United States Patent

[11] 3,522,792

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[45] Patented: **Aug. 4, 1970**
[73] Assignee: **The United States of America as represented by the Secretary of Agriculture**

[54] **DEVICE FOR APPLYING A COATING OF VARYING THICKNESS**
1 Claim, Drawing Figs.

[52] **U.S. Cl.** **118/415**
[51] **Int. Cl.** **B05c 3/18**
[50] **Field of Search** 118/415; 25/118; 401/5; 401/265

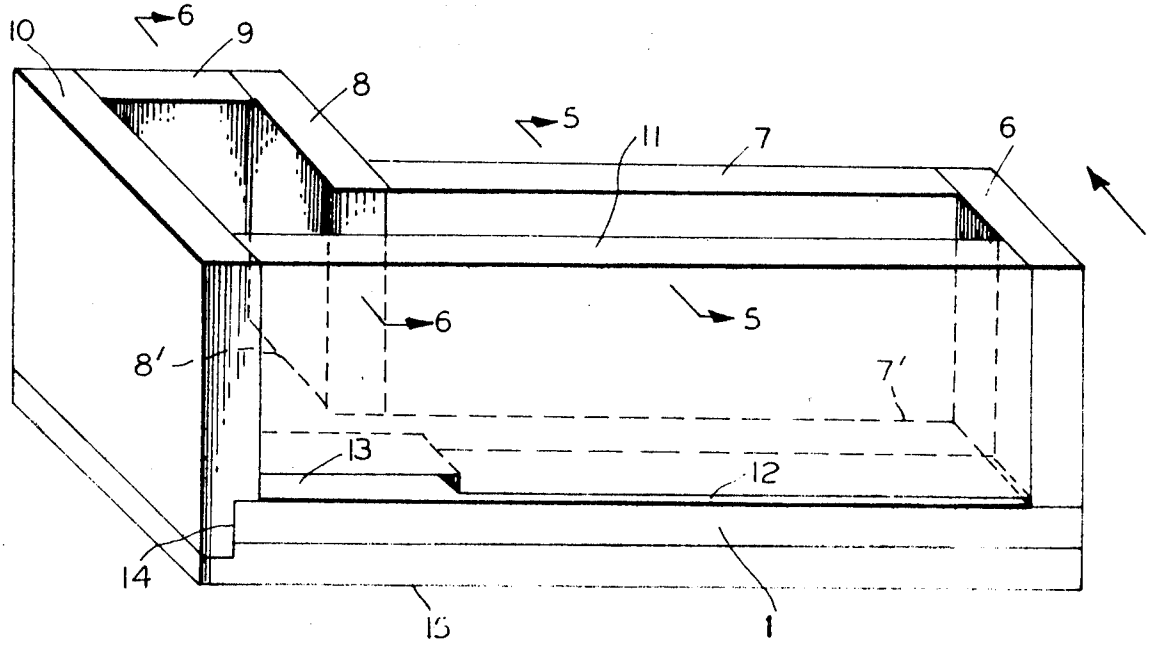
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ABSTRACT: An L-shaped laterally flanged coating device having vertical walls and adapted to be moved over a substrate. The bottom edge of the trailing wall is spaced from the substrate a distance to provide a thin coating for about 75-80 percent of its length while the remainder of the trailing wall is further elevated from the substrate by a factor of four to five. The portion of the wall opposite the enlarged gap being recessed to provide additional coating capacity for the enlarged gap area.



