

[54] CHROMATOGRAPH INLET WITH
ROTATING SEPTUM

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161/206; 215/38 R

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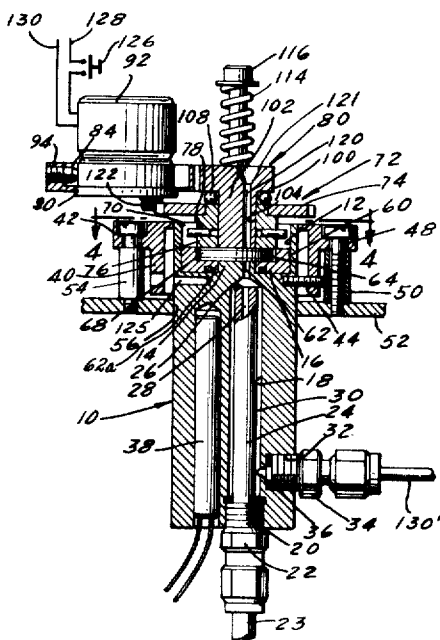
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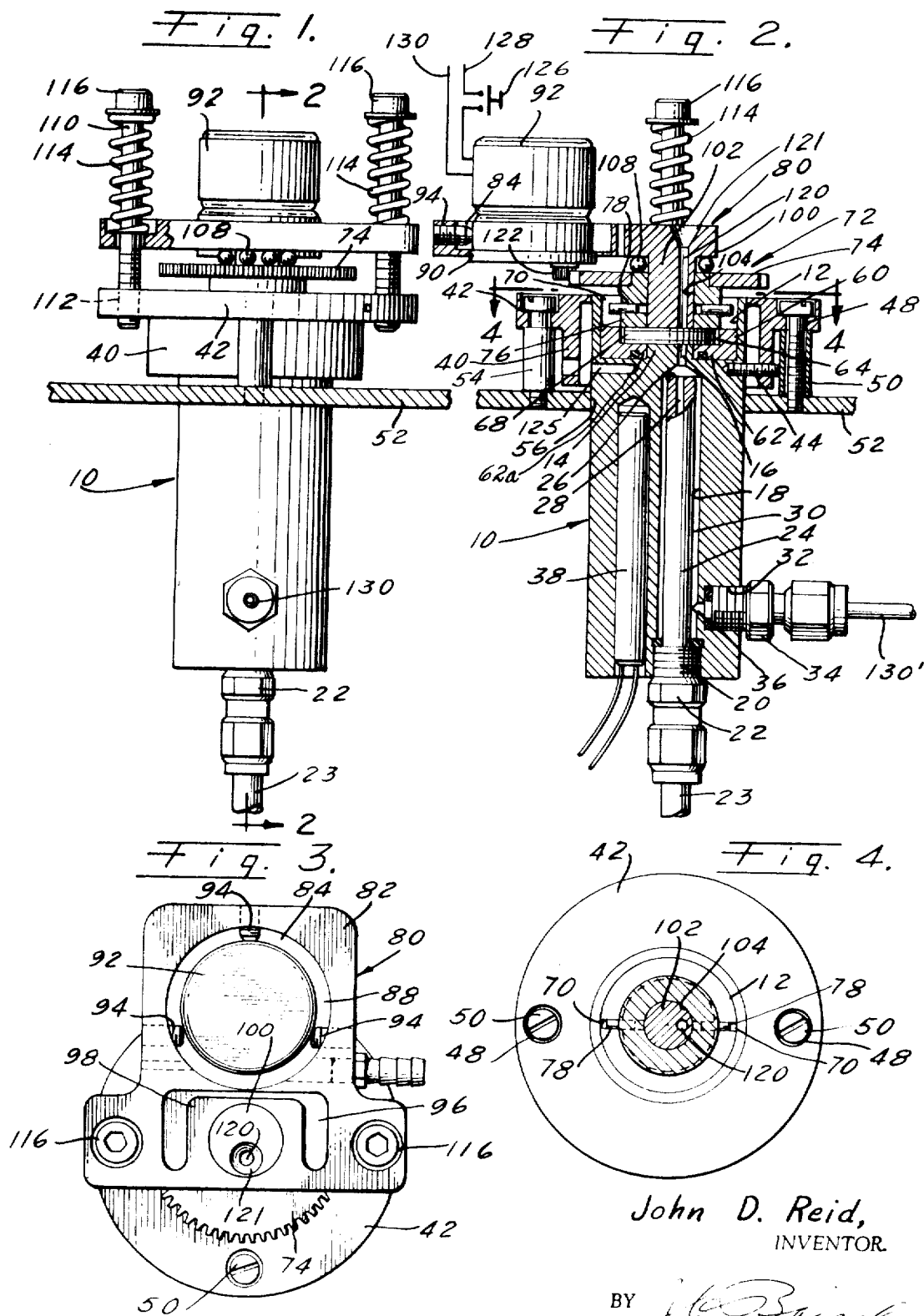
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[57] ABSTRACT

A septum inlet apparatus for chromatographs having a ported body and a septum disc carried by a rotatably mounted septum carrier driven by an electric motor through speed reduction gearing. The septum is rotatably moved a predetermined amount by operating the motor for a predetermined period of time to move a used portion of the septum out of operative position and bring into operative position an unused portion.

