloring materials, plasticizers or the like.

In the preceding discussion, the formation of particles of polymerized resins has been primarily stressed, and the application of the invention to such substances involves generally the two phases of the polymerization reaction and the micronic reduction occurring simultaneously. But the production of micronic moldable particles of non-adherent character need not involve a chemical reaction at all as is, in fact, exemplified above by the treatment of already polymerized materials in solution. This matter of spraying a solution to disperse droplets, secure instantaneous drying, and separate dry and non-adherent particles as a powder may be applied not only to fully polymerized materials (or those polymerized to such extent as to be definitely hard and non-adherent) but to plastic materials such as those comprising cellulose esters, for example, the nitrates or acetates, rubber derivatives, natural resins (such as shellac, copal, casein, zein, or the like), etc., which may be sprayed in the form of solutions in solvents to secure end products in the form of powders. Or any of the types of materials mentioned which melt or soften without decomposition may be dispersed by the high velocity jets to form molten or softened globules which upon cooling in the cold gas currents or cold gas from dispersing jets will form fine particles which will no longer adhere together. In the case of artificial rubber-like products, the particles may be permitted to adhere in suitable collectors, the gummy product being periodically scraped therefrom; for example, the lower portion of the shell of Figure 2 may be provided with rotating scrapers.

In the case of thermosetting resins, for example those of the phenol-formaldehyde, urea-formaldehyde, glyceryl-phthalate, or melamine-formaldehyde types, the practice of the improved methods resembles to a considerable extent what is described above in connection with polymerization and simultaneous dispersion. In this case, however, what is dispersed is a mixture of the reagents, including suitable catalysts, while they are in an incompletely reacted state in such fashion that the reaction is completed during the period of dispersion to form the moldable stage powder in dispersed condition so that the latter is recovered in the form of a fine powder or non-adherent granules; for example in the formation of a phenol formaldehyde molding powder, the mixed reagents, together with water, and containing ammonia or hexamethylenetetramine as catalyst, may be preliminarily brought to a stage of partial condensation, but only to such extent that the product is still sufficiently fluent to be extruded under pressure into the region of the high velocity jets. At the temperature of the air or other gas surrounding the minutely dispersed droplets, the condensation will be completed while simultaneously the water and catalyst, as well as the excess of either of the reagents, will be vaporized so that the powder in a dry non-adherent physical form, and chemically in the moldable stage of condensation, will separate

17

out. A similar reaction can be caused to take place to form a urea-formaldehyde molding powder or molding powders of other similar types produced by condensation reactions.

Where one of the reagents is a gas, for example the formaldehyde in the formation of a phenol formaldehyde or urea formaldehyde resin, the formaldehyde gas, or other gas in corresponding cases, may be used as the dispersing gas either alone or in admixture with air or some other inert material. In fact, the phenol may also be vaporized and the reaction may occur between the phenol formaldehyde vapors with no liquid droplets introduced into the apparatus as such, these being formed by way of a mist in the reaction chamber as condensation occurs.

Other materials, as mentioned above, such as pigments, lubricants, plasticizers, coloring materials, fillers, or the like, may be incorporated in the reacting mixture or may be dispersed in the apparatus to form a uniform homogeneous mixture with the final particles or to coat the latter. In the case of the phenol-formaldehyde resin and urea-formaldehyde resin, the moldable stages thereof are not particularly soluble in solvents, and hence it is not so feasible to produce the powders by evaporation of solvents as in the cases mentioned above of the thermoplastic resins. However, in the partially condensed states, these resins and other similar thermosetting resins are soluble at least to a limited extent, and it is possible, therefore, to recover the molding powders from solutions as described above.

