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Matocha

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[54] **HOMOGENEOUS FUSION SYSTEM AND METHOD**

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[52] **U.S. Cl.** .... **432/11;** 432/44; 432/157; 266/36 P

[51] **Int. Cl.** **F27b 3/06**

[58] **Field of Search** .... 432/11, 12, 44, 156, 157; 266/36 P

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[57] **ABSTRACT**

A fusion system is provided whereby material in comminuted form or the like may be automatically fused under conditions which provide a homogeneously fused sample which then may be analyzed by analytical techniques to determine content of the material. The apparatus for carrying out the fusion provides control of the initial heating time, fusion time, and cooling time, as well as controlling the mixing or blending of the sample material during the fusion to ensure the homogenuity of the mixture.

10 Claims, 6 Drawing Figures

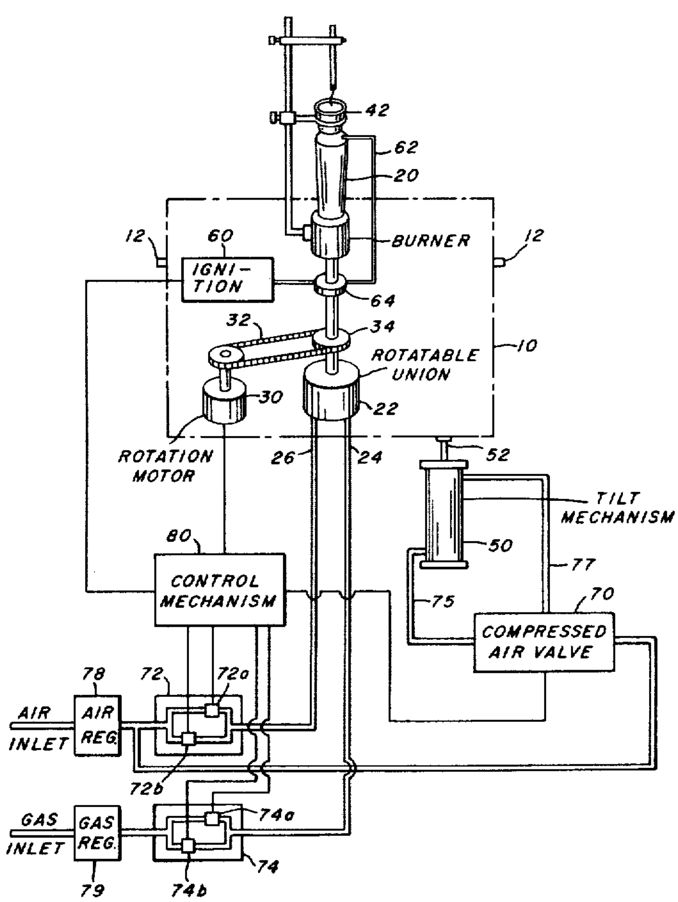
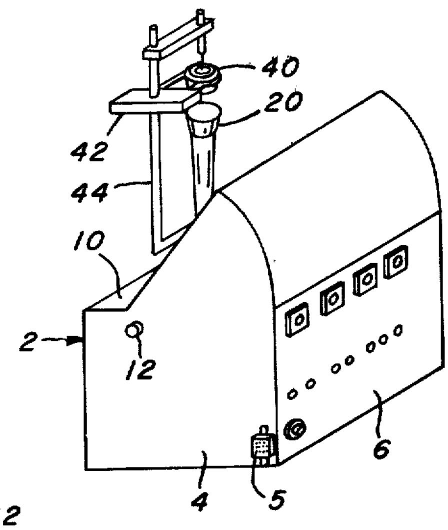


