EP.0048742

PCT WORLD INTELLECTUAL PROPERTY ORGANIZATION
International Bureau

INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

(51) International Patent Classification ^{3}: B01F 3/04

A1

(11) International Publication Number: **WO 81/02848**

(43) International Publication Date: 15 October 1981 (15.10.81)

(21) International Application Number: PCT/US81/00351

(22) International Filing Date: 20 March 1981 (20.03.81)

(31) Priority Application Number: 137,148

(32) Priority Date: 4 April 1980 (04.04.80)

(33) Priority Country: US

(81) Designated States: DE (European patent), FR (European patent), JP, NL (European patent).

Published
With international search report
With amended claims

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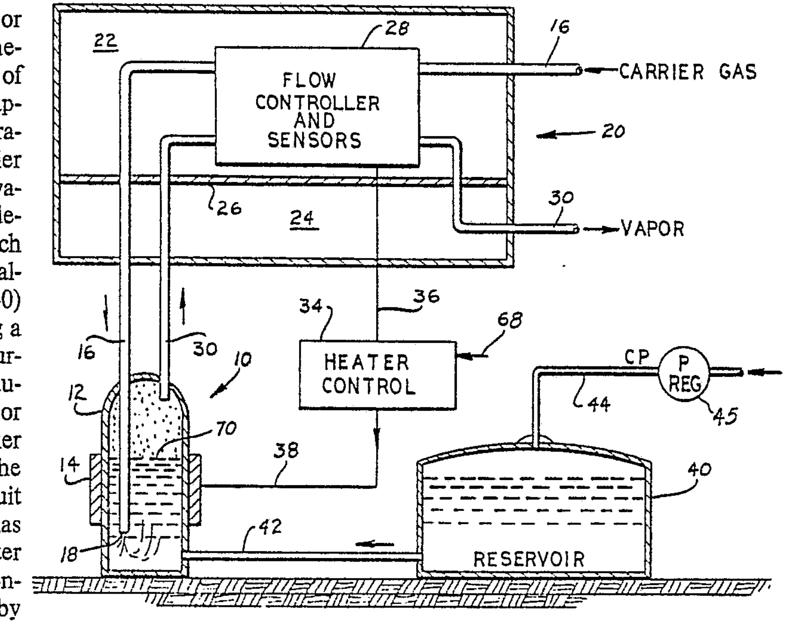
(54) Title: VAPOR DELIVERY CONTROL SYSTEM AND METHOD

(57) Abstract

Method for controlling the delivery of vapor from a bubbler (12) containing a supply of liquid through which a carrier gas is bubbled and from which bubbler vapor is delivered in a vapor stream entrained with the carrier gas. The method comprises the steps of sensing the ratio of vapor to carrier gas in the vapor stream and applying heat to the liquid within the bubbler at rates relative to the sensed ratios of vapor to carrier gas being delivered from the bubbler in the vapor stream to hold the ratio constant. A vapor delivery control system is also disclosed which comprises a vaporizer (10), a heater (14) thermally coupled with the vaporizer, a reservoir (40) and a pressure regulator (45) for maintaining a supply of liquid in the vaporizer. The system further includes an intake conduit (16) for introducing a carrier gas into the vaporizer, a vapor stream conduit (30) for conducting the carrier gas and vapors of the liquid entrained with the carrier gas from the vaporizer, a bridge circuit (74) for sensing the ratio of vapor to carrier gas being conducted from the vaporizer, a heater controller (34) for controlling the heater responsive to the ratio of vapor to carrier gas sensed by the bridge circuit to hold the ratio constant.



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VAPOR DELIVERY CONTROL SYSTEM AND METHOD

Technical Field

This invention relates to methods and systems for controlling the delivery of vapors.

Background of the Invention

With the advent of optical waveguides for use in the communications industry much emphasis has recently been placed on vapor deposition as a materials forming technique. In constructing preforms from which optical fibers may be drawn, vapors of materials such as $SiCl_4$, $GeCl_4$ and $POCl_3$ must be precisely delivered at controlled mass flow rates to the preform construction site where they are reacted and deposited on or in a support. This can be done by passing carrier gases such as H_2, He, N_2, O_2, or Ar through a supply of the material in liquid form and to the deposition site as a mixture with the vapors entrained with the carrier gas. In performing this operation a vaporizer is ordinarily used of the type known as a bubbler which has a carrier gas intake conduit that terminates with an outlet orifice located below the surface of the liquid materials and an outlet conduit extending from the space above the surface of the liquid within the bubbler to the deposition site.