While admixture of the resins with pigments, etc., has been stressed, it will also be evident that various resins may be mixed to form powders of homogeneous compositions. For example, the reagents condensable to form a urea-formaldehyde resin or a phenol-formaldehyde resin may be mixed with a partially polymerized styrene, vinyl acetate, methacrylate, or the like and simultaneously disperse in which case condensation and polymerization will occur simultaneously in the dispersed condition giving a homogeneous powder of a combination thermoplastic-thermosetting nature. The initial mixture in such case may be an emulsion produced by conventional emulsifying procedure or the mixture may be formed by the intermixture of two dispersions formed by adjacent nozzles and subject to turbulence and flow paths to insure homogeneity as in the apparatus of Figure 4. It may be pointed out that the exothermic heat of polymerization of the styrene or the like may be used to provide the heat necessary for starting the condensation reaction, control being exercised thereafter by introduction of air or other gas at proper temperature to prevent the reaction's getting out of control which would tend to carry the thermosetting resin beyond the moldable stage. However, the thermosetting resin may be carried to a final stage so that it becomes, essentially, an inert filler of a thermoplastic resin whose plastic constituent is a polymer.

The reactions described above are typical of a broader class of quite general character. The speed of chemical reaction is dependent largely upon the surface contact of reacting materials, particularly in organic reactions, which are frequently very slow when occurring between liquids, liquids and solids, or solids and solids in solutions or suspensions. If such materials are finely dispersed, and, in such state, admixed, or alternatively, partially or completely admixed and then immediately dispersed, the reactions are greatly

18

accelerated. The speeding up of reactions, however, is not the sole advantage. If a reaction, for example between two salts, results in the formation of a precipitate, the final product may only be secured from a reaction in solution through the medium of filtration, washing and drying, and if a finely comminuted product is required, this drying is generally necessarily followed by grinding because, in the precipitation in solution, and in the filtration, agglomeration occurs. But if the materials are brought together in finely comminuted form while wet (either in solution or suspension) the reactions will take place with the formation of products in finely comminuted form. If drying then occurs, a fine powder is produced which, unlike precipitates, even if thereafter wetted, will not agglomerate. Since agglomeration is a matter of time, it is possible usually to achieve similar results by causing reaction to occur and then, before agglomeration may happen, dispersing the product. If there is produced in this reaction no material which need be washed from the solid product, the result is the direct production of an extremely fine powder. If, on the other hand, a soluble salt remains which must be washed out, the dried powder can be subjected to washing and can then be filtered, washed and dried, generally without further agglomeration, since it has already passed into a stable physical state, non-conducive to the further growth of the particles. Such a wet washed powder can be dried by a subsequent operation in the apparatus illustrated.

Generally, in reactions in which one material is not a gas, it is necessary for economy, if not for the obtaining of a desired final product, that the reacting materials be fed in rather closely related proportions. These proportions need not necessarily be chemical equivalents, but may involve predetermined excesses of one or more materials to secure most effective reaction in accordance with the law of mass action. In conventional batch processes or even continuous processes in which the time of reaction is indefinitely long and thorough intermixture may be leisurely caused to occur, it is sufficient that the materials be measured out in desired proportions and mixed together either at one time or progressively. In the described apparatus, however, it will be evident that a particular small amount of material passing from one nozzle assembly will be completely out of the reaction zone in a time of the order of a fraction of a second to not more than a few seconds, and hence it is necessary to feed the materials continuously in continuously closely regulated proportions to insure that the reaction will be completed or have proceeded to the desired extent before drying occurs and, at any rate, while the materials are in suspension, i. e., before they come to a condition in which agglomeration can occur in a separator or collector. To this end there is provided the proportioning apparatus illustrated in Figure 9.

As an example of the type of chemical reaction which may be produced, there may be mentioned the production of lithopone by the spraying into a common reaction zone of an aqueous paste of zinc sulphate and an aqueous paste of barium sulphide. In the feeding of these materials, stirring may be used to maintain the material fed of uniform composition and adjustment of feeding means such as that of Figure 8 made upon analysis of the materials to insure their feed in equivalent quantities. The reaction between the