FIG. 1.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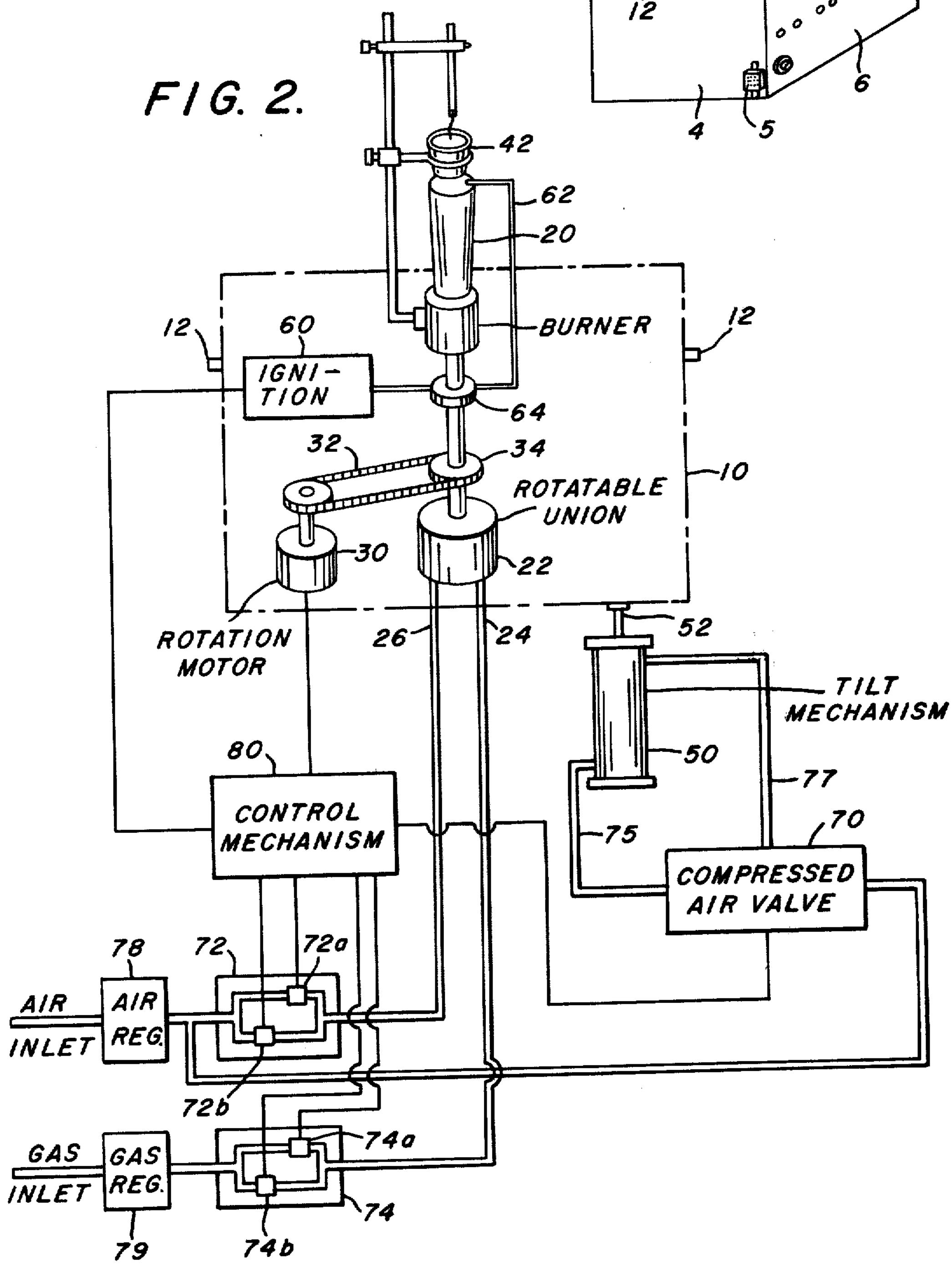
6
4
5

FIG. 2.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62
20
12
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IGNI-TION
BURNER
12
64
32
34
ROTATABLE UNION
10
30
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ROTATION MOTOR
26
24
52
TILT MECHANISM
50
80
CONTROL MECHANISM
77
70
75
COMPRESSED AIR VALVE
78
72
72a
AIR INLET
AIR REG.
72b
74a
GAS INLET
GAS REG.
74
79
74b

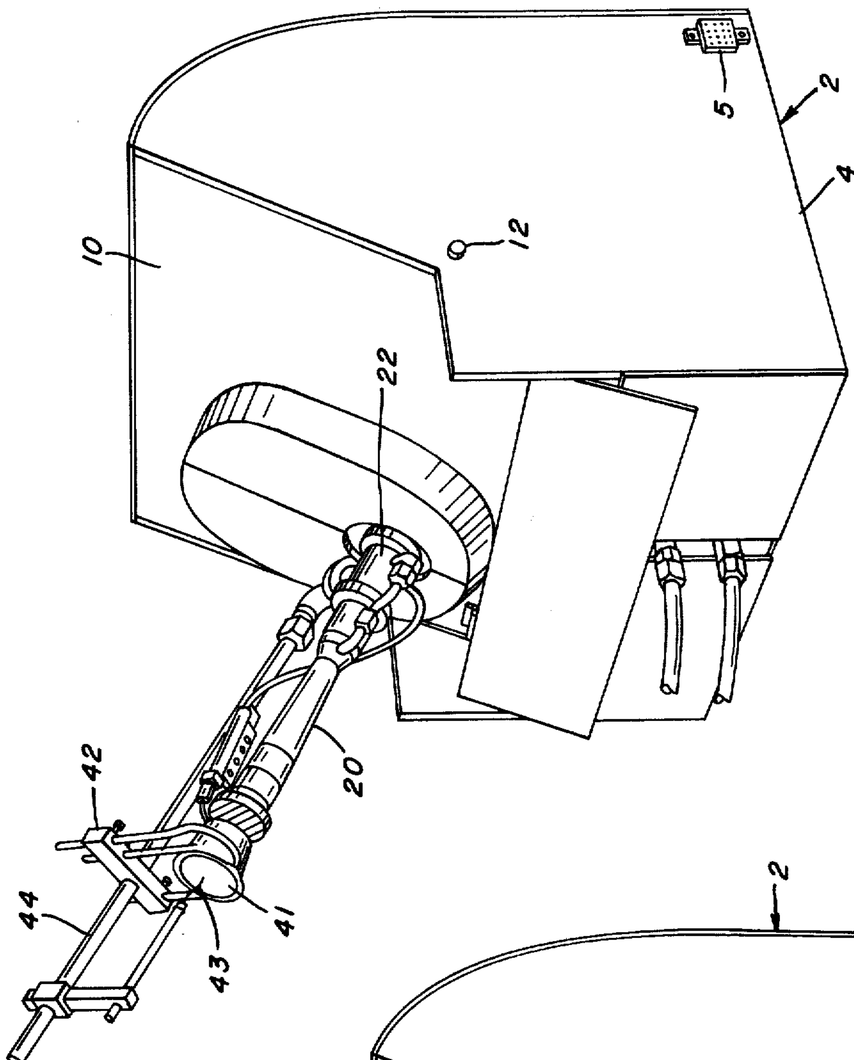


FIG. 4.