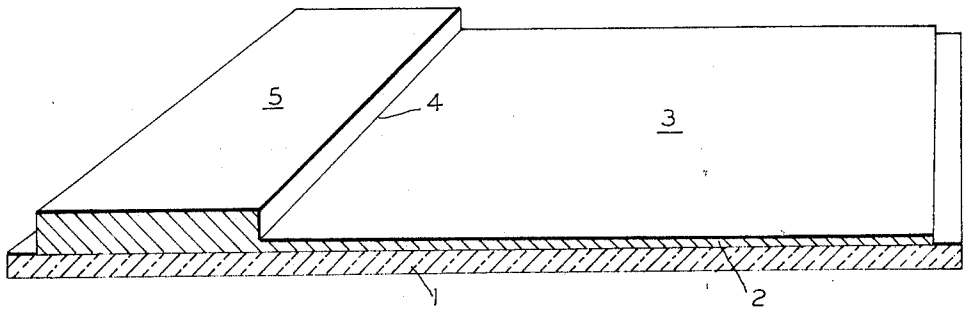


FIG. 1

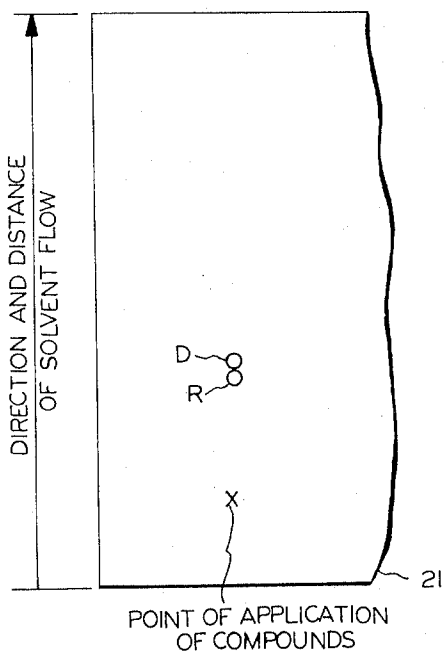


FIG. 3B

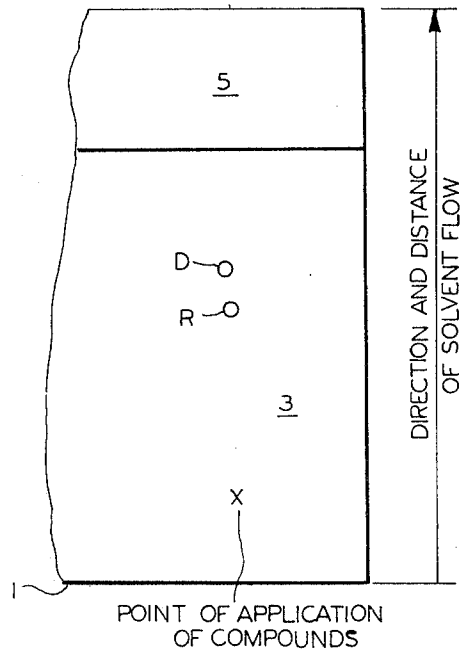


FIG. 3A