To construct an optical waveguide preform properly the mass flow rate of the vapor must be carefully programmed and accurately controlled. Heretofore control has been achieved with vaporizer controllers which employ a carrier gas mass flow rate sensor and a vapor to carrier gas ratio sensor.

The carrier gas flow rate sensor operates on the theory that the heat added to a known mass of gas is proportional to its temperature rise at relatively constant pressure. It employs two resistance heating elements which are part of a bridge circuit, positioned in series with each other on the outside of a sensor tube. Gas is passed through the tube which creates a bridge imbalance, the signal from which is proportional to the mass flow rate.

The vapor to carrier gas ratio sensor also operates as a function of heat transfer. This sensor employs one electrical resistance element located in the carrier gas intake stream and another in the vapor and carrier gas stream, hereinafter termed "vapor stream". Again the sensors are elements of a bridge circuit which indicates an imbalance as soon as the properties of the gas and vapor stream differ. This difference is proportional to the ratio of source to carrier gas.

With these controllers the electrical signals from the carrier gas flow rate and vapor to carrier gas ratio bridge circuits are electronically multiplied and the product compared with a preselected set point for vapor mass flow rate. An error signal is then fed through an amplifier to an electrically controlled valve located in line with the carrier gas intake conduit. When an insufficient mass flow is detected the valve in the carrier gas intake conduit is opened further to increase the flow of carrier gas into the vaporizer which, in theory, serves to pick up more vapor and increase the mass flow rate. Conversely, if too great a mass flow rate is detected the valve is closed somewhat.

Though the just described system and method for controlling vapor delivery has been found to be the best available, it nevertheless is quite inaccurate with deviations from optimum set points ranging as great as 30% over both long and short terms. This is attributable at least in part to the fact that this method assumes a steady state condition of vapor pressure. In actuality however the system is not in a steady state since vapor pressure depends on numerous criteria such as carrier gas retention time within the liquid, the depth at which the bubbles are released within the liquid, total pressure, carrier gas temperature, localized temperature inhomogentities surrounding the bubbles as they travel toward the liquid surface, and heat flow into the bubbler from its environment. These effects all become more important as flow rates increase and the liquid levels in the bubbler decrease, since retention by carrier gases also decreases as localized cooling takes place.

It is thus seen that control of the flow rate of carrier gas into a vaporizer is only a relatively crude method of controlling mass flow rate of vapor from the vaporizer because of the dynamics of such systems. Some investigators have sought to overcome this problem by placing an array of temperature sensors in the liquid housed within the bubbler and controlling heat into the bubbler responsive to sensed temperatures. This approach however has also failed to produce a high a degree of accuracy which again is believed to be attributable at least in part to the dynamics of the system.

Summary of the Invention

In one form of the present invention a method is provided for controlling the delivery of vapor from a bubbler containing a supply of liquid through which a carrier gas is bubbled and from which bubbler vapors are delivered in a vapor stream entrained with the carrier gas. The method comprises the steps of sensing the ratio of vapor to carrier gas in the vapor stream and applying heat to the liquid within the bubbler at rates relative to the sensed ratios of vapor to carrier gas being delivered from the bubbler in the vapor stream to hold the ratio constant.

In another form of the invention a vapor delivery control system is provided which comprises a vaporizer, a heater thermally coupled with the vaporizer, and means for maintaining a supply of liquid in the vaporizer. The system also comprises means for introducing a carrier gas into the vaporizer, means for conducting the carrier gas and vapors of the liquid entrained with the carrier gas from the vaporizer, means for sensing the ratio of vapor to carrier gas being conducted from the vaporizer, and means for controlling the heater responsive to ratios of vapor to carrier gas sensed by the sensing means.

Brief Description of the Drawing

Figure 1 is a schematic diagram of a vapor control delivery system embodying principles of the invention in one preferred form which may be used in practicing a method of the invention in a preferred form.

Figure 2 is a schematic diagram of the flow controller and sensors unit of the system shown in Figure 1.