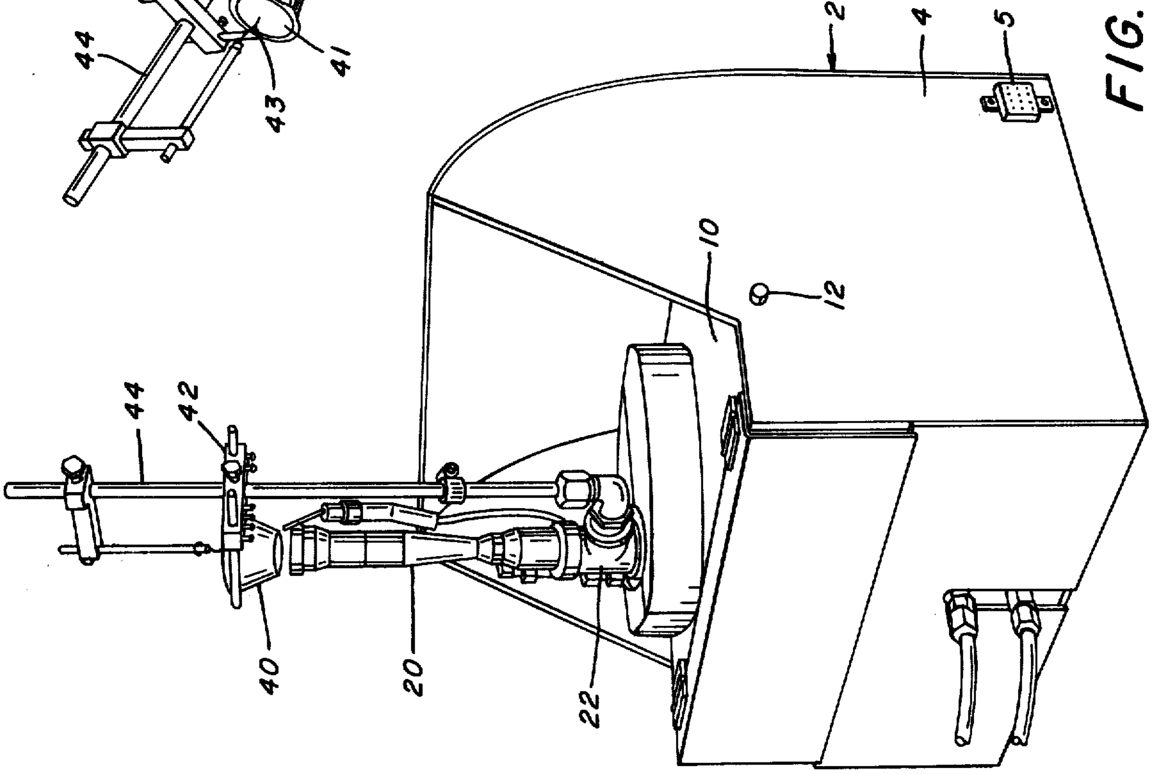
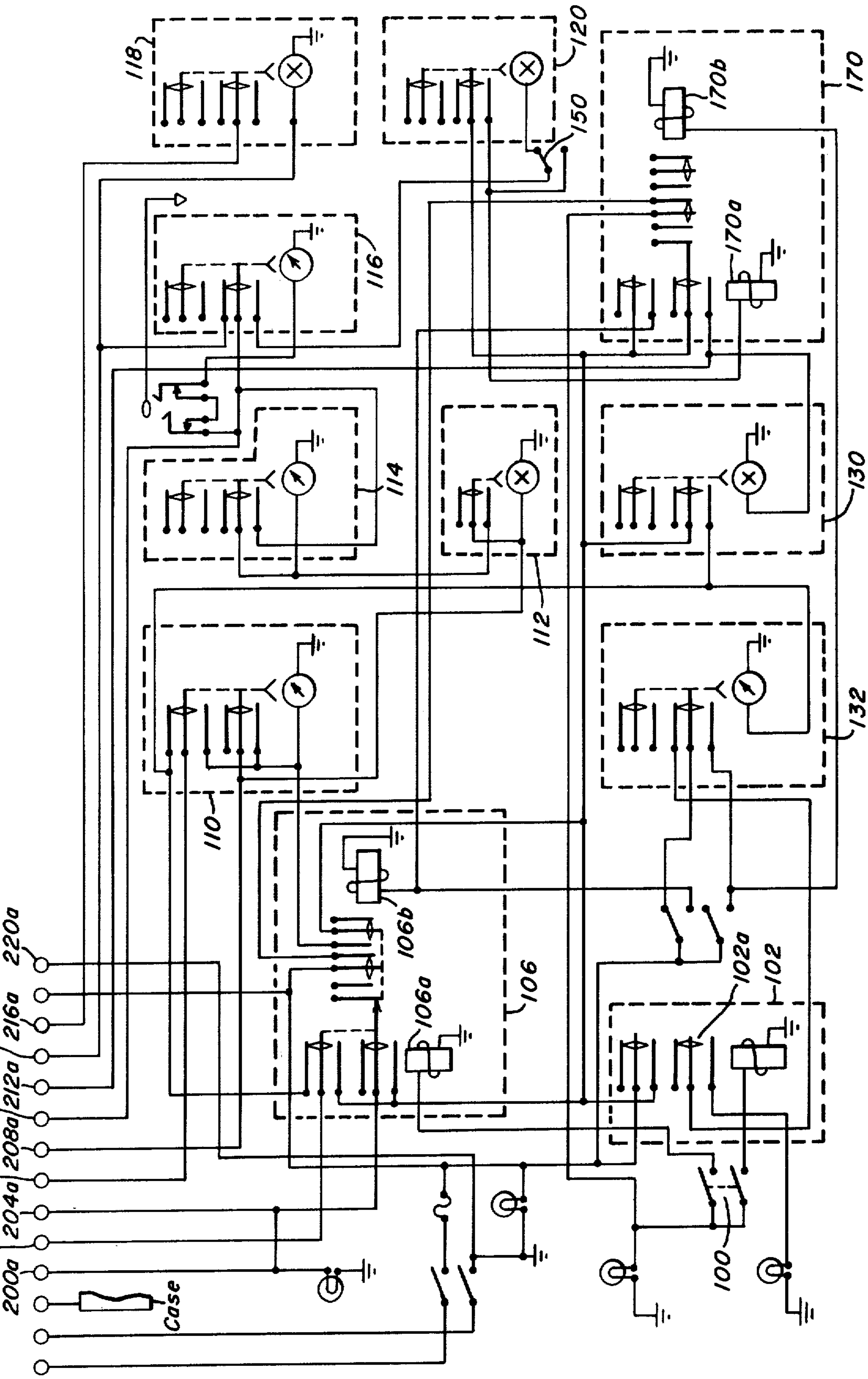


