

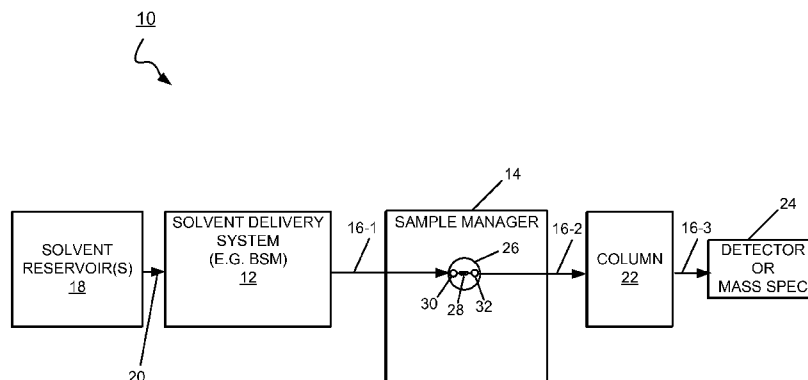


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(54) Title: CORROSION PROTECTION IN TUBING USED IN CHROMATOGRAPHY



(57) Abstract: An apparatus for use in a liquid chromatography system includes a chromatography port and a tubing assembly having a chromatography tube coupled at one end to the chromatography port. The end of the tube has an end face covered with a corrosion-resistant material, for example, gold. The corrosion-resistant nature of the material protects the end of the tube from corrosion or erosion, which improves the quality and reliability of a seal between the end face of the tube and a sealing surface of the port. Alternatively, or in addition to covering the end face of the tube with the corrosion-resistant material, a gasket covered with or made of the corrosion-resistant material can be disposed between the end face of the tube and the port. This gasket extends the reach of the tube to facilitate bottoming out the tube within the port.



CORROSION PROTECTION IN TUBING USED IN CHROMATOGRAPHY

RELATED APPLICATION

This application claims the benefit of and priority to co-pending U.S. provisional application no. 61/606,525, filed March 5, 2012, titled "Corrosion Protection in Tubing Used
5 in Chromatography," the entirety of which application is incorporated by reference herein.

FIELD OF THE INVENTION

The invention relates generally to liquid chromatography systems. More specifically, the invention relates to tubing treatments for protection from corrosion in chromatography systems.

10 BACKGROUND

Chromatography is a set of techniques for separating a mixture into its constituents. Well-established separation technologies include HPLC (High Performance Liquid Chromatography), UPLC (Ultra Performance Liquid Chromatography), and SFC (Supercritical Fluid Chromatography). HPLC systems use high pressure, ranging
15 traditionally between 1,000 psi (pounds per square inch) to approximately 6,000 psi, to generate the flow required for liquid chromatography in packed columns. In contrast to HPLC, UPLC systems use columns with smaller particulate matter and higher pressures approaching 20,000 psi to deliver the mobile phase. SFC systems use highly compressible mobile phases, which typically employ carbon dioxide (CO₂) as a principle component.

In general, in a liquid chromatography (LC) application, a solvent delivery system
20 takes in and delivers a mixture of liquid solvents to an autosampler (also called an injection system or sample manager), where an injected sample awaits the arrival of this mobile phase. The mobile phase carries the sample through a separating column. In the column, the mixture of the sample and mobile phase divides into bands depending upon the interaction of
25 the mixture with the stationary phase in the column. A detector identifies and quantifies these bands as they exit the column.

Tubing between equipment used in liquid chromatography, such as valves, pumps, and the column is typically made of stainless steel. Such material is readily available and capable of handling the elevated pressures and temperature range required for
30 chromatography. Despite its hardness, however, this material may corrode or erode at connection points to the detriment of the chromatographic results. The effects of the

corrosion or erosion may include the loss of pressure or of flow, which affect retention times. Other negative effects may include carryover, and tailing and fronting of peaks.

SUMMARY

5 In one aspect, the invention features an apparatus comprising a chromatography port and a tubing assembly having a chromatography tube with a bore extending therethrough. The chromatography tube is coupled at one end to the chromatography port. The one end of the chromatography tube has an end face covered with a corrosion-resistant material.

10 In another aspect, the invention features an apparatus comprising a chromatography port and chromatography tubing coupled at one end to the chromatography port. The end of the chromatography tubing has an end face. The apparatus further comprises a gasket disposed between the end of the chromatography tubing and the chromatography port. One side of the gasket abuts the end face of the chromatography tubing and the opposite side of the gasket abuts a sealing surface of the chromatography port. The gasket is covered with or made of corrosion-resistant material.

15 In still yet another aspect, the invention features a tubing assembly comprising a chromatography tube with a bore extending from one end of the tube to an opposite end of the tube. One of the ends of the tube has an end face covered with a corrosion-resistant material.

20 In another aspect still, the invention features a liquid chromatography system comprising a component with a chromatography port and a tubing assembly with a chromatography tube having a bore extending therethrough. The chromatography tube is coupled at one end to the chromatography port with the bore of the chromatography tube axially aligned with the fluidic channel at the chromatography port. The end of the chromatography tube has an end face covered with a corrosion-resistant material.

25 BRIEF DESCRIPTION OF THE DRAWINGS

The above and further advantages of this invention may be better understood by referring to the following description in conjunction with the accompanying drawings, in which like numerals indicate like structural elements and features in various figures. The drawings are not necessarily to scale, emphasis instead being placed upon illustrating the principles of the invention.

