Aug. 16, 1966 E. R. FENSKE ETAL 3,266,321

SAMPLE VALVE

Filed Dec. 28, 1964 2 Sheets-Sheet 1

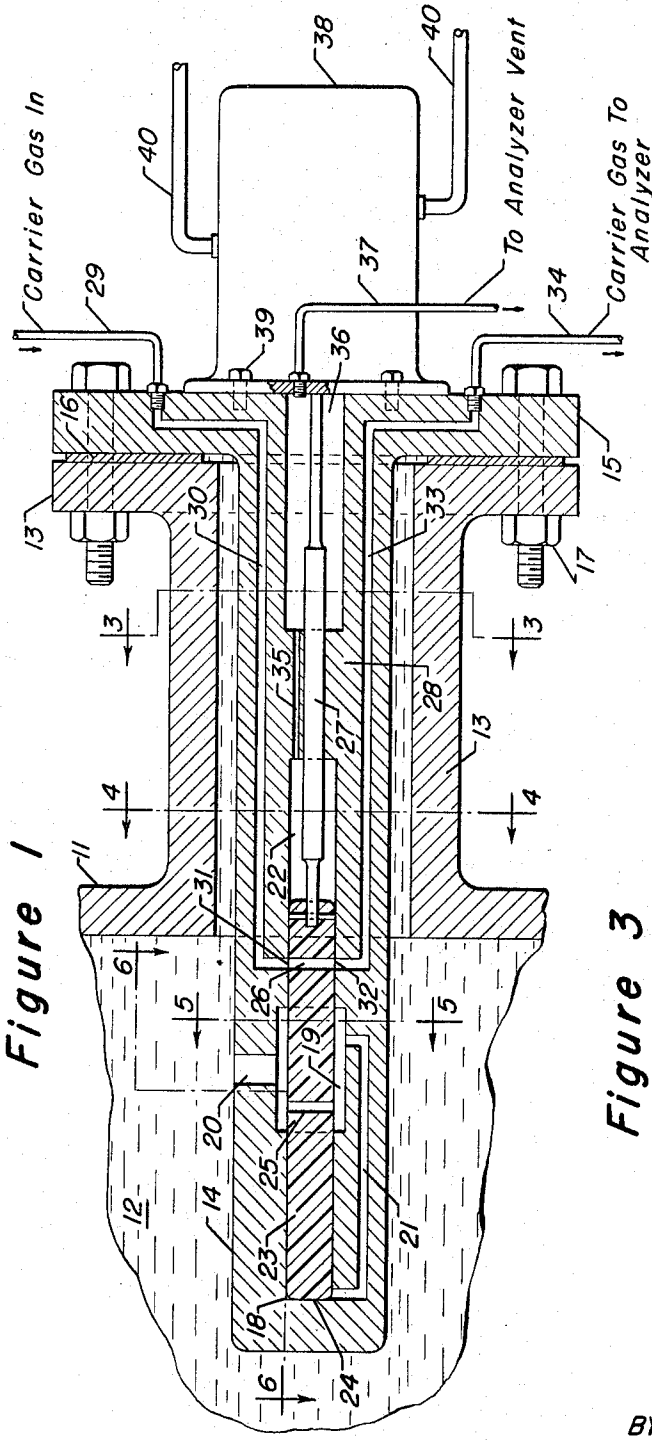



Figure 1

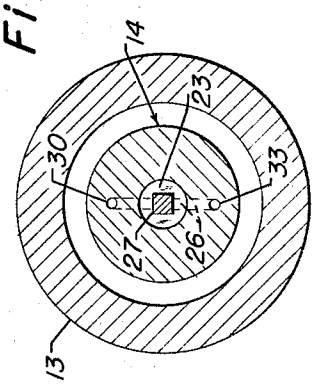



Figure 4

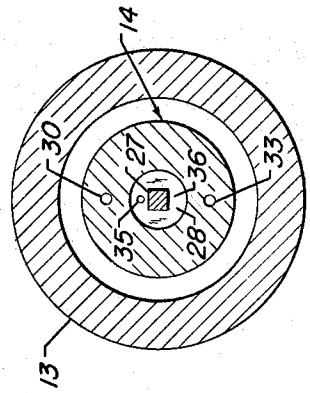



Figure 3

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Figure 2

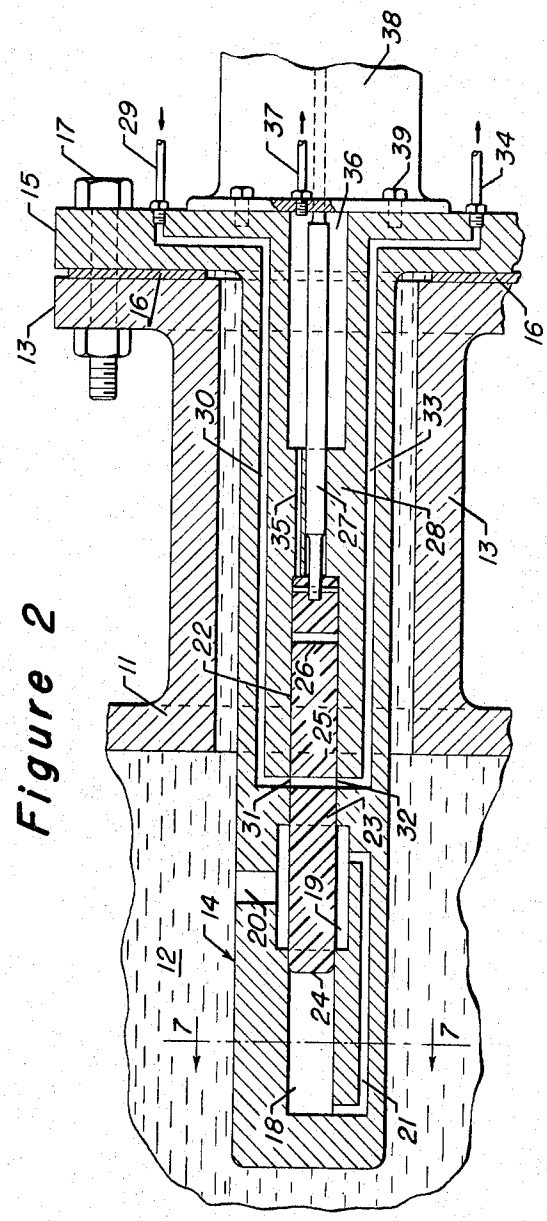



Figure 7

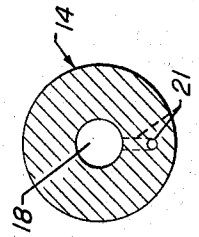



Figure 6

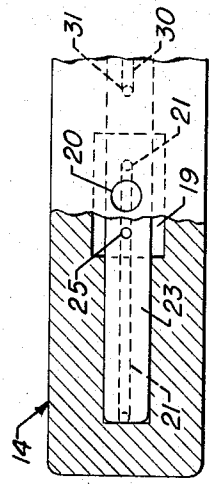
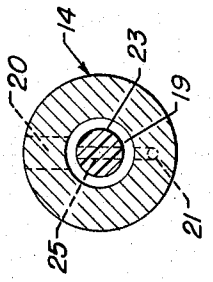



Figure 5




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# SAMPLE VALVE

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This invention relates to a fluid sampling apparatus for use in analysis systems in which repetitive samples of fluid are taken from one fluid stream and introduced into another fluid stream. More particularly, the invention is directed to a sample valve for process stream chromatographs, as well as other types of analyzers requiring periodic injection of fixed volume samples, especially designed for in-line installation.