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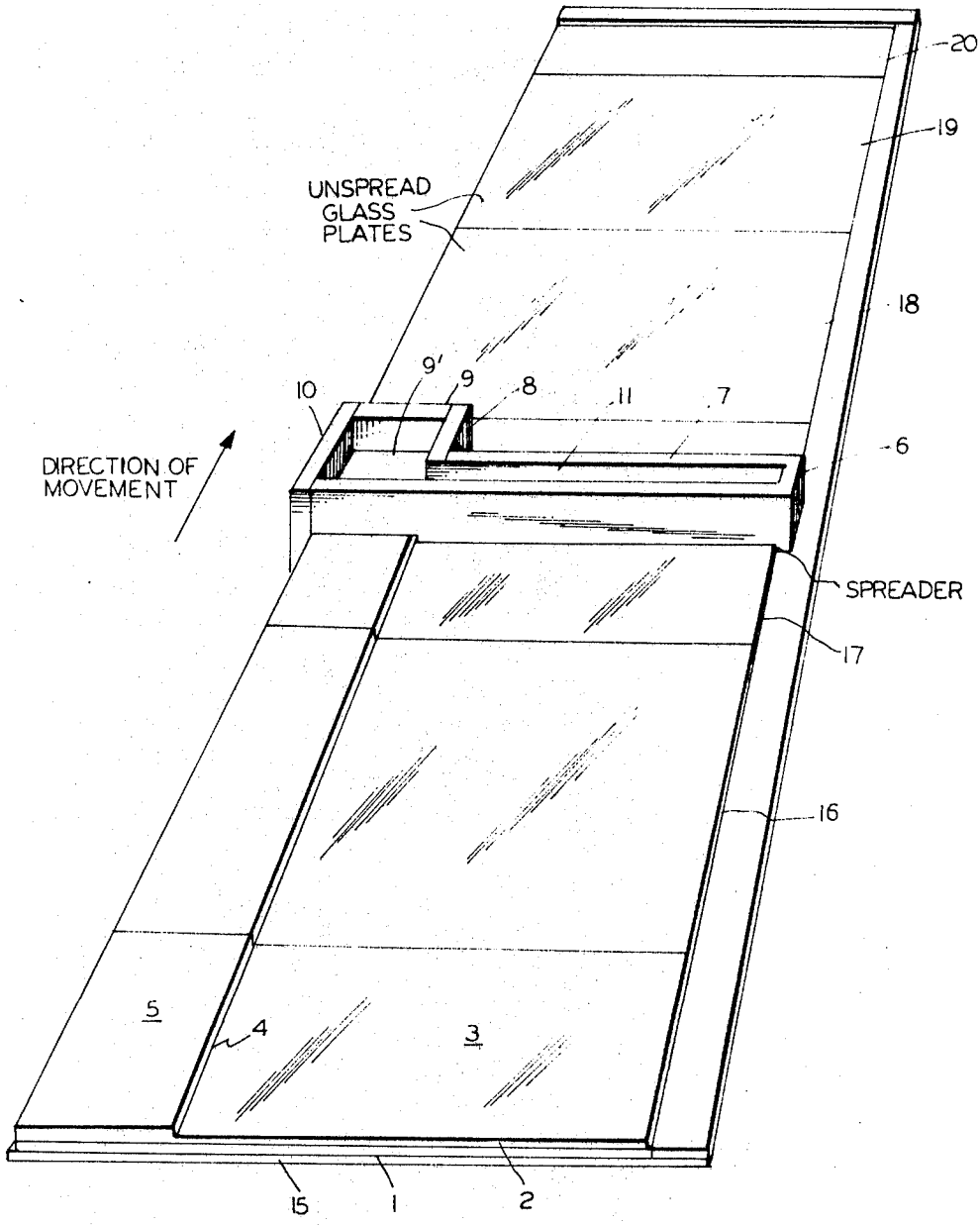


FIG.2

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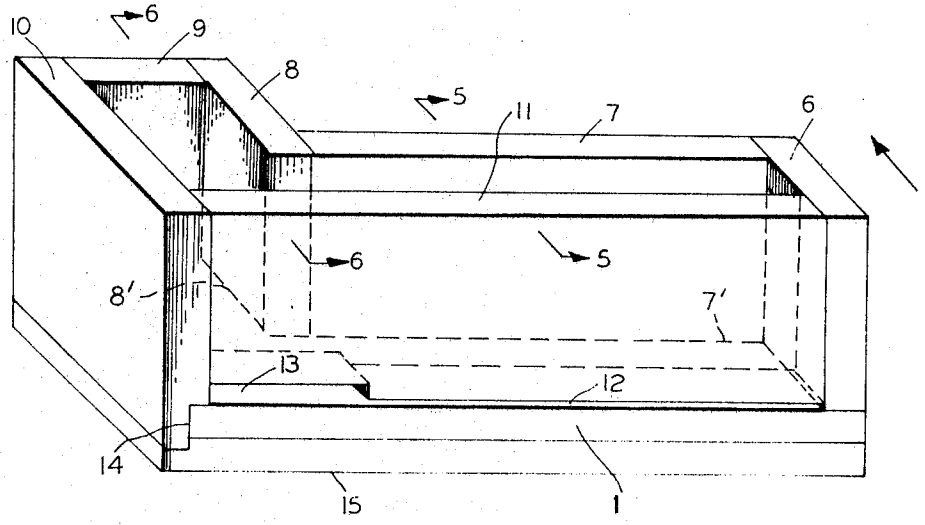