Description of the Preferred Embodiment

Referring now in more detail to the drawing, there is shown in Figure 1 a vapor delivery control system comprising a vaporizer 10 in the form of a bubbler 12 having a resistance heater 14 jacketed about the exterior surface of the bubbler. A carrier gas intake conduit 16 extends from an unshown source of pressurized gas laterally into the upper space 22 of a temperature controlled enclosure 20 and through a flow controller and sensors unit 28. After passing from the unit 28 the conduit 16 is directed downwardly through a partition 26 and through a lower space 24 within the enclosure 20. The conduit then exits the enclosure and extends further downwardly into the bubbler 12 terminating with a lower outlet 18 located adjacent the bottom of the bubbler.

A vapor stream conduit 30 extends upwardly from an intake orifice located within and adjacent the top of the bubbler 12 into the heat-controlled enclosure 20 through the flow controller and sensors unit 28 and into enclosure 24 where it junctures with other, unknown vapor stream conduits from other bubblers of the type shown and then out of the enclosure 20 to a vapor deposition station (not shown). The flow controller and sensors unit 28 is electrically coupled to a heater control 34 by control signal line 36. A load line 38 extends from the heater control 34 to the resistance heater 14 jacketed about the bubbler 12. A reservoir 40 is also provided together with conduit 42 in fluid communication between lower portions of the reservoir 40 and bubbler 12. A gas intake line 44 extends upwardly from the top of the reservoir 40 through a pressure regulator to a source of compressed gas (not shown). The pressure of the gas is adjusted to maintain constant reservoir head pressure.

With reference next to Figure 2 the flow controller and sensors unit 28 is schematically seen to include both a flow rate sensor and a vapor to carrier gas ratio sensor. The flow rate sensor includes a pair of electric resistance elements 50 wound in series around a sensor tube in fluid communication with the carrier gas intake conduit 16. In conjunction with other elements 78, the resistance elements form a bridge circuit 72. The flow rate sensor utilizes the principle that the temperature rise of a gas at a relatively constant pressure is a function of the amount of heat added, the mass flow rate and other properties of the gas. Constant power is supplied to the sensor unit which in turn changes the relative resistances of the unit resistors when gas is flowed through the sensor tube. The upstream sensor is cooled at a higher rate than the downstream sensor producing a bridge unbalance. The bridge output can then be calibrated with actual gas flow that is monitored with a standard such as a Brooks volumeter.

The vapor to carrier gas ratio sensor unit also employs the just described principle by utilizing sensor elements 52 which are exposed to the flow of both the input carrier gas and to the vapor stream and whose resistance changes are functions of thermal properties of the two streams. Similarly, these elements 52 together with other elements 76 form a bridge circuit 74. Bridge output versus ratio curves are producable by mixing known mixtures and plotting calibration curves. The bridge output voltages curves, which are not completely linear and stable, thus provide representations of the vapor to carrier gas ratios.

The output signal from the vapor to carrier gas ratio sensor bridge circuit 74 is transmitted over line 36 to the signal input terminal of the heater control 34. The heater control 34 is a conventional power controller. It compares the input signal with a preselected vapor to carrier gas ratio set point 68 and, in response thereto, controls the current flow to the bubbler heater 14 over power line 38 which is connected to the power output terminal of the heater control 34.

The signal from bridge circuit 74 is fed by line 57 to a voltage multiplier 58. The signal from the carrier gas flow rate sensor bridge circuit 72 is also fed to the multiplier 58. The output signal from the multiplier 58 is in turn conducted to a voltage comparator 60, where the signal is compared with a pre-established voltage via a set point terminal 62. The output signal of the comparator is directed through an amplifier 64 to a valve V positioned within the carrier gas input conduit 16. Alternatively, the signal from circuit 74 may not be transmitted to the multiplier 58 and the signal from the circuit 72 merely transmitted directly to the comparator 60, thereby eliminating the multiplier from the circuitry and controlling carrier and ratio separately.