FIG. 3.

FIG. 5.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120
150
170
170b
170a
116
114
112
130
110
132
106b
106
106a
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204a
202a
200a
Case

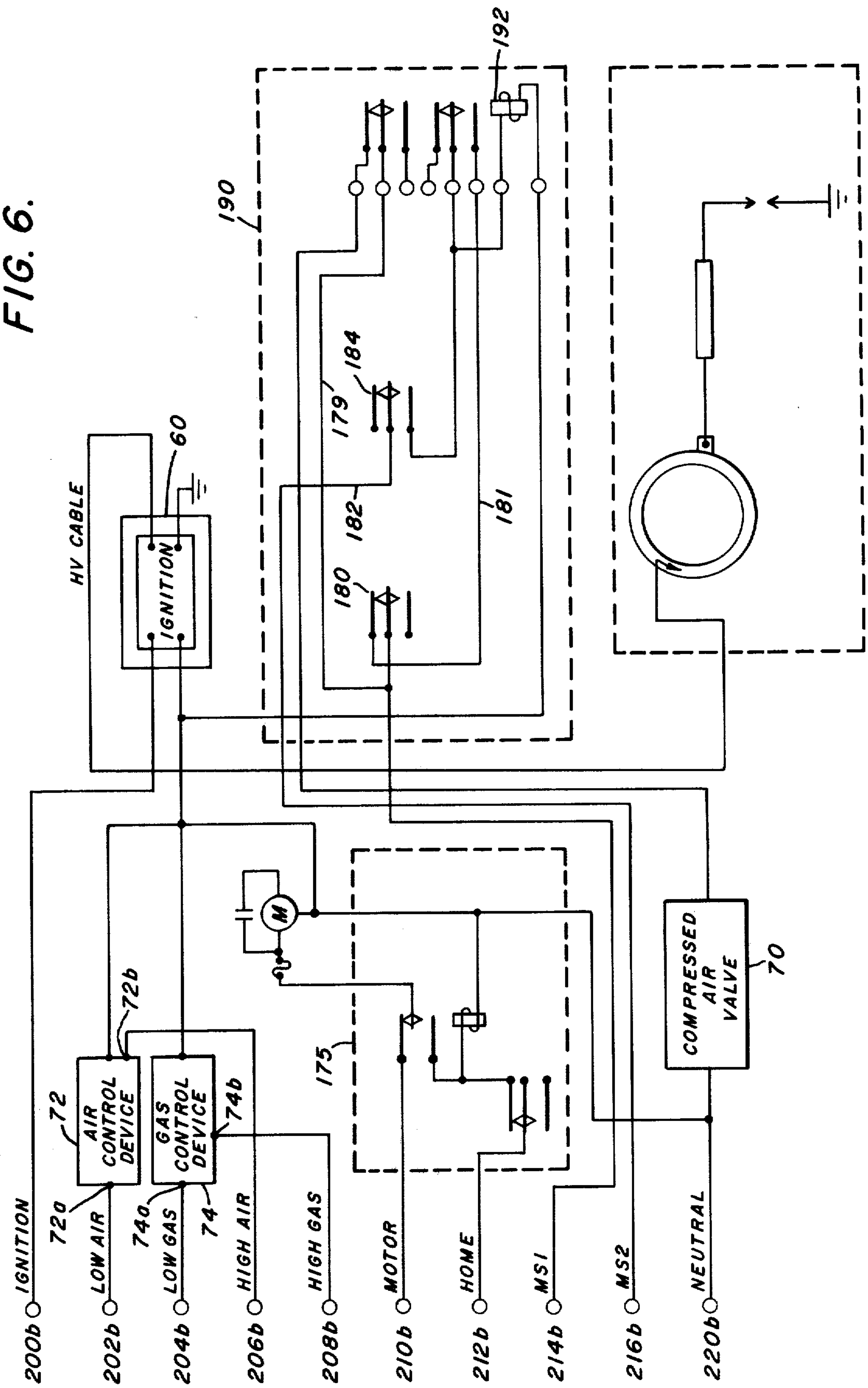
HV CABLE
IGNITION
60
190
192
184
179
182
181
180
175
M
AIR CONTROL DEVICE
72
72a
72b
GAS CONTROL DEVICE
74
74a
74b
COMPRESSED AIR VALVE
70
IGNITION
LOW AIR
LOW GAS
HIGH AIR
HIGH GAS
MOTOR
HOME
MS1
MS2
NEUTRAL
200b
202b
204b
206b
208b
210b
212b
214b
216b
220b

