Aug. 25, 1964 E. STAHL 3,145,410

APPLICATOR APPARATUS

Filed May 7, 1962 3 Sheets-Sheet 1

FIG. 1.

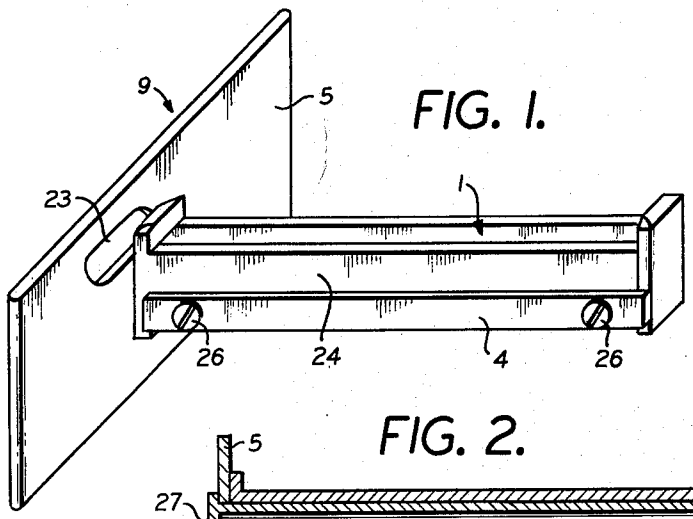


FIG. 2.

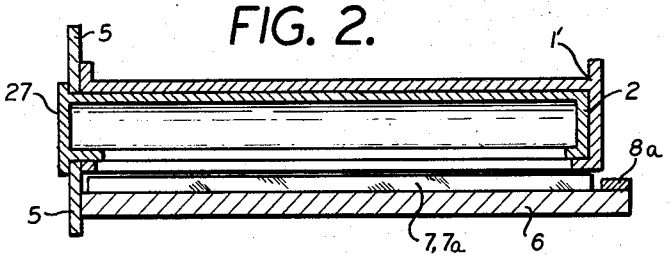


FIG. 3.

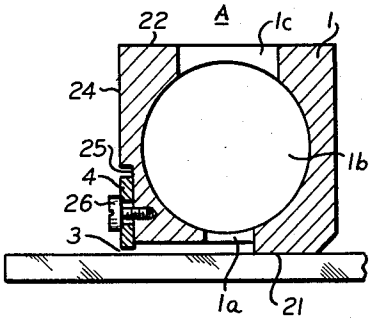


FIG. 4.

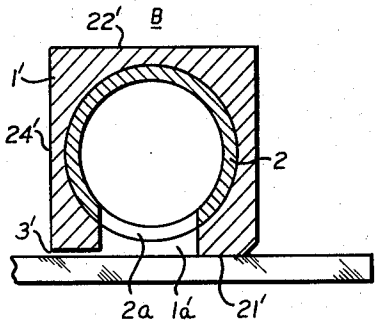
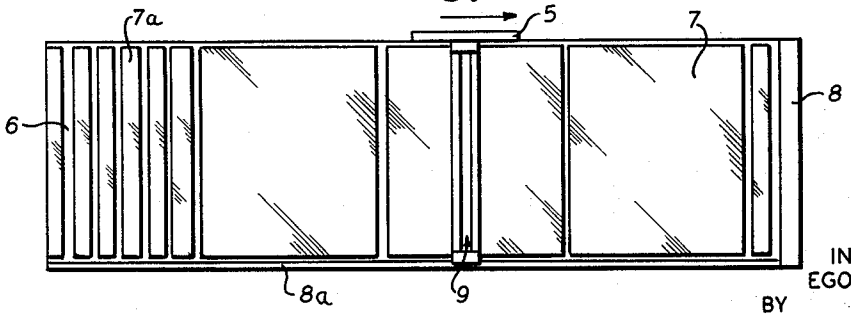


FIG. 5.



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APPLICATOR APPARATUS

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FIG. 6.