Sample valves of the prior art, generally comprising a multiported linear reciprocable plug or rotary disc, are conventionally installed within the analyzer case. The analyzer in turn is located at grade, either adjacent to the operating equipment from which the sampled fluid is taken or more remotely in a control house. To bring the process fluid sample to the anlyzer, it is customary to utilize a sample loop, comprising a lengthy run of pipe extending from the sample withdrawal point to the analyzer and thence back to the process. The flow rate through the sample loop is maintained many times the actual quantity required by the analyzer in order to reduce transport lag, a small sample stream being continuously bled from the sample loop at a point contiguous to the analyzer and passed to the sample valve. If a substantial pressure difference is not available across the sample loop, a pump must also be provided to achieve a reasonably high velocity therethrough. Notwithstanding these precautions, such installations still suffer a serious transport lag, or delay time in bringing a representative fluid sample from the process to the analyzer, ranging from a matter of seconds to several minutes or more. If the analyzer is employed to effect closed loop control, the transport lag may be of such magnitude as to present serious stability problems and in all cases will slow down the system response in the event of an upset. In addition, long sample lines accompanied by high frictional loss and/or changes in elevation may result in partial vaporization of a liquid sample stream enroute to the analyzer, with the consequence that its composition will vary erratically and will not be representative of fluid composition at the process sampling point. Another disadvantage of a long sample line is that it is not amenable to vapor samples near dewpoint temperature because of partial condensation during passage to the analyzer. If vapor samples are required, it is necessary to totally condense the sample at the field withdrawal point and transport it to the analyzer as a liquid, a procedure which further increases transport lag.

The present invention is directed to a novel sample valve adapted to be inserted into a processing vessel or conduit from which it is desired to take samples. Carrier fluid is delivered to the valve at point of use and carrier fluid plus sample is withdrawn therefrom. Sampling and flushing lines are completely eliminated as are the above cited disadvantages inhering therein. It is desirable that the sampling valve be close coupled with the analyzer whereby, in the case of a process stream chromatograph, the instrument case or housing containing the chromatographic column, detector block and heater may be bolted to or otherwise unitarily assembled with the sample valve body. Carrier fluid and utilities are supplied directly to this field-mounted apparatus; bridge power and signal leads need be the only connection with remotely located readout or control hardware.



The valve of this invention incorporates a reciprocable plug in combination with a positive displacement action to obviate the possibility of obtaining a stagnant sample. This is provided by a variable volume pocket within the valve body which communicates with the process fluid through the metering port of the valve plug and which is arranged to expand in volume as the plug is stroked towards its sample inject position. The increasing volume produces a suction effect causing a positive flow of sampled fluid through the metering port into the variable volume pocket. The sample trapped in the metering port is thus immediately extracted from the process fluid just prior to injection, so that the sample composition is identical with that of the process fluid at the instant the sample inject command is given. During the plug return stroke to its bypass position, the process fluid within the variable volume pocket is forced back out through the metering port and into the processing vessel to prepare the valve for the next injection command.

Briefly stated, the valve comprises a probe-like casing or stator adapted to extend through an opening of a fluid container and into a body of fluid to be sampled therein. A plug-retaining cavity is formed within the tip portion of the stator and extends longitudinally thereof. The cavity is shaped to define a forward piston chamber, an intermediate sampling chamber and a rear plug guiding bore. A sample intake port is provided in the stator wall connecting with the sampling chamber and affording open fluid communication with the body of process fluid surrounding the stator. A pair of spaced carrier gas ports are formed in the longitudinal wall of the plug guiding bore. A suction conduit connects the piston chamber with the sampling chamber at a point spaced from the sample intake port. A reciprocable plug is slidably disposed within the plug-retaining cavity, said plug having forward and rear adjusted plug positions and being in sliding, substantially fluid-tight contact with the longitudinal walls of the piston chamber and the plug guiding bore. The forward end of the plug cooperates with the piston chamber to form the variable bottom pocket. A metering port extending through the plug cooperates with the sampling chamber to provide a continuous fluid flow path through the metering port from the sample intake port to the suction conduit, as the plug travels from forward to rear adjusted positions, the metering port coming into registry with the carrier gas ports when the plug is in its rear adjusted position.

The invention is more explicitly described hereinbelow in connection with the accompanying drawings in which:

FIGURE 1 is a sectional elevation view of the sample valve in one operating position.

FIGURE 2 is a sectional elevation view of the sample valve in a second operating position.

FIGURE 3 is a transverse view of the valve taken along line 3—3 of FIGURE 1.

FIGURE 4 is a transverse view of the valve taken along line 4—4 of FIGURE 1.

FIGURE 5 is a transverse view of the valve taken along line 5—5 of FIGURE 1.

FIGURE 6 is a sectional plan view of the valve taken along line 6—6 of FIGURE 1.

FIGURE 7 is a transverse view of the valve taken along line 7—7 of FIGURE 2.

