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METHOD OF AND APPARATUS FOR PREPARATION OF
HIGHLY FLAMMABLE MIXTURES

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2 Sheets-Sheet 1

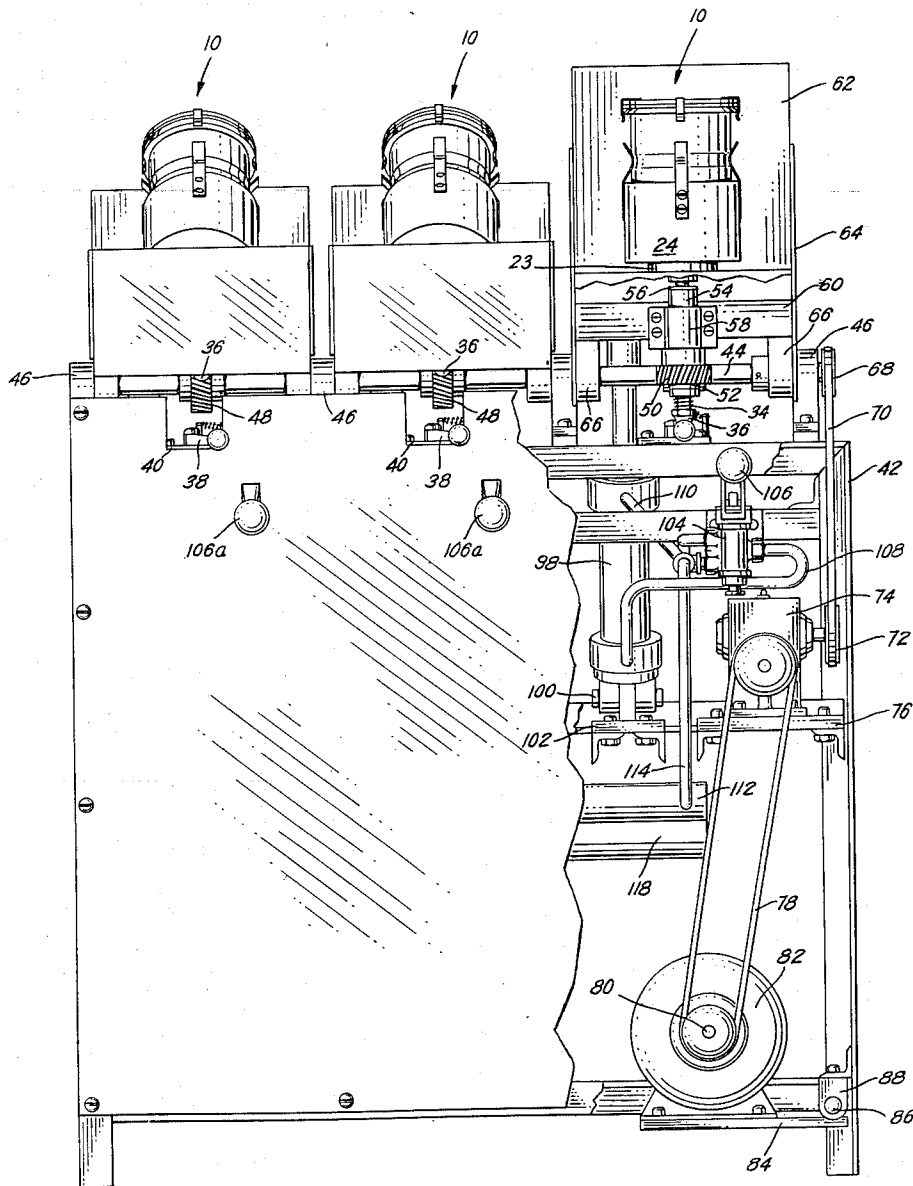


Fig. 1

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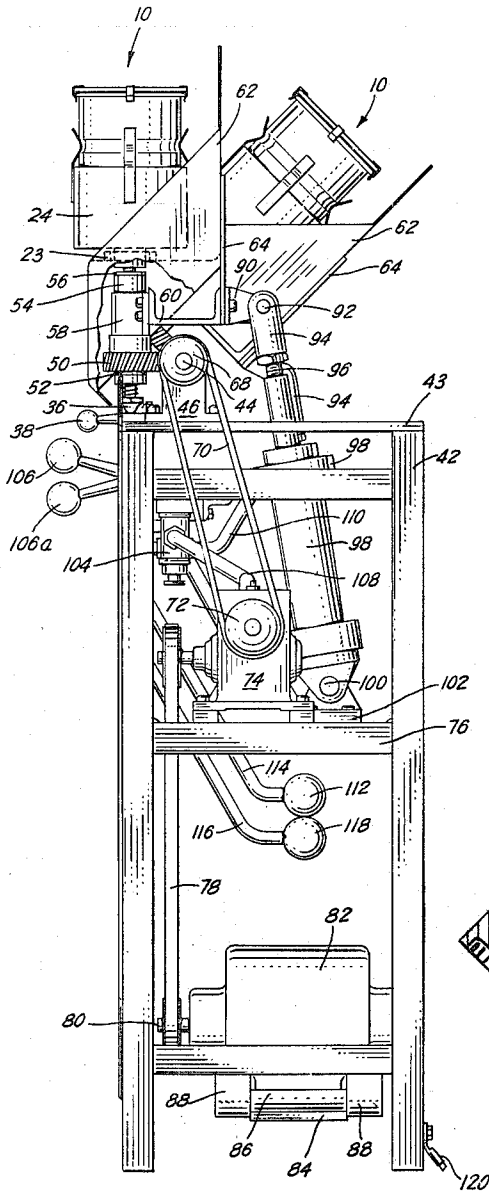


Fig. 2

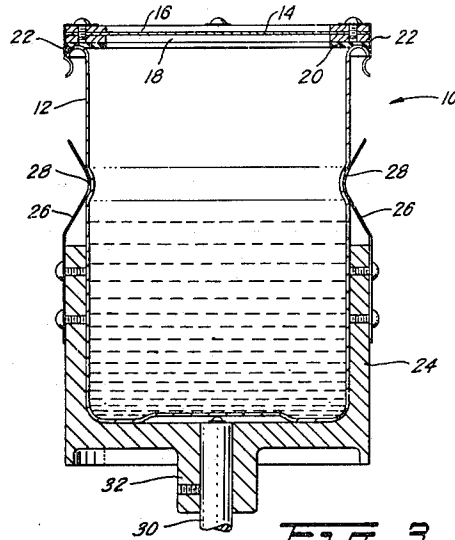


Fig. 3

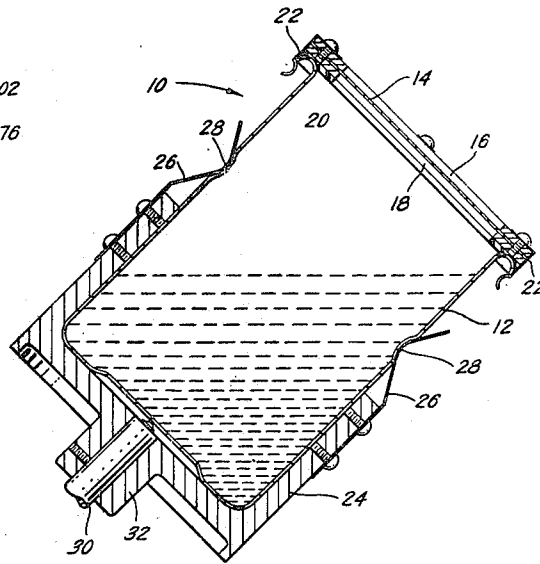


Fig. 4

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METHOD OF AND APPARATUS FOR PREPARATION OF HIGHLY FLAMMABLE MIXTURES

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7 Claims. (Cl. 52-19)

This invention relates to the preparation of highly flammable ignition paste employed in the manufacture of photographic flash lamps which provide a short duration, high intensity light source. The light emission has to be precisely timed to synchronize with the opening of the shutter of a photographic camera. The flash in the lamp is started by closing an electric circuit which heats a small tungsten filament to incandescence. The heated filament in turn ignites two small beads of ignition paste which are agglutinated to the ends of two lead wires in close proximity to the filament.

The ignition paste employed in the manufacture of photographic flash lamps usually includes very fine, dry zirconium metal powder with an average particle size of two microns because it has been found that this material provides the desired reliability and precision in timing the peak development of light emission when shredded foil or only zirconium metal powder is burned in an oxygen atmosphere. However, dry zirconium metal powder is highly flammable and must be handled with great care in order to avoid serious accidents.

The preparation of an ignition paste for use in the manufacture of photographic flash lamps usually comprises mixing zirconium metal powder, magnesium metal powder and potassium perchlorate in a solution of $\frac{1}{2}$ sec. nitrocellulose in amyl acetate. Great care must be exercised, not only in handling the dry zirconium powder, but also in the preparation of the mixture to obtain the ignition paste. Usually a separate laboratory constructed and equipped to handle the zirconium metal powder and mixtures of it is employed. Personnel handling the metal powders and preparing the mixtures must wear certain safety apparel and use certain safety equipment.