two constituents will take place with great rapidity, in view of the large surfaces offered for reaction by the droplets or particles, and the result will be a dry cloud of fine particles composed of zinc sulphide and barium sulphate. This cloud may be passed through a calcining zone provided either in a separate apparatus or by the introduction of sufficiently hot gases, for example, in the bottom of the apparatus of the type of Figure 1. If chilling of the particles is desired, large quantities of air at ordinary temperature may be admixed with the suspension prior to its reaching the separator. It will be evident that the reaction may take place in inert gas or in a reducing gas if the temperatures used are such that detrimental oxidation might possibly take place in air. In the case of chemical reactions, not only can there be removed by evaporation liquid solvent, but there may also be removed volatile solid products of a reaction if the temperature required is not too high to cause damage to the other particles. For example, in the precipitation of chemical bases by the use of ammonium hydroxide, the resulting ammonium salt may be volatilized together with the water used for solution or suspension and the base in a dry form and free of ammonium salt recovered. In such case, the volatilizing temperature must be maintained through the dust collector, and the spent vapors may be frictionally or wholly condensed to recover material of value such as, in the example just mentioned, ammonium salts. Evaporation or volatilization of products of many reactions will cause them to approach substantial completion according to the law of mass action.

In using the apparatus of Figure 4 for accomplishing a chemical reaction, the proportioned amounts of materials may be introduced to the nozzles 152 and 160. Generally speaking, the materials will be initially moist with aqueous or other suspending liquid or solvent though, of course, either or both may be completely in solution. As the reaction proceeds, evaporation of the solid or suspending liquid may be caused to occur simultaneously by the introduction of hot air or other gas at 168, the evaporation being substantially complete before the suspended material reaches the bottom of the apparatus. Alternatively, in the form of apparatus illustrated in Figure 1, a pair of nozzles such as 152 and 160 may be provided in which case a vortical flow of gas may meet the downwardly moving suspension to carry the final particles outwardly through the top of the apparatus. Quite heavy particles may reach the receiver 38.

Not only chemical reactions but physical admixture or coating and quasi-chemical reactions may be produced. For example, lakes may be formed by spraying together a metallic base and a dye solution, the resulting pigment in a fine state resulting directly as a product. Or particles intended to form the disperse phase of an emulsion may be coated with a dispersing agent, such as a soap, to produce a fine powder which forms an emulsion directly upon introduction into a liquid.

As further examples of coating operations which may be effected, there may be cited coating filler particles with small amounts of plastic materials, coating pigments or dyes with salts or the like to prevent adherence of particles and more ready dispersion or solution, the coating of asphalt particles with powders to avoid cohesion or with glazes to produce essentially solid particles, the coating of fine materials to prevent adherence of

particles or the coating of fine materials with emulsifying agents, wetting out agents or the like.

For the purpose of coating, the apparatus of the type illustrated in Figure 7 is advantageous, particularly when considerable grinding is desired in the case of both materials. This apparatus is also designed for the staging of operations such as, for example, concentration of a solution followed by sudden cooling to produce crystallization whereupon further evaporating action of the carrying fluid will dry the crystals.

It may be pointed out that the simultaneous feeding of two materials into an apparatus with or without successive drying or grinding is conducive to the formation of homogeneous mixtures achievable in batch operations only through a lengthy mixing procedure. For example, if a plastic molding powder is to be mixed with a filler, it is very difficult to prevent stratification in batch mixing, which would result in local regions of substantial size being deficient in one or the other of the constituents. In accordance with the procedures herein outlined, homogeneity is a readily attainable result. In the specific case of the apparatus of Figure 7, a dry material may be introduced at 320 and ground in the lower portion of the apparatus with the production of a fine suspension of the material. When this material reaches the region 306, a different material may be introduced in at least a partially wet state, for example in suspension in liquid, though it may be in solution. The mist produced by the second dispersion will coat the fine particles resulting from the grinding to produce a dry material suitably coated. For example, if the first material is of a hygroscopic nature, a protective coating may be thus provided so that the first material may be kept in a moist atmosphere.