FIG. 6.

HOMOGENEOUS FUSION SYSTEM AND METHOD

BACKGROUND OF THE INVENTION

This invention relates to a system for automatically producing homogeneous fused materials suitable for use in analytical equipment.

Preparation of mixtures of materials for analysis including both metallic and non-metallic samples can be made in a variety of ways. An approach has been to pulverize materials such as ores, slags, or the like. The pulverized materials are then analyzed using various techniques such as, for example, x-ray fluorescence or the like.

However, while preparation of this type is simple and quick, possibilities of error arise for example, in x-ray fluorescense, which restrict its application especially when a high level of accuracy is desired. This is because the samples are themselves not fully homogeneous after grinding. Furthermore, the samples are affected by the different crystal structures which can exist in similar chemical compositions. In addition, materials of differing hardness have different size distribution after pulverizing.

It has thus become preferable to prepare a fused sample. If the materials are properly mixed together during the fusion, the resulting fused sample is homogeneous. The fused sample can then be ground or used directly in tablet form and, in either event, is permanently homogeneous. Other materials such as internal standards or the like can be added to the mixture prior to the fusion and, then upon fusion, will become homogeneously mixed throughout the sample.

Despite the advantages of use of the fused sample for analytical purposes, the cost and inconvenience of producing such a sample has greatly inhibited the use thereof. It has previously been necessary for one to individually heat a crucible containing the fusible materials and then to attempt to mix the materials while in a fused state. This usually necessitates removing the crucible, using tongs or the like, from the heating zone and manually mixing the molten material by a swirling action to effect the stirring. This will also help remove entrapped bubbles as well as ensure complete incorporation of all materials on the sidewalls of the fusion container into the fused material. However, such manual mixing is almost inevitably done outside the heating zone and, particularly when working at such elevated temperatures, circa 1100°C, rapid cooling, therefore, occurs. This, in turn, results in some undesirable solidification requiring a remelting period.

SUMMARY OF THE INVENTION

It is therefore an object of this invention to provide a system whereby fusible materials previously loaded into a container may be automatically melted, mixed while fusion is occuring to provide a homogeneous melt, and then cooled, all via predetermined timed sequences to prepare a homogeneous sample.

It is yet another object of the invention to provide apparatus for including programmed electrical control means for controlling the preparation of fused samples.

In accordance with the invention, apparatus for the preparation of homogeneously fused material is provided comprising means for holding the material to be fused and means for heating the material for a preselected time at a temperature below the fusion temperature to expel, for example, entrapped air and low temperature volatiles; means for heating the material to fusion temperature for a predetermined period of time; means for rotating the material during heating; means for tilting the material during fusion to a predetermined angle; and means for cooling the fused material after the cessation of heating. In a preferred embodiment, the tilting and rotating of the material are controlled so that the material can be independently tilted or rotated; the material may be constantly rotated while at an angle; or the material may be rotated while being simultaneously moved back and forth between a vertical position and a tilted position. Each of the periods of heating, rotating, tilting, and cooling may be variably timed to obtain the optimum respective periods to thereby provide the desired homogeneous fusion.

BRIEF DESCRIPTION OF THE DRAWINGS

FIG. **1** is an isometric view of the apparatus.

FIG. **2** is a diagrammatic chart functionally illustrating the apparatus.

FIG. **3** is a fragmentary isometric view of a portion of the apparatus.

FIG. **4** is a fragmentary isometric view similar to FIG. **2** showing the same apparatus in a different position.

FIG. **5** is a schematic of the programmed electrical control circuitry for the apparatus.

FIG. **6** is an electrical schematic of certain components of the apparatus which electrically interface with the electrical control circuitry in FIG. **5**.