Various means have been utilized in an effort to develop a safe method of preparing this ignition paste. In accordance with one method, the dry powders are charged into a nitrocellulose solution in a beaker and this mixture is kept under a nitrogen atmosphere by bubbling N_2 through amyl acetate first and then discharging it through a pipe inside the beaker. All the material in the beaker is then mixed with a mechanical, propeller-type, stirrer. Care must be exercised to make sure that the N_2 , carrying amyl acetate fumes, is removed properly. The mechanical stirrer must be controlled to prevent it from striking any obstacles or creating heat by friction which could ignite the mixture. The ignition paste can burn or explode under a nitrogen or argon atmosphere because it contains oxidizers like potassium perchloride. The zirconium metal powder will also react quickly with N_2 at about $500^\circ C$. Thus the N_2 atmosphere only retards the ignition; it does not eliminate the hazard of an explosion.

In accordance with another method, the dry powders are charged into a porcelain ball mill jar containing a quantity of small flint pebbles and is then covered by a solution of nitrocellulose in amyl acetate. A heavy porcelain lid, which is employed as a closure, is tightly

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secured to the jar to prevent leakage of the binder and drying of the paste when the jar is rotated or rolled with its longitudinal axis in a horizontal position. One of the disadvantages of this method is that the mixing is done in a heavy wall vessel which is tightly closed, and a high pressure can be built up without any noticeable change which could serve as a warning of danger. Thus, if explosions of mixtures in a porcelain ball mill jar occur, serious accidents usually result.

In view of the foregoing one of the principal objects of this invention is to effect the preparation of a highly flammable ignition paste in a safe manner.

In accordance with the principles of this invention, the paste is prepared by pouring a part of the binder solution into a beaker of non-sparking material, such as stainless steel for example, which contains a number of porcelain balls. The beaker is then rotated so that a major portion of the inside wall of the beaker and the balls are wetted down. The metal powders are charged into the beaker, and finally the remainder of the binder solution is added. The final addition of the binder solution insures the wetting down of all parts of the mixture. The beaker is then closed by a lid which comprises a thin sheet of metal or foil of non-sparking material. The foil lid constitutes an excellent venting membrane which will rupture before any substantial pressure is built up inside the beaker. The beaker is then tilted at an angle from the vertical and rotated to work the mixture into a smooth suspension ready for use. The quantity of materials mixed in each batch and the angle at which the beaker is rotated are controlled so that none of the materials being mixed touch the lid. By doing this, the danger of spillage and the need for carefully cleaning the lid for safety reasons after each batch has been mixed are minimized.

An apparatus with which the method of this invention may be employed is illustrated in the accompanying drawings in which:

Figure 1 is a front elevational view of the apparatus.

Figure 2 is an end elevational view of the apparatus.

Figure 3 is a transverse sectional view of a mixing container shown in its rest position, with the mix disposed therein.

Figure 4 is a transverse sectional view of a mixing container shown in the position in which it is disposed during the mixing operation, with the mix disposed therein.

Referring now to the drawings, particularly Figure 3 thereof, container 10 comprises a beaker 12 provided with a foil lid 14 held by two concentric rings 16 and 18, ring 18 being provided with a gasket 20. Spring clips 22, attached to the top of ring 16, hook under the lip of the beaker 12 and draw the lid down tightly on the beaker, the gasket 20 providing a gas-tight seal of the lid to the beaker. The container 10 is disposed in a pot chuck 24 and is securely retained therein by spring fingers 26 which are attached to the pot chuck 24 and which interlock with indentations 28 in the wall of beaker 12. The pot chuck 24 is provided with a centering and positioning rod 30 depending from the neck 32 thereof. The rod 30 is spring loaded by compression spring 34 (Figs. 1 and 2) which engages cap 36 on the end of the rod. The cap 36 rides on the cam surface of a container lift lever 38 which is pivotally mounted on a plate 40 mounted on table 42.

Referring now to Figures 1 and 2, several of the container assemblies just described are located on the table 42. Rotation of the containers 10 is effected by rotation of shaft 44 which is supported by shaft supports 46 mounted on the table 42. Since substantially similar means are employed to effect rotation of each of the containers from the common drive shaft 44, only one will

be described. Helical drive gear 48 mounted on shaft 44 meshes with driven gear 50. Driven gear 50 is mounted on the lower end of sleeve 52 which is concentric with the rod 30, on which the pot chuck 24 is mounted, and through which the rod 30 freely extends. The upper end of the sleeve 52 has a shaft collar 54 attached thereto and a friction drive washer 56 is mounted on the upper end of the collar 54. The sleeve 52, driven by gear 50 mounted thereon, rotates within bushing housing 58 which is mounted on channel iron support 60.