30 FIG. 1 is a functional block diagram of an embodiment of a liquid chromatography system.

FIG. 2 is a functional diagram of an embodiment of a solvent manager of a liquid chromatography system.

FIG. 3 is a side view of an embodiment of a fitting for joining fluidic pathways, the fitting comprising a tubing assembly, the tubing assembly comprising a tube, a portion of which is coated with a corrosion-resistant material.

FIG. 4 is a side view of the tubing assembly and a compression member coupled to the tubing assembly, the tubing assembly including a tube having a gasket at one end, the gasket being coated with or made of a corrosion-resistant material.

FIG. 5 is a cross-section view of the tubing assembly.

FIG. 6 is a detail view of the end of the tubing assembly with the gasket coated or made of a corrosion-resistant material.

FIG. 7 is a cross-section view of the fitting of FIG. 2 installed within a port.

FIG. 8 is cross-section view of the fitting installed within a port, the fitting having a tubing assembly comprising a tube with a gasket at one end, the gasket being coated with or made of a corrosion-resistant material.

DETAILED DESCRIPTION

Applicant recognized that erosion or corrosion of tubing used in a liquid chromatography system can lead to sample carryover, peak fronting and tailing, or pressure loss, which are phenomena known to affect negatively the chromatography results. Such erosion or corrosion is thought to occur primarily at the joints where the tubing connects to a component of the liquid chromatography system, such as an injector valve or a column, although corrosion of the tubing can conceivably occur anywhere along its length.

To minimize or eliminate the erosion or corrosion at connection points, portions of the ports and/or tubes are covered (i.e., coated, plated, or treated) with a malleable, corrosion-resistant relatively chemically inert material, one that can withstand the temperature and pressure requirements of liquid chromatography systems. Herein, the terms coated, plated, and treated (and variations thereof) are interchangeably used, without any implicit limiting of the principles described herein to the particular process used to produce the layer of corrosion-resistant material on the tube. Preferably, the plating covers the end face at one or both ends of the tube. In addition, the plating can extend to the inside surface of the tube, preferably for the entire tube's length, or as far into the tube as the plating process and the inner diameter (ID) of the tube will allow. In addition, the facing surface of the ports that contact the tubes can be plated with the corrosion-resistant material. For those connection

joints where the thickness of the plating on the end face of the tubing may not be sufficient for the tube to “bottom out” in a port, a gasket coated with or made of the corrosion-resistant material can be placed at the tube end, in effect, extending the tube to make contact with a sealing surface of the port. This gasket can be bonded to the end face of the tube or placed
5 separately within the port before insertion of the tube.

In one embodiment, the corrosion-resistant material is gold (either hard gold or soft gold). Gold is useful in this application because of its high ductility, malleability, resistance to corrosion, and relative chemical inertness. An example of another corrosion-resistant, relatively chemically inert material for use in covering the tubing is tantalum. Another
10 example is platinum.

FIG. 1 shows an embodiment of a liquid chromatography system 10 for separating a sample into its constituents. The liquid chromatography system 10 can be, for example, an HPLC, UPLC, or a CO₂-based system. In brief overview, the liquid chromatography system 10 includes a solvent delivery system 12 in fluidic communication with a sample manager 14
15 (also called an injector or autosampler) through tubing 16-1. The solvent delivery system 12 includes pumps (not shown) in fluidic communication with solvent (or fluid) reservoirs 18 from which the pumps draw solvents through tubing 20. A chromatography column 22 is in fluidic communication with the sample manager 14 through tubing 16-2. Tubing 16-3 couples the output port of the column 22 to a detector 24, for example, a mass spectrometer.
20 Through the tubing, the detector 24 receives the separated components from the column 22 and produces an output from which the identity and quantity of the analytes may be determined. As described herein, the tubing 16-1, 16-2, 16-3 (generally, 16) are partially or fully covered with a corrosion-resistant material, used in conjunction with a plated end-face gasket, or both. Each tubing 16 refers generally to a section of tubing rather than to a single
25 tube; each tubing section may comprise one tube or multiple tubes (e.g., by valves, tees, etc).

The sample manager 14 includes an injector valve 26 with a sample loop 28. In one embodiment, the injector valve is a rotary shear valve. The solvent manager 14 operates in one of two states: a load state and an injection state. In the load state, the position of the injector valve 26 is such that the solvent manager 14 loads the sample into the sample loop
30 28; in the injection state, the position of the injector valve 26 changes so that solvent manager 14 introduces the sample in the sample loop 28 into the continuously flowing mobile phase arriving from the solvent delivery system 12. With the injector valve 26 in the injection state, the mobile phase carries the sample into the column 22, the mobile phase arriving at the

injector valve 26 through an input port 30 and leaving with the sample through an output port 32.

The various connection joints benefitting from the treated tubing, ports, and/or gasket include, but are not limited to, where the tubing 16-1 connects to the input port 30 of the injector valve 26, where the tubing 16-2 connects to the output port 32 of the injector valve 26 and to the column 22, and where the tubing 16-3 connects to the output end of the column 22 and to the detector 24.

