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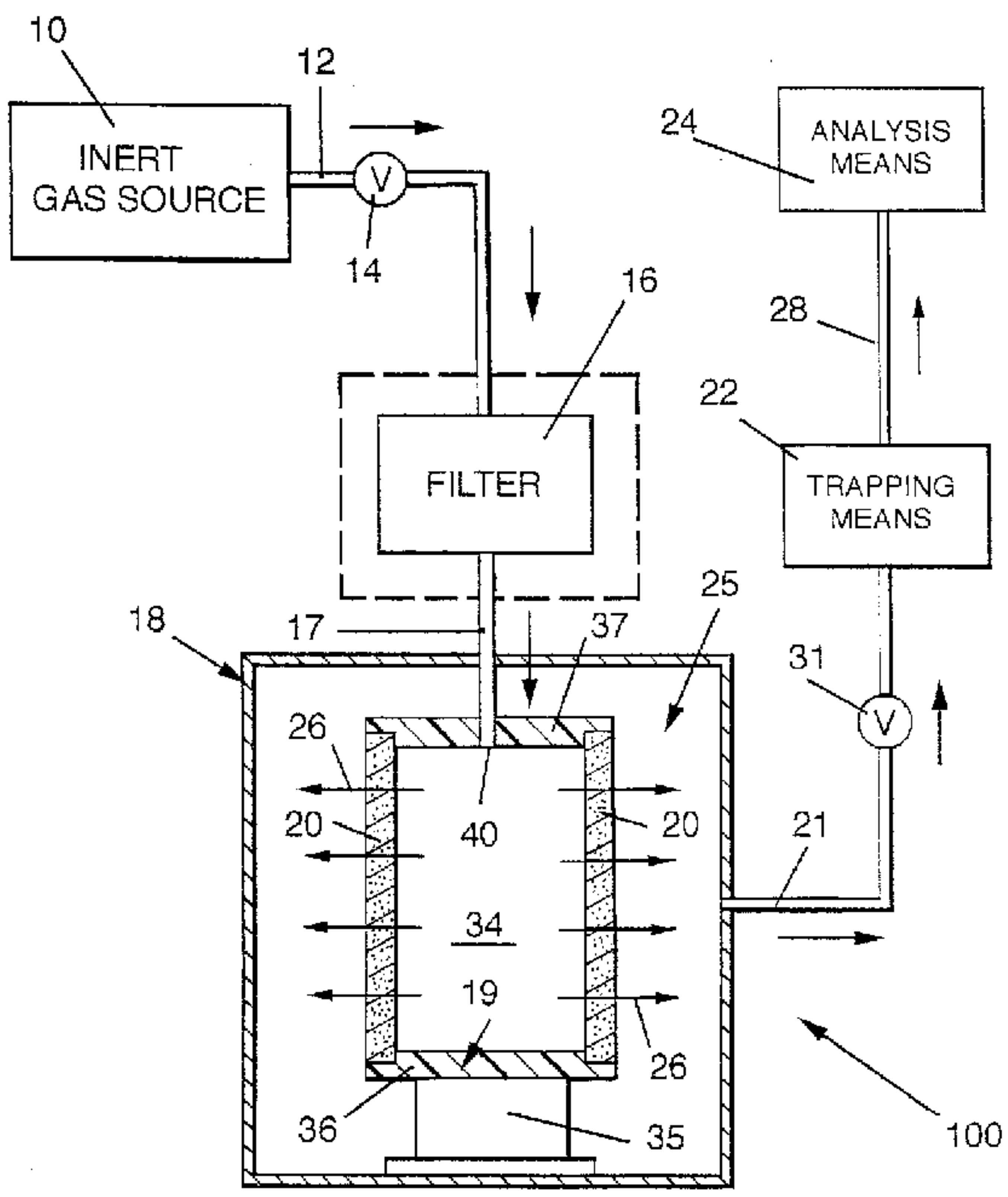
(54) **APPAREIL SERVANT A ANALYSER UNE COMPOSITION VOLATILE DE MATIERE FIXEE DE MANIERE NON PERMANENTE A UN SUPPORT POREUX PLIABLE ET METHODE D'UTILISATION DUDIT APPAREIL**

(54) **APPARATUS FOR ANALYZING A VOLATILE COMPOSITION OF MATTER RELEASABLY BONDED TO A PLIABLE POROUS SUBSTRATE AND PROCESS FOR USING SAID APPARATUS**



(57) Described is apparatus and a process for analyzing a volatile composition such as a perfume composition located on the surface and/or in the interstices of a planar pliable porous substrate such as a towel section. A planar surface of the substrate which contains the volatile composition is initially juxtaposed adjacent a solid wall (e.g., glass frit) porous to a nonreactive carrier gas such as air, nitrogen or carbon dioxide, and fully and tightly covers the porous section of the wall. The carrier gas is passed through the porous section of the wall and then through the pliable porous substrate section which is adjacent to the wall; after which the carrier gas contains molecules of each component of the volatile composition. The composition-carrier gas mixture is then passed through a trapping substance (e. g., TENAX®) which entraps the molecules of each component of the volatile composition. The volatile composition is then analyzed (e. g., using GLC and NMR techniques) after removing the trapping substance containing the entrapped molecules from the apparatus.

ABSTRACT OF THE DISCLOSURE

Described is apparatus and a process for analyzing a volatile composition such as a perfume composition located on the surface and/or in the interstices of a planar pliable porous substrate such as a towel section. A planar surface of the substrate which contains the volatile composition is initially juxtaposed adjacent a solid wall (e.g., glass frit) porous to a nonreactive carrier gas such as air, nitrogen or carbon dioxide, and fully and tightly covers the porous section of the wall. The carrier gas is passed through the porous section of the wall and then through the pliable porous substrate section which is adjacent to the wall; after which the carrier gas contains molecules of each component of the volatile composition. The composition-carrier gas mixture is then passed through a trapping substance (e.g., TENAX®) which entraps the molecules of each component of the volatile composition. The volatile composition is then analyzed (e.g., using GLC and NMR techniques) after removing the trapping substance containing the entrapped molecules from the apparatus.

IFF-13608F

S P E C I F I C A T I O N

TO ALL WHOM IT MAY CONCERN:

BE IT KNOWN THAT I, **WALTER O. LEDIG,** a citizen of the United States of America and resident of 12 Maple Avenue, Matawan, County of Monmouth, State of New Jersey 07747, have invented certain new and useful improvements in:

"APPARATUS FOR ANALYZING A VOLATILE COMPOSITION OF MATTER RELEASABLY BONDED TO A PLIABLE POROUS SUBSTRATE AND PROCESS FOR USING SAID APPARATUS"

of which the following is a specification.

BACKGROUND OF THE INVENTION

My invention covers apparatus for analyzing a volatile composition of matter which is releasably bonded to a pliable porous substrate such as a towel, as well as a process for using such apparatus.

The properties of a pliable substrate (including physical, chemical and microbiological properties) affect the behavior of the substrate with respect to chemicals, particularly volatile chemicals, either naturally present in or on the substrate in a substance applied thereto and so affect the chemicals present in a headspace above the substrate. Conversely, the chemicals entrapped in the interstices of a porous substrate or on the surface of the porous substrate have an effect upon the properties of the substrate, for example, if the substrate is a towel and the towel contains a fragrance, the concentration of that fragrance and the nature of the fragrance in the particular substrate will affect the ultimate aroma in the headspace above the substrate after the substrate is utilized, washed and dried.

Considerably complicated techniques exist in the prior art for analysis of volatile materials contained within a substrate. Thus, U.S. Letters Patent No. 5,891,729 issued on April 6, 1999 ("METHOD FOR SUBSTRATE CLASSIFICATION") discloses a substrate (e.g., skin of unknown type, fabric or hard surfaces) as being characterized by analyzing chemicals emanating from the substrate or from a substance (e.g., a test formulation comprising a mixture of volatile chemicals) applied to the substrate. U.S. Letters Patent No. 5,891,729 indicates that analysis is preferably done using a volatile chemicals sensor, desirably a sensor comprising an array of conducting polymer sensors. The chemcial analysis data obtained in this way, according to U.S. Letters Patent No. 5,891,729, may be statistically analyzed, e.g., by Euclidian distance mapping or principal component analysis for ease of handling. It is further indicated in U.S. Letters Patent No.

5,891,729 that having characterized a surface in this manner, products, e.g., cosmetic and cleaning products, may be formulated for optimized performance on that substrate. More specifically, U.S. Letters Patent No. 5,891,729 discloses a method of characterizing a substrate which comprises applying a test formulation to said substrate, subsequently collecting volatile chemicals in a headspace above the substrate, determining a profile of the volatile chemicals so emanated and using said profile to characterize the substrate. Specific examples of substrates in U.S. Letters Patent No. 5,891,729 are skin, wood, hair, clothing, carpets, plastics, surfaces, ceramic tiles, wool, fabric or perfumed products.

Noting the complexity of the methods and techniques of U.S. Letters Patent No. 5,891,729, it is apparent that a need exists for a more standardized and simplified technique for analysis of a volatile composition of matter releasably bonded to a pliable porous substrate such as a towel.

Thus, for example, U.S. Letters No. 5,891,835 issued on April 6, 1999 discloses a cleaner impregnated towel comprising a flexible porous substrate and impregnated into the substrate a cleaner formulation comprising d-limonene, dibasic acid ester, N-methyl-2-pyrrolidone, secondary alcohol ethoxylate, sodium lauryl sulfate, polysorbate 80, a salt of a coconut oil, fatty acid ester of isethionic acid, glycerine, ethyl alcohol, an antimicrobial preservative and, optionally, water. Although the composition impregnated into the substrate is known initially, after the substrate is utilized, there is no teaching of the nature or concentration of the components impregnated into the substrate after initial use or after repeated use of the substrate, nor is there any teaching in U.S. Letters Patent No. 5,891,835 or 5,891,729 of apparatus or processes for analyzing the contents of such substrate quantitatively or qualitatively using a standardized, simplified procedure as is the case with the instant invention.