A container guard 62, which is a fabricated member designed to form a partial enclosure for container 10, is mounted on a guard support 64 which is in turn attached to the channel iron support 60.

The container guard 62 is provided with a washer 23 brazed thereto and extending up into the recess in the bottom of the pot chuck 24, thus preventing any accidental spillage which might occur during mixing from running down into the mechanisms below.

The channel iron support 60 is provided with a pair of depending bushing housings 66 through which drive shaft 44 extends. Thus the container assembly is pivotally supported on the drive shaft 44.

Drive shaft 44 is provided with a driven pulley 68 which is connected by belt 70 to drive pulley 72 on gear reduction unit 74 mounted on support 76. Belt 78 connects the gear reduction unit 74 to the drive shaft 80 of motor 82. Motor 82 is mounted on a support 84 which is fixedly attached to shaft 86 and shaft 86 is rotatably supported in bracket 88. Thus the motor assembly is pivotally mounted to insure constant pressure on the belt 78 and elimination of slippage in the drive.

The channel iron support 60, to which the bushing housing 58 and the guard support 64 are attached, has a base pivot 90 attached thereto. Clevis pin 92 connects the base pivot 90 to clevis 94 which is threaded into shaft 96 of hydraulic cylinder 98. The lower end of hydraulic cylinder 98 is pivotally mounted through clevis pin 100 on block 102. Thus the hydraulic cylinder pivots about clevis pin 100 when the container assembly is tilted. Valve 104, which controls the operation of hydraulic cylinder 98, is mounted on bearing support strap 43. When the control arm 106 of valve 104 is disposed in the "up" position, oil flowing through line 108 moves the cylinder shaft 96 upwardly and moves the container assembly into vertical position. When the control arm 106 of valve 104 is disposed in the "down" position, as 106a, oil flowing through line 110 moves the cylinder shaft 96 downwardly and tilts the container assembly into a canted position. Oil is admitted to the valve 104 from supply manifold 112 through line 114. Oil is removed from the valve 104 through line 116 and return manifold 118. Electrical grounding of the entire apparatus, including the foil lid 14, the beaker 12 and pot chuck 24, is effected at 120 on one of the legs of table 42 (Fig. 2).

The exact composition of the materials used in the preparation of the ignition paste and their relative weights do not constitute a part of this invention and hence are not described herein in detail, it being well known in the art that the composition and the proportions thereof may be varied depending on the characteristics desired in any given case. In each instance, in accordance with the principles of this invention, a portion of the binder solution is poured into the stainless steel beaker 12 which contains a quantity of a mixing medium (not shown) such as one-half inch diameter porcelain balls for example. The beaker is then rotated in such a manner that all the balls and a major portion of the inside wall of the beaker are wetted. Then the dry metal powder is charged cautiously into the beaker. Finally, the remainder of the binder solution is poured into the beaker. The foil lid 14, supported by the concentric rings 16 and 18, is placed on top of the beaker and secured thereto by spring clips 22. After waiting for sev-

eral minutes to permit soaking of the metal powders by the binder solution, the beaker 12 is placed in the pot chuck 24, the spring fingers 26 offering sufficient resistance to the insertion of the container in the pot chuck to minimize the danger of dropping the container into the pot chuck.

Rotation of pot chuck 24 is now initiated. This is accomplished by moving lever 38 to the right (in Fig. 1) to effect a lowering of the pot chuck until the neck 32 thereof seats on friction drive washer 56 mounted on the upper end of collar 54. Since the lever 38 is provided with an inclined cam surface on which the cap 36 on the end of rod 30 rests, movement of lift lever 38 to the right eases the pot chuck 24 downwardly and the spring 34 contributes to this downward movement. When the neck 32 of the pot chuck 24 seats on the friction drive washer 56, rotation of drive shaft 44 is transmitted to the pot chuck through drive gear 48, driven gear 50, sleeve 52 and shaft collar 54 on which the friction drive washer 56 is mounted.

With the pot chuck 24 rotating, the entire pot chuck assembly is then tilted, to an angle of about forty-five degrees from the vertical for example, where it is disposed and maintained during the mixing operation. Tilting of the pot chuck assembly is accomplished by actuation of the shaft 96 of the hydraulic cylinder 98. When the control arm 106 of valve 104 is moved to the "down" position, as shown at 106a in Figures 1 and 2, oil under pressure is admitted into the cylinder 98 through line 110 and drives the shaft 96 downwardly within the cylinder. As the shaft 96 moves downwardly, the cylinder 98 swings clockwise (Fig. 2) pivoting about clevis pin 100. As the shaft 96 moves downwardly, the clevis 94, which is threaded into shaft 96, causes the base pivot 90 to effect a tilting of the pot chuck assembly away from an operator since the base pivot 90 is attached to channel iron support 60, the member to which both the bushing housing 58 and the guard support 64 are attached.

