United States Patent [19]

Neel et al.

[11] **3,788,331**

[45] **Jan. 29, 1974**

[54] **SOLVENT RECOVERY IN TOBACCO TREATING PROCESS**

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[73] Assignee: **R. J. Reynolds Tobacco Company,** Winston-Salem, N.C.

[22] Filed: **June 26, 1972**

[21] Appl. No.: **266,416**

[52] **U.S. Cl.** ..... **131/143,** 131/140 P, 55/30

[51] **Int. Cl.** **A24b 03/18**

[58] **Field of Search**... 23/270, 283; 62/9; 55/81, 30; 131/143

[56] **References Cited**

UNITED STATES PATENTS

3,195,292	7/1965	Skarstrom	55/32
3,612,066	10/1971	Jones et al.	131/143

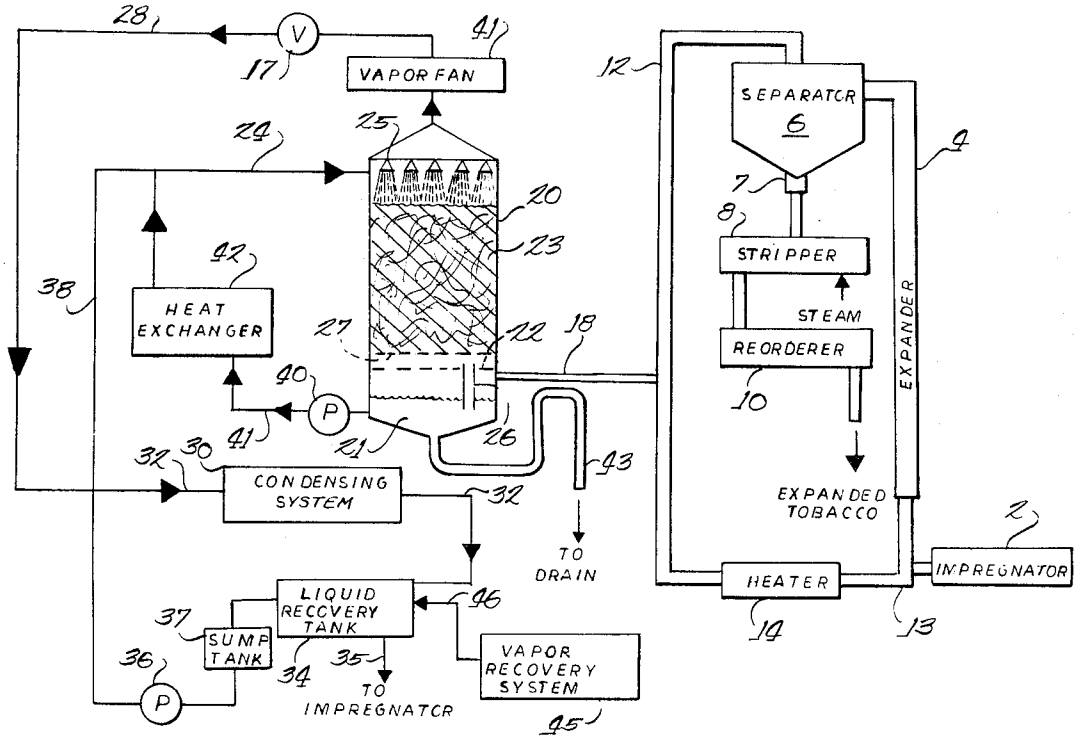
Primary Examiner—Melvin D. Rein

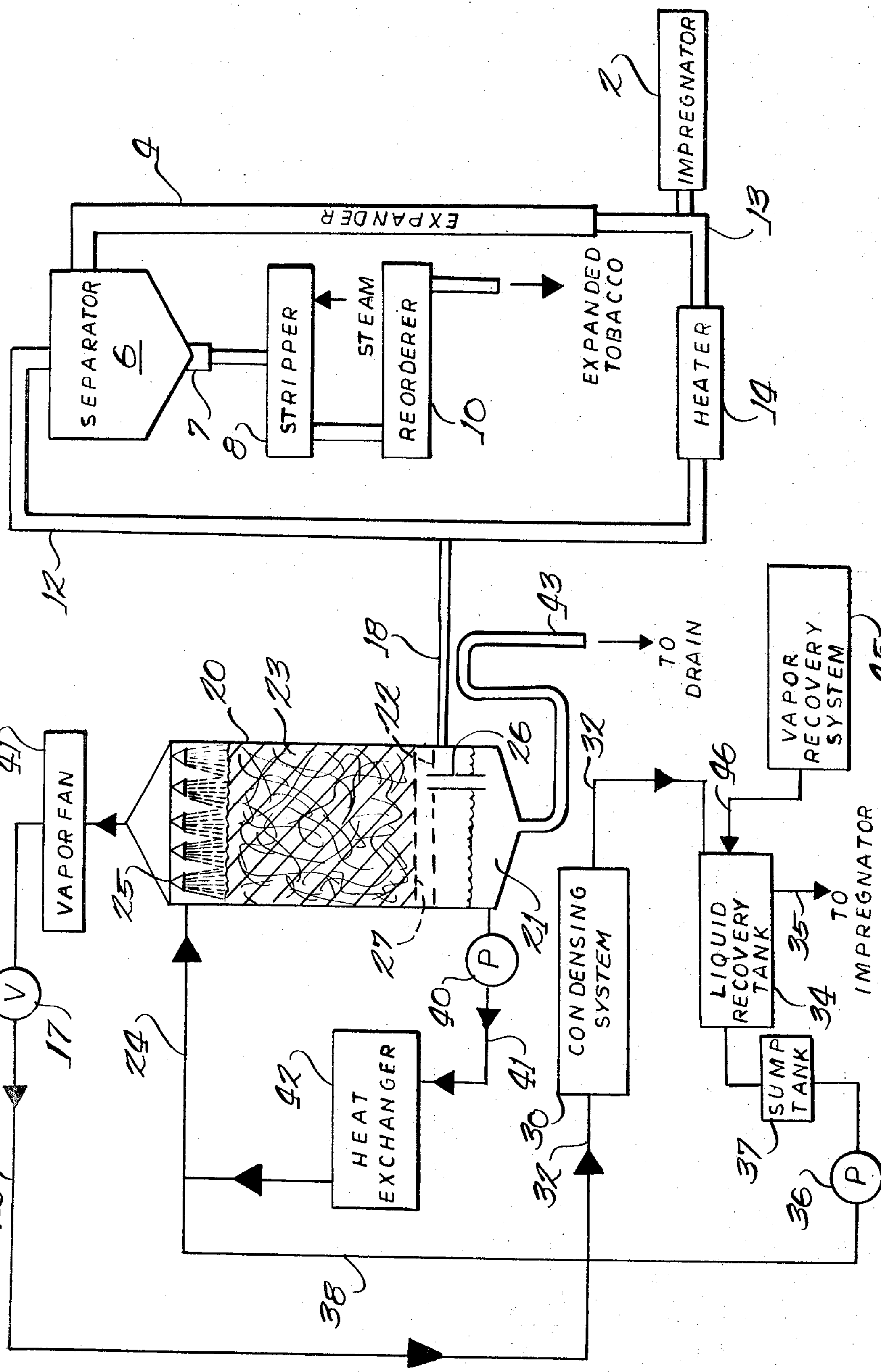
Attorney, Agent, or Firm—Newman, Williams, Anderson & Olson

[57] **ABSTRACT**

Recovery of impregnant in a process in which tobacco is impregnated with a water-immiscible organic impregnant and then subjected to a stream of gas heated to a temperature substantially above the boiling point of the organic impregnant whereby the organic impregnant is volatilized and the tobacco is expanded, with the expanded tobacco being then separated from the resulting impregnant-containing gas stream. Recovery of the organic impregnant is accomplished by passing a portion of the gas stream into a lower section of a packed tower and introducing into the upper section of said tower water at a controlled temperature to effect removal of tobacco fines in the gas stream, to cool the stream and condense a substantial proportion of the water present therein and to strip organic impregnant from process waste water introduced into the upper section of said tower. The organic impregnant is then condensed and recovered by conventional procedures for subsequent reuse.

