

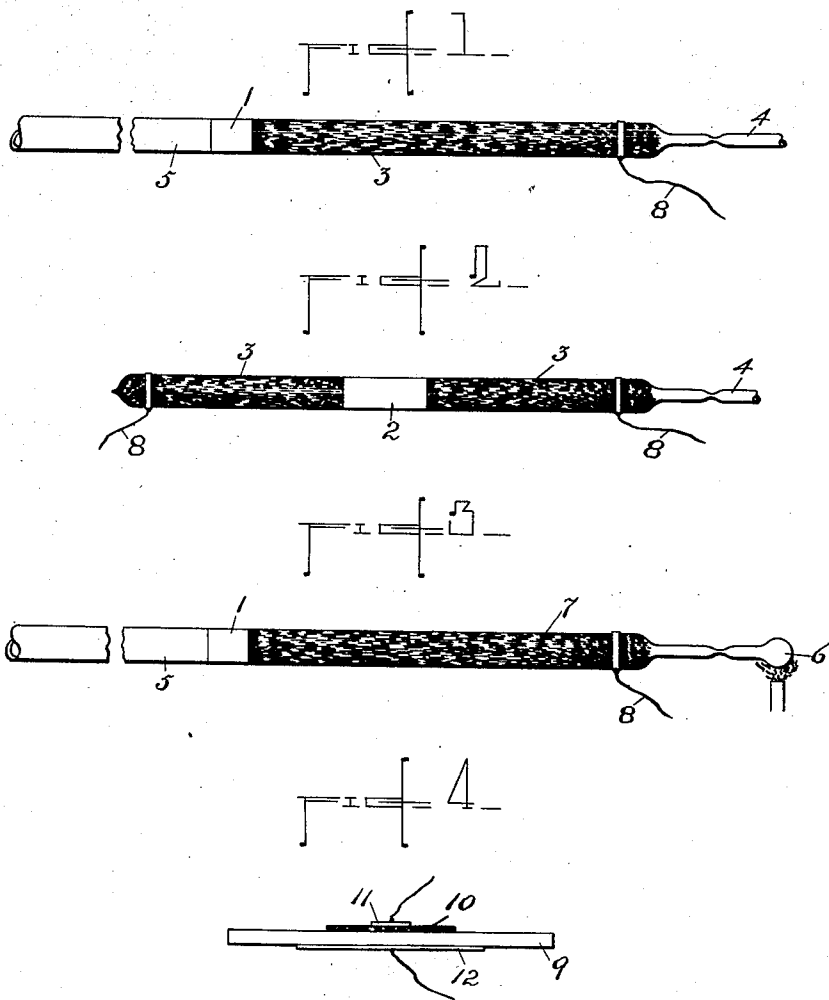
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D. McF. MOORE.

METHOD OF PRODUCING VACUUM LAMPS.

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WITNESSES:

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# UNITED STATES PATENT OFFICE.

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## METHOD OF PRODUCING VACUUM-LAMPS.

No. 814,794.

Specification of Letters Patent.

Patented March 13, 1906.

Original application filed September 2, 1903, Serial No. 171,587. Divided and this application filed April 21, 1904. Serial No. 204,173.

*To all whom it may concern:*

Be it known that I, DANIEL MCFARLAN MOORE, a citizen of the United States, and a resident of Newark, in the county of Essex and State of New Jersey, have invented certain new and useful Improvements in Methods of Producing Vacuum-Lamps, of which the following is a specification.

My present invention relates to an improved method of producing electrically-operated gas or vapor tubes; and it consists more particularly in a method of manufacture whereby a tube may be produced having a contained material of such character that it shall form a source of supply of or for regenerating the gas which forms the source of light within the tube.

My present application is a division of my prior application for patent filed September 2, 1903, Serial No. 171,587, wherein I have set out certain advantages in respect to the life of the device, quality of light when the tube is employed as a source of light, efficiency, and other advantages which are secured by the presence in the tube or receptacle of a suitable material comprising an electrolytically-treated organic substance, preferably one of which nitrogen is a constituent.

The special object of my invention is to secure the presence of the desired material within the manufactured device by a simple and cheap method or process of manufacture.

To this end the invention consists, substantially, in applying to the interior of the tube or receptacle or to a section of tubing or receptacle employed in the manufacture of the device the particular organic substance which is to be electrolytically treated, then subjecting said tube or section of tubing to the action of the electric current and employing said tube or section of tubing with the converted material converted upon the surfaces thereof as the tube or section of tubing for the completed device.