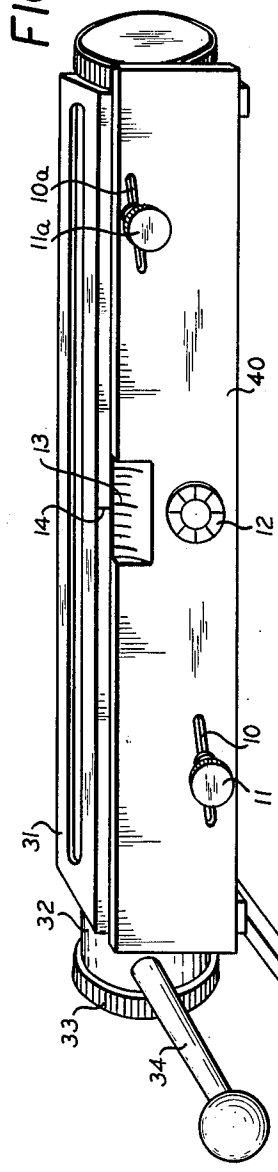


FIG. 8.

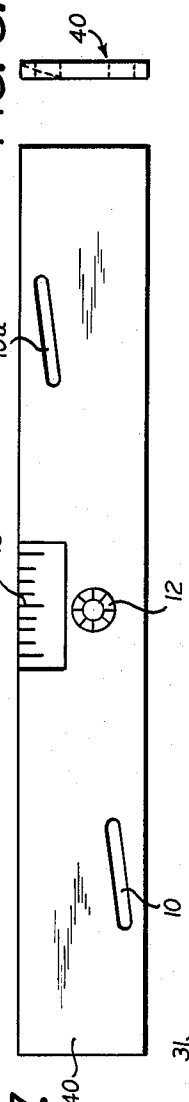


FIG. 7.

FIG. 9.

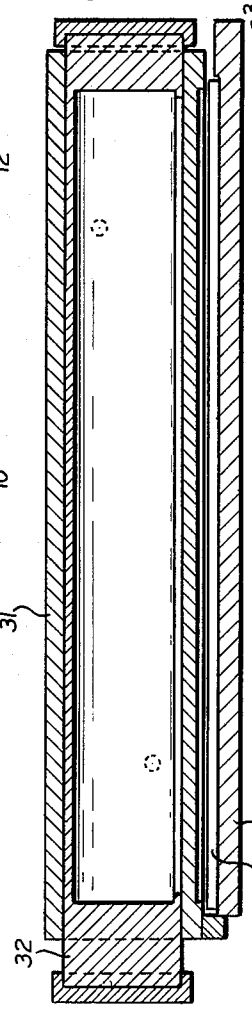
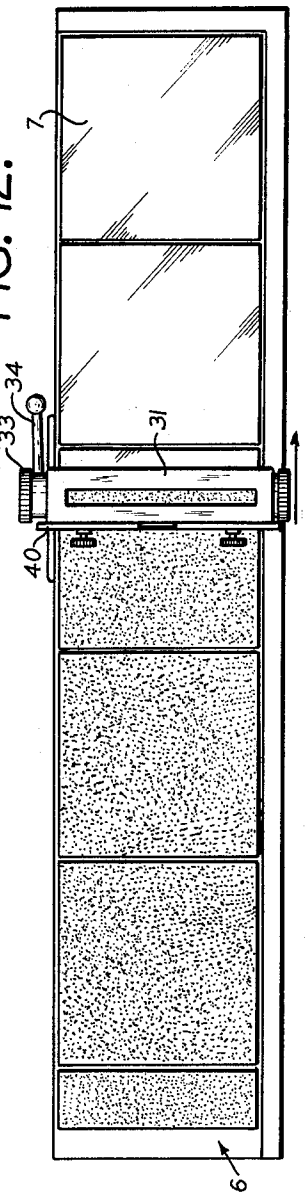


FIG. 12.



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APPLICATOR APPARATUS

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FIG. 13.