This matter of securing homogeneity of a composition containing fine particles is of particular importance in the case of plastic-filler compositions. If filler particles stick together they may form a weak region, deficient in the binding plastic. No amount of stirring will ordinarily break up such an agglomeration of particles. In proceeding according to this invention, however, not only may complete homogeneity be readily attained, but by successive coating there may be built up what amount to adherent structural units of minute size. For example, a filler material may be ground and presented in suspension to a dispersion of pigment which will settle on, or at any rate, depending upon, the relative state of subdivision, will intimately and uniformly mix with, the filler particles. This combination in turn, still in suspension, may be sprayed with a thermoplastic or thermosetting binder in the form of molten droplets or in solution in a volatile solvent, and the new, and again completely homogeneous mixture, of which each particle is coated, passed through a cooling or solvent-evaporating zone in which, by freezing or drying, the binder is caused to leave a solid coating on each particle. Finally, any desired admixture may be made with a suspension of dust, lubricant, plasticizer, coloring material or the like, with further evaporation if necessary and final separation and collection of the particles. Thus a completely homogeneous moulding powder may be obtained. Such successive coatings may be accomplished in any of the various types of apparatus described above.

What I claim and desire to protect by Letters Patent is:

1. Apparatus for the treatment of materials comprising a casing, means for dispersing mate-

rial in a high velocity elastic fluid jet having in at least a portion thereof a velocity of flow at least equal to the velocity of sound in the fluid of the jet having the same pressure and temperature as said portion of the jet, within said casing, and means for dispersing a second material into the first mentioned dispersion.

2. Apparatus for the treatment of materials comprising a casing, means for dispersing material in a high velocity elastic fluid jet having in at least a portion thereof a velocity of flow at least equal to the velocity of sound in the fluid of the jet having the same pressure and temperature as said portion of the jet, within said casing, means for maintaining said dispersion throughout flow through a substantial region of said casing by the introduction of further elastic fluid, and means for dispersing a second material into the first mentioned dispersion.

3. Apparatus for the treatment of materials comprising a casing, means for dispersing material in a high velocity elastic fluid jet having in at least a portion thereof a velocity of flow at least equal to the velocity of sound in the fluid of the jet having the same pressure and temperature as said portion of the jet, within said casing, means for maintaining said dispersion throughout flow through a substantial region of said casing by the introduction of further elastic fluid to flow in a spiral path, and means for dispersing a second material into the first mentioned dispersion.

4. Apparatus for the treatment of materials comprising a casing, means for dispersing material in a high velocity elastic fluid jet having in at least a portion thereof a velocity of flow at least equal to the velocity of sound in the fluid of the jet having the same pressure and temperature as said portion of the jet, within said casing, and means for producing along the walls of the casing a substantially smooth spiral flow of elastic fluid to prevent contact of the suspension with the casing walls.

5. Apparatus for the treatment of materials comprising a casing, means for dispersing material in a high velocity elastic fluid jet within said casing to produce a rotating dispersion, said jet having in at least a portion thereof a velocity of flow at least equal to the velocity of sound in the fluid of the jet having the same pressure and temperature as said portion of the jet, and means for producing along the walls of the casing a substantially smooth spiral flow of elastic fluid in the opposite direction of rotation to prevent contact of the suspension with the casing walls.

6. Apparatus for the treatment of materials comprising an upwardly diverging passage, means providing a flow of elastic fluid upwardly through said passage, means for introducing material to be dispersed into said passage, and means for providing therein at least one high velocity jet to affect the dispersion, said jet having in at least a portion thereof a velocity of flow at least equal to the velocity of sound in the fluid of the jet having the same pressure and temperature as said portion of the jet.

7. A method including passing elastic fluid into a receiver through a nozzle to produce a high velocity jet having in at least a portion thereof a velocity of flow at least equal to the velocity of sound in the fluid of the jet having the same pressure and temperature as said portion of the jet, introducing material into the jet to produce a fine suspension of said material,

passing an elastic fluid into a region containing said fine suspension through a second nozzle arranged to produce a high velocity jet having in at least a portion thereof a velocity of flow at least equal to the velocity of sound in the fluid of the jet having the same pressure and temperature as said portion of the jet, introducing into the second jet a different material to produce a fine suspension of the second material admixed with that of the first named material, and separating resulting material from the elastic fluid.