As shown in FIG. 2, in some embodiments, for example, those in which the liquid chromatography system 10 employs a CO₂-based mobile phase, the sample manager 14 can further include an auxiliary valve 40 interposed between the solvent delivery system 12 and the injector valve 26 and between the injector valve 26 and the column 22. Although shown to be part of the sample manager 14, the auxiliary valve 40 can be implemented in another piece of equipment remote to the injector valve 26. Like the injector valve 26, the auxiliary valve 40 can be implemented using a rotary shear valve. In general, the auxiliary valve 40 provides a fluidic pathway through which the injector valve 26 may vent. In this embodiment, the tubing 16-1 couples the solvent delivery system 12 to an input port 42 of the auxiliary valve 40 and the tubing 16-2 couples an output port 44 of the auxiliary valve 40 to the column 22. Tubing 16-4 and 16-5 couples the auxiliary valve 40 to the injector valve 26; tubing 16-4 connects port 46 of the auxiliary valve 40 to the input port 30 of the injector valve 26, and tubing connects port 48 of the auxiliary valve 40 to the output port 32 of the injector valve 26.

When the valves 26, 40 are configured for sample injection, the arrows on the tubing 16-1 and 16-4 show the direction of flow of the mobile phase towards the injector valve 26; those arrows on the tubing 16-5 and 16-2 correspond to the flow of the mobile phase carrying the sample from the injector valve 26 towards the column 22.

Like the tubing 16 described in connection with FIG. 1, the tubing 16-4 and 16-5 are also partially or fully treated with a corrosion-resistant material, used in conjunction with a plated end-face gasket, a plated port, or any combination thereof. The additional connection joints benefitting from the treated tubing and/or end-face gasket include where the tubing 16-4 connects to the input port 30 of the injector valve 26 and to the port 46 of the auxiliary valve 40, and where the tubing 16-5 connects to the output port 32 of the injector valve 26 and to the port 48 of the auxiliary valve 40.

FIG. 3 shows an embodiment of a fitting 50 for coupling two fluidic paths, for example, to couple the tubing 16-1, 16-2 of FIG. 1 or the tubing 16-4, 16-5 of FIG. 2 to a port

of a rotary shear seal valve. The fitting 50 includes a tube assembly 60 having a tube 62 surrounded by a sleeve 64. Either or both the tube 62 and the sleeve 64 can be made of stainless steel. Materials other than stainless steel can be used for the tube 62 and the sleeve 64. A weld may join the tube 62 to the sleeve 64. As an example, the inner diameter (ID) and outer diameter (OD) of the tube 62 can be 0.007 in. and 0.025 in., respectively, and the outer diameter of the sleeve 64 can be 0.062 in. The end face 66 of the tube 62 is substantially normal to the longitudinal axis 72 of the tube 62 and has a low surface roughness. A portion of the tube 62 protrudes from the end of the sleeve 64. The length of the protrusion allows the end face 66 of the tube 62 to contact a sealing surface of a port (e.g., of the injector valve 26), which may also be covered with a corrosion-resistant material.

In general, any portion of the tube 62, its end faces, inner surface, and outer surface, and the sleeve 64 can be plated with the corrosion-resistant material. Use of the term “plating” or “plated” in connection with the tube is not intended to limit the principles described herein to the particular process used to produce the layer of corrosion-resistant material on the tube. It is to be understood that any one of various conventional processes, including, for example, electroplating and vapor deposition, can be used to cover, coat, treat, or plate the tube, partially or fully, without departing from the principles described herein.

Preferably, at least the end face 66 is plated. In addition, the plating of the corrosion-resistant material can extend to the inside of the tube 62 to a depth of approximately equal to or greater than the inner diameter (ID) of the tube. For example, for a tube with a 0.007-inch ID, the plating of the corrosion-resistant material extends at least 0.007 inches into the tube 62. Preferably, a plating thickness in the range of 2-4 μ m is generally thick enough to achieve corrosion resistance and to fill any surface imperfections on the end face 66 of the tube 62 and on the sealing surface of the port. Plating thicknesses that far exceed this range can produce a “gasket” between the end face 66 of the tube 62 and the sealing surface of the mating port. This gasket can serve to span any gap between the end face 66 of the tube 62 and the sealing surface of the port.

The fitting 50 further includes a two-part compression member 68A, 68B (generally 68) and a compression screw 70, each of which encircle the tube assembly 60. The two-part compression member 68 has a front-coned portion 68A and a back ring 68B. The compression member 68 can be, for example, a stainless steel ferrule set (e.g., part no. SS-100-SET available from Swagelok Company of Solon, OH). The compression screw 70 has threads 74 for engaging threads of the receiving port.

FIG. 4 shows the fitting 50 of FIG. 3 without the back ring 68B of the two-part compression member 68A, 68B and without the compression screw 70, and FIG. 5 shows a cross-section of the fitting 50 along the line A-A in FIG. 4. In addition, the tube assembly 60 here includes a washer 80 plated with or made of a corrosion-resistant material and attached to the end face 66 (FIG. 3) of the tube 62. Plating limitations might not allow for a thick enough plating layer to produce a gasket on the end face of the tube 62. Accordingly, this washer 80 provides that gasket. The washer (hereafter, gasket 80) can be used instead of or in addition to plating the end face 66 (and/or the inner surface) of the tube 62.

Known techniques, for example, laser weld, e-beam weld, and thermal compression bonding, can join the gasket 80 to the end face 66 of the tube 62. A purpose for joining the gasket 80 to the end face 66 is to prevent the gasket 80 from becoming stuck in the bottom of the receiving port (e.g., upon removal of the fitting 50).

FIG. 6 shows a detail view corresponding to the circle B in FIG. 5. The detail view shows the placement of gasket 80 on the end face 66 of the tube 62. The bore 82 of the tube 62 aligns with the central opening 84 in the gasket 80. The inner diameter of the gasket 80 is approximately equal to or slightly greater than the inner diameter of the tube 62, and the outer diameter of the gasket 80 is optionally greater than the outer diameter of the tube 62. In one embodiment, the thickness of the gasket 80 is 0.010 in.