SUMMARY OF THE INVENTION

My invention is directed to apparatus which will permit determination of compounds and their concentrations in the headspace over dry cloth as well as moist cloth and other substrates. The compounds are releasably bonded to the surface and/or in the interstices of the porous substrate.

More specifically, my invention is directed to apparatus and a process for analyzing a volatile composition such as a perfumery composition located on the surface and/or in the interstices of a planar pliable porous substrate such as a towel section.

In practicing my invention, a planar surface of the substrate which contains the volatile composition is initially juxtaposed adjacent a solid wall (e.g., glass frit) porous to a nonreactive carrier gas such as air, nitrogen or carbon dioxide and fully and tightly covers the porous section of the wall. The carrier gas is passed through the porous section of the wall and then through the pliable porous substrate section which is adjacent the wall, after which the carrier gas will contain molecules of each component of the volatile composition. The composition-carrier gas mixture is then passed through a trapping substance (e.g., TENAX®) which entraps the molecules of each component of the volatile composition. The volatile composition is then analyzed (e.g., using GLC, NMR and mass spectral techniques) after removing the trapping substance containing the entrapped molecules from the apparatus.

Thus, my invention is directed to apparatus for quantitatively and qualitatively analyzing a volatile substance such as a perfumery material releasably bonded to a substantially planar pliable porous substrate having an inner surface and an outer surface. The substrate is porous to the passage of a carrier gas therethrough in a direction substantially perpendicular to the inner and outer surfaces of the planar pliable porous substrate. The apparatus comprises:

(a) hollow enclosure means (which can be cylindrically shaped or elliptical-cylindrically shaped) having:

(i) hollow outer enclosure means comprising a hollow outer enclosure means first void space, and surrounding said outer enclosure means first void space, an outer gas-impermeable enclosure means wall having exit port means therethrough and entirely surrounding inner enclosure means whereby a second void space exists having volume $\mathbf{V_2}$ within said outer enclosure means and without said inner enclosure means; and

(ii) inner enclosure means located entirely within said outer enclosure means first void space comprising an inner enclosure means third void space, and having two spaced-apart oppositely situated end sections and a central section juxtaposed and communicating with each of said oppositely-situated end sections, said central section consisting essentially of support means having a substantially gas-permeable outer geometric laminar surface with at least one laminar gas-permeable section for both (a) supporting the pliable porous substrate whereby, when the apparatus is in use, said porous substrate covers said laminar permeable section of said central section in an all-encompassing manner; and (b) enabling carrier gas to flow from within said inner enclosure means third void space to the second void space within said outer enclosure and without said inner enclosure, in a direction substantially perpendicular to and through said porous substrate, each of the two end sections being impervious to the flow of gas therethrough and one of said end sections having an entry port means communicating from without said hollow outer enclosure means to the first void space within said inner enclosure means, said first void space having a volume V_1;

(b) analytical apparatus means located downstream from said hollow enclosure means and communicating with the exit port means thereof, comprising tube trapping means whereby volatile substance molecules emitted from said pliable porous substrate during gas flow therethrough (for example, perfume molecules such as phenyl ethyl alcohol molecules emitted from a towel section) are entrapped in said tube trapping means; and

(c) upstream from said hollow enclosure means or downstream from said analytical apparatus means, inert gas flow effecting means for effecting the flow of inert gas sequentially (i) through said entry port means; (ii) through porous pliable substrate means located on said support means; (iii) through said exit port means; and (iv) through said anlytical apparatus means.

My invention is also directed to a process for quantitatively and qualitatively analyzing a volatile substance (such as a fragrance composition or an insect-repelling composition) releasably bonded to a substantially planar pliable porous substrate (for example, a cloth or a towel fabricated from cotton or polyester) having an inner surface and an outer surface comprising the step of:

(a) providing the apparatus as set forth, supra;

(b) providing a substantially planar pliable porous substrate having releasably bonded thereto a volatile substance such as a perfumery substance;

(c) juxtaposing in an all-encompassing manner said porous substrate with said support means of said central section of said inner enclosure means of said apparatus whereby the inner surface of said porous substrate is removably adhered to and intimately adjacent to the entirety of the laminar gas-permeable section of the outer geometric laminar surface of the support means;

(d) effecting the flow of carrier gas sequentially (i) from a location upstream from the entry port means; (ii) into the inner enclosure means through said entry port means; (iii) past said support mean; (iv) through said porous substrate means in a direction substantially perpendicular thereto, in a substantially evenly distributed manner across the inner and outer surface thereof; (v) into and through said second void space; (vi) through said exit port means of said hollow outer enclosure means and into and through said analytical apparatus means; and (vii) whereby volatile substance molecules (e.g., perfumery composition molecules) emitted from said porous substrate are trapped in a trapping means of the analytical apparatus means; and

(e) employing the analytical apparatus means whereby analysis of the releasably bonded volatile molecules is effected.

Preferably, the hollow enclosure means of the above-described apparatus contains two concentric cylindrical enclosures, with the outer cylindrical enclosure being impervious to gas except for an exit port and with the inner enclosure means having an entry port and having a centrally located solid porous surface (e.g., glass frit, or solid microporous polymer).

Preferably, after the pliable porous substrate is in place on the inner enclosure, carrier gas is forced through the inner enclosure past the porous substrate into the outer enclosure and then out of the outer enclosure into the analytical means which preferably contains a trapping material. The carrier gas, such as nitrogen, air or carbon dioxide, is inert and nonreactive with the porous substrate or with the volatile substance releasably bonded to the porous substrate. The carrier gas can either be forced through from a pressurized device upstream from the hollow enclosure means (e.g., a pressurized carbon dioxide cylinder), or the carrier gas can be pulled through using means downstream from the analytical apparatus means such as a vacuum pump.

Whether the inert gas flow effecting means is upstream from the remainder of the apparatus or downstream from the remainder of the apparatus, it is preferable to have a gas filter in place in the apparatus of my invention, upstream from the hollow enclosure means so that the inert gas is free of any contaminants which would interfere with the analysis of the composition releasably bonded to the substantially planar pliable porous substrate (e.g., towel section).

Preferably, the hollow outer enclosure means of the hollow enclosure means part of the apparatus of my invention is cylindrical and has a height dimension of from about 4 cm up to about 20 cm and a diameter dimension of from about 4 cm up to about 12 cm. Preferably, the inner enclosure means of the hollow enclosure means part of the apparatus of my invention is cylindrical and has a height dimension between from about 50% up to about 85% of the height dimension of the hollow outer enclosure means and a diameter dimension of from about 40% up to about 70% of the diameter dimension of the hollow outer enclosure means.

The support means part of the inner enclosure means (that is, the central section of the inner enclosure means) is preferably cylindrical or substantially cylindrical in shape and as stated, supra, is preferably glass frit or microporous polymer. However, other suitable support means are useful in the practice of my invention, for example, the material which is marketed as cylindrical filter screens by the B.C. McDonald & Company of St. Louis, Missouri 63132 under the description of "Ronningen-Petter Woven Wire Screen"; or Ronningen-Petter Woven Synthetic Screen (illustrated in Figure 1E which is described in the Brief Description of the Drawings and in the Detailed Description of the Drawings sections, infra); or the Ronningen-Petter Perforated Screen. The Ronningen-Petter Screens are manufactured by the Dover Corporation/Ronningen-Petter Division, P.O. Box 188, Portage, Michigan 49081. The Ronningen-Petter Cylindrical Screens useful as support means in the practice of our invention are specifically described in literature published by Ronningen-Petter entitled ***"how to select filter screens* for the removal of trace contaminants in a closed liquid system."**

Other support means useful in fabrication of the central section of the inner enclosure means of the apparatus of my invention are described in U.S. Letters Patent No. 5,762,797 issued on June 9, 1998 entitled "ANTIMICROBIAL FILTER CARTRIDGE" and U.S. Letters Patent No. 5,868,933 issued on February 9, 1999 entitled "ANTIMICROBIAL FILTER CARTRIDGE."

With respect to the analytical apparatus means located downstream from the hollow enclosure means and communicating with the exit port means of the hollow outer enclosure means, the analytical means part of the apparatus of my invention as stated, supra, comprises tube trapping means whereby volatile substance molecules emitted from the pliable porous substrate during gas flow therethrough are entrapped in the tube trapping means. The tube trapping means preferably consists of a tube having a length in the range of from about 2 cm up to about 4 cm and a diameter of from about 0.1 cm up to about 0.4 cm. Thus, various trapping materials are useful in the practice of my invention. As stated, supra, TENAX® is a preferable material. Various forms of TENAX® are useful, for example, TENAX®-GC. TENAX® is a registered trademark of ENKA, N.V. of the Kingdom of the Netherlands (CAS Registration No. 2438-68-9). Other forms of TENAX® and methods of production of such forms of TENAX® are described in the following U.S. Letters Patents:

U.S. Letters Patent No. 3,400,100 issued on September 30, 1968 ("PROCESS FOR THE PREPARATION OF POLYPHENYLENE ETHERS");

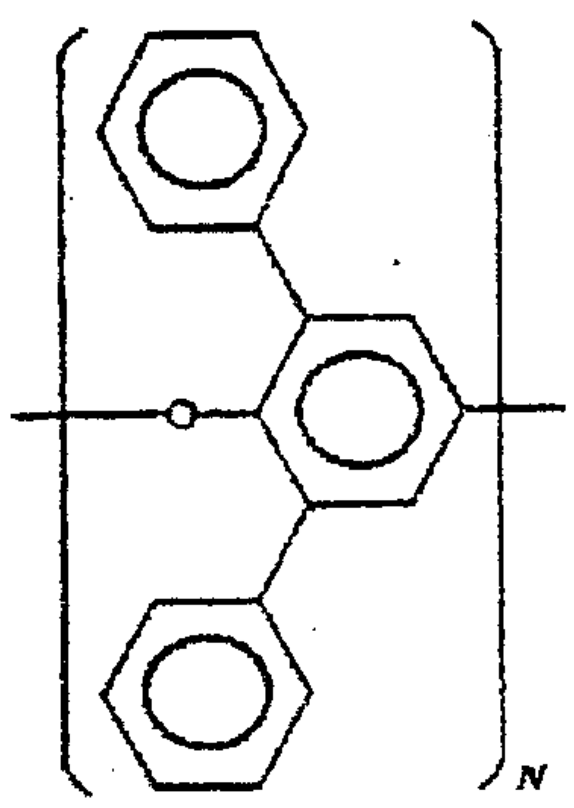
U.S. Letters Patent No. 3,644,227 issued on February 22, 1972 ("SEPARATION OF POLY(2-6-DIMETHYL-1,4-PHENYLEOXIDE") FROM ITS BLENDS WITH OTHER POLYMERS);

U.S. Letters Patent No. 3,703,564 issued on November 21, 1972 (BIS-POLYPHENYLENEOXIDE]ESTER BLOCK COPOLYMERS");

U.S. Letters Patent No. 4,431,779 issued on February 14, 1984 (POLYETHERAMIDE-POLYPHENYLENE ETHER BLENDS"); and

U.S. Letters Patent No. 4801,645 issued on January 31, 1989 ("THERMOPLASTIC RESIN COMPOSITION").