DESCRIPTION OF THE INVENTION

Referring now to FIGS. **1** and **2**, the apparatus of the invention is shown generally at **2**, comprising a case **4** having a front control panel **6** containing a number of controls which will be described in more detail below. A tiltable platform portion **10** has mounted thereon a burner **20** such as a Fisher-Meker burner or the like. Burner **20** is rotatably mounted on platform **10** via a rotatable union **22** which is appropriately journaled through platform **10**. Rotatable union **22**, in turn, contains mechanisms for feeding gas via gas line **24** and air from an air line **26** to burner **20** while allowing burner **20** to rotate with respect to the lines. Devices of this kind are commercially available and are well known to those skilled in the art. For example, such a union is commercially available from the Perfecting Service Company. Rotation of burner **20** is provided by a motor **30** which, in the illustrated embodiment, is connected to burner **20** via a chain drive **32** which drives a sprocket at **34** mounted to an extension of burner **20** coupled to union **22**.

The sample to be fused is placed in a crucible **40** and covered by a lid **41**. Crucible **40**, in turn, is retained by a clamp or support **42** which may conveniently comprise any suitable supporting means such as a circular clamp, three-dimensional or three-pronged, or the like. Lid **41** is retained in place by a clamp **43**. Clamps **42** and **43** are supported by a bar **44** which is, in turn, mounted to the bottom portion of the burner to allow the entire mechanism to rotate together.

In the illustrated embodiment, platform **10** may be tilted to a position of, for example, about 30° from the horizontal by an air cylinder **50** coupled to platform **10** at **52**. Platform **10** is pivoted at **12** to permit the tilting of the platform via movement of air cylinder **50**.

Ignition of burner **20** is provided by an ignition mechanism **60** which provides an electrode **62** which terminates at a point adjacent the outlet of the burner. The burner is grounded to provide, together with electrode **62**, the terminals of a spark gap through which a spark passes to ensure ignition of the gas. The electrical connection of the ignition control **60** with electrode **62** is accomplished via a wiper ring **64** electrically isolated, but mechanically mounted to the burner which permits the electrode **62** to rotate with burner **20**.

Compressed air for air cylinder tilting mechanism **50** is provided from a pressure regulated source adjusted to provide a sufficiently low pressure to permit a damped or gradual movement of the piston within the air cylinder.

Control of the air flow to the air cylinder tilting mechanism is provided by a solenoid valve **70**. Similarly control of the flow of air and gas to burner **20** is provided respectively by control devices **72** and **74**. Air control device **72** and gas control device **74** each have two separate solenoid valves **72***a*, **72***b* and **74***a* and **74***b* controlling parallel paths and which are separately controlled to permit a low or high rate of flow through the device depending upon whether only one or both of the solenoid valves are energized. The temperature of the burner during either the low heat or fusion heat cycles is determined by the respective volumes of gas and air passed through the respective valves by varying either the pressures at gas pressure regulator **79** and air pressure regulator **78** or the needle valves (not shown) associated with the respective solenoid valves.

The ignition, rotation, tilting, and combustion are all controlled by an electrical control circuit mechanism **80**. This mechanism comprises timing and control means which may be electrical or mechanical. In the illustrated embodiment mechanism **80** comprises a series of solid state timers and electrical relays (which will be described in more detail) permitting one to program the rotation, tilting, initial heating, fusion, and cooling of the fused material. Control mechanism **80** is designed to permit one to interconnect the terminals to a plurality of such fusion units or apparatus containing the various valves and rotation, ignition, and tilting mechanisms to permit a number of fusion samples to be prepared simultaneously using the same programming and electrical controls. Such units, which might be designated as slave fusion units, would be very similar to that which has just been described except for the omission of control mechanism **80** including the controls shown on the front panel of the apparatus of FIG. **1** at **6**.

Turning now to FIGS. **3** and **4**, the apparatus is shown with platform **10** positioned horizontally in FIG. **3** to place burner **20** in a vertical position and in a tilted position in FIG. **4**. Initial rotation of burner **20** and crucible **40** in the tilted position permits the fusible material therein to lap the wall of crucible **40** thus picking up or incorporating any powdered granular or globules of sample material which may stick to the sidewalls of the crucible. It will be seen that the rotation of burner **20** can be accomplished in the vertical position as well as the tilted position and, furthermore, that the rotation may be maintained while imparting a rocking motion to burner **20** and platform **10** by alternatively tilting platform **10** to the tilted position shown in FIG. **4** and cyclically returning platform **10** to a horizontal position and retilting the platform. While the rotation of the burner and the crucible in the tilted position tends to impart some stirring of the material in crucible **40**, the additional rocking motion of mechanism during rotation imparts a swirling action to the material within crucible **40**. This swirling action also assists in the removal of entrapped bubbles of gases which may evolve as the material is fused. In any event, the rotation and tilting provide a means for automatically mixing the fused materials as they are fused. This type of mixing, as discussed above, was previously accomplished manually, by removing a crucible, for example, from a furnace using tongs and then manually swirling the crucible. In accordance with the invention, however, it is possible to maintain this mixing and swirling during the actual application of heat. Thus, the fused material does not cool during the swirling action.

After the fusion has been completed, the heat optionally is shut off and the platform **10** is returned to a horizontal position. Alternatively, a short additional heating period is available after swirling action has ceased to ensure that the melt settles in the bottom of the crucible to avert any solidification on the wall and to obtain the desired solidified bead. As will be discussed with respect to FIG. **5**, the rotation of the crucible preferably continues until a predetermined position is reached.