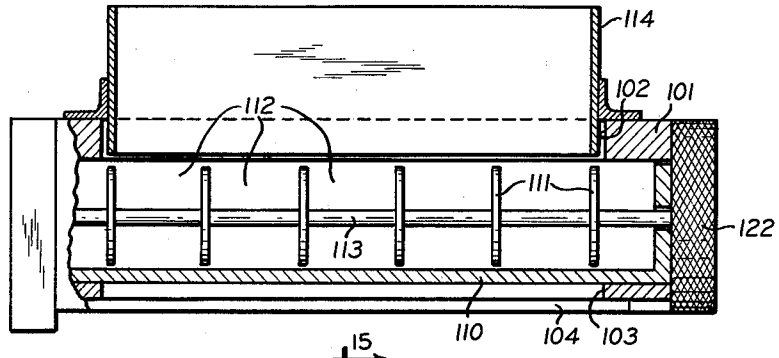


FIG. 14.

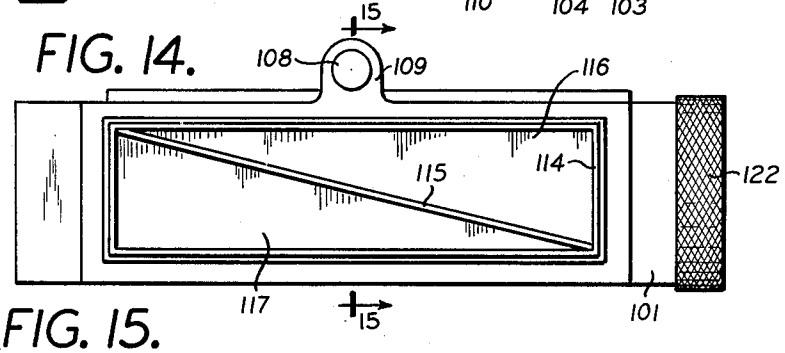


FIG. 15.

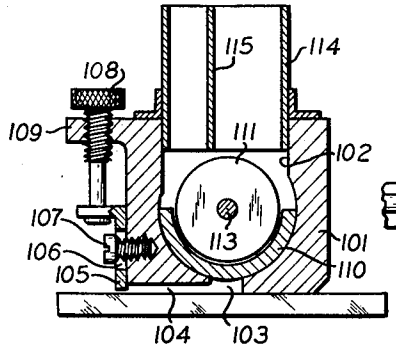


FIG. 16.

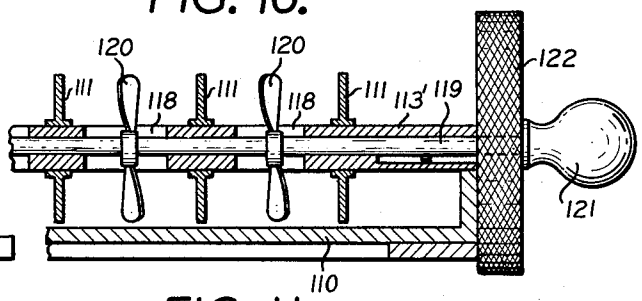


FIG. 10.

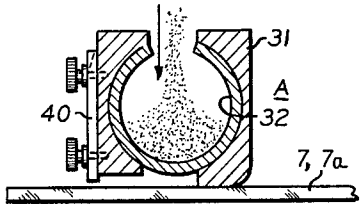
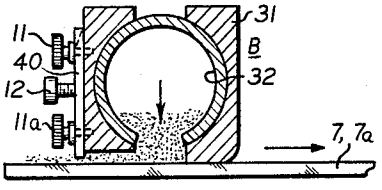


FIG. 11.



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3,145,410

APPLICATOR APPARATUS

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Claims priority, application, Germany, Dec. 6, 1961, St 18,646
14 Claims. (Cl. 15—510)

This invention relates to the application of thin layers onto flat plates or other flat surfaces, and more particularly to an improved device for applying thin layers of controllable and adjustable thickness to such plates or surfaces.