8 Claims, 1 Drawing Figure




IMPREGNATOR
EXPANDER
SEPARATOR
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STRIPPER
STEAM
REORDERER
EXPANDED TOBACCO
HEATER
TO DRAIN
VAPOR RECOVERY SYSTEM
VAPOR FAN
LIQUID RECOVERY TANK
TO IMPREGNATOR
CONDENSING SYSTEM
HEAT EXCHANGER
SUMP TANK
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SOLVENT RECOVERY IN TOBACCO TREATING PROCESS

This invention relates generally to treatment of tobacco to increase or expand its filling capacity. More particularly, the invention relates to the recovery of fluids employed in tobacco expanding processes.

U.S. Pat. No. 3,524,452 to Glenn P. Moser and Grant Mathews Stewart which issued Aug. 18, 1970 describes a method of increasing the filling capacity of tobacco which involves impregnating tobacco with an organic liquid and then subjecting the impregnated tobacco to a stream of hot gas whereby the liquid is quickly vaporized with the simultaneous puffing or expanding of the tobacco particles. The impregnating liquid employed is one which is chemically inert with respect to the tobacco and falls generally into the classes of aliphatic hydrocarbons, aromatic hydrocarbons, alkanols, ketones, aliphatic esters, ethers, halogenated hydrocarbons and mixtures thereof.

Copending application Ser. No. 138,039 filed Apr. 28, 1971 by Arnold G. Moore and Donald A. Newton relates to a process which is a modification of the process described in the above patent. In accordance with the latter process, tobacco and a stream of vapors of an inert organic compound are introduced into one end of an impregnating zone and moved in concurrent flow relationship to the other end of the impregnating zone during which time the tobacco becomes thoroughly impregnated with the compound. After the impregnation has occurred, the merged stream comprising tobacco and the impregnating fluid, including vapors and any condensed impregnant, is withdrawn from the other end of the impregnating zone and suddenly subjected to vapor-expanding conditions whereby the impregnant in the tobacco is vaporized causing the tobacco to expand. The expanding conditions are preferably achieved by rapidly increasing the temperature of the impregnated tobacco by introducing it into a stream of hot gas in the manner described in said U.S. Pat. No. 3,524,452. The volatile compound or mixture employed for impregnating the tobacco is preferably one which is organic in nature, is chemically inert to the tobacco being treated and has a boiling point at atmospheric pressure between about −50° C. and +80° C. Preferably, the atmospheric pressure boiling point of the compound is between −40° C. and +40° C. Illustrative inert organic compounds are: ketones such as acetone and methyl ethyl ketone; aliphatic or cyclic ethers such as methyl ethyl ether, diethyl ether, diisopropyl ether, methyl butyl ether, dimethoxy methane, furan and tetrahydrofuran; aliphatic alcohols such as methanol, ethanol and 2-propanol; esters such as methyl formate, ethyl formate and methyl acetate; aliphatic hydrocarbons such as butane, pentane, isopentane, hexane and the corresponding unsaturated hydrocarbons; the cyclo aliphatic hydrocarbons such as cyclobutane, cyclohexane and cyclopentane; the halohydrocarbons ethyl chloride, propyl chloride, isopropyl chloride, sec-butyl chloride, t-butyl chloride, methyl bromide, ethyl bromide, t-butyl bromide, methylene chloride, chloroform, carbon tetrachloride, ethylene dichloride, ethylidene chloride; and the fluorinated hydrocarbons represented by trichloromonofluoromethane, dichlorodifluoromethane, monochlorodifluoromethane, 1,1-difluoroethane, chloropentafluoroethane, octafluorocyclobutene, 1,1,1-trichlorodifluoroethane and 1,2-dichlorotetrafluoroethane.