With reference to FIGURE 1, there is shown a section of a wall 11 of a fluid container which encloses a process fluid 12. Such process fluid may comprise a relatively fast moving stream or a relatively quiesent zone. The container, for example, may be a pipe run, a processing vessel such as a reactor, fractionating column, liquid-liquid extraction column or fluid-solid adsorption column, storage tank, vapor-liquid separator and the like from which it is desired to withdraw successive samples of a

process fluid. A flanged nozzle **13**, extending outwardly from wall **11**, provides a mounting means for the sample valve. The valve itself comprises an elongated probe-like stator member **14** terminating in an outer mating flange **15**. Flange **15** is held in compression through an annular gasket **16** against the flange of nozzle **13** by throughbolts **17**. The lefthand or tip portion of stator **14** extends well into the body of process fluid **12**.

Formed within the tip portion of stator **14** is a plug-retaining cavity comprising a forward cylindrical piston chamber **18** (FIGURES 2, 7), an intermediate sampling chamber **19** (FIGURES 1, 5), and a rear cylindrical plug guiding bore **22** (FIGURES 1, 4). For the purpose of clarifying the detailed description of the valve as well as the definition thereof in the claims, the locative expression "forward" connotes that portion of the valve which is more closely adjacent to the tip of stator **14**, while the expression "rear" connotes that portion of the valve which is more remote from the tip of stator **14**. A sample intake port **20** in the stator wall provides free fluid communication between process fluid **12** and sampling chamber **19**. A U-shaped channel or passageway **21** connects the forward end of piston chamber **18** with the sampling chamber **19**; its area of intersection with chamber **19** is spaced 180° from intake port **20**. A pair of diametrically spaced carrier gas ports **31**, **32** are formed in the longitudinal wall of plug guiding bore **22**. A suitable carrier gas, such as helium, is supplied to port **31** through tubing **29** and a longitudinal carrier gas supply passage **30**. Carrier gas or carrier gas plus sample are taken from port **32** through a longitudinal carrier gas removal passage **33** and tubing **34** to the process stream analyzer.

Slidably disposed within the plug-retaining cavity is a cylindrical, longitudinally reciprocable plug **23** having a forward piston face **24**, a transverse metering port **25** and a transverse bypass port **26**. Plug **23** is sized so that the forward and rear portions thereof are continuously maintained in sliding but substantially fluid-tight contact with the longitudinal walls respectively of piston chamber **18** and plug guiding bore **22**. Plug **23** has a forward adjusted position in which bypass port **26** registers with ports **31**, **32** and a rear adjusted position in which metering port **25** registers with ports **31**, **32**.

Plug **23** is stroked by means of a square shanked shaft **27** keyed thereto. Shaft **27** is supported by a guide sleeve **28** which also prevents rotation of the shaft and plug. Shaft **27** extends through an outer shaft bore **36** to connect with a pneumatic operator **38**. Operator **38**, secured to the face of flange **15** by machine screws **39**, may be of the double diaphragm type actuated by instrument air delivered through lines **40**. Lines **40** are alternately pressured and vented through programmed solenoid valves in the usual manner so that shaft **27** is driven back and forth in response to appropriate electrical commands.

To accommodate the small but practically unavoidable leakage of process fluid past the plug, a pressure relief vent **35**, drilled through shaft guide sleeve **28**, allows process fluid to escape into outer shaft bore **36** which in turn is vented via tubing **37** to the main analyzer vent or other suitable sink. This arrangement prevents accumulation of process fluid within bore **22** which would otherwise impede rearward movement of plug **23** or create a potential explosion hazard.