In operation, the reservoir 40 is partially filled with liquid chemicals which are to be deposited by vapor deposition at an unshown vapor deposition station where a fiber optics preform or rod is to be constructed. As shown in the drawing the reservoir 40 may be mounted at the same height as the bubbler 12. As a result the liquid chemicals flow from the reservoir (40) through conduit 42 into the bubbler 12 until the surface level 70 of the liquid in the bubbler 12 equals that of the surface level of the liquid within the reservoir 40. A gas is then introduced under pressure into the reservoir 40 through conduit 44, and the pressure set with the pressure regulator to establish a preselected level for the surface 70 of the liquid within the bubbler 12. The pressure is then maintained constant within the reservoir 40 by the pressure regulator. As the reservoir 40 contains a substantially larger supply of liquid than the bubbler 12, the rate at which the level of liquid 70 in the bubbler 12 changes remains quite low. Periodically, however, significant lowering of level 70 occurs, whereupon the level is adjusted by increasing the pressure head of reservoir 40. In deriving the selected level the effervescent condition of the liquid with carrier gas being bubbled up therethrough is taken into account. It is important to insure that the level 70 of the vaporizable liquid in the bubbler 12 is maintained constant as this system provides.

With a constant level of liquid in the bubbler 12, the carrier gas, which may be oxygen for example, is introduced through the flow controller 28 and through the vaporizer 10 and bubbled up through the liquid. Since the liquid within the bubbler 12 is maintained substantially constant the rise time and size of the bubbles is pre-established by the size of the outlet 18 of the conduit 16. In turn, both the surface area and time of exposure of the bubbles of carrier gas to the liquid as the gas rises to the surface 70 is also preselected. Vapors thus diffuse into the bubbles themselves as well as occupying the space above the surface 70 within the bubbler 12. Vaporization also occurs at the surface of the liquid. As a result it is not mandatory that the vaporizer 10 here be of the bubbler type. Finally, the carrier gas carries the vapors out of the bubbler 12 through the inlet orifice conduit 30 and then upwardly through the conduit to the ratio sensor and an unshown manifold where it is mixed with gases and other vapors entering the enclosure from other unshown bubblers of the same type.

It should be noted that the orientation of the conduit 30 is vertical as it leaves the bubbler 12. This is done so that any condensation or aerosol which might occur within this portion of the conduit 30 gravitates back into the bubbler 12. The conduit 30 passes through the lower space 24 of the enclosure 20 and into the upper space 22 where it turns laterally and passes through the flow controller and sensors unit 28 to space 24 and then out of the enclosure. The temperature of the lower air space 24 is preferably maintained approximately 5° to 10°F above that of the upper space 22. Due to this elevation in temperature over the ambient room temperature environment of the bubbler there is little tendency for condensation to form within the vapor stream as it approaches and after it has departed from the flow controller and sensors unit 28, since it is constantly being heated.

The vapor stream now passes over a vapor to carrier gas ratio sensor 52 and then out of the delivery system through conduit 30 to the vapor deposition station. If the vapor deposition station is located at any significant distance from enclosure 20, that portion of conduit 16 between the station and enclosure 20 should also be heated. As the stream passes over sensor 52, the sensor and associated circuitry detect the ratio of vapor to carrier gas being delivered to the vapor deposition station. A corresponding signal is transmitted from the sensor circuit 74 over line 36 to the heater control 34 to hold it constant. At the heater control 34, this input signal is compared with a set point established at terminal 68. If the sensed ratio is found to be too low, the heater control 34 increases the current delivered over line 38 to the resistance heater 14 mounted about the bubbler 12, thereby causing a higher rate of heat to be applied to the liquid chemicals housed within the bubbler 12. Conversely, if the ratio of the vapor to carrier gas is found to be too low, the heater control 34 decreases the flow of current to the heater 14. The quantitative relationship here may be expressed as:

$$P=K_p \;.\; \text{error} + K_i \int_{0}^{t_1} \text{error}\; dt \;+.\; K_r \frac{{}^{d}\text{error}}{d^{t}}$$

where P is power, K_p the proportionality constant, K_i the integration constant, K_r the rate constant, and t is time.

As the sensor circuit 74 and heater control 34 operate in controlling heater 14, the carrier gas flow rate sensor circuit 72 simultaneously controls the flow rate of the carrier gas to the bubbler. The signals from the ratio sensor circuit 74 are combined with those of the flow rate sensor circuit 72 at the voltage multiplier 58. This signal is compared to a voltage at a control set point 62 for vapor mass flow rate and small deviations are trimmed by automatically making slight adjustments to the carrier gas flow as a means for controlling mass flow rate. It is however emphasized that it is the heater circuit 34 which holds the ratio constant. The expression which relates the vapor flow rate to ratio and carrier flow rate is as follows:

$$\dot{V} = K \times R \times \dot{C}$$

where; $\dot{V}$ = mass flow rate of vapor
K = molecular weight of vapor
R = mole ratio of vapor to carrier
$\dot{C}$ = carrier flow rate to moles.