FIG. 7 shows the fitting 50 mated with a port 90, the tube assembly 60 and the two-part compression member 68A, 68B being disposed within a cavity of the receiving port 90. When securing the fitting 50 to the port, the user tightens the compression screw 70, which causes the compression member 68A, 68B to grip the sleeve 64 and urges the end face 66 of the tube 62 towards the sealing surface 92 of the port 90. In this example, the end face 66 physically contacts the sealing surface 92 (i.e., “bottoms out”), with the bore 82 of the tube 62 aligning with the fluidic channel 94 at the bottom of the port 90. With the compression screw 70 adequately tightened, the end face 66 of the tube 62 produces a fluid-tight seal against the sealing surface 92 of the port 90. Because the end face 66 and, optionally, a portion of the inner surface of the tube 62 are plated with a malleable, corrosion-resistant relatively chemically inert material, the quality and reliability of the fluid-tight seal is improved by filling the surface imperfections on the end face 66 and on the sealing surface 92 and by resisting corrosion and erosion at the tube’s end.

FIG. 8 shows another example of the fitting 50 mated with the port 90. In this example, the compression screw 70 and the two-part compression member 68A, 68B seal the joint between the two fluidic pathways 82, 94 before the end face 66 of the tube can

physically contact the sealing surface 92 of the port 90. The tube 62 does not bottom out. In this instance, the chromatography results could be subjected to carryover because sample or analytes can fill the sealed cavity 98. As shown, use of the gasket 80, plated with or constructed of malleable, corrosion-resistant, relatively chemically inert material, spans the distance between the end face 66 and the sealing surface 92 of the port 90, and produces a fluid-tight, imperfection-filling, corrosion-resistant seal against the sealing surface 92. The gasket 80 couples the bore 82 of the tube 62 directly to the fluidic channel 94 of the port 90. Consequently, sample or analytes cannot spill into the cavity 98, where they could subsequently contribute to carryover.

Reference in the specification to “one embodiment” or “an embodiment” means that a particular, feature, structure or characteristic described in connection with the embodiment is included in at least one embodiment of the teaching. References to a particular embodiment within the specification do not all necessarily refer to the same embodiment.

While the invention has been shown and described with reference to specific preferred embodiments, it should be understood by those skilled in the art that various changes in form and detail may be made therein without departing from the spirit and scope of the invention as defined by the following claims.

What is claimed is:

CLAIMS

- 1 1. An apparatus comprising:
2 a chromatography port; and
3 a tubing assembly having a chromatography tube with a bore extending
4 therethrough, the chromatography tube being coupled at one end to the
5 chromatography port, the one end of the chromatography tube having an end face
6 covered with a corrosion-resistant material.
- 1 2. The apparatus of claim 1, further comprising a gasket covered with the corrosion-
2 resistant material, attached to the end face of the chromatography tube, and abutting a
3 sealing surface of the chromatography port.
- 1 3. The apparatus of claim 1, wherein the corrosion-resistant material comprises gold.
- 1 4. The apparatus of claim 1, wherein the chromatography tube further comprises an
2 outer surface and an inner surface, the inner surface near the end face being covered
3 with the corrosion-resistant material.
- 1 5. The apparatus of claim 4, wherein at least a portion of the outer surface adjacent the
2 end face of the chromatography tube is covered with the corrosion-resistant material.
- 1 6. The apparatus of claim 1, wherein the chromatography tube is made of stainless steel.
- 1 7. The apparatus of claim 1, wherein the chromatography port is covered with the
2 corrosion-resistant material.
- 1 8. An apparatus comprising:
2 a chromatography port; and
3 chromatography tubing coupled at one end to the chromatography port, the
4 end of the chromatography tubing having an end face; and
5 a gasket disposed between the end face of the chromatography tubing and the
6 chromatography port, one side of the gasket abutting the end face of the
7 chromatography tubing and the opposite side of the gasket abutting a sealing surface

- 8 of the chromatography port, the gasket being covered with or made of the corrosion-
9 resistant material.
- 1 9. The apparatus of claim 8, wherein the port is covered with a corrosion-resistant
2 material.
- 1 10. The apparatus of claim 8, wherein the end face of the chromatography tube is covered
2 with a corrosion-resistant material.
- 1 11. A tubing assembly comprising a chromatography tube with a bore extending from one
2 end of the tube to an opposite end of the tube, one of the ends of the tube having an
3 end face covered with a corrosion-resistant material.
- 1 12. The tubing assembly of claim 11, further comprising a gasket with one side attached
2 to the end face of the chromatography tube and an opposite side covered with a
3 corrosion-resistant material.
- 1 13. The tubing assembly of claim 11, wherein an inner surface of the tube within the bore
2 near the end face is covered with the corrosion-resistant material.
- 1 14. The tubing assembly of claim 13, wherein an outer surface of the tube near the end
2 face is covered with the corrosion-resistant material.
- 1 15. The tubing assembly of claim 11, wherein the chromatography tube is made of
2 stainless steel.
- 1 16. The tubing assembly of claim 11, wherein the corrosion-resistant material comprises
2 gold.
- 1 17. A liquid chromatography system, comprising:
2 a component with a chromatography port to a fluidic channel; and
3 a tubing assembly with a chromatography tube having a bore extending
4 therethrough, the chromatography tube being coupled at one end to the
5 chromatography port with the bore of the chromatography tube axially aligned with
6 the fluidic channel at the chromatography port, the one end of the chromatography
7 tube having an end face covered with a corrosion-resistant material.