3 Claims, 4 Drawing Figures





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CHROMATOGRAPH INLET WITH ROTATING SEPTUM

CROSS-REFERENCE TO RELATED APPLICATION

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BACKGROUND OF THE INVENTION

1. Field of the Invention

This invention relates generally to inlets for chromatographs and relates more particularly to chromatograph inlets having a septum that can be quickly and easily moved to bring various parts thereof into operative position.

2. Description of the Prior Art

Conventional inlets for chromatographs have various problems, disadvantages and operating difficulties. As is well known, septums retain their sealing reliability for from six to ten injections and then must be replaced. When such septums are replaced, the chromatograph instrument must be shut down and there is, of course, an interruption of carrier gas flow and a loss of carrier gas while the used septum is removed from the inlet and a new septum installed. Then, there is a waiting period while the instrument regains its operating temperature after a shutdown. There is, also, always the danger that the operator will have his fingers burned while changing the septum.

The Vannus Automatic Septum Changing Inlet provides septum changer wherein the septum disc is carried by a rotatably mounted ratchet wheel having a plurality of annularly spaced outer ports which are successively moved into alignment with a port of the body, there being means for successively rotating the ratchet wheel to move said outer ports respectively into alignment with the port in the body.

SUMMARY OF THE INVENTION

The present invention has a body, for connection with a chromatograph column, and has a heater. A collar provides thermal isolation from the body to the chromatograph panel. A septum carrier is provided with a Teflon O-ring serving as a secondary seal, the septum being the primary seal. An electric motor effects rotation of the septum carrier through speed reduction means, there being a needle guide offset from the axis of the septum carrier. There is spring loaded means which keeps the septum under compression and compensates for the thermal expansion of the septum under the operating temperature conditions of the chromatograph and inlet.

OBJECTS AND ADVANTAGES OF THE INVENTION

It is an object of the invention to provide an inlet for chromatographs having means for moving the septum to bring unpunctured or unused portions, respectively, into operative position as portions lose their sealing reliability by being punctured a number of times.

Another object of the invention is to provide a chromatograph inlet having means for easily and quickly changing effective parts of a septum.

Still another object of the invention is to provide an inlet of this character that is simple in construction and reliable in operation.

A further object of the invention is to provide an inlet of this character that has relatively few parts and is relatively inexpensive to manufacture.

A still further object of the invention is to provide an inlet of this character wherein the movement of the septum from station to station is effected without shutting down the instrument.

Another object of the invention is to provide apparatus of this character which eliminates interruption of carrier gas flow and loss of carrier gas.

Still another object of the invention is to provide apparatus of this character which eliminates waiting for the instrument to regain its operating temperature after a shut down.

A further object of the invention is to provide a chromatograph inlet apparatus that eliminates the danger of the operator burning his fingers.

The characteristics and advantages of the invention are further sufficiently referred to in connection with the following detailed description of the accompanying drawings, which represent one embodiment. After considering this example, skilled persons will understand that many variations may be made without departing from the principles disclosed and I contemplate the employment of any structures, arrangements or modes of operation that are properly within the scope of the appended claims.

BRIEF DESCRIPTION OF THE DRAWING

Referring to the drawings, which are for illustrative purposes only:

FIG. 1 is a side elevational view of a chromatograph inlet embodying my invention;

FIG. 2 is a sectional view thereof taken on line 2—2 of FIG. 1;

FIG. 3 is a top plan view; and

FIG. 4 is a sectional view taken on line 4—4 of FIG. 2.

DESCRIPTION OF THE PREFERRED EMBODIMENT

Referring more particularly to the drawings, the inlet comprises a body, indicated generally at 10, of stainless steel, aluminum, or other suitable material. Body 10 is shown as being cylindrical but it may be of any other suitable shape. The body has a cylindrical recess 12 in the upper end, with a short cylindrical boss 14 upstanding axially from the bottom of said recess 12. Boss 14 has a longitudinally extending needle bore 16 of such diameter as to operably receive the needle of a fluid measuring syringe, said needle bore 16 being offset from the axis of the boss 14.