In various operations, such as that of chromatographically separating mixtures of chemical substances into thin sorption layers, means are required capable of applying uniform thickness layers of the materials or mixtures to the plates, the materials or mixtures at this time being either in paste form or highly viscous. Various arrangements are known for applying these layers, including one method in which a predetermined amount of a viscous mixture is applied to a glass plate and is thereafter thinned out with a suitable tool, such as a spatula, to a thickness of about 2 to 3 mm. Other arrangements have involved a funnel-shaped device secured to a base plate, and through which glass strips, having a width of about 1.5 cm., are pushed so that the plates will be coated with a layer of about 3 mm. thick. However, for chromatography employing very thin layers of materials, with thicknesses of between 20 and 30 microns, such techniques are not suitable.

Additionally, the higher development of thin layer chromatography, and particularly two-dimensional chromatography, has resulted in relatively large plates of square or polygonal shape being employed in the place of the previously used relatively narrow glass strips, these relatively large plates having a number of advantages. A prerequisite for successful chromatography with such relatively large plates is that the layers have a uniform thickness at all areas of the plates, and that there be no grooves, cleavages or beads on the layer surface so that the latter will be homogeneous and even. Known devices are not capable of covering a large plate with a layer of material of uniform thickness as, in all known prior art arrangements, there always occur frequent "set off" areas where the apparatus is applied to or lifted off from the plate. At these set off areas, the layer will be thicker than in other areas. Careful studies have indicated that difficulties of this nature cannot be overcome simply by enlarging known apparatus as, if the known apparatus and devices are enlarged, then the difficulties are increased.

Another known disadvantage of prior art devices, particularly in chromatography and in other applications where rather thin viscous masses are used, is that these thin pastes or masses cannot be applied with the known devices because they tend to flow out of the device before the application procedure is initiated. Additionally, prior art known devices for thin layer chromatography can be used only with strips having a predetermined width and, when the width of the strip is changed, another device has to be used which is constructed and dimensioned in accordance with the width of the new strip.

The present invention is directed to a device by means of which large square or polygonal plates, as well as a series arrangement of narrow or wide strips, either per se or in combination with quadratic plates, can have a relatively thin paste-like substance applied thereto in a uniform thickness layer in a single working operation. It is, accordingly, an object of the invention to provide a device which is capable of applying uniform thin layers of a relatively thin paste-like substance to relatively large area plates or surfaces.

While devices are known in other arts for applying a thin layer of material along a path of a predetermined width to be coated, it has been found that these are not particularly applicable to applying thin layers of the type just mentioned above and consisting of relatively thin or free-flowing material. Also, these known devices are not effective for a large volume operation wherein it is desired to coat a relatively large number of strips or plates in a relatively short time. This latter factor is important, particularly when using silica-gypsum materials which solidify rapidly, or when using any other materials or mixtures which solidify rapidly.

Thus, another object of the invention is to provide an applicator having a substantially flat undersurface in which there is formed an accurately dimensioned horizontally extending outwardly opening notch formed by undercutting the relatively flat support surface. This notch constitutes a discharge notch for the material to be applied to a glass strip or plate of any desired width and which rests on a base plate, in such a manner that the glass plate can be coated with a uniformly thick layer of the material in a single operation and in a relatively short time.

A further object of the invention is to provide such an applicator including a sleeve rotatably mounted in the applicator and movable between a material receiving position and a material discharge position, this sleeve having a slot extending longitudinally thereof and having parallel edges and which may be selectively wholly or partially registered with a substantially rectangular slot in the applicator and leading to the horizontal discharge notch. By relative angular adjustment of the sleeve, the rate of outflow of the material may be accurately controlled. Furthermore, due to the provision of this angularly adjustable sleeve, the pressure head of the material in the applicator does not act entirely at the horizontal exit notch, and by rotating the sleeve to a position in which its slot is out of registry with the slot leading to the horizontal discharge notch, the material can be retained against running out of the applicator except when it is desired to discharge the same. Furthermore, with the sleeve "closed," the applicator may be lifted from the base plate without any material running out of the applicator.

A still further object of the invention is to provide an applicator of the foregoing type in which a vertically adjustable slide is provided to control the effective vertical extent of the discharge opening of the horizontal discharge notch. Thereby, the thickness of the layer of material applied to the plate can be very accurately controlled.