- 1 18. The liquid chromatography system of claim 17, further comprising a gasket attached
2 to the end face of the chromatography tube, the gasket abutting a surface of the
3 chromatography port, the gasket being covered with the corrosion-resistant material.
- 1 19. The liquid chromatography system of claim 17, wherein the corrosion-resistant
2 material comprises gold.
- 1 20. The liquid chromatography system of claim 17, wherein the component is a
2 chromatography column.
- 1 21. The liquid chromatography system of claim 17, wherein the component is an injector
2 valve.
- 1 22. The liquid chromatography system of claim 17, wherein the component is an auxiliary
2 valve.
- 1 23. The liquid chromatography system of claim 17, wherein the chromatography tube is
2 made of stainless steel.
- 1 24. The liquid chromatography system of claim 17, wherein a sealing surface of the
2 chromatography port is covered with a corrosion-resistant material.

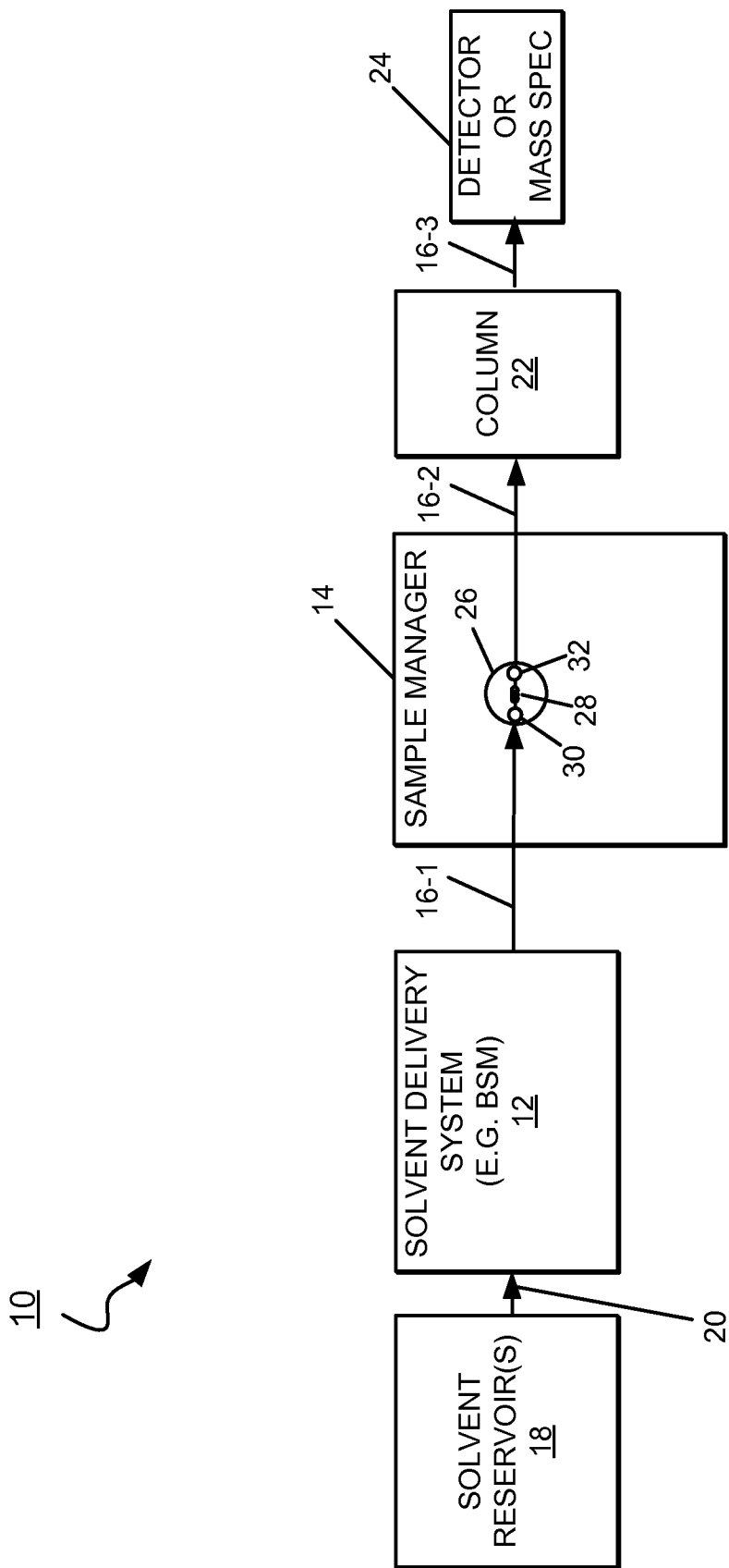


FIG. 1

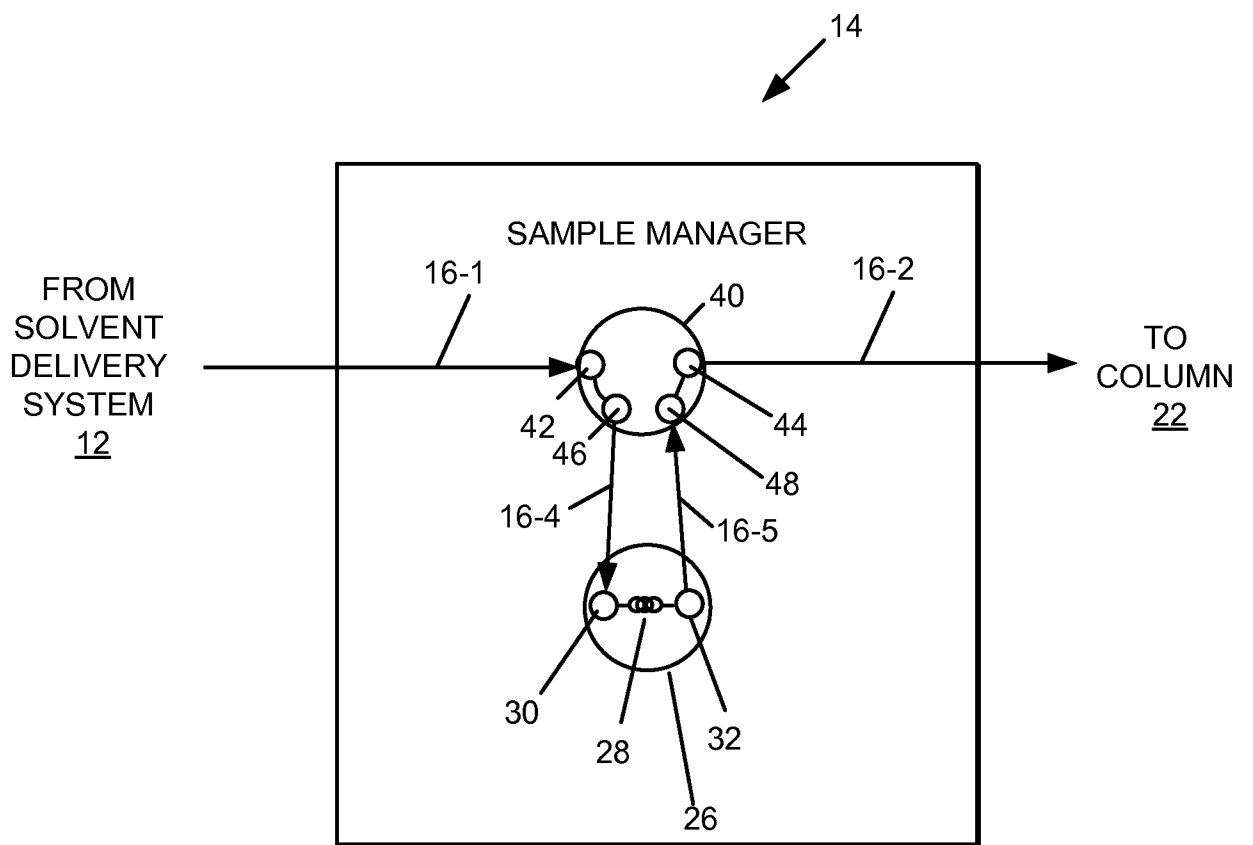


FIG. 2

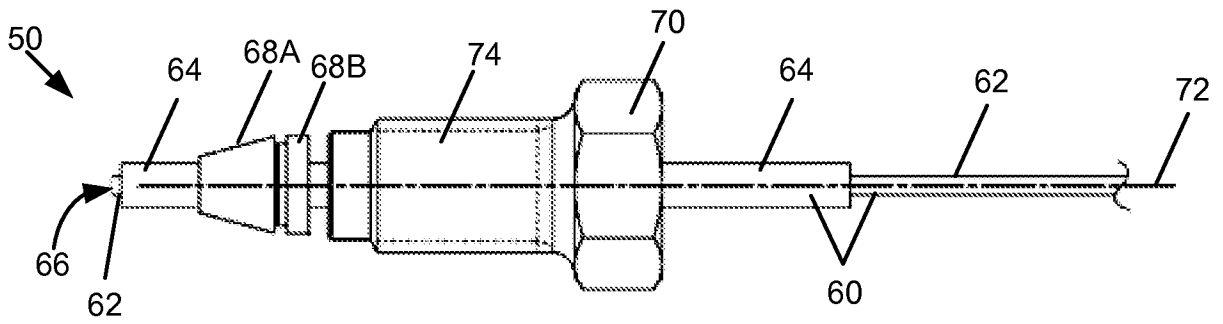


FIG. 3

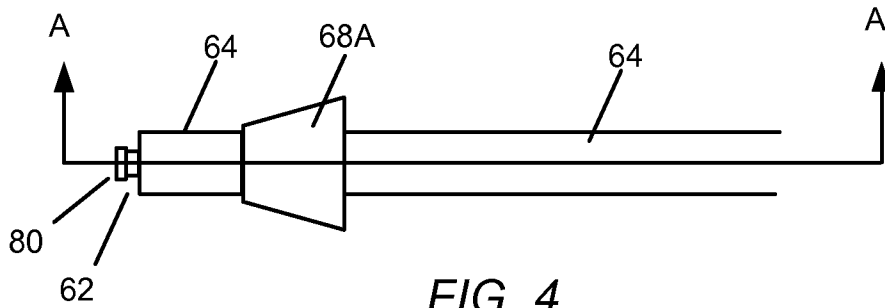


FIG. 4

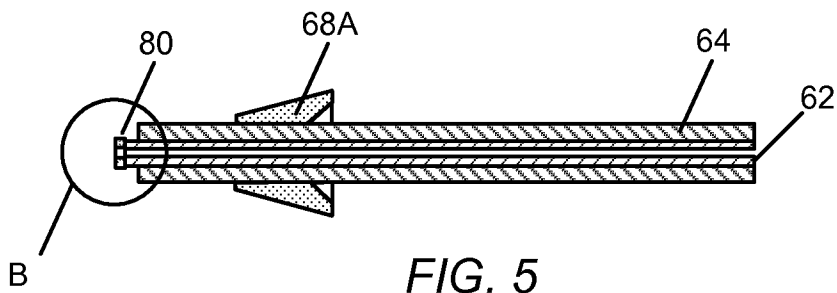


FIG. 5

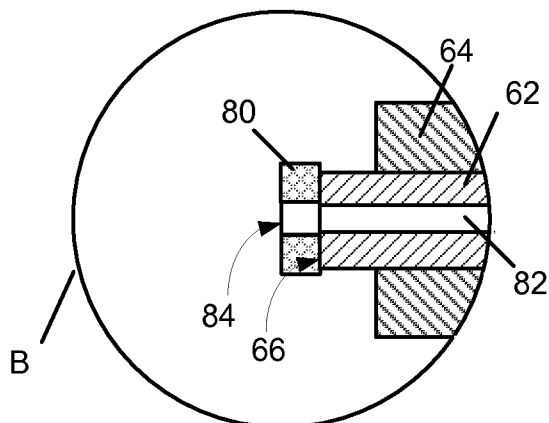


FIG. 6

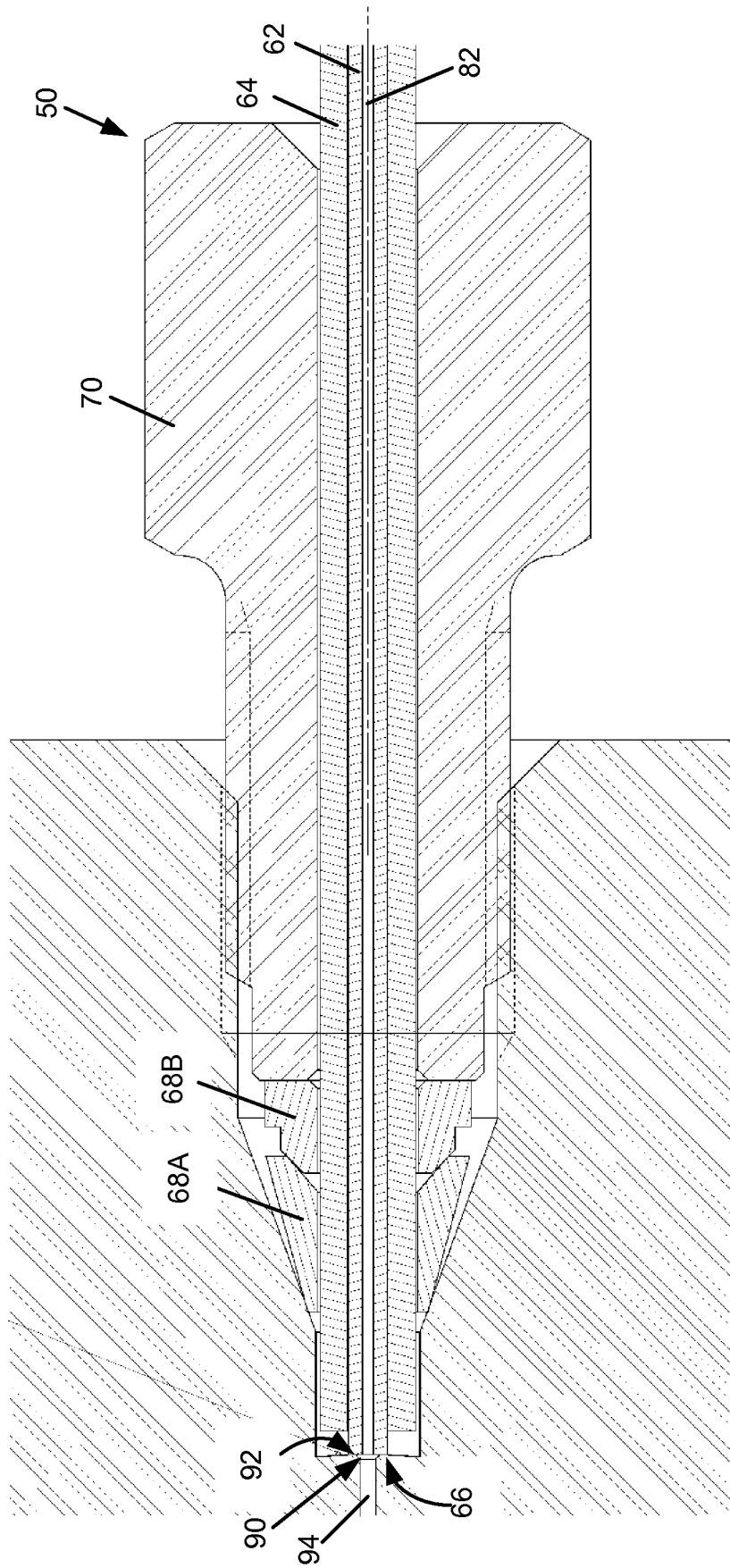


FIG. 7

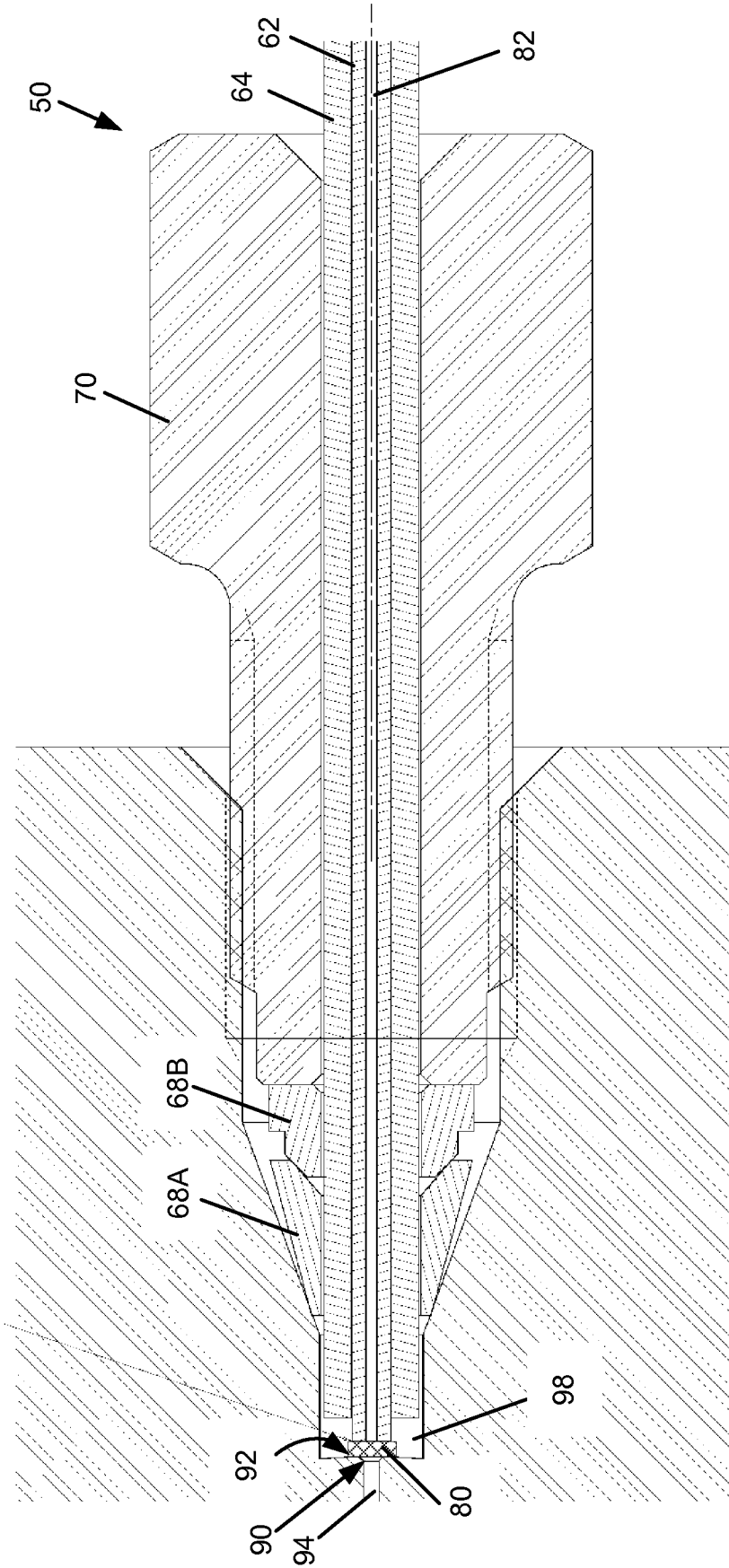


FIG. 8

INTERNATIONAL SEARCH REPORT

International application No.

PCT/US 13/28798

A. CLASSIFICATION OF SUBJECT MATTER

IPC(8) - G01N 30/02 (2013.01)

USPC - 422/70

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

IPC (8) - G01N 30/02

USPC - 422/70

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched
USPC: 251/129.11 (Search terms - See Below)Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)