My invention consists also in a novel process of producing a vacuum-tube lamp or similar vacuum-tube device with a contained substance produced by electrolytic action upon an organic material, consisting in first constructing the tube with the proper amount of such substance contained within it and then exhausting the tube in the usual manner to produce the desired degree of rare-

faction of its contained gas and then finally sealing off.

As stated in my prior application, electric tube-lamps and other devices having a contained gas or vapor and through the interior of which an electric current circulates are of comparatively short life, and in many cases where the device is used as a source of light the quality thereof is poor and the device is inefficient. This difficulty I have attempted to obviate by introducing chemicals into the tube, which theoretically should be the source of fresh gas supplied as rapidly as the gas deteriorates under the action of the current, but at first met with only a measurable degree of success, because apparently all chemicals, solid, liquid, and gaseous, if of suitable quantity for the purpose act to change the necessary degree of rarefaction within the tube, since they would give off their gases too fast. The principal object of the present invention is to overcome this difficulty and to secure a manufactured lamp in which the vacuum shall be of the desired degree of rarefaction when the lamp is put into use and which shall thereafter maintain a suitable degree of rarefaction, as well as to secure other advantages herein referred to.

I have found that the difficulties heretofore experienced may be overcome by the introduction in any suitable manner into the tube of an organic substance of any kind which previously to the sealing off of the lamp has been subjected to electrolytic action. The material from which I have secured the best results is obtained by subjecting an organic compound, animal or vegetable, but preferably an aromatic hydrocarbon—such as benzoin, toluene, &c.—or a derivative thereof—such as anisic, benzoic, (without nitrogen,) hippuric acid, and benzamid (with nitrogen)—to electrolytic action. While it is possible to form this material in one tube or receptacle and remove it therefrom and place it in the manufactured tube or receptacle, I prefer to secure its presence in the manufactured device in one of the following ways: I take the length of tube or form of receptacle such as is to be employed for the complete device and into the same I introduce the substance to be treated, which in the case of a solid is preferably powdered and introduced into the tube

and uniformly distributed over the interior surface of the same by rotating the tube, heat being externally applied when necessary to cause the substance to adhere to the tube.

5 In some cases where the substance to be treated will sublime without decomposition (anistic or benzoic acid) I prefer to place the tube in a slanting position with the substance occupying the lower end or in a suitable tubular  
10 projection, to which heat is applied. The tube fills with vapor, which condenses on the cold walls, and the tube becomes coated internally with the substance employed in a finely-divided crystalline condition. If the  
15 substance is a liquid, I introduce a fixed amount and cover the walls of the tube with a thin film by rotating in a horizontal position. The tube is then sealed up, and being provided with suitable caps or electrodes by which electric  
20 current of high tension may be passed through the interior thereof is connected to a suitable source of electricity and current passed through the same, while at the same time, if desired, the air or other gas is exhausted  
25 from the interior thereof, the exhaustion of the air or gas aiding somewhat in the celerity of the conversion of the material forming the coating upon the interior walls of the tube or otherwise existing therein. In some cases  
30 also decomposition of the material may be hastened and aided by heating the tube, which may be resorted to in the case when crystals of hippuric acid are employed, when it is found that even with the continued application  
35 of the current no apparent decomposing effect takes place and that the light in the tube instead of changing from a red color at the beginning of the operation to a thick white as the exhaustion progresses  
40 changes from a red to a very thin white. In ordinary cases near the commencement of the exhaustion the light in the tube is a deep red color changing to a pure white, this change in color probably marking the fixation  
45 of the nitrogen and the production of an amid or other organic compound having nitrogen for one of its constituents. I also employ pure nitrogen in the place of air for this purpose.

50 By the above operation there is produced within the tube a fixed solid residue forming a more or less spongy but still firmly adherent coating upon the walls of the tube. The composition of this coating is uncertain, but in  
55 all preferred forms benzamid and in many cases acetamid is present in considerable quantity. In the case of benzoic or anistic acid or other substance containing no nitrogen and similar hydrocarbons these amids are formed  
60 by the fixation of the nitrogen of the air in the tube while the current is traversing the tube. In this operation the production of the coating seems to be assisted by lowering the pressure of the air or other gas within the tube.

65 The same treatment may be applied to a

substance already embracing nitrogen as a constituent—for example, benzonitril, benzamid, acetamid, &c.