TENAX®-GC is actually a polyphenyleneoxide defined according to the structure:



wherein **N** is an integer of from about 100 up to about 150.

Other trapping materials useful in the practice of my invention are as follows:

Activated Carbon marketed by Aldrich Chemical Company of 1001 West Saint Paul Avenue, Milwaukee, Wisconsin 53233 (Catalog Nos. 16, 155-1; 29, 259-1; 24, 223-3; 24, 224-1; and 24, 227-6);

Activated Alumina marketed by Sigma Chemical Company of St. Louis, Missouri (Catalog Nos. A8753; A8878; A9003; A1522; and A2272);

Silica Gels marketed by Sigma Chemical Company (for example, Catalog Nos. S4004; S6628; and H8506); and

CHROMOSORB® (registered trademark of the Johns-Manville Company of Manville, New Jersey), such as CHROMOSORB® LC-2; CHROMOSORB® LC-3; AND CHROMOSORB® LC-7, marketed by Sigma Chemical Company under Catalog Nos. C 0641; C 0766; C 5517 and C 6269.

The analytical apparatus means useful in the practice of my invention may contain, in place of the TENAX® trapping substance, solid phase microextraction materials ("SPME" materials) such as those described in *Bulletin 869* published by SUPELCO, INC., Supelco Park, Bellefonte, Pennsylvania 16823-0048. An SPME example useful in the practice of my invention is 100 μm polydimethylsiloxane fiber, Catalog No. 5-7300 of Supelco, Inc.. An additional description of the SPME (solid phase microextraction) technique useful in conjunction with the practice of my invention is the paper, Elmore, et al, *J. Agric. Food Chem.*, 1997, Volume 45, pages 2638-2641, entitled "Comparison of Dynamic Headspace Concentration on Tenax [TENAX®] with Solid Phase Microextraction for the Analysis of Aroma Volatiles."

As stated, supra, the means for effecting the flow of inert gas sequentially (i) through the entry port means of the inner enclosure means; (ii) and through the porous pliable substrate means located on the support means of the apparatus of my invention can be located downstream from the analytical apparatus means. If that is the case, the inert gas flow effecting means is a negative pressure pump means, preferably a vacuum pump of the "low flow" variety, for example, "Low Flow" pumps marketed by the Ametek Company of Largo, Florida 34643 (the "Ametek Constant flow Sampler").

The flow rate of inert carrier gas past the porous pliable substrate is preferably at a rate in the range of from about 20 ml per minute up to about 200 ml per minute of carrier gas, e.g., nitrogen, air or carbon dioxide.

At the indicated rates of carrier gas flow, a range of molar rates of release of volatile composition will occur from the porous substrate, e.g., towel section, in accordance with the following algorithm:

$$\Delta n = n_1\left[\frac{V_1}{V_2}\,e^{-\frac{2C_V}{zR}\left[\frac{T_2-T_1}{T_2+T_1}\right]} - 1\right]$$

wherein n_1 is the carrier gas flow rate in gram moles per hour;

Δn is the molar flow rate (in gram moles per hour) of release of volatile composition from the pliable porous substrate;

V_1 is the volume of the inner enclosure;

T_1 is the temperature of the void space of the inner enclosure in °K (degrees Kelvin);

$\mathbf{V_2}$ is the volume between the porous pliable substrate and the outer enclosure;

$\mathbf{T_2}$ is the temperature of the carrier gas and volatile composition released from the pliable porous substrate (that is, the temperature of volume $\mathbf{V_2}$) in °K;

R is the gas constant $\left[0.08206 \dfrac{\text{liter} - \text{atm}}{\text{gm mole} - {}^{\circ}\text{K}}\right]$;

z is the compressibility factor of the carrier gas; and

$\mathbf{C_V}$ is the heat capacity of the carrier gas defined as $\left(\dfrac{\partial \mathbf{E}}{\partial \mathbf{T}}\right)_{\mathbf{V}}$, wherein **E** is the internal energy of the carrier gas during flow through the apparatus of my invention.

BRIEF DESCRIPTION OF THE DRAWINGS

Figure 1A is a schematic block flow diagram showing the operation of the apparatus of my invention and the process of my invention.

Figure 1B is another schematic block flow diagram showing the operation of the apparatus of my invention as well as the process of my invention and showing the use of pressure measuring devices in conjunction with the apparatus of my invention; and, in addition, showing the use of inert gas flow effecting means for effecting the flow of inert gas through the apparatus of my invention, upstream from the hollow enclosure portion of the apparatus of my invention, specifically as a pressurized gas source (e.g., cylinder of pressurized air).

Figure 1C is another schematic block flow diagram showing the operation of the apparatus of my invention and the process of my invention and also showing inert gas flow effecting means for effecting the flow of inert gas through the apparatus of my invention, which flow effecting means is in the form of vacuum pump means downstream from the analytical apparatus means.

Figure 1D is another schematic block flow diagram showing the use of the apparatus of my invention when in actual operation analyzing a pliable porous substrate material containing material to be analyzed (e.g., a fragrance composition).

Figure 1E is a cutaway perspective diagram of an example of a laminar gas-permeable section of the central section of the inner enclosure means of the apparatus of my invention ("Ronningen-Petter Woven Synthetic Screen" manufactured by the Ronningen-Petter Division of the Dover Corporation, P.O. Box 188, Portage, Michigan 49081).

Figure 2 is a detailed cutaway side elevation view of a preferred embodiment of the apparatus of my invention showing the employment of fritted glass as a laminar gas permeable section of the central section of the inner enclosure means of the apparatus of my invention.

Figure 3A is a perspective view of a preferred embodiment of the apparatus of my invention, showing the outer enclosure means fabricated from ceramic quartz glass and showing the central part of the inner enclosure means fabricated from fritted glass.

Figure 3B is a perspective view of a preferred embodiment of that part of the apparatus of my invention which is the inner enclosure means wherein the central section consists of a fritted glass laminar gas-permeable section and wherein the porous pliable planar substrate to be analyzed is a towel section about to be placed fully covering and adjacent to the fritted glass section of the inner enclosure means.

Figure 3C is a top cutaway schematic view of the inner enclosure means of the apparatus of my invention having juxtaposed and adjacent thereto the porous pliable substrate to be analyzed for a volatile composition contained thereon or in the interstices thereof.

Figure 4 is the GC-mass spectrum of a fragrance composition releasably bonded to a towel section, which composition was analyzed for using the apparatus and process of my invention according to the procedure of Example I, infra (conditions: 50 meter x 320 μ x 0.52 μ bonded fused silica methyl silicone column programmed from 80-220°C at 8°C per minute).

DETAILED DESCRIPTION OF THE DRAWINGS

Referring to Figures 1A, 1B, 1C and 1D, gas from gas source 10 is passed through line 12 past valve 14 through carrier gas filter 16 (optionally) through line 17 into the inner enclosure means 19, which is a support means for porous material 20. The carrier gas passes through entry port 40 into void 34. The inner enclosure means has top 37 and base 36. The inner enclosure means is supported via support 35 within the outer enclosure means 18. Carrier gas flows from void 34 into void 25 of the outer enclosure means (with the flow being shown by reference numeral 26). The carrier gas is then passed through line 21 past valve 31 into and through trapping means 22 wherein molecules of volatile material from the porous material at 20 are trapped. The trapping substance containing trapped molecules is then conveyed via route 28 to analysis means 24 (e.g., NMR, IR and mass spectral analytical equipment). Overall, the apparatus is indicated by reference numeral 100.

Specifically referring to Figure 1B, pressurized gas (e.g., air) from, for example, a pressurized air vessel 11, is passed through line 12 into the apparatus of my invention, initially through line 17 via entry port 40. In Figure 1B, pressure indicator 13 is located on line 12, and pressure indicator 27 is located in the outer enclosure means 18 whereby a pressure drop between line 14 primarily across porous wall 20 is measured.

Referring specifically to Figure 1C, carrier gas from gas source 10 is pulled through the apparatus by means of vacuum pump means 23 located downstream from the trapping means 22. Inert carrier gas is pulled through the apparatus using vacuum pump means 23 through line 28 which is connected to trapping means 22. The resulting trapped molecules are then conveyed on the trapping substance via conveying means 30 to analysis means 24.

Referring specifically to Figure 1D, the central section of inner enclosure means 19 is composed of glass frit shown by reference numeral 42. Carrier gas entering at entry port 40 into void 34 within the inner enclosure means then passes through the glass frit 42 and through the pliable porous substrate 20. The passage of the inert gas again is shown by reference numeral 26 wherein the carrier gas now containing molecules of volatile substance is passed into void 25 of outer enclosure means 18.