In FIGURE 1 plug **23** is shown occupying its forward or bypass position wherein bypass port **26** is aligned with carrier gas ports **31** and **32**. Piston end **24** of the plug preferably abuts flush against the forward end of piston chamber **18** so that the forward part of the plug occupies substantially the entire volume of the piston chamber; however, in some instances it may be desirable to space the piston end of the plug from the end wall of the piston chamber to provide a small clearance pocket. In FIGURE 2 plug **23** is shown occupying its rear or sample inject position wherein metering port **25** is aligned with carrier gas ports **31** and **32**. Since the diameter of sampling chamber **19** is slightly greater than that of the plug, it will be seen that as the plug is moved from forward to rear positions, metering port **25** in moving thereacross provides a fluid flow path across the sampling chamber from intake port **20** to passageway **21** for a portion of the plug travel equivalent to the length of chamber **19**. At the same time the volume of chamber **18** is increasing which induces a suction effect through passageway **21**, drawing in process fluid through port **20**, metering port **25** and passageway **21** to chamber **18**. The operative elements are preferably so proportioned that the effective volumetric displacement, or increase in volume of chamber **18** during the time that metering port **25** is within chamber **19**, is greater than the volume of the metering port, preferably 5 to 1000 times the metering port volume. For example, typical metering volumes may range from 1 to 5 microliters, while the corresponding effective volumetric displacement of piston chamber **18** may range from 5 microliters to 5 milliliters. In fact, preferred design considerations indicate that the combined free volume of sampling chamber **19**, metering port **25** and suction passageway **21** should be much less than the effective volumetric displacement of the piston chamber. The objective here is to insure an adequate flushing flow through the metering port to sweep out any residual material from the preceding injection stroke and to provide a trapped sample whose composition is identical with that of the process fluid at the time of injection. When metering port **25** is moved into alignment with carrier gas ports **31** and **32**, the sample trapped therein is swept out and conducted to the process stream analyzer. Upon the return stroke of the plug to its forward adjusted position, the process fluid within chamber **18** is forced back out through passageway **21**, metering port **25** and intake port **20**, and the apparatus is prepared for the next sample injection command.

The materials of construction may be any of those generally employed by those skilled in the valve arts. The stator, for example, may be fabricated of carbon steel, stainless steel, brass, bronze and the like. Similarly the plug may be fabricated of carbon steel, stainless steel, brass, bronze or a solid polyfluoroethylene resin such as Teflon or Kel-F; also, the plug may be a Teflon-coated metal block. Polyfluoroethylene resins are well known, of course, for their low coefficient of friction and good thermal stability characteristics which make them ideally suited for use in valve members having sliding bearing surfaces.

In connection with the configuration of the sampling chamber **19**, as specifically disclosed by FIGURES 1, 5 and 6, it will be seen that the annular space **19** permits some of the process fluid to bypass the metering port during the injection stroke. However, it may be advantageous in some instances to arrange the geometry thereof so that the total flow of process fluid is necessarily forced through the metering port. This can be accomplished by making the diameter of the sampling chamber the same as that of the piston chamber and plug guiding bore, and providing longitudinal grooves at the top and bottom of the sampling chamber, one groove connecting with the intake port and overlying the metering port of the plug, and the other groove connecting with the suction passageway and overlying the opposite end of said metering port.

If the fluid is substantially incompressible, some passage from chamber **18** to port **20** must be provided at all times when the plug **23** is in motion. One way to do this is to drill another bypass port in the forward end of the plug.

Many other variations of and substitutions in the above specifically described apparatus will be apparent to those skilled in the art and are deemed within the scope of the claims. For example, the plug may have a shape other than cylindrical: square, rectangular or polygonal cross sections may be provided. If the plug is square or rec-

tangular, the shaft can be cylindrical. Similarly the exterior contour of the probular stator may be other than circular such as tapered, square and the like. Passageways **21**, **30** and **33** have been depicted as ducts wholly formed within the stator and, although such arrangement is desirable for the sake of appearance and unitary construction, it is nevertheless within the scope of the invention to substitute, for these internal passageways, exteriorly disposed conduits such as copper or stainless steel tubing. In addition, heating means such as steam coils or electric resistance elements may be embedded in or wrapped around the stator between the carrier gas ports and the outer flange member to provide a means for vaporizing the sample before it reaches the analyzer. Other types of valve operators may be utilized such as, for example, those incorporating a single diaphragm with spring return and those utilizing the solenoid principle.

It is also within the purview of this invention to eliminate the bypass port of the plug. The purpose of the bypass port is to insure continuity of carrier gas flow for chromatographic analysis. However, such flow may alternatively be provided by an external valve programmed to open and close at the proper time; and for certain other types of batch analyses, a continuous flow of carrier medium is not required.