When the electrical treatment is applied to a substance originally containing nitrogen, 70 the fixation of nitrogen by the electric current as set forth by the electrolytic action is of less importance.

While it is practicable to use any of the substances already mentioned in practice, I 75 find it preferable to use the material produced by electrolytically treating the aldehyde of anise known as "aubepine."

The tube or other receptacle with the coating or material secured by this process may 80 then be used entirely as the complete manufactured device, although, and especially when it is desired to secure illumination, the opaque nature of the coating on the interior of the tube is objectionable. Hence I ordinarily 85 prefer to use a section of tubing coated on its interior by the process described as a part of the tube of a complete or commercial lamp or other device, and by preference also I use such section as the end portion of the 90 tube, especially when, as is frequently the case, the said end section is provided with an external conducting layer of material, usually opaque, by means of which the current is passed into and through the gaseous 95 contents of the tube by electrostatic action; or, if desired, and especially when the complete tube is of very great length, a section of tubing having a coating produced upon its inner surface as already described may be 100 inserted into and employed as a section of the tube forming the complete or commercial device. In this case it is desirable that the coating of material should be rather thick in order to supply a sufficient amount thereof 105 to produce the results desired.

The presence of the material produced as described secures a smooth or perfectly steady uniform light, with an efficient life for the tube several hundreds of times as long as 110 a similar tube not having such new material within it.

The action of the new substance and the particular chemical constitution of the same resulting from the electric treatment described 115 cannot be fully set forth; but apparently the gases held by the solid material are decomposed by the electric current and recombined by the catalytic action of the spongy residue, and since this constantly and 120 rapidly recurring action is nearly perfect the life of the tube is very great, being only limited by the slow breaking down or clogging of the porous residue, with a consequent loss of the necessary amount of surface exposed 125 to regenerate the gases as fast as they are decomposed by the current—that is, this new material becomes the agent in a train or cycle of actions in which the gases forming the light are decomposed or affected by the light- 130

ing - current and transformed into a gas or gases whose chemical constitution is such that they can from the new substance or material be changed or recombined into the gas which is effective in producing the light. In some cases—as, for example, when variously-colored tubes are required—it is desirable to use chemicals within the tube in accordance with any of the methods herein described and introduce also into the tube various gases, either singly or in mixture, and I have found that the presence of the chemical causes such gases to last many hundreds of times longer.

By first treating the substance to be employed in the lamp or tube by electrolytic action until it is decomposed to its final form and exhausting the tube with the contained material to the required degree or to secure the desired vapor or gas density for producing light the difficulties heretofore met with in this class of devices are obviated to a considerable extent—namely, the difficulty that the chemical or material, solid, liquid or gaseous, when employed directly in light-tubes and decomposed by the action of the electric current when the lamp is in use and form objectionable spots or discolorations deposited on the inner walls thereof along the light-giving area. It is obviously, therefore, possible in order to secure this result to remove the coating secured in the manner already herein described from the interior wall of the tube and introduce it into the tube to be used as the light-tube, after which the latter may be sealed up and exhausted in the ordinary way to produce the required density of contained gas or vapor.

Although I have described the manner of manufacturing tubes for lighting and have set forth the use of external electrodes or caps therefor, I do not wish to limit myself to this class of tubes, since the invention is equally applicable for the production of long life in all classes of vacuum-tubes by maintaining the vacuum at a stable degree of rarefaction, it being understood that this includes X-ray tubes and light-tubes, either with internal or external electrodes.

Another great advantage of the use of chemicals as described is that a comparatively low vacuum can be used, and yet the light is satisfactory in all respects. Heretofore in order to obtain such a light a high vacuum, comparatively speaking, was absolutely essential, and therefore required suitable vacuum-pumps for the production of the high vacuum, and this involved the use of mercury-pumps; but with the chemicals as described the tubes can be exhausted by using only so-called "mechanical" pumps—that is, various kinds of vacuum-pumps that do not employ mercury.

In the description of the method of preparing these chemicals I have stated that a vacuum-pump is attached to the tube containing

the chemical which is being acted upon by high-tension electricity; but I do not wish to limit myself to the use of the vacuum as a necessary factor, since the material may be prepared by spreading the carbon compound or other chemical on a plate of glass and then passing the electrolyzing-current through it by placing one terminal of the high-tension source of electricity beneath the plate and another above it and close to the film or layer of material. This causes high-tension sparks to radiate all over and through the chemical, causing part of it to evaporate and after some time leaving the residue in the form of a coating on the plate that is similar to that produced within the tube which has a high-vacuum pump attached to it. However, I have found that this latter method is far more laborious, more expensive, and not nearly as preferable as the vacuum process.