One example here is provided by 800 cc/min carrier gas intake, a 60% ratio, and 4 gms/min of chemical vapor.

We thus see that whereas before accurate control of vapor delivery over a range of values has not been achievable by solely controlling the flow rate of carrier gas, the present invention does provide a highly accurate control system and method of effecting such by sensing and controlling the ratio of vapor to carrier gas in conjunction with control of carrier gas flow. Indeed, vapor mass flow rate accuracy of ±2% of full scale has now been achieved over the flow range (5% to 100% of full scale). Thus, by directly controlling source to carrier gas ratios, vapor mass delivery rates may be maintained in a highly accurate manner and changed from time to time by changing carrier gas flow rates with predictably accompanying delivery rate changes.

Claims

1. A method of linearly controlling mass flow rates of delivery of vapor from a bubbler (12) containing a supply of liquid through which a carrier gas is bubbled at preselected mutually-diverse_flow rates in which vapor pressure is not in steady state and from which bubbler vapor is delivered in a vapor stream entrained with the carrier gas, the method comprising the step of sensing the ratio of vapor to carrier gas in the vapor stream and characterized by the step of applying heat to the liquid within the bubbler at rates in direct relationship to the sensed ratios of vapor to carrier gas being delivered from the bubbler in the vapor stream to hold the ratio constant.

2. A vapor delivery control method in accordance with claim 1

CHARACTERIZED IN THAT

the ratio of vapor to carrier gas is detected by sensing the heat transfer characteristic of the vapor stream.

3. A vapor delivery control method in accordance with claim 1 or 2

CHARACTERIZED IN THAT

heat is applied to the liquid by thermal conduction into the bubbler (12).

4. A vapor delivery control system comprising a vaporizer (10); a heater (14) thermally coupled with the vaporizer; means (40, 45) for maintaining a supply of liquid in the vaporizer; means (16) for introducing a carrier gas into the vaporizer; means (30) for conducting the carrier gas and vapors of the liquid entrained with the carrier gas from the vaporizer; and means (74) for sensing the ratio of vapor to carrier gas being conducted from the vaporizer; characterized by means (34) for controlling the heater in direct response to ratios of vapor to carrier gas sensed by the sensing means, the controlling means including means (38) for applying heat to the liquid in the vaporizer at rates in direct relationship to the sensed

ratios to hold the ratios constant.

5. A vapor delivery control system in accordance with claim 4

CHARACTERIZED IN THAT

the means (40, 45) for maintaining a supply of liquid in the vaporizer (10) includes means (45) for maintaining the surface of the liquid at a generally constant level within said vaporizer.

6. A vapor delivery control system in accordance with claim 5

CHARACTERIZED IN THAT

means (16) for introducing a carrier gas into the vaporizer (10) includes a conduit having an outlet (18) positioned within the vaporizer below the liquid surface level.

7. A vapor delivery control system in accordance with claim 4

CHARACTERIZED IN THAT

the means (74) for sensing the ratio of vapor to carrier gas includes means (52, 76) for sensing the heat transfer characteristic of the combined carrier gas and vapors being conducted from the vaporizer (10).

AMENDED CLAIMS

(received by the International Bureau on 28 July 1981 (28.07.81))

1. (Amended) A method of controlling the delivery of vapor from liquid into which a carrier gas is introduced, the vapor being entrained with the carrier gas to form a vapor stream, the method comprising the step of sensing the ratio of vapor to carrier gas in the vapor stream and characterized by the step of controlling the application of heat to the liquid in accordance with the sensed ratio.

2. (Amended) A method according to claim 1

CHARACTERIZED IN THAT

the ratio of vapor to carrier gas is detected by sensing the heat transfer characteristic of the vapor stream.

3. (Amended) A method according to claim 1 or 2

CHARACTERIZED IN THAT

the carrier gas flow rate is also sensed and controlled in accordance with the sensed ratio.

4. (Amended) Vapor delivery control apparatus comprising a vaporizer (10), a heater (14) for the vaporizer, means (40, 45) for maintaining a supply of liquid in the vaporizer, means (16) for introducing a carrier gas into the vaporizer, means (30) for conducting from the vaporizer the vapor stream resulting from vapor of the liquid becoming entrained with the carrier gas, and means (74) for sensing the ratio of vapor to carrier gas in the vapor stream,

CHARACTERIZED IN THAT

means (34) control the heater in accordance with the sensed ratio.