FIG.4

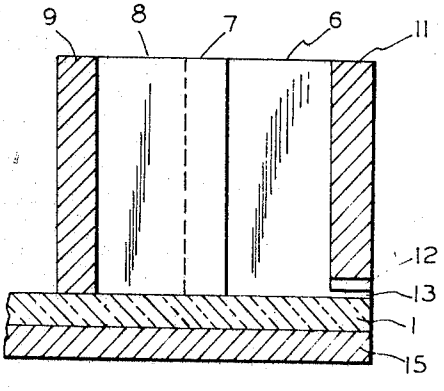


FIG.6

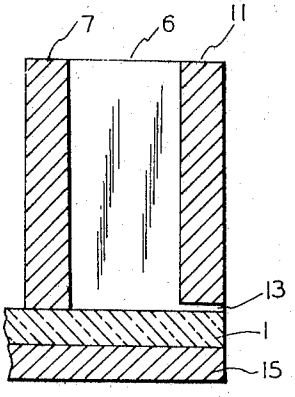


FIG.5

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U.S. PATENT 3,522,792

DEVICE FOR APPLYING A COATING OF VARYING THICKNESS

The invention relates to a novel spreader for simultaneously depositing the adsorbent in the form of two contiguous zones characterized by inversely related and distinctly different widths and depths of adsorbent, whereby is produced my improved dual zones TLC plate. In general, the spreader comprises a vertically walled, otherwise open enclosure having a reversed open block L-shaped configuration, which enclosure becomes a bottomed reservoir or hopper when the empty enclosure is placed at the margins of a gap-free series of the uncoated plates (which temporarily serve as a bottom for the enclosure in addition to being the substrate to be coated). When the reservoir is filled with a slurry comprising a siliceous adsorbent, e.g., purified silica gel, and is manually advanced to the opposite end of the series of glass plates, there are deposited simultaneously, and in only single transit, both the wide normal depth band and immediately adjacent thereto a correspondingly narrowed but much more deeply coated band.

Although a more detailed description of the adsorbent spreading device for preparing my novel dual zone TLC plates will be given in connection with the drawings, the device may be broadly described as being a somewhat L-shaped, vertically-walled enclosure, a functional bottom for which is temporarily provided by placement upon the plate that is to be coated. The lower (functionally trailing) edge of the longest wall is raised above the plate just enough to permit depositing a conventionally thin layer of silica gel excepting for a minor (relatively narrow) terminal segment where the edge of the spreader is abruptly further raised so as to form a gap that is capable of depositing preferably about four times as great a depth of adsorbent, the polydimensional gap thus being capable of simultaneously forming a wide major zone of adsorbent having the usual TLC depth and, in continuous side-by-side adjacency thereto, a much narrower but correspondingly more heavily deposited strip or zone. To provide a properly increased supply of silica gel adsorbent for the more deeply layered zone, the hopper is suitably enlarged across from but directly in line with the more widely open gap segment, thereby giving the L-shaped configuration of the hopper.

With reference to the drawings, **FIGURE** 1 represents an isometric view of a dual-zone TLC plate according to the present invention. **FIGURE** 2 represents an isometric view of the partially emptied dual discharge hopper of the invention in place on the surface of a series of side-by-side glass plates over which it has completed a partial transit. **FIGURE** 3A is a representation of the complete resolution of the rotenone-deguelin mixture referred to in column 2 hereof using the dual zone TLC plate of the invention while **FIGURE** 3B is a representation of the overlapping resolution obtained on a conventionally coated **20** cm. TLC plate. **FIGURE** 4 is an isometric view of the applicator. **FIGURE** 5 is a section taken along lines **5-5** of **FIGURE** 4. **FIGURE** 6 is a section taken along lines **6-6** of **FIGURE** 4.

As seen in **FIGURE** 1, the major portion of glass plate substrate **1** is coated with a uniformly thin layer **2** of siliceous adsorbent to provide thin layer zone **3** which at demarcation line **4** is abruptly thickened to provide thickly coated narrow zone **5**.