The material produced in the above-described way will generally be in the form of a fixed solid residue forming a more or less spongy but still firmly adherent coating upon the plate or wall of glass, extending in some cases for several inches beyond the point of application of the current or electrolyzing agent. When desired, it may be removed from the surfaces to which it adheres by scraping it off, or it may be washed off by the use of various liquids and then recovered by evaporation.

In the accompanying drawings, Figure 1 shows a tube or receptacle that may be employed in producing the substance and at the same time be a part of the light-tube. Fig. 2 shows a tube specially designed for preparing the chemical. Fig. 3 shows a tube for introducing the chemical in a modified way. Fig. 4 shows a modified form of apparatus that may be used for producing the substance.

Referring to Fig. 1, the terminal of a tube of translucent material, like glass, is indicated at 3, which terminal is provided with a suitable conducting-cap—like, for instance, a coating of plumbago—provided with an attached conductor 8, by means of which electric energy may be supplied electrostatically to the contents of the tube. 4 is a small tube attached to the larger receptacle for the purpose of permitting the receptacle to be exhausted by the application of a gas-pump. The body of the receptacle of translucent material is indicated at 5. It will be understood that the opposite end of the tube is provided with a similar cap or terminal, (either inside or outside.) The substance to be treated having been introduced into the tube in either of the manners described or in any other suitable manner, preferably by spreading it over the interior surface by shaking the tube, said tube may then be allowed to drain off if the material introduced is of liquid form, after which it is properly

sealed and the electric current applied, the exhaustion of the air or other gas present in the tube at such time being carried on, preferably, during the application of the current.

5 After the completion of the process, which will be indicated in the manner already described, the tube may be cut and the material recovered therefrom, as already set forth. The material will then be collected, if the tube

10 is of considerable length, near the points of application of the current and under the cap, the coating extending, however, slightly beyond the gap into the translucent portion of the tube. The material removed may then

15 be introduced into another tube to be used as a completed device and said latter tube exhausted during the application of the current until the desired degree of exhaustion is obtained, as indicated by the color of the

20 light, or, if desired, the presence of the substance in the tube having been secured as already described it may then be allowed to remain and the tube itself employed as the operating device, it being understood, however, that this is generally a desirable procedure only when the tube is of such length

25 that the opaque or slightly-opaque coating does not extend very far into the translucent portion. In general the completion of the process will be indicated by the changed appearance of the coating in the translucent parts where said coating exists, such change being generally a change of color to a brown color. The material removed will appear as

30 a granular sawdust-like substance somewhat resembling brown sugar in appearance, but readily reduced by attrition to an almost impalpable powder that also sometimes has a chocolate color.

35 As indicated in Fig. 2, the material may be manufactured in a tube or receptacle of comparatively short length having a translucent section 2, in which a coating of the material on the inner walls may be secured in the manner already described. This section 2 of tubing or any other portion of said tube in which

40 the coating exists in sufficient amount may be employed as the section of the completed device in obvious manner. Thus, for instance, section 2 may be introduced, as at 1,

45 near the cap portion 3, Fig. 1, or the terminal portion of the device, Fig. 2, having the opaque conducting-cap, may be employed as the terminal portion 3 of the completed device. A section of the apparatus, Fig. 2,

50 having been sealed in place to make a part of the complete device, the manufacture of the same may be completed by exhaustion of the contents to the desired degree of rarefaction in the proper manner either with or without

55 the simultaneous application of the electric current.

In Fig. 3, 6 indicates a separate receptacle in which the substance to be treated may be placed, so that when heated its vapors will

pass into and condense on the walls of 7, the exterior of which is coated with graphite or other suitable material, which is connected with the wire 8, leading to a source of electricity of suitable potential.

In Fig. 4, 9 indicates a plate upon which the substance 10 to be treated may be spread, and 11 and 12 the electrodes for electrolyzing said material in one of the manners previously mentioned—viz., in the open air.

In the foregoing specification and annexed claims the term "tube" is used as synonymous with a sealed receptacle of any form or contour.

My invention is not limited to tubes containing only a rarefied gas acting and acted upon as herein described, but may be used with tubes in which there is also a mercury-vapor or other metallic or mineral vapor or gas.