In both the above processes the tobacco after being impregnated with the selected fluid is subjected to vapor-expanding conditions by being withdrawn from the impregnating zone and subjected to a hot gas to vaporize the volatile organic impregnant and to expand the tobacco. Thus, referring to the drawing which shows in diagrammatic form the flow of various streams, the impregnated tobacco from impregnator **2** is passed into conduit **13** and then into expander **4** in which it is contacted with a hot gas to vaporize the impregnant and to simultaneously expand the tobacco. The expanded tobacco is carried by the hot gas to a separator **6** such as a cyclone separator, in which the expanded tobacco separates from the hot expanding gas, vapors of the impregnating fluid and steam vapors. The expanded tobacco is passed through an air lock **7** to steam stripper **8.** Residual impregnating fluid is removed by vaporization from the tobacco in steam stripper **8** and the tobacco effluent is passed to a reorderer **10** in which the product is adjusted to the desired moisture content. The gases including steam and vapors of the impregnating fluid are withdrawn from separator **6** through conduit **12,** are heated to desired extent in heater **14** and recycled to the expander **4** through conduit **13.**

The present invention relates to the recovery of organic water-immiscible impregnating fluids used in such processes as above for expanding tobacco. Thus, in accordance with the present invention, a side stream of excess gas consisting principally of hot vapors of the impregnating fluid together with some air, water vapor and tobacco dust is withdrawn through line **18** and is subjected to preliminary recovery operations as will now be described. This side stream is withdrawn through line **18** at a suitable rate as controlled by valve **17** to maintain a slight suction in separator **6** and is passed into the lower section of tower **20** at a point above a liquid-collecting or liquid seal section **21** in the bottom section thereof. It then passes through a perforated plate **22** where it bubbles through a shallow layer of water to remove tobacco particles and dust. It then passes through a bed of packing designated by numeral **23** which is supported by screen **27** and which occupies approximately 60 to 80 percent of the height of the tower **20.** The packing material is preferably made of materials which are essentially inert under the operating conditions employed. Polypropylene, ceramic or stainless steel packing having a "Berl saddle" or "intalox saddle" configuration are examples of packing materials suitable for the present invention. Water at a controlled temperature is introduced through line **24** into the top section of tower **20** and is discharged through manifold **25** so as to flow downwardly in countercurrent relationship to the upwardly flowing gas stream rising from the perforated plate **22.** The temperature of the water introduced through line **24** and manifold **25** is controlled so that it is at a temperature somewhat, say 5° to 20° F., above the boiling point of the organic impregnating fluid but below the boiling point of water at the pressure prevailing in tower **20.** The gas stream flowing upwardly within tower **20** is cooled to within a few degrees of the temperature of the water flowing downwardly within tower **20** and a major proportion of water vapor in the gas stream is thereby caused to condense and collect with the circulating water stream in the liquid-collecting section **21** of the

tower 20. Small particles of tobacco or dust which are removed at the perforated plate 22 or, to a lesser degree, in the packed section 23, either settle out of the water in the lowest part of the tower 20 or leave in the water draining from the tower through drain 43.