Still a further object of the invention is to provide novel means, including cooperating indicating means, for accurately adjusting the position of this slide in accordance with the desired thickness of the layer of material to be applied.

A still further object of the invention is to provide such an applicator in which the composition of the material may be gradually and uniformly changed throughout the length of the working stroke of the applicator.

Another object of the invention is to provide such an applicator including agitator means operable to maintain the material in the applicator thoroughly mixed to prevent settling and sedimentation thereof.

A still further object of the invention is to provide such an applicator in which the mixture may be prepared within the applicator and then discharged in a regulated manner therefrom.

For an understanding of the principles of the invention, reference is made to the following description of typical embodiments thereof as illustrated in the accompanying drawings.

In the drawings:

FIG. 1 is a perspective view of one form of applicator embodying the invention;

FIG. 2 is a longitudinal sectional view through another form of applicator embodying the invention, and including an angularly adjustable flow control sleeve;

FIG. 3 is a transverse sectional view through the applicator shown in FIG. 1;

FIG. 4 is a transverse sectional view through the applicator shown in FIG. 2;

FIG. 5 is a plan view illustrating the base plate with the plates to be coated arranged thereon, and further illustrating the applicator in operative association with the base plate;

FIG. 6 is a perspective view of another form of applicator embodying the invention;

FIG. 7 is an elevation view of a control slide used in the applicator shown in FIG. 6;

FIG. 8 is an end elevation view of the control slide shown in FIG. 7;

FIG. 9 is a longitudinal sectional view through the applicator shown in FIG. 6;

FIGS. 10 and 11 are transverse sectional views through the applicator shown in FIG. 6, FIG. 10 illustrating the angularly adjustable sleeve in the material receiving position and FIG. 11 illustrating the angularly adjustable sleeve in the material discharging position;

FIG. 12 is a plan view of the base plate with the plates to be coated arranged thereon, and illustrating the applicator of FIG. 6;

FIG. 13 is a longitudinal sectional view through another form of applicator embodying the invention;

FIG. 14 is a plan view of the applicator shown in FIG. 13;

FIG. 15 is a transverse sectional view of the applicator, taken on the line 15—15 of FIG. 14; and

FIG. 16 is a longitudinal sectional view through an applicator of the type shown in FIGS. 13 and 14, and illustrating an agitator device.

Referring first to the embodiments of the invention shown in FIGS. 1 through 5, each applicator includes a relatively elongated body, of essentially quadrilateral or other polygonal cross section, illustrated at **1** (FIGS. 1 and 3) and **1'** (FIGS. 2 and 4), and having a flat undersurface **21** or **21'**. Each block **1**, **1'** may be made of metal, synthetic resin, or any other desired material, and is formed with a cylindrical bore **1***b* extending longitudinally thereof. In the embodiment of the invention shown in FIGS. 1 and 3, the block **1** is formed with a preferably rectangular and longitudinally extending discharge slot **1***a*, opening through the bottom surface **21** of the block, and with a filling slot **1***c* opening through the top surface **22** of the block.

The applicator as a whole is indicated at **9**, and each block **1** or **1'** has secured to one end thereof a relatively large handle plate **5** provided with an opening **23**. Plate **5** is arranged to engage one longitudinal edge of a support surface or plate **6** on which the plates **7** and the relatively narrow strips **7***a* are positioned, locating strips **8** and **8***a* being provided to properly position the plates **7** and the strips **7***a* on the support surface **6**, as best seen in FIGS. 2 and 5.

In the embodiment shown in both FIGS. 1 and 2, the undersurface **21** or **21'** of the block **1** or **1'** is undercut to form a horizontally extending notch **3** or **3'** which is preferably rectangular in plan and opens through a side surface of the block. Material within the block **1**, as in the bore **1***b* of FIGS. 1 and 3, will flow through the slot **1***a* into the notch **3** and will be distributed by the latter as a thin layer over the surface of the plates **7** and **7***a*. The thickness of the layer of material thus distributed is controlled, in the embodiment shown in FIGS. 1 and 3, by a slide **4** which is vertically adjustable in a notch **25** in a side surface **24** of the block **1**, the slide **4** being held in adjustable position by clamping bolts or screws **26**.