Referring to Figure 1E, inner support means 42a (Ronningen-Petter Woven Synthetic Screen) supports the pliable porous substrate containing volatile composition therein and/or thereon 20.

Referring to Figure 2, inert carrier gas, e.g., air, passes through tube 17 past apparatus entry location 50 through entry port 40 (the entry port for the inner enclosure means) into void 34 and then through fritted glass 42 into the void between the outer container means 18 and the inner container means 19. The flow of carrier gas is shown by reference numeral 26. The top of the outer enclosure means is sealed to the lower section thereof 18 (which has base 18a) with Teflon seal 49. The carrier gas containing molecules of volatile substance is then passed through line 21 past Swageloc connector 48 into TENAX® trap 22. The fritted glass support 42, in the case of the apparatus of Figure 2, is 4" in length x 1.5" in diameter and will hold a piece of cloth 4" in length x 5.25" in width.

Referring to Figure 3A, the apparatus 100 contains the upper inlet tube 18 and an inner enclosure means 42 having base 36 and outer enclosure means 18 having base 18a. Carrier gas flows through tube 21 into TENAX® trap or SPME trap 22.

Referring to Figure 3B, the pliable porous substrate 20 is a section of a towel which is to be juxtaposed immediately adjacent to and fully covering the fritted glass central part of the inner enclosure means 42.

Referring to Figure 3C, the void space of the inner enclosure means 34 has carrier gas flowing therethrough in a direction perpendicular to the support means 42 for the pliable porous substrate 20 containing volatile composition (e.g., perfume composition) to be analyzed with the carrier gas flow being shown by reference numeral 26.

* * * * *

The detailed description of the operation of the apparatus of Figure 3A is set forth in the description of Example I, infra.

Thus, the following Example I is illustrative of my invention, but my invention is only limited by the scope of the claims following said example.

EXAMPLE I

ANALYSIS OF CONTENTS OF FRAGRANCE COMPOSITION RELEASABLY ADHERED TO TOWEL

OBJECTIVE:

To analyze the contents of a fragrance material originally situated in the interstices of a cotton towel.

PROCEDURE:

A 4" x 5.25" cotton towel section containing 0.005% by weight fragrance composition is tightly wrapped around the central section of the inner enclosure of the apparatus of Figure 3A. The inner enclosure thereof is composed of a porous fritted glass. Air from location 11 (Figure 1B) is passed through the apparatus at a rate of 40 ml per minute for a period of 7 hours. Trapping means 22 contains a TENAX®-GC trap. At the end of the 7-hour period, the air flow was terminated and the TENAX®-GC trap was opened and the contents analyzed. The contents of the trap were analyzed by GC-MS analysis using a 50 m x 0.32 mm OV-2 fused silica column having conditions: 80-220°C at 8°C per minute.

Figure 4 is the GC mass spectrum for the perfume composition located on the towel, which is the subject of this example.

WHAT IS CLAIMED IS:

1. Apparatus for quantitatively and qualitatively analyzing a volatile substance releasably bonded to a substantially planar pliable porous substrate having an inner surface and an outer surface, said substrate being porous to the passage of a carrier gas therethrough in a direction substantially perpendicular to the inner and outer surfaces of said planar pliable porous substrate comprising:

(a) hollow enclosure means having:

(i) hollow outer enclosure means comprising a hollow outer enclosure means first void space and, surrounding said outer enclosure means first void space, an outer gas-impermeable enclosure means wall having exit port means therethrough and entirely surrounding inner enclosure means, whereby a second void space exists having volume V_2 within said outer enclosure means and without said inner enclosure means; and

(ii) inner enclosure means located entirely within said outer enclosure means first void space comprising an inner enclosure means third void space having volume V_1 and having two spaced-apart oppositely situated end sections and a central section juxtaposed to and communicating with each of said oppositely-situated end sections, said central section consisitng essentially of support means having a substantially gas-permeable outer geometric laminar surface with at least one laminar gas-permeable section for both (A) supporting the pliable porous substrate whereby, when in use, said porpous substrate covers said laminar gas-permeable section of said central section in an all encompassing manner and (B) enabling carrier gas to flow from within said inner enclosure means third void space to the second void space within said outer enclosure and without said inner enclosure, in a direction substantially perpendicular to and through said porous substrate, each of the two end sections being impervious to the flow of gas therethrough and one of said end sections having an entry port means communicating from without said hollow outer enclosure means to the first void space within said inner enclosure means;

(b) analytical apparatus means located downstream from said hollow enclosure means and communicating with the exit port means thereof, comprising tube trapping means whereby volatile substance molecules emitted from said pliable porous substrate during gas flow therethrough are entrapped in said tube trapping means; and

(c) upstream from said hollow enclosure means or downstream from said analytical apparatus means, inert gas flow effecting means for effecting the flow of inert gas sequentially (I) through said entry port means; (II) through porous pliable substrate means located on said support means; (III) through said exit port means; and (IV) through said analytical apparatus means.

2. A process for quantitatively and qualitatively analyzing a volatile substance releasably bonded to a substantially planar pliable porous substrate having an inner surface and an outer surface comprising the steps of:

(a) providing the apparatus of Claim 1;

(b) providing said porous substrate;

(c) juxtaposing in an all encompassing manner said porous substrate with said support means of said central section of said inner enclosure means of said apparatus whereby the inner surface of said porous substrate is removably adhered to and intimately adjacent to the entirety of the laminar gas-permeable section of the outer geometric laminar surface of the support means;

(d) effecting the flow of carrier gas sequentially (I) from a location upstream from the entry port means; (II) into the inner enclosure means; (III) past said support means through said entry port means; (IV) through said porous substrate means in a direction substantially perpendicular thereto in a substantially evenly distributed manner across the inner and outer surface thereof; (V) into and through said second void space; (VI) through said exit port means of said hollow outer enclosure means and into and through said analytical apparatus means; and (VII) whereby volatile substance molecules emitted from said porous substrate are trapped in said analytical apparatus means; and

(e) employing the analytical apparatus means whereby analysis of the volatile molecules is effected.

3. The apparatus of Claim 1 wherein the central section of the inner enclosure means comprises porous glass frit.

4. The apparatus of Claim 1 wherein carrier gas filtering means is immediately upstream from said entry port of said inner enclosure means.

5. The apparatus of Claim 1 wherein carrier gas flow is effected by means of positive pressure from a source upstream from said inner enclosure means.

6. The apparatus of Claim 1 wherein carrier gas flow is effected into the inner enclosure means by means of negative pressure means downstream from said trapping means.

7. The apparatus of Claim 1 wherein the analytical apparatus means comprises a tube containing TENAX® trapping material.

8. The apparatus of Claim 1 wherein the analytical apparatus means comprises a solid phase microextraction means.

9. The process of Claim 2 wherein in step (e) a TENAX® trap is used and molecules extracted from the TENAX® trap are analyzed by means of NMR and IR analyses.

10. The process of Claim 2 wherein in step (e) solid phase microextraction is used, and the molecules obtained from the solid phase microextraction procedure are analyzed by means of NMR and IR analyses.

11. The process of Claim 9 wherein the pliable porous substrate is a cotton towel having fragrance molecules located thereon.

12. The process of Claim 10 wherein the pliable porous substrate used is a towel having fragrance molecules located thereon.

13. The process of Claim 11 wherein the carrier gas used is air.

14. The process of Claim 12 wherein the carrier gas used is air.

15. The apparatus of Claim 1 wherein the outer enclosure means and the inner enclosure means are in the shape of cylinders.