It will be understood that the embodiment herein described is simple and uncomplicated, the drawings illustrating generally the principal features of novelty. Taking into account the desirability of access to the valve internals, especially for repairing and replacing the plug, it will be feasible to construct the valve in two mating sections, cutting it along line **4—4** of FIGURE 1, for example, and assembling the two sections with a union fitting. If onstream maintenance or replacement of the entire valve is anticipated, the simple flanged nozzle supporting member may be replaced with a double block valve lock seal arrangement to permit removal of the sampling valve without the necessity of shutting down the process.

It has previously been indicated that the invention may be utilized to maximum advantage when it is close coupled to the analyzer receiving the successive samples of process fluid. This may readily be accomplished, for example, by bolting the analyzer housing to the outer face of flange **15**, which housing may enclose the sample valve operator as well as the other operative elements of the analyzer including, for example, the chromatographic column, detector block, sample vaporizer and thermostatically controlled heating means for the analyzer as a whole. Carrier fluid, instrument air for actuating the valve operator and electric power may be supplied directly to this field-mounted apparatus, while detector bridge power and signal leads may be run through electrical conduit to a remotely located indicator, recorder and/or controller.

We claim as our invention:

**1.** A fluid sampling valve comprising a probe-like stator member adapted to extend through an opening of a fluid container and into a body of fluid to be sampled therein; a plug-retaining cavity formed within the tip portion of said stator and extending longitudinally thereof, said cavity defining a forward piston chamber, an intermediate sampling chamber and a rear plug guiding bore; a sample intake port through the stator wall connecting with said sampling chamber and providing open fluid communication with said body of fluid; a pair of spaced carrier gas ports formed in the longitudinal wall of said plug guiding bore; suction conduit means connecting said piston chamber with the sampling chamber at a point spaced from said intake port; a reciprocable plug within said cavity having forward and rear adjusted plug positions and being in sliding, substantially fluid-tight contact with the longitudinal walls respectively of said piston chamber and said plug guiding bore; and a metering port extending through said plug located to provide a fluid flow path therethrough and across said sampling chamber from the intake port to the suction conduit during a portion of the plug travel from forward to rear adjusted positions, and to register with said carrier gas ports when the plug is in its rear adjusted position.

**2.** A fluid sampling valve comprising a probe-like stator member adapted to extend through an opening of a fluid container and into a body of fluid to be sampled therein; a plug-retaining cavity formed within the tip portion of said stator and extending longitudinally thereof, said cavity defining a forward piston chamber, an intermediate sampling chamber and a rear plug guiding bore; a sample intake port through the stator wall connecting with said sampling chamber and providing open fluid communication with said body of fluid; a pair of spaced carrier gas ports formed in the longitudinal wall of said plug guiding bore; suction conduit means connecting said piston chamber with the sampling chamber at a point spaced from said intake port; a reciprocable plug within said cavity having forward and rear adjusted plug positions and being in sliding, substantially fluid-tight contact with the longitudinal walls respectively of said piston chamber and said plug guiding bore, the forward end of the plug coacting with the piston chamber to form a variable volume pocket; and a metering port extending through said plug and cooperating with said sampling chamber to provide a continuous fluid flow path through the metering port from the intake port to the suction conduit, as the plug travels from forward to rear adjusted positions, for a plug displacement producing a corresponding increase in said pocket volume greater than the volume of the metering port, said metering port registering with said carrier gas ports when the plug is in its rear adjusted position.

**3.** The valve of claim **2** further characterized in that said suction conduit means connects with the piston chamber at the forward end of the latter.

**4.** The valve of claim **3** further characterized in that the forward part of said plug occupies substantially the entire volume of the piston chamber when the plug is in its forward adjusted position.

**5.** The valve of claim **2** further characterized in that a bypass port extends through said plug and registers with said carrier gas ports when the plug is in its forward adjusted position.

**6.** The valve of claim **2** further characterized in that said suction conduit means is a passageway wholly contained within the stator member.

**7.** The valve of claim **2** further characterized in the provision of carrier gas supply and removal conduits connecting with said carrier gas ports.

**8.** The valve of claim **7** further characterized in that said carrier gas supply and removal conduits are longitudinal passageways wholly contained within the stator member.

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LOUIS R. PRINCE, *Primary Examiner.*