The tilted beaker 12 is rotated until the balls therein bring the mixture to a smooth suspension. In the specific example of a mixture described above, forty-five minutes has been found to be a satisfactory period of rotation at about 60 R. P. M. The quantity of the materials to be mixed is kept sufficiently small with respect to the size of the beaker so that at no time, even when the beaker is in its tilted position, do they contact the lid. This eliminates the necessity for cleaning the lid after each mixing cycle, as must be done when mixing is done in a ball mill jar.

What we claim is:

1. The method of preparing a highly flammable mixture of a zirconium metal powder and a cellulose binder solution which comprises: charging the powder and binder solution into a ball mill; affixing a ventable membrane lid to said ball mill; tilting said ball mill so that its longitudinal axis is disposed at an angle to the vertical; and rotating said ball mill while it is so angularly disposed to effect a mixing of the materials disposed therein.

2. The method of preparing a highly flammable mixture of a zirconium metal powder and a cellulose binder solution which comprises: pouring a portion of the binder solution into a ball mill; rotating said ball mill to effect a wetting of the mixing medium and the inside wall of said ball mill; charging the powder into said ball mill while it is maintained stationary; pouring the remainder of the binder solution into said ball mill; affixing a foil lid to said ball mill; tilting said ball mill so that its longitudinal axis is disposed at an angle to the vertical; and rotating said ball mill while it is so angularly disposed to effect a mixing of the materials disposed therein.

3. The method of preparing highly flammable ignition paste employed in the manufacture of photographic flash lamps from a zirconium metal powder and a cellulose binder solution which comprises: pouring a portion of the binder solution into a ball mill; rotating said ball

mill to effect a wetting of the mixing medium and the inside wall of said ball mill; charging the powder into said ball mill while it is maintained stationary; pouring the remainder of the binder solution into said ball mill; affixing a foil lid to said ball mill; tilting said ball mill so that its longitudinal axis is disposed at an angle to the vertical; and rotating said ball mill while it is so angularly disposed to effect a mixing of the materials disposed therein.

4. Apparatus for preparing highly flammable ignition paste employed in the manufacture of photographic flash lamps comprising: a container in which the materials to be mixed are disposed; a foil lid covering the top of said container; positive rotational drive means disposed beneath said container and spaced therefrom; means for lowering said container into frictional engagement with said positive rotational drive means to effect rotation of said container; and means for tilting said container so that its longitudinal axis is disposed at an angle to the vertical, while maintaining said container in frictional engagement with said positive rotational drive means to effect a mixing of the materials disposed therein.

5. Apparatus for preparing highly flammable ignition paste employed in the manufacture of photographic flash lamps comprising: a container in which the materials to be mixed are disposed; a foil lid covering the top of said container; helical gear drive means disposed beneath said container and spaced therefrom; means for lowering said container into frictional engagement with said helical gear drive means to effect rotation of said container; and means for tilting said container so that its longitudinal axis is disposed at an angle to the vertical, while maintaining said container in frictional engagement with said helical gear drive means to effect a mixing of the materials disposed therein.

6. Apparatus for preparing highly flammable ignition paste employed in the manufacture of photographic flash lamp comprising: a container in which the materials to

be mixed are disposed, said container having a rod depending therefrom; a tubular member concentric with said rod and through which said rod extends, said tubular member being spaced from the bottom of said container; means for rotating said tubular member; means for lowering said rod to move said container into frictional engagement with said tubular member to effect rotation of said container; and means for tilting said container so that its longitudinal axis is disposed at an angle to the vertical, while maintaining said container in frictional engagement with said tubular member to effect a mixing of the materials disposed therein.

7. Apparatus for preparing highly flammable ignition paste employed in the manufacture of photographic flash lamps comprising: a container in which the materials to be mixed are disposed, said container having a rod depending therefrom; a tubular member concentric with said rod and through which said rod extends, said tubular member being spaced from the bottom of said container; helical gear drive means mounted on said tubular member for effecting rotation thereof; means for lowering said rod to move said container into frictional engagement with said tubular member to effect rotation of said container; and means for tilting said container so that its longitudinal axis is disposed at an angle to the vertical, while maintaining said container in frictional engagement with said tubular member to effect a mixing of the materials disposed therein.

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