8. A method including passing elastic fluid into a receiver through a nozzle to produce a high velocity jet having in at least a portion thereof a velocity of flow at least equal to the velocity of sound in the fluid of the jet having the same pressure and temperature as said portion of the jet, introducing material into the jet to produce a fine suspension of said material, passing an elastic fluid into a region containing said fine suspension through a second nozzle arranged to produce a high velocity jet having in at least a portion thereof a velocity of flow at least equal to the velocity of sound in the fluid of the jet having the same pressure and temperature as said portion of the jet, introducing into the second jet a different material in at least a partially wet state to produce a fine suspension of the second material admixed with that of the first named material, and separating resulting dried material from the elastic fluid and vapor.

9. A method including passing elastic fluid into a receiver through a nozzle to produce a high velocity jet having in at least a portion thereof a velocity of flow at least equal to the velocity of sound in the fluid of the jet having the same pressure and temperature as said portion of the jet, introducing into the jet material to be dried to produce a fine suspension of said material in at least a partially dried state, passing an elastic fluid into a region containing said fine suspension through a second nozzle arranged to produce a high velocity jet having in at least a portion thereof a velocity of flow at least equal to the velocity of sound in the fluid of the jet having the same pressure and temperature as said portion of the jet, introducing into the second jet a different material to produce a fine suspension of the second material admixed with that of the first named material, and separating resulting material from the elastic fluid and vapor.

10. A method including passing elastic fluid into a receiver through a nozzle to produce a high velocity jet having in at least a portion thereof a velocity of flow at least equal to the velocity of sound in the fluid of the jet having the same pressure and temperature as said portion of the jet, introducing into the jet material to be dried to produce a fine suspension of said material in at least a partially dried state, passing an elastic fluid into a region containing said fine suspension through a second nozzle arranged to produce a high velocity jet having in at least a portion thereof a velocity of flow at least equal to the velocity of sound in the fluid of the jet having the same pressure and temperature as said portion of the jet, introducing into the second jet a different material in at least a partially wet state to produce a fine suspension of the second material admixed with that of the first named material, and separating resulting dried material from the elastic fluid and vapor.

11. A method including passing elastic fluid into a receiver through a nozzle to produce a

23

high velocity jet having in at least a portion thereof a velocity of flow at least equal to the velocity of sound in the fluid of the jet having the same pressure and temperature as said portion of the jet, introducing into the jet material, subject to chemical change under proper temperature conditions, to produce a fine suspension of said material, and maintaining said suspension in an elastic fluid atmosphere at a temperature at which chemical change of said material occurs for a sufficient period to effect said chemical change to a substantial degree.

12. A method including passing elastic fluid into a receiver through a nozzle to produce a high velocity jet having in at least a portion thereof a velocity of flow at least equal to the velocity of sound in the fluid of the jet having the same pressure and temperature as said portion of the jet, introducing into the jet material, having constituents capable of reaction under proper temperature conditions, to produce a fine suspension of said material, and maintaining said suspension in an elastic fluid atmosphere at a temperature at which reaction of constituents of said material occurs for a sufficient period to effect said reaction to a substantial degree.

13. A method including passing elastic fluid into a receiver through a nozzle to produce a high velocity jet having in at least a portion thereof a velocity of flow at least equal to the velocity of sound in the fluid of the jet having the same pressure and temperature as said portion of the jet, introducing polymerizable material into the jet to produce a fine suspension of said material, and maintaining said suspension in an elastic fluid atmosphere at a temperature at which polymerization of said material occurs for a sufficient period to effect substantial polymerization.

14. A method including passing elastic fluid into a receiver through a nozzle to produce a high velocity jet having in at least a portion thereof a velocity of flow at least equal to the velocity of sound in the fluid of the jet having the same pressure and temperature as said portion of the jet, introducing material containing condensable constituents into the jet to produce a fine suspension of said material, and maintaining said suspension in an elastic fluid atmosphere at a temperature at which condensation of constituents of said material occurs for a sufficient period to effect substantial condensation.

15. A method including passing elastic fluid into a receiver through nozzles to provide a plurality of high velocity jets each having in at least a portion thereof a velocity of flow at least equal to the velocity of sound in the fluid of the jet having the same pressure and temperature as said portion of the jet, introducing into the several jets materials capable of chemical reaction to produce a fine suspension of each and admixture of said suspensions, maintaining the resulting admixture under proper conditions and for a sufficient time to effect substantial reaction of the constituent materials, and separating at least one reaction product from the elastic fluid.