FIG. 1-A

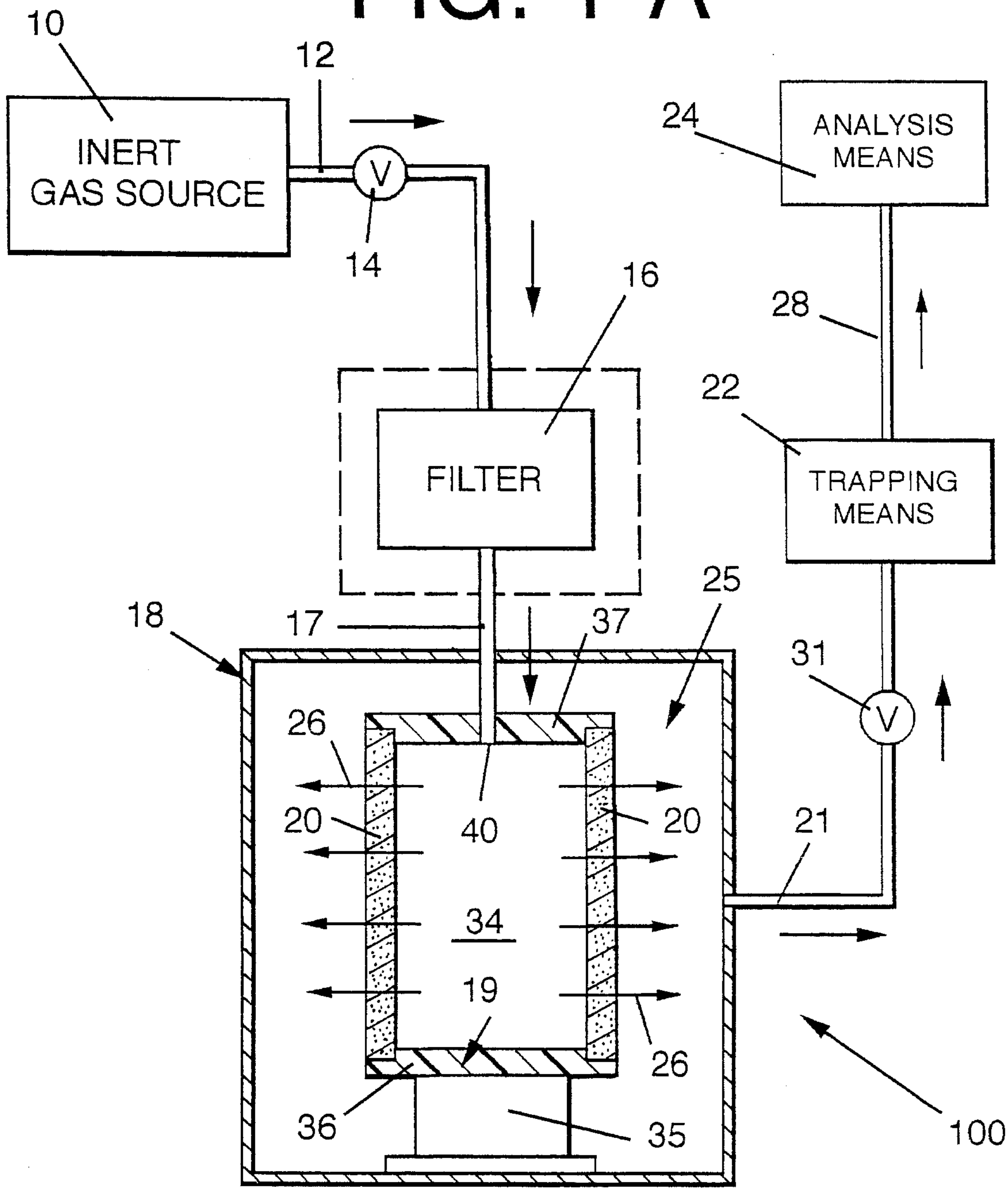


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FIG. 1-B

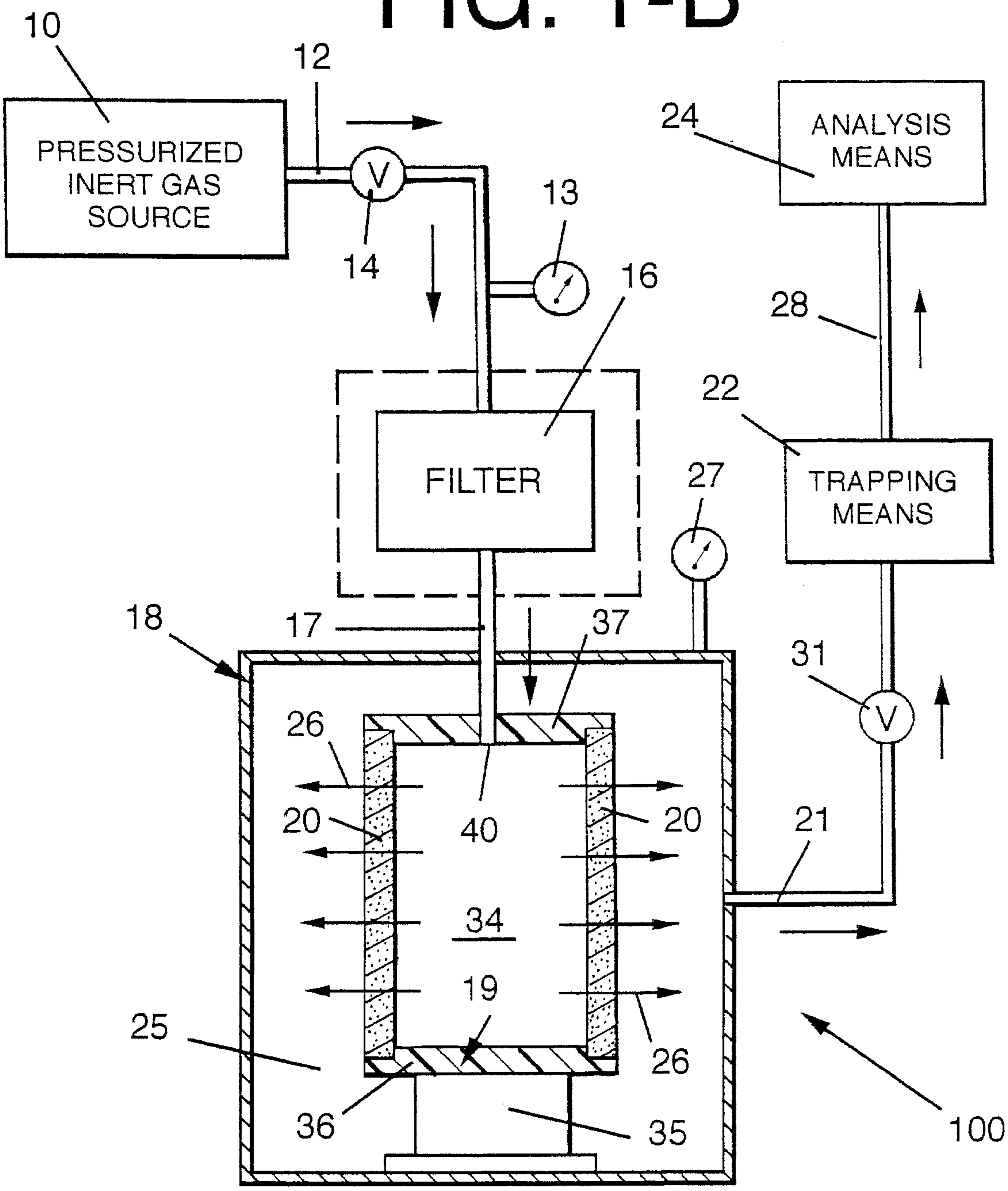
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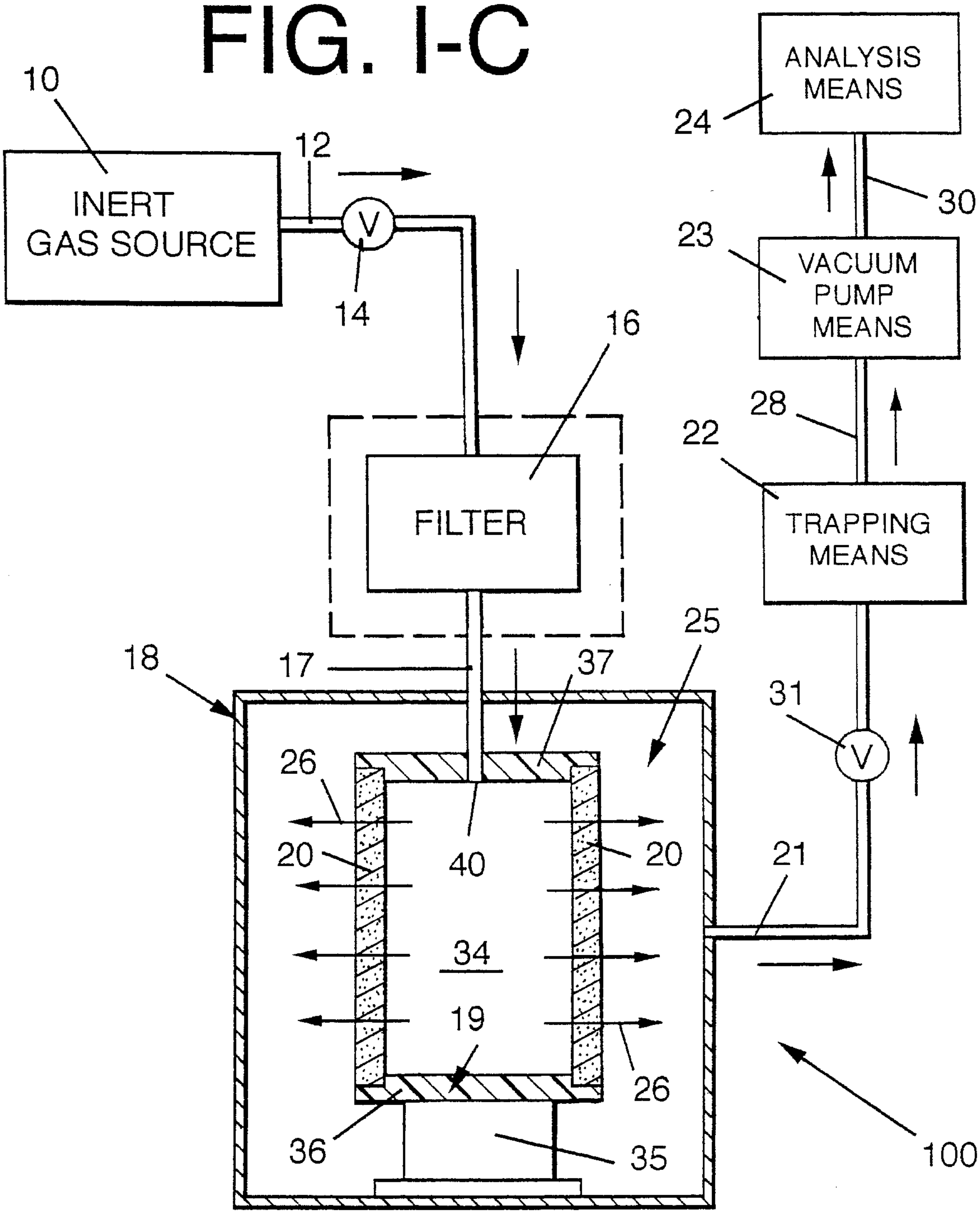
FIG. I-C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ANALYSIS
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FIG. I-D