5. (Amended) Apparatus according to claim 4,

CHARACTERIZED IN THAT

the heater (14) is an electrical resistance heater thermally coupled to the vaporizer (10), and the controlling means (34) controls the flow of current to the heater.

6. (Amended) Apparatus according to claim 4 or 5

CHARACTERIZED IN THAT

the maintaining means (40, 45) comprises means (45) for maintaining the surface of the liquid at a substantially constant level within the vaporizer.

7. (Amended) Apparatus according to claim 4, 5 or 6

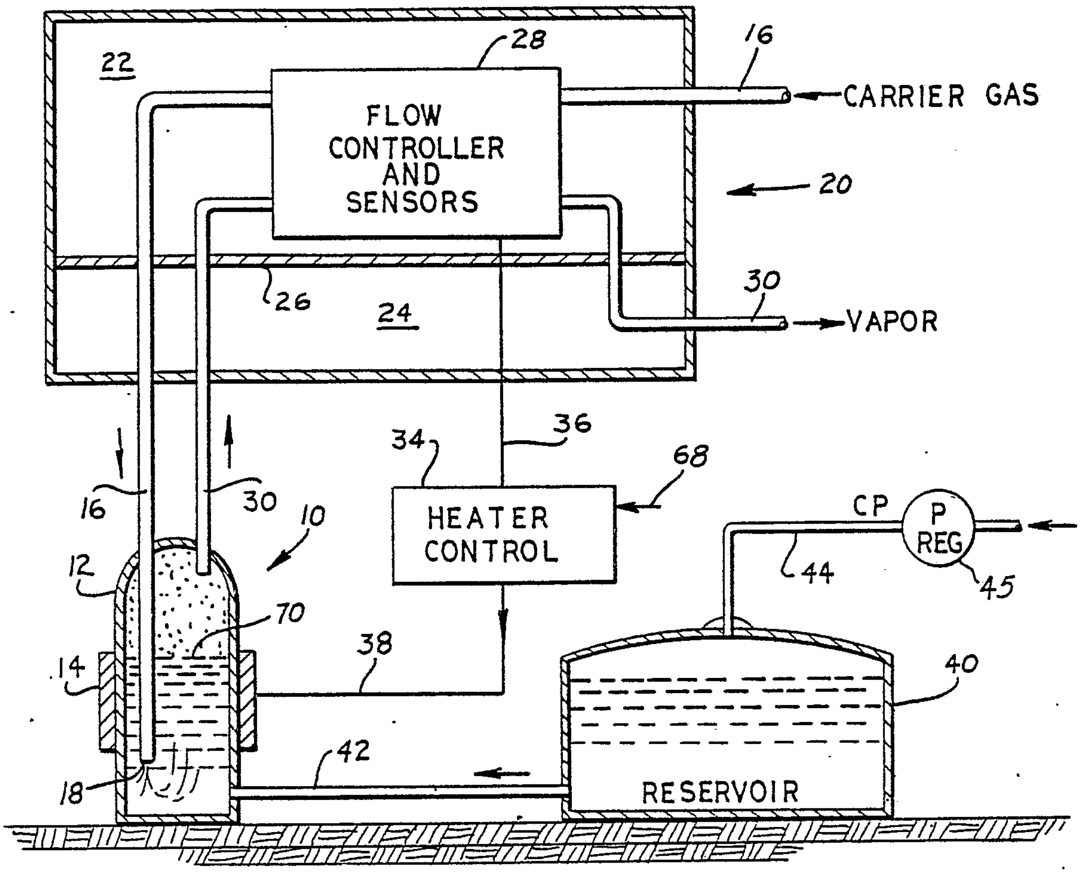
CHARACTERIZED IN THAT

the introducing means (16) has an outlet (18) positioned within the vaporizer (10) below the surface level (70) of the liquid.

8. (New) Apparatus according to claim 4, 5, 6 or 7

CHARACTERIZED IN THAT

means (58, 60, 64) for adjusting the flow rate of the carrier gas are coupled to the sensing means (74).