The block **1'** shown in FIGS. 2 and 4 is essentially the same as the block **1** of FIGS. 1 and 3, but in this case the upper surface **22'** of the block is not formed with a filling opening. Instead thereof, an angularly adjustable cylindrical sleeve **2** is rotatably mounted within the bore and may have an enlarged end **27** for relative angular adjustment of the sleeve. This sleeve **2** is formed with a longitudinally extending substantially rectangular slot **2***a* which is selectively registrable, to a greater or less degree, with the slot **1***a'* opening through the lower surface **21'** of the block **1**. Thereby, the sleeve **2** may be rotated to a position in which the slot **2***a* is in full registry with the slot **1***a'*, and with the block **1'** in the inverted position the material to be applied as a layer may be discharged into the sleeve **2**. The sleeve **2** may then be rotated so that the slot **1***a'* is completely blocked, so that the material may thereby be retained within the applicator at all times. To discharge material from the applicator, the sleeve **2** is rotated to bring the slot **2***a* into a desired degree of registry with the slot **1***a'* in the block **1'**, the degree of registry being determined by the viscosity of the material and the desired thickness of the layer to be applied. In this case, the thickness of the layer applied onto the plates **7** and **7***a* is determined by the spacing or height of the horizontal discharge slot **3'**. For example, the height of this slot may be a few millimeters, in one case, or in another case it may be 250 microns, which is optimum for many purposes.

It should be noted that the guide strips **8** and **8***a*, which are arranged at right angles to each other, preferably have a thickness as shown in FIG. 2, which is less than that of the plates **7** and **7***a* to which the coating or layer is to be applied. The length of the plate **6** is selected in accordance with the desired operating stroke, and a length of about 1 meter will generally be sufficient. With a base plate or support surface **6** having a length of about 1 meter, about 65 strips each 1.5 cm. in width, or 40 strips each 2.5 cm. in width, can be coated in a single operation. Alternatively, 5 quadratic plates 20 cm. wide can be coated in a single operation.

While the invention is particularly applicable to the coating of thin sorption layers for chromatography, it will be understood that it may well be applied to any other operation in which layers of a predetermined thickness are to be applied to a flat relatively wide surface in a single operation. It is particularly applicable where relatively thin mixtures are to be applied onto carrier plates. Examples of this are the production of thin agar or starch gel layers, such as used in electrophoresis in order to determine the characteristics of colored or uncolored layers of synthetic material. Furthermore, the applicator can be used for applying thin layers of powder which are to be spread in a thin and uniform layer, such as used in measuring reflection properties.

Referring to the embodiment of the invention shown in FIGS. 6 through 12, the block **31** illustrated therein corresponds substantially to the block **1'** previously described, and is provided with a cylindrical slotted sleeve **32** which is substantially identical to the sleeve **2**. The sleeve **32** may be provided with an operating knob **33**, which is preferably knurled, and with an operating handle **34**, as best seen in FIG. 6.

In this embodiment of the invention, the adjustable slide **40** is formed with a pair of obliquely extending and relatively aligned longitudinal slots **10** and **10***a* through which extend bolts **11** and **11***a* which are threaded into the block **31**. A clamping bolt **12** is provided for securing the slide in adjusted position. By loosening the bolts **11**, **11***a* and **12**, the slide may be moved to the right or to the left to raise or lower the same, after which the several bolts may be tightened. A scale **13** is provided on the slide **40** and is cooperable with a fixed indicating mark **41** on the body **31**, and this scale **13** may be calibrated in millimeters indicating the height of the lower edge of the slide **40** above the surface or plate to be coated.