Referring to **FIGURE** 2, it will be seen that the hopper comprises six successively integral vertical walls, respectively, **6**, **7**, **8**, **9**, **10**, and **11**, which together define an enclosure having a configuration approximating that of a block letter upper case L. Wall **11** is the trailing wall when spreader is moved across the plates and is distinguished by its failure to extend downward quite to the reference plane of the glass that is to be coated and also to the plane of the lower edges of walls **6**, **7**, **8**, and **9** (shown as **6'**, **7'**, **8'**, and **9'**) whereby are formed, with reference to the said plane, a nearly full width standard aperture or gap **12** and continuous therewith, abruptly widened

short segment aperture **13**. Vertical wall **10** not only contributes to the formation of said segmental aperture but also extends further to define flangelike edge **14**, which extends below the glass plate and serves as a guide or stop for maintaining the spreader device in position.

Although I prefer a **4:1** ratio of the respective gap or aperture widths with inverse **1:4** ratios of gap heights, the invention is not strictly limited to these values which will be influenced by the particular compounds to be separated, the adsorbent to be deposited on the glass plates and the solvent system.

Those skilled in the preparation of uniformly coated TLC plates will recognize the impractability of making one or two plates at a time and are accustomed to place about a half-dozen or so uncoated plates in successive side-by-side contact on a flanged support and then progressively coating the series of plates with a uniformly thin layer of adsorbent by moving the spreader only once from one end of the series to the other. The dual zone spreader of the invention is employed in an identical manner to provide the dual zone TLC plates.

To illustrate the beneficial operativeness of my dual zone TLC plates, **10** microliter amounts of an acetone solution containing per milliliter **1** mg. of rotenone and **1** mg. of deguelin were spotted **3** cm. from the bottom of each of two **20** cm. x **10** cm. TLC plates, one plate being conventional in having been uniformly coated with a subsequently dried **0.25** mm. thickness of a silica gel slurry in **12%** $AgNO_3$ (**1** g. silica/**2**ml. of the $AgNO_3$ solution) whereas the other plate was identical with respect to the presence of a **0.25** mm. depth silica gel major zone but differed in that the deposit of silica gel on the last **5** cm. of the **20** cm. length of the plate furthest from the source of solvent was abruptly increased by a factor of four to a depth of **1** mm. The plates were exposed for one hour to a solvent system consisting of chloroform containing **2.5%** acetone and **0.5%** acetic acid, the conventionally (**0.25** mm.) coated end of the dual zone plate being introduced into the solvent mixture. Then to develop the color, the plates were in turn exposed first to HNO_3 vapor and then to NH_3 vapor. On the conventional TLC plate the centers of the two spots were only **0.9** cm. apart, and the resulting overlap prevented effective use of an optical desitometer. By contrast the **1.5** cm. center-to-center distance of the developed spots on the dual zone plate avoided overlapping of the spots and permitted an accurate quantitation.

A nonexclusive, irrevocable, royalty-free license in the invention herein described, throughout the world for all purposes of the United States Government, with the power to grant sublicenses for such purposes, is hereby granted to the Government of the United States of America.

I claim:

1. A laterally flanged spreader adapted for being easily moved across, and simultaneously depositing a silica gel coating from gaps in the bottom of a trailing wall of said moving spreader onto a substrate, said coating being in the form of two abutting zones that have different thicknesses that are independently uniform with reference to the substrate as well as unequal widths that are inversely proportional to the relative thicknesses of the coatings thereon, said spreader being a vertically walled, open, generally L-shaped hexagonal receptacle having a leading wall and a trailing wall, for which a functional bottom is obtained by placement on the substrate to be coated, about **75-80** percent of the length of the bottom edge of said trailing wall being only sufficiently elevated with reference to said substrate to form a thin gap that will deposit a TLC thickness of the silica gel coating material, the remainder of said trailing wall being further elevated with reference to said substrate by a factor of four to five, the portion of receptacle wall directly opposite the enlarged gap segment being recessed four to five times the wall opposite the thin gap so that the portion of the receptacle that supplies the enlarged gap segment has sufficient capacity to provide a proper amount of silica gel coating material.