Vapors of the organic impregnating fluid plus some water vapor and air are removed from tower 20 by vapor fan 41 through line 28 controlled by valve 17 and sent to condensing system 30. Condensing system 30 can involve one or more conventional liquid condensing stages at successively higher pressures. After condensation the organic impregnating fluid and water are sent via line 32 to liquid recovery tank 34 where phase separation between the organic impregnating fluid and water takes place. In the case of organic liquids having a density greater than water, the organic impregnating fluid is withdrawn through line 35 (this line being appropriately located in the case of less dense organic liquids) and recycled for reuse in expanding additional quantities of tobacco. Water plus small amounts of entrained and dissolved organic fluid from recovery tank 34 overflows into sump tank 37 and is sent by means of pump 36 via line 38 to water inlet line 24 for use in tower 20. In line 24 it is combined with a much larger volume of cooled, recycled water emerging from heat exchanger 42. This combined water stream should be large enough to keep the packing 23 in tower 20 thoroughly wet and to cool the gas stream to the desired temperature. As this water flows down over the packing 23 countercurrent to the rising gas stream, it cools the hot gas and causes much of the water vapor in the rising gas stream to condense. The recycled water is thereby heated causing entrained and dissolved organic liquid to vaporize therefrom and to combine with the gas stream which leaves the top of tower 20 through vapor fan 41. The heated water flows over the perforated plate 22 where it washes tobacco particles out of the gas stream. Perforated plate 22 is provided with a vertically-arranged pipe 26 which protrudes slightly above the top surface of plate 22 and extends downwardly to a point below the normal water level maintained in liquid seal section 21 by an inverted U-shaped drainline 43. Excess water condensed in the process can be removed via drain 43 but the greater portion of the water stream emerging from the packed section 23 through the perforated plate 22 is recycled by pump 40 through heat exchanger 42 back to the top of tower 20 through line 24.

A further feature of the present invention is that it permits, if desired, the use of organic impregnant-saturated water obtained from an auxiliary or complementary vapor recovery system indicated generally by the numeral 45. Condensate from vapor recovery system 45 consisting of water substantially saturated with organic impregnant is routed to liquid recovery tank 34 via line 46. Water, which represents the major portion of this condensate, is thus combined with water obtained from condensing system 30 for subsequent use in tower 20 as previously described. It can be seen that this feature serves to further minimize loss of organic impregnant by the reuse of water that is normally discarded. An auxiliary vapor recovery system 45 is described in copending application Ser. No. 266,433 filed of even date by Robert M. Neel and Robert C. Johnson.

The process of the present invention will be further described with reference to a specific example. Thus, a gaseous stream consisting of 20 to 30 percent by volume organic impregnating fluid such as trichloromonofluoromethane, 20 to 30 percent air, 40 to 60 percent water vapor and tobacco fines or dust is passed through line 18 into tower 20 at a rate of 5,000 pounds per hour and at a temperature of about 240° F. Tower 20 contains a cylindrical bed of 2-inch polypropylene intalox saddle packing having a height of 5 feet and a diameter of 3 feet. Water at a temperature of about 90 to 105° F. is introduced through water inlet line 24 into tower 20 at a rate of about 100 gallons per minute. This water, discharged through manifold 25, flows downwardly in countercurrent relationship with the upwardly flowing gaseous stream introduced through line 18. This causes water vapor in said latter stream to condense and it also removes tobacco fines which are carried by the condensed water flow from plate 22 to section 21 of the tower.

Vapors containing 45 percent trichloromonofluoromethane together with about 10 percent water vapor and 45 percent air are withdrawn by fan 41 at a temperature of about 90° to 105° F. and are passed through line 28 to condensing system 30. The condensed water and trichloromonofluoromethane are transferred via line 32 to liquid recovery tank 34 where the water and organic phases separate by gravity. The recovery tank 34 is cooled to a 50°–60° F. temperature range. The liquid trichloromonofluoromethane is withdrawn from tank 34 via line 35 and used for impregnating additional quantities of tobacco. The water is allowed to overflow to sump 37 and is pumped by pump 36 via line 38 to water inlet line 24. Recycle water from section 21 of tower 20 is withdrawn by means of pump 40, sent through heat exchanger 42 and passed through water line 24 and manifold 25 with the water from tank 34 into the top section of tower 20.

By operation in the above manner, good recovery of the organic tobacco impregnating fluids is achieved efficiently. The present invention can be advantageously employed for recovery of any water-immiscible impregnating fluid used for expanding tobacco. The removal of substantial quantities of water in tower 20 significantly reduces the load on condensing system 30. Likewise, the load on vapor fan 41 and on condensing system 30 is reduced by virtue of the reduced temperature of the organic impregnating fluid being conveyed through line 28 as compared with its temperature in line 18. Also, the entrained and most of the dissolved trichloromonofluoromethane is stripped from the water overflow from tank 34 as it passes through tower 20. Moreover, the removal of tobacco fines in tower 20 reduces the interference of such particles with respect to operation of the condenser system 30. It is also apparent that the recycle of water through lines 38 and 24 for reuse in tower 20 affords significant reduction in the water requirements of the system for scrubbing out the tobacco particles.