Following solidification of the sample, and cooling under a timed ambient condition, both solenoid valves of air control device **72** are permitted to reopen to provide cooling air through burner **20** to crucible **40**. This greatly assists in obtaining more rapid cooling of the sample to permit removal of the sample from the apparatus in a shorter period of time, thus allowing one to commence preparation of subsequent samples. Alternatively only one of the solenoid valves could be opened if desired.

Turning now to FIGS. **5** and **6**, the actual control of the various sequential steps via control mechanism **80** will be described in more detail. FIG. **5** schematically illustrates the control mechanism **80** in FIG. **2** while FIG. **6** schematically illustrates the components in FIG. **2** energized by control mechanism **80**. Control mechanism **80** in the illustrated embodiment in FIG. **5** comprises a series of relays and timers which program the entire sequential fusion steps of the apparatus. The circuitry of FIGS. **5** and **6** are interconnected by terminals **200***a*–**220***a* and **200***b*–**220***b* respectively. The combined circuitry of FIGS. **5** and **6** as shown in FIG. **2** comprises an independent or master unit as will be described further below.

After appropriate mounting of crucible **40** with the fusible material therein, the fusion is initiated by depression of a start button **100** on the front panel of the master unit **4**. This in turn energizes power holding relay **102** and a heating holding relay **106**. Power holding relay **102** includes an electrical holding circuit through terminals **102***a* in the drawing. Heating relay **106** has a mechanical latch mechanism which is latched by energization of coil **106***a* and unlatched by coil **106***b*. Latching of relay **106** provides energizing signals to gas solenoid valve **74***a* and air solenoid valve **72***a* causing the solenoid valves to open. The ignition circuit is also completed and the low heat cycle therefore commences. In addition to the expulsion of low temperature volatiles, or occluded gases, this low heat capability of the apparatus is of particular interest when a low heat roasting is desired, for example, when a sample is mixed with an oxidizing agent.

Simultaneously, with initiation of the low heat cycle, adjustable timer relay **110** is energized to control the time period for provision of low heat through burner **20** to crucible **40.** In a preferred embodiment the timer relay **110** and other such relays which will be referred to below each comprise an electronic timer which is energized and then, subsequent to the expiration of a preselected time period, an associated relay coil is energized to move the relay contacts from their illustrated rest positions. Timer relay **110** and the other timer relays to be described in the illustrated embodiment do not have mechanical latching but are rather held in a closed or latched position when desired by appropriate electrical holding circuits. Timer relay **110** and similar adjustable timers to be described below are adjustable via appropriate control knobs on the front panel **6** of unit **4** to provide variable time periods for the respective cycles which they control.

After expiration of the preselected low heat time period on time relay **110,** its coil is energized to respectively energize gas solenoid valve **74***b* and air solenoid valve **72***b* to initiate the fusion heating or high heat cycle as well as to initiate the sequential timing of timers **112** and **114.**

Timer **112** in the illustrated embodiment is a preset timer which provides a preset heating time of four minutes, following which variable timer relay **114** is energized. The total time period measured by these timers thus provides an adjustably timed premix fusion cycle.

After the expiration of the premix cycle the closing of relay contacts on timer relay **114** completes a circuit through timer relay **116** and terminals **214***a* and **214***b* to tilting circuit **190** to energize a compressed air solenoid valve **70** allowing air to flow to air cylinder **50** to tilt platform **10** to a tilted position. The closing of the relay contacts on timer relay **114** commences the timing function of timer relays **116** and **118** as well as energizing motor **30** to commence rotation of the crucible while platform **10** remains in the tilted position. Timer **118** is a preset timer providing preliminary mixing for a predetermined period. In the preferred embodiment illustrated, rotation of the crucible for about 90 seconds while in a tilted position, as determined by timer relay **118,** allows the fused material to lap the walls of the crucible to prevent adherence thereto of any particle which may not have been incorporated into the main mass.

After the expiration of time on relay **118,** its relay coil is energized to provide an interconnection via terminals **216***a* and **216***b* to tilt circuit **190** allowing the signal previously transmitted to valve **70** to be passed to microswitch **184** via line **182** to initiate the cyclic tilting.

Solenoid valve **70** is a conventional normally-open normally-closed four-way pneumatic solenoid valve which is interconnected to a double acting air cylinder **50** by air lines **75** and **77** to alternatively control the direction of movement of the piston within cylinder **50** as is well known to those skilled in the art.

When platform **10** reaches full tilt, microswitch **184** is tripped, closing the normally open contacts. This in turn momentarily energizes the coil of relay **192** causing it to open its normally closed contacts to de-energize air valve **70** and therefore switches the pressure to the other side of air cylinder **50** causing platform **10** to return to its horizontal rest position. While the contact with microswitch **184** is only momentary, the energization of relay **192** completes a holding circuit through the relay coil of relay **192** via the normally closed contacts of microswitch **180** and line **181.**

Platform **10** continues to return toward the horizontal position, and, at its terminal position, the contacts of microswitch **180** are opened breaking the holding circuit to relay coil **192** thereby again energizing cylinder valve **70.**

This cyclic action continues until the expiration of the time period of timer **116.** In a preferred embodiment the rotation of crucible **40** via motor **30** and drive members **32** and **34** continues thereafter until the mechanism reaches a preferred home point comprising a desired orientation of the holder (which assists in easy removal of crucible **40** from holder **42**) via the circuitry outlined in box **175.**

At the same time, heating may be continued if desired by activation of switch **150** to the position shown in the drawing to provide an additional **30** seconds of heating until timer **120** times out energizing coil **106***b* to break the heating cycle by unlatching relay **106** and at the same time latching cooling relay **170** by energization of coil **170***a* and energizing a preset timer **130** which provides a predetermined cooling period of ambient conditions of about three minutes.