In some operations, it is desired to apply the coating in such a manner that the effective characteristics or composition of the coating will vary in a continuous manner, or in a quasi-continuous manner, such as stepwise, throughout the length of the operating stroke. For example, it may be desired to attain a stepwise variation of the basicity of the sorption layer. In such case, the characteristic of the coating may vary from a strongly acid condition through the neutral point to a strongly basic condition, or, alternatively, the variation can be within a certain range of pH values. As another typical operation, part of the sorption band may contain an active sorption agent such as aluminum oxide or silica gel, whose concentration is increased toward one end of the sorption band and decreased toward the other end thereof, while the concentration of an inactive sorption agent, such as cellulose, may be increased in correspondence with the decrease in the active sorption agent. The variation in the layer may take place from left to right or vice-versa along the length of the layer, or it may vary throughout the length. Furthermore, the variation may be either continuous or stepwise.

FIGS. 13 through 16 show an applicator by which such variable composition or variable characteristics of the applied layer can be attained. Generally speaking, in this embodiment of the invention, the angularly adjustable sleeve is subdivided by axially spaced partitions into a plurality of adjacent chambers, while the filling slot or opening of the block member is divided by a diagonally extending partition, and which may form part of a detachable funnel arrangement for filling the applicator. Thus, a relatively acid mass can be placed in one compartment of the funnel and a relatively basic mass in the other compartment, the one compartment decreasing in width from one end of the applicator to the other and the other compartment increasing in width from one end of the applicator to the other. Thus, the relative proportions of basic material and acid material in the respective compartments of the sleeve will be varied continuously throughout the axial length of the sleeve.

Preferably, a hollow shaft extends through the partitions in the sleeve, and a rod is reciprocable axially of this hollow shaft. This rod has agitating wings extending therefrom, through slots, in the hollow or tubular shaft, into each of the separate compartments. Thus, by rotating or reciprocating the rod, the individual masses can be kept thoroughly mixed and prevented from sedimentation.

In FIGS. 13 through 16, the block **101** is formed, in the same manner as previously described, with a filling slot **102** and a discharge slot **103** which leads to a horizontally extending undercut notch **104** in the undersurface of the block, such as described in connection with FIGS. 1 through 5. The discharge slot **103** opens into such undercut portion **104**. Regulation of the thickness of the layer applied to the material is effected by a vertically adjustable slide **105** which has vertical slots **106** receiving screws or bolts **107**, and which is vertically adjusted by a micrometer screw **108** threaded through an eye or opening in an ear **109** of the applicator block. By observation of the reading of the micrometer screw **108**, the height of the lower edge of the slot **105** above the upper surface of the plates **7** or **7***a* to be coated can be readily and accurately adjusted and set.

A semi-cylindrical sleeve **110** is angularly adjustably mounted in a cylindrical bore in the block **101** and is provided with a knurled operating knob **122**. This sleeve can be brought selectively into alignment or registry with a filling slot **102** in the block **101**, or into any desired degree of registry with the discharge slot **103**. The interior of the sleeve **110** is divided into individual chambers **112** by means of axially spaced discs **111** fixed on a shaft **113**.

A filling funnel **114** is disengageably mounted in registry with the slot **102** of the block **101**, and is divided into two chambers by a diagonally or obliquely extending partition **115**. By means of the partition **115**, a pair of compartments **116** and **117** are provided which reversely vary in width throughout the length of the slot **102**. Thus, an acid phase of the material may be placed into the space **116** of the funnel, and a basic phase of the same material may be supplied to the space **117**. Thereby, chambers **112** in the sleeve **110**, formed by the discs **111**, will contain variable proportions of the basic and acid phases of the material, considered longitudinally of the slot **102**, and the mixing in each chamber is in a predetermined proportion which is preset by the diagonal partition **115** and the number of chambers **112**. Thus, the mixture may vary in pH values from an acid phase through a neutral phase to a basic phase, serially of the chambers **112**.

In order to maintain good mixing with individual chambers **112**, and as best shown in FIG. 16, the shaft extending axially of the sleeve **110** may be a tubular shaft **113'** having slots **118** opening through its wall in each of the chambers **112**. A rod **119** is mounted through the tubular shaft **113'** for rotation and axial reciprocation, and has vanes or blades **120** extending upwardly through the slots **118** into the chambers **112**. The rod **119** may be provided with an operating knob or head **121** so that it may be readily rotated, oscillated or axially reciprocated. By virtue of this arrangement, the material in the different chambers **112** may be thoroughly and continuously agitated to prevent any sedimentation thereof. As an advantageous feature, the slots **118** may extend helically so that, upon a reciprocation of the rod **119**, the vanes **120** may be oscillated through a predetermined angle.