22
28
FLOW CONTROLLER AND SENSORS
16
CARRIER GAS
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VAPOR
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HEATER CONTROL
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RESERVOIR

Fig. 1

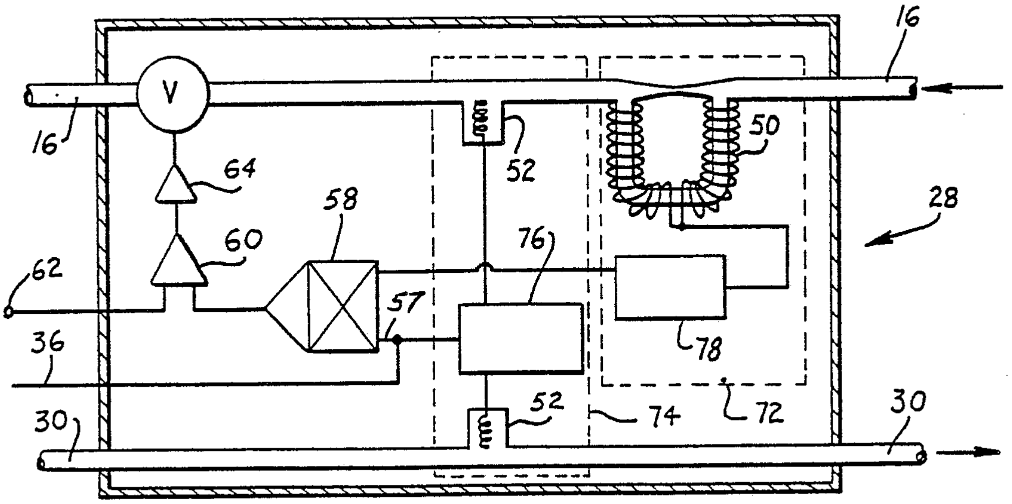
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Fig. 2

INTERNATIONAL SEARCH REPORT

International Application No PCT/US81/00351

I. CLASSIFICATION OF SUBJECT MATTER (if several classification symbols apply, indicate all) [3]

According to International Patent Classification (IPC) or to both National Classification and IPC

INT. CL^3 B01F 3/04

U.S. CL. 261/128,121R,131,142,DIG. 65; 73/204; 137/90; 219/272,273

II. FIELDS SEARCHED

Minimum Documentation Searched [4]

Classification System	Classification Symbols
U.S.	261/121R,128,130,131,142,DIG. 65; 73/29,204; 137/90; 219/271-275

Documentation Searched other than Minimum Documentation to the Extent that such Documents are Included in the Fields Searched [5]

III. DOCUMENTS CONSIDERED TO BE RELEVANT [14]

Category *	Citation of Document, [16] with indication, where appropriate, of the relevant passages [17]	Relevant to Claim No. [18]
X	US,A, 3,583,685, PUBLISHED 08 JUNE 1971, SEE FIG. 1, REF. NUM. 11-13 AND COL. 1, LINES 1-59, BOERGER ET AL.	1-7
X	US,A, 3,939,858, PUBLISHED 24 FEBRUARY 1976, SEE COL. 1, LINES 64-68 AND COL. 2, LINES 1-8, LE MAY.	2-3, 7
X	US,A, 3,938,384, PUBLISHED 17 FEBRUARY 1976, SEE COLUMN 1, LINES 32-34, BLAIR.	2-3, 7
X	US,A, 3,650,151, PUBLISHED 21 MARCH 1972, SEE COL. 2, LINES 23-56, DREXEL.	2-3, 6-7
X	US,A, 3,178,159, PUBLISHED 13 APRIL 1965, SEE FIG. 1, REF. NUM. 22, 24-29, JOHNSON.	5
A	US,A, 3,323,784, PUBLISHED 06 JUNE 1967, FAZIO.	5, 7
A	US,A, 3,528,418, PUBLISHED 15 SEPTEMBER 1970, GROSHOLZ ET AL.	1-7
A	US,A, 3,937,063, PUBLISHED 10 FEBRUARY 1976, KETHLEY.	2-3, 7

* Special categories of cited documents: [15]

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"X" document of particular relevance

IV. CERTIFICATION

Date of the Actual Completion of the International Search [2] 04 JUNE 1981	Date of Mailing of this International Search Report [2] 16 JUN 1981
International Searching Authority [1] ISA/US	Signature of Authorized Officer [20] Richard L. Chiesa RICHARD L. CHIESA