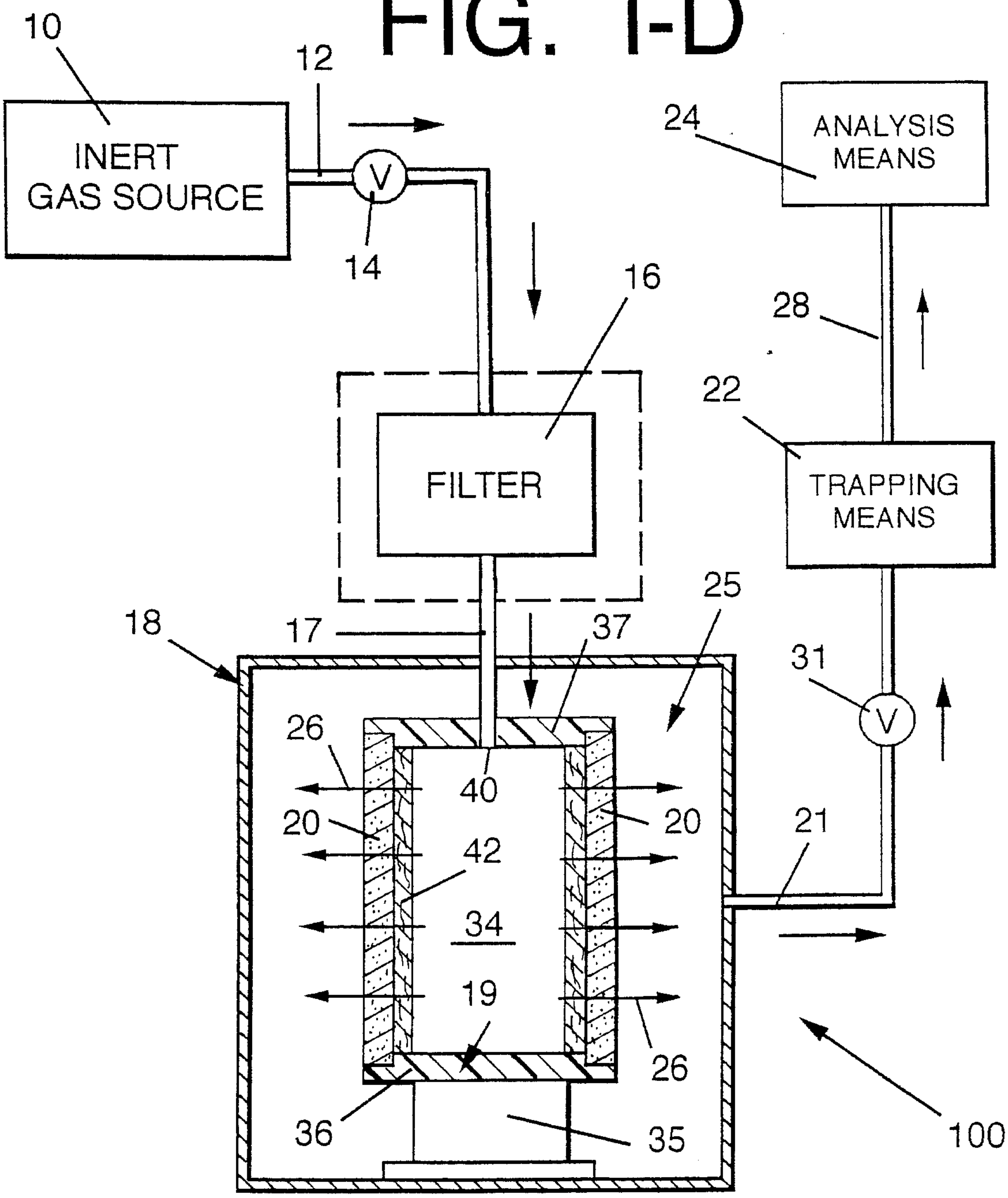
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FIG.1-E
PRIOR ART