While specific embodiments of the invention have been shown and described in detail to illustrate the application of the principles of the invention, it will be understood that the invention may be embodied otherwise without departing from such principles.

What is claimed is:

1. Apparatus for applying thin, uniform thickness layers of flowable material to the relatively flat upper surfaces of plural plates of equal thickness comprising, in combination, means providing a substantially flat support surface on which said plates are positioned in adjacent relation; a relatively elongated applicator body positionable to extend transversely of the support surface and having a substantially flat undersurface engageable with said plates; said undersurface being undercut to form a substantially horizontal discharge notch opening through a longitudinal edge of the body, and said body having a substantially cylindrical bore extending longitudinally thereof and being formed with an elongated slot connecting said bore to said discharge notch for flow of material from the interior of said bore outwardly through said discharge notch; and means, including said discharge notch, regulating the thickness of the layer material discharged through said discharge notch.

2. Apparatus as claimed in claim 1, including a substantially cylindrical sleeve extending coaxially of said bore and angularly adjustable therein; said sleeve having a slot extending longitudinally thereof and selectively registrable, to a predetermined degree, with said slot connected to said discharge notch to regulate the flow area of material from said bore into said discharge notch.

3. Apparatus as claimed in claim 2, in which said body has an upper surface formed with a material filling slot extending longitudinally thereof; said sleeve being angularly adjustable to register its slot with said filling slot for filling material into said sleeve.

4. Apparatus as claimed in claim 1, in which said longitudinal edge is the lower edge of another surface of said body extending upwardly from said undersurface; and a relatively elongated flat slide mounted on said upwardly extending surface for adjustment vertically thereof to regulate the effective flow height of said discharge notch.

5. Apparatus as claimed in claim 4, in which said slide is formed with a pair of longitudinally spaced obliquely extending and aligned slots therein; clamping means extending through said slots and engaged in said block; whereby, by loosening said clamping means and moving said slide longitudinally of said block, the effective height of said slide may be readily adjusted.

6. Apparatus as claimed in claim 5, including cooperating scale and indicia means on said slide and said body for indicating the adjusted height of said slide.

7. Apparatus as claimed in claim 1, in which said means providing a substantially flat support surface is a supporting plate; and guide means secured to at least one end of said body and engageable with a rectilinear edge of said support plate for guided movement of said body along said support plate.

8. Apparatus as claimed in claim 7, including positioning ledges on said flat support surface engageable by said plates for location thereof on said flat support surface, said positioning ledges having a height above said flat support surface less than the thickness of said plates.

9. Apparatus as claimed in claim 3, including plural axially spaced partitions subdividing the interior of said sleeve into axially adjacent chambers; and a diagonally extending partition operatively associated with said filling slot to subdivide the latter into two filling openings each communicating with each of said chambers.

10. Apparatus as claimed in claim 9, including a filling funnel disengageably securable in registry with said filling slot, said diagonally extending partition being provided in said funnel.

11. Apparatus as claimed in claim 9, in which said partitions are secured to a shaft extending coaxially of said sleeve.

12. Apparatus as claimed in claim 9, including agitator means disposed in each of said chambers; and externally accessible means operable to conjointly operate said agitator means.

13. Apparatus as claimed in claim 11, in which said shaft is tubular and is formed with a pair of opposing slots in each of said chambers; an agitator rod extending through said tubular shaft and reciprocable axially thereof; and blades on said agitator shaft extending through said last-named slots into said chambers.

14. Apparatus as claimed in claim 13, including an operator on said agitator shaft externally of said body.

References Cited in the file of this patent

UNITED STATES PATENTS

95,974	Benton	Oct. 19, 1869
2,022,733	Parks	Dec. 3, 1935
2,108,952	Urban	Feb. 22, 1938
2,303,120	Greaves-Walker	Nov. 24, 1942
2,526,991	Biddle	Oct. 24, 1950