PatBase and Google. Search terms: chromatograph, port, flange, end, terminus, tube, tubing, column, capillary, corrosion, rust, deteriorate, oxide, resist, oppose, block, fitting, gasket, Teflon, gold, pte and polytetrafluoroethylene

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
Y	US 4,751,004 A (Stevens et al.) 14 June 1988 (14.06.1988) Entire document, especially, Fig. 1 and col 4, ln 27-31; col 8, ln 39-65 and col 9, ln 19-48 and col 10, ln 38-41.	1-24
Y	US 2008/0283458 A1 (Ishii et al.) 20 November 2008 (20.11.2008) Entire document, especially, Fig. 4a, it 2, 15 and 19 Para [0025], [0027], [0028], [0040], [0043], [0052] and [0053].	1-24
A	US 2004/0236083 A1 (Libert et al.) 25 November 2004 (25.11.2004) Entire document, especially para [0005], [0008], [0014], [0016], [0018] and [0019]	1-24

 Further documents are listed in the continuation of Box C.

* Special categories of cited documents:

"A" document defining the general state of the art which is not considered to be of particular relevance

"E" earlier application or patent but published on or after the international filing date

"L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)

"O" document referring to an oral disclosure, use, exhibition or other means

"P" document published prior to the international filing date but later than the priority date claimed

"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention

"X" document of particular relevance: the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone

"Y" document of particular relevance: the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art

"&" document member of the same patent family

Date of the actual completion of the international search

09 April 2013 (09.04.2013)

Date of mailing of the international search report

07 MAY 2013

Name and mailing address of the ISA/US

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