Extending longitudinally from the lower end of the body 10, as shown in FIGS. 1 and 2, is an adaptor bore 18 which is in axial alignment with the needle bore 16 but of greater diameter. An outer end portion 20 of the adaptor bore 18 is tapped for threadable reception of a column fitting 22 at the end of a chromatograph column 23, said fitting carrying a column adaptor 24 disposed in the adaptor bore 18, said column adaptor 24 being of smaller diameter than the adaptor bore 18, the inner end 26 of said column adaptor 24 clearing the inner end of the adaptor bore so that carrier gas can flow into the fluid flow passage 28 of the column adaptor from the annular space 30 between the wall of the adaptor bore 18 and the external wall of the column adaptor. The fluid flow passage may flare at its inner end to facilitate insertion of the needle.

The body 10 has a laterally extending tapped bore 32 for reception of the externally threaded end portion of a carrier gas fitting 34, a passage 36 extending from the inner end of the tapped bore 32 to provide a connection with the annular flow passage 30 between the wall of the adaptor bore and the adaptor 24.

Body 10 also has a heater bore for an electric heater 38, of well known character, for heating the inlet body to the proper operating temperature.

There is a collar 40 having a radially extending annular flange 42 at the upper end, said collar having a greater inside diameter than the external diameter of the body 10, the collar being of stainless steel, aluminum, or other suitable material. Collar 40 is positioned at the upper end of the body 10 and is secured in spaced relation thereby by setscrews 44 disposed in tapped openings provided therefor in the collar and aligned tapped bores provided therefor in the body adjacent the upper end thereof. There may be any suitable number of such setscrews but three equally spaced about the collar and body are used. Thermal isolation of the collar from the body is provided by having the collar spaced from the body.

The flange 42 has an opening 46 therethrough of smaller diameter than the internal diameter of the collar 40, the upper end portion of the body being received in said opening 46. Flange 42 also has openings 48 therethrough for reception of screws 50 for securing the inlet to the chromatograph panel 52, there being tubular spacers 54 on said screws 50 for properly spacing the outer (upper) end of the inlet from said panel. It is to be noted that the panel 52 has an opening 56 therein for reception of the body 10.

Within the recess 12, there is rotatably disposed a cup shaped septum retainer having a peripheral wall 60 and a bottom wall 62, said walls defining a recess 64. The bottom wall has an axial bore 62a therethrough in which is received the boss 14, the height of the boss being the same as the thickness of the bottom wall 62 of the septum retainer so that the upper surface of said boss is in substantially the same plane as the upper surface of said bottom wall 62. The underside of the bottom wall has an axial, annular groove about the axial bore in the bottom wall 62 in which an O-ring is disposed to provide what is termed a secondary seal between the septum retainer and the bottom of recess 12 of the body. A septum 68 is press fitted, or otherwise secured in the recess 64 of the septum retainer, and the under side of said septum sealingly engages the top surface of boss 14 and comprises the primary seal, said top surface of said boss providing back-up means for the septum. The septum is of any suitable, well known character, various septums being available on the market. However, the septum shown is laminated, there being a relatively soft central layer of silicone rubber with layers of harder silicone rubber secured to each side of said central layer. The septum is self-sealing.

A gear subassembly, indicated generally at 72, comprises a gear 74 having a hub 76 from which project a pair of oppositely disposed radial pins 78. Hub 76 is received within the recess 64 of the septum retainer and rests on the septum, the pins 78 being received within the notches 70 and are disposed intermediate the top and bottom thereof, and can take care of expansion and contraction of the septum. Also, this connection between the septum retainer and the gear subassembly 72 provides means whereby the septum retainer is rotated with rotation of the gear subassembly, to thereby move and thereby replace unpunctured portions of the septum for punctured portions, as described more fully hereinafter.

There is a motor mount and needle guide, indicated generally at 80, which comprise a generally T-shaped plate 82, the stem of the T having a bore 84 therein, there being a flange 88 at the lower end of the bore 84 defining an opening 90 in which an electric motor 92 is disposed. The motor is secured in operating position by screws 94, three screws spaced annularly apart relative to the bore 84 being used.

Plate 82 has a U-shaped opening 96 in the cross part of the T leaving a tongue 98 which has an opening therein for reception of the enlarged head 100 of a needle guide 102. The opening 96 serves to minimize heat transference to the tongue 98. The needle guide is disposed in an axial bore 104 through the hub 76 of the gear subassembly 72, and friction is minimized between the upper end of the hub 76 and its gear by a thrust ball bearing assembly 108 disposed on the needle guide 102.