An electrically-operated tube in which a material produced by electrolytically treating an organic substance is employed is not claimed herein, but forms the subject of claims in my prior application for patent hereinbefore referred to.

While I have found that all organic compounds, especially those of the hydrocarbon class, will be suitable for increasing the life of a vacuum-tube as above described, those organic compounds which come under the head of benzene derivatives have a decided advantage over others. About seventy-five per cent. of all organic compounds come under this head—that is, they do not belong to the "aliphatic" class, which is composed principally of such compounds as beeswax, butter, lard, &c., the carbon chains of which can be readily broken up; but the residues from benzene derivatives have the highest melting and boiling points of any organic compounds, (not metallic compounds,) and this indestructible feature of the benzene derivatives is that they all have six (6) carbon atoms permanently linked together in rings.

What I claim as my invention is—

1. The herein-described method of manufacturing electrically-operated vacuum-tubes, consisting in coating the interior of a tube or receptacle of any desired form adapted for use in the completed device with an organic substance, electrolyzing said substance to form an adherent coating of an electrolytically-produced substance upon the interior of the receptacle or tube and utilizing said tube as a tube or section of tube in the complete device.

2. The herein-described method of manufacturing electric lights in which the light-giving source is a rarefied gas, consisting in treating an organic substance contained in a section of the tube or lamp by an electrolytic process, producing thereby a fixed solid residue forming an adherent coating upon the wall of the tube and utilizing said section for

a section of the complete lamp and then exhausting the latter in the ordinary way.

3. The herein-described method of manufacturing electric lights in which the light-giving source is a rarefied gas, consisting in treating an organic substance contained in a section of the tube or lamp by an electrolytic process with the application of external heat, producing thereby a fixed solid residue forming an adherent coating upon the wall of the tube and utilizing said section for a section of the complete lamp and exhausting the latter in the ordinary way.

4. The herein-described method of producing vacuum-tubes for electric lighting consisting in coating the interior of a glass receptacle with a liquid hydrocarbon, passing an electric current through said receptacle while at the same time exhausting air and gas from the tube, producing thereby a fixed solid residue forming an adherent coating upon the wall of the tube and utilizing said receptacle as a section or portion of another tube or receptacle designed for employment as a complete lamp and exhausting the latter in the ordinary way.

5. The herein-described method of manufacturing vacuum-tube lamps containing a material designed to form a source of gas-supply during operation of the lamp, consisting in first subjecting an organic substance to electrolytic action, exhausting the lamp and subsequently and while the electrolytically-treated substance is contained therein sealing off the lamp.

6. The herein-described method of producing vacuum-tube lamps without spots or discolorations arising from the decomposing substance, consisting in first electrolyzing an organic substance designed to form a source of gas-supply during operation of the lamp and finally sealing off the lamp after exhaustion and while the previously electrolytically decomposed substance is contained therein.

7. The method of producing vacuum-tube lamps with a stable vacuum and with a contained material designed to form a source of gas-supply during operation of the lamp, consisting in introducing an organic substance

into the lamp, passing an electric current through the lamp to decompose such substance and at the same time exhausting the lamp and after decomposition has been practically completed, sealing the lamp off.

8. The herein-described method of securing a stable vacuum in vacuum-tube lamps containing an electrolyzed organic substance, consisting in first subjecting such substance to electrolytic action to completely decompose it, and applying the exhaust-pump, and finally sealing off.

9. The method of producing vacuum-tube lamps with stable vacuum and with a contained material, adapted to maintain the desired degree of rarefaction, consisting in reducing an organic material by electrolytic action to its final form and sealing off the lamp after exhaustion to the degree of rarefaction desired for commercial operation.

10. The herein-described method of manufacturing vacuum-tube lamps, containing a substance designed to form the source of gas during operation of the lamp; consisting in introducing an organic substance into the lamp, passing an electric current through the lamp to decompose the substance to its final form while at the same time exhausting the lamp, and finally sealing off.

11. The herein-described method of manufacturing vacuum-tube lamps containing electrolytically-treated aubepine forming during operation of the lamp a source of a gas by which the desired degree of rarefaction and luminosity is maintained, consisting in first subjecting the aubepine to the action of an electric current by which it may be reduced to its final form and sealing off the lamp while it contains the completely-reduced aubepine and subsequently to the exhaustion of the tube to secure the desired degree of rarefaction of its gaseous contents.

Signed at New York, in the county of New York and State of New York, this 4th day of March, A. D. 1904.

DANIEL McFARLAN MOORE.

Witnesses:

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