16. A method including passing elastic fluid into a receiver through nozzles to provide a plurality of high velocity jets each having in at least a portion thereof a velocity of flow at least equal to the velocity of sound in the fluid of the jet having the same pressure and temperature as said portion of the jet, introducing into the several jets materials capable of chemical reaction to produce a fine suspension of each and

24

admixture of said suspensions, at least one of said materials being in at least a partially wet state, maintaining the resulting admixture under proper conditions and for a sufficient time to effect substantial reaction of the constituent materials, and separating at least one reaction product from the elastic fluid.

17. A method including passing elastic fluid into a receiver through nozzles to provide a plurality of high velocity jets each having in at least a portion thereof a velocity of flow at least equal to the velocity of sound in the fluid of the jet having the same pressure and temperature as said portion of the jet, introducing into the several jets materials capable of chemical reaction to produce a fine suspension of each and admixture of said suspensions, maintaining the resulting admixture under proper conditions and for a sufficient time to effect substantial reaction of the constituent materials, and separating at least one reaction product in at least a partially dried state from the elastic fluid.

18. A method including passing elastic fluid into a receiver through nozzles to provide a plurality of high velocity jets each having in at least a portion thereof a velocity of flow at least equal to the velocity of sound in the fluid of the jet having the same pressure and temperature as said portion of the jet, introducing into the several jets materials to be admixed to produce a fine suspension of each and admixture of said suspensions, and separating non-gaseous material from the elastic fluid.

19. A method including passing elastic fluid into a receiver through a nozzle to produce a high velocity jet having in at least a portion thereof a velocity of flow at least equal to the velocity of sound in the fluid of the jet having the same pressure and temperature as said portion of the jet, introducing into the jet a material in at least a partially wet state to produce a fine suspension of the material subject to being at least partially dried by its suspending elastic fluid, and subjecting the suspension of the partially dried material to a second suspension of the material in at least a partially wet state whereby further drying effects the production of larger particles of partially dried material.

20. A method including passing elastic fluid into a receiver through a nozzle to produce a high velocity jet having in at least a portion thereof a velocity of flow at least equal to the velocity of sound in the fluid of the jet having the same pressure and temperature as said portion of the jet, introducing into the jet a material in wet state to produce a fine suspension of the material subject to being at least partially dried while suspended in the elastic fluid, separating from the suspension at least some residually wet material, and subjecting the last mentioned material to another jet action to produce a suspension thereof in admixture with at least part of the original suspension remaining after said separation.

21. A method of producing a resinous moulding powder including dispersing in a high velocity jet of elastic fluid, having in at least a portion thereof a velocity of flow at least equal to the velocity of sound in the fluid of the jet having the same pressure and temperature as said portion of the jet, a wet material which becomes at a proper temperature a substantially dry resinous material, said dispersion having a temperature at which the droplets thereof become substantially dry particles of said resinous material, and separating from the elastic fluid said substantially dry particles.

22. A method of producing a resinous moulding powder including dispersing in a high velocity jet of elastic fluid, having in at least a portion thereof a velocity of flow at least equal to the velocity of sound in the fluid of the jet having the same pressure and temperature as said portion of the jet, a wet material which, by evaporation of solvent, becomes a substantially dry resinous material, said dispersion having a temperature at which the droplets thereof become, by evaporation of solvent, substantially dry particles of said resinous material, and separating from the elastic fluid said substantially dry particles.

23. A method of producing a resinous moulding powder including dispersing in a high velocity jet of elastic fluid, having in at least a portion thereof a velocity of flow at least equal to the velocity of sound in the fluid of the jet having the same pressure and temperature as said portion of the jet, a molten material solidifiable to a substantially dry resinous material, said dispersion having a temperature at which the droplets thereof become, by solidification, substantially dry particles of said resinous material, and separating from the elastic fluid said substantially dry particles.