Motor mount and needle guide 80 are secured to the flange 42 by means of oppositely disposed screws 110 disposed in openings 112, provided therefor in the plate 82 adjacent the free ends of the cross part of the T, the lower ends of said screws being threadably received in tapped bores provided therefor in said flange 42. These screws are of substantial length and have coil springs 114 disposed thereon above the plate 82. The lower ends of said springs are received in spring retaining counter bores at the upper ends of said openings 112. The upper ends of said springs abut against the heads 116 of said screws 110 so that said springs react between said screw heads 116 and the bottoms of the counter bores of openings 112, and yieldingly urge the plate downwardly so that the lower end of the needle guide is pressed sealingly

against the upper surface of the septum. The springs 114 allow movements of the plate and, hence, the needle guide, with expansion and contraction of the septum.

The needle guide is provided with a needle guide passage 120, flared at its outer end 121. Needle guide passage is parallel to but radially offset from the axis of the needle guide and axis of the septum and septum retainer, when the above described parts of the inlet are operably assembled. Also, when said parts are thus assembled there is a gear 122 on the rotatable shaft of the electric motor 92 which meshes with the gear 74, gear 122 being of much smaller diameter than the diameter of the gear 74, so that when the motor is energized there is a speed reduction that results in a slow rotation of the septum retainer and septum.

Thermocouple holes 125 are provided in the body 10, and there are, also, thermocouple holes, not shown, in the collar, motor mount and needle guide.

Since the needle passage 120 in the needle guide and the needle bore 16 are offset from the axis of the septum retainer, septum rotation thereof by the motor 92 will cause an annular part of the septum spaced from the axis thereof to be brought under or into alignment with the needle passage 120 and needle bore 16.

OPERATION

Carrier gas is supplied through a conduit 130' and fixture 34 therefor. The carrier gas flows through the annular passage 30 between the wall of the adaptor bore 18 and the exterior of the column adaptor 24, past the upper free end of said column adaptor, into the space 132 at the end of said column adaptor and into the fluid flow passage 28 of the column adaptor, and, hence, to the chromatograph column 23.

With the inlet at operating temperature, the operator inserts the needle into the needle passage 120, through the septum, needle bore 16 and into the fluid flow passage 28 of the adaptor. The operator then injects the sample. The carrier gas continues to flow into and through the fluid flow passage 28 of the adaptor, since said fluid flow passage is larger in diameter than the outside diameter of the needle. As the sample is injected into the fluid flow passage 28, it is entrained by the carrier gas and carried along into the chromatograph column.

After a number of injections, the sealing reliability of the septum is lost.

The part of the septum that has been repeatedly punctured and lost its sealing reliability is then rotated out of alignment with the needle passage 120 and needle bore 16 by closing switch 126 of the electric circuit for the motor, wires 128 and 130 of said electric circuit being connected to a suitable source, not shown, of electric power. The motor is operated for a predetermined period of time to rotate the septum into alignment with said needle passage 120 and needle bore 16. An example of the amount of rotation of the septum to effect the above described change is 0.06545 inch, the motor being operated but a few seconds to effect this amount of rotation.

I claim:

1. A septum inlet apparatus for chromatographs, comprising:
 - A. a body having an adaptor bore therein adapted to receive carrier gas;
 - B. a column adaptor operably disposed in said adaptor bore, said column adaptor having a fluid flow passage therein adapted to be connected to the column of a chromatograph;
 - C. a rotatable septum carrier operably carried by said body;
 - D. a septum carried by said septum carrier and rotatable therewith;
 - E. a needle guide having a needle guide passage therethrough parallel to the axis of said septum carrier but radially offset from said axis, the fluid flow passage in the column adaptor being aligned with the needle guide passage;
 - F. resilient means for applying yielding pressure to the outer side of said septum;

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G. means for rotating said septum carrier;

H. the body having a cylindrical recess in one end in which the septum carrier is rotatable, said septum carrier being cup-shaped and having a peripheral wall and an end wall having an axial bore therethrough, said end wall having an axial groove in the bottom thereof concentric with said bore; and a secondary seal in said groove providing a seal between the septum carrier and the bottom of the recess in the body.

2. The invention defined by claim 1, wherein the peripheral wall of the septum carrier has a pair of oppositely positioned radial notches; and the means for rotating the septum carrier includes a gear having a hub, a pair of oppositely extending

radial pins carried by said hub and slidably disposed in said notches to permit axial movement of said hub relative to said septum carrier.

3. The invention defined by claim 2, wherein there is a cylindrical lug upstanding from the bottom of the recess in said body and extending in the axial bore in the bottom wall of the septum carrier, the free end of said lug being flat and normal to the axis of said lug and providing a backing for the septum, said lug having a needle bore therethrough offset from the axis of said lug and in alignment with the needle guide passage of the needle guide, said needle bore also being in alignment with the fluid flow passage of said column adaptor.

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