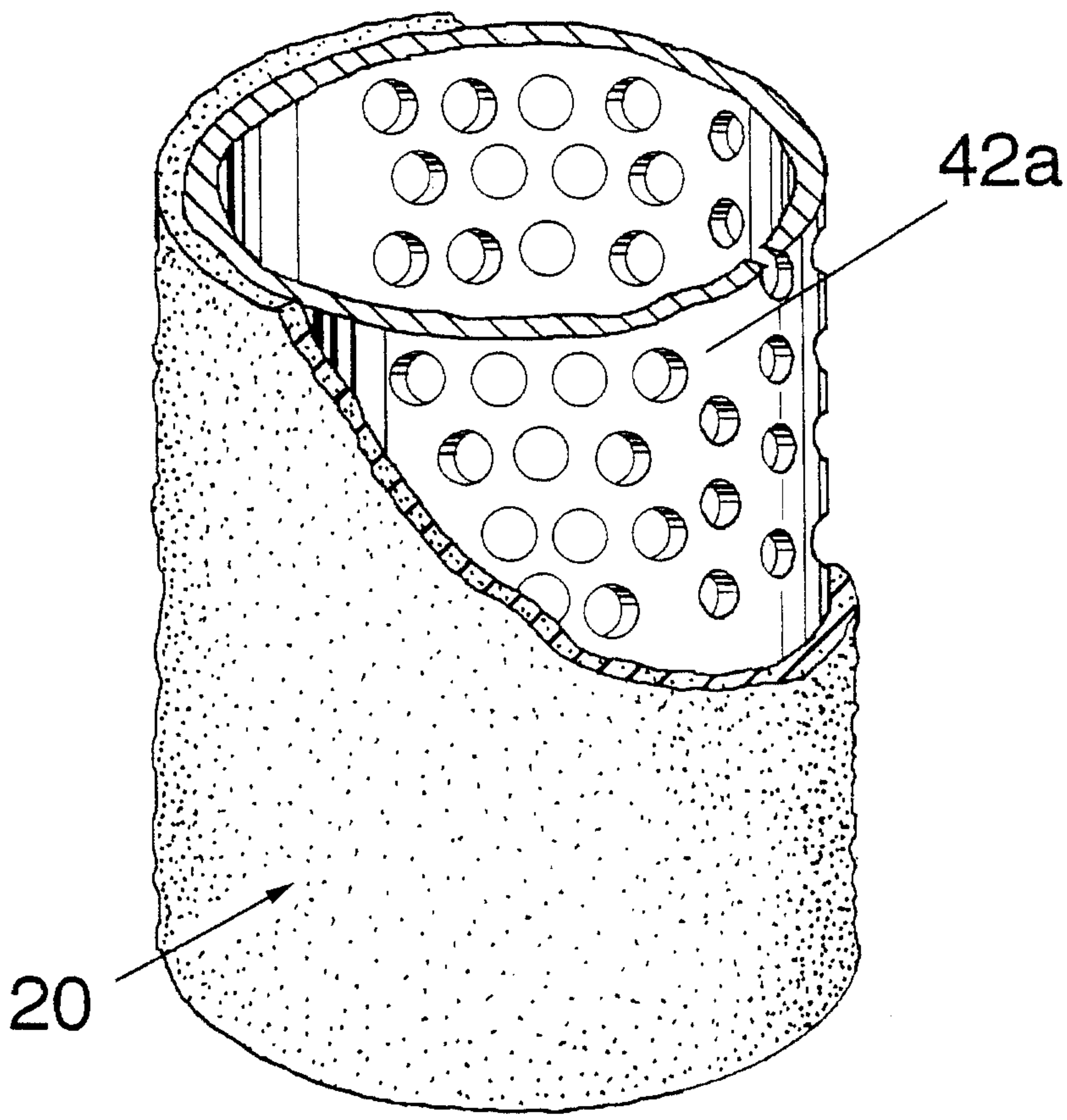


FIG. 2

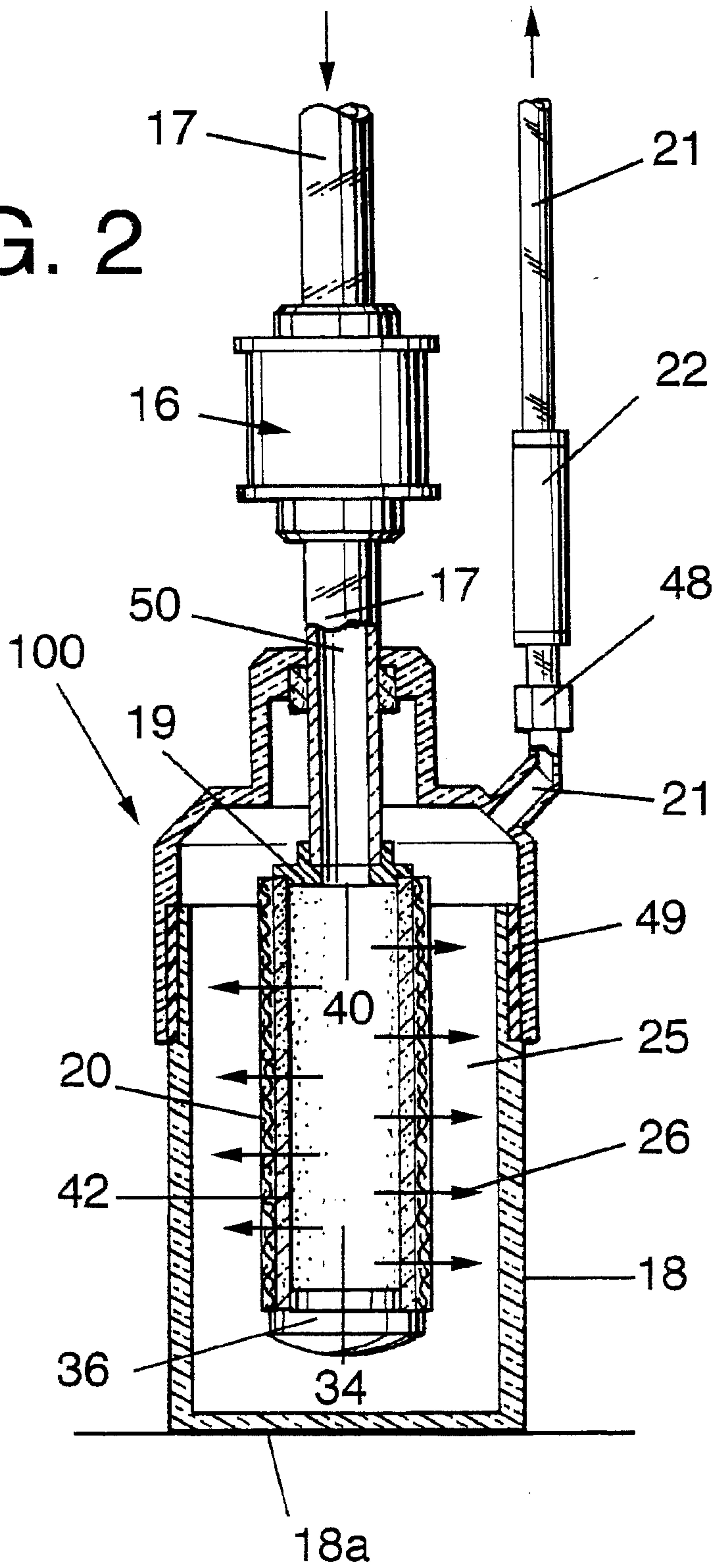


FIG. 3-A

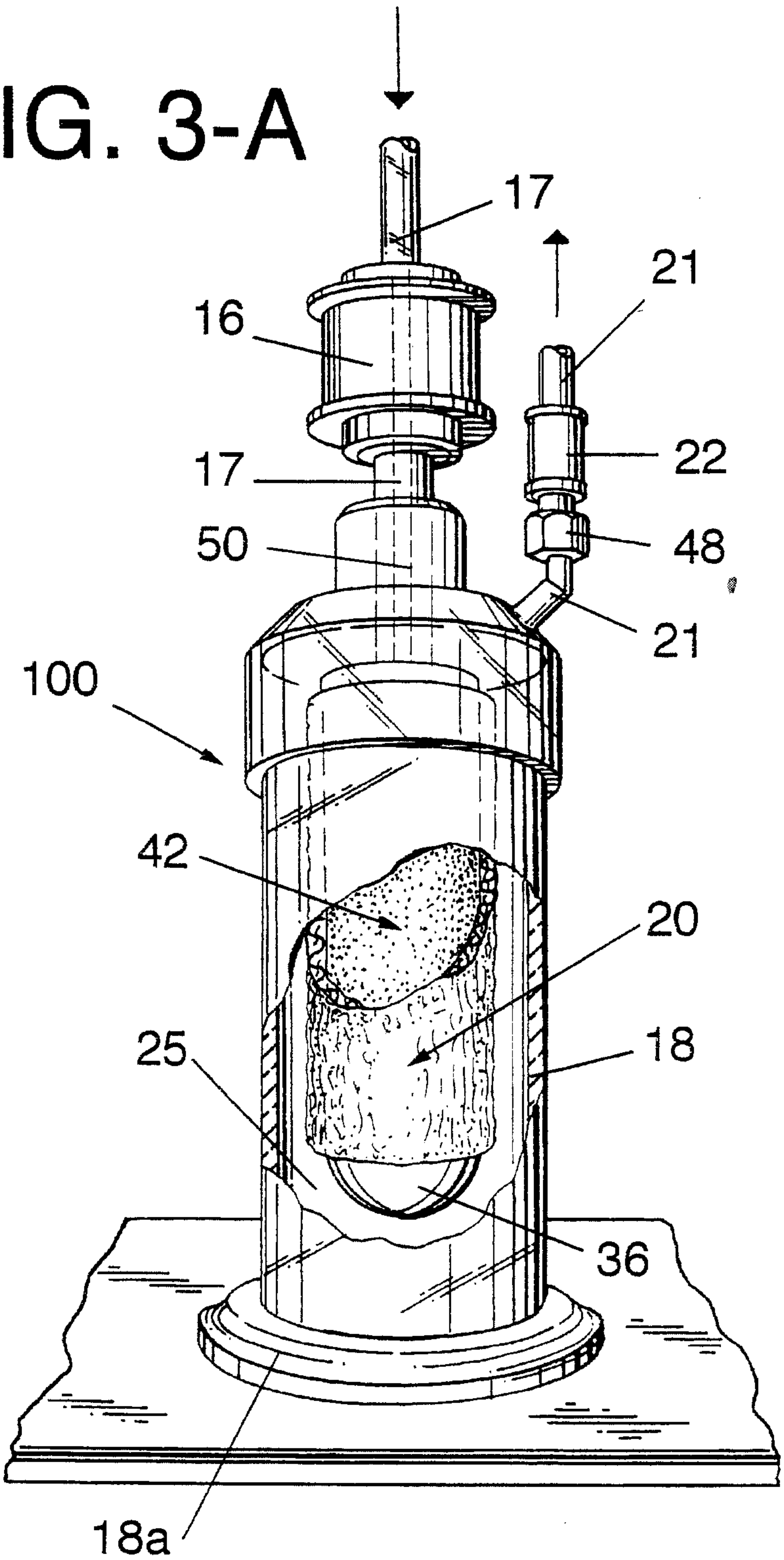


FIG. 3-B

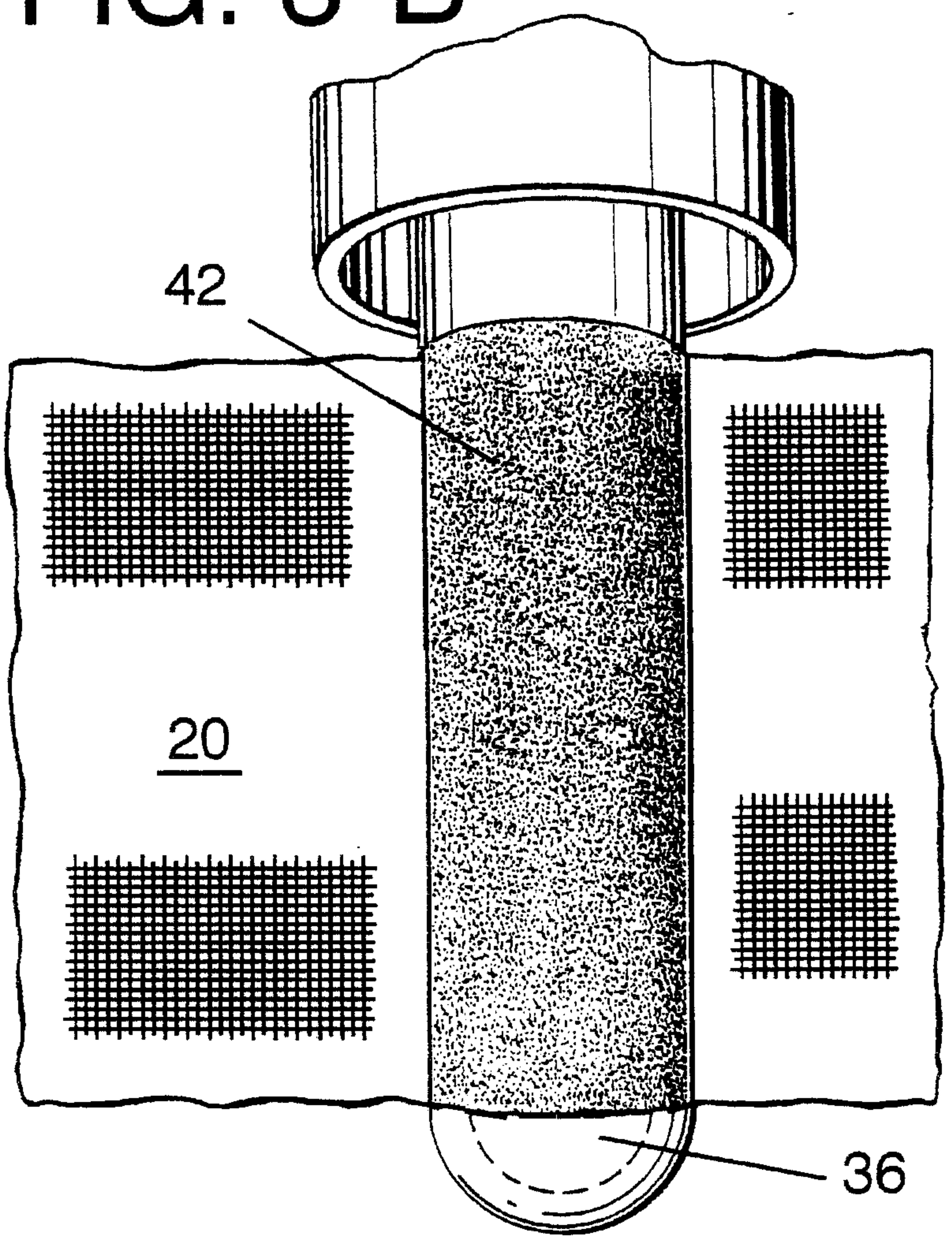