24. A method of producing a resinous moulding powder including dispersing in a high velocity jet of elastic fluid, having in at least a portion thereof a velocity of flow at least equal to the velocity of sound in the fluid of the jet having the same pressure and temperature as said portion of the jet, a polymerizable material, said dispersion having a temperature at which the droplets thereof become, by polymerization, substantially dry particles of said resinous material, and separating from the elastic fluid said substantially dry particles.

25. A method of producing a resinous moulding powder including dispersing in a high velocity jet of elastic fluid, having in at least a portion thereof a velocity of flow at least equal to the velocity of sound in the fluid of the jet having the same pressure and temperature as said portion of the jet, a material containing condensable constituents, said dispersion having a temperature at which the droplets thereof become, by condensation, substantially dry particles of said resinous material, and separating from the elastic fluid said substantially dry particles.

26. A method of producing a resinous moulding powder including dispersing in a high velocity jet of elastic fluid, having in at least a portion thereof a velocity of flow at least equal to the velocity of sound in the fluid of the jet having the same pressure and temperature as said portion of the jet, a material containing reaction constituents, said dispersion having a temperature at which the droplets thereof become, by reaction of the constituents, substantially dry particles of said resinous material, and separating from the elastic fluid said substantially dry particles.

27. A method including passing elastic fluid into a receiver through nozzles to provide a plurality of high velocity jets each having in at least a portion thereof a velocity of flow at least equal to the velocity of sound in the fluid of the jet having the same pressure and temperature as said portion of the jet, introducing in predetermined proportions into the several jets materials to be admixed to produce a fine suspension of each and admixture of said suspensions, and separating non-gaseous material from the elastic fluid.

28. A method including passing elastic fluid into a receiver through nozzles to provide a plurality of high velocity jets each having in at least a portion thereof a velocity of flow at least equal to the velocity of sound in the fluid of the jet having the same pressure and temperature as said portion of the jet, introducing substantially continuously in predetermined proportions into the several jets materials to be admixed to produce a fine suspension of each and admixture of said suspensions, and separating non-gaseous material from the elastic fluid.

29. A method including dispersing in a high velocity jet of elastic fluid having in at least a portion thereof a velocity of flow at least equal to the velocity of sound in the fluid of the jet having the same pressure and temperature as said portion of the jet, a material which at the temperature of the dispersion becomes partially dry but adherent to solid walls, and providing along a wall which otherwise would be reached by said dispersion a barrier comprising a mass of elastic fluid flowing along said wall.

30. A method including dispersing in a high velocity jet of elastic fluid having in at least a portion thereof a velocity of flow at least equal to the velocity of sound in the fluid of the jet having the same pressure and temperature as said portion of the jet, a material which at the temperature of the dispersion undergoes a compositional change, and subjecting said dispersion to successive actions of spiral flows of elastic fluid.

31. A method including dispersing in a high velocity jet of elastic fluid having in at least a portion thereof a velocity of flow at least equal to the velocity of sound in the fluid of the jet having the same pressure and temperature as said portion of the jet, a material which at the temperature of the dispersion undergoes a compositional change, and subjecting said dispersion to the action of a spiral flow of elastic fluid.

32. A method including passing elastic fluid into a receiver through a nozzle to produce a high velocity jet having in at least a portion thereof a velocity of flow at least equal to the velocity of sound in the fluid of the jet having the same pressure and temperature as said portion of the jet, introducing into the jet a material in at least a partially liquid state to produce a fine suspension of the material, and subjecting said suspension to admixture with a second suspension of the same material.

NICHOLAS N. STEPHANOFF.

REFERENCES CITED

The following references are of record in the file of this patent:

UNITED STATES PATENTS

Number	Name	Date
920,334	Hughes	May 4, 1909
1,830,174	Peebles	Nov. 3, 1931
1,914,895	Peebles	June 20, 1933
2,032,827	Andrews	Mar. 3, 1936
2,101,635	Bender	Dec. 7, 1937
2,187,877	Ferris et al.	Jan. 23, 1940
2,188,506	Hall	Jan. 30, 1940
2,202,481	Cox et al.	May 28, 1940
2,253,319	Batterman	Aug. 19, 1941
2,290,393	Thomas	July 21, 1942
2,297,726	Stephanoff	Oct. 6, 1942