Those modifications and equivalents which fall within the spirit of the invention are to be considered a part thereof.

What is claimed is:

1. In a process in which tobacco is impregnated with a water-immiscible organic impregnant and then is subjected to a stream of a gas heated to a temperature substantially above the boiling point of the organic impregnant whereby the organic impregnant is volatilized and the tobacco is expanded and wherein the expanded tobacco is subsequently separated from the resulting vol-

atilized stream, the improvement which consists in passing a portion of said volatilized stream into a lower section of a packed tower, introducing into the upper section of said tower water at a controlled temperature which is above that of the boiling point of the organic impregnant and below that of water, flowing said water downwardly through said packed tower in countercurrent relationship to said volatilized stream, removing water from a lower section of said packed tower and removing vaporized organic impregnant from the top of said tower and condensing the so-removed organic impregnant.

2. The process of claim **1** wherein the water which is introduced into the upper section of said packed tower is at a temperature approximately 5° to 20° F. above the boiling point of the organic impregnant.

3. The process of claim **1** wherein water introduced into the packed tower is collected in a liquid pool in said lower section of the packed tower.

4. The process of claim **3** wherein water from said liquid pool is recycled to the upper section of said tower.

5. The process of claim **1** wherein the packed tower is provided with a perforated plate in the lower section thereof through which said volatilized stream passes upwardly prior to passage through a bed of packing material in said packed tower.

6. The process of claim **5** wherein the bed of packing material occupies from about 60 to about 80 percent of the height of said packed tower.

7. In a process in which tobacco is impregnated with a water-immiscible organic impregnant and then is subjected to a stream of a gas heated to a temperature substantially above the boiling point of the organic impregnant whereby the organic impregnant is volatilized and the tobacco is expanded and wherein the expanded tobacco is subsequently separated from the resulting volatilized stream, the improvement which consists in passing a portion of said volatilized stream into a lower section of a packed tower having

an upper section comprising vapor exit means and a plurality of nozzles for introducing water at a controlled temperature which is above that of the boiling point of the impregnant and below that of water,

an intermediate section comprising packing material suspended above the lower section by means of a porous support and

a lower section comprising means for maintaining a predetermined maximum level of liquid in said lower section together with means for withdrawing said liquid, a horizontally-disposed perforated plate located above said predetermined maximum level of liquid and means for introducing said volatilized stream into said lower section of the tower at a point intermediate between said perforated plate and said predetermined maximum level of liquid,

introducing water through said nozzles for downward flow through said packing material to condense at least a portion of water vapor contained in said voltatilized stream which is moving upwardly through said packing material, withdrawing an organic impregnant-containing vapor through said vapor exit means and recovering the withdrawn impregnant vapors by condensation.

8. The process of claim **7** wherein a portion of the water introduced through said nozzles is derived from an organic vapor recovery system.

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UNITED STATES PATENT OFFICE

CERTIFICATE OF CORRECTION

Patent No. 3,788,331 Dated January 29, 1974

Inventor(s) Robert M. Neel, Clarence Robert Lloyd and Robert C. Johnson

It is certified that error appears in the above-identified patent and that said Letters Patent are hereby corrected as shown below:

Column 2, line 1, "fluorocyclobutene" should be

-- fluorocyclobutane --

Signed and sealed this 30th day of July 1974.

(SEAL)
Attest:

McCOY M. GIBSON, JR.
Attesting Officer

C. MARSHALL DANN
Commissioner of Patents

FORM PO-1050 (10-69)

USCOMM-DC 60376-P69
* U.S. GOVERNMENT PRINTING OFFICE : 1969 O—366-334