Following expiration of this fixed time period, timer relay **132** is energized to control the amount of time that cooling air will be permitted to pass through the burner via activation of the valves of air control device **72.**

After the cooling cycle, which may be as long as eight minutes, coil **170***b* is energized (which unlatches the cooling relay **170**) and power relay **102** is de-energized by interrupting the holding circuit thereto. The system is thus returned to a ready position whereby, upon removal of the sample and insertion of a new sample, the cycle can be commenced again.

The circuitry illustrated in FIGS. **5** and **6** is usually incorporated into a unitary apparatus as generally shown at **80** in FIGS. **1** and **2** which then may be termed a master or independent unit. This master unit can also be used to control several units which omit this control circuitry. Such units can be referred to as slave units. In constructing such slave units, the components generally shown in FIG. **2,** (exclusive of control mechanism **80**) and shown schematically in FIG. **6,** will be preferably incorporated into the slave unit. For its operation the slave unit is connected to the master unit by an electrical cable or other suitable connecting means which electrically communicates with the control mechanism **80** of the master unit. This connection (illustrated at **5** in FIG. **1**) conveniently can be at the junction of terminals **200–220,** *a* and *b*, respectively, as previously described.

While the preferred embodiment of the mechanism has been described hereinabove, it should be readily recognized that minor modifications may be made. For example, the ignition may be carried out through means other than a spark. The various timers may be electronic or electromechanical or the like and may be all made adjustable or fixed depending upon the circumstances. The rotation may be controlled by an air motor rather than an electrical motor; the tilting mechanism could be controlled by an electrical motor operated with a camming mechanism rather than the air cylinder shown. Other modifications will also be readily discernible by those skilled in the art.

While the invention has been described in terms of preferred embodiments, the claims appended hereto are intended to encompass all embodiments which fall within the spirit of the invention.

What is claimed is:

1. Apparatus for the preparation of homogeneously fused material comprising:

a. means for holding material to be fused;
b. means for heating said material for a preselected period of time at a temperature below the fusion temperature;
c. means for heating said material to fusion temperature for a predetermined period of time;
d. means for establishing and controlling said respective temperatures;
e. means for rotating said holding means;
f. means for tilting said holding means to a predetermined angle;
g. means for cooling said fused material after cessation of heating; said means for heating and means for cooling including solenoid operated valves.

2. The apparatus of claim **1** including means for maintaining said holding means at said predetermined angle while simultaneously rotating said holding means.

3. The apparatus of claim **1** including means for simultaneously rotating said holding means while cyclically tilting said holding means to said predetermined angle and returning said holding means to a vertical position.

4. Apparatus for the preparation of homogeneously fused material comprising:

a. a housing;
b. a tiltable platform mounted on said housing;
c. heating means mounted to said platform to provide fusion heat to said material to be fused;
d. means on said tiltable platform for holding material to be fused including means to rotate said fusible material with respect to said housing;
e. means for tilting said platform from a first position to a second position to move said fusible material with respect to said holding means to thereby aid in mixing of said material to form a homogeneous sample and removing of air bubbles; and
f. cooling means to cool said fusible material after exposure to said fusion heat.

5. The apparatus of claim **4** wherein said cooling means comprise ambient cooling means to control annealing of the solidified specimen and means for subsequently providing a cooling stream to the annealed specimen.

6. The apparatus of claim **4** wherein said heating means comprise a gas burner and solenoid operated valves provide gas and air to said burner at predetermined volumes to control the heat supplied to said fusible material.

7. The apparatus of claim **6** wherein programmed electrical control means are included to control the heating time by electrically controlling said solenoid valves.

8. The apparatus of claim **6** wherein said means to rotate said fusible material include a motor operatively coupled to said holding means to rotate said fusible material with respect to said housing.

9. The apparatus of claim **8** wherein programmed electrical control means are included to control said motor and said tilting means to cyclically tilt said fusible material during at least a portion of the time said material is rotating to impart a swirling action to said fusible material whereby fusible material adhering to sidewalls of said holding means will be incorporated into the melt to provide a homogeneously fused sample upon subsequent cooling of the sample.

10. A process for the preparation of homogeneously fused material comprising:

a. loading fusible material into holding means;
b. heating said material within said holding means for a preselected period of time to a preselected temperature below the fusion temperature to expel entrapped air and low temperature volatiles;
c. heating said material to fusion temperature for a predetermined period of time;
d. rotating said holding means for a predetermined period of time at a predetermined angle of tilt while simultaneously heating said material at fusion temperature to lap the interior wall of the holding means to clean said wall of any fusible material;
e. rotating said holding means while cyclically tilting said holding means to a predetermined angle and returning said holding means to a vertical position while simultaneously heating said material at fusion temperature to provide homogeneous mixing of said fusible material and elimination of gas bubbles; and
f. cooling said fused material at one or more predetermined temperatures for a predetermined period of time.

* * * * *