FIG. 3-C

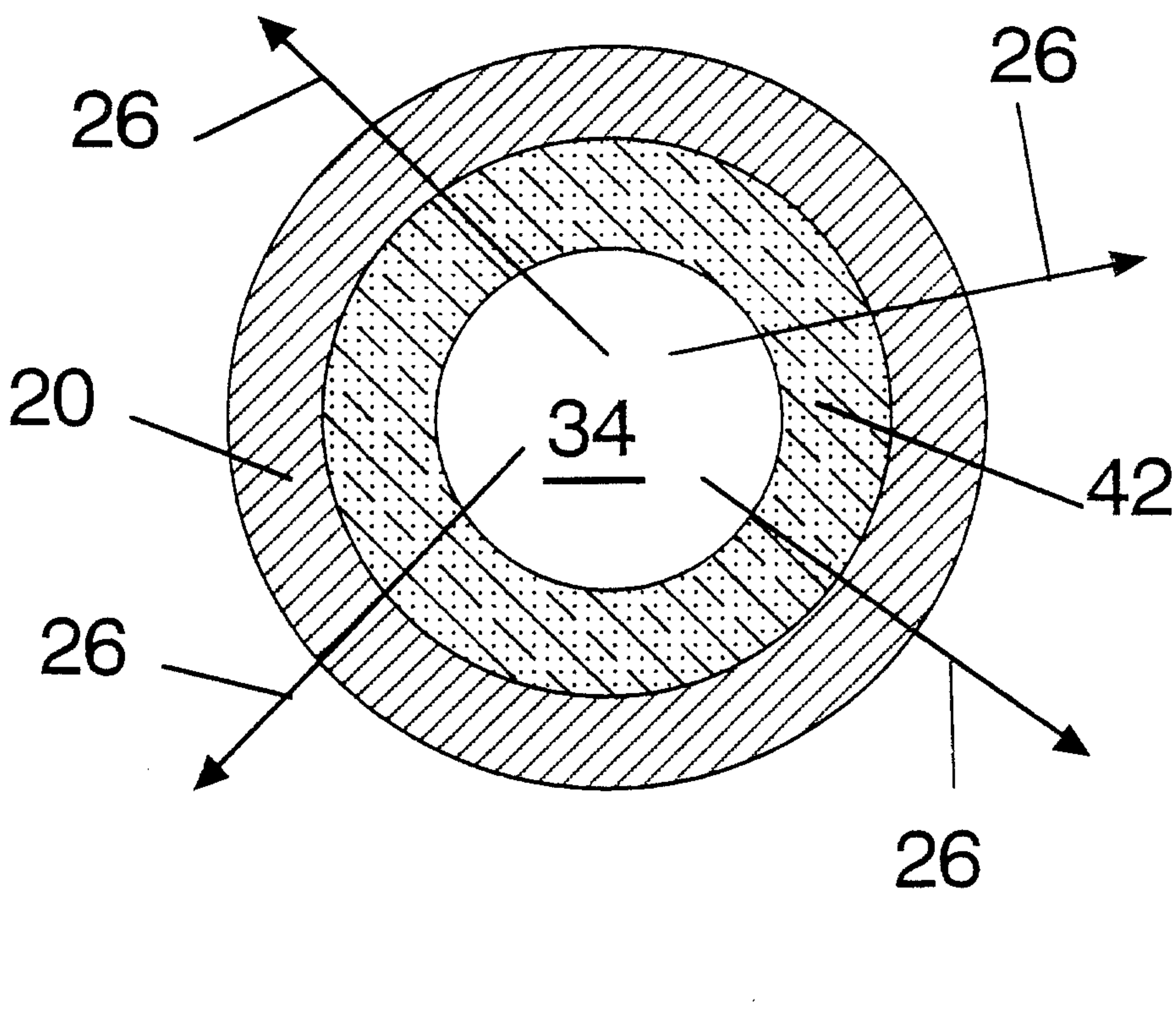
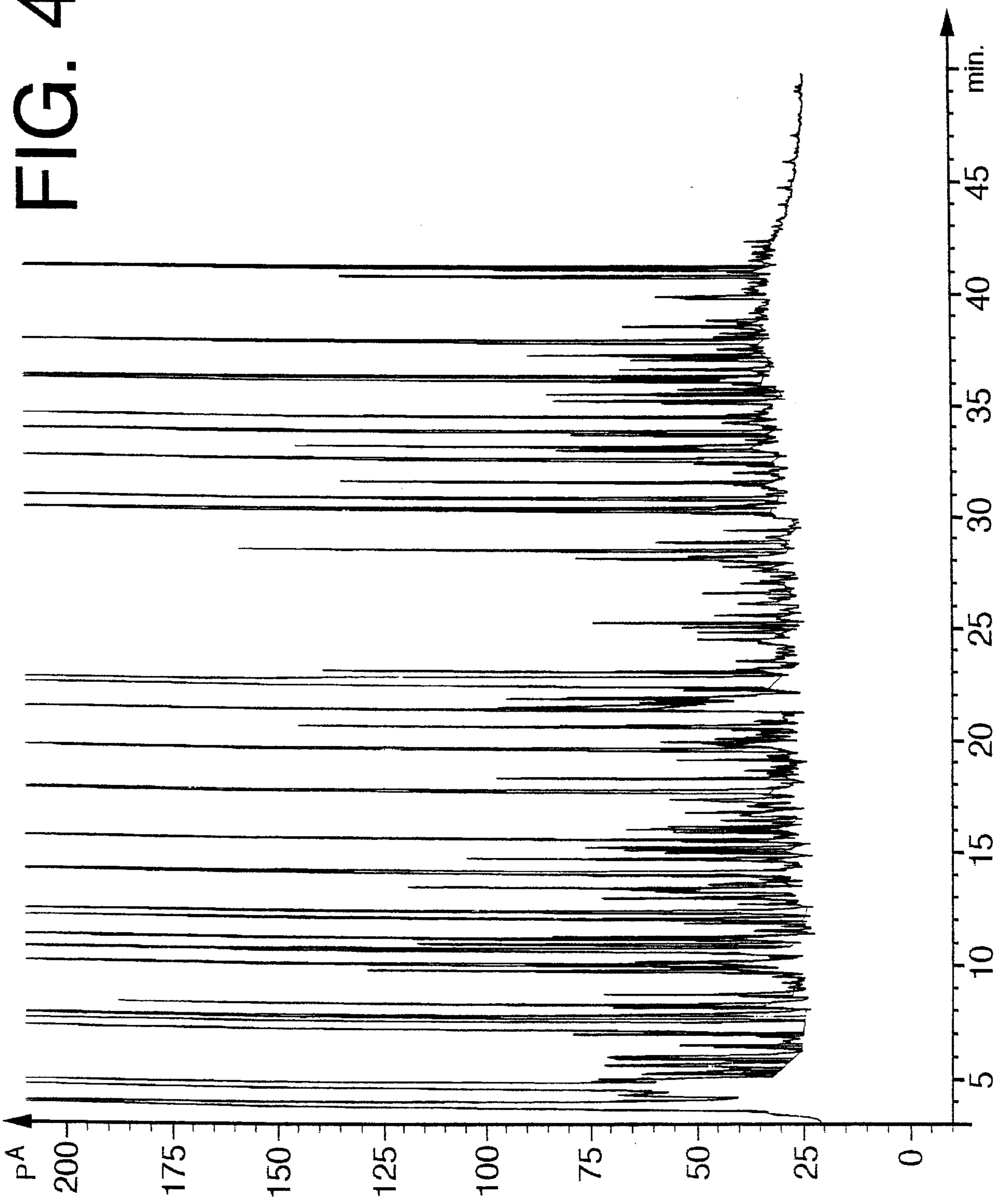
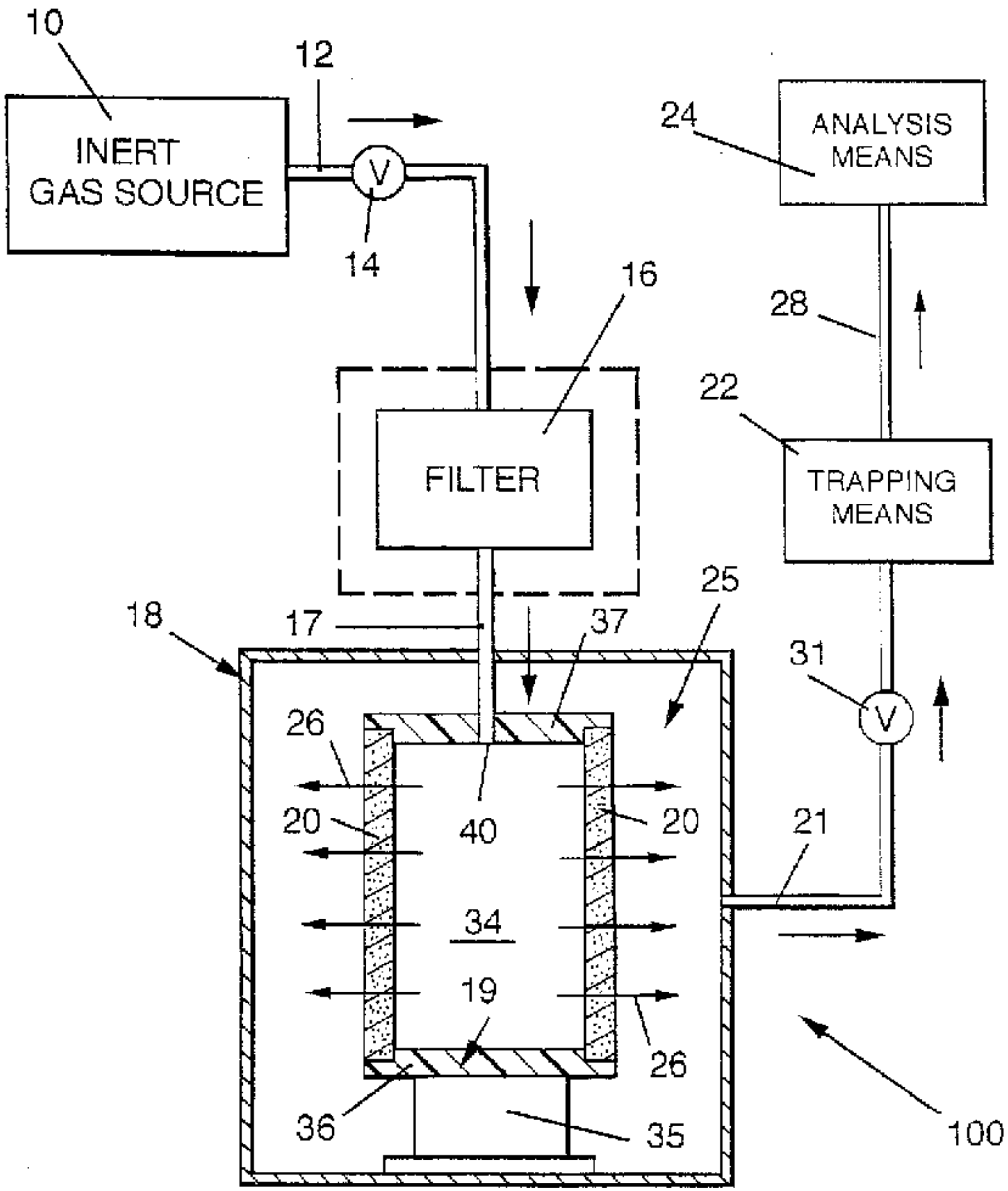


FIG. 4

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